PRACTICAL PHARMACY

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AN ACCOUNT OF THE

METHODS OF MANUFACTURING AND DISPENSING
PHARMACEUTICAL PREPARATIONS

WITH

A CHAPTER ON THE ANALYSIS OF URINE

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OF GREAT BRITAIN

WITH TWO HUNDRED AND EIGHTY-THREE ILLUSTRATIONS

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PREFACE

The intention of the author has been to write a book for students, which, while not going deeply into the details of operations that can only be conducted on a manufacturing scale, would yet afford such information as pharmacists are expected to possess. No attempt has been made to transfer the substance of the matter of the Pharmacopœia to these pages. The chemical substances are scarcely more than touched upon, as lengthy treatment would not only have enormously increased the size of the book, but would also have been out of place in a work dealing with pharmacy.

Considerable prominence has been assigned to pharmacopœial tests with explanations of the reactions involved, as well as to volumetric and gravimetric analysis, for it becomes increasingly necessary for pharmacists to examine and value for themselves the materials and preparations they are required to handle. The author makes no apology for the introduction of a chapter on urine analysis, as he believes that in the future, pharmacists will be called upon to undertake not only examinations of urine, but possibly many other operations of a similar character.

The author has to express his acknowledgments to Mr. J. C. Umney for much information concerning essential oils, and to Mr. J. Hamerton for valuable assistance in proof-reading.

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ILLUSTRATIONS

In a book of this description it would have been impracticable to prepare fresh illustrations of all the apparatus described. The author has, therefore, drawn largely upon his friends, and a list is appended of those who have kindly placed blocks, *many of them copyright,* at his disposal. To these and to Professor Clowes, who has allowed a number of illustrations to be taken from his work on Quantitative Analysis, the author expresses his best thanks.

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Mr. William Toogood, London.
Messrs. Werner, Pfleiderer & Perkins, London.
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The York Glass Company, York.

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PART I

GENERAL PROCESSES AND DESCRIPTIONS OF APPARATUS

INTRODUCTION

PHARMACY (*pharmakon*, a drug) is the art of identifying, preparing, and dispensing medicinal substances.

A pharmacopœia (*pharmakon*, a drug; and *poieo*, I make) is a dispensatory or book of directions published by authority, which defines and fixes the standards of purity of materials used in medicine; it also describes the physical and chemical properties of these materials, their methods of preparation, and their means of identification.

Table of Pharmacopœias.

Country.	Title.	Language.	Date.
Austria	Pharmacopœa Austriaca	Latin	1889
Belgium	Pharmacopœa Belgica	Latin and French	1885
Chili	Farmacopœa Chilena	Spanish	1886
Denmark	Pharmacopœa Danica	Danish	1893
Finland	Pharmacopœa Fennica	Latin	1885
France	Codex Medicamentarius; Pharmacopée Française	French	1884 (add. 1895)
Germany	Pharmacopœa Germanica; Arzneibuch für das Deutsch Reich.	German	1890 (add. 1895)
Great Britain	British Pharmacopœia	English	1898
Greece	*Ελληνικὴ Φαρμακοποιΐα*	Latin and Greek	1868
Hungary	Pharmacopœa Hungarica	Latin and Hungarian	1888
India	Pharmacopœia of India	English	1868
Italy	Farmacopœa ufficiale del regno d' Italia	Italian	1892
Japan	Pharmacopœa Japonica	Latin	1891
Mexico	Nueva Farmacopœa Mexicana	Spanish	1884
Netherlands	Pharmacopœa Neerlandica	Latin and Dutch	1889
Norway	Pharmacopœa Norvegica	Norwegian	1894
Portugal	Pharmacopœa Portugueza	Portuguese	1876
Russia	Rossüskaja Pharmakopeya	Russian	1891
Spain	Pharmacopœa official Española	Spanish	1884
Sweden	Pharmacopœa Suecica	Swedish	1894
Switzerland	Pharmacopœa Helvetica	German, French, Italian	1894
United States	Pharmacopœia of the United States of America	English	1893

In earlier times the pharmacopœias of many countries were only published in Latin, but the tendency of later years has been to print the titles in Latin and the text in the vulgar tongue, as exemplified in the British Pharmacopœia. In this the arrangement is as follows:

1. The official Latin title.
2. The English title.
3. The official synonym (if any).
4. The chemical formula.
5. A short description or definition.
6. The characters and tests.

CHAPTER I

WEIGHTS AND MEASURES *

The imperial system of weights and measures is based on two arbitrarily fixed units,—the *yard measure,* the unit of length; and the *pound weight,* the unit of mass. These standards, from which all others are derived, are deposited at the Standard Office, Old Palace Yard, Westminster.

The *yard* is the straight line or distance between the centres of two gold plugs recessed in a bronze bar, measured at a temperature of 62° F. To avoid flexure, and at the same time facilitate free expansion and contraction from variations of temperature, the bar is supported on bronze rollers. By the Weights and Measures Act of 1878 this measure is declared to be the only unit or standard measure of extension, and all other measures of extension, whether linear, superficial, or solid, must be derived from it.

One third part of the imperial standard yard is called a *foot,* and one twelfth part of the foot an *inch.*

The *pound* is the weight *in vacuo* of a platinum cylinder, and from this unit all measures having reference to weight must be derived. One sixteenth part of the imperial standard *pound* is called an *ounce,* and one sixteenth part of such an ounce a *dram,* and one seven-thousandth part of the imperial standard pound a *grain. These weights are called avoirdupois weights.*

The unit or standard measure of capacity is the imperial *gallon,* containing ten imperial pounds weight of distilled water weighed in air against brass weights, the water and air both being

---

* *Yard,* from the Anglo-Saxon *gyrd,* a rod.
Inch, from the Anglo-Saxon *ynce,* or the Latin *uncia,* a twelfth part.
Pound, from the Anglo-Saxon *pund,* or the Latin *pundus,* a weight.
Grain, from the Latin *granum,* a seed.
Quart, from the Latin *quartus,* a fourth.
Pint, from *picta,* painted on, or marked, as the ancient measuring vessels.

at a temperature of 62° F., and the barometer standing at 30 inches. The *quart* is the one fourth part, and the *pint* the one eighth part of the imperial standard gallon.

By the Weights and Measures Act of 1878 it was made unlawful to use for trade purposes any weight or measure other than those derived from the imperial standards. The following exceptions were, however, made:—Drugs when sold by retail (dispensed) being allowed to be vended by *apothecaries'* weight, and precious metals and precious stones by *troy* weight.

In the spring of 1897 a bill legalising the use of *weights and measures of the metric system* in trade came into operation, and standards derived from the *metre-international* and *kilogram-international* were deposited with the Board of Trade. These standards, which assure the invariability and accuracy of metric weights and measures throughout the civilised world, were adopted by a committee of representatives from the twenty-one states belonging to the convention.

The unit of length, the *metre-international*, is the length (measured at 0° C.) of an iridio-platinum bar deposited with the Board of Trade. The present legal equivalent of the metre is 39·370113 inches. If a brass metre is, however, compared *not at its legal temperature* (0° C. = 32° F.), but at the temperature of 62° F., with a brass yard also at 62° F., then the apparent equivalent of the metre would be nearly 39·382 inches.

The unit of weight, the *kilogram-international,* is the weight *in vacuo* of an iridio-platinum cylinder deposited with the Board of Trade. The *litre* contains one kilogram weight of distilled water at its maximum density (4° C.), the barometer registering 760 millimetres.

It will be noticed that all the standards mentioned are purely arbitrary. Thus the yard measure is no longer required as formerly to be restored, if lost, by reference to the length of a pendulum vibrating seconds of mean time in the latitude of Greenwich at sea level, nor the pound to be restored by computation from the weight of a cubic inch of distilled water at 62° F. Neither is the theoretical metre any longer regarded as the one ten-millionth part of the elliptic quadrant of the meridian passing through Paris, nor the kilogram as the weight of distilled water contained in a cubic decimetre. The metre-international has, however, been found to contain 1,553,163·5 wave-lengths of the red ray of the spectrum of cadmium measured in air at 15° C., and under an atmospheric pressure of 760 mm., so that the length of the metre bar at Paris could be redetermined, if necessary, to within a micron. The determination of standards from natural constants has thus given way to material standards constructed by hand as being more accurate and desirable.

The passing of the Weights and Measures (Metric System) Bill of 1897 has proved of immense advantage to pharmacists, as prior to that date it was illegal to use any metric weight or measure in dispensing foreign prescriptions, or in filling orders for or making contracts with countries using the above system. True, their use was not interfered with for purposes of scientific research, or for determinations found necessary in manufacturing processes, but for purposes of trade they were absolutely forbidden. In their stead a table was given which set forth the equivalents of imperial weights and measures, and of the metric weights and measures, and all trade operations involving the use of the latter system were required to be performed by the use of the equivalents.

As it would obviously be undesirable that the original standards should be in every-day use for purposes of comparison, the Board of Trade are provided with secondary standards consisting of exact copies of the imperial ones, as well as with standards of all denominations of weights and measures which are either multiples or aliquot parts of the same.

Every weight and measure used in trade is required to have its denomination stamped upon it in legible figures and letters, in addition to an inspector's stamp of verification. Wire weights below half a scruple need not be so marked.

DENOMINATION OF STANDARD WEIGHTS AND MEASURES IN COMMON USE IN PHARMACY UNDER THE DIRECTION OF THE BOARD OF TRADE

Apothecaries' Weight

Denomination.	Weight in grains in terms of the imperial standard pound, which contains 7000 such grains.
OUNCES:	
10 ounces	4800 grains.
8 „	3840 „
6 „	2880 „
4 „	1920 „
2 „	960 „
1 ounce...	480 „

Denomination.	Weight in grains in terms of the imperial standard pound, which contains 7000 such grains.	Abbreviations allowed.
DRACHMS:		
4 drachms, or half an ounce	240 grains	ʒiv.
2 ,, ...	120 ,,	ʒij.
1 drachm	60 ,,	ʒj.
SCRUPLES:		
2 scruples	40 ,,	℈ij.
1½ ,, or half a drachm	30 ,,	ʒss.
1 scruple	20 ,,	℈j.
Half a scruple	10 ,,	℈ss.
GRAINS:		
6 grains	6 ,,	
5 ,, ...	5 ,,	
4 ,, ...	4 ,,	
3 ,, ...	3 ,,	
2 ,, ...	2 ,,	
1 grain	1 grain.	
Half a grain	0·5 ,,	

Apothecaries' Measure

Denomination.	Containing the following weight of distilled water: Temperature = 62° Fahrenheit. Barometer = 30 inches. Imperial pound = 7000 grains.
A fluid ounce and the multiples thereof from 1 to 40 fluid ounces Half a fluid ounce	One fluid ounce contains 437·5 grains weight, or $\frac{1}{160}$ of the imperial gallon.
A fluid drachm and the multiples thereof from 1 to 16 fluid drachms Half a fluid drachm	One fluid drachm = $\frac{1}{8}$ fluid ounce, = 54·6875 grains of water.
A minim and the multiples thereof from 1 to 60 minims	One minim = $\frac{1}{60}$ fluid drachm, = ·9114583 grain of water.

Avoirdupois Weight

Denomination.	Abbreviation or symbol allowed.
56 pounds, or half hundredweight	56 lbs., or ½ cwt.
28 ,, or quarter hundredweight	28 ,, or ¼ ,,
14 ,, or stone	14 ,,
7 ,, ...	7 ,,
4 ,, ...	4 ,,
2 ,, ...	2 ,,
1 pound, or 7000 grains	1 lb.
8 ounces, or half pound	8 oz., or ½ lb.
4 ,, or quarter pound	4 ,, or ¼ ,,
2 ,, ...	2 ,,
1 ounce, or 437½ grains	1 ,,
8 drams, or half ounce	8 drs., or ½ oz.
4 ,, ...	4 ,, or ¼ ,,
2 ,, ...	2 ,,
1 dram	1 dr.
½ ,, ...	½ ,,

Grains Weight

Denomination.	Abbreviation allowed.
4000 grains	4000 gr.
2000 „	2000 „
1000 „	1000 „
500 „	500 „
300 „	300 „
200 „	200 „
100 „	100 „
50 to 0·01 grain	50 to 0·01 gr.

Troy Weight

Decimal troy ounce *bullion* weights are legal from 500 ounces troy down to 0·001 ounce troy, but these weights are rarely, if ever, employed in pharmacy. Apothecaries' weights are often incorrectly termed troy weights.

Imperial Measure

Denomination.	Abbreviation.	
1 gallon	1 gall.	Cj*
½ „	½ „	Css
1 quart	1 qt.	Oij
1 pint	1 pt.	Oj
½ „	½ pt.	Oss
1 gill	gill.	

A cubic inch of water at 62° F., free from air and weighed in air against brass weights of the density 8·143, with the barometer at 30 inches, weighs 252·286 grains, and is the value adopted in an Order in Council dated November 28th, 1889.

A cubic foot of water, free from air, under precisely the same conditions, weighs 62·278601 lbs.; that is to say, a cubic foot of water measures very nearly 6¼ gallons.

To convert pounds troy into pounds avoirdupois × ·822857.
„ „ avoirdupois into pounds troy × 1·21528.

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* The sign for the gallon, Cj, is derived from the Latin *congius*, a Roman measure containing 6 sextarii. The sign for the pint, Oj, is derived from *octarius*, a coined Latin word. The Roman sextarius was the sixth part of the congius, but as our gallon is divided into 8 parts, it became necessary to insert a word to meet the case.

Metric Weights and Measures, with their legal Equivalents

1 millimetre (mm.) ($\frac{1}{1000}$ metre), ·001 ... = 0·03937 inch.
1 centimetre ($\frac{1}{100}$ metre), 0·01 ... ... = 0·3937 ,,
1 decimetre ($\frac{1}{10}$ metre), 0·1 ... ... = 3·937 inches.
1 metre, 1·0 ... ... = 39·370113 ,,

1 cubic centimetre (c.c.) ... ... = 16·9315 minims.

1 litre (or 1000 cubic centimetres) ... = 1·7598 pints.

1 milligram (mgr.), 0·001 ... ... = 0·01543235639 grain.
1 centigram ($\frac{1}{100}$ gram), 0·01 ... ... = 0·1543235639 ,,
1 decigram ($\frac{1}{10}$ gram), 0·1 ... ... = 1·543235639 grains.
1 gram, 1·00... ... ... = 15·43235639 ,,

1 kilogram ... ... – 15432·35639 grains.

1 inch... ... ... = 25·39997 millimetres.
1 foot (12 inches) ... ... = 0·30479967 metre.
1 yard (3 feet) ... ... = 0·914399 ,,

1 gallon (160 fl. ounces) ... ... = 4·5459631 litres.
1 fluid ounce (℥j) ... ... = 28·4123 cubic centimetres.
1 ,, drachm (ʒj) ... ... = 3·552 ,,
1 minim (♏j) = (·91146 grain) ... ... = 0·05919 cubic centimetre.

1 grain ... ... = ·064798918 gram.
1 ounce avoirdupois (437·5 grains) ... = 28·34953 grams.
1 pound (7000 grains) ... ... = 0·45359243 kilogram.
1 hundredweight (1 cwt.) ... ... = 50·80235216 kilograms.

1 ounce, apothecaries' weight (480 grains) = 31·1034806 grams.
1 drachm ,, ,, (60 grains)... = 3·887935 ,,
1 scruple ,, ,, (20 grains)... = 1·2959783 ,,
1 grain ,, ,, ... = 64·798918 milligrams.

The following are *approximate equivalents,* and may be used for rough calculations.

Length.

1 millimetre (·001) = $\frac{1}{25}$ inch approx.
1 centimetre (·01) = $\frac{2}{5}$,,
1 decimetre (·1) = 4 inches ,,
1 metre = 3·28 feet. ,,

Weight.

1 gram = 15½ grains approx.
1 kilogram = 2$\frac{1}{5}$ pounds ,,

Capacity.

1 cubic centimetre = 15 minims approx.
1 litre = 1¾ pints ,, or 61 cubic inches approx.

Tables of Errors tolerated in Weights and Measures

Errors tolerated in avoirdupois weight.	Allowance in grains ; heavy, or in excess only.	
	Iron weights.	Brass weights.
56 lbs.	50 grains	20 grains.
28 ,,	40 ,,	15 ,,
14 ,,	30 ,,	10 ,,
7 ,,	20 ,,	5 ,,
4 ,,	20 ,,	5 ,,
2 ,,	10 ,,	5 ,,
1 lb.	10 ,,	2 ,,
8 oz.	5 ,,	2 ,,
4 ,,	5 ,,	2 ,,
2 ,,	—	1 grain.
1 ,, and under	—	1 ,,

Errors tolerated in apothecaries' weight.	Allowance in grains ; heavy, or in excess only.
10 ounces	0·5 or ½ grain.
8 ,,	
6 ,,	
4 ,,	0·2 or $\frac{2}{10}$,,
2 ,,	
1 ounce	
4 drachms	0·1 or $\frac{1}{10}$,,
2 ,,	
1 drachm	
2 scruples	0·05 or $\frac{5}{100}$,,
1½ ,,	
1 scruple	
½ ,,	
6 grains	0·02 or $\frac{2}{100}$,,
5 ,,	
4 ,,	
3 ,,	
2 ,,	0·01 or $\frac{1}{100}$,,
1 grain	
½ ,,	

Errors tolerated in Apothecaries' Graduated Glass Measures

Approximate internal diameter of measure.	Error allowed in excess or in deficiency.
4 inches	25 minims.
3½ ,,	20 ,,
3 ,,	18 ,,
2½ ,,	14 ,,
2 ,,	10 ,,
1¾ ,,	8 ,,
1½ ,,	6 ,,
1¼ ,,	5 ,,
1 inch	4 ,,
⅞ ,,	3 ,,
¾ ,,	2 ,,
⅝ ,,	1 minim.
½ ,,	½ ,,

Errors tolerated in larger Measures of Capacity

	Largest error permitted, in excess only. Liquid measures.
Gallon	2 fluid ounces.
Half gallon...	1 fluid ounce.
Quart	1 ,,
Pint	4 fluid drachms.
Half pint	3 ,,
Gill	2 ,,

CHAPTER II

WEIGHING AND MEASURING

The balance.—The balance is an instrument for determining the relative weight of bodies; in other words, the weight of a body is the measure of its gravitating force.

The ordinary prescription balance, Fig. 1, consists of a lever of the first kind, with its fulcrum in the middle. This lever is called the beam. At the extremities of the beam are suspended two pans: the one on the right generally made of glass, and

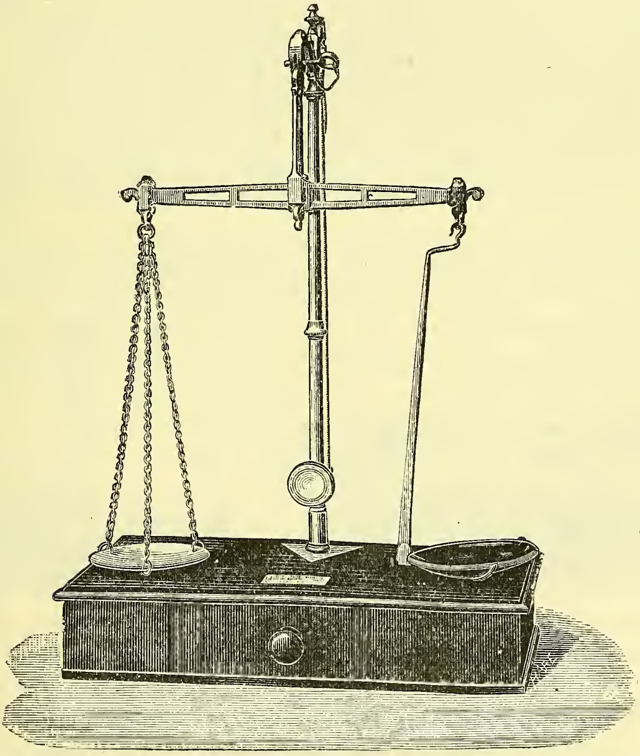

FIG. 1.

intended to receive the substance to be weighed ; the one on the left, the weights. The fulcrum consists of a hardened steel prism called the *knife-edge,* which passes through the beam at right angles, and rests with its sharp edge or axis of suspension upon two supports made in hardened steel to diminish friction.

Since two equal forces in a lever of the first kind cannot be in equilibrium unless their leverages are equal, the length of the arms ought to remain equal during the process of weighing. To secure this the pans are suspended from hooks, each of which is dependent on a knife-edge enclosed in the box ends of the beam. By this means the pans are suspended from mere points, which are practically unmoved during the oscillations of the beam.

When a good balance is placed on a level plane it ought to satisfy the following conditions :

1. *The two arms of the balance ought to be equal ;* otherwise, according to the principle of the lever, unequal weights will be required to produce equilibrium. To test whether the arms of the balance are actually equal, weights are placed in either pan until the beam becomes horizontal : the contents of the pans are then interchanged, when if the arms are equal, the beam will remain horizontal ; but if not, it will descend on the side of the longer arm.

2. *The balance ought to be in equilibrium when the pans are empty,* for otherwise unequal weights must be placed in the pans to produce equilibrium. It must be borne in mind, however, that the arms are not necessarily equal even if the beam remains horizontal when the pans are empty, for this result might be brought about by giving the longer arm the lighter pan.

3. *The beam being horizontal, its centre of gravity ought to be in the same vertical line with the edge of the fulcrum, and a little below the latter,* otherwise the beam would not be in stable equilibrium. Further, the beam should be perfectly rigid, of moderate length, and as light as is consistent with strength.

In many dispensing establishments the *hand balance,* Fig. 2, is preferred for general use. Its advantages depend upon the rapidity with which weighings may be made, as well as the fact that, owing to its portable form, such a balance may be carried from one place to another for the purpose of making a weighing, instead of bringing all substances to the balance. The hand balance should be made of the best materials, the beam about 7 inches long. The knife-edges and rings at the ends of the beam should be contained in boxes to protect them from injury.

The pans may be made of well-nickelled brass, suspended by silken cords. If money is no great object, it is best to have the pans made of platinum, as this metal is only attacked by very few chemicals. The manner of holding the balance is shown in Fig. 2, the tassel being grasped between the thumb and forefinger of the left hand, the other fingers resting against the frame to ensure steadiness. Hand balances should only be raised a short distance from the counter, and on no account should the practice of raising the balance to the level of the eye be encouraged. It is not only unworkmanlike, but it tends to inaccuracy in weighing.

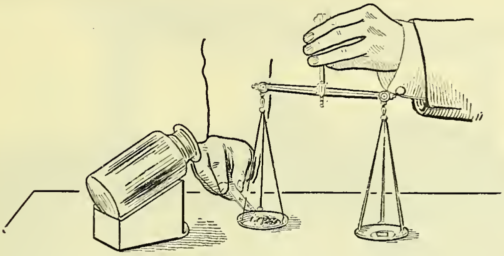

Fig. 2.

For weighing quantities of less than 2 grains a more sensitive balance than either of the foregoing is necessary. Fig. 3 shows a very sensitive balance constructed on the Dutch system. The bearings are of hardened steel working on agate planes, and the instrument is provided with a steel pointer and index. With such a balance it is quite competent to weigh to the $\frac{1}{10}$ of a grain.

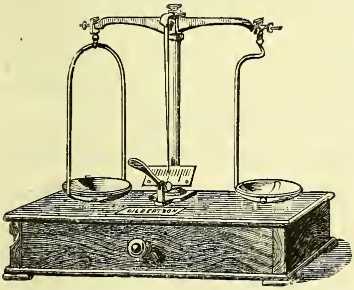

Fig. 3.

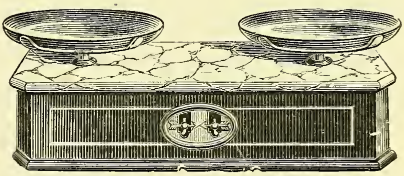

Fig. 4.

For weighing large quantities of material, balances of stronger construction are required. The forms in most common use are those known as "inverted" or counter machines, and possess more than three bearings. When the load does not exceed 1 to 2 lbs. the pattern shown in Fig. 4 finds many applications, while Fig. 5 is designed for loads from 2 to 14 lbs.

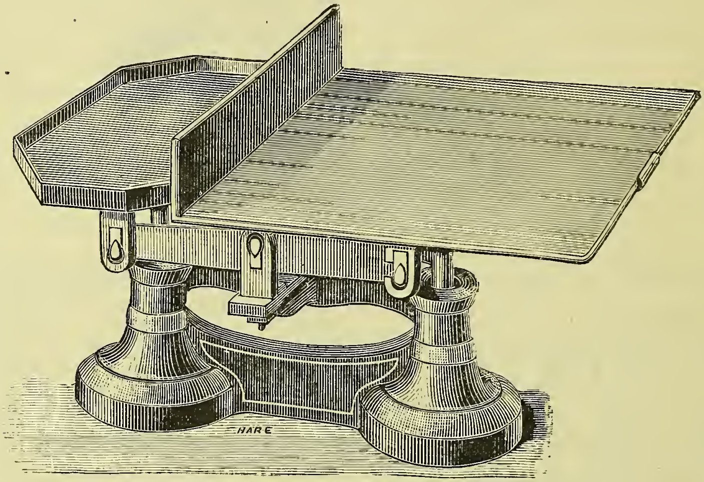

FIG. 5.

Trade balances.—Every weighing instrument used in trade is required to have the manufacturer's name as well as its maximum load marked in some conspicuous place. The maximum load indicates the greatest weight the machine is constructed to carry. In addition, every instrument is required to bear an inspector's stamp of verification, which is usually placed on a small stud or plug of soft metal under or above the central bearing.

All scale beams and counter weighing machines for weighing under 1 cwt. should turn accurately to the following amounts:

Maximum load of the weighing instrument.	Scale beam balances.	Counter weighing machines and spring balances.
1 cwt.	4 drams avoir.	8 drams avoir.
56 lbs.	2 ,, ,,	4 ,, ,,
14 ,,	1 dram ,,	2 ,, ,,
7 ,,	20 grains	1 dram ,,
4 ,,	5 ,,	20 grains
1 lb.	2 ,,	10 ,,
1 oz.	1 grain	5 ,,

The chemical balance.—For general pharmaceutical assaying the balance shown in Fig. 3 is sufficiently exact, but for operations requiring a higher degree of accuracy a proper chemical balance must be employed. Fig. 6 illustrates a quick-moving balance, constructed on Bunge's system. The triangular beam, which is very short, is made out of one piece of metal, and all the knife-edges and planes are made in agate, thus reducing friction to a minimum. The pans are platinised to prevent rust, and are provided with compensating stirrup suspenders to support them, thus preventing shifting of the knife-edges when the load is not placed in the centre of the pans. The centre agate plane in this class of balance, unlike all others, is made to rise perpendicularly, and lift the beam, so that it swings quite free from its supports. A balance of this kind in good order should be sensitive to $\frac{1}{10}$ milligram (·0001) with a load of 200 grams.

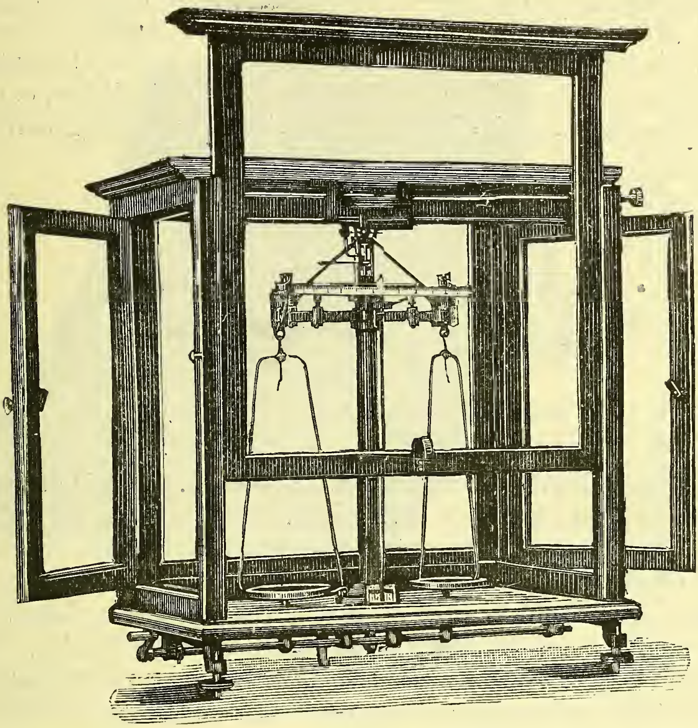

Fig. 6.

Balances should be kept free from acid fumes and moisture. A bottle containing quicklime or dried potassium carbonate ought to be kept in the balance case, and replenished as often as required. When working, the motion of the beam should be gently arrested when the pointer is at zero, and on no account should anything be added to or removed from the pans while the beam is free to oscillate. Substances to be weighed should not be placed directly on the pans; liquids are always weighed in stoppered glass weighing bottles; and the same applies to solids, unless they are quite unaffected by exposure to the atmosphere, when they may be weighed in platinum or porcelain dishes, or even in watch-glasses. Fig. 7 shows the kind of weighing bottle in general use; Fig. 8, a weighing tube; and Fig. 9, a pair of watch-glasses held together by a spring clip.

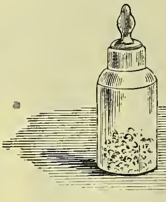

Fig. 7.

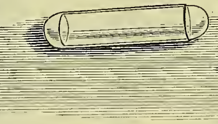

Fig. 8.

Fig. 9.

The weights used in analytical operations are almost exclusively those of the metric system. The usual shape for weights of 1 gram upwards is that of a short cylinder, with a handle at the top, which enables them to be lifted by small forceps. The weights from 0·5 gram downwards are either made of platinum or aluminium, the latter being preferred on account of its low gravity. The specific gravity of aluminium is 2·58, while that of platinum is 21·5. These small weights are usually flat, square in shape, with one corner turned up to facilitate lifting by the forceps. Fig. 10 shows such a set of analytical weights, contained in a velvet-lined box, and provided with forceps for lifting them.

The *rider* is a piece of aluminium wire bent in the form shown in Fig. 11, so that it lies astride the graduated beam, with its loop at right angles to it. As a general rule, balances employed in ordinary chemical analysis have each arm of the beam graduated into ten equal main divisions, each of which is divided into ten again. Using a *centigram* (·01) *rider*, each main division on the beam corresponds to 1 milligram (·001), and each of the subdivisions to the tenth of a milligram (·0001).

In practice the weights are rarely kept in the box, but are placed on a sheet of white cardboard, ruled in as many squares as there are weights, each square having the denomination of its weight plainly marked in ink (Fig. 12). When a weighing has been made, a double check on the weights is obtained by observing the value of those on the scale pan, and comparing with the uncovered spaces left on the card. This card of weights should

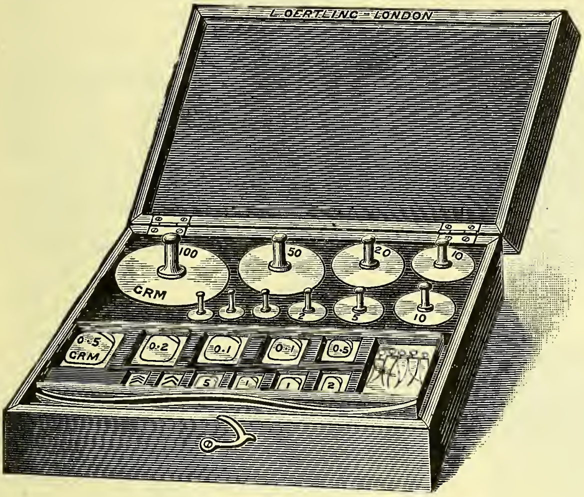

FIG. 10.

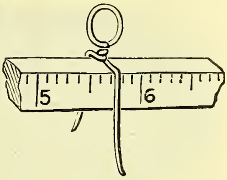

FIG. 11.

50	20	20	10
5	2	2	1
·5	·2	·2	·1
·05	·02	·02	·01

FIG. 12.

be kept on the floor of the balance-case, on the right-hand side of the pedestal.

Measures.—All measures and graduates used in trade are required to bear an inspector's stamp of verification. For measuring large volumes of such liquids as alcohol or water, which are without chemical action on the metal, copper vessels tinned inside are usually employed, as Fig. 13. In measures of this description the

FIG. 13.

FIG. 14.

proper volume is indicated when the surface of the liquid corresponds with the constricted portion of the neck. Measures of the type illustrated in Fig. 14 contain the designated volume when filled to the brim. For measuring hot liquids the graduated earthenware jug shown in Fig. 15 is exceedingly useful. This class of measures can never be used for dispensing purposes, or even for measuring potent remedies in bulk, on account of the comparatively large errors tolerated. Thus a one gallon metal measure may hold two fluid ounces in excess and yet be a legal measure; the half-gallon and quart may hold one fluid ounce, and the pint half an ounce in excess. As will be seen from the table given in the preceding chapter, apothecaries' measures are required to be much more exact.

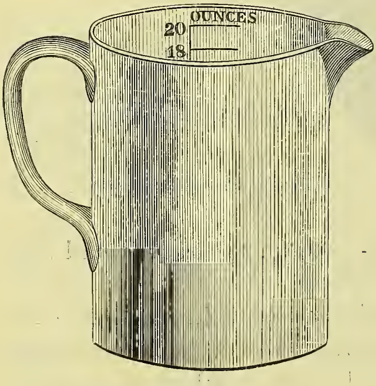

Fig. 15.

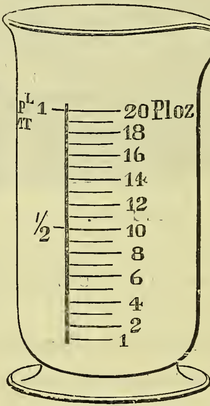

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Fig. 16.

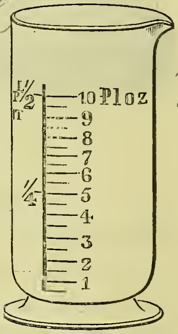

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Fig. 17.

For dispensing purposes glass measuring vessels are alone employed. For quantities from 40 to 10 fluid ounces the graduates may take the shape shown in Figs. 16 and 17, but for smaller quantities the measures should be conical in form (Figs. 18 and 19). It is not well to have the graduations too close together in any measure, as they only tend to confusion. Thus 40 fl. oz. and 20 fl. oz. graduates cannot accurately be used to measure smaller fractions than 1 fl. oz., nor 10 fl. oz. measures to indicate less quantities than half an ounce. The following graduates are almost absolutely necessary for the equipment of the practising pharmacist:—40 fl. oz., 10 fl. oz., 16 fl. drachm, and 4 fl. drachm. For measuring small quantities of strong medicines such as prussic acid, solution of strychnine, &c., a minim pipette should alone be used (Fig. 20). This consists of an ordinary graduated pipette fitted with a piston like the ordinary syringe.

When measuring with glass graduates, the bottom of the meniscus of the liquid should be made to coincide with the graduation. At first considerable difficulty will be experienced in holding the measure level, and until this art is acquired, the student should check every measurement by placing the glass on a level surface, and observing the volume again.

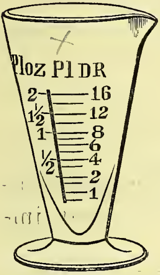

Fig. 18.

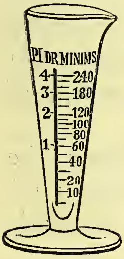

Fig. 19.

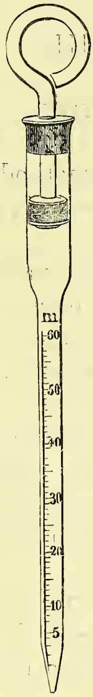

Fig. 20.

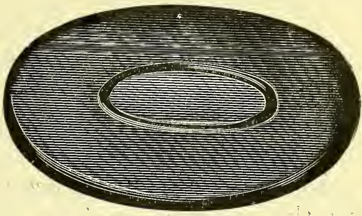

Fig. 21.

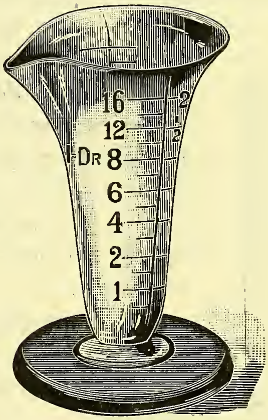

Fig. 22.

Conical measures of the types Figs. 18 and 19 are generally very weak in the stem, and if set down sharply on any hard surface, break off short. To prevent this rubber pads may be obtained which can be slipped over the measure foot (Figs. 21 and 22), and are known as Gilbertson's measure pads.

CHAPTER III

SPECIFIC GRAVITY

An intimate acquaintance with the methods used for ascertaining the specific gravities of solids and liquids is of great importance to the practical pharmacist.

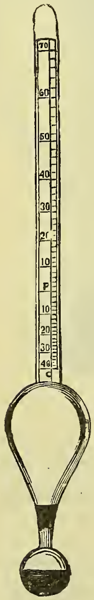

Fig. 23.

Specific gravity may be defined as the number which expresses the relation of the weight of a given volume of the body to the weight of the same volume of distilled water at a definite temperature. In this country specific gravities are determined at 60° F. In order to calculate the specific gravity of a body it is sufficient to find out its weight and that of an equal volume of water, and then to divide the first weight by the second ; the quotient is the specific gravity of the body.

Three methods are in use for determining the specific gravity of liquids—(1) by a hydrometer ; (2) by direct weighing in a flask ; (3) by the hydrostatic balance.

(1) *By means of the ordinary hydrometer* (Fig. 23).—Float the hydrometer in the liquid and read off the number indicated at the surface of the liquid. The specific gravity is obtained by dividing this number by 1000.

Examples.—An ordinary hydrometer floats in sulphuric acid 1843 ; what is the specific gravity ? 1843 ÷1000 = 1·843 sp. gr.

An ordinary hydrometer floats in ether at 720 ; what is the specific gravity ? 720 ÷ 1000 = ·720 sp. gr.

For liquids heavier than water hydrometers are graduated from 1000 to 2000 ; for liquids lighter than water from 700 to 1000.

Specific gravity beads are frequently employed by manufacturers who require to constantly adjust the specific gravity of the same class of liquid. They are of hollow glass, pear-shaped in form, and have the specific gravity they indicate engraved on them. These beads float indifferently in any liquid of the same gravity, and are especially useful as indicators when a liquid has to be evaporated to a given density. They may be regarded as hydrometers which indicate but one gravity.

(2) *By the specific gravity flask.*—As a rule, flasks holding 1000 grains or 50 grams of water are employed. If of the former capacity the operation simply consists in counterbalancing the flask, filling it with the liquid under examination, and weighing. The weight of the liquid divided by 1000 gives the specific gravity.

Example.—A flask weighed 260 grains; when filled with water 1260 grains; filled with spirit 1098 grains. What is the specific gravity of the spirit? 1098 − 260 = 838. Then 838 ÷ 1000 = ·838 sp. gr.

The bottle shown in Fig. 24 is commonly used for taking specific gravities, but in the author's opinion is not by any means to be recommended. It is of slight blown glass, fitted with a perforated stopper. Liquid is introduced into the bottle until quite full, the stopper is then inserted, the surplus liquid finding its way out through the perforation. The bottles are accurate enough when the temperature is below 60° F.; but it is manifest that if the bottle is filled at 60° F., and the room in which the operation of weighing is conducted is 65° or 70°, expansion of the liquid will take place, and the experiment will be ruined by reason of the contents oozing out through the perforated stopper.

Fig. 24.—Specific gravity bottle and counterpoise in case.

Fig. 25.—Regnault's specific gravity bottle.

Fig. 25 shows a specific gravity bottle constructed on a much better principle, designed by Regnault. In this form the liquid is filled in above the mark on the neck, the temperature determined by inserting a thermometer, and the liquid accurately adjusted to the mark by a pipette or fold of filter-paper. The stopper is then inserted, and the bottle weighed in the ordinary manner. The advantages of a bottle of this description are obvious, as once it is filled to the mark with liquid at the proper temperature, the weighing may be conducted at any higher temperature, as the neck of the bottle is sufficiently large to allow for expansion within any reasonable limits without loss. Moreover, as the stopper is accurately ground, there can be no loss by evaporation.

(3) *Determination of the specific gravity by means of the Westphal balance.*—The apparatus known as the Westphal specific gravity balance (Fig. 26) now used in all modern laboratories,

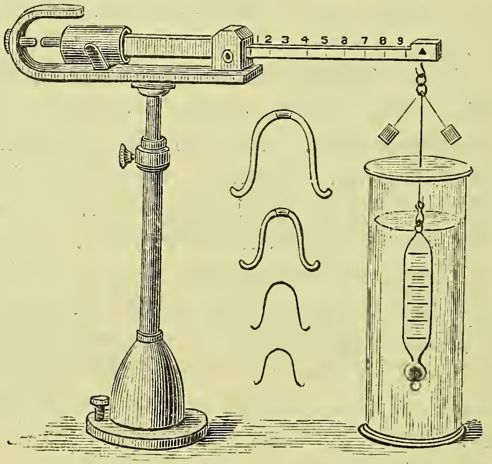

Fig. 26.—Westphal specific gravity balance.

depends upon the application of the principle that a solid body immersed in a liquid loses exactly the weight of its volume of that liquid. A small thermometer counterbalanced by the sliding weight at the opposite end of the beam is placed in the liquid and the loss of weight restored by rider weights, which indicate the specific gravity without further calculations. Fig. 27 illustrates the position of the weights for a few specific gravities.

Fig. 28 illustrates Mohr's method. Any accurate balance may be used by detaching one of the scale pans and suspending a thermometer in its place, and then counterpoising. The further operation does not differ from that described above.

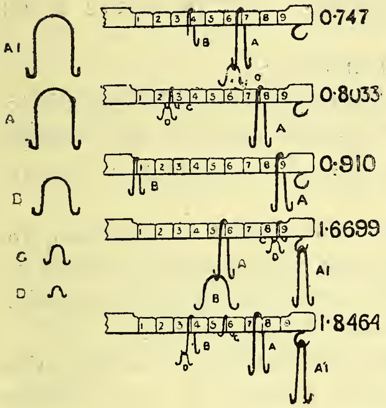

Fig. 27.

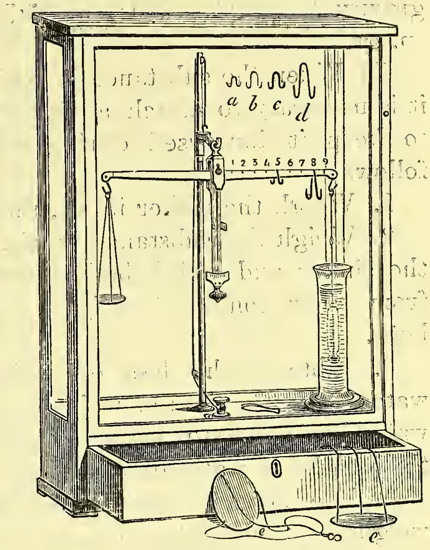

Fig. 28.—Mohr's balance.

Specific Gravity of Solids.

(*a*) When the substance is heavier than, and insoluble in water. The substance is weighed in air; it is then suspended by fine silk to one arm of a balance, as Fig. 29, and weighed again in distilled water. The loss of weight which it experiences is, according to the law of Archimedes, the weight of a volume of water equal to its own volume; consequently, dividing the weight in air by the *loss of weight* in water gives the specific gravity. Strictly speaking, the weighings should be performed *in vacuo*, and when great accuracy is required this is done, or corrections are applied for the weights of the unequal volumes of air displaced by the substance and by the weights in the opposite scale pan. Example:

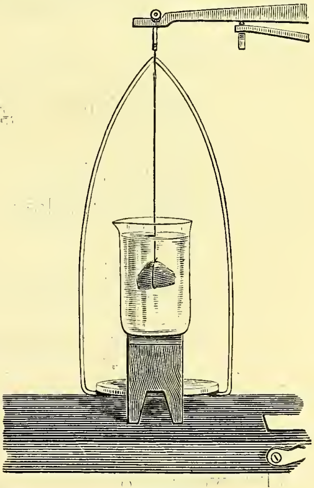

Fig. 29.

A piece of metal weighs 120·4 grains in air, and when suspended in distilled water 114·7 grains. What is its specific gravity ? 120·4 – 104·7 = 15·7. Then 120·4 ÷ 15·7 = 7·66 sp. gr.

(*b*) When the substance is insoluble in, but lighter than water, it is necessary to attach a sinker of known weight to the body to keep it immersed during weighing. The procedure is as follows :

1. Weigh the sinker in air, then in water.
2. Weigh the substance in air, then fasten it with fine silk to the sinker and weigh both in water, and subtract the weight from their combined weights in air. This gives the loss of both.
3. Subtract the loss of weight the sinker experienced in water from the loss of weight of both sinker and substance in water ; this gives the loss of weight experienced, or in other words, the weight of water displaced by the substance alone.
4. The specific gravity is now obtained by dividing the weight of the substance in air by the weight of water it displaces. Example :

A piece of wax weighs 30 grains in air, the sinker used weighs 120 grains. Their combined weight in water is 106·8 grains. The sinker alone in water weighs 108 grains. What is the sp. gr. of the wax ?

Weight of sinker	120·0
„ of wax	30·0
	150·0
Combined weight in water	106·8
Loss of both	43·2
Loss of sinker	12·0
Loss of wax in water	31·2
Weight of sinker in air	120·0
„ „ in water	108·0
Loss of sinker in water	12·0

The weight of the wax in air was 30 grains ; the weight of an equal bulk of water is 31·2 grains. Therefore 30 ÷ 31·2 = ·96, the specific gravity of yellow beeswax.

(*c*) For the determination of the specific gravity of solids soluble in water some other liquid of known specific gravity must be used for immersion. The substance is weighed in air, then in the liquid; the weight in air is divided by the loss of weight in the liquid, and the quotient multiplied by the specific gravity of the liquid. One of the most convenient liquids to use for the above purpose is rectified oil of turpentine, which has a specific gravity of ·87. Example:

A crystal weighs 35 grains in air, and 15·95 grains when immersed in oil of turpentine (sp. gr. ·87). What is its sp. gr.?

Weight of crystal in air . . .	35·00
,, ,, turpentine .	15·95
Loss of weight in turpentine .	19·05

Then 35·00 ÷ 19·05 = 1·83 sp. gr. of crystal as compared with oil of turpentine. Then 1·83 × ·87 = 1·59 sp. gr. of crystal.

Baumé's and Twaddell's hydrometers are commonly employed in manufacturing operations on a large scale, and as it is sometimes necessary to convert degrees according to these scales into specific gravities, or *vice versâ*, the following rules are given.

To reduce degrees of Twaddell's hydrometer to specific gravity.—Multiply by 5, add 1·000.

Example.—80° Tw. × 5 = 400. Then 400 + 1·000 = 1·400 sp. gr.

To convert sp. gravity into degrees Twaddell.—Subtract 1·000 and divide by 5.

Example.—1·400 sp. gr. − 1·000 = 400. Then 400 ÷ 5 = 80° Twaddell.

To reduce degrees Baumé to specific gravity.—(*a*) *For liquids lighter than water.*—To degrees Baumé add 134, and divide into 144. The answer will be the sp. gr.

Example.—Find the sp. gr. corresponding to 20° B.

$$\frac{144}{20^\circ\,\text{B.} + 134} = \cdot 935 \text{ sp. gr.}$$

To convert specific gravities into degrees Baumé.—Divide the sp. gr. into 144, and subtract 134 from the quotient. The answer will be degrees B.

Example.—Find the degrees B. corresponding to ·935 sp. gr.

$$\frac{144}{\text{sp. gr.}} - 134 = 20^\circ\,\text{B.}$$

To reduce degrees B. to specific gravity.—(*b*) *For liquids heavier than water.*—From 144 subtract degrees Baumé, and divide the remainder into 144. The quotient will be the sp. gr.

Example.—Find the sp. gr. corresponding to 10° Baumé.

$$\frac{144}{144 - 10^\circ \text{B.}} = 1{\cdot}074 \text{ sp. gr.}$$

To convert specific gravities into degrees Baumé.—Divide the sp. gr. into 144, and subtract the quotient from 144. The remainder will be in degrees B.

Example.—Find the degrees Baumé corresponding to 1·074 sp. gr.

$$144 - \frac{144}{1{\cdot}074} = 10^\circ \text{ B.}$$

CHAPTER IV

MELTING-POINT

THE accurate determination of the melting-point of many solid organic bodies is an important factor in estimating their purity. If a substance *begins* to melt at a certain temperature, but does not *completely* liquefy at the same temperature, the probability is that it contains some impurity. Various methods have been devised for determining the melting-points of substances, and notwithstanding the apparent simplicity of the operation considerable differences are noted between operators even when working with the same material.

The official process is as follows:—A minute fragment of the substance to be examined is placed in a thin-walled tube having an internal diameter of about 1 millimetre ($\frac{1}{25}$ inch), and sealed at the lower end. This tube should be attached to the thermometer (*a*, Fig. 30) so that the substance is near the middle of the

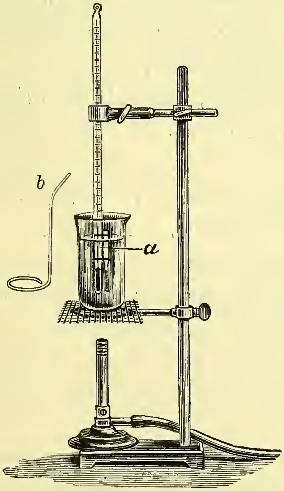

FIG. 30.—Apparatus arranged for taking the melting-point of a wax.

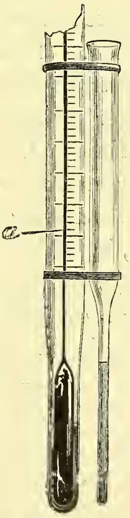

FIG. 31.—Enlarged view of capillary tube and thermometer bulb.

bulb, and the thermometer with the attached tube dipped in a liquid contained in a beaker placed over a small lamp flame. Water may be used for substances melting below 212° F. (100° C.), sulphuric acid or glycerine for higher temperatures. The liquid should, by means of a glass ring, be constantly (*b*) moved up and down until the substance is seen to melt. The temperature is noted, the tube cooled till the substance solidifies, and the operation repeated. *The latter reading of the thermometer should be taken as the melting-point.* To obtain accurate results the whole of the mercury column of the thermometer should be immersed in the heated liquid, but, as this is seldom practicable, the mean temperature of the emergent column—that is, of that portion above the surface of the heated liquid—should be ascertained, and the necessary correction applied. To obtain the mean temperature of the emergent column, a small thermometer is fixed in such a position that its bulb is about the middle of the emergent column. The correction may be calculated with approximate accuracy from the formula—

$$\mathrm{T\ (corr.)} = \mathrm{T} + \cdot 000143\ (\mathrm{T} - \mathrm{t})\ \mathrm{N},$$

in which—

T = observed, *i. e.* uncorrected temperature.
t = mean temperature of the emergent column.
N = the length of the emergent column in scale degrees.

To insure concordant results the capillary tube must be very closely watched, and the temperature noted the instant liquefaction takes place. It is better to make three or four determinations, and take the mean as the true melting-point.

Another accurate method is Dr. Cook's, which is described in the 'Journal of the Chemical Society' as follows:—"A beaker is filled to the brim with water; inside this, and separated from it on all sides, is a smaller one. The smaller beaker is partly filled with mercury, in which is placed a thermometer. A stirrer is used to keep the water in the large beaker of uniform temperature. A cardboard or other disc covers the smaller beaker when the operation is in progress. The whole is heated from below by means of a sand-bath. When the melting-point to be determined is under 30° C. it is better to replace the sand-bath by an evaporating dish containing water."

"The process is conducted as follows:—The material whose melting-point is to be taken is placed on three or four small pieces of thin ferrotype plate, or on the cover-slips which are used for microscope slides. If ferrotype or other metallic slips are used, care must be taken to remove the varnish or other coating, in order that good metallic contact can be had with the mercury. The slips with the substance on them are now placed on the surface of the mercury, and the heat applied until the substance melts. The solidifying point is obtained by raising the temperature above the melting-point and allowing the beaker to cool, noting the thermometer when the first solidification takes place.

"For temperatures between 100° C. and 200° C. the larger beaker is filled with paraffin wax.

"The following precautions have been found to be necessary:—(1) The temperature must be made to rise very slowly. (2) The liquid in the outer beaker must be frequently stirred. (3) Not less than 2·5 cm. in depth of mercury must cover the inner beaker. (4) Sufficient volume of water must be allowed between the two beakers. The minimum distance to give good results is one inch between them laterally, and an inch and a half at the bottom. (5) The inner beaker must be immersed a sufficient depth in the water. This point is of great importance, the least between the top of the mercury and the top of the water being three inches; a greater distance is, however, to be preferred. (6) The whole apparatus should be protected from draughts. (7) The disc should be kept on the smaller beaker during the determination.

"The following examples show the degree of accuracy to be obtained in ordinary working:

"1. Paraffin wax 49·8, 49·7, 49·6, 49·8.
"2. Paraffin wax 46·2, 46·0, 46·1, 46·0.
"3. Paraffin wax 46·5, 46·3, 46·5.
"4. Ortho-mono-nitro-phenol 44·5, 44·7.
"5. Urea 131·0, 131·5, 131·2."

BOILING-POINT

A liquid is said to boil when, by raising the temperature, the tension of its vapour overcomes the pressure of the surrounding atmosphere.* It follows from this that if the pressure becomes

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* The point at which the tension of the vapour of a liquid overcomes the pressure of the surrounding atmosphere is made clear by the following experiment:—Procure a piece of glass tubing about 34 inches long, and seal one end in a Bunsen flame. Entirely fill with mercury and insert it over a small dish, when the mercury will fall until the weight of the column left is equivalent to that of the atmospheric pressure. If now by means of a piece of bent glass tubing a little water is introduced into the lower end of the tube, it at once rises to the surface of the mercury and begins

reduced by any means, a liquid can be made to boil at a lower temperature. Advantage is taken of this in the construction and working of vacuum pans. An apparatus arranged for the determination of the boiling-point of a liquid is shown at Fig. 32.

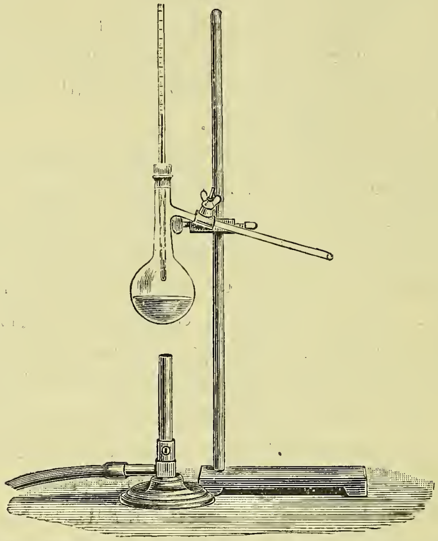

FIG. 32.—Apparatus arranged for determination of boiling-point.

The thermometer should extend well below the side tube, but should not touch the surface of the boiling liquid. In accurate determinations the *entire length* of the mercurial column should be surrounded by the vapour, but for ordinary observations the thermometer may project some distance if protected from currents of hot air by a disc of cardboard resting on the cork of the flask. The liquid is introduced into the flask until about one third full, and if it be of an inflammable nature the side tube is connected with an efficient condenser.

On the application of heat the mercury in the thermometer rises until the liquid is in free ebullition, when it remains stationary. If the atmospheric pressure be normal this tempera-

to vaporise, the evident result of which will be the depression of the mercurial column. This depression of the mercury represents the tension of the vapour of water for the particular temperature at which the experiment is made. If now the mercury tube is surrounded by a second tube of wide bore, and steam blown freely through, the mercury will become more and more depressed, until when the temperature of the steam reaches 212° F. (100° C.) the level corresponds with that of the mercury in the dish. At this point the tension of the vapour within the tube will equal that of the atmospheric pressure.

ture is noted on the exact boiling-point, otherwise correction must be made.

If any considerable length of the mercurial column is not surrounded by the vapour, the temperature of the emergent column should be ascertained as directed under Melting-point, and the necessary correction applied.

Fractional distillation.—If the liquid be a mixture of bodies of different boiling-points, it will begin to boil at a temperature near to the boiling-point of its most volatile constituent. If the heat is carefully managed distillation will proceed at this temperature until ebullition ceases, when the receiver is changed. If the heat be now increased ebullition will recommence, and a more or less complete separation of the constituents be effected, provided the receivers be changed at intervals, so that the several fractions are collected between certain limits of temperature.

CHAPTER V

HEAT

According to the undulatory theory, heat is not a substance, but a condition which can be transmitted from one substance to another. The heat of a body is caused by the extremely rapid vibration of its molecules, and the hottest bodies are those in which the vibrations have the greatest velocity and amplitude. With the exception of india rubber and one or two other substances, when solid bodies are heated the force of molecular cohesion is overcome, and expansion takes place by the increase of size of the intermolecular spaces. The effects of heat, therefore, are to cause bodies to expand, and to bring about a change of state, converting solids into liquids and liquids into gases.

Measurement of heat.—Instruments for comparing the sensible heat of various bodies are termed *thermometers.* The liquid used in their construction may be either mercury or alcohol; the former because it only boils at a very high temperature, the latter because it does not solidify under extreme cold. The mercurial thermometer, which is almost exclusively used in pharmacy, consists of a capillary glass tube, one end of which is blown out to form a cylindrical or spherical reservoir. This reservoir or bulb is filled with mercury, and heat applied until the mercury expands and fills the capillary tube, when it is sealed. After filling, the tubes are laid aside for a period ranging from several months to two or three years, to allow the glass to completely contract, when it is ready for calibration and graduation. For this latter, two points must be fixed, which represent identical temperatures, and can always be reproduced. It has been found by experiment that ice always melts at the same point, and that distilled water under normal conditions always boils at the same temperature. In the Centigrade (Celsius) and Réaumur thermometers the interval between these fixed points is divided into a number of parts of equal capacity called degrees. In the former the scale is divided into 100 degrees, in the latter into 80 degrees only,—that is, one degree Centigrade indicates $\frac{4}{5}$ of a degree Réaumur. Consequently degrees Centigrade may be converted into degrees Réaumur by multiplying by four and dividing by five. Degrees below zero on either the Centigrade or Réaumur scale are designated by prefixing the minus sign; thus $-10°$ C. means ten degrees below the freezing-point of water.

Although the Centigrade scale is generally used by chemists and physicists, that invented by Fahrenheit is in common use in this country. In this the higher fixed point is, like the other two, that of the temperature of boiling water; but the zero is obtained by mixing equal parts of sodium chloride and pounded ice, the interval between the two being divided into 212 degrees. According to Fahrenheit's scale ice melts at 32°, therefore 180° are equal to 100° Centigrade, or 1° is equal to $\frac{5}{9}°$ C. In converting degrees Fahrenheit into degrees Centigrade the number 32 must first be subtracted, in order that the degrees may both count from the same part of the scale. Then, as 1° F. is equivalent to $\frac{5}{9}°$ C., it is only necessary to multiply by 5 and divide by 9. Conversely, to convert degrees Centigrade into degrees Fahrenheit we multiply by 9, divide by 5, and add 32.

Thermometers employed in taking specific gravities, melting-points, or boiling-points should have been compared with a standard thermometer, and their errors recorded in a table, by means of which the readings of the instrument used are to be corrected. The zero points of the instrument should be verified from time to time.

Applications of heat.—Omitting coal fires, the chief sources of heat employed in pharmaceutical operations are gas, alcohol, and steam, the first named being the most generally used. Gas is used in one of the many types of Bunsen burners; in these gas is burnt with sufficient air to produce an almost colourless and smokeless flame, possessing great heating power. Fig. 33 shows a standard rose Bunsen; the flame holes are protected by a rim, and slope downwards to prevent wet running into the burner, as so often happens when liquids boil over. The correct distance between the bottom of the vessel and the top of the flame is half an inch. Fig. 34 illustrates what is known as a safety Bunsen; in this burner the gas can be turned down to a mere flicker without lighting back, but when turned fully on a flame seven or eight inches high is obtained. For heating vessels of large size

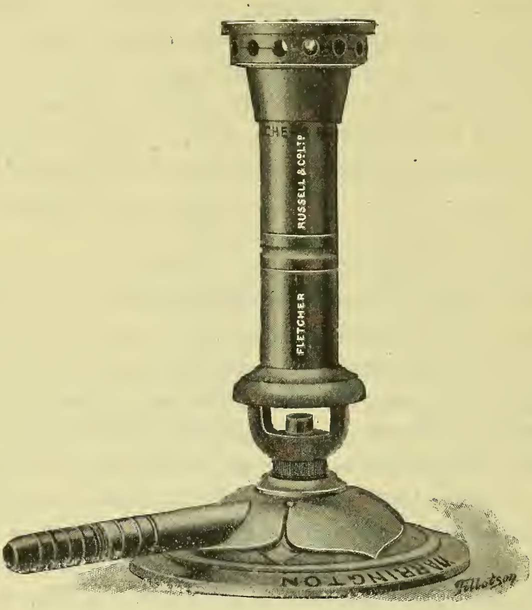

FIG. 33.—Standard rose Bunsen.

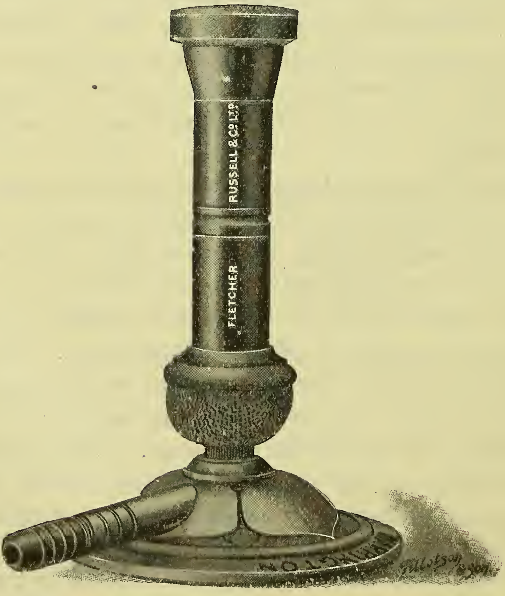

FIG. 34.—Patent safety Bunsen.

the triple concentric burner (Fig. 35) is one of the most powerful made. This burner is 8½ inches in diameter, and is very substantial, being made in one casting. Each ring is provided with a separate tap, so that the heating capacity may be regulated at will.

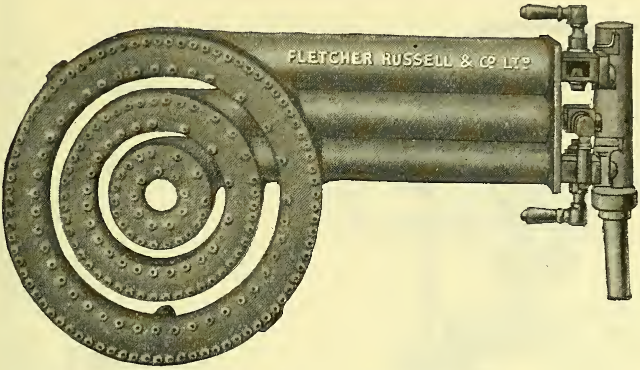

Fig. 35.—Triple concentric burners.

At the dispensing counter small vessels may be supported by tripods (Fig. 36), or the convenient apparatus designed by Mr. Thompson may be used (Fig. 37). This consists of a small atmospheric gas jet (Fig. 38 shows exact size) affixed to a standard. The burner may be either used for heating flasks, &c., for supporting which a ring is supplied which may be raised or depressed as desired, or the burner may be bent over at a right angle, when it may be utilised for a sealing jet. This is a most useful arrangement, as the wax, being melted in a flame free from carbon, is not darkened in colour. For heavier work a retort stand may be used. That illustrated at Fig. 39 has the iron supporting rod surrounded by a stout brass tube to which the rings are clamped. By means of this tube the rings may all

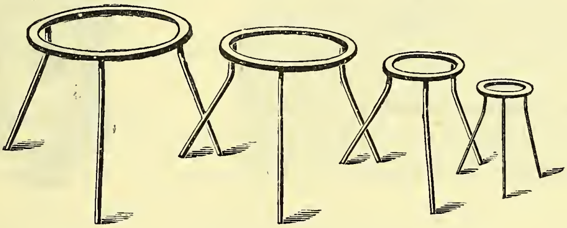

Fig. 36.—Tripods.

be raised or lowered simultaneously, while each ring, being provided with a straight arm and thumb-screw, may be removed separately.

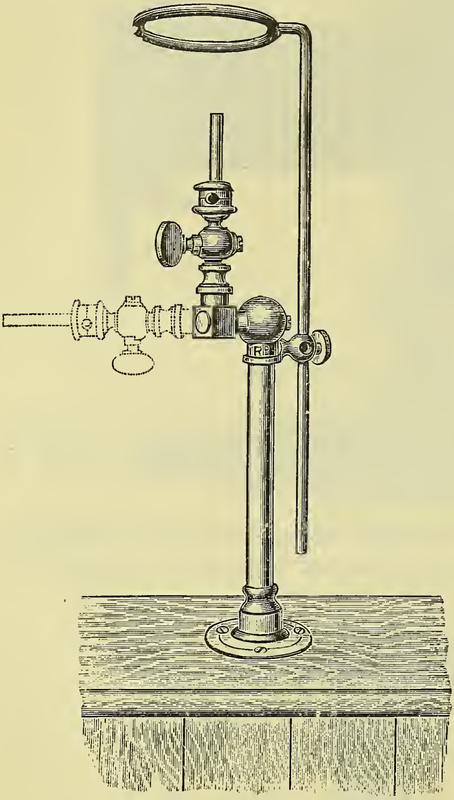

Fig. 37.—Thompson's burner and support.

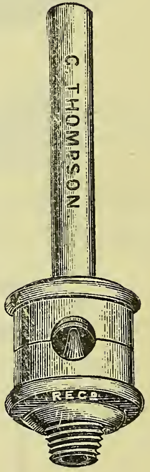

Fig. 38.

The burners hitherto described require the vessels to be supported by tripods or other device at the correct distance above the flame. This is occasionally inconvenient, and the type of burner shown in Fig. 40 was specially designed to overcome the objection. This stand carries flasks or dishes of almost any size with equal steadiness; while the flame, although of great power, does not light back even when turned down to its lowest point.

Spirit lamps.—When the gas supply is not convenient, spirit lamps are frequently employed for small operations. For melting sealing-wax, &c., the familiar glass lamp Fig. 41 is generally used; but since the introduction of the "Barthel" and other lamps of the same type, when alcohol can be obtained cheaply enough, heating operations of much greater magnitude are possible. Barthel's methylated spirit burner is absolutely safe, and is said to give an approximate heat of 1300° C. Fig. 42 is an illustration of Barthel's spirit Bunsen burner ; it gives a flame about ten inches in length, and is said to be equal to three or four ordinary gas Bunsens. The container is filled with methylated spirit, connected with the burner by flexible metallic tubing, and hung up about three feet above the bench. To light up, first open the tap beneath the container, allow time for the spirit to fill the tube, then give the regulating screw on the burner half a turn to the left, and let some spirit overflow into the saucer until it is about one third filled; then close the regulating screw and set light to the spirit in the saucer. This having burnt out, open the regulating screw again, and set light to the spirit vapour escaping at the mouth of the burners. By the regulating screw the flame can be made large or small, but it should never be less than an inch and a half. When the spirit in the burner has been lit, any spirit remaining in the

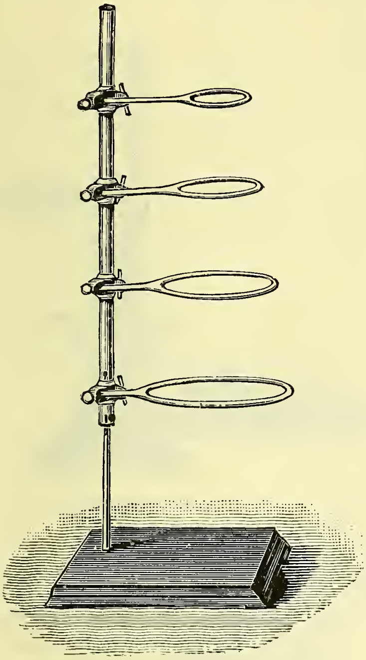

FIG. 39.—Griffin's retort stand.

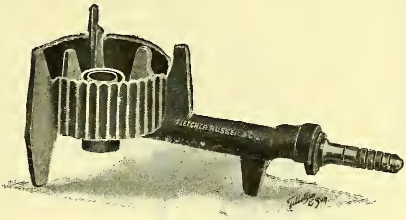

FIG. 40.—Argand Bunsen.

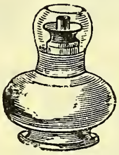

FIG. 41.—Spirit lamp.

parts of the burner this side of the regulating screw will flare up, but soon burn out. The flame may be unsteady just at first, but soon becomes perfect. Such unsteadiness must not be mistaken for the irregular yellowish flame which appears if the burner has not been sufficiently warmed up; in the latter case turn the flame down a little until the burner is sufficiently heated, and vapour comes off uniformly. Fig. 43 shows the same burner in section.

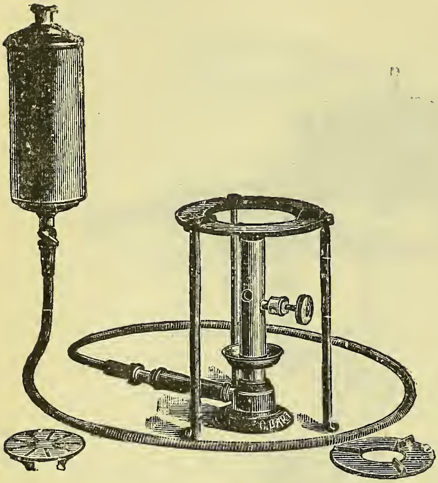
Fig. 42.—Barthel's methylated spirit Bunsen burner.

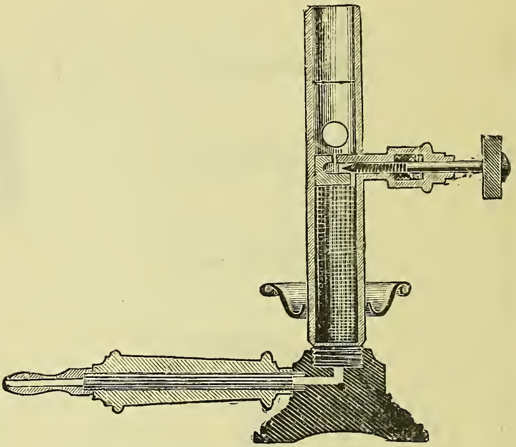
Fig. 43.—Section of Barthel's burner.

Sand-baths.—When for any reason it is undesirable to allow the flame to impinge directly on the bottom of the vessel, an iron or copper dish of slightly concave shape containing sand is intervened. Asbestos cloth or wire gauze may be used for a similar purpose. The author however prefers to use either aluminium or copper filings instead of sand; the heat is conducted much more readily, and is, moreover, under better control.

Furnaces.—For the operations in which a greater heat is required than an ordinary Bunsen burner affords, small gas furnaces are employed. Griffin's muffle furnace (Fig. 44) is a most useful adjunct to a pharmaceutical laboratory, as it can be utilised at all temperatures from a cherry-red to a white heat. The burner of this furnace consists of a circle of gas jets arranged so as to leave a central space, in which a fireclay block is fitted to form a solid support for a crucible, or over which a muffle may be placed exactly in the focus of heat. To obtain the best results the flue should not be less than six feet high. The furnace has two clay-stoppered holes at each side, and can be used with two combustion tubes at once. For ordinary work porcelain or platinum crucibles are employed, but for fusing caustic alkalies *silver* dishes or crucibles must be employed (Fig. 45). Care must be taken not to use too high a temperature, to avoid danger of melting the silver. Moist precipitates should never be introduced directly into the furnace, but must first be dried in a water or air oven. If this is not attended to, the loss by spurting will be very considerable, besides the danger of cracking the muffle. A coarser form of crucible is illustrated at Fig. 46.

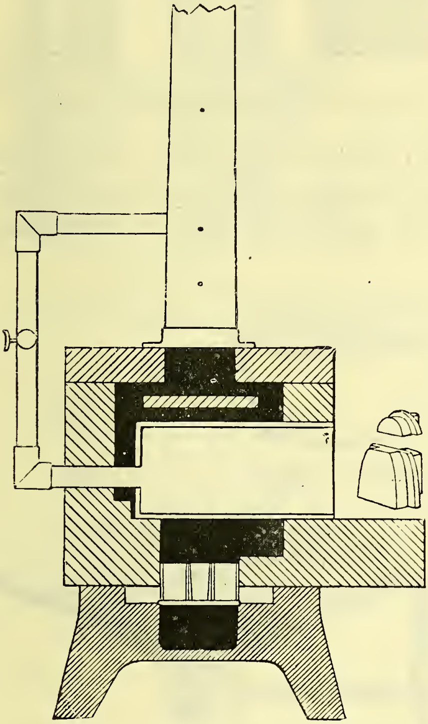

Fig. 44.—Gas muffle furnaces.

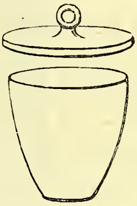

Fig. 45.—Porcelain crucible.

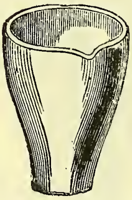

Fig. 46.—Battersea crucible.

Should a gas furnace such as the above not be available, ignitions and fusions may be conducted over a powerful Bunsen, the heat being confined by building an extemporaneous furnace round the crucible, as illustrated at Figs. 47 and 48. Over a Bunsen such as Fig. 34 place an iron tripod, as Fig. 36; on this burner build a furnace to suit the height of the crucible. First

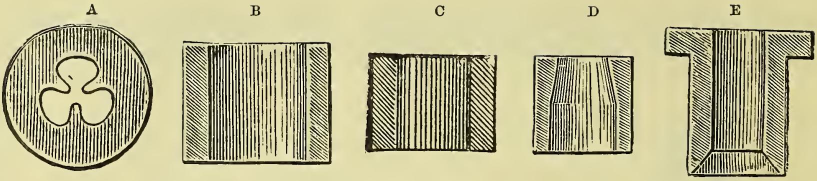

FIG. 47.—Fireclay cylinders, &c., for building extemporaneous furnaces.

place the grate A (Fig. 47), and on this the cylinders B and C, lastly the cone D. A tail-piece, E, may be added with advantage for high temperatures, but is not absolutely necessary. Fig. 48 shows such a furnace in operation.

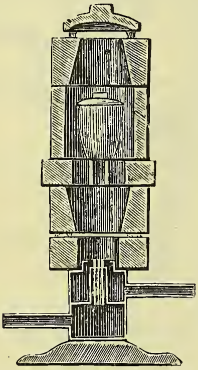

FIG. 48.—Extemporaneous furnace in use.

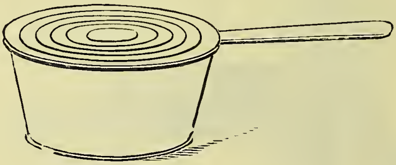

FIG. 49.—Copper saucepan with rings.

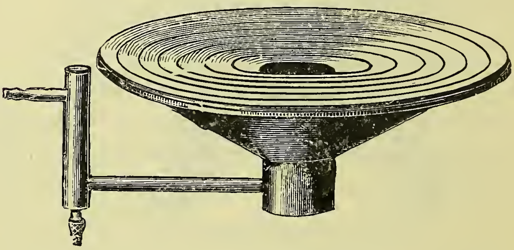

FIG. 50.—Pechi's water-bath.

Water-baths.—For operations requiring a temperature never exceeding 212°F. water-baths are commonly employed. These may consist of an ordinary copper saucepan (Fig. 49), provided with concentric rings, to adapt it for use with dishes and flasks of varying size. Fig. 50 shows a conical water-bath, which is useful for melting ingredients for suppositories, &c.; it only holds a small quantity of water, and is very rapidly heated, the water being kept at a constant level by the apparatus shown at the side. This form is well adapted for the laboratory for pharmaceutical assay-operations, &c. Figs. 51 and 52 illustrate two excellent forms of water-bath specially designed for melting ointments. In all the above forms of apparatus the upper vessel is heated by the vapour of the boiling water, consequently its contents never reach a temperature much above 200° F., the exact point varying with the freedom with which the steam is permitted to escape. Frequently it is found necessary to drill one or two small holes through the largest ring to relieve the internal pressure, otherwise the dishes may bump so violently as to spill their contents. When convenient the water-bath should be provided with a constant level apparatus, as Fig. 50, to avoid the necessity for constant refilling. A home-made apparatus that will answer every purpose may be constructed as follows:—Procure a length of glass tubing about one inch bore and six inches long, and fit it with two rubber corks. A piece of glass tubing, bent as shown in Fig. 53, is passed through a hole in the upper cork, and serves for the supply. Two tubes, one straight and of wide bore, the other narrower and bent at right angles, are fitted to the lower cork. The wide tube serves as the waste, the narrow tube is intended to feed the water-bath. When the connections are made and water turned on, it enters by the upper tube, and is directed against the sides of the glass reservoir, which it partially fills, finally overflowing by the waste pipe. The feed is connected with the water-bath, which may be at any distance away; by raising or lowering the apparatus the

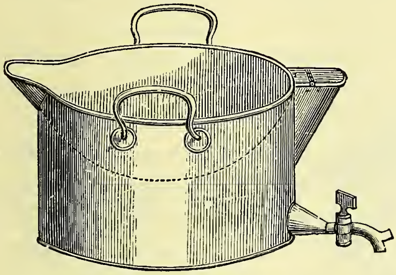

FIG. 51.—Maw's water-bath.

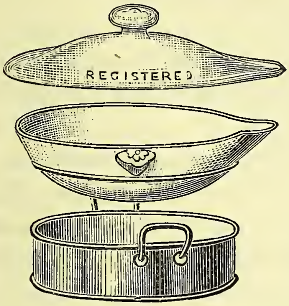

FIG. 52.—May Roberts's water-bath.

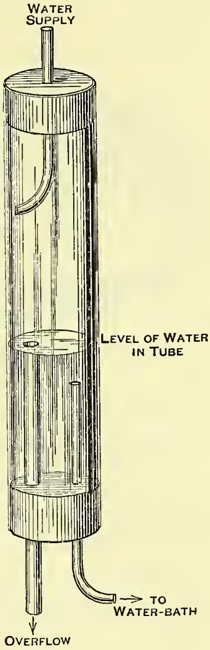

FIG. 53.—Constant level apparatus.

water may be maintained at any desired level, the final adjustment being conveniently made by pushing the waste-pipe further in or withdrawing it, as the water level is to be increased or diminished.

Vaporisation.—The ordinary term for the conversion of a volatile liquid into the gaseous state, by the more or less rapid disengagement of vapour at the surface, is *evaporation.* Unlike ebullition, which under normal conditions always takes place at a definite temperature, evaporation takes place between widely different temperatures, probably ceasing altogether below a certain point. In pharmacy the term is restricted to the concentration of solutions by the vaporisation of the more volatile portions, which are disregarded; and is distinct from *distillation,* in which the volatile portions are after vaporisation condensed and preserved, and from *desiccation,* by which solid bodies are deprived of moisture at comparatively low temperatures. *Exsiccation* is the name applied to the removal of the water of crystallisation from crystallised salts.

Spontaneous evaporation consists in effecting vaporisation at the ordinary temperature of the atmosphere. Practically, artificial heat is generally employed, and the process is still called spontaneous if conducted at the temperature of the drying-room (90°—100° F.). The evaporation of liquids at temperatures below their boiling-points is due to the diffusion of their vapours into the superincumbent atmosphere. All gases and vapours have a tendency to diffuse into—that is, to become intimately mixed with—other gases or vapours with which they may be brought in contact.

In effecting spontaneous evaporation, the rapidity with which the process will take place will depend—(1) *On the previous state of dryness of the air;* for as air can only hold a certain limited quantity of vapour of water in suspension, whatever it may already contain will so far limit its power of inducing diffusion, and if it be already saturated no further diffusion can take place into it. (2) *On the temperature of the air;* the higher the temperature, other circumstances being equal, the greater the amount of vapour it is capable of taking up. (3) *On the removal of the superincumbent air as soon as diffusion has taken place into it;* for the speed with which the vapour rises is greatly retarded as the air becomes partly saturated.

The most effectual means for promoting spontaneous evaporation consists in causing a current of warm dry air to pass over the surface of the evaporating liquid, proper arrangements being made at the same time for its removal as it becomes water-laden.

For quantities up to one gallon, evaporating basins may be made of porcelain or earthenware, as Fig. 54. Larger sizes are made of tinned copper or enamelled iron. Fig. 55 illustrates a new form of iron dish which has been turned perfectly smooth before enamelling. Each dish is furnished with a pair of detachable handles, easily affixed when it is required to be removed from the source of heat.

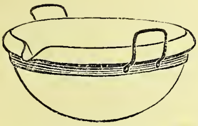

Fig. 54.—Evaporating dish.

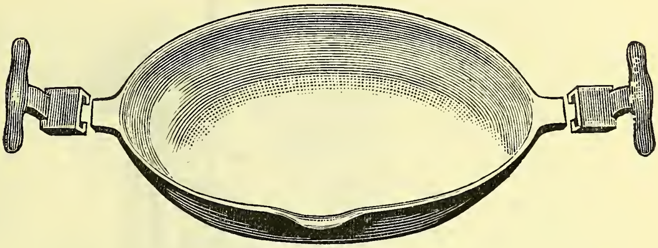

Fig. 55.—Enamelled iron evaporating dish with loose handles.

Steam-baths.—In the steam-bath the steam is employed under pressure, and, consequently, at a temperature above 212° F. The temperature will be 227° F. with a pressure of 5 lbs. to the square inch. At 10 lbs. to the inch it will be about 239° F., while with a pressure of 20 lbs. to the inch a temperature of 260° F. is reached. It will be seen from this that very much higher temperatures are attainable by the steam-bath than the water-bath ; thus steam at a pressure of only 5 lbs. to the inch, circulating in the jacket of a steam-pan, will heat it sufficiently to boil water and effect rapid evaporation. Pans for dealing with quantities of liquid from 5 to 200 gallons and upwards are made of either copper, which may or may not have a lining of block tin, or of solid block tin. Fig. 56 shows a jacketed copper pan of the form commonly used in pharmaceutical operations provided with a moveable mixing and stirring apparatus. This latter pan is carried on trunnions for tilting, and has many applications. It is estimated that every ten feet of heating surface in a pan requires one horse-power of steam per hour to evaporate one cubic foot of water. For the preparation of vegetable extracts the pans should either be made of block tin or enamelled iron. If copper vessels are employed the plant juices may attack the metal so rapidly, that if a bright steel knife is left in contact with the finished extract for a few hours it becomes covered with a thin coating of copper.

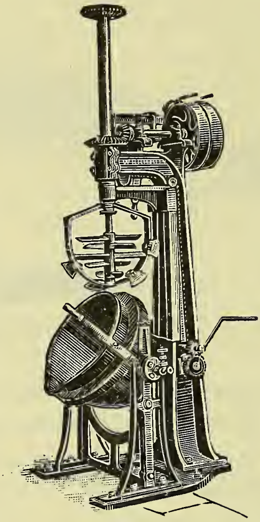

FIG. 56.—Tilting steam-pan and mechanical stirrer.

The disadvantage of using steam-pans in which the jacket is carried up to the rim is that, as the volume of liquid diminishes, the extractive matter is apt to bake on the exposed surface. This may be obviated by evaporating the liquid in a convenient vessel, not necessarily of metal, by a concentric coil of steam piping fixed at the bottom. It has, however, been pointed out that if the coil is placed just beneath the surface, the upper layers become concentrated by evaporation, and being heavier sink to the bottom, where they are exposed to a lower degree of heat, fresh and less concentrated portions of liquid rising, to be evaporated in turn.

Evaporation is accelerated by keeping the liquid in constant motion. Small quantities of liquids may be stirred by hand with a glass rod, or by one of the many forms of water-motor, of which Chicago's top is one of the most suitable (Fig. 57). The construction is exceedingly simple. It is composed of three principal parts: the metal casting forming the drum and base of the machine; the driving-wheel, consisting of a plate carrying twelve steel floats and a steel spindle, to which a pulley with three grooves is attached; and lastly, the bell-shaped distributor which directs the water under pressure against the floats of the turbine. The waste water is discharged through an orifice at the base.

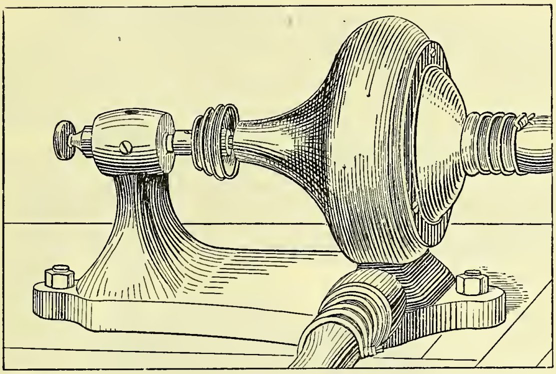

Fig. 57.—Water-motor.

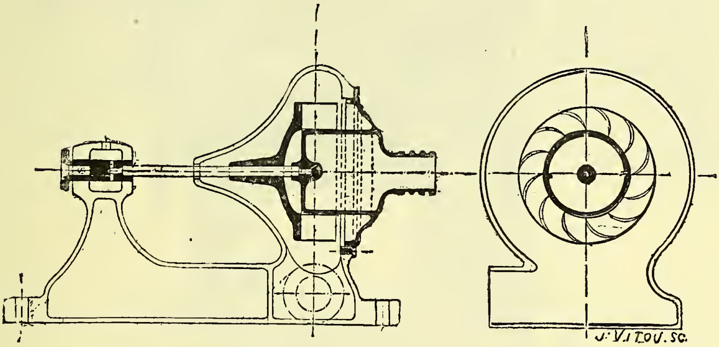

Fig. 58.—Section of ditto.

The supply pipe may be of canvas-lined rubber, and must be securely wired to the inlet pipe. The waste-pipe must be kept very free to prevent accumulation of water in the drum, which would check the speed of the wheel. To utilise the power to the best advantage a speed of about 2000 revolutions a minute should be maintained, one of the grooves of the driving pulley being connected by a flexible cord to the groove of a larger pulley at least 20 inches in diameter. Power may then be taken from the spindle of the larger pulley, or from a smaller pulley fixed on it. What is lost in speed is gained in power. With a head of 65 feet of water the motor uses 50 gallons of water an hour, giving 285 foot-pounds per minute. With a head of 195 feet, consuming 80 gallons an hour, over 3000 revolutions a minute are obtained, equivalent to 870 foot-pounds a minute. For stirring considerable volumes of liquids wooden paddles actuated by steam power are used.

Much loss and annoyance is sometimes caused by liquids boiling over during evaporation. This may not only be prevented, but vaporisation may be hastened, by inverting a funnel having holes in its base, and provided with a hood at the top of the stem, over the *actual heating surface*. The boiling liquid enters the funnel by the holes, rises up the tube, where it strikes the hood and is returned to the pan. The name *boiling fountain* is sometimes applied to this device.

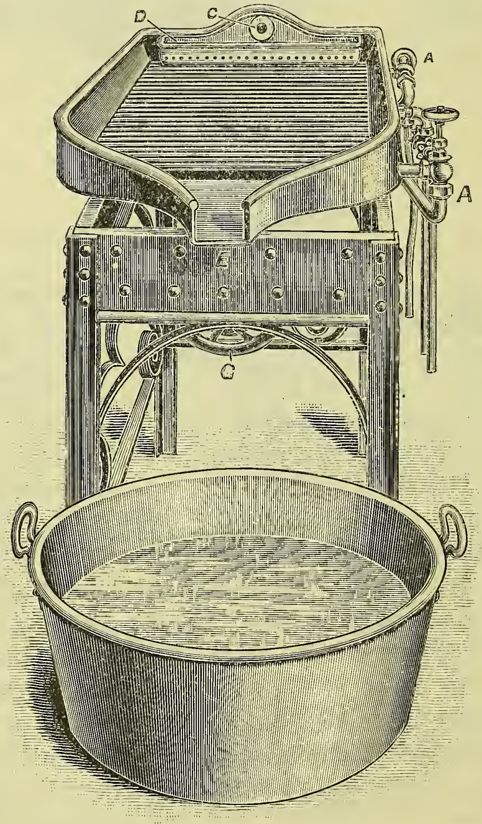

Fig. 59.—Sumner's evaporating tray—front view.

The most modern and perhaps most efficient form of evaporator is that known as Sumner's evaporating tray (Figs. 59 and 60). This consists of a series of copper tubes brazed together, and the ends so connected that steam circulates throughout the entire length. The tray stands on a rigid iron frame, to which

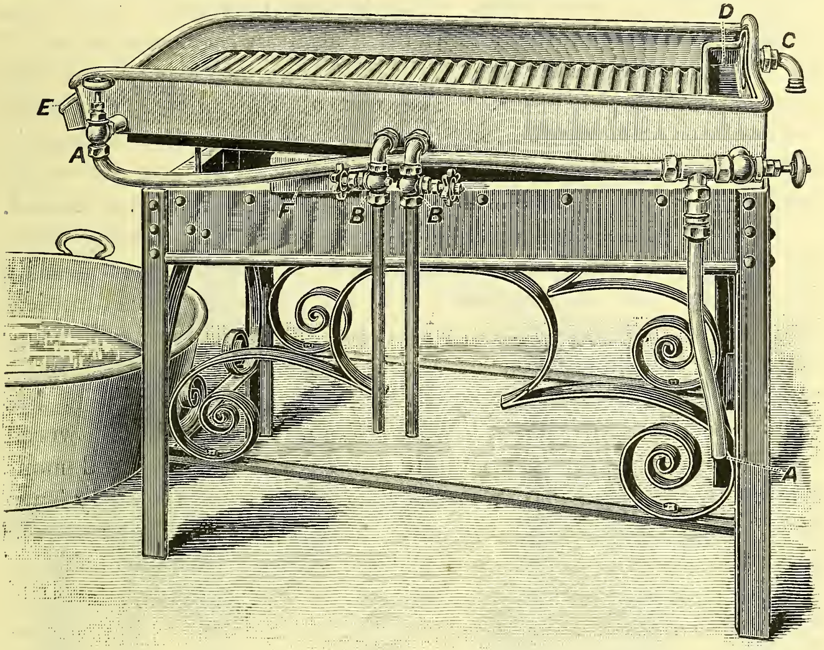

Fig. 60.—Sumner's evaporating tray—side view.

is fixed the elevating screw G, by means of which it can be tilted at any angle. The angle at which the tray is used depends upon the density to which it is desired to bring the liquid.

The liquid to be evaporated is carried by means of a pipe from the macerator, through the connection C into the receptacle D, which latter is perforated, so that the liquid falls upon the whole width of the tray, and travels slowly towards the delivery point E. Steam enters at A A and leaves at B B.

A tray of the capacity illustrated will evaporate twenty gallons of water an hour. This form of evaporator possesses several advantages over ordinary steam pans, the chief of which are its inability to boil over, the short time liquids are in contact with the heating surface, and its almost automatic action. Once the feed is regulated to the angle at which the tray is tilted, evaporation proceeds without attention, no stirring being required, as the liquid is in constant motion the whole time.

The steam bath or pan offers great facilities for regulating the temperature by the admission of more or less steam into the jacket, or if necessary it may be instantly excluded, when the temperature at once falls. The heat is completely under the control of the operator, and a little skill in the management of the valves will enable him to guard against injury resulting from injudicious heating. The use of the stirrer especially necessitates some caution, as, if there were only a little extract in a pan, rapid stirring would spread a thin stratum over the entire surface, where it would be liable to become baked and over-heated.

Vacuum pans.—Evaporation in open pans will probably never be superseded for the concentration of liquids not readily injured by heat or exposure; but all modern pharmaceutical laboratories are now fitted with vacuum pans, for evaporating vegetable infusions such as those of senna, malt, &c., under reduced pressure at comparatively low temperatures. It has before been stated that the boiling-point of a liquid is lowered by reducing the atmospheric pressure. This is readily demonstrated by boiling a little water for some minutes in a fairly large flask, so as to expel all the air. If the flask is now removed from the source of heat, and at once securely corked, ebullition may be started again by placing a cloth that has been dipped in cold water over the upper part of the glass, when owing to condensation of the vapour a partial vacuum is formed. Fig. 61 is an illustration of a vacuum pan which is actually worked com-

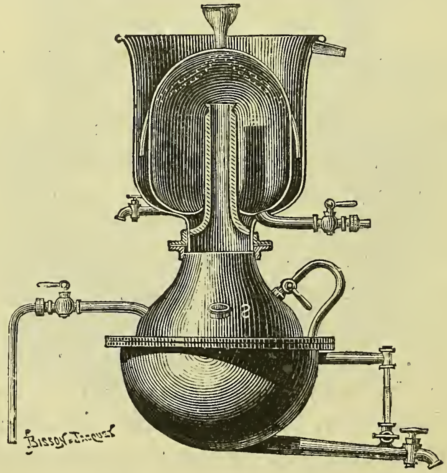

Fig. 61.

mercially on these lines, the vacuum being obtained by the condensation of the vapour. Fig. 62 shows a small apparatus in which the vacuum is created by a water-pump. The steam-jacketed boiler (1) has a capacity of about 1½ gallons, and is connected by a glass dome and arm to the condenser (2), which in its turn is connected to the receiver (3). The condensing water enters at W, and serves at the same time to create the vacuum.

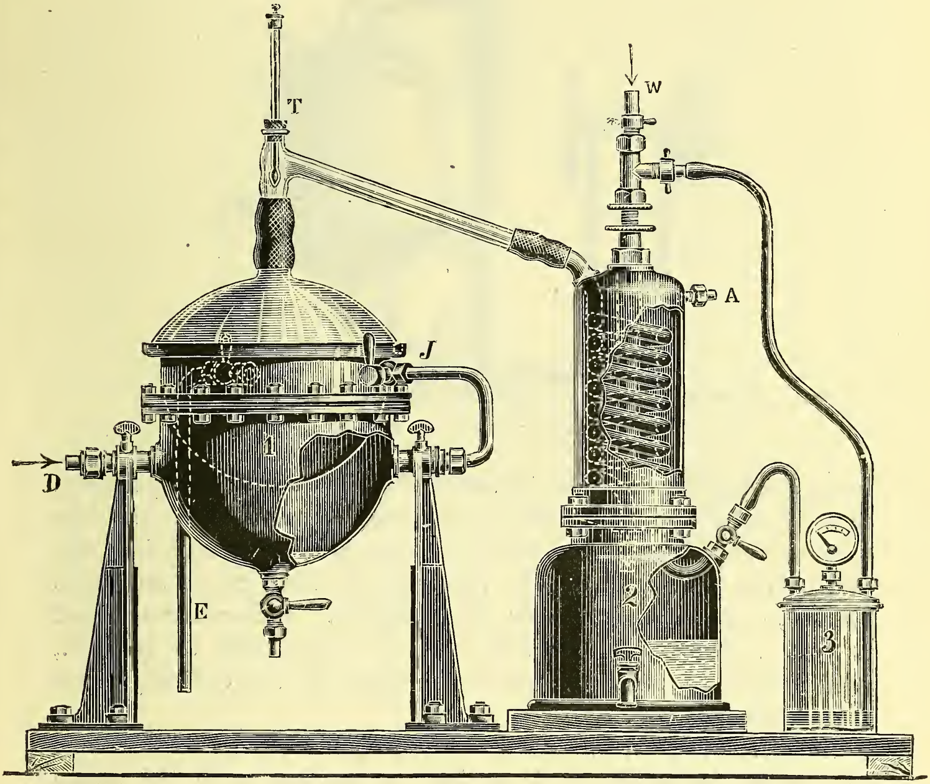

FIG. 62.—Vacuum apparatus. (Gallenkamp's pattern.)
D. Steam inlet to the jacket. J. Glass dome. T. Thermometer. W. Filter-pump.
E. Tube for refilling boiler without disconnecting the apparatus.

Large vacuum pans are generally made in copper, heated by either a steam jacket or internal steam coil. The vapour is pumped out as quickly as formed by a wet air-pump, and runs to waste with the condensing water (Fig. 63). In *vacuum stills* the vapour is pumped out, and condensed in a closed tubular condenser, from which it passes to a closed receiver. A glass window is let into the top of the pan, so that by the aid of an electric lamp the operator is able to watch the progress of an operation, and by means of the thermometer and steam-cock control the evaporation. The liquid to be concentrated is sucked into the pan by the pipe seen on the right of the illustration, and once started, evaporation proceeds automatically. It is calculated that a good vacuum pan will boil water at about 120° to 130° F., and evaporate twice as much per hour as an ordinary open steam-jacketed pan.

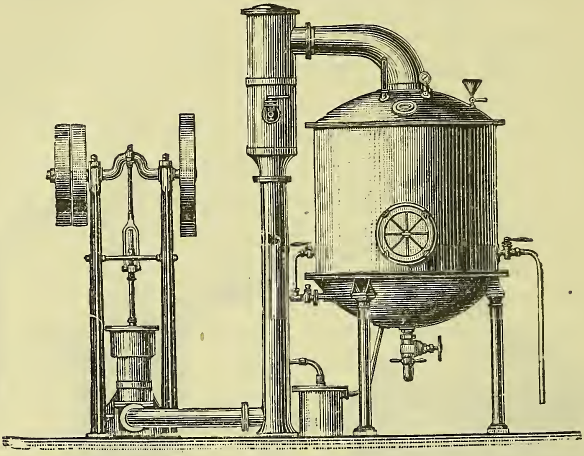

FIG. 63.—Large vacuum pan with steam-pump.

Temperature of steam-jacketed pans.—The temperature obtained in a steam-jacketed pan is entirely dependent upon the steam pressure. If there were no leakage at the joints or unions, and no loss by surface condensation, the temperature would be readily ascertained by reference to the following table. But as steam pipes frequently leak, and surface condensation varies, the exact temperature can only be known by fixing a steam gauge to the jacket.

Table of Temperatures of Steam in Degrees Fahrenheit, corresponding to Pressure in Pounds per Square Inch above *Atmosphere.*

Pounds per square inch above atmosphere, as shown by the gauge.		Degrees Fahrenheit.
0 (= 14·706 absolute pressure)	=	212·0
1	=	215·4
2	=	218·6
3	=	221·6
4	=	224·5
5	=	227·2
6	=	229·8
7	=	232·4
8	=	235·8
9	=	237·1
10	=	239·3
11	=	241·5
12	=	243·7
13	=	245·9
14	=	248·0
15	=	249·9
16	=	251·7
17	=	253·5
18	=	255·3
19	=	257·1
20	=	258·8
21	=	260·5
22	=	262·1
23	=	263·7
24	=	265·3
25	=	266·8

These calculations may be extended by applying the formula—

$$T. = \left(\frac{2938{\cdot}16}{6{\cdot}1993544 - \log.\ P.}\right) - 371{\cdot}85,$$

where T. = temperature of steam in degrees F.
P. = pressure including atmosphere.

Distillation.—Distillation is the name given to the operation by which liquids, after conversion into a state of vapour by application of heat, are condensed to the liquid state again. The process is made use of for separation of liquids from solids as well as liquids from liquids; the latter separation can only be effected when their boiling-points differ considerably. The apparatus in which this operation is performed is called a retort or still, and the condensed liquid the distillate. Quantities of liquids from an ounce to several pints may be distilled from glass flasks or retorts. When a flask is used a perforated cork carrying a glass tube is adapted to act as a condenser. This tube may be wrapped with several folds of linen rag dipped in cold water, which is changed from time to time as it becomes heated. Retorts may be either plain as Fig. 64 or tubulated and stoppered as Fig. 65. The latter are much more convenient, as the liquid to be distilled may be easily introduced without risk of contaminating the neck of the retort, which so frequently happens when plain retorts are filled. The shape of the retort is of great importance, as if bent at too wide an angle there is danger of liquid being carried over with the vapour ; the tubulure, moreover, should be so situated that when a thermometer is used it may be inserted by means of a perforated cork *in a vertical position*.

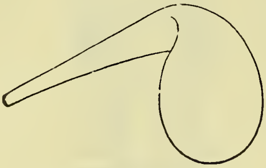

Fig. 64.—Plain retort.

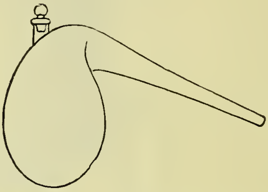

Fig. 65.—Tubulated retort.

Adapters.—It occasionally happens that the base of the condenser is too small to admit of the introduction of the neck of the retort. In this case glass adapters of the form shown in Fig. 66 are used to make the connection.

Fig. 66.—Adapter.

Condensers.—Formerly a glass tube bent in the shape shown in Fig. 67 was usually employed, but nowadays the Liebig form of condenser is almost invariably used. This consists (Fig. 68) of a long and somewhat tapering glass tube, narrowed at one extremity and somewhat funnel-shaped at the other. This tube is fixed by means of corks or rubber bands inside a large cylindrical tube of glass or copper, the space between the two tubes being constantly supplied with cold water. Fig. 69 shows the most improved form of Liebig's condenser. The body is made of

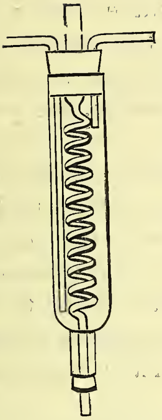

FIG. 67.—Worm condenser.

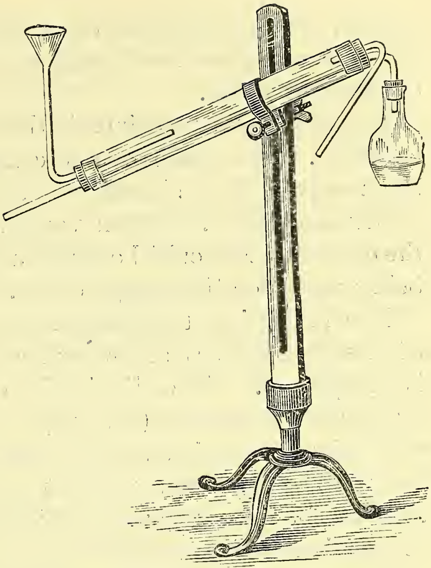

FIG. 68.—Liebig's condenser.

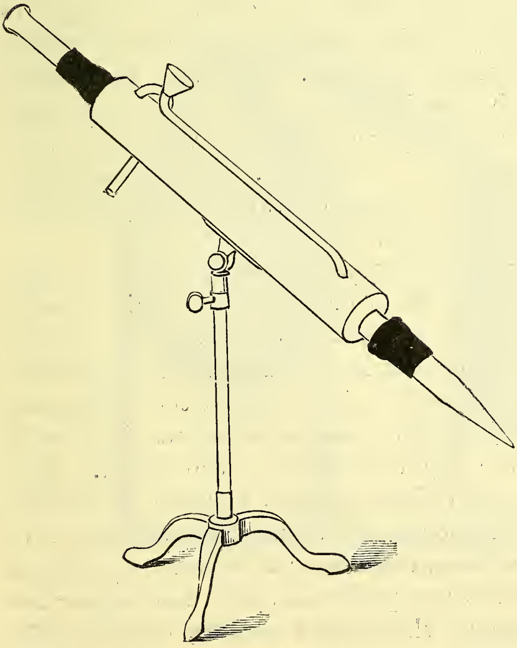

FIG. 69.—Griffin's form of condenser.

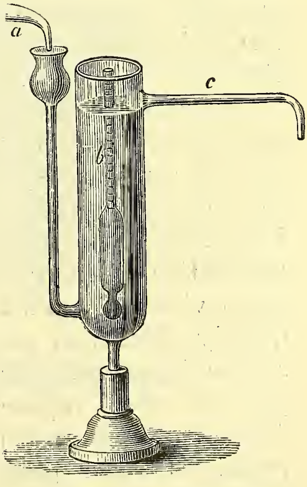

FIG. 70.—Mohr's still watcher.

copper, and is mounted on a telescopic stand with a knuckle-joint; metal pipes are brazed on for supplying and removing the condensing water.

Receivers.—The receiver is the vessel in which the distillate is collected. The nose of the condenser should extend well into the receiver, but should not dip beneath the surface of the condensed liquid. When very volatile distillates are collected the connections should be made as tight as possible, due provision being made for the escape of air.

For indicating the progress of a distillation by the density of the distillate, a still-watcher of the form shown in Fig. 70 is employed. The liquid is delivered from the condenser by the bent adapter (*a*), and accumulates in the reservoir (*b*), where its density is tested by the hydrometer. When the distillate rises to the side arm (*c*) it overflows into another vessel, distillation being continued until the hydrometer indicates the completion of the operation.

Notes on the use of glass retorts and flasks for distillation.—Glass retorts ought never to be heated by a naked flame, but should always be protected by a piece of thin nickel or copper gauze. For many operations it is preferable to use a bath of sand or copper filings, the grains being heaped round the glass to a height of one or two inches. Many liquids when boiled in glass vessels give rise to the phenomenon of *bumping*. This may be lessened or altogether prevented by the introduction of one or two small coils of platinum wire, or a few pieces of pumice which have been previously heated to redness in a crucible. These merely act mechanically by promoting the escape of vapour and preventing the formation of explosive bubbles.

The phenomenon of "bumping" is peculiarly liable to occur when ebullition takes place in glass or glazed porcelain vessels, more rarely in polished metal vessels. It is generally assumed to be caused by the sudden conversion of the particles of liquid immediately next the heated surface into vapour, the sudden accumulation of energy projecting the superincumbent liquid with great violence against the top and sides of the vessel. Frequently distillation proceeds quietly enough, because the heated surface is not perfectly clean or smooth, but offers a number of points at which vapour collects, dispersing almost as quickly as formed. If no such points are presented, then the vapour accumulates under pressure at the heated surface, owing to the great adhesion which naturally exists between many liquids and their vapours, until, becoming superheated, disruption of the vaporous particles occurs with considerable violence.

In large steam boilers this phenomenon is called "priming," the vapour accumulating at the heating surface and lifting the water just as it does in a glass flask, but rarely with sufficient force to rupture the boiler-plates. When a boiler under great pressure explodes, it is frequently caused by the falling in of the fire-box or by a large fitting blowing off. The effect of this is to liberate the steam, which rushes out of the hole thus formed. The pressure on the water in the boiler then being suddenly removed, the watery layer next the hot plates is instantly converted into steam, which not being able to escape quickly enough, discharges the remainder of the water with such violence against the plates of the boiler as to rip it open from end to end. In this case it is the water acting as a water-hammer which does the damage, and not the steam which was primarily present *as steam.*

Earthenware stills.—Owing to the fragility of glass retorts, it is customary when considerable bulks of liquid have to be dealt with to employ earthenware stills for the distillation of certain liquids,—sweet spirit of nitre, for instance. In the illustration (Fig. 71) the body of the still is contained in a cast-iron steam jacket, the joints being made steam-tight with large soft rubber

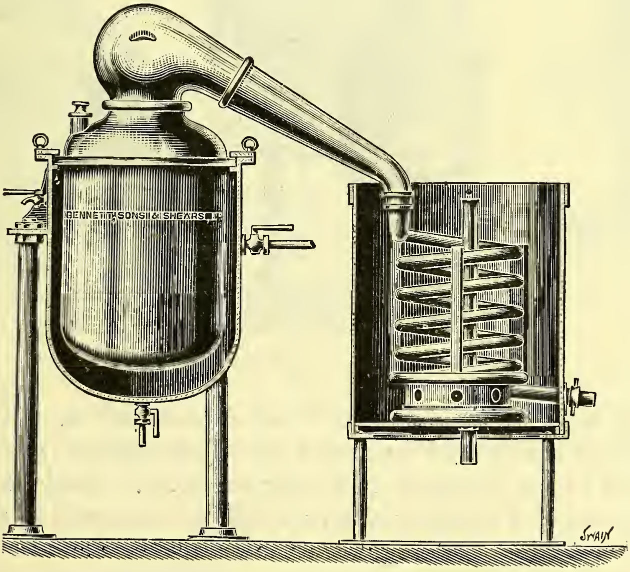

FIG. 71.—Earthenware still and condenser.

washers. The head and arm are separate, and require to be luted on to the condensing worm, which is contained in a metal tank. Great care must be taken in the fitting together of these stills to allow for the unequal expansion of the earthenware and metal when heated.

Water stills.—A good supply of distilled water is of the first importance in pharmaceutical laboratories. In manufacturing concerns this is obtained either from the steam boiler, or the waste steam from the jacketed pans is condensed and utilised. If the boiler is fed by means of an injector, the condensed water may be sufficiently pure for all ordinary operation; but if fed by means of a pump the distilled water nearly always contains traces of oil used for lubricating the working parts of the engine. Nor is this the only drawback, as many engineers are in the habit of using all sorts of compounds for preventing scale, none of which tend to improve the quality of the water obtained by condensing the steam. Fig. 72 shows an automatic water still especially designed to meet the requirements of retail pharmacists. It consists of a copper still heated by means of a low-pressure gas burner, the steam being condensed in a block tin worm. Between the still and condenser is a supply cistern which is connected to the main by rubber tubing, and regulates the supply by means of a ball-cock. The water enters the condenser at the bottom, and overflows by a pipe near the top into the still. By this arrangement warm water is automatically fed into the still, accelerating distillation and economising gas. The great drawback to constant distillation is

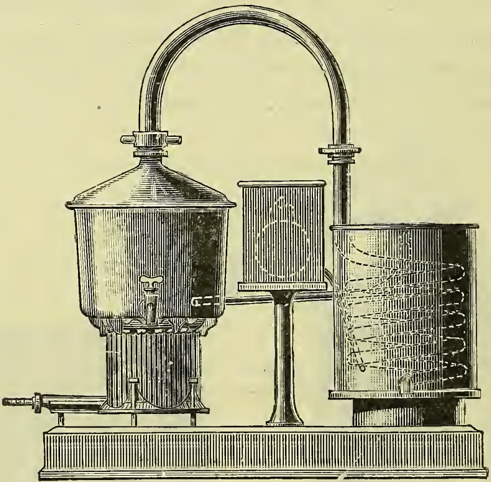

FIG. 72.—Shear's registered auto-still.

that no provision is made for rejecting the first portion of distillate, which always contains ammonia. Another very convenient water still is shown at Fig. 73. The water is boiled in the lower part and condensed in the conical upper part, which is supplied with cold water. The liquid as it condenses flows into a trough, and is discharged through a small pipe. This apparatus is well suited for recovering alcohol from tincture marcs, &c. When not used as a still the upper part may be detached and a pan inserted, transforming it into a water-bath.

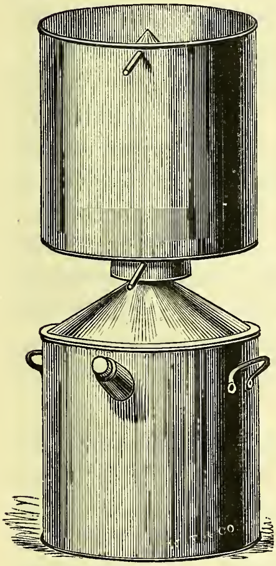

Fig. 73.—Phœnix water still and condenser.

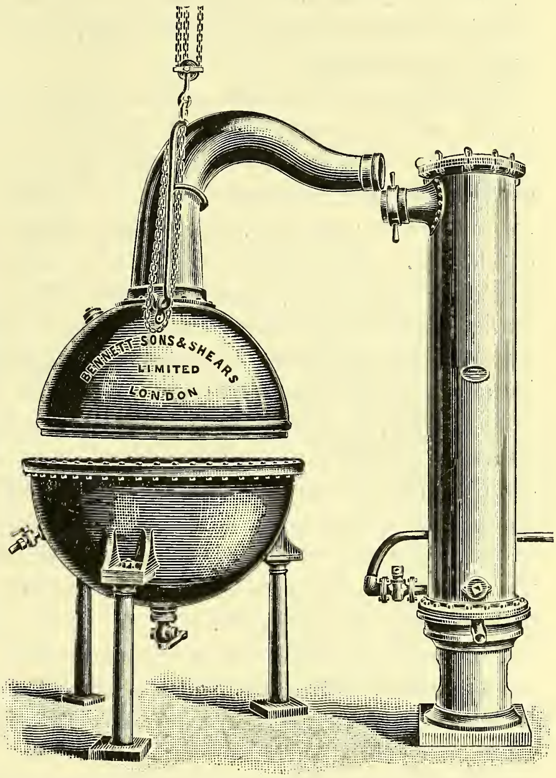

Fig. 74.—Combined steam pan and still.

Large pharmaceutical stills are nearly always made of copper, and are provided with cast-iron or copper steam jackets. Fig. 74 illustrates a still and evaporating pan combined. When the dome and head are removed the pan may be used for ordinary evaporation. The condensers for these large stills are rarely of the old-fashioned worm type, but consist of an upright cylinder containing a number of straight tubes, between which as little space is left as possible. The condensing water enters at the bottom and flows away to waste from the top. As the condensing tubes do not contain any bends, they are readily cleansed by removing the cover, when they may be brushed out. This last is a great advantage over the old form of condensers, which could only be cleansed by blowing steam continuously through, often a tedious and unsatisfactory proceeding.

Distillation of volatile oils.—Volatile oils, such as those of cloves and caraway, are best distilled in stills heated by steam, the charge being placed on wire gratings, so as to be out of contact with the heating surface. Live steam, which is then blown in beneath the grating, carries the volatile constituents into the condenser. Peppermint, chamomile, lavender, &c., are generally distilled from large copper stills heated by naked fires, the charge being placed on a false bottom along with a large quantity of water. The mixture of oil and water distils together, and is collected in large receivers; the oil which floats on the surface is skimmed off, and the saturated water reserved for a fresh charge. Dense woods such as sandal are first reduced to fine raspings, and distilled in a current of steam, the mass being constantly roused by mechanical stirrers, actuated by overhead gearing. Salt was occasionally added by some distillers to raise the boiling-point of the water, but the practice has been mostly abandoned in favour of the preceding methods.

Destructive distillation is the process of decomposing organic substances in retorts at a high temperature, the volatile products which pass over either escaping as gases, or condensing as liquids of varying constitution and physical characters. The process is one that more nearly concerns the manufacturing chemist than the pharmacist. On account of the high temperatures employed, and the nature of the residues, iron retorts of large size are invariably employed. The preparation of acetic acid with its accompanying by-products affords a good example of the application of this process. Billets of wood are packed in iron retorts or cylinders heated by furnaces. The volatile products are passed through condensers of large bore cooled by water, the condensible portions collecting in appropriate receivers, while the gases, consisting of ethylene, acetylene, marsh gas, and other hydrocarbons, are led into the furnace, and serve to economise the fuel. The condensed portions consist of a tarry mixture of empyreumatic oils and phenylic compounds floating on an aqueous substratum. By proper treatment acetic acid, wood spirit, acetone, &c., are obtained from the watery solution, and creasote from the oily layer.

Still licence.—By an Act to prevent the use of stills by unlicensed persons, an annual excise duty of ten shillings is imposed on every person, not being a licensed distiller, rectifier or compounder of spirits, or vinegar maker, who keeps or uses any still or retort. Under a general order dated October 31st, 1892, the Inland Revenue authorities allow professors of chemistry, teachers of chemistry and analytical chemists who carry on no business involving the manufacture of any article for sale containing spirit, to use a still or stills in the *bonâ fide* exercise of their profession without taking out a still licence.

Exsiccation.—This name is specially given to the process for the removal of water from crystallised salts. Pharmacopœial examples are found in dried alum and dried ferrous sulphate. The process simply consists in exposing the salts in a porcelain dish to a temperature from 230° to 250° F. over a naked flame or sand-bath. When cold the mass is powdered and preserved in stoppered bottles. These anhydrous salts may be made to reassume their original form by dissolving them in water, concentrating, and setting aside to crystallise.

Desiccation consists in depriving solid bodies of their moisture at comparatively low temperatures. For drying substances in analysis, and for small operations, water ovens as Fig. 75 are generally employed. This illustrates the combined water-bath and oven designed by Mr. Wills, of the Somerset House laboratory. It consists of a hot-water bath, the air-chamber of which measures ten inches wide, seven inches high, and eight inches from back to front, a pair of hot-water funnels, a hot-air box for drying test-tubes, &c., and four openings with covers on the top, for evaporating-capsules. It is provided with a water-gauge beside the door, and is supported by a four-legged stool, and is without doubt a most useful piece of apparatus. To avoid the necessity for frequent refilling a constant level apparatus should be attached as described on page 43. It is impossible to raise the temperature of these ovens higher than that of boiling water, but occasionally it becomes necessary to maintain the temperature within certain narrow limits. For this purpose thermostats controlling the supply of gas to the burner are employed. Fig. 76 shows Bunsen's regulator. In this the temperature is controlled by increasing or diminishing the supply of gas to the burner *d*.

The gas enters by *a*, passes into a narrow tube, which is continued for about two thirds of the length of *b*, and is open at the lower end, where the gas escapes, passing out by the tube *c* to supply the burner *d*. The lower part of the tube *b* is cut off from the upper by a diaphragm containing mercury. A small tube, open at both ends, passes through the diaphragm to the bottom of the mercury. There is a scale and a screw at the upper end of the tube *b*, by which the proper supply of gas can be regulated at the commencement. Supposing the apparatus to be in action as represented by the figure, if the heat becomes greater than is required, the air enclosed above the mercury in the lower chamber expands, drives out the mercury which rises in the tube *b*, and closes the lower end of the continuation of the supply pipe *a*. In order to prevent extinction of the flame the inner supply pipe is either pierced with a small hole which always remains open, or the lower part of the tube is provided with fine slits which permit a slight passage of gas when the bottom is closed. When the heat falls in the bath the air enclosed in the tube *b* contracts, the

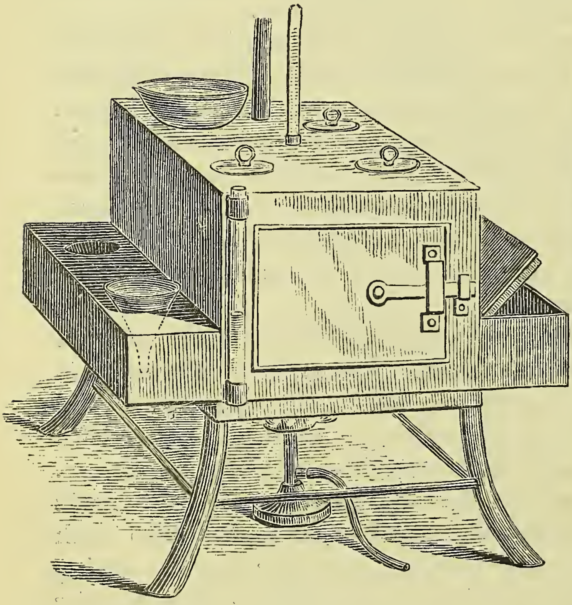

Fig. 75.—Griffin's combined hot-water bath and oven.

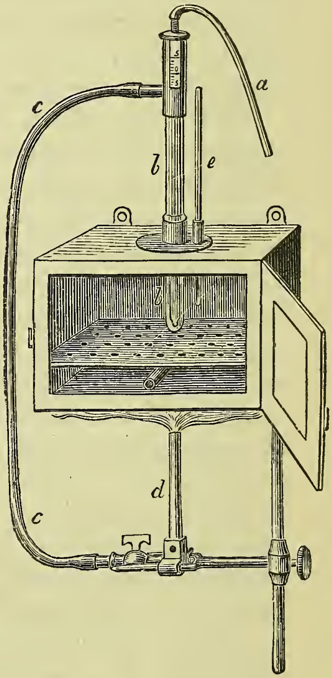

Fig. 76.—Hot-air oven with thermostat.

mercury falls, and a greater supply of gas is admitted to the burners.

When the temperature at which the substance is to be dried exceeds 200° F., hot-air ovens are often used, as Fig. 76. These practically only differ from water ovens in that, in the space between the walls, air circulates instead of water.

Precipitates, &c., after drying in water or air ovens are often placed in desiccators of the form shown in Fig. 77, where they not only cool, but lose the last traces of moisture. In the form shown the drying agent is fused calcium chloride, but sulphuric acid may be used if preferred.

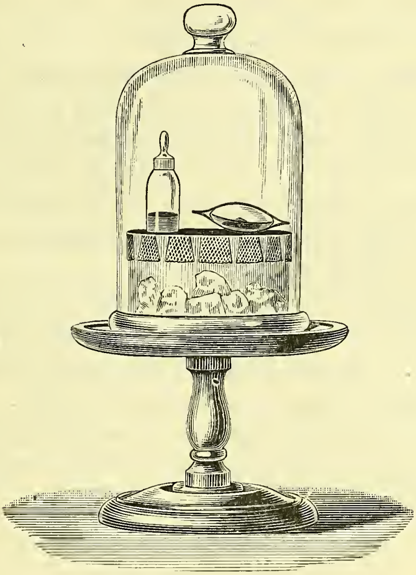

Fig. 77.—Desiccator.

A very excellent form of desiccator is easily made by cutting an ordinary Winchester-quart bottle about halfway, by filing a deep nick, and then leading a crack around the middle by means of a red-hot wire. The sharp edge of the upper half must then be ground perfectly smooth on a glass plate with emery and water. A rubber cork, fitted with a glass tube provided with a stopcock, is then placed in the neck. Now take a shallow ointment jar, and half fill it with fused calcium chloride, cover it with a piece of perforated zinc, and stand under the improvised bell jar. The desiccator is now ready for use, and may be employed in the usual way; but if it is required to hasten desiccation or effect evaporation of small quantities of volatile liquids at ordinary temperatures, as in the assaying of alkaloids, the stopcock may be opened and the glass tube connected with a Korting's or other filter-pump. In this way a vacuum corresponding to twenty or twenty-eight inches of mercury is rapidly attained, in which volatile bodies evaporate at a great rate. To prevent entrance of air the surface of the glass plate must be smeared with soft paraffin.

For drying drugs for powdering, &c., large chambers, heated by steam pipes or brick flues, are made use of. The drugs are exposed in shallow trays placed on perforated shelves, means being adopted for the removal of the air as it becomes laden with moisture. The most convenient temperature for general work of this description is from 100° to 110° F. For drying certain substances injured by heat on a large scale, vacuum desiccators are employed, but their description hardly comes within the scope of this work.

Calcination consists in subjecting a substance to intense heat, with the object of driving off volatile constituents, as the burning of chalk in kilns. In this instance the chalk loses carbonic acid gas, friable calcium oxide remaining in the kiln. Another well-known example of the application of calcination is the pharmacopœial process for the manufacture of heavy and light magnesia from the carbonate.

CHAPTER VI

SUBLIMATION

SUBLIMATION is the operation of raising a solid substance to a state of vapour, and again condensing by cooling; in other words, it is the distillation of volatile solids. The product of sublimation is known as the "sublimate."

The chief sublimates of pharmaceutical interest are ammonium carbonate and chloride, arsenious acid, benzoic acid, camphor, mercurous and mercuric chlorides, and sulphur.

An experiment for the sublimation of benzoic acid may be conducted as follows:—Benzoin in coarse powder is mixed with dry sand and spread evenly over the bottom of an iron dish, which is then covered with coarse filtering-paper pasted round the rim to prevent loss. A conical cap of stiff glazed paper is then fitted closely over the rim, and heat applied by means of a sand-bath until vapours cease to rise. The paper cap must be lifted off occasionally, and the benzoic acid removed from the inside as well as from the surface of the filter, which is rather apt to clog from the presence of moisture and volatile oils. Most of the benzoic acid passes over about 300° F., but towards the end of the operation the temperature may be increased to 400° F. Even then all the benzoic acid is not sublimed, and a further quantity may be obtained by allowing the dish to cool, breaking up the fused mass into small pieces, and again subliming through a fresh piece of filter-paper. The foregoing yields a product of great purity, having a pleasant odour, and very different from that obtained artificially. Artificial benzoic acid is generally prepared by oxidising toluol with chlorine, and heating the compound formed (trichlormethyl-benzene) with water under pressure, when it splits up into benzoic and hydrochloric acids—$C_6H_5CCl_3 + 2H_2O = C_6H_5COOH + 3HCl$. Large quantities are also made by oxidising naphthalin with nitric acid, and converting the phthalic acid thus obtained into the calcium salt, which latter on being heated yields calcium carbonate and benzoate. From the latter benzoic acid is liberated in the usual way by treatment with a suitable acid.

Camphor is easily sublimed from a copper retort provided with a short wide neck, which is luted into a hole cut in the side of a large wooden box lined inside with glazed paper. The vapours, if the temperature has been properly managed, will condense as a beautiful white powder, easily concreting into balls under pressure. Care must be taken that the vapours do not come in contact with a naked flame, as camphor is extremely inflammable.

Of the other sublimates mentioned, some are obtained as by-products, others being manufactured on an enormous scale for use in the arts, and suitable text-books should be consulted for information concerning them.

CHAPTER VII

COMMINUTION

UNDER the head of Comminution (Latin *minus,* less) is included the reduction of drugs and chemicals to small particles by any mechanical means. All substances to be comminuted should be freed from moisture by drying at suitable temperatures (see Desiccation). Small quantities of crystalline salts and neutral principles may be reduced to a state of fine powder in an ordinary Wedgwood-ware mortar. Larger quantities may be dealt with in marble or granite mortars ; the pestles for these mortars (Fig. 78)

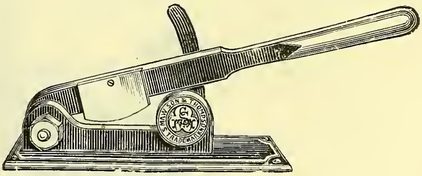

FIG. 79.—Root cutter.

FIG. 78.—Large granite mortar.

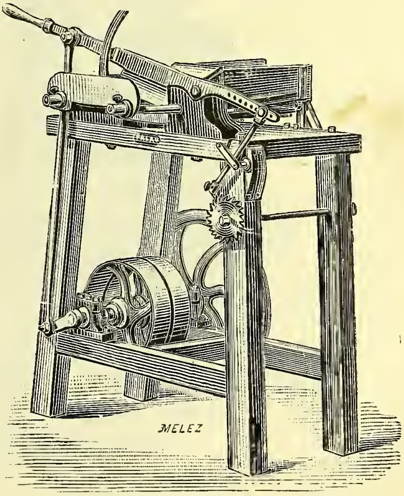

FIG. 80.—Automatic root cutter.

are provided with long shafts for use with both hands, so that considerable pressure may be exerted. Mortars of the size illustrated should be secured on solid supports on the ground-floor to prevent jarring.

Tough and fibrous plant structures may be sliced with a root cutter (Fig. 79) before drying. Fig. 80 shows an automatic root cutter, which may be either worked by hand or by power. The material is placed on a smooth inclined plane, down which it slides to the cutting blades, between which the pieces freely engage themselves, their length being regulated by a stop plate, which can be altered at will. The sliced material after drying is often bruised in metal mortars provided with heavy iron pestles or beaters, the operation being known as contusion. To lessen the labour when worked by hand the pestle may be suspended from the ceiling, or from a beam by two or three of the strong rubber springs used for closing doors. The free ends are fixed at such a height that when hanging loosely the knob of the pestle reaches to within about six inches of the bottom of the mortar.

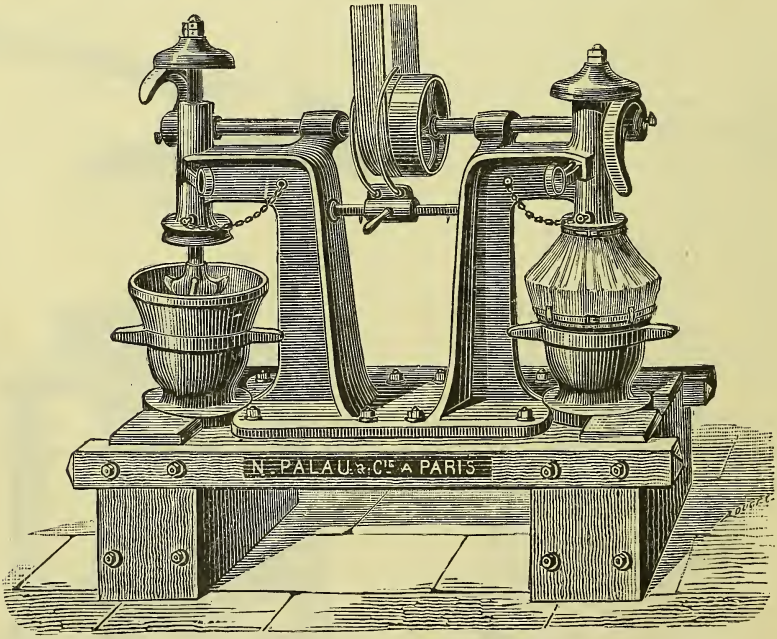

Fig. 81.—Mechanical crushers.

Fig. 81 illustrates a special form of mechanical crusher worked by power. The pestles are of peculiar shape, and are raised by a cog at each revolution of the spindle. The result is that all the bottom of the mortar is successively worked over, the substance operated on undergoing a tearing as well as a beating action. Friable substances may be reduced to powder in mortars of granite or iron. To prevent loss from the fine particles being blown away during the operation the mortar is covered by a circular piece of wood, with a hole in the centre for the pestle, and provided with a broad wooden flange, as Fig. 82.

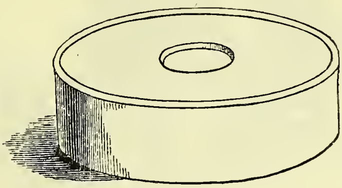

Fig. 82.—Mortar cover.

The chief means employed by pharmacists as well as drug-millers for reducing fibrous drugs to powder is by grinding. Fig. 83 shows a metal disc mill well adapted for general work;

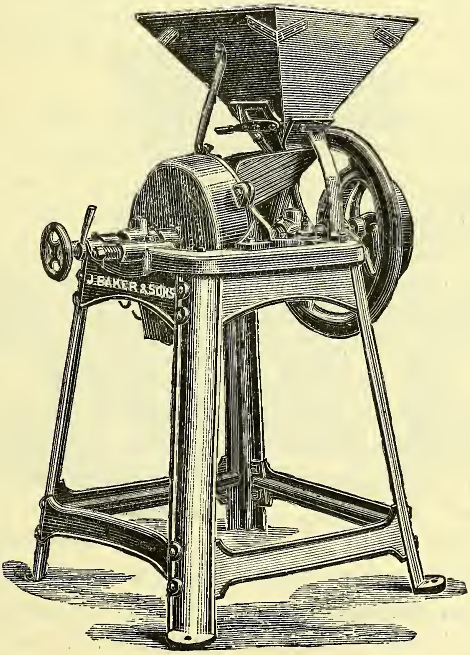

Fig. 83.—Metal disc mill.

the cutting plates are of chilled iron, and are provided with a pair of spiral springs which allow nails or other hard substances to drop through without stopping the mill. The mill is so constructed as to allow a current of air to pass through whilst at work, thereby preventing the material being ground from becoming overheated. Fig. 84 shows one of the most useful mills for druggists, viz. the edge-runner mill. In this a pair of stones revolve inside a shallow iron basin. The material is not only crushed, but actually torn, by the peculiar movement of the stones, each of which revolves on its own axis while both travel round the basin. The material is kept in the path of the stones by suitable arrangements.

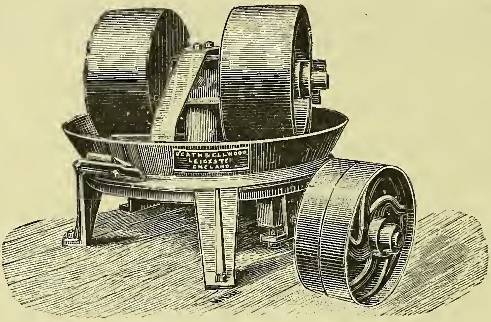

FIG. 84.—Edge-runner mill.

Many drugs are now-a-days reduced to powder in disintegrators. The best of these act by percussion. The material is fed through the side hopper into the grinding chamber, where it falls on the extremity of the beaters, which make about three thousand revolutions a minute. These strike it with such force that it is either pulverised at once or beaten against the serrated chilled iron lining of the chamber. Figs. 85 and 86 illustrate one of the best of these disintegrators. Considerable power is required to drive these machines, as they only act properly when worked at very high speeds; one having a grinding chamber of about two feet in diameter requires from five to eight horse power to obtain the best results. Ordinary frictional grinding mills, such as the American and Universal, operating with two corrugated iron surfaces, require these surfaces to be set close together in order to produce fine powders; the friction then set up soon polishes the sharp iron corrugations, and when this occurs grinding can only be carried on by using great pressure, engendering considerable heat and absorbing enormous power.

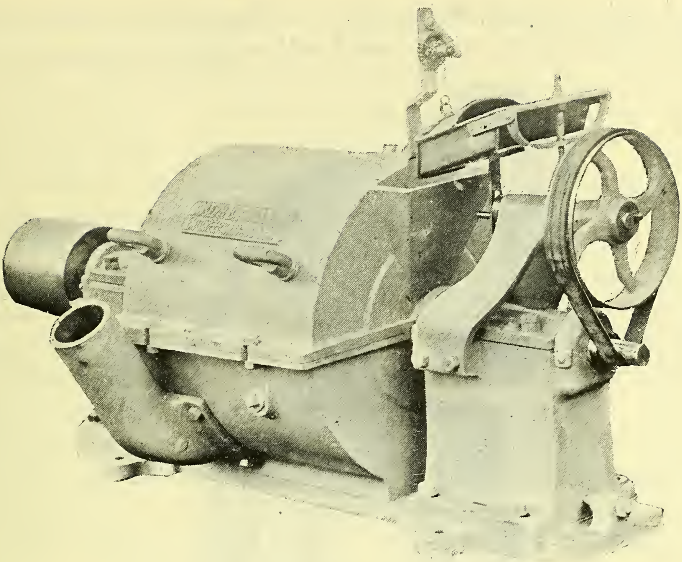

FIG. 85.—Baker's disintegrator.

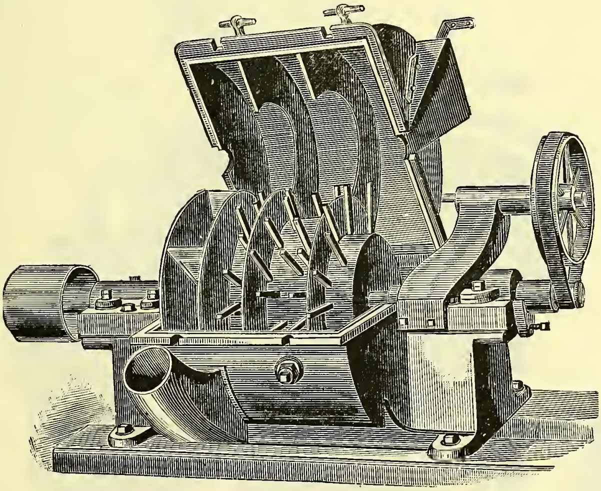

FIG. 86.—Baker's disintegrator (open).

For powdering certain classes of drugs, such as euphorbium, &c., French pot-mills, Fig. 87, are generally used. These, as will be seen from the illustration, consist of a pot made of heavy boiler steel, without any corners for drugs to lodge in, and provided with a closely fitting cover. The material to be powdered is placed in the pot with a steel ball weighing from ten to thirty

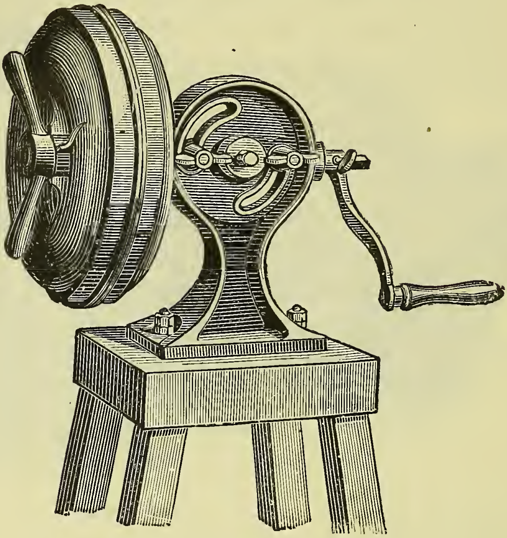

FIG. 87.—French pot-mill.

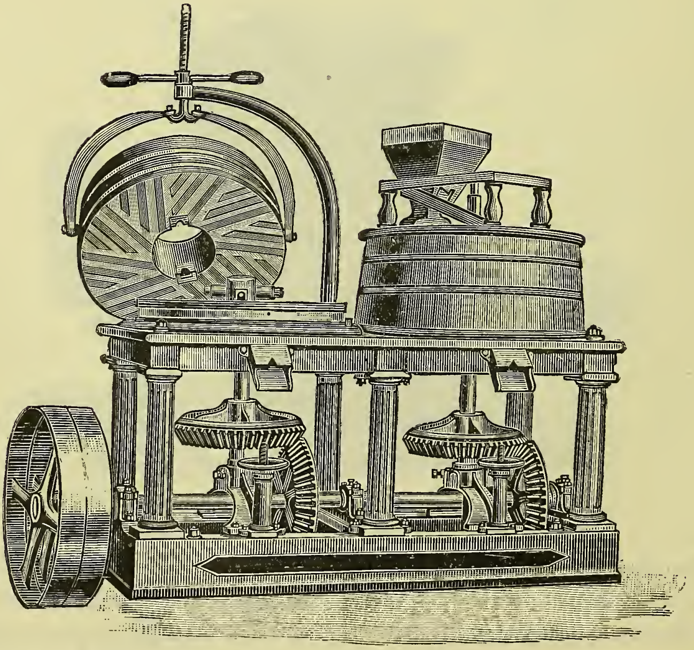

FIG. 88.—Buhr mill.

pounds, and the lid clamped on. The pot is then turned at a speed of twenty-five revolutions a minute; when the machine has been running sufficiently long the mill is brought to rest, and after a little interval the lid may be removed and the powdered substance taken out.

Fig. 88 illustrates Gardner's buhr mill as used for grinding soft fibrous drugs and spices. The upper buhr is stationary. In the illustration the mill on the left hand is raised for the purpose of dressing. The material to be operated upon is fed in through the hopper, and finds its way on to the lower stone, which grinds it against the upper stationary stone. The two stones almost touch at their periphery, and the material cannot escape until it has been reduced to the required degree of fineness.

Milburn's patent "conoidal" buhr-stone mills have perhaps the simplest construction of any stone mills adapted for pharmaceutical grinding, as they are comparatively cheap, and do not require skilled labour to dress the stones. These are illustrated

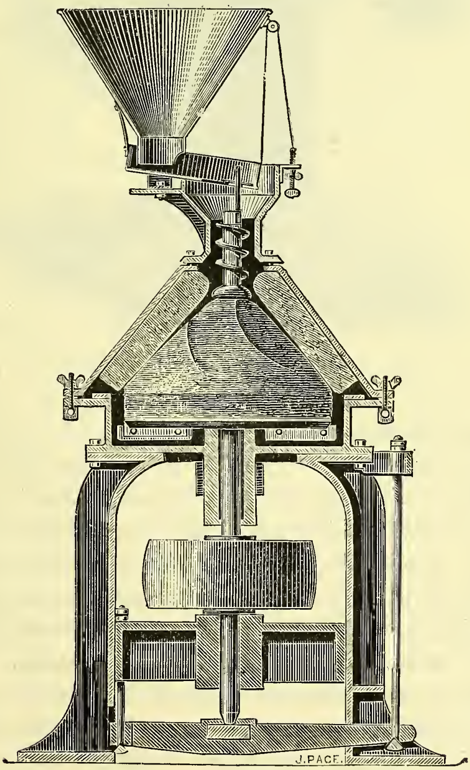

Fig. 89.—Conoidal buhr mill (section).

in Figs. 89 and 90, the former showing the mill in section, the latter as fitted up for use, with a lever arrangement for automatically relieving the stones from nails and other obstructions likely to injure the mill. As will be gathered from the name, these mills are cone-shaped; the upper stone is a hollow cone fixed inside a cover, inside which the lower furrowed cone, which is attached to a spindle, works. The space between the faces increases towards the top, where the material to be ground is introduced through a hopper—the stones only approaching one another closely from one to two inches from the bottom. It is evident from this arrangement that no material can escape from between the stones until it has acquired a minimum grade of fineness, the degree of which is only regulated by the closeness with which the stones approach at their bases. When the stones become

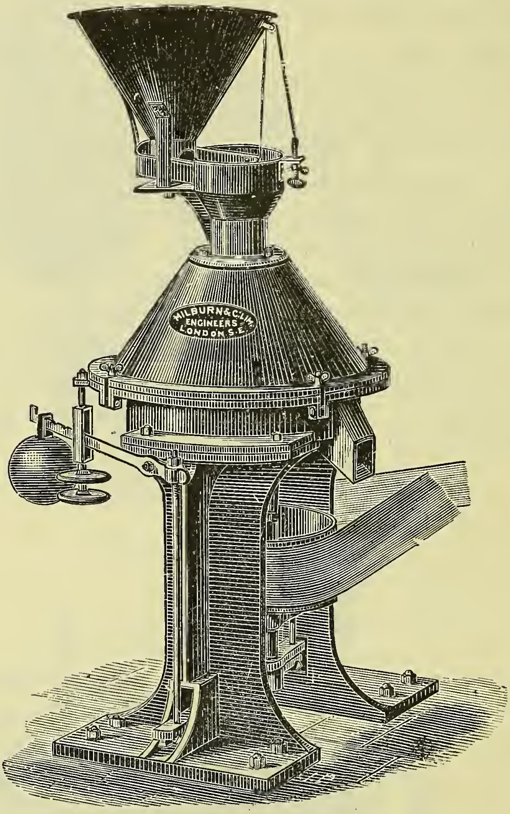

Fig. 90.—Conoidal buhr mill.

too smooth from long use they only require roughening with a mill-bill, which is easily done after loosening the wing nuts and removing the cover attached to the fixed stone.

Sieves.—Although in milling the material has apparently been all subjected to an equal amount of grinding and bruising, yet all the particles are not of the same degree of fineness, therefore to obtain the powder in a nearly uniform state of disintegration it is passed through a sieve of a particular mesh. The degrees of disintegration are represented by numbers ranging from 12 to 180. These numbers indicate the numbers of parallel wires of a definite gauge within a linear inch, forming the meshes of the sieves used. Thus a No. 30 sieve contains 30 wires in a linear inch, or 900 meshes in a square inch. Ingredients required for infusion, &c., are reckoned sufficiently fine if passed through a very coarse sieve, while powdered rhubarb is generally passed through a No. 120 sieve; while an extreme example is seen in powdered boric acid, which for many pharmaceutical purposes is ground until it can be shaken through a silk bolting cloth having about 173 threads in a linear inch. The question of sieves will be again referred to in the article on Powders.

The first operation connected with the grinding of drugs consists in drying them. If in large masses these are cut or broken into small pieces to facilitate the drying, and also to prepare them for the final grinding. When thoroughly dry the substance is either powdered in a mortar or ground in a mill, the powder being sifted from time to time, and the residue which does not pass through the sieve returned to the mill for further treatment, and this method of alternate grinding and sifting continued until the process is finished. The facility with which drugs may be reduced to powder depends upon the care bestowed on their previous drying. If this last operation has been incompletely carried out it will be impossible to produce fine powders. Some drugs, such as squill and aloes, absorb moisture again so rapidly that it becomes necessary to return them to the drying room for some time before the operation can be completed.

Certain drugs, no matter how carefully the drying operation has been performed, can scarcely be reduced to powder by the ordinary methods. Nux vomica and St. Ignatius beans belong to this class. These tough and horny seeds are exposed to the action of steam until they have swelled to about twice their natural size, when they are rapidly dried at a temperature of about 200° F. Other substances which require a little management in powdering are camphor, spermaceti, shellac, &c. Camphor is readily powdered by the aid of a few drops of rectified spirit; spermaceti by the help of a trace of oil. Shellac is powdered fairly easily, but not sifted, and to assist this last operation the powder is slightly wetted with water before being rubbed through the sieve. Phosphorus is obtained in a state of fine division by shaking with warm spirit.

All substances which contain volatile principles lose a portion during the process of drying and powdering, and if the efficacy of the substance depends upon these principles the drying must be conducted at as low a temperature as possible. Thus myrrh, cardamoms, cinnamon, and spices generally lose some of their efficacy in being reduced to a fine powder, which is not the case with such drugs as rhubarb, scammony, ipecacuanha, &c.

When a parcel of a drug has been ground and sifted it does not necessarily follow that the whole powder is in a state of uniformity. Far from this being the case, it is found by experience that the medicinal activity of the powder varies greatly, the last portion of *gruffs* often containing only a trace of the active principles, while the first siftings contain an undue proportion. To remedy this the whole batch of powder should be lightly mixed and then again passed through the sieve.

LEVIGATION

Levigation (L. *lævis,* smooth, and *ago,* I make) is the operation of reducing substances to an impalpable or very fine powder while in a moist condition. Originally this process was often carried out on a porphyry slab, the moistened material being ground beneath a large flat stone held with both hands (Fig. 91). This stone was called a *muller,* and considerable dexterity was required to use it properly. During the operation a circular motion is given to the sweep of the muller, circles gradually becoming smaller being intersected with figures of 8. The pasty substance should be constantly scraped off the edges of the muller and extremities of the slab, and spread in the path of the stone. This process is eminently adapted for the reduction of such substances as red oxide of mercury to a fine powder. Instead of using a slab and muller, shallow mortars with flat-bottomed pestles are sometimes used; but as considerable pressure must be exerted, the pestle is either weighted or connected with a spring.

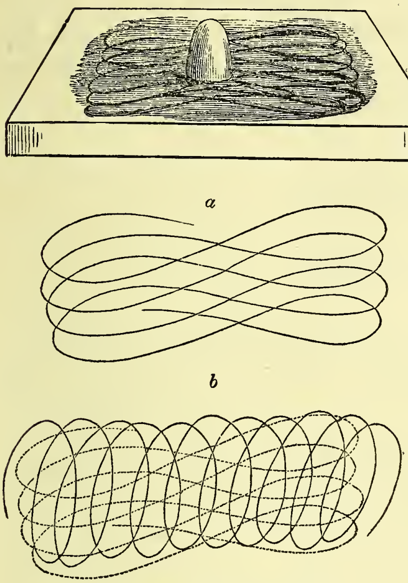

FIG. 91.—Slab and muller.

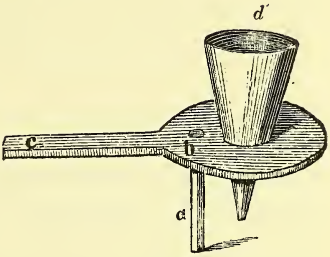

FIG. 92.

ELUTRIATION

Elutriation (L. *e*, and *luo*, I wash) is the name given to the operation of separating the coarse from the fine particles of an insoluble powder, by suspension and partial subsidence in water. Elutriation frequently follows the process of levigation. The material, whether in a pasty or dry condition, is thoroughly agitated with a considerable bulk of water, and set aside to deposit. After a time, depending upon the coarseness and relative gravity, the heavier particles will have subsided; the upper layer, containing the finer and lighter particles still in suspension, is carefully decanted and again set aside to deposit. If desired, the coarse particles are again subjected to levigation, and again elutriated until the whole is brought to a state of fine division, when the mixed deposits are collected, dried, and sifted.

The process of elutriation is made use of in the preparation of calamine, chalk, &c. The little conical masses in which prepared chalk comes into the market are formed by placing the pasty substance in a funnel fixed in a wooden handle, to which is attached a short peg, as Fig. 92. The apparatus is then gently tapped on a slab of chalk or on other porous substance ; each tap causes a conical drop to fall out. The porous block absorbs the moisture before the cones have time to lose their shape.

CHAPTER VIII

EXTRACTION

The chief methods employed for extracting the active principles of drugs are decoction, infusion, maceration, percolation, and re-percolation.

Decoction is applied when the active principles chiefly consist of extractive matter, readily dissolved out but not injured on boiling with water. Besides the official decoctions, examples are seen in the extracts of chamomile and gentian, both of which are evaporated decoctions. (See Decoctions.)

Infusion is also applied for dissolving out extractive matter from vegetable tissues by treatment with water, but the temperature employed is lower. At the highest, boiling water is sometimes poured on the drug, but the temperature rapidly falls; in other instances only tepid or cold water is used. In addition to the official infusions, a modification of the process is employed in the preparation of the extracts of aloes, ergot, liquorice, &c. (See Infusions.)

Maceration.—This is really prolonged infusion, except that alcohol, either strong or dilute, is substituted for water as a menstruum. Frequently the process is combined with that of percolation. As its name implies, maceration consists in steeping the material in the liquid contained in a closed vessel for a definite period, usually seven days, agitating at intervals. The liquid is then strained off, the marc strongly expressed, and the liquids mixed and filtered. Examples of its application are seen in the preparation of many tinctures and liquid extracts.

Percolation.—In the majority of instances percolation is considered the most perfect method for obtaining the soluble parts of drugs. Briefly it consists in allowing a liquid (the menstruum) to slowly trickle through a column of the material in such a way that every solid particle is in turn submitted to the solvent action of the gravitating fluid. Its applications in pharmacy are almost endless.

Re-percolation is a modification of the foregoing, the difference being that instead of submitting the whole bulk of material to the action of the menstruum at once, it is divided into two, three, or more equal portions, and packed in percolators. The menstruum having been poured on the *first* percolator, percolation is proceeded with until a small amount of percolate collects in the receiver. This percolate is then poured on the drug contained in the *second* percolator, and the percolate from this used to exhaust the contents of the *third* percolator. This process is continued, every fresh addition being made to pass through each percolator in turn until the drug is exhausted, or a sufficiency of strong percolate collected. By this means a small volume of liquid may be made to exhaust a comparatively large amount of material, doing away in many instances with the necessity for concentration at the end of the process. A good example of the application of re-percolation is seen in the preparation of liquid extract of belladonna.

The liquid remaining in the marc may be recovered by pressure and used to exhaust the next batch, instead of taking fresh menstruum.

Pressure maceration.—This term may be applied to a process sometimes employed for exhausting certain drugs with a minimum quantity of menstruum, and is exemplified in the official process for syrupus sennæ. In this, senna leaves are moistened by rubbing the leaves with 60 per cent. alcohol, and then immediately pressed up tightly in a press box. As a rule a very little liquor comes away; a further small quantity of menstruum having been poured on, the pressure is somewhat relaxed, with the result that the fluid is instantly sucked up. After macerating for a short time the pressure is increased and the liquor forced out; fresh menstruum then being added, pressure is once more relieved, and this process of alternately sucking up liquid, macerating, and pressing out continued until the marc is exhausted, or a sufficient quantity of extract is obtained. Operating on the pharmacopœial quantities the density of the expressed liquids was—

1st expression . .	sp. gr.	1·068
2nd ,, . .	,,	1·035
3rd ,, . .	,,	1·013

As the specific gravity of 20 per cent. alcohol is ·976, it is evident that the last expression contains but little extractive, consequently it would be useless to continue the extraction.

Percolation

Percolation (L. *per,* and *colo,* I strain) is the name given to the process employed for the extraction of the active principles and soluble matter of drugs by displacement with a suitable liquid. The vessel in which the operation is conducted is called a *percolator,* the liquid used for extraction is known as the *menstruum,* and the product which is collected in a *receiver* is termed the *percolate.* The exhausted material is sometimes called the *marc.*

The Pharmacopœia directs percolation to be carried out as follows :—After moistening the solid material with the prescribed quantity of menstruum, it is set aside in a closed vessel for twenty-four hours. The mixture is then packed, lightly or closely according to the nature of the materials, in a percolator, and the menstruum poured on, care being taken that a layer of liquid is maintained above the level of the material. Percolation is then allowed to slowly proceed until three quarters of the finished preparation has been collected, or until the solid material has been exhausted. The marc is then removed from the percolator and submitted to pressure, the expressed liquid filtered and mixed with the percolate, and sufficient menstruum added to produce the prescribed volume.

The details and minor variations of the processes are left to the judgment of the pharmacist, the only recommendation being the use of a glass or earthenware percolator of such dimensions that a column of solid material at least six times as high as wide is presented to the menstruum. The shape of the percolator may be either cylindrical or conical; if the latter, the lower diameter of the percolator should be not less than half that of the upper.

The process of percolation described in the United States Pharmacopœia, consists in subjecting a substance or substances in more or less fine powder contained in a vessel called a percolator, to the solvent action of successive portions of menstruum, in such a manner, that the liquid as it traverses the powder in its descent to the receiver shall be charged with the soluble poriton of it, and pass from the percolator free from insoluble matter. When the process is successfully conducted the first portion of the liquid or percolate passing will be nearly saturated with the soluble constituents of the substance treated; and if the quantity of menstruum be sufficient for its exhaustion the last portions will be destitute of colour, odour, and taste other than those possessed by the menstruum itself.

The percolator most suitable for the quantities contemplated by the Pharmacopœia should be nearly cylindrical or slightly conical, with a funnel-shaped termination at the smaller end. The neck of this funnel-shaped termination should be rather short, and should gradually and regularly become narrower toward the orifice, so that a perforated cork bearing a short glass tube may be tightly wedged into it from within, and the end of the cork is flush with the outer end of the orifice. The glass tube, which must not project above the inner surface of the cork, should extend about an inch and a half beyond the outer surface of the cork, and should be provided with a closely fitting rubber tube at least one fourth longer than the percolator itself, and ending in another short glass tube, whereby the rubber tube may be so suspended that its orifice shall be above the surface of the menstruum in the percolator, a rubber band holding it in position.

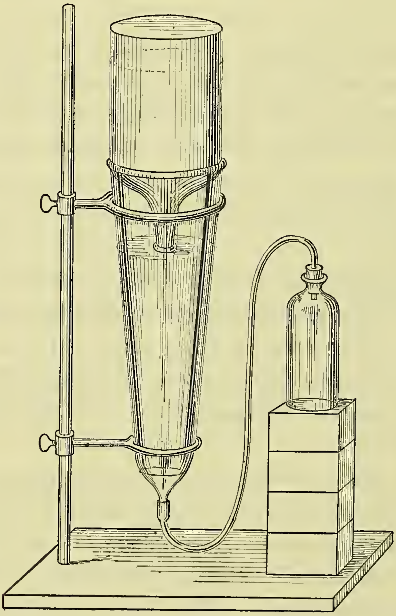

Fig. 93.—Percolation.

The percolator should be adapted to the nature of the drug to be operated upon; for drugs which are apt to swell, particularly when a feebly alcoholic or an aqueous menstruum is employed, a *conical* percolator is preferable. A *cylindrical* or only slightly tapering percolator may be used for drugs which are not liable to swell, when the menstruum is strongly alcoholic, or when ether or some other volatile liquid is used for extraction. The size of the percolator selected should be in proportion to the quantity of drug extracted. When properly packed in the percolator, the drug should not occupy more than two thirds of its height. The percolator is best constructed of glass or stoneware, but unless otherwise directed may be made of any suitable material not affected by the drug or menstruum.

A small tuft of cotton is directed to be placed in the neck of the percolator above the cork, and a layer of clean dry sand poured upon its surface. The objection to cotton is that when wetted it falls together, and under pressure becomes clogged, the liquid refusing to filter through it. Glass wool is better for the purpose, retaining its spongy condition even under considerable pressure. "Slag wool" is equally good for this purpose, and is much cheaper, the price per pound being less than the price per ounce of glass wool. It is commonly used as a packing to retain the heat of steam boilers and pipes. Sometimes the slag wool contains a number of glassy beads. If desired, these may be got rid of by washing the wool in a large volume of water, when the beads sink to the bottom. Asbestos has been recommended, but it has no advantage over slag wool, and is much more expensive.

The powdered substance to be percolated (which must be uniformly of the fineness directed in the formula, and should be perfectly air-dry before it is weighed), is put into a basin, the specified quantity of menstruum is poured on, and thoroughly stirred with a spatula or other suitable instrument until it appears uniformly moistened. The moist powder is then passed through a coarse sieve, No. 40 powders and those which are finer requiring a No. 20 sieve, whilst No. 30 powders require a No. 15 sieve for this purpose. Powders of a less degree of fineness usually do not require this additional treatment after moistening. The moist powder is now transferred to a sheet of thick paper, and the whole quantity poured from this into the percolator. It is then shaken down lightly, and allowed to remain in that condition for a period varying from fifteen minutes to several hours, unless otherwise directed; after which the powder is pressed by the aid of a plunger of suitable dimensions, more or less firmly in proportion to the character of the powdered substance and the alcoholic strength of the menstruum, strong alcoholic menstrua as a rule permitting firmer packing of the powder than the weaker. The percolator is now placed in position for percolation, and the rubber tube having been fastened at a suitable height, the surface of the powder is covered by an accurately fitting disc of filter-paper or other suitable material, and a sufficient quantity of the menstruum poured on through a funnel reaching nearly to the surface of the paper. If these conditions be accurately observed the menstruum will penetrate the powder equally until it has passed into the rubber tube, and has reached in this a height corresponding to its level in the percolator, which is now closely covered to prevent loss by evaporation. The apparatus is now allowed to stand at rest for the time specified in the formula. To begin percolation the rubber tube is lowered, and its glass end introduced into the neck of a bottle previously marked with the quantity of liquid to be percolated if the percolate is to be measured, or of a tared bottle if the percolate is to be weighed. By raising or lowering this receiver the rapidity of percolation may be increased or decreased as may be desirable, care being taken, however, that the rate of percolation, unless the quantity of material be largely in excess of the pharmacopœial quantity, shall not exceed the limits of ten to thirty drops in a minute. [The rate or speed of percolation may be increased in proportion to the amount of drug percolated.] A layer of menstruum must constantly be maintained above the powder, so as to prevent the access of air to its interstices, until it has all been added, or the requisite quantity of percolate has been attained. This is conveniently accomplished, if the space above the powder will admit of it, by inverting a bottle containing the entire quantity of menstruum over the percolator, in such a manner that its mouth may dip beneath the surface of the liquid, the bottle being of such shape that its shoulder will serve as a cover for the percolator.

The object in moistening the drug is to allow it to swell before being packed in the percolator, thus avoiding blocking. In nine cases out of ten if the drug were packed in a dry state, or even before its tissues had fully expanded from the imbibition of moisture, percolation would proceed very slowly, or perhaps with some drugs cease altogether. Too much care cannot be exercised in packing the material evenly, and for this purpose a plunger of relatively large size should be used. If unevenly packed the menstruum will select the course that offers least resistance, the more tightly packed portions being almost unaffected by the solvent. If a glass percolator is used the descent of the menstruum can be watched. The reason so much stress is laid upon the necessity for keeping a layer of liquid above the surface of the solid material is that if allowed to run dry, air enters and fills the interstices, and if left long enough, cracks and channels are formed, down which the menstruum trickles without coming in contact with the bulk of material at all. To obviate any danger of this occurring there is no simpler device than that described in the United States Pharmacopœia. A percolator of such a size is selected, that when the material has been properly packed about one third of its depth should remain unoccupied. The menstruum may then be poured down a glass rod to avoid disturbance of the upper layer of powder, and a flask containing the remainder of the menstruum inverted over the percolator, taking care that the mouth of the flask just dips beneath the surface of the liquid. The degree of fineness to which the material must be reduced varies greatly. Drugs of a loose, spongy nature, such as gentian or rhubarb, need not be passed through a finer sieve than a No. 30 ; while others of a horny nature, such as the seeds of nux vomica, are required to be in the very finest powder to ensure complete exhaustion. Again, the degree of firmness with which a drug may be safely packed depends largely upon the character of the menstruum to be used. If the latter be only feebly alcoholic, and especially if it happens to consist of water only, the packing must be much more lightly done than if the liquid consisted of strong alcohol or other volatile solvent. The nature of the menstruum also influences the shape of the percolator to be selected. If an aqueous menstruum is to be used, the percolator should be distinctly conical to allow of the lateral and vertical expansion of the material as its tissues become saturated. If, on the other hand, the menstruum is strongly alcoholic, a cylindrical percolator is to be preferred, as a longer column of material may be exposed to the solvent without risk of clogging.

Recovery of spirit from marcs.—After exhaustion the material may still contain considerable quantities of spirit. All residues should therefore be retained and stored in a stone jar with a closely fitting bung for future treatment. The usual method of dealing with marcs when a sufficient quantity has accumulated is to place them in a still with a considerable volume of water, and distil as long as any alcohol comes over in the distillate, which may afterwards be concentrated by re-distillation and purified in the usual way. In the 'Year-book of Pharmacy' for 1895, Bird describes an inexpensive apparatus for the recovery of the alcohol from tincture marc without addition of water, thus saving subsequent re-distillation. The details will be readily apparent from the following sectional sketch. The lower part of the apparatus forms the water-bath; it is of large area and very shallow, and is furnished with a side tube for a thermometer. The condensing cover is a circular water tank having the bottom dished up in a cone, the edge of which terminates in a V-shaped trough round the inner edge of the water-bath. This trough is cooled by a cold-water jacket underneath, and is inclined so that all liquid runs out by the tube leading from it. The water-pipes are so arranged that a current of cold water passes first round the water jacket of the collecting trough, and then into the condensing cover which rests on a flange in the water-bath. The joint is secured by luting. It will be noticed that the condensing surface is very near the material from which alcohol is to be recovered, and the whole construction of large area and shallow, the dimensions being—diameter 18 inches, depth of water-bath 2 inches, height of cone in centre 2 inches. The marc having been spread evenly over the bottom of the water-bath, the condensing cover luted on, and a small stream of cold water turned into the apparatus, the application of heat is quickly followed by the condensation of spirit. This takes place at a remarkably low temperature. When dealing with a proof spirit marc, for instance, a fairly rapid succession of drops falls from the delivery tube at 80° F., and at 140° F. the greater part of the spirit may be recovered.

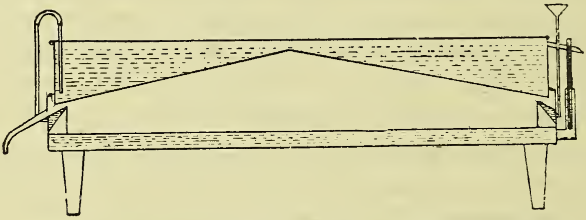

Fig. 94.—Apparatus for recovering alcohol from marcs.

Upward displacement by water is also occasionally resorted to for the recovery of the alcohol. Although theoretically water should easily displace spirit in a spongy material, owing to the difference in gravity, yet it is found by experiment that results vary con-

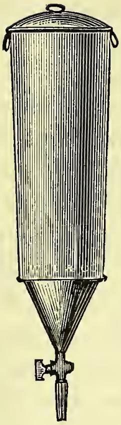

Fig. 95.—Cylindrical percolator.

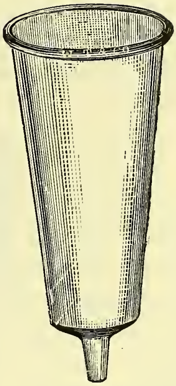

Fig. 96.—Conical percolator.

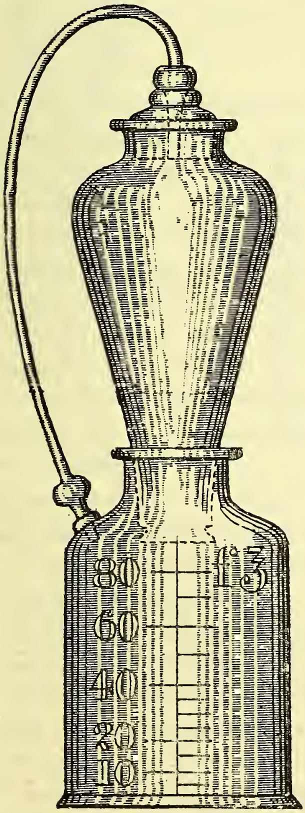

Fig. 97.

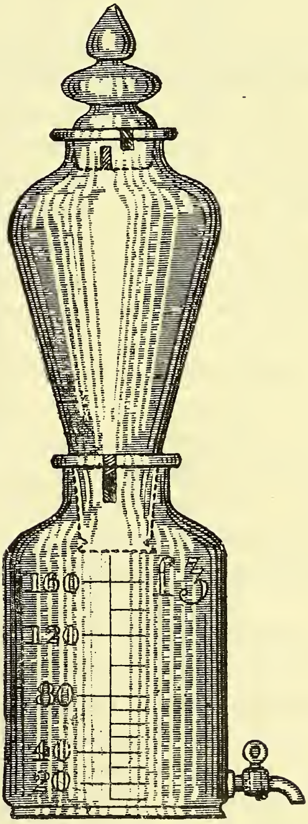

Fig. 98.—York glass percolator.

siderably, much alcohol being wasted owing to the two liquids mixing.

Various forms of percolating apparatus.—When working with small quantities of material glass percolators are desirable; these may be cylindrical as Fig. 95, or conical as Figs. 96, 97, and 98. Perhaps one of the most useful of its kind, and one of the most largely used by retail pharmacists in this country, is the York Glass Co.'s percolator (Fig. 98). Their original form consisted of two glass vessels fitted together as shown in Fig. 97. The lower vessel or receiver had two openings, a wide one for the ground neck of the percolator and a second narrow one for the escape of the displaced air, which passed up a flexible tube into the perforated stopper. In the present form the flexible air-tube has been done away with, the escape and entrance of air being provided for by an ingenious arrangement of grooves cut in the fitting parts, and for convenience in drawing off the percolate a glass tap is fitted to the receiver. The groove in the stopper extends a little more than halfway down the ground surface; that in the corresponding upper part of the percolator extends for the same length upwards; consequently when the two grooves are in line a small air-passage is formed, but when the stopper is turned either to the right or left the grooves do not affect the air-tight character of the stoppering. The grooves in the other fitting surfaces are precisely similar to those just described. Fig. 98 shows the groove of the stopper in such a position as to effectually prevent escape or entrance of air, that of the lower junction arranged so as to allow the displaced air to escape from the receiver. It will be obvious that when each pair of grooves is in a straight line percolation will proceed freely, as air will pass into the percolator and out of the receiver, whereas if the grooves are turned out of line percolation will cease. If, therefore, all the joints are made air-tight maceration of the solid substance goes on, but no percolation takes place, providing a practically perfect apparatus of its kind.

For the preparation of very small quantities of tinctures by percolation, excellent percolators may be made by cutting the bottoms off champagne bottles. This is an operation requiring some little skill. One method is to file a deep notch on the side of the bottle about an inch from the bottom, and then lead a crack in the required direction by a red-hot nail. An easier and quicker method, which is frequently successful, is to stand the perfectly dry bottle on a very hot sand-bath for about a minute, remove it, and before the heat has time to spread, rapidly pour in a little cold water by means of a funnel reaching nearly to the bottom. As a general rule the bottom of the bottle comes away quite cleanly; the secret of success consists in making the bottom of the bottle very hot, and in pouring the water taking care that none splashes against the sides. The sharp edge is then ground smooth on a sheet of glass with emery and water.

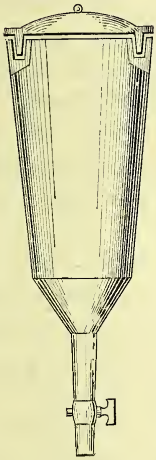

Fig. 99.—Percolator with water-seal cover.

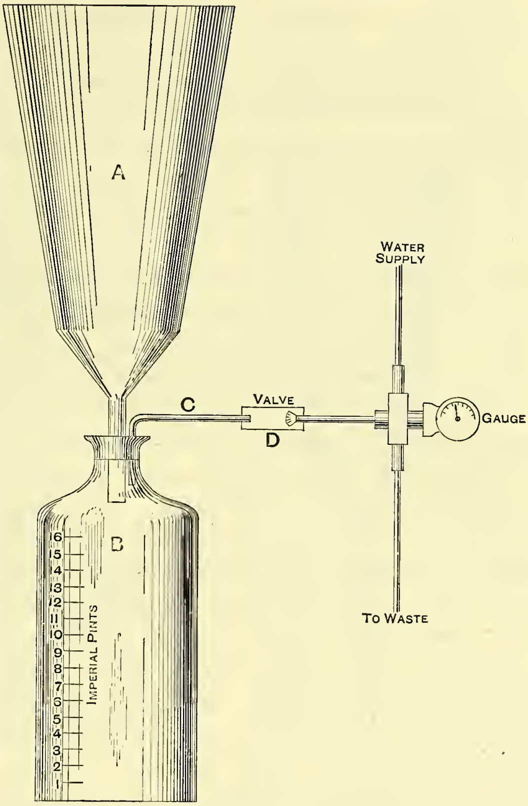

Fig. 100.—Percolator connected to water-pump.

To prevent loss by evaporation when a very volatile menstruum is employed, a stoppered glass percolator of the form shown in Fig. 98 may be used, or if large quantities are under operation a tinned copper percolator with a cover fitting into a water or mercury seal, as Fig. 99. The method of construction is easily seen from the diagram, the apparatus consisting of an ordinary conical or cylindrical percolator, with a hollow rim at the top containing either mercury or water, into which the loosely fitting flanged cover drops.

Percolation under pressure.—It occasionally happens that a substance has to be exhausted by percolation, which, no matter what precautions are taken in grinding, moistening, and packing, becomes so impacted as to unreasonably delay or altogether stop the process. Recourse is then had to pressure-percolation. It is obvious that if the pressure exerted upon the surface of the liquid in the percolator be increased, the liquid will be forced to percolate through the material at a greater rate. The pressure may be increased in several ways, the most simple being to increase the height of the column of menstruum above the surface of the material. When the volume of liquid is insufficient for this purpose the air pressure may be increased. There are two ways of doing this; either by exhausting the air from the receiver by a pump, or by forcing air under pressure into the upper portion of a percolator provided with an air-tight lid.

Fig. 100 shows an apparatus in which a water-pump is employed for creating the vacuum. A is an ordinary glass percolator, B the receiver. The receiver is fitted with a good bung pierced with two holes. The exit tube from A passes through one of the holes, while a piece of glass tubing (C), of about one eighth of an inch or less bore, is inserted in the second. The joints are made tight by painting with strong gelatin solution containing 5 per cent. of glycerin. The glass tube (C) is connected with thick-walled rubber tubing to a Korting's or Geissler's filter-pump. As soon as water runs through the pump, air is drawn out of the receiver, the percolate in most cases beginning to drop at once. With a Korting's pump an almost complete vacuum can be maintained for any length of time, provided the joints remain tight. If the receiver is of thin glass it is not advisable to create a vacuum sustaining more than ten to fifteen inches of mercury, otherwise there is considerable danger of the vessel collapsing under the atmospheric pressure. A glass valve must be inserted between the receiver and the pump, as shown at D, to prevent any possibility of water from the tap being sucked back into the receiver should the pressure in the main be lowered from any cause. To construct the valve, take a short length of glass tubing of fairly wide bore, and fit with rubber plugs. Through one pass a narrow glass tube and connect to the filter-pump; through the other pass a similar tube, but tie over one end an infant's feeding-bottle teat pierced with leech-bites, and connect the other end to the receiver. As soon as the pump is started a partial vacuum is formed, and air drawn through; but should the pressure at the pump fall, no water can be drawn into the receiver on account of the cuts in the teat being pressed tightly together. If a constant supply of water is not available to work a filter-pump, a partial vacuum may be obtained by the aid of an aspirator arranged as Fig. 101.

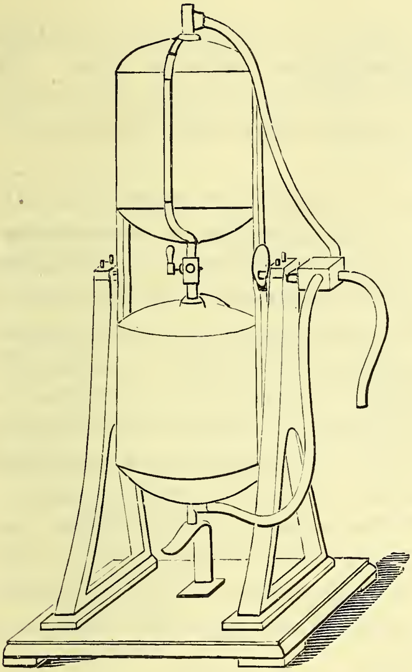

Fig. 101.—Double reversible aspirator in frame for continuous use.

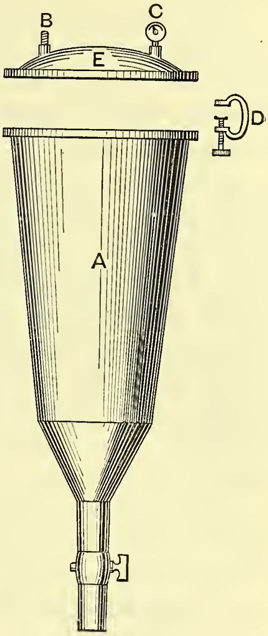

Fig. 102.—Pressure percolator.

The upper cylinder is filled with water and the air-tube connected with the receiver. As soon as the water is allowed to flow into the lower cylinder a partial vacuum is created in the upper one, and air is sucked in from the receiver.

Fig. 102 shows a very simple pressure percolator, which can be fitted up at very small cost. A is an ordinary tinned copper percolator with a broad flange at the top instead of the usual rounded rim; the lid (E) has a corresponding flange, and is held in place by four strong clamps (D), a soft rubber washer between the two metal surfaces serving to make the joint air-tight. An ordinary pneumatic-tyre valve is soldered into the lid at B, and, if desired, a cycle pressure gauge at C. All that now remains to be done is to screw a cycle pump on the valve (B), and force air into the upper part of the percolator until the desired pressure is attained.

The receiver of a percolator may be of any convenient shape or dimensions, but should preferably be made of glass. All receivers should, however, be accurately graduated, either temporarily with a paper mark, or permanently with a diamond or hydrofluoric acid.

Exhaustion.—Many drugs are directed to be percolated until exhausted. Absence of colour in the percolate is no indication, as the active principles of many substances are extracted long before the percolate becomes colourless. In the case of drugs possessing a markedly bitter taste, as cascara and quassia, exhaustion is indicated by the absence of bitterness. Alkaloidal drugs may be considered exhausted when the percolate no longer turns cloudy on the addition of Thresh's or Mayer's reagent. The Pharmacopœia directs cinchona to be percolated until the percolate no longer yields a precipitate with ammonia.

Note.—As an illustration of the process of percolation, one pint of compound tincture of rhubarb was started, and the percolate collected in fractions of 4 fluid ounces. The ingredients were first macerated 48 hours,—afterwards, 24 hours' maceration was allowed between each fraction :

1st fraction was very dark coloured and had a sp. gr. of	·986
2nd fraction, still dark coloured, had a sp. gr. of	·931
3rd fraction, much lighter in colour, sp. gr.	·918
4th fraction (of 6 ounces) nearly colourless, had a sp. gr. of	·915
The menstruum (60 per cent. alcohol) had a sp. gr. of	·913

CHAPTER IX

DIALYSIS

Dialysis (from the Greek *dia,* from; and *lusis,* a loosing) is a process for the separation of bodies based on their unequal diffusibility. Dialysis was first discovered by Graham, and was described by him in his memoir on "Liquid Diffusion applied to Analysis" in the 'Transactions of the Royal Society' for 1861. Ten per cent. solutions of the following substances were subjected to dialysis for twenty-four hours, and the amounts of each substance which passed through the septum bore the following relations to one another :

Gum arabic	4
Starch-sugar	266
Cane-sugar	214
Glycerin	440
Alcohol	476
Sodium chloride	1000

Graham's dialyser consists of two gutta-percha hoops, one of them 2 inches deep, the other 1 inch deep. The 2-inch hoop is slightly conical, and the 1-inch hoop fits over the smaller end of the 2-inch hoop. The parchment paper that forms the bottom must be about 3 inches wider than the small end of the 2-inch hoop.

To prepare the dialyser the parchment paper is soaked for a few minutes in water and stretched evenly over the smaller end of the 2-inch hoop, and strained tightly by pushing on the second hoop. The paper must be pressed smoothly round the outside of the hoop, and the bottom must be quite flat and even, and quite free from holes. To test this, place some distilled water in the dialyser to the depth of about half an inch, and stand it on clean white blotting-paper. If any dark or wet spots appear, they indicate the existence of small holes. To close such holes, paint them over with white of egg, put on a small patch of parchment paper, and iron the patch with a hot iron. This coagulates the albumen, fixes the patch, and closes the hole (Fig. 103).

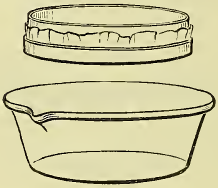

Fig. 103.

The dialyser being prepared, the liquid to be operated upon is introduced to a depth of not more than half an inch, and the apparatus floated on distilled water contained in a flat basin. A dialyser of 12 inches diameter will hold about 30 ounces of liquid. In the course of time a more or less complete separation of the component parts of the mixture will be effected; thus, if a solution of arsenious acid mixed with bread paste is subjected to dialysis, in a few days most of the poison will pass through the diaphragm into the distilled water, from which it may be recovered in the usual way. Substances such as arsenic, salt, &c., which are capable of diffusion, are called *crystalloids,* in contradistinction to those incapable of assuming the crystalline form, such as starch and gelatin, and which are known as *colloids.* The term *dialysate* has been proposed for the residue left on the diaphragm after dialysis, the solution of the crystalloids being known as the *diffusate.*

Dialysis is made use of in the process for Liquor Ferri Dialysatus. Solution of ferric chloride is saturated with freshly precipitated ferric hydrate, and the filtered liquid placed in a dialyser. Very little iron passes through the septum, and dialysis is continued until the diffusate gives no reaction for chlorides. The colloid matter left at the end of the operation has scarcely any taste of iron; it is neutral to litmus paper, and does not give any precipitate with potassium ferrocyanide unless previously warmed with hydrochloric acid; the specific gravity should be about 1·407.

If the operation of dialysis is to be successful the following conditions must be adhered to. The surface of the liquid in the dialyser should not be below the level of the water in the outer vessel, otherwise the bulk of the dialysate becomes unnecessarily increased. In fact, the under side of the dialyser should be only *just below* the surface of the water. If a constant stream of water cannot be made to pass through the outer vessel the contents should be changed at least twice a day, so as to prevent accumulation of soluble salt in the diffusate. Should this latter be allowed to take place dialysis proceeds very slowly, and ultimately stops when equilibrium has been established between the amount of crystallisable matter held in solution by diffusate and dialysate respectively.

CHAPTER X

EXPRESSION

Many pharmaceutical operations require the removal of small quantities of liquids from relatively large bulks of solid material. When this cannot be accomplished by filtration (q. v.) the moist material is squeezed in a machine called a press, and the liquid forcibly expelled. This is termed *expressing*. When working on small quantities screw presses are generally used, while for very large quantities, or when great pressure is required, hydraulic presses are employed.

There are two kinds of screw presses—one having two screws, the other only a single one. In the former the screw acts horizontally, the bag being placed in a vertical position. The screw in the latter is vertical, and effects a downward pressure on the bag, which is placed horizontally. Of late years the double screw press has gone out of fashion, yet it has many advantages over the single screw pattern. In the first place, the press bag is easily placed in the required position, equality of pressure being easily regulated should one side of the bag be thicker than the other; moreover the expressed liquid runs down the vertical sides of the press plates and from the sides of the bag without impediment; and last, but not least, the power of the press is exerted in the most advantageous manner, the male screw being fixed while the female screw or nut is moved. The principal objection to the single screw pattern is that the press-block attached to the screw frequently loses its horizontal position, being turned on one side in consequence of defective packing of the material, so that it sometimes becomes necessary to stop in the middle of an operation, unscrew the press, and rearrange the bag. In this press the male screw alone moves, while the female screw or nut remains stationary. Under these circumstances the screw suffers great torsion, frequently becoming bent and forced out of the perpendicular.

In all operations requiring the use of a press, the pressure should be increased only very slowly; after a time as the liquid drains away more force may be used, until as the material becomes dry, it may be squeezed as strongly as possible without rupturing the press cloth.

Screw presses.—Fig. 104 illustrates the ordinary form of hand press, which is operated by a vertical screw fitting into a recess at the top of the plunger. The body of the press consists of a tinned iron cylinder provided with an opening for the escape of the expressed liquid. Fitting snugly inside this is a second cylinder, perforated with numerous holes. The wet material is wrapped in a cloth and placed on the perforated bottom of the press, the plunger being screwed down as strongly as desired. Fig. 105 shows a double-action tincture press. This is a much more powerful machine than the preceding. After the material has been placed in position the plunger is screwed down as far as it will go by the top lever; greater pressure is then exerted by the worm and wheel action. Screw presses can be made of

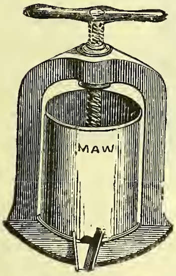

Fig. 104.—Tincture press.

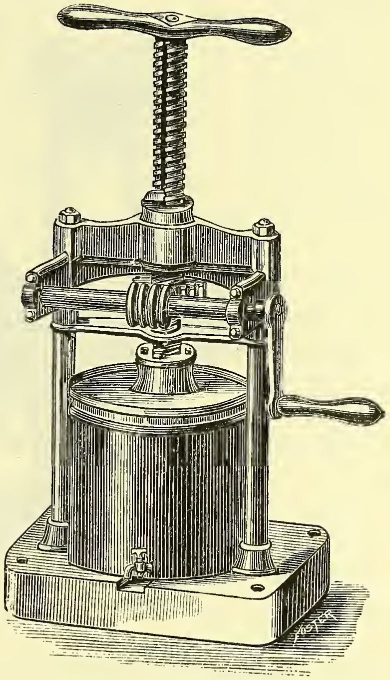

Fig. 105.—Double-action tincture press.

almost any power, and for some purposes are preferred to hydraulic, as there is no possibility of leakage.

The hydraulic press is the most powerful of all presses. It depends for its action on the application of Pascal's law of the equality of pressures. Pressure exerted anywhere on a mass of liquid is transmitted undiminished in all directions, and acts with the same force on all equal surfaces, in a direction at right angles to those surfaces. An approximate verification of this statement may be effected by taking two cylinders of different diameter and connecting them with a tube, the whole being partly filled with water. Each cylinder is provided with a closely fitting piston. Let the area of the larger piston be fifty times that of the smaller. That being assumed, let a two-pound weight be placed upon the small piston; this pressure will be transmitted to the water and to the large piston, and as this pressure amounts to two pounds on each portion of its surface equal to that of the small piston, the large piston must be weighted with one hundred pounds to counterbalance it. It is important to observe that in speaking of the transmission of pressures to the sides of the containing vessel, these pressures must always be supposed to be perpendicular to the sides. An ordinary hydraulic press consists

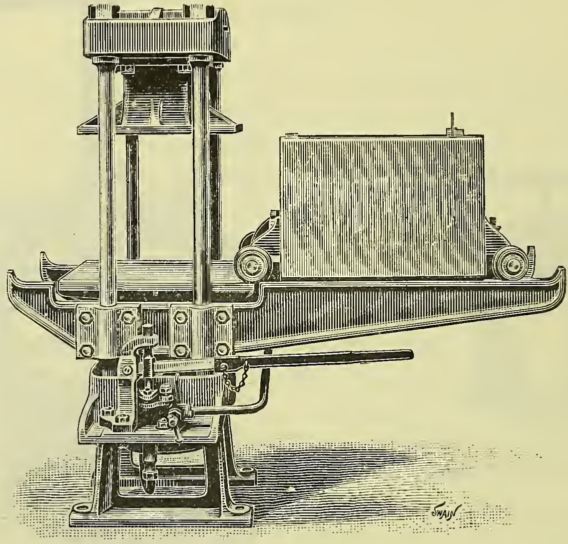

FIG. 106.—Modern hydraulic press.*

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* Bennett, Sons, and Shear's patent.

of a powerful frame fixed on a base, and having a very massive head, which is secured in its place by bolts and nuts. Inside this frame is an iron cylinder containing the ram which carries the bed-plate. The cylinder is connected with the force-pump by a pipe of small bore and thick sides. The press plates are kept in position by guiding rods. When the pump is set going the water enters the cylinder, and forces the ram upwards in accordance with the law previously stated. The pressure must be increased very gradually to avoid bursting the bags. A safety-valve is fixed at one side of the press to relieve the pressure on the cylinder should it be over-pumped. Fig. 106 is a convenient form of press for pharmaceutical use. Instead of placing the material between plates it is laid in a press-box mounted on wheels, arranged to run out on rails for charging and emptying. The press-box is fluted down its sides, and is fitted with a perforated lining to permit of the ready escape of liquid.

In using the presses described, some means are usually required for confining the material during the process of expression. Frequently the substance may be put directly into the press-box, but many substances require to be enclosed in a press cloth or bag previous to introduction into the press. Small tincture presses are sometimes constructed to be used alone, but with all large presses, cloths or bags are used.

Press-bags are very commonly made of horsehair cloth, a material which possesses great strength and durability, yet does not absorb liquids to the same extent as linen or woollen bags. Horsehair bags are, moreover, very readily cleansed. Strong canvas or unbleached linen cloth is sometimes substituted for horsehair, but is inferior in strength, although closer in texture and more flexible. Many substances can be effectively pressed if simply enclosed in the coarse material commonly known as cheese-cloth; but no matter what material is used, the operation can only be successfully conducted by applying the pressure gradually. If the substance is very soft and pulpy the pressure must be exerted very cautiously, for according to the law of hydrostatics the force is communicated equally in every direction, and the bag will inevitably give way if much force be exerted at first. When part of the liquid has been pressed out the pressure may be increased until ultimately, as the contents of the bag become nearly solid, the full force of the press may be safely exerted.

As previously stated, the pressure exerted by a hydraulic press depends upon the relation of the superficial area of the plunger of the pump, to the area of the ram. Suppose for instance, the length of the lever is 30 inches, the distance between the fulcrum and the piston 3 inches, and the power applied equivalent to 100 lbs. Then $\frac{100 \times 30}{3} = 1000$ lbs.; if the area is 50 times that of the plunger of the ram, then the total lifting capacity will be 1000 × 50 = 50,000 lbs.

CHAPTER XI

FILTRATION

Filtration is the process by which liquids are separated from substances mechanically suspended in them ; it is also resorted to for the separation of colouring matters, *e. g.* the decoloration of alkaloidal solutions by filtering through animal charcoal. Various substances are employed as filtering media, the chief being, cotton and linen fabrics, woollen cloth (flannel and felt), absorbent cotton, glass wool, slag wool, kieselguhr, powdered glass, sand, and charcoal.

The simplest application of filtration in pharmacy is the removal of suspended matter from aqueous and alcoholic solutions. This is generally accomplished on the small scale by folding a circular piece of fine unsized paper across its diameter, then again at right angles, and opening it out in the form of a hollow cone, Figs. 107 and 108 ; this is then placed in a funnel and the liquid to be filtered poured gently in, taking care that the stream is directed against the side and not on the apex of the filter. Frequently however, filtration proceeds very slowly, owing either to the faulty shape of the funnel, or to the filtering-paper lying too closely against its sides. The triangle formed by the sides of a

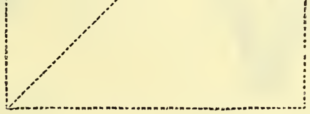

Fig. 107.

Fig. 108.

funnel and a line joining them should be equilateral, so that circular filter-papers folded as directed should fit accurately, as Fig. 109.

The funnels usually supplied by the makers are sometimes ribbed or fluted inside to facilitate the flow of the filtrate. The ribs or flutings, however, are not nearly prominent enough. To be of any service they should be in comparatively high relief, as shown in Figs. 110 and 111. These funnels are moulded uniformly to an angle of 60°, and possess either prominent spirals or deep corrugations, thus offering very few points of contact with the paper, so that filtration is very rapid. It would be an advantage if the funnels were made with a projection on the stem, thus permitting ready escape of the displaced air when filtering into narrow-necked vessels. To prevent splitting the necks of these

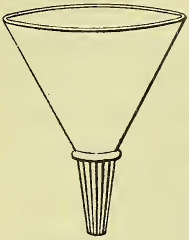
FIG. 109.

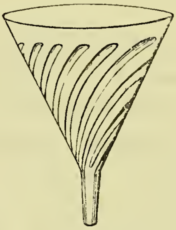
FIG. 110.

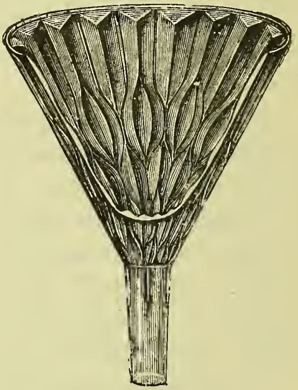
FIG. 111.

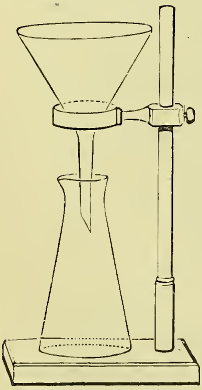
FIG. 112.—Funnel support.

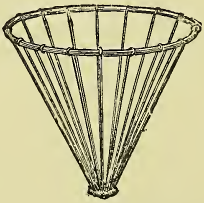
FIG. 113.—Filtering rack.

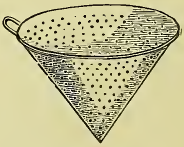
FIG. 114.—Zinc filtering cone.

latter it is frequently necessary to use funnel supports, as Fig. 112. These may be made of either wood or iron, but in either case the foot should be sufficiently massive to prevent toppling over.

Funnels of small size are usually made of glass or earthenware, large sizes of tinned iron or tinned copper. Lately aluminium funnels have appeared in the market, and they specially recommend themselves to the pharmacist on account of their lightness. The metal is, however, difficult to work, and as the funnels are spun on a lathe they are neither ribbed nor fluted. Filtration accordingly proceeds slowly unless some device such as the filtering rack, Fig. 113, or the zinc cone, Fig. 114, is adopted to keep the filtering-paper from adhering to the sides of the funnel.

The most delicate portion of a paper filter is the apex, and it is here they generally break if roughly treated. Filtering cones pierced with holes made of aluminium or zinc are easily obtained, and one of these may be placed in the funnel and the paper filter carefully adjusted if the volume of liquid is large.

Up to now plain paper filters have alone been dealt with, but on account of their slowness they are rarely used by practical pharmacists, unless filtration is made use of simply for collecting the precipitate without regard to the filtrate ; in that case the fewer the folds the more easily the precipitate is detached from the paper. Liquids pass through a plaited filter-paper very much quicker than through a plain one. The method of folding may be explained by the aid of the following diagrams.

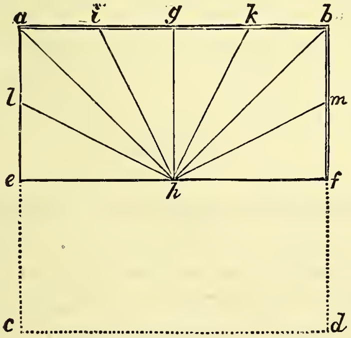

Fig. 115.

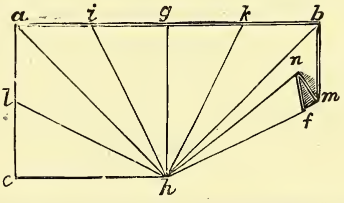

Fig. 116.

A square piece of paper (*a b c d*, Fig. 115) is folded in the line *e f*, the edge *c d* being placed over *a b*. This double sheet is then creased as represented in the drawing. In the first place the crease *g h* is produced by laying *b f* over *a e*, and pressing the thumb-nail over the folded edge so as to produce a sharp crease. Then placing *f* over *g*, the crease *b h* is formed ; in like manner the crease *a h* is formed by laying *e* over *g*, and by similar means the intermediate creases *l*, *m*, *i* and *k*. These creases are all in one direction, forming seven receding angles, and in making them it is desirable not to bring the creases quite to the point *h*, but to leave about half an inch or less through which they do not pass, otherwise the frequent foldings of the paper at this point would so weaken the texture as to cause it to break with the weight of the liquid introduced into the filter. In the next place, an equal number of creases are to be made in the opposite direction, dividing each of the eight sections repre-

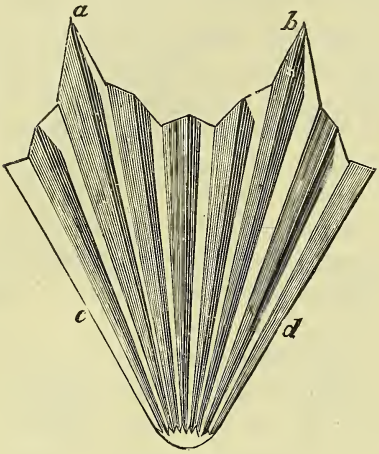

Fig. 117

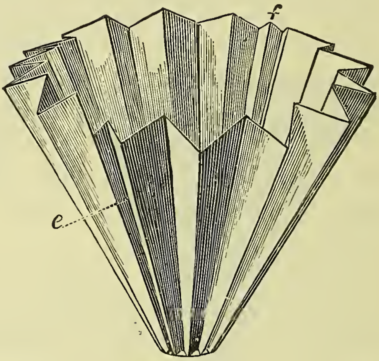

Fig. 118.

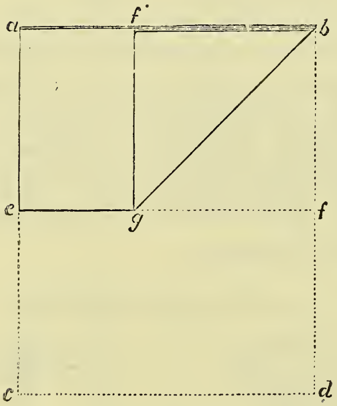

Fig. 119.

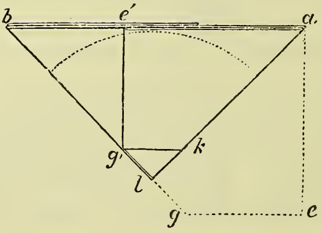

Fig. 120.

sented in the upper part of Fig. 115 in half. In doing this the edge *f h* is laid on the crease *b h*, and then turned back as shown in Fig. 116, producing the crease *n h*. In like manner an intermediate crease is made in each of the other sections, so as to form a sort of fan, as represented in Fig. 117. The points *a b* are cut off with scissors, and the filter opened to its proper angle by separating the originally doubled halves of the paper without disturbing the sharpness of the creases. It will now be found to consist of alternately projecting and receding angles, forming a uniform zigzag circumference excepting at the points *c* and *d* (Fig. 117), at each of which places two projecting angles come together. The intermediate portion of paper between these two angles should be folded so as to form a small receding angle, as shown at *e* and *f*, Fig. 118. This figure represents the appearance of a completed filter.

When a filter breaks, the fracture generally occurs in the apex of the cone. This is the part on which the liquid exerts the greatest pressure, and it also receives the least support from the funnel. The plaited filter especially often breaks at this point, and to obviate this result it may be folded so as to give increased strength where most needed. The paper used is oblong, and not square. It is folded so as to bring the two short ends *a b* and *c d* (Fig. 119) together. The edge *b f* is then laid over *b f′*, producing the fold *b g*. The paper thus folded is now turned over, as shown in Fig. 120, and the edge *a e* laid over *a e′*, producing the fold *a l*. Finally, the projecting points *a* and *b* are cut off in the direction of the dotted line. This filter will then have a double thickness of paper at the apex *g′ k l*.

Another method of supporting the filter consists in placing a little tow or absorbent cotton in the nozzle of the funnel, so as to form a bed on which the point of the filter may rest. If, however, it is considered worth the trouble the apex of the filter-paper may be dipped in strong nitric acid and then thoroughly washed in water. This makes the paper vastly stronger, and yet does not altogether stop filtration, the paper being converted into altogether a different substance from that produced by the action of sulphuric acid, as in the preparation of parchment paper. The edge of a filter-paper should never project above the edge of the funnel, but should rather be below it, so as to permit a closely fitting cover being adapted to prevent evaporation.

When considerable quantities of liquids have to be filtered, a constant supply may be maintained by inverting a flask over the filter in the manner described under Extraction, Fig. 93. Another good method is that described by E. Robinson in the 'Chemical News,' Fig. 121.

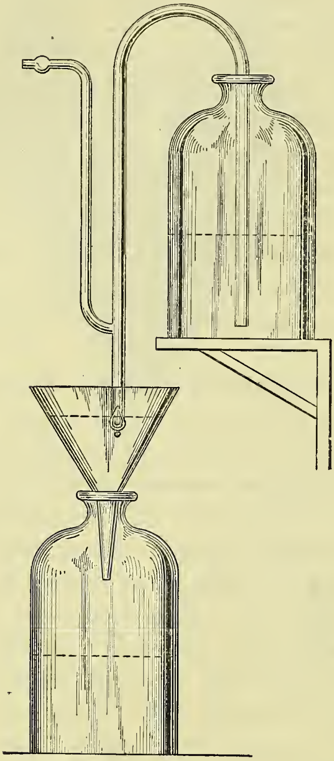

FIG. 121.

FIG. 122.

To the longer limb of the syphon is attached a short rubber tube. Operating vertically within the lower end of the tube is the narrow conical stem of a glass bulb float, in the bottom of which are two or three leaden shot. As the liquid in the funnel filters out the glass bulb descends and opens the bottom of the rubber tube, permitting the liquid in the syphon to flow out over the float. If the liquid from the syphon flows faster than that through the filter the float rises, and by its conical form wedges the inside of the rubber tubing and stops the outflow. By this means the funnel is kept constantly supplied until all the liquid in the upper bottle has been syphoned out. An important advantage of this arrangement is that when once started it can be left without attention until filtration is complete.

To accelerate filtration through paper of small quantities of liquid, as in the washing of precipitates during the application of group reagents, &c., the following simple plan may be resorted to:—Select a perfectly smooth-sided funnel of about 3 inches diameter, fold a circular filter as Fig. 108, and place in the funnel; moisten with water and press gently with the finger so as to ensure a good fit. Pour in the liquid to be filtered and cover the funnel with a perforated glass disc, through which a piece of bent tubing is passed and held in place by a cork above and below. Now blow gently into the filter, holding the glass cover tightly against the edge of the funnel to prevent escape of air. If the filter-paper has been properly adapted the filtration of an ounce or two of liquid need not take more than as many minutes. Usually it is necessary to grind the funnel on the glass, using a little emery powder and oil.

Filtration of volatile liquids.—Various methods have been devised to prevent the loss by evaporation that usually attends the filtration of volatile liquids. One of the most simple is to fit a suitable sized wide-mouthed bottle with a tightly fitting cork pierced with two holes. Into one of these the funnel is fixed, a piece of glass tubing passing through the other. A cover fits closely over the top of the funnel, and as it is also fitted with a glass tube, a short length of soft rubber tubing at once prevents access of air and affords an opening for the escape of the air displaced by the filtrate (Fig. 122).

The glass percolators made by the York Glass Company are most useful for filtering volatile fluids (see Fig. 98). A plaited filter-paper is placed within the percolator and prevented from slipping by a filtering-rack, Fig. 113, placed beneath its apex; the liquid is then poured in by means of a guiding rod, and the grooves in the neck of the receiver and stopper turned out of line so as to prevent entrance of air.

Hot filtration is occasionally resorted to for the clarification of substances which are solid or very viscid at the ordinary temperatures, such as waxes, fats, and ointments. The substance should be melted over a water-bath and poured into a paper filter, supported by a metal funnel kept warm by one of the following contrivances:

In Fig. 123 the funnel is kept hot by a ring of gas jets, a method which is not to be preferred to Fig. 124. Here the funnel is also provided with a hot-water jacket, but the heat is maintained by a Bunsen flame beneath the projecting arm.

Another simple device for keeping a funnel warm is to wind round it a coil of ordinary lead piping, through which steam generated in a Florence flask may be forced. This steam should enter at the top of the coil, and the lower end should dip

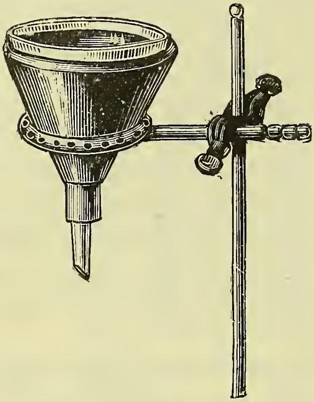

Fig. 123.

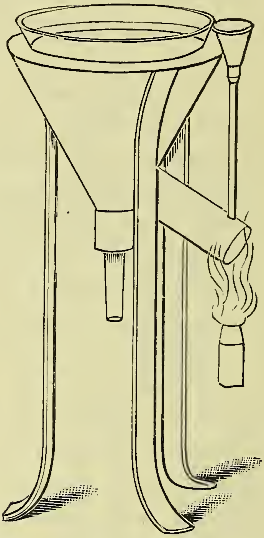

Fig. 124.—Hot-water copper funnel with side arm and small filling funnel, mounted on three legs.

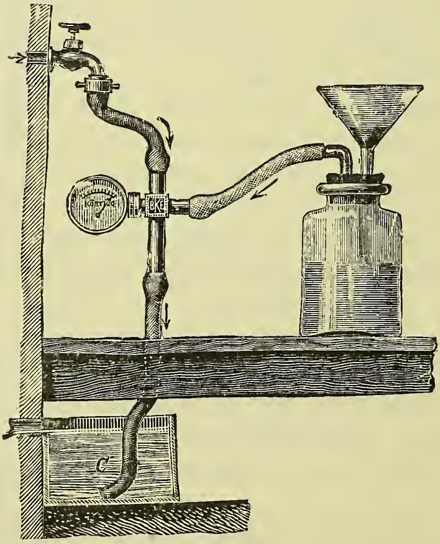

Fig. 125.

into a beaker of water. This serves the purpose of both condensing the steam and increasing the temperature by slightly raising the pressure.

Filtration under pressure.—For accelerating the filtration of liquids that under normal conditions filter very slowly, numerous expedients are resorted to, depending either on artificial pressure derived from a "head" of liquid, the use of a filter press, or increase of atmospheric pressure on the surface of the filtering liquid by the production of a more or less complete vacuum under the filter bed. The last method is most commonly made use of in pharmaceutical laboratories.

The vacuum may be obtained by connecting the air-tight receiver with a Korting's filter pump as Fig. 125. Ordinary paper filters however, are not sufficiently strong of themselves to bear any great increase of pressure, but require support. This may be afforded by fitting a perforated metal cone over the apex of the filter-paper, afterwards pressing the wetted paper tightly against the sides of the funnel to prevent suction of air. But it is found that if the atmospheric pressure is increased ever so little, turbid liquids are drawn through the pores of the paper almost unaltered. To remedy this, scraps of white blotting or filtering paper may be rolled into a ball and rubbed through a coarse wire sieve, and a little of the powder obtained by mixing this paper pulp with ten times its weight of kieselguhr (infusorial earth) shaken up with the liquid to be filtered. The suspended matter becomes entangled in the filtering powder, the whole being deposited on the surface of the filter, through which the liquid passes quite bright. Kaolin and calcium phosphate have both been recommended for the above purpose, but in the author's hands they have not been found as serviceable as kieselguhr mixed with paper pulp. When the vacuum is obtained by a water-pump it is absolutely necessary to fit a valve between the pump and the receiver to prevent water being sucked back into the latter in case the pressure on the water main falls. For description of this valve see Percolation under pressure, page 90.

Filter presses.—An excellent form of portable filter press for comparatively low pressures is shown at Fig. 126, and in section at Fig. 127. Two discs of asbestos filtering-paper are firmly clamped between the ebonite plates A and B, being held apart by the ring D, through an orifice in which the turbid liquid finds entrance, leaving the press at *a*, *b*. With a head of five feet a pressure equivalent to about a sixth of an atmosphere is exerted, and most liquids filter at a very rapid rate.

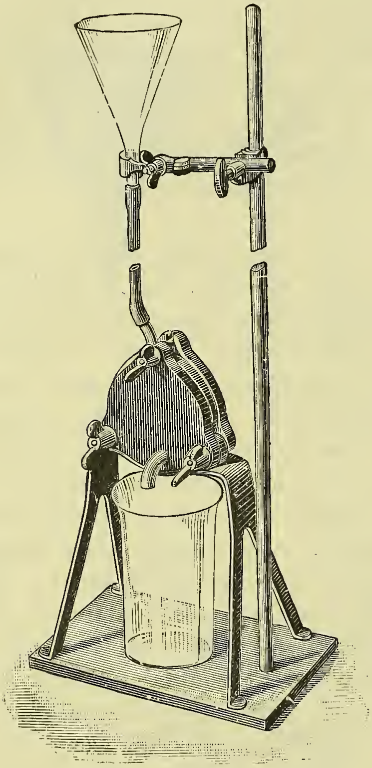

Fig. 126.—Ebonite filter press.

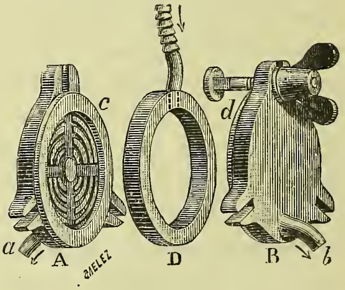

Fig. 127.

When, however, very refractory liquids have to be filtered the filter press illustrated at Fig. 128 is employed. This has a capacity of about four gallons, and can be used for a variety of purposes. The liquid being introduced into the reservoir by the funnel is forced by the pump shown on the right of the engraving into the filter proper. The filtering material, which should be of the stoutest asbestos cloth, weighing about $2\frac{3}{4}$ lbs. to the square yard, is supported by diaphragms similar to Fig. 127, and is capable of supporting enormous pressure. The filtered liquid finds its way out by the two taps situated on the left of the machine. To obtain the necessary pressure and prevent leakage it is important to screw the filter-plates tightly together, for which purpose a powerful screw and lever are attached. When an operation has been completed, the whole machine can be cleaned out by simply unscrewing the clamps and removing the head and tube connections.

Filtration of oils.—The filtration of oils does not differ in any way from ordinary filtration. Great care should, however, be taken to cover the funnel, so as to prevent access of air. When possible, all viscid liquids should be allowed to deposit before placing on a filter.

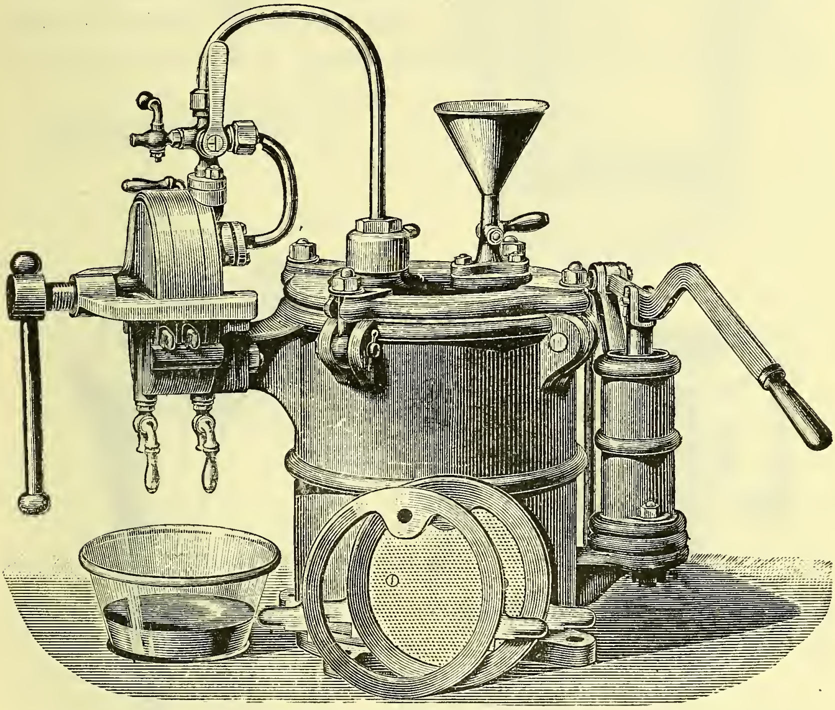

Fig. 128.—Griffin's laboratory filter press.

Filtration of corrosive liquids.—The clarification of strong acids and alkalies is brought about as much as possible by subsidence, the clear liquid being drawn off by a safety syphon (see Decantation). It is a fortunate fact that such liquids deposit readily, and rarely require other treatment. They may, however, in emergency be filtered through a plug of glass wool, pushed well into the nozzle of an ordinary earthenware funnel.

Colation.—Colation or straining differs from filtration in that it is made use of for the removal of particles of comparatively large size. The ordinary straining materials are muslin, calico, flannel, and horsehair. A very useful support or frame is shown in Figs. 129. Muslin and calico may be stretched and adapted for straining small quantities of liquids by the use of the metal strainers, Figs. 131 and 132. A very convenient piece of apparatus is shown in Fig. 133 for adapting funnels to wide-mouthed jugs, jars, &c. For many purposes it suffices to place the straining cloth in an ordinary funnel in the same way as a filter-paper, when, if the liquid passes through slowly, the four corners may be tied together with a piece of string, and the whole suspended from a nail or hook at a convenient height above the receiver, in a similar manner to the straining bag, Fig. 130.

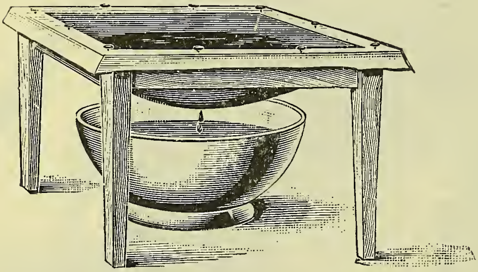

Fig. 129.—Straining frame.

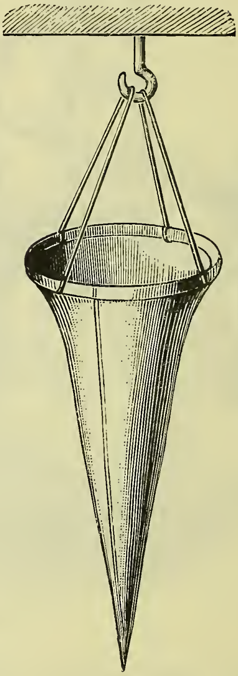

Fig. 130.—Straining bag.

Bath brick filters.—Some time ago the author had occasion to deal with a thick muddy deposit which altogether refused to be filtered under ordinary conditions, and if filtered under pressure invariably passed through muddy. A trial was then made with a Berkefeld filter connected with a vacuum-pump, the best results being obtained, but the " candles " were so expensive and fragile that some cheaper method had to be devised. After numerous experiments it was decided to establish a battery of

Fig. 131.—Straining funnel for muslin or flannel.

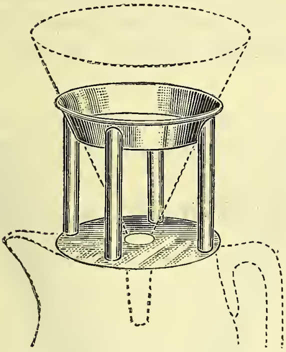

Fig. 133.—Metal funnel adapter.

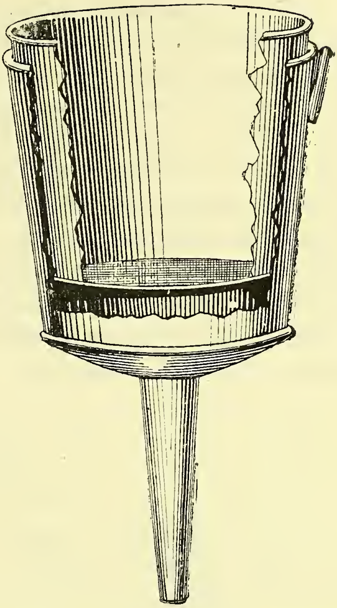

Fig. 132.—Metal strainer and funnel combined.

filters made of bath brick, and such excellent results were obtained with these as to appear to warrant the publication of the process adopted.

Sound bath bricks were selected, and after rubbing perfectly smooth against each other were sawn across the middle. A hole was then bored three quarters of the length into one of the blocks with a carpenter's brace and bit, and a glass tube roughened on the outside cemented firmly in. It was then only necessary to place the block beneath the surface of the liquid to be filtered, and to connect the glass tube by pressure tubing with a filter-pump to obtain a steady flow of perfectly clear liquid, filtration proceeding automatically. It was found advantageous to pass the short glass tube through a hole bored in a large bung, the filter block being securely fastened underneath. By this means *the block always floated just beneath the surface of the liquid* instead of lying buried in the thick deposit at the bottom of the vessel. When the filter block at length showed signs of clogging it was withdrawn, the pump turned off, and without disconnecting scrubbed with a stiff brush in a stream of water and replaced. As many such filter blocks as desired, provided all the joints are tight, can be connected up to the same pump, so that almost any quantity of intractable liquid can be dealt with at a very small outlay.

As a proof of the efficacy of the filtration one example may suffice. Half a pint of peptone broth was inoculated with a strong culture of *Bacterium termo*, and left in a warm place for three days, at the end of which time it was filtered through a sterilised filter similar to Fig. 134 into a tube of sterile nutrient gelatin. No liquefaction took place in fourteen days, so that it may be fairly assumed that a mass of ordinary bath brick, if free from flaws, is at least as efficient a filtering medium as the more expensive Berkefeld material.

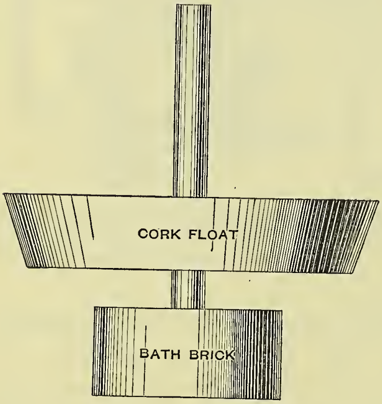

Fig. 134.

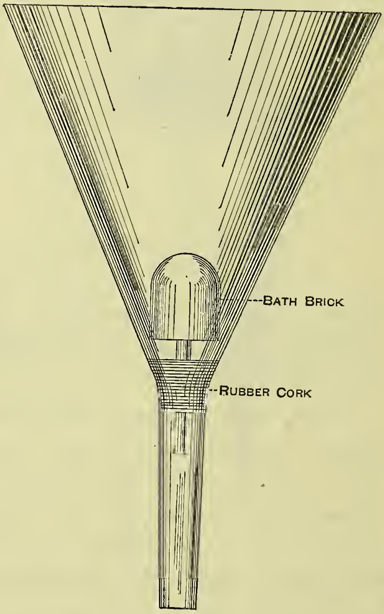

Fig. 135.

For ordinary liquids the filters need not be connected with a vacuum-pump, but may be used under the normal atmospheric pressure. The most convenient method of forming filters is perhaps the following. With a sharp knife cut a solid cone out of a bath brick, and rub the sides perfectly smooth with coarse sand-paper; now cement it firmly into a glass funnel, or slip a broad rubber band cut from a piece of soft rubber tubing of wide bore over the cone, and push it firmly into the funnel. This latter is the better method, as the cones can be used for funnels of different sizes. Instead of the above, sound bath brick may be trimmed into a cylinder, bored and fitted with a glass tube as Fig. 135. It is then adapted by means of an india-rubber cork to a large funnel.

For dealing with larger quantities, a bed of bath bricks could be laid down on a surface of sand or fine gravel, the only precaution being to insure that the sides and ends of each brick were perfectly smooth and in intimate contact. When foul, the surface of such a filter bed could be readily cleansed by scrubbing with a stiff broom, the muddy liquid being drawn off at a side exit.

Clarification.—In some cases it is not easy to apply any of the foregoing processes for obtaining clear liquids. Honey, for instance, cannot well be filtered, but if it is rapidly brought to the boil, a scum rises to the surface, which may be removed when cool by straining through felt. Again, solutions of gelatin may be obtained perfectly bright by adding white of egg that has been well stirred up with water, and bringing rapidly to the boil. In this case the albumen coagulates, and carries with it any finely divided matter held in suspension. This is called clarification.

CHAPTER XII

DECANTATION

Decantation (*de,* from ; *canthus,* an edge) is the name given to the process of separating a fluid from a solid as practised in the washing of precipitates, when, the insoluble substance having subsided, the supernatant fluid is poured off or decanted as completely as possible. In the pharmacopœial process for the preparation of saccharated ferrous carbonate, the precipitate obtained by the decomposition of ferrous sulphate and ammonium carbonate is washed out of contact with air, by agitating with water, allowing the precipitate to subside, and decanting. The operation is repeated until the washings give no reaction for sulphates. When dealing with comparatively small volumes, fluids may be decanted by slightly greasing the rim of the pouring vessel. This keeps the fluid in a single stream, preventing it from dividing and running down the side of the vessel. The liquid may also be guided into the receiver by placing a glass rod close to the lip or rim of the pouring vessel, as shown in Fig. 136.

When large quantities of liquids have to be decanted from one vessel to another, or when the precipitate is very light and easily disturbed, the transference is usually effected by the aid of a syphon (Fig. 137).

In the illustration the syphon is provided with a safety suction tube to avoid danger of corrosive liquids entering the mouth. The shorter leg of the syphon is placed in the liquid, and fastened at such a height that the extremity reaches to within an inch or two of the precipitate, a short length of rubber tubing provided with a burette clip being at the same time slipped over the upper end of the suction tube. The longer leg of the syphon is next closed with a cork, the burette clip opened, and suction applied until the liquid appears in the suction tube.

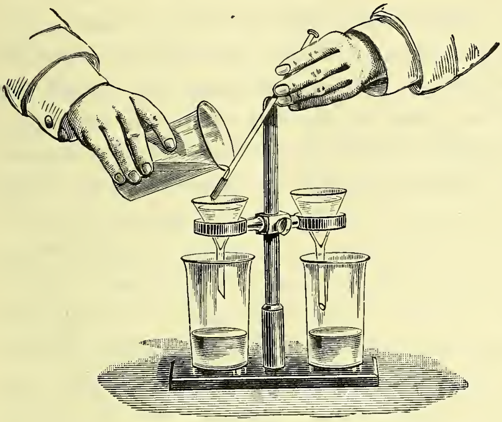

Fig. 136.

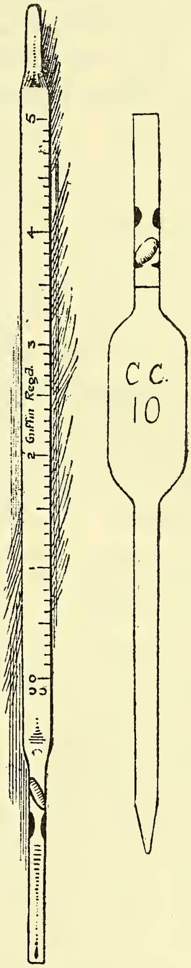

Fig. 138.—Safety pipettes.

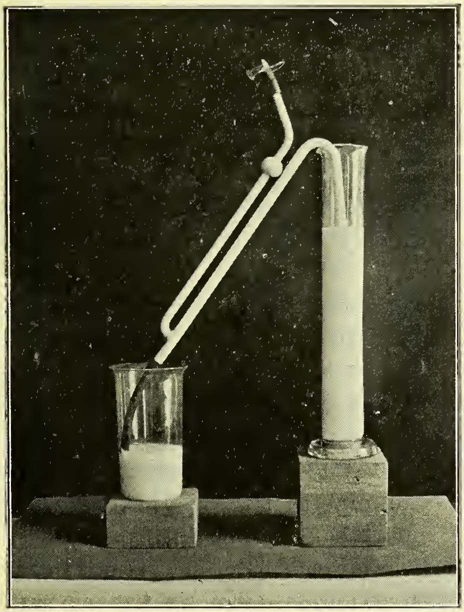

Fig. 137.—Syphon with safety-tube.

The burette clip is then released and the flow of liquid started by removal of the cork.

The action of a syphon may be explained as follows:—The shorter leg is first placed in the liquid, which rises until the levels inside and outside the tube correspond. Communication with the outer air is then cut off by the cork at the bottom of the delivery tube and the clip on the suction tube. Now if the clip is opened, and suction applied, a vacuum is formed inside the tubes, and the liquid rushes in to take the place of the exhausted air owing to the pressure of the atmosphere on the surface of the liquid. The syphon is now filled. When the clip is released and the cork withdrawn a flow of liquid ensues, which is more rapid in proportion, as the difference of level between the *aperture* of the delivery tube and the *surface* of the liquid in the beaker is greater. This is explained by the column of liquid in the outer limb of the syphon being longer and therefore heavier than the corresponding column in the immersed limb; being heavier it falls by gravitation, the liquid in the shorter limb being instantly pressed after it by the pressure of the atmosphere. The shorter limb of the syphon is only reckoned from surface of the liquid to the bend, the part below the surface playing no part in the operation.

For decanting small quantities of liquid, such as the ethereal layer in the estimation of opium, glass instruments called pipettes are used. These consist primarily of a glass tube having a very narrow discharge outlet. When a liquid is sucked up it can be retained by simply closing the upper orifice. The chief drawback to their use, however, lies in the fact that if a noxious or corrosive liquid is incautiously sucked up into the mouth disastrous results may ensue.

The author in order to overcome this difficulty devised an instrument which was made by Messrs. Griffin and Sons, consisting of an ordinary graduated pipette with a somewhat elongated mouthpiece with two constrictions about an inch apart. The upper constriction is ground smooth inside, the lower one is imperfect, while between the two is a loosely working glass bead. The action is as follows:—The pipette is placed in the liquid, which is sucked up by the mouth in the usual way. As soon as the liquid reaches the constriction the bead is carried upwards and forced into the second constriction, into which it fits accurately, thus preventing the liquid from entering the mouth. There are no delicate parts to get out of order, and being made entirely of glass it can be safely used for measuring corrosive liquids of all kinds (Fig. 138).

Separation of Immiscible Liquids.

In many pharmaceutical operations it becomes necessary to separate immiscible liquids, such as essential oils, chloroform, petroleum, ether, &c., from water or watery solutions. When the operation is of frequent occurrence, and the immiscible liquid is not volatile, separating funnels as Fig. 139 are used. If the liquid is of a volatile nature, stoppered separators as Fig. 140 must be employed. If neither of these forms of apparatus are at hand, or if the quantities to be dealt with are so great as to preclude the use of the usual sizes, decantation by means of a syphon may be resorted to (see Decantation), or a funnel of convenient size may be converted into a separator by the device suggested by Currier, the liquid being drawn off by means of a glass tube sliding through a cork. The stem of a funnel is plugged with a closely fitting cork, through which passes a glass tube closed at its upper end, but having a slit filed through about an inch from the top, so as to afford egress to the liquid. If the tube is pulled down so that the slit is closed by the cork, no liquid can escape; but if pushed up, liquid can be drawn off from any level. Attached to the lower end of the glass tubing is a short piece of rubber tube provided with a pinch-cock. The same device may be made use of for the separation of volatile liquids, but instead of being adapted to a funnel the tube is adapted to a convenient-sized flask. In addition to the draw-off tube a second tube of very fine bore is pushed through the cork until it reaches almost to the bottom of the flask, and serves as an air supply.

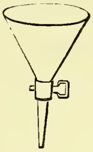

Fig. 139.—Separating funnel.

Fig. 140.—Stoppered separator.

Dr. Mitchell's device for separating immiscible liquids is even simpler than the foregoing. A stout penholder is firmly attached to a rubber plug which fits snugly into the funnel stem. The liquids are introduced and allowed to separate, the lower layer being drawn off by gently raising the plug.

CHAPTER XIII

CRYSTALLISATION

Almost all solid substances assume under favorable conditions distinct geometrical figures, such as the cube, prism, or rhombohedron; these are called crystals. All crystals owe their figure to the regularity of internal structure with which they are built up, as a crystal placed in a saturated solution undergoing spontaneous evaporation increases by a constant deposition of fresh matter on its sides, so that the angles remain unaltered. Generally speaking, all solids have a particular crystalline form by which they may often be recognised, although the same substance may assume two different forms at different temperatures, when it is said to be *dimorphous*. All bodies capable of assuming the *crystalline* form are spoken of as *crystallisable;* while uncrystallisable bodies, such as gelatin, &c., are termed *amorphous*. The plane surfaces with angles of constant value which bound a crystal are called *planes* or *faces;* the intersection of two adjacent planes forms the *edge*.

All crystalline forms may be divided into six systems, depending upon the length and position of their *axes*. These axes are imaginary lines passing through the central point of the crystal, from end to end or from side to side or from opposite angles, around which lines the particles forming the crystal may be imagined to have been deposited. For a full description of the different systems the reader is referred to text-books specially dealing with crystallography.

Substances more soluble in hot than cold water may be crystallised by allowing hot saturated solutions to cool very slowly and quietly. If the substance be almost equally soluble in hot and cold water, concentrated solutions may be left to evaporate spontaneously in the air, or they may be slowly evaporated in a partial vacuum. The more slowly and quietly the operation of crystallisation is conducted the larger and more regular will be the deposited crystals.

Other methods of effecting crystallisation than by deposition from solution are by the cooling of vapours, as described under Sublimation, benzoic acid affording an example; the slow cooling of fused masses, such as molten sulphur; melted bismuth especially frequently affords beautiful crystals as it cools. Crystals are sometimes produced by the decomposition of solutions, as when solution of potash is added to solution of tartaric acid. Again, crystallisation often takes place when a concentrated solution is mixed with a liquid exercising little or no solvent action on the dissolved body, as alcohol and strong syrup.

Solution of substances not very soluble are usually concentrated until a pellicle or crust forms on the surface, but in any case the proper degree of concentration is entirely dependent upon the nature of the substance, the quantity of the solution, and the temperature of the surrounding air. Large well-formed crystals can only be formed in solution of considerable bulk when cooled slowly.

Alum for instance is crystallised on an enormous scale in large vats. After a thick crust has formed around the sides and bottom, the iron hoops with which the staves are held in position are removed, and a hole cut through the mass to allow the mother liquor to escape.

It has been noted (under the head of Solution) that supersaturated solutions can be made and kept under certain conditions without crystallising; but in all cases crystallisation may be started by dropping in a crystal of the same form, and in many instances by simply rubbing the sides of the crystallising vessel, or by placing rough sticks or yarn in the cooling solution; milk-sugar and sugar-candy are generally crystallised under these latter conditions.

The liquid remaining after crystallisation has taken place is called the *mother liquor*, and on further evaporation yields a second crop of crystals, usually less pure than the first. They may, however, be purified by re-solution and re-crystallisation. If more than one substance be present in a solution, and their solubilities are appreciably different, they may be separated by *fractional crystallisation*; that is, the least soluble crystallises first and the most soluble last, and by re-crystallising each crop, pure crystals are readily obtained.

The temperature at which crystals are formed largely determines the composition. Glauber's salt, for instance, crystallises with ten molecules of water from cold solutions, and from hot water with seven molecules, or even none, according to the temperature.

Many crystals, such as potassium chlorate, mechanically enclose water during formation. This is known as *interstitial water*, and must be distinguished from water of crystallisation. These crystals decrepitate when heated, owing to the explosive force with which the steam ruptures the crystals.

Granulation may now be interpreted in more than one way. When associated with crystallisation we understand the production of granules or coarse grains obtained by stirring concentrated solutions while cooling. The official variety of Epsom salts is an example; if the hot supersaturated solution instead of being disturbed is allowed to cool and crystallise quietly, large rhombic prisms would be formed instead of the minute crystals with which we are so familiar. Solutions of other freely soluble salts, such as potassium carbonate, are concentrated, and then slowly evaporated to dryness on a steam-bath, stirring constantly all the time. This causes the salt to separate in a finely granular condition. The granular form of ferrous sulphate, formerly official, was ordered to be made by pouring the filtered solution into strong alcohol, by which means very small crystals were obtained. The term granulation has also been adopted for the preparation of a certain class of effervescent salts, as well as for the process to which powders are subjected previous to compression into tablets. These will be treated under special headings. To add to possible confusion, the name *granule* is also applied to pills of small size, generally, although not always, sugar-coated.

Deliquescence.—Many salts, such as potassium carbonate, lithium bromide, &c., possess the property of absorbing water from the atmosphere, becoming damp, and in some instances actually dissolving in the amount of moisture attracted. Such salts are said to be deliquescent.

The following substances, as well as all granular effervescent salts and scale preparations, attract moisture from the air:

Acid, chromic.
,, sulphuric.
Aluminium chloride.
Ammonium hypophosphite.
,, iodide.
,, nitrate.
Ammonium valerianate.
Caffeine citrate.
Calcium bromide.
,, chloride.
,, iodide.
Calcium nitrate.

Chloral hydrate.
Cocaine salicylate.
Glycerin.
Gold chloride.
Iron ,,
Lithium acetate.
  ,, bromide.
  ,, chloride.
  ,, citrate.
  ,, iodide.
  ,, salicylate.
Lysidine.
Magnesium bromide.
  ,, iodide.
Manganese chloride.
Pancreatin.
Pepsin.
Physostigmine sulphate.
Pilocarpine hydrochloride.
Piperazine.
Platinic chloride.
Potassium acetate.
  ,, carbonate.
  ,, citrate.
  ,, cyanide.
  ,, hydroxide.
  ,, hypophosphite.
  ,, phosphate.
  ,, sulphite.
Sodium chloride.
  ,, hydroxide.
  ,, iodide.
  ,, nitrite.
  ,, peroxide.
  ,, valerianate.
Strontium bromide.
  ,, iodide.
Zinc bromide.
  ,, chloride.
  ,, iodide.
  ,, permanganate.

Efflorescence is the exact opposite of deliquescence. Efflorescent salts give up their water of crystallisation on exposure to air, losing their transparency and becoming powdery on the surface. The following are efflorescent:

Acid, citric.
Borax.
Codeine.
Copper acetate.
  ,, sulphate.
Ferrous sulphate.
Lead acetate.
Magnesium sulphate.
Potassium ferrocyanide.
Sodium acetate.
  ,, arsenate.
  ,, carbonate.
  ,, hypophosphite.
  ,, phosphate.
  ,, sulphate.
Zinc acetate.
  ,, sulphate.

Note.—It will be evident from a study of the foregoing lists of deliquescent and efflorescent bodies that careful attention must be paid to their storage. Deliquescent substances especially should be preserved in stoppered bottles.

Drying of crystals.—Small quantities of crystals may be practically freed from adhering water by lightly squeezing them between folds of blotting-paper. A good deal of the water drains away if the moist crystals are placed on porous tiles or on well-washed and dried bath bricks.

Larger quantities may be almost entirely freed from water by means of a centrifugal machine, as Fig. 141. The wet crystals are placed in the inner perforated cylinder lined with enamel, and revolved at a high speed, when the water flies off and is collected in the outer vessels.

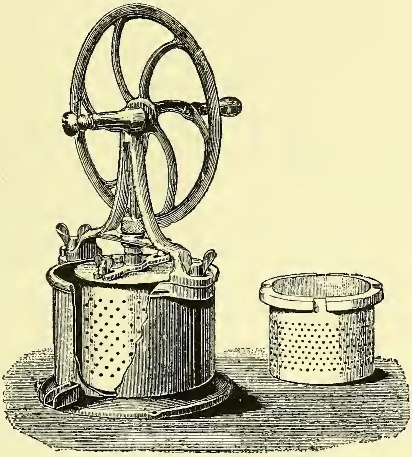

FIG. 141.

PART II

PHARMACOPŒIAL PREPARATIONS

CHAPTER XIV

ETHYLIC ALCOHOL

The manufacture of alcohol forms a very important branch of industry. Alcohol was formerly obtained by distillation from wine, and the fact that it was also contained in beer, naturally led to its preparation by the action of yeast on infusion of malted grain.

Ethylic alcohol is always a product of vinous fermentation; its manufacture may be divided into three principal operations:

1. The preparation of a saccharine fluid.
2. The fermentation of this fluid.
3. Separation of the alcohol by distillation.

All saccharine fluids, and all substances which yield alcohol by fermentation, can be employed in the manufacture of spirit. These materials contain either ready formed alcohol, as wine and beer; sugar, as sugar-cane and beetroot; or, substances which by the influence of diastase or dilute acids are converted into sugar, as starch.

The chief sources of the alcohol produced in this country are the cereals. The grain is moistened and allowed to germinate. During germination changes take place in the embryo, and an enzyme (diastase) is formed which is capable of converting the insoluble starch in the endosperm into a soluble sugar, intended for the use of the seedling until the latter is capable of supporting itself from the soil. Germination is then interrupted by heating the grain, but at a temperature below that likely to injure the diastase. The dried product is then ground, and constitutes *malt*; it not only contains sufficient diastase to convert its own unaltered starch, but also the starch of a considerable quantity of unmalted grain, when the whole is infused with warm water.

1. *Preparation of the saccharine fluid or "mash."*—It has been found that a *mixture* of several sorts of grain yields more alcohol than one kind mashed by itself. The most common mixture is that of barley with oats or wheat. Commonly one part of malted

barley is added to three parts of unmalted grain, and the whole ground as evenly as possible between ordinary millstones. As it is not essential in the manufacture of alcohol that a clear infusion (wort) should be prepared, the grain may be broken up very small, whereby formation of sugar is rendered more complete. The crushed grain is then put into the mash-tub, generally a large wooden vat, where it is intimately mixed with an equal weight of warm water (temperature about 60° C.), and carefully stirred so as to avoid the formation of lumps. After standing for half an hour the first mash is drawn off, a second supply of water added, and the mash heated to 65° C. The mashing this time occupies several hours, so as to make certain of the conversion of the whole of the starch into sugar. When completed the second mash is drawn off and mixed with the first in a large tub called the fermenting vat, and yeast added in the proportion of 2 to 3 pounds to every 100 pounds weight of mash or wort. During the mashing the starch is converted first into dextrin, and finally into maltose, a readily fermentable sugar.

2. *Fermentation.*—The most favorable temperature for fermentation is about 23° C. After standing four or five hours at this temperature fermentation commences, and usually lasts for four days. During this period alcohol is formed and carbonic anhydride given off, the insoluble substances sinking to the bottom of the vat.

3. *Distillation.*—In this country the weak alcoholic liquid is usually distilled in a "Coffey's still." This consists essentially of two parts, the "analyser" and the "rectifier." The first depends upon the principle that when mixtures of the vapours of alcohol and water are cooled by suitable arrangements, the condensed liquid separates into two parts, one containing relatively a large proportion and one a small proportion of alcohol—the latter being allowed to run away. (This principle is made use of on a small scale in laboratories in the process of fractional distillation with Wurtz's bulbs.) In the second part of the apparatus the alcoholic portion is again heated and condensed in the rectifier, but none of the liquid is allowed to run back as in the first part, the whole being heated by a current of steam to boiling-point, when vapour rich in alcohol is given off. This is condensed, again brought to the boiling-point by the action of steam, and condensed in a third vessel as strong alcohol.

During vinous fermentation there are formed, in addition to ethylic alcohol, varying quantities of its homologues, such as amylic, propylic, and butylic alcohols. These alcohols with fatty acids and ethereal salts constitute what is technically known as "fusel oil." To separate the pure spirit from the strongly smelling fusel oil it is either treated with freshly burnt charcoal or submitted to further rectification, when ethylic alcohol passes over first, leaving the fusel oil, which possesses a higher boiling-point, behind.

Properties of alcohol.—According to Mendelejeff, absolute alcohol boils under normal pressure at 78·3° C.; and its specific gravity at 15° C., compared with water at 3·9° C., is ·79367.

Alcohol has a pleasant ethereal smell; it mixes with carbon disulphide in all proportions, and causes no turbidity when shaken with benzene.

For accurately determining the percentage of absolute alcohol in spirit its specific gravity must be determined by means of a specific gravity bottle. For excise and general purposes however, hydrometers are usually employed.

In England the legal temperature is fixed at 51° F., the spirit not being valued according to its percentage of real alcohol, but according to the amount of "proof spirit" it contains. This term is defined by the Act 58 George III as "being such as shall, at a temperature of 51° F., weigh exactly $\frac{12}{13}$ part of an equal measure of distilled water." Proof spirit, therefore, contains 49·3 per cent. by weight, or 57·09 per cent. by volume, of absolute alcohol. Weaker spirits are termed "under proof," and stronger spirits "over proof." Thus 25° over proof means that 100 volumes of this spirit diluted with water yield 125 volumes of proof spirit, whilst 25° under proof means that it contains in 100 volumes 75 volumes of proof spirit and 25 volumes of water.

Note.—It is possible that the views hitherto held with regard to the nature of the action of yeast upon sugar in the formation of alcohol, will require modification. Lately Buchner has stated that alcoholic fermentation may be produced without the presence of yeast cells. Fresh beer yeast was freed as completely as possible from adhering water, and after mixing with ground quartz, submitted to hydraulic pressure, a strong solution of the soluble contents of the yeast cells being thus obtained. The liquid after filtration had a specific gravity of 1·0416. On warming, carbonic anhydride was given off, and on raising the temperature to 40° C. the liquid coagulated. Buchner found that this liquid, although absolutely free from yeast cells, was capable of inducing alcoholic fermentation in saccharine liquids.

Professor J. R. Green, who has repeated Buchner's experiments, has failed to produce the enzyme from samples of English yeast, but he does not regard this as conclusive proof of its non-existence, as Buchner himself failed to produce it from certain samples of yeast.

Table of the strength of alcohol ordered in the Pharmacopœias of the more important countries.

Pharmacopœia.	Specific gravity at 60° F.	Official name.	Percentage of absolute alcohol by volume.
Austrian	·830 —·834	Sp. Vini Concentratus	90—91
	·894 —·896	„ Dilutus	68—70
Belgian	·794	Alcohol Anhydrus	100
	·8276	„ @ 92°	92
British	·794 —·7969	„ Absolutum	100
	·834*	Spiritus Rectificatus	90
Danish	·830 —·834	„ Concentratus	90—91
	·893 —·895	„ Dilutus	68—69
	·940 —·942	„ Tenuis	46—47
Dutch	·831 —·837	„ Fortior	89—91
	·887 —·892	„ Dilutus	69—74
French	·8164	Alcool à 95°	95
	·8338	„ 90°	90
	·8639	„ 80°	80
	·9135	„ 60°	60
German	·830 —·834	Spiritus (Weingeist)	90—91
	·892 —·896	„ Dilutus (verdünnta Weingeist)	68—69
Hungarian	·831 —·834	„	90—91
	·892	„ Dilutus	70
Italian	·8344	Alcool	90
	·7937	„ Assoluto	100
	·9139	„ Diluito	60
Norwegian	·8311—·8344	Spiritus Concentratus	90—91
	·9026—·9049	„ Dilutus	64—65
	·9346	„ Tenuis	50
Portuguese	·834	Alcool á 90°	90
	·850	„ 85°	85
	·905	„ 65°	65
Russian	·813 —·816	Sp. Vini Alcoholisatus	95
	·831 —·834	„ Rectificatissimus	90
	·888 —·890	„ Rectificatus	70
	·952 —·955	„ Dilutus	38
Spanish	·796	Alcohol Anhidro	100
	·833	„ de 90°	90
	·913	„ de 60°	60
Swedish	·830 —·834	Spiritus Concentratus	90—91
	·901 —·905	„ Dilutus	64—65
	·935	„ Tenuis	50
Swiss	·812 —·816	„	95—96
	·892 —·892	„ Dilutus	69—70
U.S.A.	·820	Alcohol	94
	·797	„ Absolutum	100
	·816	„ Deodoratum	95
	·936	„ Dilutum	48·6

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* Sp. Rectificatus is sold by the rectifiers as 58° over proof.

Rules for making alcohol of any required lower percentage from alcohol of any given higher percentage.

I. *By volume.*

Let the volume percentage of the stronger alcohol be V.
,, ,, ,, weaker ,, *v*.

Rule.—Mix *v* volumes of the stronger alcohol with sufficient water to produce V volumes of product. Allow the mixture to stand until cold, and full contraction has taken place, then make up any deficiency with water. *Example.*—An alcohol of 30 per cent. by volume is to be made from alcohol of 90 per cent. by volume.—Take 30 volumes of the 90 per cent. alcohol, and dilute with water to 90 volumes.

II. *By weight.*

Let the weight percentage of the stronger alcohol be W.
,, ,, ,, weaker ,, *w*.

Rule.—Mix *w* parts by weight of the stronger alcohol with sufficient water to produce W parts by weight of product. *Example.*—An alcohol of 50 parts by weight is to be made from alcohol of 90 per cent. by weight.—Take 50 parts by weight of the 90 per cent. alcohol, and add enough water to produce 90 parts by weight.

Rule for making any required quantity of alcohol, of any percentage by volume, from a stronger alcohol.

As the percentage of the alcohol given is to that of the alcohol required, so is the quantity desired to the quantity of alcohol to be taken; to this quantity of alcohol must be added sufficient water to make up the required quantity. *Example.*—It is desired to make 80 fluid ounces of 75 per cent. alcohol from that of 90 per cent. As 90 : 75 :: 80 : 66⅔. That is, 66⅔ fluid ounces of 90 per cent. alcohol are to be diluted to 80 fluid ounces with distilled water. This rule makes no allowance for any subsequent contraction, and a mixture after being made should be allowed to stand until it has acquired a temperature of 60° F., when the volume must be finally adjusted by the addition of water.

To convert percentage of alcohol by volume into percentage of alcohol by weight.

Rule.—Divide ·79367 by the specific gravity, and multiply the quotient by the percentage by volume. Thus 57·3 per cent. by volume = 49·5 per cent. by weight (·79367 ÷ ·920 × 57·3 = 49·5).

To convert percentage of alcohol by weight into percentage of alcohol by volume.

Rule.—Multiply the percentage by weight by the specific gravity, and divide by ·79367. Thus 49·5 per cent. by weight = 57·3 per cent. by volume (49·5 × ·920 ÷ ·79367 = 57·3).

To convert percentage of alcohol by volume into percentage of proof spirit (Sikes).

Multiply the former by 1·7525. Thus rectified spirit B.P. (90 per cent. by volume) = 157·725 (Sikes) or 57·725 over proof. Conversely, percentage proof spirit (Sikes) may be converted into percentage of alcohol by volume by dividing the former by 1·7525.

Methylated spirit

Under certain conditions two kinds of methylated spirit are permitted to be received by licensed persons; these are *mineralised* methylated spirit and *ordinary* methylated spirit (old kind). Any person not being a distiller or rectifier of spirits, or a dealer in or retailer of beer, spirits, wines, or sweets, may obtain a licence to receive and sell *mineralised* methylated spirit. The licence duty is 10*s.* per annum, and is renewable on the 1st of October in each year. The penalty for selling this kind of spirit without a licence is £50 in addition to any other liability which such unlawful sale may involve.

It is also unlawful to sell *mineralised* methylated spirit between the hours of 10 p.m. on Saturday night and 8 a.m. on the following Monday. Contravention of this provision renders the offender liable to a fine of £100 for each offence.

Mineralised methylated spirit may be used for the preparation of the four following pharmacopœial liniments, provided they are plainly labelled "methylated:"—Linimentum Saponis, Linimentum Camphoræ Compositum, Linimentum Aconiti, and Linimentum Belladonnæ. Besides these, it is not lawful to be in possession of or sell any mineralised spirit containing any essential oil or other flavouring matter, or in which the proper quantity of mineral naphtha ($\frac{3}{8}$ of 1 per cent.) has not been dissolved.

The foregoing applies only to the *mineralised* methylated spirit used for lamps and heating and cleaning purposes generally, and it is the only kind allowed to be sold by retailers.

Persons however, obtaining the sanction of the Revenue Commissioners may receive the old kind of methylated spirit (which contains about 10 per cent. wood naphtha only, and no mineral oil) for use in the arts and manufactures, security being given for its proper use. This kind of methylated spirit must only be used for purposes which are definitely specified, and if at any time it is required to be used for any other fresh, although similar purpose, permission must first be obtained from the Commissioners. By contravention of these conditions a fine of £50 is incurred for each offence.

The use of methylated spirit in the preparation of absolute alcohol is illegal, but it may be used for the manufacture of ether, chloroform, and chloral hydrate. Permission may also be obtained for its use in the preparation of vegetable extracts, medicated cottons and gauzes, &c., provided the whole of the spirit is completely removed from the finished product. The spirit may be recovered by distillation if permission is first obtained, but care must be taken that the recovered spirit does not become too pure by repeated distillation.

Detection of methylated spirit in tinctures, liniments, &c.—The process devised by Miller is as follows:—Distil 5 c.c. from the suspected sample, and place in a small retort with 2 grams of potassium dichromate, 15 c.c. of water, and 2 c.c. of strong sulphuric acid. Mix by rotating gently, and set aside for half an hour. Distil off 10 c.c., add to the distillate a slight excess of sodium carbonate, boil to about 5 c.c., and then acidify slightly with acetic acid. Pour the mixture into a perfectly clean test-tube containing solution of silver nitrate, and warm gently for a few minutes. If the contents of the tube merely darken but continue translucent, no methylic alcohol is present; but if a dark brown or black precipitate of metallic silver is thrown down, the sample must be regarded as methylated. In the latter case the sides of the tube generally show a metallic mirror when viewed by transmitted light.

Explanation.—(A) When ethylic alcohol is treated with potassium dichromate and sulphuric acid, it is oxidised first to aldehyde and then to acetic acid, the latter of which has practically no action on silver salts. Any aldehyde remaining unoxidised is boiled away. (B) Methyl alcohol when similarly treated yields formic aldehyde, and finally formic acid, a powerful reducing agent. Thus:

$$(\text{A})\begin{cases} 1.\quad CH_3.CH_2.OH + O = CH_3.CHO + H_2O. \\ 2.\quad CH_3.CHO + O = CH_3.COOH. \end{cases}$$

$$(\text{B})\begin{cases} 1.\quad CH_3.OH + O = H.CHO + H_2O. \\ 2.\quad H.CHO + O = H.COOH. \end{cases}$$

SPIRITS

Of the eighteen official spirits the following are simple solutions of the medicament in alcohol, and do not require any special description:

Spiritus Ætheris.
,, Anisi.
,, Cajuputi.
,, Camphoræ.
,, Chloroformi.
,, Cinnamomi.

Spiritus Juniperi.
,, Lavandulæ.
,, Menthæ Piperitæ.
,, Myristicæ.
,, Rosmarini.

Several of the others are, however, of considerable interest, and will be dealt with at length.

Spiritus Ætheris Compositus.—The process followed aims at the production of heavy oil of wine, which after purification is dissolved in a mixture of alcohol and ether. In the first step alcohol and sulphuric acid are allowed to react in the cold for twenty-four hours, producing some ethyl-hydrogen sulphate.

$$C_2H_5.OH + H_2SO_4 = C_2H_5.HSO_4 + H_2O.$$

On the application of heat complicated decompositions occur, ether being formed and distilling over with water, sulphur dioxide, ethylene, and a little oil of wine. The distillate is collected and poured into a separator, the acid aqueous layer withdrawn, the oil of wine washed with solution of sodium bicarbonate to remove acidity, and finally with distilled water; it is then dissolved in the mixture of alcohol and ether.

Oil of wine is regarded as consisting chiefly of an ethereal salt—normal ethyl sulphate—with polymers of ethylene, as when distilled with water or alkalies it is decomposed into sulphovinic acid and a liquid hydrocarbon having the same composition as olefiant gas. A liquid obtained by distilling the residue left after the manufacture of ether has occasionally been sold as heavy oil of wine, but this liquid has an entirely different composition, being a mixture of hydrocarbons, ethers, and ketones of varying boiling-points.

Spiritus Ætheris Nitrosi.—The present process (Redwood's) does not differ from that of the 1885 Pharmacopœia. The reactions that take place are very complicated; probably copper nitrate is first formed, and hydrogen set free. This latter reduces some nitric acid to nitrous acid, and this again reacts with ethyl alcohol, forming ethyl nitrite and water. The copper nitrate formed in the earlier reaction is decomposed by the sulphuric acid, nitric acid being regenerated and copper sulphate produced.

The reactions may be expressed by the following equations:

1. $Cu + 2HNO_3 = Cu(NO_3)_2 + H_2$.
2. $HNO_3 + H_2 = HNO_2 + H_2O$.
3. $HNO_2 + C_2H_5.OH = C_2H_5NO_2 + H_2O$.
4. $Cu(NO_3)_2 + H_2SO_4 = CuSO_4 + 2HNO_3$.

Or combining the equations in one:

$$2C_2H_5.OH + 2HNO_3 + 2H_2SO_4 + Cu_2 = 2C_2H_5NO_2 + 4H_2O + 2CuSO_4.$$

The distillate, consisting of a mixture of ethyl nitrite, alcohol, and its oxidation products, is dissolved in as much alcohol as will make the product contain 2½ per cent. of ethyl nitrite.

Small quantities of sweet spirit of nitre can be prepared without difficulty in a glass retort heated by means of a water-bath. Care must be taken that the temperature does not exceed 180° F. (82·2° C.) at any period of the operation, or violent decomposition will take place. Larger quantities are prepared in stoneware retorts heated by steam jackets similar to the one illustrated in the chapter on Distillation. A more modern apparatus consists of a large stoneware or glass vessel heated by means of a coil of *platinum* steam-piping, but owing to the high value of platinum, such stills are only to be found in the best equipped laboratories.

Pure ethyl nitrite may be kept for almost any length of time without decomposing, but the pharmacopœial preparation just described soon becomes acid, either from oxidation of aldehyde to acetic acid or from decomposition of the ethyl nitrite into alcohol and nitrous acid. To obviate this difficulty the spirit should be preserved in small bottles, hermetically sealed and kept in the dark. It is a matter of common knowledge that much fictitious spirit of nitrous ether is sold, which simply consists of a solution of ethyl nitrite in alcohol. Such a solution probably keeps better, but certainly does not represent the pharmacopœial article. Spirit of nitrous ether when properly prepared according to the official directions contains, as previously mentioned, notable quantities of aldehyde, and it is generally considered that to this latter much of the efficacy of the preparation is due.

Spiritus Ammoniæ Aromaticus.—According to the pharmacopœial directions a spirituous solution of the essential oils is first prepared by distillation, and reserved. Next the distillation is pushed a little further, and the aqueous distillate thus obtained used to dissolve the ammonium carbonate, with which is mixed the strong solution of ammonia. Finally the liquids are mixed and filtered through cotton wool.

Ammonii Carbonas B.P. is regarded as consisting of one molecule of acid carbonate of ammonium ($NH_4.HCO_3$) and one of ammonium carbamate ($NH_4.NH_2CO_2$). On the addition of water and solution of ammonia 2 molecules of neutral carbonate are formed, thus :

$$NH_4.HCO_3 + NH_4.OH = (NH_4)_2CO_3 + H_2O.$$
$$NH_4.NH_2CO_2 + H_2O = (NH_4)_2CO_3.$$

So that Spiritus Ammoniæ Aromaticus consists of an alcoholic solution of normal ammonium carbonate with a large excess of ammonium hydroxide.

Although the Pharmacopœia requires solution of ammonia to be free from tarry matter, it rarely is so; and it not infrequently happens that from this cause spirit of sal volatile turns slightly brown on keeping, especially in sunlight. The only remedy is to distil the solution of ammonia with the alcohol and oils. This requires the use of an earthenware still, such as described under Spirit of Nitrous Ether. As during the manufacture of spirit of sal volatile by this method some ammonia is unavoidably lost, it becomes doubly necessary to titrate the product as described under Volumetic Analysis.

Spiritus Ammoniæ Fœtidus.—In the first part of this process a tincture of asafœtida is prepared. This is then distilled without straining, and the distillate, which contains most of the volatile oil, mixed with the solution of ammonia.

Spiritus Armoraciæ Compositus.—The Pharmacopœia simply directs the scraped horseradish root, orange peel, and nutmeg to be added to the alcohol and water, and distilled. Oil of horseradish, which is the chief constituent of the distillate, has the same composition as volatile oil of mustard, and like the latter does not pre-exist in the plant, but is formed by the fermentation of the glucoside potassium myronate with myrosin, allyl isothiocyanate, glucose and a potassium salt being formed. Having regard to this, it would appear better pharmacy to allow the scraped horseradish to macerate in water at the ordinary temperature for several hours, so as to ensure completion of the decomposition, as the ferment myrosin is destroyed on heating.

CHAPTER XV

TINCTURES

The tinctures of the Pharmacopœia may be divided in two ways, either according to their methods of preparation or according to their dosage.

Methods of Preparation

1. *Maceration.*—The bruised material is allowed to remain in contact with the whole of the menstruum in a closed vessel for a definite period, agitating at intervals. When exhaustion is considered to be complete the liquid is strained off and the marc submitted to pressure, when more liquid is obtained. These liquids are finally mixed and filtered. In maceration simple the tincture is not made up to any definite bulk, it being argued that any liquid retained in the marc must be of the same strength as the expressed tincture.

The following table gives the specific gravity and product of tincture obtained by maceration in one pint of the official menstruum :

	Sp. gr.	Volume.
Tinctura Aurantii	·876	21·6 fluid ounces.
,, Calumbæ	·918	19·2 ,,
,, Cantharidis	·834	20·0 ,,
,, Capsici	·894	19·6 ,,
,, Cardamomi Compositus	·945	19·4 ,,
,, Catechu	·976	18·6 ,,
,, Cocci	·949	19·1 ,,
,, Croci	·920	18·4 ,,
,, Gentianæ Compositus	·965	18·5 ,,
,, Lavandulæ ,,	·837	... ,,
,, Limonis	·876	21·6 ,,
,, Lupuli	·931	18·1 ,,
,, Pruni Virginianæ	·840	18·0 ,,
,, Quassiæ	·941	19·4 ,,
,, Scillæ	·972	16·8 ,,
,, Sennæ Compositus	·991	18·5 ,,
,, Sumbul	·900	18·8 ,,
,, Valerianæ Ammoniatæ	·936	18·0 ,,

Tinct. Aurantii	The rind of the fresh ripe fruits contains about 60 per cent. of water.
Tinct. Limonis	

Tinct. Pruni Virginianæ.—The bruised material is first allowed to macerate in cold water, fermentation being set up, and a volatile oil containing hydrocyanic acid being formed (compare Ol. Amygd. Essent.).

2. The following tinctures are prepared by maceration, with the difference that the product is made up to a definite bulk :

	Sp. gr. of finished tincture.		Sp. gr. of finished tincture.
Tinctura Aloës	·975	Tinctura Myrrhæ	·850
,, Asafœtidæ	·915	,, Opii Ammoniatæ	·900
,, Guaiaci Ammoniatæ	·899	,, Podophylli	·844
,, Kino	·998		

3. *Percolation.*—The bruised material after thoroughly moistening with a portion of the menstruum is packed in a percolator, and exhausted by allowing the remainder to trickle slowly through (see Extraction). The marc is then removed, submitted to pressure, and the expressed liquid which is necessarily weak in extractive, filtered and mixed with the percolate, sufficient of the original menstruum being added to produce the required volume.

	Sp. gr. of the finished tincture.		Sp. gr. of the finished tincture.
Tinctura Aconiti	·893	Tinctura Hydrastis	·923
,, Arnicæ	·974	,, Hyoscyami	·950
,, Buchu	·928	,, Jaborandi	·950
,, Cascarillæ	·898	,, Krameriæ	·930
,, Chiratæ	·922	,, Lobeliæ Ætherea	·815
,, Cimicifugæ	·928	,, Pyrethri	·905
,, Cinnamomi	·900	,, Quillaiæ	·921
,, Colchici Seminæ	·950	,, Rhei Compositus	·971
,, Conii	·896	,, Senegæ	·935
,, Cubebæ	·845	,, Serpentariæ	·895
,, Digitalis	·928	,, Stramonii	·952
,, Ergotæ Ammoniatæ	·937	,, Strophanthi	·890
,, Gelsemii	·925	,, Zingiberis	·840
,, Hamamelidis	·948		

4. The following tinctures are standardised (see Assay) :

	Sp. gr. of the finished tincture.	Proportion of active ingredient.
Tinctura Belladonnæ	·917	·05 gram in 100 c.c.
,, Cinchonæ	·916	1·00 ,, ,,
,, ,, Compositus	·913	·50 ,, ,,
,, Jalapæ	·910	1·50 ,, ,,
,, Nucis Vomicæ	·912	·25 ,, ,,
,, Opii	·953	·75 ,, ,,

5. The following tinctures are prepared by simple admixture:

	Sp. gr. of the finished tincture.
Tinctura Camphoræ Compositus	·915
,, Cannabis Indicæ ...	·846
,, Chloroformi et Morphinæ Co.	1·011
,, Ferri Perchloridi ...	1·086
Tinctura Iodi	·878
,, Quininæ	·887
,, ,, Ammoniata	·928
,, Tolutani	·867

Tinct. Ferri Perchloridi.—When this tincture is exposed to sunlight it gradually becomes lighter in colour, owing to reduction of some of the iron to the ferrous state.

Tinct. Iodi.—It has been stated that iodine slowly reacts with alcohol at ordinary temperatures, hydriodic acid and other compounds being formed.

Dosage.—According to their potency the tinctures fall into two great classes,—the stronger with dose from 5 to 15 minims, and the weaker dose half to one fluid drachm. Tincture of iodine forms the solitary exception, dose 2—5 minims. The following tinctures have an ordinary dose, ranging from 5 to 15 minims; all the others half to one fluid drachm:

Tinctura Aconiti.
,, Belladonnæ.
,, Cannabis Indicæ.
,, Cantharidis.
,, Capsici.
,, Chlorof. et Morphinæ Comp.
,, Cocci.
,, Colchici Seminæ.
,, Croci.
Tinctura Digitalis.
,, Ferri Perchloridi.
,, Gelsemii.
,, Lobeliæ Ætherea.
,, Nucis Vomicæ.
,, Opii.
,, Podophylli.
,, Scillæ.
,, Stramonii.
,, Strophanthi.

EXTRACTS

The extracts of the Pharmacopœia may be generally divided into three classes: (1) those in which the active principles are readily dissolved out by water; (2) those in which the active principle is of an alkaloidal or resinous nature, soluble in alcohol; (3) evaporated fruit juices. Besides the above there is one ethereal extract, that of male fern, and one alcoholic extract combined with other ingredients, viz. compound extract of colocynth, which if placed in its proper category should find a place with the pills.

The *watery extracts* may be subdivided as follows:

Evaporated infusions:
Extract of aloes, dry.
„ ergot, liquid.
„ liquorice, soft.
„ „ liquid.
* „ opium, liquid.
* „ „ soft.

Evaporated decoctions:
Extract of chamomile, soft.
„ gentian, soft.

Evaporated aqueous solutions obtained by infusion followed by percolation:
Extract of cascara, dry.
„ „ liquid.
* „ cinchona, liquid.
„ krameria, dry.
„ pareiræ, liquid.
„ rhubarb, dry.

The alcoholic extracts may be looked upon as evaporated strong tinctures, prepared by the ordinary processes of maceration and percolation. Two—the extracts of belladonna and nux vomica—are obtained by re-percolation.

Evaporated strong tinctures:
*Extract of belladonna, soft.
„ „ liquid.
„ cannabis indica, soft.
„ cimicifuga, liquid.
„ coca, liquid.
„ ergot, soft.
„ hamamelis, liquid.
„ hydrastis, liquid.
* „ ipecacuanha, liquid.
„ jaborandi, liquid.
„ jalap, soft.
* „ nux vomica, soft.

Evaporated strong tinctures:
*Extract of nux vomica, liquid.
„ calabar bean, soft.
„ sarsaparilla, liquid.
„ stramonium, soft.
„ strophanthus, dried.
„ taraxacum, liquid.

Evaporated plant juices:
Extract of belladonna, green.
„ hyoscyamus, green.
„ colchicum, soft.
„ taraxacum, soft.

The green extracts of belladonna and henbane are prepared by crushing the flowering tops of the plant between granite rollers, or on a small scale by pounding in a stone mortar; the bruised material is then wrapped in press cloths or packed in horsehair bags and submitted to pressure. The expressed juice is next gradually heated in earthenware or block-tin steam-pans to 130° F. (55·4° C.); this effects separation of the green colouring matter, which is removed by straining through a calico filter and preserved. The filtrate is then heated to 200° F. (93·3° C.) for five minutes, to coagulate albuminous matter which is filtered out. The clear brownish coloured filtrate is then carefully evaporated with constant stirring at a temperature not exceeding 140° F. (60° C.) to the consistence of thin syrup. The green colouring matter, which should in the meantime have been squeezed through muslin or pressed through a fine hair sieve, is then

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* These extracts are standardised (see chapter on Alkaloidal Assay).

added and carefully mixed in, and the whole evaporated to a firm extract.

A somewhat different procedure is adopted for the preparation of the extracts of colchicum and taraxacum, as no green colouring matter is present. The material is crushed as before, the juice expressed and set aside for an hour to deposit; the decanted liquor is then heated to 212° F. (100° C.) for ten minutes to coagulate albuminous matter, which is removed by filtration, and the clear liquid evaporated at a temperature not exceeding 160° F. to the consistence of a firm extract.

Extract of malt is prepared by evaporating an infusion of malt to the consistence of a soft viscid extract. The malt should only be crushed as required, as it rapidly absorbs water and becomes heated. The crushing mill should not produce much powder, or subsequent filtration is rendered very tedious. The temperature at which the malt is infused, or, as it is technically called, mashed, must not be above 160° F., to avoid injury to the diastase. It is curious that distilled water exerts scarcely any solvent action on the albuminous matter; for this reason hard water is always preferred, or at any rate water containing calcium sulphate.

When the infusion is complete the clear liquor is syphoned off, the grain submitted to hydraulic pressure, and the mixed liquors filtered by means of a vacuum filter. The filtration should be hastened as much as possible, as acetification soon sets in and impairs the product. The clear infusion is next rapidly evaporated in a vacuum-pan, the temperature not being allowed to rise above 125° F., until the specific gravity of the cold extract reaches 1·375, when it is almost semi-solid, but still perfectly transparent. The yield varies from 90 to 95 per cent.

According to J. C. Umney, *liquid extract of malt* is prepared by mixing the thick extract with 70 per cent. alcohol, added very cautiously so as to prevent precipitation of the diastase. The product should contain about 7 per cent. of alcohol, and have a specific gravity of 1·25 at 60° F.

Estimation of the diastasic value.—Extract of malt prepared as above should be capable of digesting its own weight of gelatinised starch in from ten to fifteen minutes.

Mix one gram of sifted arrowroot with a little water until perfectly smooth; dilute with about 120 c.c. water and boil for twenty minutes, cool, and make up to about 120 c.c. Place the gelatinised starch in a flask capable of holding 200 c.c, and heat in a water-bath until the temperature is constant at 100° F.

Now add one gram of malt extract dissolved in a little water, and continue the heat, shaking occasionally until 1 c.c. of the mixture when cooled ceases to give a blue colour with solution of iodine. With good samples this conversion of starch into dextrin and maltose takes from five to ten minutes, but should never exceed fifteen minutes.

Malt extract with cod-liver oil.—A good malt extract is capable of emulsifying 25 per cent. of cod-liver oil without any addition of mucilage, &c. The mixing is generally effected by machinery, and takes about six hours to complete, with the mixers making twenty revolutions a minute. The air should be excluded as much as possible ; indeed, with the newest forms of apparatus the mixing takes place in a vacuum, the finished product being nearly transparent when viewed in the field of a microscope. It is not, however, to be regarded as a solution of oil in malt extract, the fact being that the refractive indices of the two bodies are almost identical, so that it is practically impossible to see the globules of oil.

CHAPTER XVI

LINIMENTS

Liniments (from the Latin *lino,* I smear) are applications for external use, and are intended to be rubbed or painted over affected parts of the body. The terms embrocation and liniment are now commonly used indifferently, but the former term, from its Greek derivation, ought to be restricted to such preparations as are partly lotions and partly liniments.

The liniments of the Pharmacopœia, with the exception of liniment of potassium iodide and soap, are liquid compounds, composed of oil, soaps, or alcoholic preparations, either alone or in combination.

Aconite liniment is really a very strong tincture of the root, containing camphor, prepared in the usual way by maceration and percolation. Ammonia and lime liniments partake of the nature of soaps, and form creamy emulsions in which the excess of oil is suspended. Lime liniment is commonly known as "carron oil," probably after the great Carron Ironworks, where it was supposed to have been originally introduced for the treatment of burns and scalds.

Liniment of potassium iodide and soap.—In the preparation of this liniment particular attention should be paid to the shredding of the curd soap. This is best accomplished by fixing a bar of the soap against a short nail or screw in the working bench, and then taking off thin broad shavings with an ordinary joiner's plane. If managed properly the shavings will be thin enough to dissolve rapidly on warming with the water, but in any case the tare of the vessel should be carefully noted, so that the loss by evaporation may be afterwards made up. When the solution is complete, it is strained through muslin on to the powdered potassium iodide in a large mortar. Trituration must be continued until cold, when the oil of lemon should be added. The addition of the last ingredient not only covers the odour of the soap, but it produces a much more creamy liniment.

If soft soap is used, a transparent liniment of the consistence of thin honey results. The author has found that a liniment prepared with curd soap only, very soon becomes granular, but if a little soft soap is substituted for an equal weight of curd soap the liniment retains its creamy consistence for a much longer period.

Turpentine liniment.—The formula now given for this liniment produces a thick creamy white emulsion, which can only be dispensed in wide-mouthed bottles. To obtain a good result the solution of camphor in turpentine must only be added in very small quantities at a time to the mixture of soap and water. Each addition must be thoroughly incorporated before adding more. Everything depends upon this, as a liniment started badly cannot afterwards be set right by any amount of trituration. The student is recommended to read the chapter on Emulsions before attempting this liniment for the first time, as it really requires considerable skill in manipulation.

CHAPTER XVII

INFUSIONS

At one time fresh infusions were very largely prescribed, and it is much to be regretted that they are gradually falling into disuse, especially as it is not possible to substitute any other class of preparations in their stead.

Infusions, as the name implies, are preparations made by pouring water, generally boiling, but in some cases tepid or even cold, upon coarsely comminuted vegetable substances, and allowing to stand (infuse) for a definite period, the solid particles being subsequently removed by straining. The period for infusion varies; it is shortest where the properties are chiefly due to volatile bodies, and longest when it is desired to take up extractive or mucilaginous matter. It is noteworthy that the period for infusion has been considerably shortened, substances which in the 1885 edition were infused for two hours now being considered sufficiently exhausted after standing a quarter of that time. It is not an infrequent practice to leave infusions in the vessels in which they are made, in contact with the solid ingredients for a much longer time than that specified in the Pharmacopœia, the infusion being poured off from time to time as wanted, until the whole has been used, or a fresh set of ingredients are put under operation. This practice, by which a continued cold maceration is maintained, is very objectionable, as many infusions assume a very altered character from prolonged contact with the solid ingredients.

The preparation of infusions is exceedingly simple, the water being simply poured upon the requisite quantity of material in an earthenware jar, as Fig. 142 or Fig. 143, covering closely, and allowing to stand, with an occasional stir, for the prescribed period, after which the clear infusion is strained off. Fig. 143 is sometimes known as Squire's infusion pot. It is constructed on the principle originally suggested by Mr. Alsop. The solid substance to be infused is contained in the upper perforated portion of the vessel instead of being allowed to subside to the bottom. Mr. Alsop observed that when infusions were prepared in a plain pot in the usual way, the water in contact with the solid ingredients became saturated or highly charged with soluble matter, and thus formed a dense stratum at the bottom, which had little tendency to diffuse through the less saturated liquor above. Mr. Alsop proposed the addition of a perforated diaphragm about halfway between the top and bottom of the pot, on which to place the solid ingredients; for this Mr. Squire substituted a perforated earthenware cup, which could be lifted clean out of the finished infusion, thereby avoiding the trouble of straining.

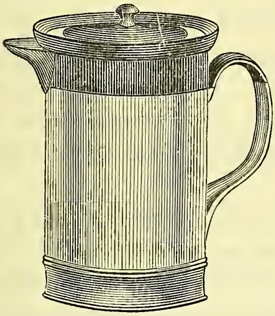

Fig. 142.—Infusion pot.

Fig. 143.—Squire's infusion pot.

As has been already stated, infusions are mostly made with boiling water, and the drugs so exhausted are generally of an aromatic character. Infusion of calumba, which contains much starch, is prepared with cold water. Infusion of quassia is also made with cold water; infusion of chiretta, if made with boiling water becomes intensely bitter and nauseous, but the Pharmacopœia disregards this.

Vegetable infusions are very prone to decomposition, and with one or two exceptions, notably those of cloves and roses, should be made freshly every morning or as required. In hot weather infusion of digitalis will scarcely keep good for more than three or four hours, and the infusions of calumba and senna are nearly as readily affected. Various methods have been suggested from time to time for the keeping of infusions, but in no case should the addition of formalin, salicylic or boric acid, or other preservative be countenanced. The least harmful process, perhaps, consists in almost filling the bottles with the strained infusion, placing in a water-bath, and heating to boiling for fifteen minutes on three successive days, during which time the mouths of the bottles are loosely plugged with sterilised cotton. On the third day the bottles may have a piece of clean paper twisted over the neck, and be set aside in a cool place. If the sterilisation has been effective such bottles may be kept for a considerable length of time, but they can hardly be said to accurately represent freshly made infusions.

Of recent years there has been a growing tendency on the part of some pharmacists and many medical men to *substitute* liquids, prepared by diluting *so-called concentrated infusions* in the proportion of 1 to 7 or 1 to 9 with water, for real infusions. Such preparations do not and cannot represent the official article, and those who dispense such do so in direct opposition to the pharmacopœial instructions.

Concentrated infusions that will keep are a pharmaceutical impossibility, those sold as such being neither more nor less than tinctures or extracts, prepared as a rule by the ordinary methods. It will be argued by some, and for obvious reasons, that since the introduction of vacuum pans vegetable infusions can be concentrated without the slightest deterioration, but let any one prepare infusion of gentian by the official process and compare it with that obtained by diluting a "concentrated infusion," and the difference will be readily apparent both to the sense of smell and of taste.

Since the above was written a new edition of the Pharmacopœia has been issued, and it is with infinite regret that the author learns that a class of remedies has been introduced (concentrated vegetable liquors) purporting to represent, when diluted, fresh infusions. Comment on them is needless.

CHAPTER XVIII

DECOCTIONS

Decoctions (L. *de*, and *coquo*, I cook) are aqueous preparations obtained by boiling drugs with water in order to extract principles which would not be readily taken up by simple infusion. The process of decoction is not applicable to substances containing aromatic or volatile principles, but is confined to the exhaustion of such drugs as owe their properties to astringent and extractive matter.

The general method for the preparation of decoctions, consists in boiling the sliced or coarsely bruised materials in a covered vessel with distilled water for a specified time, straining, and making the product up to measure with more water.

For making quantities of one to two pints earthenware vessels (Fig. 144) are to be preferred. Copper vessels, which have been well tinned inside, may, however, be used, but are not so safe as the foregoing. Saucepans made in block aluminium may now be obtained, and possess many advantages over all others. Care should be taken only to select such as are turned or spun out of a single sheet of metal, as vessels in which solder has been used deteriorate very rapidly. Iron or tinned iron vessels should never be used, on account of the discoloration occasioned in decoctions containing astringent matter.

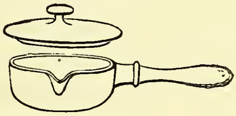

Fig. 144.

Decoctions, like infusions, are very liable to decomposition, and should only be prepared as required. The remarks under the heading of Infusions concerning addition of preservative agents apply equally to decoctions.

CHAPTER XIX

MEDICATED WATERS

The medicated waters of the Pharmacopœia consist of aqueous solutions of the volatile principles of vegetable substances. The solitary exception is Aqua Chloroformi.

Various methods have been proposed from time to time for the preparation of the above class of remedies, such as simple solution in cold or hot water, percolation through an absorbent substance impregnated with the medicament, and distillation.

Simple solution can only be made use of in the case of essential oils. *Cold* water is not a satisfactory solvent, owing to the length of time necessary for saturation; but if an essential oil is shaken up with *hot* distilled water, the latter after cooling and filtering is found to possess most of its medicinal properties.

The preparation of medicated waters by percolation through absorbent cotton strongly impregnated with an essential oil, was formerly official in the Pharmacopœia of the United States. The distribution of the oil, was however, found to be a tedious and wasteful process, the cotton requiring careful picking or carding after each addition. When uniformly impregnated the cotton was packed in a glass funnel, and exhausted by percolation with distilled water.

In the last edition of the U.S.P. the use of absorbent cotton has been discontinued, the essential oil being divided by trituration with calcium phosphate. On the addition of water a milky mixture is formed, which is filtered through a well-wetted filter-paper.

The use of absorbent powders for minutely subdividing the oil is not new, magnesium carbonate having been used for the same purpose for many years. A small quantity of the carbonate, however, dissolves in the water, and in some cases materially alters the character of the finished product. The most important objection to the use of magnesium carbonate or oxide lies in their alkaline reaction, aromatic waters prepared by their aid precipitating alkaloids from neutral solutions of their salts, which in the case of potent remedies might be a source of danger. Instead of calcium phosphate any other *insoluble* powder may be used, such as kaolin, powdered glass, or pumice.

Preparation by distillation.—This is the official process, and is undoubtedly the best. All odorous plants yield their perfume and, to a certain extent their taste, to water which has been distilled from them. This is chiefly due to volatile oils which pass over with the vapour of water, but in many instances the flavour of such distilled waters differs considerably from that of the aqueous solutions of the corresponding volatile oils. It is therefore assumed that besides the essential oils, other volatile bodies, such as acids and ethers, pass over and remain dissolved in the water, thereby modifying and generally improving the flavour.

Parts of plants which, if subjected to drying, lose their volatile constituents, are directed to be distilled in the fresh state. Examples are found in the distilled waters of orange, rose, and elder flowers. None of these flowers bear drying without deterioration; the two latter are sometimes "pickled," that is preserved with dry salt, when they keep exceedingly well for a considerable period, and may be distilled as required. This is an advantage, as all aromatic waters lose their sweetness by long keeping. On the other hand, many freshly distilled aromatic waters require to be kept in loosely covered vessels for a short time, before the "smell of the still" as it is technically called, passes off.*

In cases where the oil-cells are well protected, as in the fruits of the Umbelliferæ, cinnamon bark, &c., the material may be dried and preserved for a long time without appreciable change. Small fruits, as those of dill and caraway, and leaves which contain oil-cells near the surface, may be distilled with water without previous preparation; but when the oil-cells are more deeply situated, as in the case of cinnamon bark and pimento fruits, the material should be well bruised before placing in the still.

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* It must not be assumed that all flowers yield their fragrance to water. On the contrary, the character of most of them is completely destroyed; thus it has been found impossible to obtain the delicious perfume of the violet or wallflower by distilling them with water, or indeed by distillation at all. It has, however, been found that the odour of many flowers is extracted by macerating them at a low temperature in melted animal fat. From this fat, the perfume can be again obtained, by digesting with purified alcohol. Quite recently, animal fat has been superseded by odourless liquid hydrocarbons of high boiling-point, which are preferred chiefly on account of their generally inert character.

To obtain medicated waters of the finest quality, great care must be paid to the details of distillation, especially when it is performed over a naked fire. To avoid risk of burning, and consequent production of empyreumatic odours, the material is generally placed on a wire grating or false bottom, as described under Distillation, p. 60. If steam is used, the danger of burning is avoided; indeed, steam distillation should be made use of wherever possible, the risk of over-heating then being reduced to a minimum. In all cases, the watery vapours should be rapidly and completely cooled in the condenser, to avoid chemical changes in the volatile oil, which in its finely divided condition are more likely to take place. Moreover, waters that have been cooled under bad conditions very frequently become ropy on keeping, even when protected from the air and light.

Of the official medicated waters, six are obtained by simply distilling one pound of the material with sufficient water to produce one gallon of distillate:

Aqua Anethi	*Strength.*—1 pound to 2 gallons of water. Distil 1 gallon.
„ Anisi	
„ Carui	
„ Cinnamomi	
„ Fœniculi	
„ Pimentæ*	* ½ pound for 1 gallon.

The two mint waters are obtained, as before, by distilling the respective essential oils with water, the product in each case being a practically saturated solution:

Aqua Menthæ Viridis	*Strength.*—77 minims to 1½ gallons of water. Distil 1 gallon.
„ „ Piperitæ	

Aqua Laurocerasi is unaltered. The distillate from the freshly bruised leaves contains a varying amount of hydrocyanic acid, and possesses an agreeable odour resembling that of bitter almonds. As the medicinal virtue of this water depends upon the hydrocyanic acid, it has been suggested to substitute for it a preparation made by distilling bitter almonds with water. It is a recognised fact that dilute solutions of bitter almond oil are much more stable than the corresponding simple aqueous solutions of hydrocyanic acid, and there would be no pharmaceutical difficulty in preparing a weak alcoholic solution of oil of bitter almonds, containing 2 per cent. of hydrocyanic acid, in place of Acid. Hydrocyanicum Dilutum; or an entirely aqueous solution of 0·1 per cent. strength, to replace Aqua Laurocerasi. As the latter is commonly used for eye lotions, alcohol could not be used in its preparation. The desirability for change, is evident to many practical pharmacists, as Aqua Laurocerasi prepared according to the pharmacopœial directions, rarely, if ever, contains the proper proportion of hydrocyanic acid, and has to be fortified by the addition of chemically prepared acid in consequence.

Strength.—Adjusted to contain 0·1 per cent. of hydrogen cyanide (one twentieth the strength of Acid. Hydrocyanicum Dilutum).

Aqua Aurantii Floris.—No directions are given for the preparation of this pleasantly odorous water, but the description is intended to cover the so-called "triple" orange-flower water of commerce. This is practically a saturated aqueous solution of the volatile constituents of the orange flowers, chiefly imported from the South of France, where large quantities are obtained as a by-product in the distillation of neroli oil. It is however, a curious fact that when neroli oil itself is distilled with water, the distillate does not at all correspond with the orange-flower water as imported, a result which points to the fact, that the latter probably contains one or more fragrant principles freely soluble in water, which are not contained in the essential oil.

For dispensing purposes the imported water may be diluted with two parts of water.

Aqua Rosæ.—It is somewhat to be regretted, even if only for sentimental reasons, that the Pharmacopœia has discontinued the directions for the preparation of this water. Large quantities of *Rosa centifolia* are grown in this country, and there is no reason why pharmacists should not prepare the water for themselves, ten pounds of the fresh or salted petals being used for every gallon of distillate. For dispensing purposes the "triple rose water" of commerce may be diluted with two parts of water.

Aqua Sambuci.—Prepared by distilling ten pounds of fresh or salted elder flowers, freed from stalks, with sufficient water to produce one gallon. Elder-flower water when freshly distilled has not an altogether pleasant odour, and requires re-distillation after being kept in somewhat loosely covered vessels for a week or so.

Aqua Camphoræ.—Strength, 0·1 per cent. A radical change has been made in the preparation of camphor water, but the practical outcome is about the same, the difference being that according to the new formula it contains nearly 0·3 per cent. of alcohol.

Water *saturated* with camphor at 60° F. contains as nearly as possible 0·1 per cent.; and as the new preparation is only required to be of this strength, there seems no particular advantage to be derived from the presence of the alcohol, except perhaps to hasten solution.

Aqua Chloroformi now contains 0·25 per cent. of chloroform, and is exactly half the strength of Aq. Chlorof., B.P. 1885. This is a great improvement, as there will be less tendency for the chloroform to be thrown out of solution by the addition of salts, when prescribed in mixtures, &c.

CHAPTER XX

SOLUTION

Solutions are homogeneous mixtures which cannot be separated into their constituent parts by mechanical means (Ostwald). The ability of substances to form such mixtures or solutions, varies according to the state of aggregation of the substances.

All gases mingle with one another in all proportions to form homogeneous mixtures, except when they combine to form chemical compounds. A large number of liquids are known to dissolve in one another, but the number of pairs of liquids which mutually dissolve in all proportions is exceedingly small. The solution of solids in solids, although an established fact, need not now be considered.

Solutions of gases in liquids.—Every liquid possesses the property of mingling with every gas to form a homogeneous mixture or solution. According to the law of Henry, "*the quantity of a gas dissolved by a specified quantity of a liquid is proportional to the pressure of the gas.*" Gases with few exceptions are more soluble in cold liquids than in hot, and as a rule a gas can be expelled from a liquid simply by raising the temperature.

When a gas is brought into contact with a liquid, absorption begins at once; the surface layer of the liquid first becomes saturated, the dissolved gas mixing with the lower layers by diffusion, and the surface layer again dissolving a fresh portion. This process goes on until the whole bulk of the liquid becomes saturated with the gas. The foregoing, however, only takes place very slowly, and in commercial operations it is found necessary to hasten solution by bubbling the gas through the liquid under pressure, or by agitating the liquid so as to constantly bring fresh portions into intimate contact with the gas.

Solution of liquids in liquids.—Unlike gases, liquids show considerable difference in their behaviour to each other, the differences depending upon the nature of the liquids. Pairs of liquids are divided by Ostwald into three classes. *First,* pairs of liquids that behave to each other in the same manner as gases; that is, they mix with one another in all proportions, as alcohol and water. *Secondly* pairs of liquids that, although dissolving in one another, do not do so in all proportions, of which ether and water are good examples. *Thirdly,* those pairs of liquids which exert no mutual solvent action, as olive oil and water. Very few liquids, strictly speaking, belong to this class, as all liquids which show an appreciable vapour pressure at the temperature of the experiment must be excluded,—a solution of a vapour not being distinguishable from a solution of the liquid itself.

Solution of solids in liquids.—A solid body is said to dissolve in a liquid when their reaction results in a homogeneous mixture. When the quantity dissolved reaches a point when no more can be taken into solution under the same conditions, the liquid is said to be *saturated.* The point of saturation is entirely dependent upon temperature. When this is changed, either some of the dissolved solid passes out of solution, or the liquid dissolves a further quantity. As a general rule, solid bodies dissolve more freely in hot than in cold liquids; among others, exceptions are found in common salt, which is about equally soluble in hot and cold water; and calcium sulphate, which, though moderately soluble in cold water, dissolves but sparingly if the water be heated.

Nernst ascribes to all solid substances in solution, a certain *solution-pressure,* which is in all respects analogous to vapour-pressure.

"The molecular conception which we are able to form of the process of solution, and of the existence of a solution-pressure, is connected on one side with the theory of the mutual solubility of liquids, and on the other side with that of the processes of melting. By reason of their thermal energy, the molecules of a solid tend to separate from one another; this tendency is counteracted by the internal pressure, which acts normal to the surface, towards the interior, and is existent in solids as well as in liquids. When a solid is brought into contact with a liquid which exerts an action on the molecules situated on the surface of the solid, the internal pressure is diminished, and a number of molecules of the solid are removed from the surface and find themselves in the interior of the liquid. This process continues, until the number of molecules passing from the liquid to the solid and remaining there, is equal to the number of molecules leaving the surface of the solid. As the stationary equilibrium thus attained, is conditioned solely by the processes which occur at the surfaces in contact, it is independent of the relative quantities of the two components" (Östwald).

The solution of many bodies causes a considerable reduction in the temperature, advantage of which is taken in the preparation of freezing mixtures. Tilden observed that many liquids appear to exercise a certain selective action in the solids they take up. Thus water dissolves a number of salts which are practically insoluble in alcohol. On the other hand, alcohol dissolves a great many carbonaceous substances, such as organic bases, resins, and camphor, which are scarcely affected by water ; and without straining the idea too far, it is not too much to say, that in many cases a relationship is observable between the chemical composition of the solvent, and that of the solid it dissolves. Ether and benzene, both highly carbonaceous liquids, dissolve fats and other substances rich in carbon ; phosphorus trichloride dissolves phosphorus ; carbon bisulphide is the best solvent for the common form of sulphur. The relation cannot however, be said to be general, and at present we possess few clues to the laws regulating the solution of solids in liquids.

The rate of solution is directly proportionate to the extent of surface exposed to the action of the solvent. It follows from this that solid bodies should be reduced to small particles before adding to the liquid, when speedy solution is desired.

The ordinary method of determining the solubility of a substance in a given solvent, is to place a convenient volume of the latter in a small flask, the powdered substance being added until at last a little remains undissolved. The mixture is then warmed gently until complete solution is effected, when it is set aside for twenty-four hours, shaking and stirring occasionally. The solution is next filtered through paper into a tared capsule and weighed. After evaporating until the weight remains constant, it is again weighed, when the difference gives the weight of solid in the amount taken, and from this, the ratio of solubility may be readily calculated.

When the solvent is of a very volatile nature, considerable difficulty is experienced in obtaining concordant results, when the foregoing process is employed. Very accurate results may however be attained, by the use of the *lysimeter*, an apparatus devised by Dr. Charles Rice, of New York, and described by him in the 'Journal of the American Chemical Society' for October, 1894.

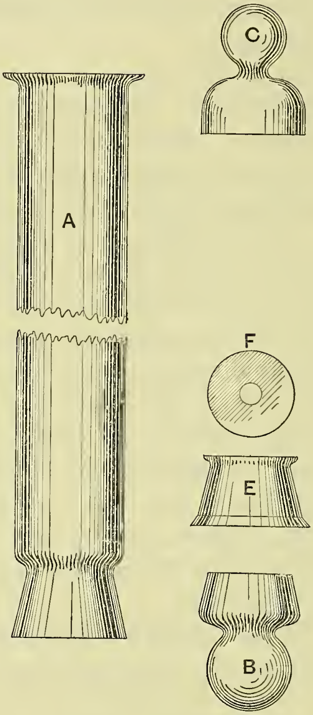

Fig. 145.—Lysimeter.

The apparatus (Fig. 145) consists of a glass tube A, 15 centimetres long and 1 centimetre in diameter, provided at one end with a well-ground stopper C, while the other end is cup-shaped, there being a contracted neck between the cup and the main tube. Into this cup a carefully-ground glass bell E is fitted, having a small perforation in its bottom (as shown in F). There is also a stopper B, which is carefully ground to fit into the cup, and which is inserted after the glass bell E has been removed. The several stoppers, &c., are all numbered to show where they belong.

The lysimeter is prepared for use by inserting the stopper C, and introducing into the cup-shaped end, the glass bell E, containing a plug of absorbent cotton. A sufficient amount of liquid having been introduced into a beaker, heat is applied and the powdered substance added until, after the boiling has been kept up for some time, a small quantity remains undissolved. The prepared glass tube is now inserted in the liquid. As long as the stopper C closes the upper end of the tube, no liquid will be able to filter in. When the tube has acquired the temperature of the boiling liquid, the stopper C is removed, when the liquid will begin to filter through the cotton and rise in the tube until the levels correspond. In order to ensure perfect uniformity of the liquid within and without the tube, it is best to allow the filtered portion to flow back through the cotton, and again filter in. The stopper C having been inserted, the tube is withdrawn, turned upside down, the filtering cup removed, and the stopper B inserted. The tube is now carefully cleaned and laid aside until cold. Its tare having been previously determined, the increase in weight represents the weight of the solution contained therein. On washing the contents into a tared dish and evaporating, the weight of the dissolved substance will be found.

Saturated solutions.—Saturated solutions are liquids impregnated to such an extent with a soluble substance, that no more can be taken up at the normal temperature. It has, however, been found that certain solutions refuse to crystallise, even when cooled below the temperature at which they ordinarily yield crystals. Such liquids are said to be *super-saturated.* Thus if water saturated with alum at 60° F. is gently warmed, and a crystal or two of the same salt added, it will dissolve, and in many cases will not crystallise out again on regaining the normal temperature. If, however, a small crystal of alum be dropped into the liquid, crystallisation at once commences, and proceeds so rapidly that in a short time the whole sets into a solid mass. In these cases, crystallisation is not started by shaking or stirring the liquid, nor by the introduction of a crystal of another salt, unless it be isomorphous with the dissolved substance.

Percentage solutions.—Strictly speaking, a percentage solution contains a definite weight of a substance, in 100 parts by weight of solution. Thus a 2 per cent. solution of cocaine hydrochloride, is made by dissolving 2 grains of the salt in 98 grains of water. The weight of substance and solvent necessary, to produce a definite quantity of a percentage solution, may be ascertained by applying the following rule:—Multiply the quantity of solution desired in grams, ounces, or grains by the percentage, and divide the product by 100. The quotient is the quantity of substance necessary; and if this figure is subtracted from the total quantity of solution desired, the remainder indicates the proper weight of liquid to be used. Thus, supposing 4 fluid ounces of cocaine hydrochloride solution, 5 per cent. strength, are required: 4 fluid ounces contain 1750 grains. Then 1750 × 5 ÷ 100 = 87·5. Therefore 87½ grains of the salt are to be dissolved in water, and the weight made up to 4 ounces.

It occasionally happens that the weights of substance and solvent are inconvenient to deal with practically, as when such a fraction as one third of a grain requires to be weighed; in this case the quantity nearest in weight to that required must be made, even when it entails slight loss.

In this country, *so-called* percentage solutions are often prepared by dissolving parts by weight in parts by measure, as 1 grain in 100 minims. It is hardly necessary to say that such solutions are not true percentage solutions; and although the error may appear slight when reckoned on 100 minims, yet if a gallon of solution were prepared on the same lines, it would be very considerable. Thus a gallon of solution of mercuric chloride, 1 in 1000, should contain 70 grains, but if prepared by dissolving the poison at the rate of 1 grain in 1000 minims it would contain 6·8 grains too much.

*Formulæ for increasing or decreasing the strength of percentage liquids**

I. Given a liquid of known weight and percentage: required to know the quantity of a liquid of the same kind, of higher or lower percentage, or of water which will have to be added to produce a desired percentage.

Let a be the quantity of the given liquid;
    b its percentage of important constituents;
    c the percentage of the liquid to be mixed with a;
    d the desired percentage of the mixture;
    x the quantity of the liquid, of higher or lower percentage, or the quantity of water to be added to a.

(1) Then $x = \dfrac{a\,(b-d)}{d-c}$ *if the liquid to be added is of higher or lower percentage*; or in words:

To find x (the quantity of liquid to be added), multiply the difference between the percentage of the liquid a and the desired

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* E. Claassen.

percentage of the mixture, by the quantity of the liquid a, and divide the product, by the difference between the desired percentage and that of the liquid to be mixed with a.

Example.—Supposing we have 40 parts of a liquid of 50 per cent., and want to mix it with a liquid of 20 per cent., in order to get one of 30 per cent., how much of the liquid of 20 per cent. should be added? x (the quantity of the liquid of 20 per cent.) =

$$\frac{40\ (50-30)}{30-20} = \frac{40 \times 20}{10} = 80.$$

(2) *If water is to be added,* $x = \frac{a\ (b\ -\ d)}{d}$; or in words:

To find x (the quantity of water to be added), proceed as described under (1), but divide the product by the desired percentage only.

Example.—Supposing we have 40 parts of a liquid of 50 per cent., and want to mix it with water in order to get one of 30 per cent., how much water must be added?

$$x \text{ (the quantity of water)} = \frac{40\ (50-30)}{30} = \frac{40 \times 20}{30} = \frac{800}{30} = 26\tfrac{2}{3}.$$

II. Given two liquids of the same kind, one of higher and the other of lower percentage; or one of any percentage, the other being water: required to know the quantity of the weaker liquid to be added to the stronger, to produce a desired weight and percentage.

Let a be the desired weight or quantity of the mixed liquids;
  b the percentage of the stronger liquid;
  c the percentage of the weaker liquid;
  d the percentage of the mixture;
  x the weight of the weaker liquid or of water to be added to the stronger.

(1) $x = \frac{a\ (b\ -\ d)}{b\ -\ c}$, *if a stronger liquid is to be mixed with a weaker one;* or in words: to find x (the quantity of the weaker liquid), multiply the difference between the percentage of the stronger liquid and the desired percentage of the mixture, by the desired weight or quantity of the mixed liquids, and divide the product by the difference between the percentage of the stronger and that of the weaker liquid.

Example.—Supposing we have a liquid of 60 per cent., and another of 20 per cent., and require forty parts of a liquid of 30 per cent., how much of the 20 per cent. liquid must be taken? x (the quantity of the weaker liquid) =

$$\frac{40\ (60 - 30)}{60 - 20} = \frac{40 \times 30}{40} = 30.$$

Then the quantity of the stronger liquid will be ten parts, to produce the required forty parts.

(2) $x = \frac{a\ (b - d)}{b}$, *if a stronger liquid is to be mixed with water;* or in words: to find x (the quantity of water to be added) proceed as described under II (1), but divide the product by the percentage of the stronger liquid only.

Example.—Supposing we have a liquid of 60 per cent., and want 40 parts of a liquid of 30 per cent., how many parts of water must be taken? x (the quantity of water) =

$$\frac{40\ (60 - 30)}{60} = \frac{40 \times 30}{60} = 20.$$

That is, twenty parts of water must be mixed with twenty parts of the given liquid.

Solubilities

The following table of the solubility of salts in water at normal temperature, was compiled by Dr. Attfield.

Acetates.—All soluble.

Arsenates. }
Arsenites. } —Insoluble, except those of the alkali metals.

Bromides.—Soluble, except mercurous and silver bromides. The bromides of antimony and bismuth, form oxy-salts when poured into water.

Carbonates.—Insoluble, except those of the alkali metals.

Chlorides.—Soluble, except mercurous and silver chlorides. Lead chloride is only sparingly soluble in cold water. The chlorides of bismuth and antimony, form oxy-salts when poured into water.

Citrates.—Manganese, silver, strontium, and mercurous citrates are insoluble. Those of the alkali metals, lithium, iron, calcium, and magnesium, are freely soluble; the remainder only sparingly soluble.

Cyanides.—Insoluble, except the cyanides of the alkali metals and alkaline earths, and mercuric cyanides.

Hydroxides.—Insoluble, except the hydroxides of the alkali metals, and the hydroxides of calcium, strontium, and barium. The latter are only sparingly soluble.

Iodides.—Antimony, bismuth, and gold insoluble. Lead, silver, mercuric and mercurous iodides are only sparingly soluble; the remainder are all soluble.

Oxalates.—Generally insoluble, except those of the alkali metals.

Oxides.—Insoluble, except the oxides of the alkali metals; barium, strontium, and calcium only slightly.

Nitrates.—Soluble.

Sulphates.—Soluble, except barium sulphate. The sulphates of calcium, strontium, antimony, lead, and mercury (-ous) are sparingly soluble.

Sulphites.—Soluble, except those of aluminium, antimony, and bismuth.

Tartrates.—Soluble, except the tartrates of antimony, barium, and bismuth.

Liquors

The pharmacopœial liquors or solutions may be roughly divided into two classes:

1. *Concentrated vegetable liquors.*—These are an innovation, and in the opinion of the author indicate a distinctly retrograde movement. Their utility is questionable, as they are neither tinctures nor fluid extracts. Infusions they certainly do not represent, for, as stated under Infusions, it is not possible to prepare them in a concentrated form. However, it is only from a pharmaceutical point of view that such preparations can be dealt with here; their method of preparation is fully explained in the chapter on Extraction, and need not be again gone into.

2. *Solutions of definite chemical substances.*—A large number of these are simple solutions. The solutions of the alkaloidal salts, are prepared by dissolving one part by weight of medicament in sufficient liquid to produce 100 parts by volume, and do not represent true percentage solutions in which both factors are in the same term. True percentage solutions of solids, contain a definite weight in 100 parts by weight of solution. Percentage solutions of liquids, may contain either, a definite volume of medicament in 100 volumes, or a definite weight in 100 parts by weight.

Liquor Ammonii Acetatis.—Formerly, this was prepared by diluting a strong solution of ammonium acetate; now a definite weight of ammonium carbonate is directed to be neutralised with acetic acid, and diluted to proper strength with water.

$$\underbrace{NH_4HCO_3.NH_4NH_2CO_2}_{156\cdot04} + H_2O + \underbrace{3HC_2H_3O_2}_{178\cdot74}$$
$$= 3NH_4C_2H_3O_2 + 2CO_2 + 2H_2O.$$

That is, 156·04 grams of B.P. salt will require 178·74 grams of real acetic acid; therefore 50 grams will require 57·2 grams of acid, or 173·3 grams of B.P. acetic acid 33 per cent. It is somewhat difficult to ascertain when neutrality is exactly reached, owing to the dissolved carbonic acid; therefore a little of the solution must be heated from time to time in a test-tube to expel the CO_2, and then tested with litmus paper.

Liquor Ammonii Citratis.—This solution is even more prone than the foregoing to develop fungoid growths, and will rarely keep good after the stopper has been opened a few times and carbonic anhydride lost. The author therefore recommends the solution to be made four times the strength, and diluted as required. Both the solutions of ammonium acetate and citrate rapidly attack common potassium-lead or "flint" glass; consequently, they should be stored in either hard-glazed earthenware vessels, or bottles made of the best potassium-calcium or potassium-sodium metal.

Liquor Arsenicalis.—Chemically this is simply a solution of arsenious acid in potassium carbonate. When these compounds are heated with a *small* quantity of water, combination is effected, and potassium arsenite (probably K_2HAsO_3) formed. This on diluting with water containing carbonic acid, rapidly breaks up, the solution containing the original compounds. Liquor Arsenicalis is very peculiar in its behaviour, as if kept for any lengthened period it deposits crystals of *arsenic* acid. Fresenius observed that when kept in *partly filled* bottles the solution always contains arsenic acid. The tincture of lavender is added to prevent fungoid growths.

Liquor Arsenici Hydrochloricus.—This only contains arsenious oxide dissolved in very dilute hydrochloric acid. Arsenious chloride, which may be obtained by distilling arsenious oxide with sodium chloride and strong sulphuric acid, is instantly decomposed by water into arsenious oxide and hydrochloric acid. A crystalline arsenic chlor-hydroxide is obtainable, but not in aqueous solution.

Liquor Arsenii et Hydrargyri Iodidi.—The solution contains the iodides dissolved in about molecular proportions. Care should be taken to see that the arsenic salt is in good condition, as if any free iodine is present, dark-coloured solutions result. When freshly made, the solution has a pale yellow colour, which on exposure to direct sunlight gradually disappears, hydriodic and arsenious acids being formed. The preparation was originally introduced by Dr. Donovan, of Dublin, who recommended it to be made by rubbing arsenious oxide, mercury, and iodine with a small quantity of alcohol, which was supposed to aid combination, the resulting dry mass being afterwards extracted with water and boiled.

Liquor Bismuthi et Ammonii Citratis.—Bismuth oxynitrate is dissolved in nitric acid and diluted with water until the oxysalt *just* begins to form again. The potassium citrate and carbonate are then added, dissolved in the least possible quantity of water. This produces a voluminous white precipitate of bismuthous citrate, which after boiling so as to render it more dense, is washed with distilled water until free from nitrates, and dissolved in solution of ammonia. If this solution is prepared in clean porcelain vessels and filtered through white filter-paper, it leaves nothing to be desired either in appearance or keeping properties. It is very important to see that the bismuth gives no reaction for either tellurium or selenium (see Tests).

Liquor Calcis Saccharatus.—When cane-sugar solution is added to certain oxides, soluble salts known as saccharosates are formed. Thus, saccharated solution of lime contains calcium saccharosate, $C_{12}H_{20}(CaOH)_2O_{11}$. These salts are not very stable, being readily decomposed by excess of water or carbonic anhydride, into sugar, and hydroxide or carbonate of the metal. If the solution is prepared from lime free from iron, obtained by burning marble, it can be preserved without appreciable change of colour for a long time; but if prepared from commercial lime it soon darkens.

Liquor Ethyl Nitritis.—This may be prepared by the following process :—Dissolve 635 grams of sodium nitrite in 1 litre of distilled water, and put the solution into a glass or earthenware retort connected with a very efficient condenser; then add 550 c.c. of 90 per cent. alcohol, and connect with a receiver kept *cold* in a mixture of pounded ice and salt. By means of a funnel passing through the tubulure and dipping beneath the surface of the liquid, gradually introduce, a *perfectly cold* mixture of 520 grams of sulphuric acid and 1 litre of water. Distillation usually commences when about half the acid has been added. When at last all the acid has been introduced, regulate distillation by the application or withdrawal of heat until no more ethyl nitrite comes over. Wash the distillate in a separator, first with 100 c.c. of *ice-cold* water to remove alcohol, and then with 100 c.c. of a 10 per cent. solution of sodium carbonate to remove acidity. Finally shake the ethyl nitrite with 30 grams of anhydrous potassium carbonate to remove last traces of water. The purified ethyl nitrite may then be filtered into a definite weight of a mixture of 5 parts of glycerol and 95 parts of absolute alcohol, finally diluting if necessary with more of the same mixture, so that the solution will contain exactly 3 per cent. by weight.

The reaction that takes place is expressed by the following equation :

$$2C_2H_5OH + 2NaNO_2 + H_2SO_4 = 2C_2H_5NO_2 + Na_2SO_4 + 2H_2O.$$

This solution does not contain any aldehyde, and, owing to the almost complete absence of water, is not so readily hydrolysed ; consequently it keeps very much better than the analogous spirit of nitrous ether.

Liquor Ferri Acetatis.—Ferric hydrate is first prepared by pouring solution of ferric sulphate into excess of dilute solution of ammonia. If the order of mixing is reversed, some oxysulphate is produced, owing to the ferric hydrate first precipitated, combining with undecomposed ferric salt. The ferric hydrate is collected on a calico filter, washed with water until free from sulphate, pressed, dissolved in glacial acetic acid, and diluted. Unless this solution is very carefully prepared it deposits an oxy-salt on keeping. To avoid this, the various operations should be conducted at as low a temperature and with as little exposure as possible, the liquid being cleared by decantation.

Liquor Ferri Perchloridi Fortis.—Iron wire is added to hydrochloric acid until at last a little iron remains undissolved. The filtered solution is then mixed with excess of hydrochloric acid, and poured in a thin stream into nitric acid, heat being applied if required, to promote reaction ; the solution is then evaporated until a precipitate begins to form. A little hydrochloric acid is added, and the whole diluted to the required volume with water.

In the first part of the operation ferrous chloride is produced :

$$6Fe + 12HCl = 6FeCl_2 + 6H_2.$$

On mixing with more hydrochloric acid and pouring into nitric acid the following reactions take place:—First, the ferrous chloride decomposes the nitric acid; nitric oxide, water, and oxygen being set free. The latter, in its nascent condition, at once attacks the hydrochloric acid, water being formed and chlorine liberated. The chlorine then unites with the ferrous chloride to produce ferric chloride. Thus:

1. $2HNO_3 = H_2O + 2NO + O_3.$
2. $O_3 + 6HCl = 3H_2O + 3Cl_2.$
3. $3Cl_2 + 6FeCl_2 = 3Fe_2Cl_6.$

Or in one equation:

$$6FeCl_2 + 6HCl + 2HNO_3 = 3Fe_2Cl_6 + 4H_2O + 2NO.$$

Liquor Ferri Pernitratis.—The action of nitric acid upon metals is sometimes of a very complicated nature, the products of decomposition depending not only upon the strength of the acid, but also upon the temperatures and presence of impurities. Generally speaking, nitric acid acts upon iron, forming ferric nitrate, nitric oxide, and water, thus:

$$Fe_2 + 8HNO_3 = 2Fe(NO_3)_3 + 4H_2O + 2NO.$$

If the action becomes too violent, water is added to prevent volatilisation of the acid. As the solution becomes cooler, and ferric salt accumulates, nitric oxide and even nitrogen itself are given off, while in certain cases ammonium nitrate is formed. The United States Pharmacopœia seeks to overcome this liability to variation, by preparing ferric hydrate, and then dissolving the well-squeezed precipitate in nitric acid, when ferric nitrate is formed without liberation of the objectionable nitric oxide fumes. However, as a ferric salt must first be prepared either by the aid of chlorine or nitric acid the objection is not a very valid one.

Liquor Ferri Persulphatis.—In the preparation of this liquor, ordinary crystallised ferrous sulphate is oxidised by means of nitric acid in the presence of an excess of free sulphuric acid, the whole of the metal being converted into normal ferric sulphate. The reaction is analogous to that described under Liq. Ferri Perchloridi. Thus:

$$\underbrace{6FeSO_4.7H_2O}_{1932\cdot7} + \underbrace{3H_2SO_4}_{292\cdot02} + 2HNO_3 = 3Fe_2(SO_4)_3 + 2NO + 46H_2O.$$

According to the above equation 1932·7 grams of crystallised ferrous sulphate require 292·02 grams of real hydrogen sulphate, equivalent to 295·9 grams of 98 per cent. acid, or 160·5 c.c. (sp. gr. being reckoned as 1·843). Therefore 400 grams (the quantity in the Pharmacopœia) would only require 33·2 c.c. of 98 per cent. acid.

When ferrous sulphate solution is poured into nitric acid a dark-coloured mixture at first results, due to the formation of an unstable compound of ferrous sulphate and nitric oxide : $2FeSO_4.NO$. This compound is readily decomposed by heat, nitric oxide being evolved.

Liquor Hydrargyri Nitratis Acidus.—When mercury is dissolved in nitric acid in the *cold*, mercurous nitrate, $Hg_2(NO_3)_2$, is formed, thus :

$$3Hg_2 + 8HNO_3 = 3Hg_2(NO_3)_2 + 4H_2O + 2NO.$$

On heating this solution, which contains excess of nitric acid, mercuric nitrate is formed, and more red fumes given off :

$$3Hg_2(NO_3)_2 + 8HNO_3 = 6Hg(NO_3)_2 + 4H_2O + 2NO.$$

A more convenient process would be to dissolve red mercuric oxide in nitric acid, thus avoiding the liberation of nitrous fumes :

$$HgO + 2HNO_3 = Hg(NO_3)_2 + H_2O.$$

Liquor Hydrogenii Peroxidi.—Hydrated barium peroxide is made into a thin cream with water, and gradually added to cold, very dilute sulphuric acid until the latter is almost neutralised. The slight excess of acid is then got rid of by adding an exact quantity of barium hydroxide solution. On a large scale silicofluoric or phosphoric acid is commonly employed, traces of the latter acting as preservatives of the hydrogen peroxide.

Liquor Magnesii Carbonatis.—Magnesium exhibits a great tendency to form oxy-salts when a soluble salt is precipitated by an alkaline carbonate. Thus :

$$4MgSO_4 + 4Na_2CO_3 + H_2O = 3MgCO_3.Mg(OH)_2 + 4Na_2SO_4 + CO_2.$$

If, however, the oxycarbonate is suspended in water, and the liquid saturated with carbonic acid gas under pressure, it dissolves, forming the bicarbonate, $MgH_2(CO_3)_2$, just as calcium carbonate is dissolved in spring waters, communicating what is known as temporary hardness. Crystals of normal carbonate, $MgCO_3$, may however, be obtained by allowing a solution of the bicarbonate to evaporate at a low temperature. In order to preserve the solution in a fit state for dispensing, it should be kept in ordinary aërated water syphons.

Liquor Plumbi Subacetatis Fortis.—This is prepared by boiling together, approximately molecular weights of lead acetate and lead oxide; the solution then contains an oxyacetate having the composition $Pb(C_2H_3O_2)_2.Pb(OH)_2$, with probably a little triplumbic acetate, $2Pb(C_2H_3O_2)_2.Pb(OH)_2$, a more basic and less soluble acetate being removed by filtration. The composition of this latter is probably $Pb(C_2H_3O_2)_2.5Pb(OH)_2$.

To facilitate combination, the lead oxide should be sifted into the water containing the lead acetate, and the mixture kept constantly stirred while boiling. If the clear solution is diluted with recently boiled distilled water, no turbidity occurs, but on exposing to the atmosphere for a short time, carbonic anhydride is absorbed, and a basic carbonate precipitated:

$$Pb(C_2H_3O_2)_2.Pb(OH)_2 + CO_2 + H_2O = PbCO_3.Pb(OH_2) + 2HC_2.H_3O_2.$$

Liquor Potassæ.—This solution may be prepared by dissolving potassium hydroxide in water, or if preferred by adding excess of slaked lime, which has been deprived of its soluble salts by washing, to a weak boiling solution of potassium carbonate:

$$Ca(OH)_2 + K_2CO_3 = 2KOH.CaCO_3.$$

Liquor Sodæ Chlorinatæ.—Prepared by double decomposition. Bleaching powder may be considered as consisting of $Ca(OCl)Cl$; on treating with water it is converted into calcium hypochlorite and chloride:

$$2Ca(OCl)Cl = CaCl_2 + Ca(OCl)_2.$$

Then on adding sodium carbonate:

$$Ca(OCl)_2 + Na_2CO_3 = CaCO_3 + 2NaOCl.$$

Solution of chlorinated soda therefore, actually contains sodium hypochlorite and calcium chloride. It has been proposed to add the alkaline carbonate solution boiling, effecting admixture in a closely stoppered vessel, so as to produce a dense precipitate; but it must be remembered that heat alone will effect the formation of a small proportion of chlorate, while if any free chlorine is present owing to decomposition of the hypochlorite, a considerable amount might be converted.

Liquor Sodii Ethylatis.—Sodium decomposes ethylic alcohol with formation of sodium ethylate or ethoxide, and liberation of hydrogen:

$$2C_2H_5OH + Na_2 = 2C_2H_5ONa + H_2.$$

This salt is soluble in absolute alcohol, but in presence of water it splits up into alcohol and sodium hydroxide.

Liquor Trinitrinæ.—This is a solution of the ethereal salt glyceryl trinitrate (nitro-glycerin or glonoin) in alcohol. When glycerol is allowed to fall drop by drop into a perfectly cold mixture of concentrated nitric and sulphuric acids, and the solution poured into cold water, nitro-glycerin is precipitated as a heavy oil; this when well washed and dried is dissolved in 90 per cent. alcohol. *On account of the highly explosive nature of nitro-glycerin, its preparation should only be attempted by experts.*

Liquor Zinci Chloridi.—If metallic zinc could be obtained perfectly pure on a commercial scale, solution of zinc chloride could be easily obtained by dissolving the metal in hydrochloric acid. As, however, zinc is frequently contaminated with both lead and iron, a somewhat complicated process becomes necessary. First the metal is dissolved in hydrochloric acid and the solution cooled. If either of the above-mentioned impurities be present, chlorine in excess is passed in; this oxidises the ferrous to ferric salt, when, on the addition of zinc carbonate, ferric and lead carbonates are precipitated and filtered out.

CHAPTER XXI

SYRUPS

Of the official syrups the following are either simple mixtures or solutions :—Syrupus, Syrupus Aromaticus, Aurantii, Aurantii Floris, Cascaræ, Chloral, Codeinæ, Glucosi, Limonis, and Scillæ.

In the three following the material is infused with the water, and the sugar dissolved in the strained infusion :—Syr. Hemidesmi, Rhæados, Rosæ. Syrupus Tolutani is prepared by exhausting tolu balsam with boiling water, allowing to cool, filtering out the cinnamic acid, and dissolving the sugar in the filtrate.

Syrupus Pruni Virginianæ.—The bark, in coarse powder, is exhausted by percolation with cold water, during which, a small quantity of hydrocyanic acid is produced. The sugar is then dissolved in the clear percolate without heat, to avoid driving off the volatile constituents.

Syrupus Rhei.—Rhubarb root and coriander fruit in No. 20 powder are exhausted by percolation with dilute alcohol. The percolate is then concentrated on a water-bath, and the sugar dissolved in the filtered liquid. A much better preparation would be obtained by exhausting the material with dilute alcohol in such proportion that no recourse need be had to evaporation. This could be easily done by the process of "pressure-maceration" (q. v.), when, if the sugar is dissolved in the cold, a syrup of fine aroma and full activity results.

Syrupus Sennæ.—Senna leaves are exhausted with dilute alcohol by the process of "pressure-maceration," the liquid sterilised by heating, and the sugar dissolved in the filtered product. The process, if properly carried out, leaves nothing to be desired either in keeping power or activity.

Syrupus Zingiberis.—This practically does not differ in composition from syrup of ginger of the 1885 Pharmacopœia, the only difference being that, as strong tincture of ginger has been deleted, it becomes necessary to prepare its equivalent for the manufacture of this syrup.

Syrupus Ferri Iodidi.—By the pharmacopœial process a strong syrup is first prepared, and solution of ferrous iodide, obtained by digesting iodine with iron wire and water, filtered into it.

Syrup prepared by the above process is of a good colour when first made, but it does not keep very well. The following process answers the official requirements, and will, moreover, keep for a very long time without change, the glucose appearing to have a distinct preservative action :

Iron wire	½ ounce.
Iodine	726 grains.
Refined sugar	16 ounces.
Glucose (as free from acid as possible)	A sufficiency.
Distilled water	

Dissolve the sugar in 6 ounces of boiling water; filter through felt and wash with distilled water until the filtrate weighs 22 ounces.

Digest the iodine and the iron wire in a small flask with 2½ fluid ounces of distilled water, and when combination is nearly complete heat until the liquid loses its brown colour. Filter while still hot through a chemically pure filter-paper into the hot syrup; wash the filter with a boiling mixture of the glucose and an equal weight of water. When all has run through, remove the funnel and allow the syrup to cool, finally making up to 20 ounces by volume with distilled water if necessary. *It is important not to wash the filter-paper with water.*

Syrup of iodide of iron that has become yellow through liberation of iodine, may be rendered colourless by placing in direct sunlight, when the iodine becomes converted into hydriodic acid. Such syrup will, however, be deficient in iron.

Syrupus Ferri Phosphatis.—Iron wire dissolves in phosphoric acid, forming ferrous phosphate and liberating hydrogen. If the proportions are strictly adhered to, the syrup remains colourless for any reasonable time; but if the amount of acid is increased at all, the syrup rapidly turns brown.

Syrupus Ferri et Quininæ, et Strychninæ Phosphatis.—This is prepared on the same lines as the foregoing, with exception that quinine and strychnia are added. It has been found necessary to introduce the former as sulphate, as syrup made with quinine phosphate so readily deposits.

CHAPTER XXII

COLLODIONS

Collodion, B.P., is a solution of cellulose nitrate in a mixture of alcohol and ether. When cellulose (cotton), $(C_6H_{10}O_5)_n$, is treated *for a short time only* with nitric acid, or better still with a mixture of sulphuric and nitric acids, it is converted chiefly into tetra- and penta-nitrate (pyroxylin), both of which, when well washed and dried, dissolve in a mixture of ether and alcohol. If the cotton were allowed to remain in the acids for several hours it would be chiefly converted into the hexa-nitrate, a highly explosive compound commonly known as "gun-cotton," insoluble in alcohol and ether. When these nitro-celluloses are decomposed by alkalies they yield nitrates of alkalies and cellulose ; they are therefore true ethereal salts.

Pyroxylin, if kept in tightly stoppered vessels, is liable to spontaneous decomposition ; but if kept in wooden boxes in a moderately dry place, it may be preserved almost indefinitely.

Collodion is principally used in surgical practice ; when allowed to dry on the skin it rapidly evaporates, and leaves a protective pellicle almost unaffected by water. Flexible collodion is similar to the foregoing, but, as its name implies, it leaves a more elastic film on evaporation. Blistering collodion is a form of liquid blister, simply made by dissolving pyroxylin in blistering liquid.

CHAPTER XXIII

FIXED OILS, FATS, AND WAXES

The fixed oils and fats are obtained from both the animal and vegetable kingdoms. They are all lighter than water, unctuous to the touch, and leave a permanent greasy stain when rubbed on paper. As they occur in nature they consist chiefly of mixtures of ethereal salts formed from the trihydric-alcohol glycerol, and the fatty acids of the acetic and acrylic series, with usually a varying proportion of free fatty acids and traces of colouring and flavouring principles.* These fatty acids are monobasic, therefore the neutral glyceryl esters contain three acid radicals, combined with the alkyl base C_3H_5: an example is seen in glyceryl stearate (stearin), $C_3H_5.(C_{17}H_{35}COO)_3$. Fats containing a relatively large proportion of olein are either liquid, as olive oil, or soft and pasty compounds, as lard; but if palmitin, stearin, or other higher members of this homologous series predominate, they are comparatively solid at ordinary temperatures, as mutton suet.

The waxes are chiefly composed of fatty acids, combined with monohydric alcohols homologous with methyl alcohol. Spermaceti for instance, contains, among other bodies, the palmitic ester of cetyl alcohol, $C_{61}H_{33}OH$, and beeswax, the palmitic ester of myricyl alcohol, $C_{31}H_{63}OH$.

The composition of oils and fats varies slightly according to the processes employed in their manufacture and purification. When they contain an undue proportion of free fatty acid they are said to be rancid, a condition partly due to oxidation, and partly to the fermentative action of bacteria.

Vegetable oils and fats are obtained from the parts of plants yielding them, either by expression or by boiling with water and

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* Oleic acid, $C_{17}H_{23}COOH$, contains two atoms of hydrogen less than stearic acid, and is an unsaturated acid belonging to the acrylic series. Its lead salt is soluble in ether, a property rarely met with in other lead salts.

skimming off the melted fat as it rises to the surface. Another method, largely practised for some seeds, consists in extracting the bruised material with either carbon bisulphide or petroleum ether, which, on distilling, leaves the fat behind. Animal tissues, rich in fat, are broken up and melted alone, and the membranous portion removed by straining, as suet and lard.

Oils and fats, as obtained by many of the *commercial* processes, are very impure, and are submitted to processes to rid them of albuminous, colouring, and other objectionable matter. The ordinary method consists in treating them with dilute sulphuric acid or solution of zinc chloride, afterwards washing with water, heating, and allowing to subside. Another process consists in blowing air through the impure oil. In addition to the above, oils are commonly bleached by exposing to light and air, or by filtering through freshly burnt animal charcoal. Oils that have been extracted by *simple expression in the cold*, do not require any other treatment than filtration to remove the suspended matter, and they are infinitely superior to those obtained in the ordinary way, by blowing steam into the material and pressing between hot plates. Commercial linseed oil, for instance, is a dark-coloured oil, very prone to deposit and of disagreeable taste, but when obtained by cold pressure it is almost colourless, or at most straw-coloured, and possesses a pleasant nutty flavour.

All oils and fats should be kept in the dark, preferably in well-sealed vessels of small capacity, and at as low a temperature as is consistent with their composition. The loosely capped oil bottles, so commonly seen on the shelves of many pharmacies, are about as *unsuitable* for the preservation of fixed oils as could well be devised, affording, as they do, every facility for rapid oxidation.

The variation in the composition of oils and fats of undoubted purity, renders their sophistication comparatively easy, and the detection of adulterants correspondingly difficult; in fact, the analysis of oils and fats requires great skill both in manipulation and interpretation of the results. By the application of the following tests a fair idea of their value may be obtained.

1. *Specific gravity.*—In the case of oils this is taken 15·5° C. (60° F.):

Almond oil	·915—·920	Cotton-seed oil ...	·922—·930
Castor oil	·950—·970	Linseed oil	·930—·940
Cod-liver oil	·920—·930	Olive oil	·914—·919
Croton oil	·940—·960		

In the case of fats and waxes, the specific gravity is generally taken at the boiling-point of water. Fill a tared specific gravity bottle similar to that shown in Fig. 24 with the melted substance, and place in a dish in which water is boiling freely. As the fat expands, it exudes by the perforated stopper. After about half an hour's boiling take out, wipe perfectly dry, and weigh.

Thus treated, the following figures were obtained by Muter:

Lard	·860—·861
Cacao butter	·857—·858
Beef suet	·857—·859

2. *Saponification equivalent* (Koettstorfer's method).—Counterbalance a strong flask of about 100 c.c. capacity, and introduce 2·5 grams of the oil or fat; then run in exactly 25 c.c. of semi-normal caustic potash in absolute alcohol, cork securely, and stand in a warm place until completely hydrolysed. When reaction is complete, add two or three drops of alcoholic solution of phenol-phthalein, and titrate the excess of alkali with semi-normal hydrochloric acid. At the same time a blank experiment on another 25 c.c. of $\frac{N}{2}$ KOH (alcoholic) is carried out, and the difference between the two titrations noted. The figure thus obtained multiplied by ·0278 (the co-efficient for 1 c.c. of $\frac{N}{2}$ KOH) gives the amount of potash in combination with the fatty acids, and is again calculated to percentage. Muter's figures for the following fats and oils are given below.

	Per cent. of KOH neutralised.		Per cent. of KOH neutralised.
Almond oil... ...	19·50	Olive oil	18·90—19·20
Castor oil	17·71—18·02	Lard	19·20—19·60
Cod-liver oil ...	18·51—18·52	Cacao butter ...	19·32—19·80
Cotton-seed oil ...	19·09—19·35	Beef suet	19·80—19·98
Linseed oil ...	19·00—19·28		

Instead of stating the results as above, the number of grams of oil which would be saponified by one litre of normal potash are given; in this case it is termed the "saponification equivalent." The most convenient way of obtaining this figure is to divide the weight of oil taken in *milligrams*, by the number of c.c. of normal solution of potash required for its saponification.

Hubl's iodine absorption equivalent.—Hubl's method depends upon the fact that all oils and fats are chiefly composed of esters of fatty acids of the acetic and acrylic acid series. While the relative proportion of these acids is constant within certain limits in any variety of oil or fat, yet, the members of the two groups of acids exhibit very different behaviour towards the halogens. Under certain conditions the members of the acetic acid series are unaffected, while those of the acrylic (oleic) series readily combine with constant amounts of the halogens, the amount being a measure of the unsaturated acids in the oil.

Hubl's solution is made by dissolving 30 grams of mercuric chloride in 500 c.c. of absolute alcohol, and adding to a solution of 25 grams of iodine in another 500 c.c. of alcohol. The solution should be prepared at least twelve hours before use.

Determination.—About ·25 gram of any oil, or about ·5 gram of a melted fat, is weighed into a stoppered glass flask capable of holding 250 c.c. To this is added, 10 c.c. of chloroform and 25 c.c. Hubl's solution. If the liquid is turbid, chloroform must be added until perfectly clear. The flask is then stoppered, shaken up, and put away in the dark for twelve hours. At the same time a blank experiment, using the same volume of Hubl's solution and chloroform, is put away with it. At the expiration of the time 20 c.c. of a 10 per cent. solution of potassium iodide and 100 c.c. of distilled water are added to each bottle. Each is then titrated with decinormal sodium thiosulphate solution, the bottle being thoroughly agitated after each addition until the colour of the uncombined iodine has nearly disappeared. A few drops of fresh, cold starch mucilage are next added, and titration continued until the blue colour just disappears. The number of c.c. of $\frac{N}{10}$ "Hypo" used for the bottle containing the oil, is deducted from that required by the blank experiment, and the difference multiplied by ·01259 (the co-efficient for decinormal iodine). This gives the amount of iodine absorbed by the oil, and from this the percentage is calculated. The following oils and fats thus treated indicate as follows :

	Percentage of iodine absorbed.		Percentage of iodine absorbed.
Almond oil ...	98·0— 99·0	Linseed oil ...	173·0—187·0
Castor oil	84·0— 84·5	Olive oil	79·0— 86·9
Cod-liver oil ...	158·7—166·6	Lard	52·0— 62·5
Cotton-seed oil ...	106·0—110·0	Beef suet	34·0— 45·0

4. *Specific heating power* (Maumené's test).—This method depends upon the rise of temperature which takes place on mixing oils with strong sulphuric acid. Allen and Archbutt found the

best strength of acid to be 97 per cent. Fifty c.c. of oil are placed in a narrow cylinder or beaker well wrapped round with flannel or lint; 10 c.c. of sulphuric acid are next added, and the mixture constantly stirred with a thermometer. When the temperature ceases to rise, a note is made. The oil and acid should both be at a temperature of 60° F. (15·5° C.). The experiment is then repeated under exactly the same conditions, using water instead of oil. The rise in temperature observed with the oil, divided by that shown with the water, furnishes the specific heating power. (*Note.*—The delivery of the acid should take exactly sixty seconds.) Linseed oil should be diluted with an equal volume of "paraffinum liquidum."

The following results have been obtained:

Water	1·00	Cotton-seed oil ...	1·63—1·70
Castor oil	·89— ·92	Linseed oil	3·20—3·49
Cod-liver oil	2·46—2·72	Olive oil	·89— ·94

Oleum Amygdale. Almond oil. (Non-drying.)—Expressed from the bitter or sweet almond. *Characters.*—Pale yellow, nearly inodorous, with a bland nutty taste. Sp. gr. ·915—·920. Soluble in ether and chloroform in all proportions. It does not congeal until cooled to nearly −4° F. (−20° C.). Almond oil is commonly adulterated with peach-kernel oil. When genuine oil is shaken with nitric acid and water equal parts, a whitish mixture is produced, which after standing separates into a solid white mass and a colourless liquid. Peach-kernel oil gives a brownish-red coloration. Sesame oil is best detected by Baudouin's test. Dissolve 0·1 gram of sucrose in 10 c.c. strong hydrochloric acid, and add 20 c.c. of the oil. Shake thoroughly and set aside. If sesame oil is present the acid liquid acquires a crimson colour. Almond oil consists principally of olein with a very small quantity of palmitin.

Oleum Crotonis. Croton oil. (Non-drying.)—Expressed from the seeds of *Croton tiglium.* *Characters.*—Brownish yellow to reddish brown. Entirely soluble in absolute alcohol, ether, or chloroform. The solution should be neutral to litmus-paper. Sp. gr. ·940 to ·960. Absence of other non-drying oils, is ensured by the oil remaining liquid for two days after shaking with a mixture of equal parts of fuming nitric acid and water. Croton oil consists of a mixture of stearin, palmitin, myristin, and laurin, and of several volatile acids of the same series, as well as tiglinic acid. The purgative and vesicating properties do not, however, exist in the acids, but in a fat which is not readily saponified.

Oleum Lini. Linseed oil. (Drying.)—Expressed from linseed (flax seed) at the ordinary temperatures. The yield is about 35 per cent. The oil should be perfectly limpid, and of a yellowish and not reddish colour. Soluble in all proportions of turpentine, and in 10 parts of 90 per cent. alcohol. The specific gravity is given as from ·930 to ·940; the author has found linseed oil pressed and filtered at a temperature of 60° F. to average ·934. Hot-pressed oil is of lower gravity, ranging from ·927 to ·930. Linseed oil gradually thickens on exposure to the air, and if spread in a thin layer dries to a transparent varnish containing linoxin, hence its use in oil painting. The dried oil is not again soluble in turpentine. Commercial linseed oil is a very different article, being obtained by hot-pressing, after subjecting the ground material to the action of steam. A greater yield of oil is thus obtained, but it is of very dark colour, and requires much purification to remove mucilaginous matter, &c. "Boiled oil" is obtained by heating linseed oil with lead acetate and lead oxide. Such oil dries very rapidly. The same result is brought about by blowing warm air through oil in which bags of manganese dioxide or manganese borate are suspended. In this case the manganese salt is not affected, it only appearing to act as an oxygen carrier. Linseed oil is a mixture of about 80 per cent. of olein with small quantities of palmitin and myristin. On oxidation, linseed oil is nearly all converted into linoxin ($C_{32}H_{54}O_{11}$).

Oleum Morrhuæ. Cod-liver oil. (Semi-drying.)—Obtained from the fresh liver of the cod, *Gadus morrhua.* Although some few pharmacists still prepare this oil for themselves in this country, the largest quantity is imported. The chief fisheries are those of Lofoden and Newfoundland. The fresh healthy livers are well washed with cold water to remove blood, and the adhering gall-bladder removed. Next they are placed in large steam pans, broken up with wooden paddles, and heated to about 85° F., when the oil rises to the surface and is skimmed off. The first skimmings constitute the finest quality, and are preserved separately; afterwards the heat is raised and a second quality skimmed off. The total yield is about 48 per cent. The skimmed oil is strained through flannel, placed in large tubs, and perfectly dry salt sifted on to the surface. This as it falls carries with it most of the adhering water. Next the oil is submitted to a temperature below the freezing-point of water, when it freezes solid; it is then placed in bags and strongly expressed; the resulting oil is allowed to deposit, and then packed in tin-lined casks for the market. The matter remaining in the press, consists principally of stearin, and is sold for making cheap soaps. Cod-liver oil readily oxidises on exposure to air, consequently the greatest care is necessary in its preservation.

Sp. gr. ·920 to ·930. It is readily soluble in ether and chloroform, and slightly in 90 per cent. alcohol. A drop of sulphuric acid added to a few drops of cod-liver oil on a white slab develops a violet colour. Nitric acid carefully added to some of the oil in a test-tube, gives a white precipitate of albumen where the liquids meet. The Pharmacopœia requires that no stearin should separate after exposure to a temperature of 32° F. (0° C.) for two hours.

Cod-liver oil consists principally of olein, with varying proportions of stearin, palmitin, and myristin. Iodine has also been found to be present in very small quantity, probably less than two parts per million.

Oleum Olivæ. Olive oil. (Non-drying.)—Expressed from the ripe fruit of *Olea europæa.* The ripe fruit is reduced to a pulp in suitable mills (care being taken not to break the seeds), placed in bags and submitted to a gradually increasing pressure. The first running constitutes the "virgin" oil. Afterwards the press cake is removed, treated with water, and again pressed, when oil of inferior quality is obtained. A still more inferior oil is obtained by treating the press cake with hot water and again pressing, or, by pressing olives which have been allowed to ferment. The finest quality oil has a pale yellow or greenish-yellow tint, and a pleasant taste. Sp. gr. ·914 to ·919. Below 50° F. (10° C.) it is liable to become pasty, and at the freezing-point of water it becomes dry and granular. If, however, at this point it is submitted to strong pressure in suitable bags, it yields upwards of 70 per cent. of pure olein; the residue consists principally of palmitin.

Adulteration.—Sesame oil is detected by the high figure for iodine absorption and by Baudouin's test. Cotton-seed oil also by the high iodine absorption and by the following test:—If 10 c.c. of olive oil is shaken with 2 c.c. of the following reagent, and then heated on a water-bath for ten minutes, a blackening occurs if cotton-seed oil is present—

Reagent.—Silver nitrate	... ...	1·00 gram.
Absol. alcohol	... ...	100·00 c.c.
Ether	... ...	20·00 c.c.
Nitric acid	... ...	1 drop.

Oleum Ricini. Castor oil. (Semi-drying.)—Expressed from the seeds of *Ricinus communis.* Until recently all the oil used in this country was imported, Marseilles being the seat of the pressing industry. The process consisted in crushing the seeds and pressing after a moderate application of heat. The oil was then freed from mucilaginous matter by treatment with water. The oil thus obtained was always of inferior quality, and required to be bleached by exposure to the sun, and afterwards filtered. Recently a very fine quality of oil has come into the market, prepared in this country by Mitchell's process. The seeds are, *without previous crushing,* fed into a specially constructed press, and submitted to very great pressure in the cold. About 35 per cent. of oil is thus obtained, which only requires filtering through flannel before bottling. This oil is practically tasteless, and is of an entirely different character from that prepared by the older methods. Cold-drawn castor oil is somewhat viscid, and has a slight yellow tinge. The sp. gr. ranges from ·960 to ·965. Colourless oils are obtained by bleaching. Castor oil is soluble in less than twice its volume of 90 per cent. alcohol. It dries *very slowly* to a transparent varnish when exposed to the air in a thin layer. If 3 c.c. of the oil be shaken with an equal volume of carbon disulphide and 1 c.c. of sulphuric acid no darkening should occur (absence of cotton-seed oil). Equal volumes of castor oil and petroleum ether B.P. yield a turbid mixture if kept at 60° F., but if other fixed oils are present a clear solution is formed.

Composition.—The oil is composed of several fats, the acids of which have been the subject of repeated investigations. The principal are, a solid fat closely related to palmitic acid, and a liquid one named ricinoleic acid. The press cake is stated by some authorities to be violently purgative, but the author has seen cattle eat it without purgation taking place.

Oleum Theobromatis. Oil of theobroma.—Expressed while hot from the seeds of *Theobroma cacao.* Cacao butter is obtained as a by-product in the manufacture of chocolate. The kernels of the seeds are crushed and pressed between hot plates; the melted fat is collected, filtered through flannel, and run into moulds. It occurs as a creamy white solid, with a pleasant odour, and bland agreeable taste. It melts between 88° and 93° F. (31·1° and 33·9° C.). If

1 gram be dissolved in 3 c.c. of ether in a test-tube, and the tube be cooled to 32° F. (0° C.), the liquid should neither become turbid nor deposit a granular mass in *less* than three minutes; if the mixture, after congealing, be again heated to 60° F., it should gradually afford a clear solution (absence of other fixed fats). The oil consists chiefly of stearin with some olein, and traces of glycerides of other acids, among which are lauric and arachadic acids. Cacao butter is almost entirely used for suppositories, as while having a low melting-point, it is sufficiently firm to be handled.

Adeps. Lard.—The perfectly fresh fat of the abdomen of the hog, known as "flare," is freed as far as possible from the adhering membrane, after which it is cut into small pieces, and either crushed in a mill or beaten in a stone mortar. The mass is then kneaded in running water until free from blood, &c.; during this process much of the peculiar smell is got rid of. After being allowed to drain, the fat is melted in a water or steam pan, and stirred occasionally to promote liberation of the adhering water. When perfectly fluid it is strained through flannel and stirred until cold. The yield is about 92 per cent. of the "flare" taken. *Benzoated lard* is simply prepared by digesting powdered benzoin with melted lard, when some benzoic acid and traces of essential oil go into solution. As noted under Ointments, this treatment yields a dark-coloured product which is distinctly irritating to tender skins, and which turns a brown colour on mixing with alkaloids. Lard * is a mixture of olein with about 40 per cent. of solid stearin and palmitin. At a temperature of 32° F. (0° C.) the last two may be obtained by expressing strongly in bags; the olein thus separated is sometimes sold as *lard oil.* Knowing this, the importance of stirring melted lard until perfectly cold, becomes apparent; but in spite of this precaution, if lard is kept for any length of time in a cold place, the solid fat assumes a crystalline character, the whole presenting a crumbly granular appearance.

Note.—Lard prepared from hog fat, other than that obtained from the omentum, is much softer in character, and on keeping the olein separates out.

Adeps Lanæ. Wool fat.—Although only recently made official, wool fat obtained from sheep's wool has been known and recommended in certain skin affections from very early times, but owing to difficulties in its purification little use has been made

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* See Glycerol.

of it. The present process employed in the industrial manufacture of wool fat is based on Schulze's method. According to Jaffé and Darmstädter, the manufacturers of lanolin, the crude wool fat is obtained in the usual way by treating the natural greasy wool with soap liquors, and then decomposing with mineral acids. This crude wool fat contains about 70 per cent. of cholesterin and isocholesterin, and about 30 per cent. of fatty acids. The first purifying operation consists in saponifying the fatty acids with caustic soda. An emulsion of the cholesterin compounds in the soap is thus obtained, which on dilution gives the so-called "wool milk." When this wool milk is submitted to centrifugal action it separates, like cow's milk similarly treated, into so-called "cream" and "skim-milk." The cream, on precipitation by a lime salt, yields crude lanolin. This is heated, water separating, and the lanolin further purified from waxy impurities (higher melting cholesterin) by a patented process.

The chemical composition of wool fat is a complex one. In a crude condition its constituents are cholesterin and isocholesterin, monatomic alcohols of unknown constitution. These exist partly free and partly as stearate and palmitate. The odour of crude wool fat is due to volatile acids, chiefly capric and caproic, which are always found in sweaty secretions. There has also been found ceryl cerotate and its homologues, and probably cholesteryl cerotate, as well as certain proportions of the glycerides of the lower fatty series. It is important to note that the Pharmacopœia requires wool fat to be almost free from these fatty acids in a free state. Pure wool fat cannot properly be called a *fat*, as it is not a glyceride. It does not form soaps when boiled with aqueous solutions of the alkalies, but it has the peculiar property of forming emulsions with alkaline solutions. (Wool fat may be hydrolysed with boiling alcoholic potash.)

When water is incorporated with pure wool fat to the extent of 30 per cent. it constitutes Adeps Lanæ Hydrosus. This is not, however, the full extent to which water is taken up, as if added in small quantities at a time there is no difficulty in incorporating at least 100 per cent.

Cetaceum. Spermaceti.—Spermaceti is the solid portion of the oil found in a large cavity in the head of the sperm whale, *Physeter macrocephalus*, a cetacean belonging to the order Mammalia. While the whale is alive, the oil is in a fluid condition, but soon after the animal is killed the oil becomes pasty. In this condition it is placed in bags to drain, and finally submitted to pressure, when most of the *sperm oil* comes away. The residue, which consists of impure spermaceti, is then boiled with very dilute alkali to purify it. Commercial spermaceti occurs as a white scaly-crystalline brittle mass. When used in ointments it is generally combined with a little beeswax to modify this tendency to crystallise. Spermaceti consists principally of cetyl palmitate, with small quantities of the salts of lauric, stearic, and myristic acids.

Cera Flava. Yellow beeswax.—Yellow wax is a secretion of the working bee. It is generally looked upon as a conversion product of sugar, and not derived directly from the flowers on which the bee feeds. The wax is formed in very thin scales between the rings of the belly, and is used by the bee in the construction of the hexagonal cells of the comb. This comb filled with honey is taken from the hives at certain intervals, broken up, and placed in muslin bags, when the honey drains away. The wax is then purified by melting in water, allowing to deposit, and running into moulds. *White wax* is ordinary beeswax that has been melted in dilute alum water, spun out into ribbons, and bleached in the sun; when it has acquired the requisite degree of whiteness it is remelted, washed with hot water, and moulded into cakes. By this bleaching process, wax loses its fragrance and acquires a rancid odour; otherwise it is unaltered in composition. Beeswax consists of cerotic acid, a homologue of acetic acid, and about 80 per cent. of myricin, the palmitic ester of myricyl alcohol, a homologue of ethyl alcohol.

Beeswax is very frequently adulterated. The most frequent adulterants are flour, stearic acid, stearin, and hard paraffin. Flour or any other added powders remain insoluble on boiling with turpentine. Stearic acid and resin are soluble in boiling alcohol, beeswax only slightly. Stearin, stearic acid, &c., are saponified when warmed with aqueous solution of potash. Beeswax requires to be boiled with *alcoholic potash* before hydrolysis is effected. Paraffin may be detected by heating with strong sulphuric acid in large excess. This carbonises beeswax, but does not affect paraffin. After cooling, the black mass is washed with water to remove acid, dried, and extracted with ether, which on evaporation leaves the paraffin in a weighable condition.

Sevum Præparatum. Prepared suet.—Mutton suet consists of the purified fat from the abdomen of the sheep, *Ovis aries.* It is prepared in a precisely similar manner to that practised for lard, the great object being to free the fat as completely as possible from blood and adhering membrane. Suet is generally credited with poor keeping properties, but if carefully prepared, and preserved in clean jars out of contact with the atmosphere, the author finds that it keeps almost as well as lard. The chief pharmaceutical use is for mercurial ointment and for cantharides plaster, but it occasionally finds employment in pomatums for the hair, besides being used in extracting the perfumes of flowers. Its use in candle-making need not be entered into here. The melting-point of mutton suet is about 114° F. when freshly prepared, but old samples may not melt much below 120° F. In the 1885 Pharmacopœia the melting-point was erroneously given as 103° F., which is that of beef suet.

If mutton suet is cooled to the freezing-point of water, wrapped in absorbent cloth, and submitted to hydraulic pressure, about 30 per cent. of olein is pressed out, the residue consisting principally of palmitin and stearin.

Hydrocarbons

Paraffinum. Paraffin.—The Pharmacopœia enumerates three varieties of paraffin, viz. hard, soft, and liquid. *Hard paraffin* is obtained from the higher boiling fractions of the tar obtained by the destructive distillation of American petroleum and Scotch shale or cannel coal. Russian petroleum yields practically no paraffin wax, although large quantities of a product termed "ozokerite" come from Galicia. Paraffin wax should be colourless and odourless, and have a crystalline structure; sp. gr. ·820 to ·940; melting-point from 130° to 135° F. (54·4° to 57·2° C.).

Soft paraffin consists of a mixture of solid and liquid hydrocarbons, the former of which are entirely without crystalline structure. Like paraffin wax, it consists almost entirely of saturated hydrocarbons, the small quantity of oxygen-containing compounds being almost negligible. A mixture of paraffin wax with viscous hydrocarbons, is sometimes represented as soft paraffin, but it always presents a granular structure with a widely diverging melting-point. Soft paraffin should melt between 96° and 102° F. (35·5° and 38·9° C.). The specific gravity at the melting-point varies from ·840 to ·870.

Liquid paraffin is obtained from the higher boiling fractions of "solar oil." The oil is first freed from water by blowing warm air through until perfectly bright and clear, when it is treated with sulphuric acid. After the evolution of sulphur dioxide (due to the decomposition of the impurities in the oil) has ceased, the acid is allowed to settle, drawn off, and the oil washed with solution of soda, and finally with warm water. Liquid paraffin should be colourless and free from fluorescence. Boiling-point not less than 680° F. (360° C.). The specific gravity is required to be not less than ·885, but as a matter of fact the oil rarely has a higher gravity than ·875 to ·880.

Bird has shown that the samples of soft and liquid paraffin met with in commerce are frequently contaminated with sulphur, and are therefore unsuited for internal use, especially when combined with hypophosphites.

Test.—Take of the suspected sample 4 c.c., absolute alcohol 2 c.c.; shake in a test-tube, and add 1 c.c. pure hydrochloric acid and a fragment of pure zinc. A cap of white filter-paper wetted with solution of lead oxyacetate is fastened over the mouth of the tube, spurting being prevented by a plug of cotton wool. The presence of sulphur compounds is indicated by the paper turning brown or black. The hydrogen evolved in contact with pure oils has a pleasant ethereal odour, which the presence of even a trace of sulphur modifies in a marked degree.

The origin of petroleum is unknown, but it is supposed to be produced by the destructive distillation in the lower layers of the earth's crust, of the fatty remains of sea animals, &c. Crude petroleum is specifically lighter than water, and varies greatly in consistence and colour, generally appearing as a thick yellow or brown liquid, shading green when viewed by reflected light. It consists almost entirely of hydrocarbons; that from Pennsylvania being chiefly composed of paraffins, that from the Baku district of hydrocarbons of the naphthene series.

The paraffins, as the name implies, are characterised by their stability. At the ordinary temperatures they are not acted upon by nitric acid, sulphuric acid, caustic alkalies, or such powerful oxidising agents as chromic acid or potassium permanganate, and even at high temperatures only a very slow action takes place. It is from this inertness that the paraffins have come into such universal use in pharmacy, as although it is known that the softer paraffins absorb oxygen in a slight degree, yet the amount is too small to have any appreciable effect on the keeping properties of ointments prepared with them.

CHAPTER XXIV

OINTMENTS

The ointments of the Pharmacopœia may be practically divided into two classes: (A) those containing medicaments intended for absorption, such as aconitine; (B) those used as dressings for wounds or sores, of which boric acid ointment may be taken as a type.

Class A requires a basis having a melting-point about 100° F. (37·8° C.), capable of ready absorption when rubbed into the skin, and which, while free from any tendency to set up irritation, possesses well-marked keeping properties. Pure lard fulfils all these requirements, except perhaps, the last. Benzoated lard, although keeping well, is distinctly irritating to certain tender skins, and is especially unsuitable for eye ointments. As this drawback is well known, it is somewhat astonishing that in the present Pharmacopœia some preservative other than benzoin has not been adopted. Prepared lard, containing either three minims of oil of cloves or two minims of oil of gaultheria to each ounce, is perfectly bland, keeps almost indefinitely, and being prepared in the cold is whiter and altogether more desirable as an ointment basis, than benzoated lard. Such a preparation might be termed "Adeps Odoratus." Class B also requires a non-irritant basis, softening but not melting at the temperature of the human body, so as to avoid the inconvenience caused by the ointment soaking into the bandages. The basis must, however, not be so hard that it cannot be spread readily on lint, &c. A mixture of solid and semi-solid hydrocarbons, such as Unguentum Paraffini, fulfils all these conditions; Unguentum Cetacei scarcely fulfils any of these requirements, and it is difficult to understand why, when the pruning-knife has been used so freely, it should have been retained in the Pharmacopœia.

In the following paragraphs it is only intended to mention those ointments which possess special interest from a manufacturing point of view.

Class A

Alkaloidal ointments.—Ung. Aconitinæ, Atropinæ, Cocainæ, Veratrinæ. An oleate of the alkaloid is first prepared, and this mixed with the lard. If benzoated lard is used the ointments turn brown on keeping.

Ung. Capsici.—Prepared by digesting capsicum fruits in olive oil; this dissolves out the oleo-resin. This ointment must not be stirred while cooling, as it is intended to present a crystalline appearance.

Ung. Gallæ cum Opio.—To obtain this ointment perfectly smooth, the opium should be moistened with a little water before mixing with the fat.

Ung. Hydrargyri.—The manufacture of this ointment is a very tedious process. In order to obtain it of good colour, as well as to facilitate the extinction of the mercury, the latter should be triturated with a little mercury oleate, prepared according to the 1885 Pharmacopœia. Another, but more objectionable plan is to use very old and rancid mercury ointment for this purpose. When required for inunction, this ointment may be prepared by rubbing sixteen ounces of mercury with mercuric oleate until killed, and then incorporating seventeen ounces of anhydrous wool fat.

Ung. Iodi.—Avoid use of steel knives during preparation.

Ung. Hydrargyri Oleati.—Prepared by mixing mercury oleate with lard. Mercury oleate is directed to be prepared by decomposing an emulsion of oleic acid in solution of hard soap, with mercuric chloride. An insoluble precipitate of mercuric oleate, palmitate, and stearate is formed, which is not even a homogeneous mixture. From a pharmaceutical point of view this preparation is not so good as that prepared by the 1885 formula. As the mercury compounds named rapidly darken on exposure to air, they should be mixed with the lard with as little delay as possible.

Ung. Zinci Oleati.—Prepared by mixing "zinc oleate" with an equal weight of soft paraffin. In the opinion of the author the product is not to be compared with the corresponding ointment of the 1885 Pharmacopœia, in which zinc oxide was dissolved in excess of oleic acid. If this ointment is intended for absorption, the zinc oleate should be dissolved in lard; if intended as an ordinary dressing, in which absorption is not desired, it is difficult to understand what advantage the rancid oleate possesses over the oxide. (Compare Ung. Hydrargyri Oleati, in which lard *is* used.)

Class B

Ung. Acidi Carbolici.—The glycerin is added to prevent the phenol crystallising out, as sometimes happens if simply dissolved in the melted hydrocarbons.

Ung. Acidi Salicylici.—Avoid use of steel knives in preparing this ointment, or the ointment will acquire a pinkish tint from formation of ferric salicylate.

Ung. Hydrargyri Nitratis.—The present process is a great improvement over that in the last Pharmacopœia, the ointment keeping better, besides being of a more typical colour. When the mercury is dissolved in the *cold* nitric acid, a mixture is formed containing mercuric and mercurous nitrate in much free acid. When this mixture is poured into the hot fats, violent reaction takes place, nitric oxide being given off; a great portion of the fat is oxidised to solid elaïdic acid, the mercury being at the same time raised to the *-ic* state. If any mercurous nitrate is left, the ointment instead of retaining its pale lemon colour, soon darkens from separation of metallic mercury. Elaïdic acid is also formed when nitrous fumes are led into olive oil, the new substance being readily obtained as yellow plates.

The great secret in making this ointment, lies in the strict observation of the temperatures. The lard and oil should be heated together in a porcelain dish on a sand-bath, to a temperature of 300° F. (148·8° C.). While this is proceeding a stout earthenware jar capable of holding ten times the quantity, is made as hot as possible in a *water-bath*. When the hot fats are poured into the jar, the temperature begins to fall, and when it has dropped to 290° F. (143·3° C.), the acid solution is added a little at a time, the mixture being briskly stirred with a glass rod or wooden spatula. If the latter has been soaked in melted paraffin for a few hours, the acid will scarcely affect the wood, and it may be used over and over again. It is important to stir the ointment while cooling, both to get rid of the last traces of fumes and to produce a uniform product. If larger quantities than those given in the Pharmacopœia are operated upon, the temperature of the melted fats may be safely lowered by 10° F.

Ung. Paraffinum is a mixture of hard and soft paraffin. In very warm weather or in the tropics, it is permissible to increase the proportion of hard paraffin, so as to obtain an ointment of convenient melting-point; while in the winter or in cold climates the proportion may be correspondingly reduced. It will be noticed that wherever possible a white ointment is to be made, and for this reason *white* soft paraffin is most generally used, the yellow variety only being employed for coloured ointments, such as that of mercuric oxide.

No especial precautions need be observed in melting the two paraffins, but to obtain a perfectly smooth and homogeneous mixture when cold, needs great attention. If possible the melting should be accomplished in a shallow metal dish, and as soon as the mixture shows the slightest tendency to congeal, it should be stirred with a broad-bladed spatula, with a regular side-to-side swaying motion, the solidifying portions being constantly scraped down and intimately mixed with the more fluid in the centre of the dish.

Straining of ointments.—Except when the greatest care is exercised, lumpy ointments result, either from too rapid cooling or faulty admixture. In such cases the ointment may be rendered perfectly smooth by straining in the cold. A piece of stout unwashed muslin is tied over a wide jar, and the ointment pressed through a little at a time by means of a flexible knife. The process is very tedious, and should only be had recourse to as a last resort.

CHAPTER XXV

SOAPS

As stated on page 176, the fixed oils and fats chiefly consist of mixtures of ethereal salts, formed by the union of fatty acids with an alcohol (glycerol). Stearic, palmitic, and oleic acids constitute the bulk of the acids, and it is chiefly with their salts that this chapter deals. When a fixed oil, as olive oil, or a fat, as tallow, is heated with a caustic alkali, double decomposition takes place, sodium salts of the acids being formed and glycerol set free; this action is termed *saponification*, and the salts thus formed soaps. The term saponification was also formerly applied to the analogous decomposition of other ethereal salts by alkalies, in spite of the fact that the products were not soaps; but the word "*hydrolysis*" has now to a great extent taken its place. Hydrolysis may be roughly defined as the decomposition of one compound into two or more, with fixation of the elements of water or some hydroxide. Thus ethyl nitrite is gradually hydrolysed by water or alkalies, alcohol and nitrous acid being formed:

$$C_2H_5NO_2 + H_2O = C_2H_5OH + HNO_2.$$

In the manufacture of soaps on an industrial scale, the alkalies used are called *leys*. Strong ley contains about 20 per cent. of alkali, medium ley 10 per cent., and weak ley from 1 to 4 per cent. The decomposition of a fat by an alkali does not take place suddenly, but passes through several stages, the first being the formation of an emulsion of ley and fat; next fatty acids and fat acid-salts are formed, which retain the rest of the fat in suspension; lastly the free fat is saponified, and the acid-salts converted into neutral salts, or, in other words, soaps. When caustic soda is used, hard soaps are produced, while the soft soaps result from the use of caustic potash.

Hard soaps are technically divided into three classes:

1. Nucleus soaps.
2. Smooth soaps.
3. Fulling soaps.

Nucleus soap is the purest form of soap, obtained by adding common salt to the boiled mass. It is practically free from water, glycerin, or excess of alkali. When nucleus soap is boiled for some time with water or weak ley it loses its nucleated or crystalline appearance, takes up water or ley, and constitutes smooth soap. Fulling soap is the poorest kind of soap, as an insufficient quantity of salt is added to entirely precipitate the soap, the consequence being that the whole contents of the pan—soap, glycerin, alkali, and impurities—are all kept together, boiling being continued until the mass will solidify, when it is removed, cooled, and cut into bars. Cocoa-nut oil soap is especially useful to the maker of this class of soap, as it is very hard, and possesses the property of taking up a large amount of water.

Besides the insoluble soaps there are three official varieties of soaps proper:—*Animal* or *curd soap*. This consists essentially of sodium stearate and palmitate, and is commonly prepared by first saponifying Russian tallow with caustic potash, and then by means of common salt converting into sodium compounds. After separation from the under-ley, the soap is run into large wooden boxes, so constructed that they can be readily taken to pieces, and the solidified soap cut into bars. After cutting, the soap is placed in a warm room, where it loses about 10 per cent. of moisture. Ordinary curd soap contains about equal weights of potash and soda soaps. One part of tallow yields a little more than one and a half parts of soap.

Hard or *Castile soap* is chiefly prepared in the south of Europe. Two kinds of ley are used; the first is ordinary potash ley, the second contains in addition, common salt to effect the separation as hard soap. Mottled Castile soap is produced by adding ferrous sulphate to the white soap, and stirring until cool. When freshly cut this soap has a blue and white appearance, but by exposure to the atmosphere the iron undergoes oxidation, and the soap presents a mottled red appearance. Castile soap consists chiefly of oleate of soda.

Soft soap.—The Pharmacopœia requires soft soap to be made by saponifying olive oil with caustic potash, and, as in the case of the other official soaps, fixes the limit of free alkali. Soft soap consists of impure oleate of potash in excess of potash ley, mixed with the glycerin separated by the process of saponification.

Soft soap can only be prepared with potash ley, although in practice a little soda is added to give firmness to the soap. The 1885 Pharmacopœia defined the soap as possessing a greenish tinge, but this has been rectified in the last edition. Pure olive oil soft-soap is distinctly yellowish ; the green tinge is imparted either by using hemp-seed oil or colouring with indigo or oleate of copper.

Toilet soaps are generally prepared by remelting a good class raw soap and perfuming, or by cutting bar soap into shreds, perfuming, and milling between granite rollers. In either case the perfumed soap is formed into bars, divided again into blocks of equal weight, and stamped in a soap press. This last machine gives the tablets their usual trade forms, any lettering being done at the same time.

Transparent soap is made by dissolving good hard soap in an equal weight of alcohol, cooling, and filtering the solution into moulds, where it has to remain for several weeks to harden.

Insoluble soaps.—Besides the foregoing several soaps are official under various names. Ammonia soap is formed in the preparation of Linimentum Ammoniæ ; Lime soap in Linimentum Calcis ; Lead soap in Emplastrum Plumbi ; Mercury soap in Hydrargyri Oleas, and Zinc soap in Zinci Oleas. Each of these are dealt with under their respective headings as liniments, plasters, and ointments.

Medicated soaps

The use of medicated soaps is now largely advocated by many members of the medical profession, and while it would hardly repay the pharmacist to manufacture all the soaps that are so frequently prescribed, yet instances are constantly occurring where only one or two tablets of a special kind are ordered, and it is to the credit of the dispenser if these can be prepared at short notice.

Procure white Castile and pure curd soap in bars, and with a small carpenter's plane cut up in thin shavings ; dry at a temperature about 100° F., powder finely and mix in equal proportions, and label "soap powder."

Example.—Ichthyol 5 per cent. ; sulphur 10 per cent. ; tar 2½ per cent.

℞	Soap powder	80 parts.
	Precipitated sulphur	10 ,,
	Ichthyol-ammonia	5 ,,
	Stockholm tar	2½ ,,
	60 per cent. alcohol	10 ,,

Mix the soap and sulphur intimately and pass through a 60 sieve into a convenient-sized mortar; add the ichthyol mixed with half the alcohol, and the tar previously shaken up with the remainder; mix thoroughly and beat up into a uniform mass. Weigh off into pieces of four ounces each, and stamp, either with a proper soap press or a home-made machine similar to that described below.

Fig. 146, *a*, is an oval collar, preferably made in stout brass, 4½ inches deep. In outline the collar should be that of an ordinary oval soap cake.

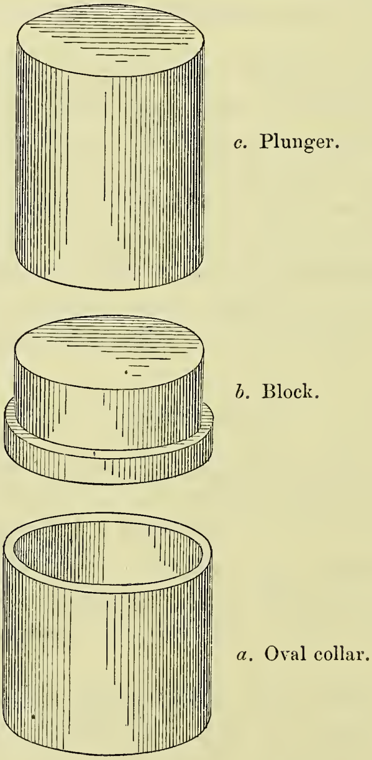

Fig. 146.

Fig. 146, *b*, is a mahogany or metal block, one inch thick, made to accurately fit into the collar, and provided with a flange at the bottom. The upper surface of the block should be hollowed out, so as to impart the usual convex surface to the soap tablet.

Fig. 146, *c*, is similar to Fig. 146, *b*, but may be about six inches long and without a flange, the under surface being scooped out so as to correspond.

To use, place the soap mass, which should weigh about four ounces, in the cylinder previously fitted over the bottom block, and having inserted the plunger, the whole is placed in an ordinary tincture press and squeezed until a firm tablet results. The inside of the mould should be oiled with a little colourless hydrocarbon before using.

3 oz Tablet about

CHAPTER XXVI

PLASTERS

In the preparation of this class of remedy for local application, the object is to mix emollient or therapeutically inert substances with various medicaments in such proportions that the resulting mixture, although firm and solid at ordinary temperatures, will, when spread in a thin layer on calico, leather, or other material, be rendered adhesive and flexible by the warmth of the body. The Pharmacopœia enumerates a fairly long list of plasters, the greater number of which contain lead plaster as a basis, the other principal basis consisting of a mixture of resins and soap. Of late years the use of plasters of this class has fallen into partial disuse, their place to a great extent having been taken, by medicated and plain adhesive plasters prepared with a rubber basis.

Some plasters are merely intended to afford support to the parts of the body to which they may be applied, while others are medicated so as to act as vesicating or anodyne applications. The spreading of plasters is one of those operations connected with the dispensing of medicines which requires more skill and experience, than almost any other branch of the art; for this reason the beginner is recommended to practise by spreading some inexpensive plaster, as Emp. Resinæ, on brown paper until a fair amount of dexterity is acquired, when a trial may be made on leather.

Plasters are generally spread on the *rough* side of sheepskin (plaster leather), but are sometimes required on wash-leather, calico, silk, or swan's-down; if the last is employed the plaster is spread on the smooth side. When a plaster is ordered in a prescription, in addition to the name of the principal medicament or pharmacopœial title, the size and shape are usually indicated; occasionally the part is named to which the plaster is to be applied, and the determination of the size and shape left to the judgment of the dispenser. There are certain shapes which are generally adopted for application to particular parts of the body, and in the absence of specific instructions, the dispenser should conform to the recognised practice in this respect. Thus, Fig. 147 represents the form of plaster usually applied to the chest; Fig. 148, that for application between the shoulders; Fig. 149, that for the small of the back; Fig. 150 is the form adopted for the side; Fig. 151, for applying behind the *left* ear; Fig. 152, for the *right* ear; Fig. 153 represents the form given to breast plasters for women. It will of course be understood that these figures

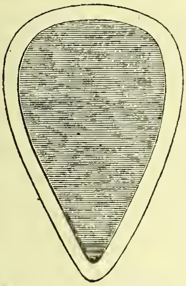
FIG. 147.

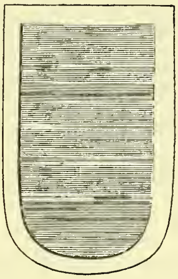
FIG. 148.

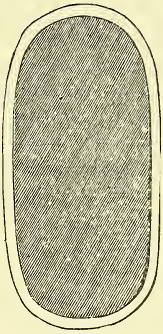
FIG. 149.

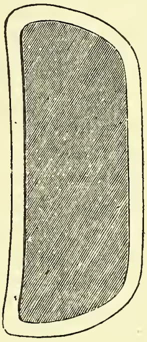
FIG. 150.

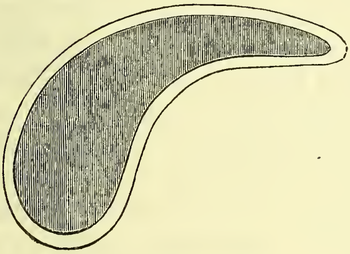
FIG. 151.

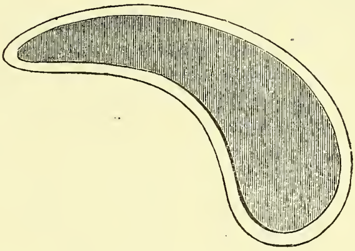
FIG. 152.

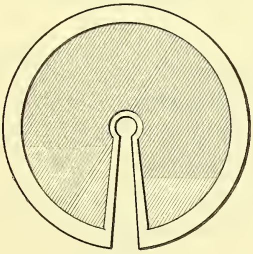
FIG. 153.

represent the forms and not the relative sizes of the respective plasters.

Blisters for applying to the back of the ear are invariably spread on adhesive plaster; all others, unless otherwise directed, on ordinary white plaster-leather. It is customary to leave a margin of unspread leather around all plasters. For ordinary sizes a margin of one inch is considered sufficient, while for very small plasters the margin may be reduced to half an inch or even a quarter of an inch.

When about to spread a plaster, say one of belladonna for the small of the back, 10 inches by 6 inches,—a piece of leather free from flaws is selected about 3 inches larger either way than the specified size. A few sheets of thick brown paper are then laid on the counter to prevent the heat being conducted away too rapidly during the operation, and the leather carefully smoothed by ironing with a moderately hot spatula. To prevent the leather from soiling, it should be protected by a piece of paper whilst being made smooth. If too great a pressure or heat is employed at the beginning the leather is sure to wrinkle and cockle, when no amount of extra ironing will flatten it out again. The use of the sheet of paper during the ironing process is an extra safeguard, for if too great a heat is used, the paper will show signs of scorching before the leather is much harmed.

Cutting the shape.—The leather having been prepared, a piece of smooth paper, say 14 inches by 10 inches, is folded equally across its longest diameter, and again across its shortest diameter. Five inches are next measured off (on the quarter-sheet) in the longer direction and 3 inches in the shorter, and the inside cut out as shown in Fig. 154. Very accurate shapes may be cut if a pencil line is first ruled as a guide, the rounded corners being made equal by placing a small coin in the angles and drawing the

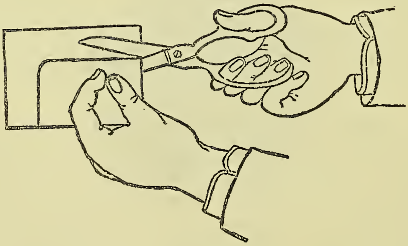

Fig. 154.

curves around it. The paper margin is then soaked for two or three minutes in water, excess of water removed by blotting-paper, and pressed evenly on the leather. The practice of smearing the paper margin with soft soap to make it adhere is not to be recommended, as the soap discolours the leather.

The next step is to melt the plaster. The time-honoured way was to press the roll of plaster against the hot iron, allowing the melted medicament to collect on a piece of paper, from which, as soon as sufficient had collected, it was transferred to the leather. Experience, however, has taught that it is preferable to take a weighed quantity of plaster (about 10 grains to the square inch), and having cut it up in small pieces, melt it in a porcelain capsule over a Bunsen flame. When completely melted, but not in a state of fluidity, the plaster is quickly transferred to the centre of the leather, and with as little loss of time as possible, spread evenly over the surface. Spreading should commence from the middle and finish up at the margin, so that when completed the plaster should be of equal thickness in all parts, and the surface even and somewhat glossy. If the melted plaster is of the right temperature it will spread easily, and yet not penetrate the leather sufficiently to show through at the back. The shape must be carefully removed before the plaster sets, as once the latter is cold, the paper leaves a broken margin when detached. Should the plaster, however, have been allowed to become quite hard before this has been done, a moderately warm plaster iron must be passed lightly over the margins before attempting the separation. To avoid accident, especially in the spreading of large plasters, it is safer to have two heated plaster irons at hand, so that if one cools unduly, the second may be at once requisitioned.

The shape of the spatula or plaster iron is not of very great importance, the most usual shape being that shown in Fig. 155. Plaster irons may be heated in an ordinary coal fire, but preferably in a Bunsen flame, as being less likely to cause injury to the blades. The exact temperature can only be learned by experience. If the application of the iron causes the plaster to smoke, the temperature is much too high. Of late years an improved form of spatula (Fig. 156) has come into use, and consists of a hollow iron of the usual shape, provided on its upper surface with a number of small holes. A flexible gas tube is attached to the handle, which is also hollow, and the gas lighted at the small holes. The amount of heat can be regulated to a nicety by increasing or decreasing the size of the gas jets, and in the hands of those accustomed to its use, the apparatus is of great utility.

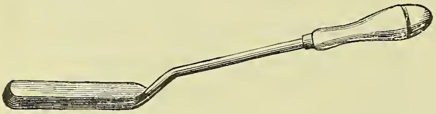

Fig. 155.

Fig. 156.

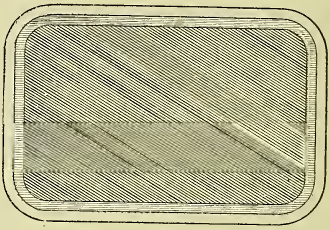

Fig. 157.

Plasters with adhesive margins (Fig. 157).—Certain plasters which possess little or no sticking power of themselves require the addition of an adhesive margin to ensure their remaining fixed to the part of the body to which they are applied. Supposing a plaster of this description, 6 inches by 3 inches, is required, the usual method of proceeding is as follows:—A suitable piece of leather having been selected, a paper shape enclosing a space say 8 inches by 5 inches is cut, and made to adhere to the leather by moistening. A little adhesive plaster (Emp. Resinæ) having been melted in a small dish is poured on the leather close to the paper edge, and a margin of about 2 inches spread all the way round. The paper shape is next removed, and the plaster laid on a cold surface to become thoroughly hard. When this has taken place, a second shape, enclosing a space 6 inches by 3 inches, is cut from waxed paper, and laid on the leather in such a way that an even margin of adhesive plaster is left on each side. The second shape must be sufficiently broad to cover not only the adhesive margin, but the plain leather one. All being ready, the centre of the leather is next spread with the medicated plaster, care being taken not to spread any more hot plaster on the waxed shape than can be helped. When the shape is removed it will probably be found that the surface of the adhesive margin appears somewhat dull where the paper was pressed on to it, but the dulness at once disappears if the finished plaster is passed rapidly face downwards over a Bunsen flame.

Breast plasters (Fig. 153).—These plasters should be spread on either "split skin" or "wash-leather." Much more difficulty will be experienced in spreading a plaster on these mediums than on ordinary sheepskin, on account of the liability to stretch and pucker. To obviate this tendency, the leather should be first stretched moderately tightly, and then tacked down over several sheets of brown paper, to a piece of smooth board; on no account should the leather be tacked down to the counter or bench, as the position of the leather requires to be constantly varied during the spreading. The melted plaster must not be poured directly on to the leather on account of its thinness, but on to a piece of brown paper, and only pushed on to the leather when it begins to cool. In fact, too much care cannot be exercised in regulating the heat both of plaster and iron, as if the temperature is allowed to rise ever so little too high, a discoloured plaster will be the result.

Sending out of plasters.—All plasters should be sent out in shallow cardboard boxes, the plaster being fastened down at each end, by one of the small brass clips used for securing documents, the ends being turned back outside the bottom of the box.

Blisters.—Blisters are invariably spread on ordinary adhesive plaster on unglazed calico. Besides those intended for the back of the ear (Figs. 151 and 152), square and circular blisters are frequently prescribed for application to the temples and other parts. The shapes are made from smooth white paper, and, a pencil line being drawn for a guide, are cut out by the aid of sharp scissors. Mr. Thompson gives the following hint on cutting shapes for ear blisters:—Bend the forefinger of the left hand, and allow it to touch the top of the thumb; the figure formed,

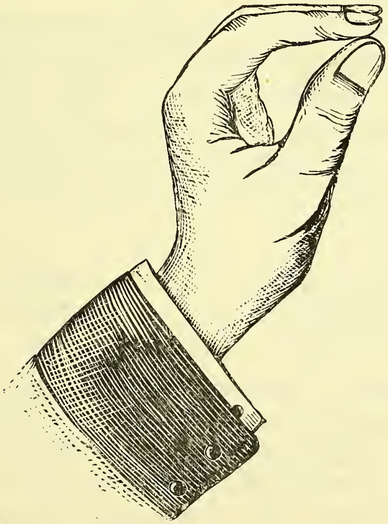

Fig. 158.

will give a fair idea of the proper form of blister for the left ear, Fig. 158. If the same is done with the right hand it gives the shape for the right ear. For blisters it is not necessary to leave so wide a margin as for other plasters, a quarter of an inch being ample. The cantharides plaster of the Pharmacopœia partakes more of the nature of a cerate than a plaster, and can be easily spread by means of a flexible knife, particularly if a little has been rubbed down on a slab previously to spreading. The classical method of blister spreading is with the thumb, but the practice has little to recommend it, and is certainly inelegant. Some dispensers are in the habit of sprinkling blisters with finely powdered cantharides, others of painting the blister with Liquor Epispasticus, with the idea of increasing the vesicating action; but such practices are extremely reprehensible, as they serve no useful purpose, and may produce the most unlooked-for results.

Court plaster consists of fine silk coated with isinglass. It is prepared by dissolving two ounces of isinglass in sixteen ounces of distilled water by the aid of a water-bath, adding by degrees when nearly cold, twelve ounces of rectified spirit. The silk having been evenly stretched on a frame, is painted over with this solution, the operation being repeated when dry, until five or six coats have been laid on, or the film appears of sufficient thickness. The silk used for this purpose may be either black, white, or flesh-coloured.

Rubber plasters.—Plasters containing a rubber basis have been in use for a number of years, and for certain purposes are a great advance on those hitherto considered. Their preparation unfortunately cannot conveniently be undertaken by the retail pharmacist on account of the expensive nature of the apparatus required, and the large quantities necessarily dealt with. The following is a usual formula:

Rubber (Para)...	2 parts.
Burgundy pitch	1 part.
Olibanum	1 ,,

The crude rubber is first washed and softened by prolonged kneading in a stream of warm water, the rubber finally leaving the machine in the form of thin sheets. These sheets are next thoroughly dried, and crushed between smooth rollers until they become plastic and soft. The next operation consists in kneading in the resins, which is accomplished by means of another pair of rollers, the upper roller of which revolves at twice the speed of the lower. When thoroughly mixed the medicaments are added, and after incorporation the whole spread on fabric by means of heavy rollers.

These plasters do not require warming to make them adhere, the normal temperature of the body being quite sufficient. For ordinary binding and strapping purposes rubber plasters are probably of great service; but it yet remains to be proved that as a means of exhibiting powerful medicaments they offer any advantage over those prepared according to the Pharmacopœia.

The official plasters.—Of the official plasters, only three need be mentioned here, viz. ammoniacum and mercury, mercury, and lead plasters. The remainder are simple mixtures. The first two are interesting from the method adopted for dividing the mercury. When sulphur is triturated with metallic mercury the latter soon becomes broken up into fine globules, each covered with a thin coating of sulphide, which effectually prevents their uniting again.

Lead plaster consists almost entirely of lead oleate with a little stearate palmitate; chemically, therefore, it is a mixture of insoluble ethereal salts.

The preparation of this plaster requires considerable care. The litharge should be in fine powder, and should be sifted into the vessel containing the oil and water. The mixture is then boiled and frequently stirred to prevent the lead oxide settling at the bottom. As much water is lost by evaporation, more should be added as required, or there is danger of overheating the plaster and darkening the product. If the operation has been carried out in a block tin or earthenware pan heated by a steam jacket this risk is practically obviated. In fact, with care, lead oxide decomposes olive oil without the presence of water at all, but the resulting plaster is rarely of a good colour. As the boiling continues, the contents of the vessel lose their red colour, and gradually become white. When this takes place heat is continued for an hour or so longer, or until a little removed from the pan and cooled, no longer becomes sticky on kneading between the fingers. When this is finished, the plaster is allowed to cool, after which it is taken out and kneaded in clean water to remove the glycerin. Lead plaster as it leaves the pan is of a greyish-white colour, but if it is kneaded and "pulled" for some time it becomes quite white. It is usual to roll the plaster into sticks of 8 ounces each, and after leaving in a trough of cold water until set, to wrap them in white calico. The yield is a little more than the weight of oil and oxide taken.

Lead plaster may also be obtained by decomposing Castile soap solution with acetate of lead, but the product is very brittle, and soon darkens on keeping. When powdered it is sometimes ordered for dusting, in certain skin affections.

CHAPTER XXVII

GLYCERINS

GLYCERIN (glycerol or propenyl alcohol), $C_3H_5(OH)_3$, is obtained as a by-product in the manufacture of soaps and candles. The chief constituents of the animal fats and the fixed vegetable oils are palmitin, stearin, and olein. These compounds consist of a fatty acid in combination with the trihydric alcohol *glycerol,* and are true ethereal salts.

When acted upon by superheated steam under pressure, or by boiling dilute mineral acids, these salts are decomposed; if the resulting mixture is then distilled in superheated steam, the mixed fatty acids and glycerin pass over, the former floating as an oily layer on the surface of the aqueous distillate in which the glycerin remains dissolved. The weak aqueous solution thus obtained is concentrated in vacuum stills, when, after one or two distillations the glycerin comes over fairly pure.

Large quantities of glycerin are also obtained by distilling soap leys after addition of a mineral acid, but much purification is required before it is fitted for medicinal use. The most frequent impurity present in cheap glycerin is butyric acid, recognised by the characteristic odour after heating with dilute sulphuric and ethylic alcohol. Arsenic has also occasionally been found in glycerin, derived probably from the mineral acid used to neutralise the soap leys. (See Arsenic in Appendix.)

Pure glycerin is crystalline below 17° F., when it has a specific gravity of 1·269. If only a trace of water is present, it refuses to crystallise, and forms a thick syrupy liquid, in which state it is official, sp. gr. 1·26. *Pure* glycerin boils at 290° C. under ordinary atmospheric pressure without undergoing decomposition, but if only traces of salts or impurities are present it partly decomposes into acrolein and water:

$$C_3H_5(OH)_3 = C_3H_4O + 2H_2O.$$

Glycerin is a very hygroscopic body, and rapidly absorbs water from the atmosphere, mixing with it in all proportions; this absorption of moisture from air continues until the glycerin has more than doubled its original bulk.

Glycerinum Acidi Borici.—This is prepared by heating glycerin with boric acid until water and excess of glycerin are driven off. The product may be looked upon as consisting of glycerin, $C_3H_5(OH)_3$, in which the three (OH) groups are replaced by (BO_3), thus:

$$C_3H_5(OH)_3 + H_3BO_3 = C_3H_5BO_3 + 3H_2O.$$

When this compound, which resembles Barff's boro-glyceride, is dissolved in water it splits up, glycerin and boric acid being regenerated.

Glycerinum Boracis.—When borax (sodium pyroborate) is heated with glycerin it is decomposed, sodium metaborate and glyceryl borate being formed:

$$2C_3H_5(OH)_3 + 2NaBO_2.B_2O_3 = 2C_3H_5BO_3 + 3H_2O + 2NaBO_2.$$

But owing to the presence of water, both as a consequence of the reaction and of that existing as water of crystallisation, a secondary reaction sets in, and boric acid is set free:

$$C_3H_5BO_3 + 3H_2O = C_3H_5(OH)_3 + H_3BO_3.$$

It would appear from this that the present formula for glycerin of borax yields a slightly different product from that obtained by the B.P., 1885, formula, which contained added water; further difference again may be caused according to the amount of heat employed in effecting solution. The longer and the greater the application of heat, the more water is evaporated, and the formation of glyceryl borate facilitated.

Mel Boracis.—Borax honey may properly be considered here, not only because the preparation contains a small quantity of glycerin, but because honey reacts with borax in precisely the same way as glycerin. Honey consists principally of dextrose, a compound possessing the properties of an aldehyde and a polyhydric alcohol. When, therefore, it is made to react with borax, sodium metaborate and an ethereal salt, analogous in composition to that formed when glycerol reacts with borax, are formed. As much water is present this salt is partly decomposed, dextrose and boric acid being formed during the secondary reaction.

The other "glycerins" of the Pharmacopœia do not call for special notice, being either simple solutions of salts in glycerin or simple mixtures.

CHAPTER XXVIII

COMPRESSED TABLETS

Compressed tablets are prepared by pressing the material between suitable dies into a disc-like or lenticular form. Until lately their manufacture has been almost entirely in the hands of a few manufacturers, but the moderate cost at which serviceable machines can now be obtained enables the pharmacist to prepare them for himself.

The simplest form of tablet apparatus is shown at Fig. 159. It is made of cast steel, and consists of a cylinder and two accurately fitting dies, the faces of which are hollowed out so as to impart the familiar lenticular appearance. In using, the cylinder is placed over the base block, the medicament introduced, and the upper die inserted, when the operation is completed by striking the latter a smart blow with a hammer. It will gene-

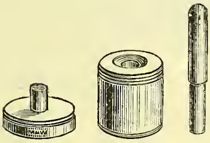

Fig. 159.

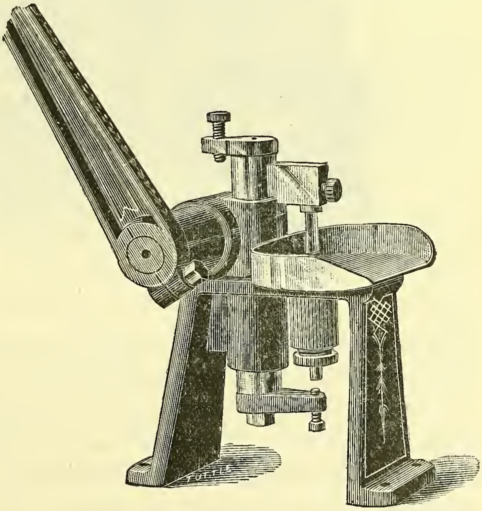

Fig. 160.

rally be found that the tablet adheres to the cylinder; in that case, by removing the base block, and holding over a sheet of clean paper, it may be expelled by gently tapping the upper die.

The next, Fig. 160, represents a machine in which a simple mechanical arrangement obviates the use of a mallet. The weighed or measured powder is slid into the cylinder, and the plunger, which is operated by a lever, is brought smartly down. Its construction is so simple that it cannot easily get out of order, and it enables the operator to turn out tablets with a sufficient degree of rapidity for dispensing purposes.

Holroyd's No. 5 machine (Fig. 161) is specially designed for pharmacists. It weighs about 2 cwt., and occupies a space 12 inches by 24 inches, and is worked by hand. The machine makes one tablet at each impression of the punch, and turns out about forty a minute. It is very easily cleaned, and the punches and dies may be changed from one size to another in about three minutes. In the feed cup to the left, is a stirrer which keeps the material in uniform motion, and the weight of the tablet is adjusted on turning a side screw. The pressure is regulated by a worm wheel on the crank pin.

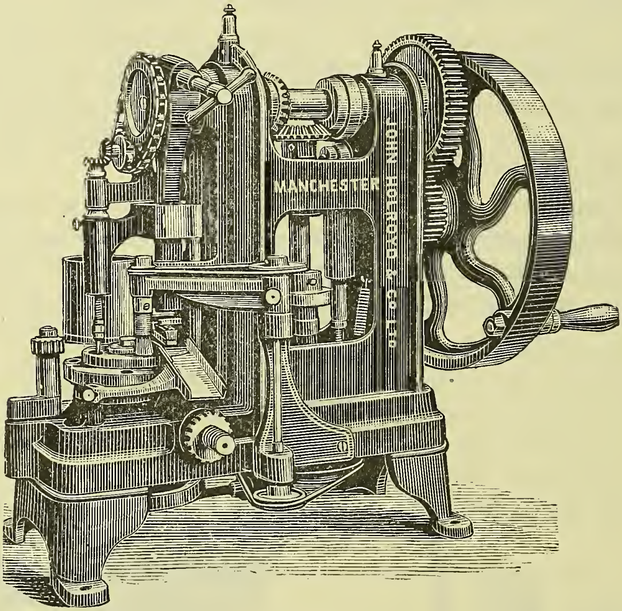

Fig. 161.

Of moderate-sized machines the Keystone (Fig. 162) is one of the simplest. Its capacity is about 70 to 100 tablets a minute. The No. 1 size figured below is adapted for hand or power. It occupies a counter space of 10 by 16 inches, stands 21 inches high over all, and weighs 150 lbs. The machine can be operated with very little instruction, and there is practically no loss of material. The dies, which vary from $\frac{1}{8}$ to $\frac{5}{8}$ inch in diameter, are of fine steel, and turn out tablets of excellent finish.

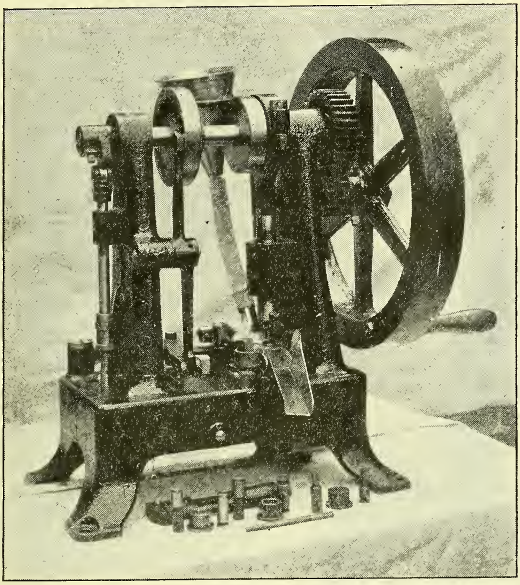

FIG. 162.

It is important that the dies and punches be perfectly smooth and well polished if good work is to be turned out. The pressure must be carefully regulated ; if excessive, the tablets disintegrate very slowly. Insoluble substances should be compressed as lightly as possible. Unduly light pressure, on the other hand, is accompanied by a greater tendency for the material to stick to the face of the dies. The three great points in tablet making are—to ensure proper cohesion of the particles of material under compression, to prevent adhesion of the particles to any part of the machine, and to carefully regulate the pressure, so as to secure ready disintegration of the finished tablet.

For the dispensing counter, the Pazo Compressor, shown in Fig. 163, is as well adapted as any. It is screwed to the edge of the counter, and movement of the upper die is effected, by pulling the lever forward. The powder is fed into the mould, through the funnel attached to the front of the machine, and falls into the lower die. At the bottom of this is an adjustable screw, by means of which the thickness and hardness of the tablet can be regulated to a nicety by raising or lowering the die, while at the back, hidden by the framework of the machine, is a rigid projecting spur. On drawing the lever forward, the piston carrying the descending die comes in contact with this spur, and the mould is automatically brought into position; at the same time the upper and lower dies firmly compress the powder between them, whilst on reversing the lever the finished tablet is automatically ejected, and falls into an open drawer or other suitable receptacle. It follows, then, that the speed of manufacture is only limited by the rate at which the operator can supply the material to be compressed and move the lever forward and backward. To adjust the machine at the outset, all that is necessary is to place the required weight of powder in the mould, depress the upper die, and screw up the lower die as tightly as possible. Then reverse the lever, give the adjusting screw another half-turn if necessary, and firmly clamp it by turning the milled head seen immediately below the mould. If substances of sparing solubility are to be compressed, it will of course be necessary to make allowance for this, either by reducing the pressure or by special treatment of the medicament. The machine, when once adjusted, will continue to deliver tablets of the selected material—compressed to an equal degree and of identical weight—until the adjustment is altered.

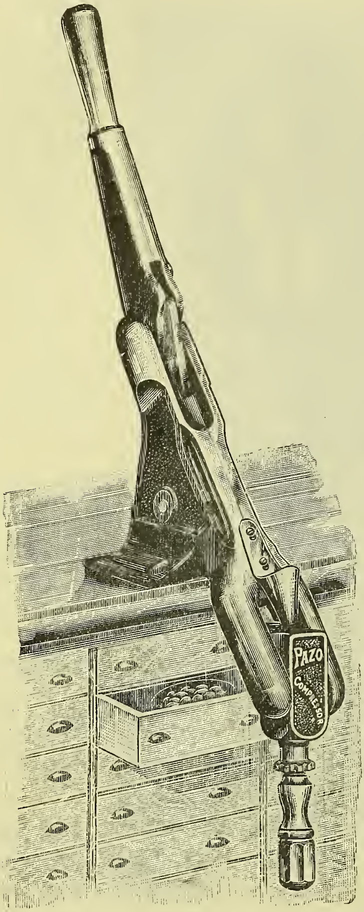

Fig. 163.—The "Pazo" Compressor.

An important point, which may be referred to here, is that the amount of pressure imparted to the descending die is quite independent of the force employed by the operator in depressing it, the advantage in this respect over other small compressors being very marked.

Preparation of the material.—It is not desirable to have the material to be compressed in a state of very fine powder, as it will neither feed properly nor lend itself to compression into uniform tablets. The condition to be aimed at, is that in which ground ammonium chloride appears, viz. in the form of small crystals. Very few substances can, however, be obtained commercially in this condition, and it is better, therefore, to reduce the material to a state of fine powder, and granulate it by moistening it with dilute alcohol, syrup, mucilage, or water, according to its nature, and press the mass through a sieve. A No. 30 sieve will be found fine enough for most purposes, but a few substances, like charcoal, pepsin, &c., should be passed first through a 20 sieve, and again when dry through a No. 50 or 60. When moistening the powder, care should be taken to add only small portions of the liquid at a time, to prevent the formation of a pasty mass. To prevent the granulated powder sticking to the dies, a lubricant of some kind must be added. French chalk in very fine powder, and a 2 per cent. solution of white vaseline in ether, are the most useful. The former should be dusted on to the dry granules, and the whole rotated gently in a wide-mouthed bottle; if ethereal solution of soft paraffin is used, it may be sprayed over the granules with an atomiser. In either case very small quantities suffice to lubricate the granules if only properly applied.

The following formulæ are given as examples of procedure.

Bicarbonate of soda, gr. v. Take of—

Bicarbonate of soda	16 ounces.
Mucilage of acacia	½ ounce.
Distilled water	a sufficiency.

Place the bicarbonate of soda in a perfectly clean mortar, add the mucilage, and mix intimately; if necessary add a few drops of water until fairly moistened, but by no means pasty. Now turn out on to a clean sieve having thirty wires to the linear inch, and press through with bottom of a gallipot; dry in a shallow dish with a gentle heat, and add a small pinch of finely powdered French chalk. Mix gently in a wide-mouthed bottle as directed; turn out on to a sheet of paper, and give a spray with the solution of paraffin; agitate gently, and stamp into five-grain tablets in the usual manner.

Soda-mint tablets. Take of—

Bicarbonate of soda	15 ounces.
Mucilage of acacia	½ ounce.

Granulate as above, dry, and add the following mixture:

Ammonium carbonate	1 ounce.
English oil of peppermint	¼ ,,
French chalk	30 grains.

Mix by shaking in a bottle, and stamp into five-grain tablets.

Voice tablets. Take of—

Cocaine hydrochloride	20 grains.
Distilled water	½ ounce.
Mucilage of acacia	¼ ,,

Dissolve, and add to—

Powdered potassium chlorate	2400 grains.
,, borax	1600 ,,

Mix thoroughly; granulate, dry, and lubricate. Stamp into five-grain tablets. Each tablet contains $\frac{1}{40}$ grain of cocaine hydrochloride, 3 grains of potassium chlorate, and two grains of borax.

Pepsin tablets. Take of—

Powdered pepsin	8 ounces.
,, sugar	½ ounce.

Moisten cautiously with a very little dilute alcohol; granulate, and dry at a temperature not exceeding 90° F. Lubricate with French chalk. The granulated pepsin should be passed through a No. 50 sieve. Tablets one or two grains each.

Ammonium chloride. This may be pressed into tablets without any preparation. The tablets should weigh 3 grains each.

Salol. Take of—

Powdered salol	8 ounces.
,, sugar	¼ ounce.
,, tartaric acid	20 grains.
Bicarbonate of soda	25 ,,

Moisten with alcohol, mix thoroughly, and dry at a temperature not exceeding 95° F. (salol melts at about 107° F.); again pass through sieve and lubricate. Stamp into five-grain tablets, using as little pressure as will make the salol firmly coherent.

Phenacetin, sulphonal, trional, and acetanilide may be prepared in the same way as salol.

Other insoluble powders, such as lithium carbonate, Dover's powder, bismuth carbonate and oxynitrate, should contain a little acid and bicarbonate of soda to promote disintegration. They may be moistened with dilute alcohol, and lubricated with French chalk.

Saccharin, citrate of lithia, antipyrin, and other freely soluble drugs simply require moistening and granulating. They should all be lubricated with the solution of paraffin in ether. Potassium bromide and iodide simply require crushing; they may then be stamped like ammonium chloride.

Hypodermic tablets should be made up to the required bulk with pure sodium chloride.

Tablet making is an art which requires considerable experience as to the selection of the moistening agent, as well as to the amount of pressure required. McFerran's advice is to learn the nature of each article to be compressed, and to take advantage of our knowledge of its solubility in different menstrua; and when the contrary nature of the different articles in a combination precludes this knowledge, to fall back upon such corrections as experience suggests to meet the particular case.

TABLET TRITURATES

Tablet triturates are rarely prescribed in this country. The name is applied to small friable tablets composed of medicated milk-sugar. The mould, Fig. 164, is usually made of vulcanite, and consists of two parts,—an upper plate perforated with holes to the number of fifty, the size of the tablet required; and a second plate carrying a number of smooth upright pegs, exactly corresponding with the holes in the upper plate, and used to displace the finished tablets.

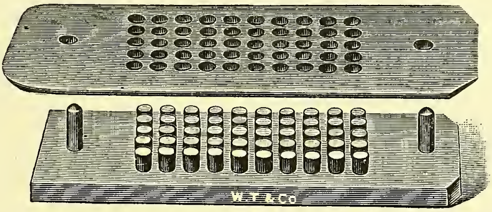

Fig. 164.

The first operation is to ascertain the exact content of each hole. This is done by moistening finely powdered milk-sugar with proof spirit, and pressing the pasty mass into the holes. The tablets are at once forced out by means of the pegs, dried, and weighed. As a rule each tablet will be found to weigh about $1\frac{1}{3}$ grains, but the total weight of the fifty plain milk-sugar tablets must be carefully noted. Suppose the total weight to be 65 grains; if fifty tablets, each containing $\frac{1}{10}$ grain of cocaine hydrochloride are required, it is only necessary to triturate 6 grains of the salt with 72 grains of finely powdered milk-sugar, moistening slightly with proof spirit and pressing into the moulds. The excess should weigh exactly 13 grains if it is all collected and thoroughly dried.

If the active ingredient is a liquid, it must be intimately mixed with the requisite quantity of milk-sugar and evaporated to dryness. Extracts should be dissolved in the smallest possible quantity of water or spirit, according to their nature, and distributed through the milk-sugar as described for liquids. It is important that the milk-sugar be in the finest possible powder, or the tablets will be too friable to keep their shape when dry. 40 or 60 per cent. alcohol is the most generally useful excipient, but the selection is entirely governed by the nature of the medicament, the rule being to use one possessing only a slight solvent action.

In practice it is found advantageous to lubricate the tops of the pegs by spraying them with a solution of 1 part of colourless petroleum (Paraffinum Liquidum) in 100 parts of absolute ether. The tablets should be pressed from the mould soon after making as if left too long they cannot be detached without crumbling.

CHAPTER XXIX

LOZENGES

A LOZENGE properly, is a four-sided figure having two acute and two obtuse angles. In pharmacy, however, the term is extended to round, oval, or other conveniently shaped flattened masses, consisting of a medicinal substance mixed with gum, sugar, extract of currants, &c., and intended for slow solution in the mouth.

Lozenges or troches afford a convenient method of administering many remedies of a mild type, especially such as are intended for continuous application to the throat, as potassium chlorate and tannin, but are not well adapted for the exhibition of potent remedies.

The Pharmacopœia gives several formulæ for the preparation of lozenges, all of which yield excellent results. The operation may be divided into three parts,—the preparation of the mass, the cutting or dividing into lozenges, and the drying.

In preparing the mass, the active medicinal ingredients are first intimately triturated with a small portion of the sugar. After thorough admixture the gum and remainder of the sugar are added, and the whole passed through a sixty-hole sieve. The massing ingredients are next added, and the whole thoroughly kneaded in a stone mortar, or in a machine similar to that used for pills. The mass need not be made so hard or adhesive as a pill mass, but at the same time must not be so soft that the lozenges fall and lose their shape before they are dry. After mixing, the mass is rolled out on a lozenge board, Fig. 165, the thickness of the cake being regulated by the height of the bearings at each side of the board. Instead of regulating the thickness of the lozenges by side bearings, the roller itself may be provided with broad flanges, several pairs of which may be stocked for the production of lozenges of different degrees of thickness. If much work is anticipated the roller should be made of nickelled brass, as wooden rollers soon become indented and

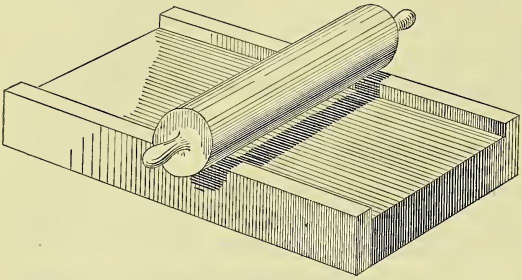

Fig. 165.—Lozenge-board and roller.

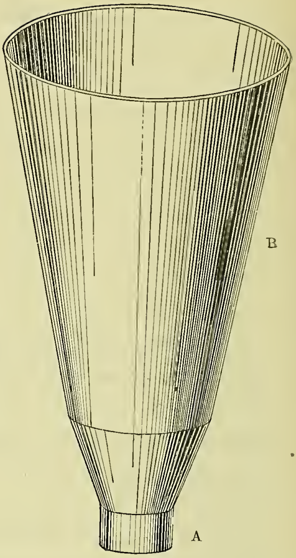

Fig. 166.—Hand lozenge cutter.

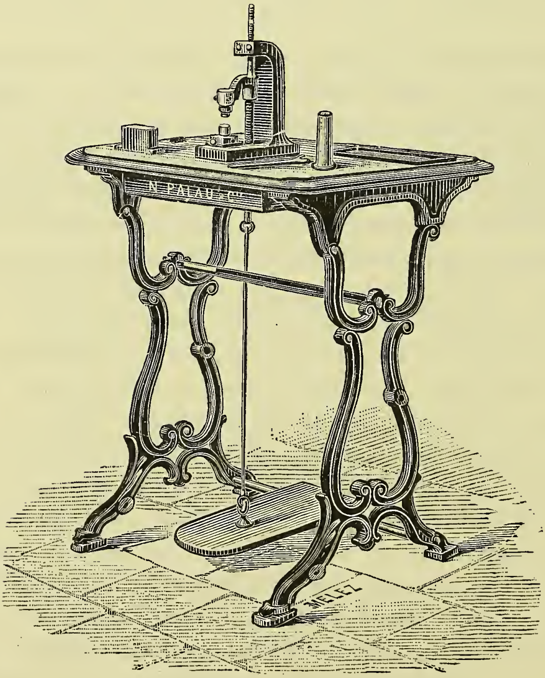

Fig. 167.—Lozenge stamper with pedal.

cracked, unless taken very great care of. To prevent the mass from sticking a mixture of two parts of starch with one of sugar is dusted from a dredger over the board. After rolling into a perfectly flat and even cake the mass may be divided into lozenges by cutters or punches of any desired shape, the most usual being ovals or circles. For hand-work the cutter consists of a short steel cylinder, Fig. 166, A, about quarter of an inch long, brazed on to a conical body, B. After cutting, the lozenges are placed on shallow trays and left to dry. The temperature of the drying-room should not be higher than 90° F.

When required, lozenges are stamped with the name and dose of the principal ingredient. This operation may be performed by hand, or in a machine actuated by a pedal, Fig. 167. The machine stamps about 14 lbs. of lozenges a day, and is especially useful to small manufacturers.

Fig. 168 is of more elaborate construction; the mass, which has been previously rolled into strips of the proper width and thickness, being fed under the cutting stamps, the lozenges ranging themselves on the bands, while the clippings fall on one side to be worked over again.

The "Climax" lozenge machine (Fig. 169) is perhaps one of the most perfect made. It has no ratchets to the pinning machine, whilst the adjustment of the skip of paste can be made without stopping, which is a great advantage. The pinning machine has six rollers (see illustration), each pair having a separate wheel for regulating the thickness, and by careful adjustment an endless sheet of paste can be fed under the cutters. Either of these machines turns out perfectly finished lozenges, the edges forming a right angle with the surfaces.

Pastilles.—Of late years pastilles prepared with a basis of glyco-gelatin have come into very general use, and this is not to be wondered at considering the ease with which twelve or twenty-four pastilles can be turned out for the filling of any special prescription. The most generally useful glyco-gelatin basis is made as follows. Take of—

Gelatin	12 ounces.
Glycerin	24 „
Distilled water	30 „
Oil of orange-peel	10 minims.

Allow the gelatin to soak in the water until softened; dissolve by the aid of a water-bath; add the glycerin, and when nearly

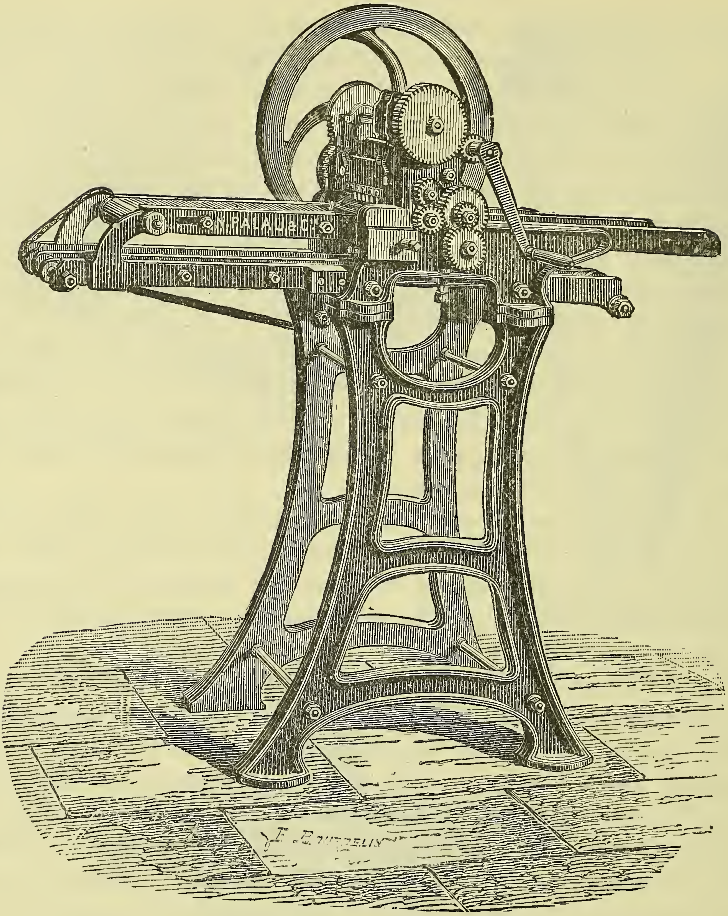

Fig. 168.—Lozenge cutting machine.

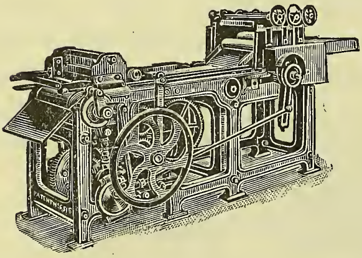

Fig. 169.—The "Climax" lozenge cutting machine.

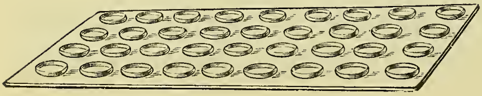

Fig. 170.—Billson's pastille mould.

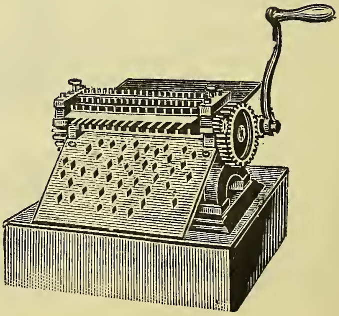

Fig. 171.—Jujube cutting machine.

cool, the oil of orange-peel. Pour out into a flat mould, and when quite cold divide into one-inch squares for convenience in weighing.

Supposing twenty-four pastilles are required, each containing one eighth grain of cocaine hydrochloride : 3 grains of the salt are dissolved in 792 grains of warm glyco-gelatin basis, and the mixture poured into moulds similar to Fig. 170. The mould illustrated was designed by Mr. Billson, and is one of the most useful of the kind for small quantities. If the tray is set in a cool place solidification takes place in a few minutes, when, if the moulds have been previously *slightly* oiled, the pastilles may be turned out smooth and bright, and ready for sending out.

Jujubes.—Jujubes are often looked upon as sweetmeats, but occasionally are used for the exhibition of certain remedies of very mild type, as eucalyptus, voice, and other jujubes. Thirty parts of gum arabic, 20 of white sugar, and 35 of water are boiled together for half an hour or more in a steam-jacketed pan, until an even mixture results. After standing a little time the scum is removed and the medicament or flavouring agent added, and the whole poured into moulds to set. If not sufficiently hard the trays containing the jujube paste are placed in the drying-room for a few days to evaporate. As soon as they are hard enough the sheets are divided by a machine similar to Fig. 171, fitted with "clearers," which revolve with the blades and prevent the jujubes from sticking. To cut diamond-shape, the strips, which have already been put through once, are passed through the second time in a diagonal direction, when the jujubes come out as shown in the cut.

Tabellæ Nitro-glycerini.—These are the only lozenges in the Pharmacopœia for which no actual formula is given. The following yields excellent results. The 10 per cent. solution of nitro-glycerin should be kept in a safe place, away from the official solution of nitro-glycerin, *than which it is ten times stronger*. Take of—

	All by weight.
Solution of nitro-glycerin in alcohol, 10 per cent. by weight ...	100
Powdered chocolate	1450
,, sugar	2500
Mucilage of acacia	390
Powdered acacia	360
Distilled water	200

Mix the powders, add the solution of nitro-glycerin, and stir *with a light hand* until evenly mixed. Add the mucilage and water, and work up to a soft mass; roll into a cake, and divide into lozenges weighing exactly 5 grains. Each lozenge will contain one hundredth grain of nitro-glycerin.

Note.—Keep the mass soft and plastic, and carefully avoid pounding or concussion during the operation. Finally dry at the ordinary temperature.

CHAPTER XXX

EYE DISCS

No directions are given in the Pharmacopœia for the preparation of lamellæ or eye discs, although three formulæ have been official since 1885. The directions now given are published for the first time.

Preparation of the basis.—Take of

Transparent gelatin	180 grains.
Glycerin	15 minims.
Distilled water	2 ounces.

Mix the glycerin and water; allow the gelatin to soak in the mixture until softened, then dissolve by gentle heat and strain through muslin.

Lamellæ Atropinæ.—Each disc contains $\frac{1}{5000}$ grain of atropine sulphate. Take of

Basis	80 grains.
Solution of atropine sulphate (1 grain in 100 minims) ...	20 minims.

Dissolve the basis by application of gentle heat, and mix. Spread a very thin coating of white beeswax (hard paraffin will not answer) over a piece of plate glass about 5 inches square. Then take a piece of thick writing-paper and cut out a shape as for a plaster, 4 inches square, and paste evenly over the waxed glass. Next place the glass on a level slab of iron heated to about 100° F. (38° C.), and when the glass has attained the same temperature pour on the medicated basis and spread it evenly over the surface, taking care that none runs over the edge of the paper shape. As soon as the film has solidified, place the coated glass in a cardboard box to keep out dust, and dry at a temperature not exceeding 100° F. for fourteen days, when the film should be easily detached from the glass. With such a punch as those used by shoemakers to punch eyelet holes, cut out discs of exactly $\frac{1}{7}$ inch diameter.

Calculation for above.—A film 4 inches square (= 16 square inches in area) would yield 784 square discs, the sides of each square being $\frac{1}{7}$ inch, therefore it would equally yield 784 circular discs of $\frac{1}{7}$ inch diameter; but as by the latter method of cutting there would be considerable loss of scrap, it is necessary to use sufficient medicament for 998 (practically 1000) discs. Thus—

The film = 16 square inches.

Each disc has an area $\frac{11}{686}$ square inch.*

Therefore 784 discs would = $12\frac{4}{7}$ square inches,

and allowance for the $3\frac{3}{7}$ square inches of film wasted, which is equal to 214 discs = in all 998 (practically 1000) discs.

Then, as practically 1000 discs are required, each containing $\frac{1}{5000}$ grain of atropine sulphate, $\frac{1}{5000} \times 1000 = \frac{1}{5}$ grain, or 20 minims of a solution 1 grain in 100 minims.

Lamellæ Cocainæ.—Each disc contains $\frac{1}{50}$ grain of cocaine hydrochloride. Take of

Basis	80 grains.
Cocaine hydrochloride	20 „
Distilled water	20 minims.

Dissolve, mix, and spread a film having an area of 16 square inches; when dry divide into circles having a diameter of $\frac{1}{7}$ inch.

Lamellæ Homatropinæ.—Each disc contains $\frac{1}{100}$ grain of homatropine hydrobromide. Take of

Basis	80 grains.
Homatropine hydrobromide	10 „
Distilled water	20 minims.

Warm until dissolved, and spread a film having an area of 16 square inches; when dry divide into discs, each having a diameter of $\frac{1}{7}$ inch.

Lamellæ Physostigminæ.—Each disc contains $\frac{1}{1000}$ grain of physostigmine sulphate. Take of

Basis	80 grains.
Solution of physostigmine sulphate (5 grains in 100 minims)	20 minims.

Mix, dissolve, and spread a film having an area of 16 square inches; when dry divide into discs, each having a diameter of $\frac{1}{7}$ inch.

Note.—For other discs, or discs of other strength, calculate that 16 square inches of film (*i. e.* 4 inches square) require enough medicament for 1000 discs, although only 784 may be cut from it.

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* To find the area of a circle multiply the square of the radius by $\frac{22}{7}$.

CHAPTER XXXI

POWDERS, CACHETS, ETC.

The Pharmacopœia recognises sixteen kinds of compound powders. The ingredients for these should be in very fine powder, being passed through a sieve having not less than 120 meshes in a linear inch. The reduction of medicinal substances to powder is fully dealt with under the head of Comminution, and need not be further considered.

It is important that all compound powders should be thoroughly mixed. Those containing potent remedies cannot have too much care bestowed on their admixture. Small quantities may be well triturated in an ordinary Wedgwood mortar, perfect uniformity being ensured by passing the product through a small sieve. In all cases, active ingredients and colouring principles should be first triturated with a small quantity of the diluent, the remainder being added by degrees. To obtain the full power of the colouring material, the powder is usually damped with alcohol. The effect of this is well seen if 5 grains of carmine be mixed dry with 1 ounce of chalk, and a corresponding quantity after moistening with a little alcohol. In the first instance the colour is scarcely appreciable, while the latter has developed a beautiful rose tint.

For sifting small quantities of powders at the counter without making a dust, the apparatus shown at Fig. 172 is very useful.

When the powder contains such incompatible substances as an alkaline carbonate with an acid (effervescing salines for example), the materials should be dried separately in a hot-air oven, and afterwards mixed lightly and sifted. As pointed out under the head of Explosive Combinations, mixtures containing potassium chlorate, or hypophosphites, and tannic acid, sugar, sulphur, &c., must never be rubbed together. If necessity actually requires the dispensing of these chemically incompatible substances, they should first be powdered separately in clean mortars, afterwards mixing very lightly with a horn spatula. If necessary such powders may be sifted through muslin stretched on a wooden hoop. To minimise danger, avoid friction and pressure.

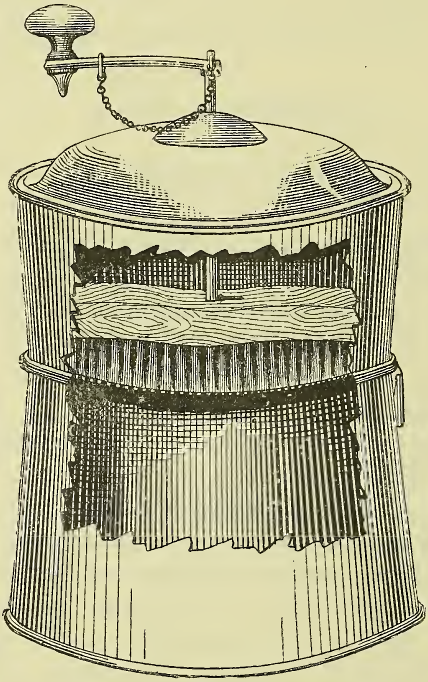


FIG. 172.—Toogood's mixer and sifter.

When quantities of more than a few ounces have to be mixed and sifted, it is better to employ one of the numerous combined-sifting and mixing machines. Much labour and time are saved, besides the entire avoidance of dust and waste during the operation. Figs. 173 and 174 illustrate convenient sizes for retail pharmacists; from one to ten pounds can be mixed and sifted in a few minutes, or only a few ounces with equal convenience. For mixing and sifting a few ounces only, the machine is placed on a sheet of paper on the counter, instead of on the metal receiver. The construction and application of the machine will be readily understood from the illustration. The lower compartment receives the mixed and sifted powder; the upper consists of a metal box containing a curved sieve. The powder is placed in this box and the lid closed, and the handle which actuates the scraper moved up and down. The scraper consists of several

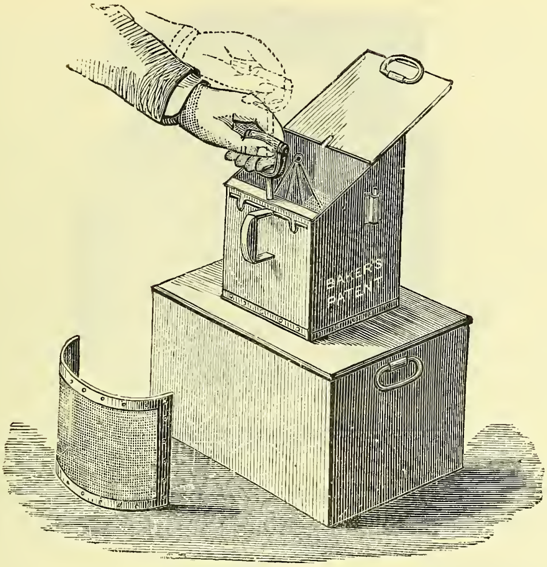


Fig. 173.—Mixing and sifting machine.

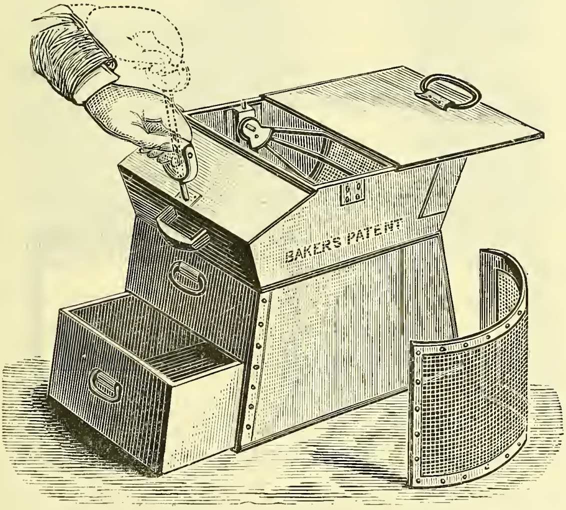


Fig. 174.—Mixing and sifting machine.

pieces of stout wire, so arranged that when the handle is withdrawn the whole of the surface of the sieve is traversed. Fig. 175 shows a somewhat larger machine of the same type, provided with a hopper, fly-wheel, and crank, the capacity and rapidity of operation being greatly increased.

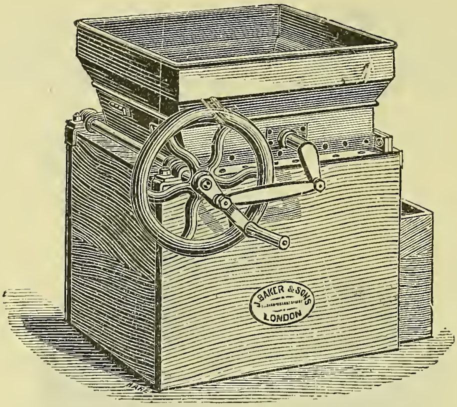
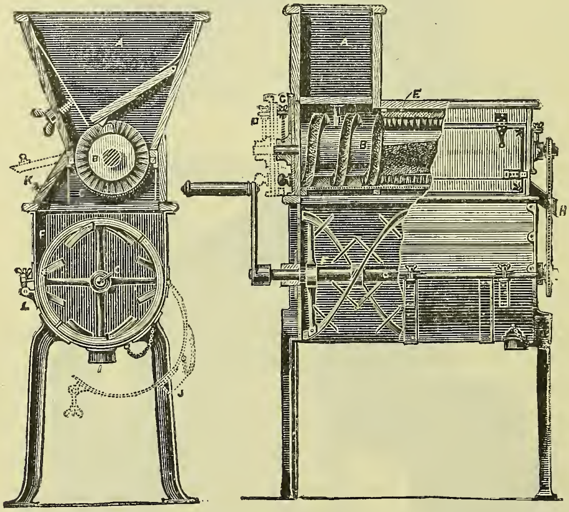


Fig. 175.—Mixing and sifting machine.

When considerable quantities of powders have to be intimately mixed and sifted, a machine similar to Figs. 176 and 177 is employed. The feed hopper A contains a regulating gate ; this can be closed or opened as desired, for controlling the supply of ingredients which pass from this to the sieve C, where they are acted upon by the spiral brush B, the first portion of which, causes the material fine enough, to pass through the meshes of the sieve, while the lumpy portions of the powder are carried along to the reducing portion of the brush. This last is of peculiar



Figs. 176 and 177.—Gardner's mixing and sifting machine.

construction, and working in conjunction with the top sieve E, reduces the lumps to powder and at the same time passes them through the sieve C; but any foreign matter such as stones, wood, and unreducible lumps is automatically thrown out at the outlet spout H at the end of the sieve. This prevents choking or cutting of the sieve, while time and labour are saved in not having to take the machine to pieces to clean it out.

After passing through the sieve C, the partially mixed powder falls into a mixing or blending chamber F, which, as will be seen from the illustration, contains an agitator G. After a short time the accurately mixed powder can be emptied by opening the slide over the outlet I. The door K is provided, for introducing fine ingredients which do not require sifting, into the chamber F, thereby economising time and labour.

The sieve C is readily withdrawn, as shown by the position of the dotted line D, and another of coarser or finer mesh can be substituted when it is necessary to sift some of the ingredients through a finer sieve than others.

Dividing of powders.—The division of all powders intended for internal administration should be performed with the utmost exactitude, and in no case should recourse be had to the lazy and inaccurate method of subdivision by means of measures, or by flattening out the powder and dividing it into *apparently* equal portions by means of a spatula. Cup-shaped measures made of boxwood are sold and recommended for measuring Seidlitz powders. The results are, however, very untrustworthy, and it is hardly necessary to remind pharmacists that vendors of Seidlitz powders that do not comply with the pharmacopœial requirements commit a breach of the law, of which some public analysts are only too ready to take advantage.

For rapid weighing, hand balances are much to be preferred to standard ones. Moreover, in the latter form of balance, the scale pan is usually made of glass, and unless great care is taken, the edges soon chip, which unless observed and readjusted, impairs the accuracy of the weighing. The paper for wrapping powders should be of good quality with a glazed surface. When one or two dozen powders have to be weighed and wrapped, it is convenient to lay down all the papers on the counter in rows of four or six, and weigh out the whole number before commencing to wrap. This is done by folding the paper over away from the operator, to within half an inch of the margin; the outside edge is next folded inwards and again on itself, when the ends are finally folded over on one of the devices figured at Figs. 178 and 179. By this means all the packets are obtained of uniform length.

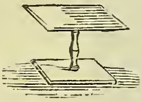


Fig. 178.—Powder folder.

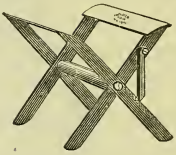


Fig. 179.—Powder folder.

Weighed powders are generally sent out in card boxes, and the appearance is considerably enhanced, if the powders are folded to such a height as to correspond exactly with the top of the box. Deliquescent substances such as lithium citrate, &c., should, after folding in white paper, be wrapped either in tinfoil or gutta-percha tissue. If the latter is employed the ends may be effectively sealed either by holding to a small gas flame, or touching with a camel-hair pencil dipped in chloroform.

Cachets.—The great advance that has been made during the last few years in synthetic remedies has helped to bring cachets into greater prominence. The desirability of introducing some of the newer remedies into the stomach without being acted upon by the salivary juices of the mouth, and the public demand for tasteless medicines, have created a need for this simple method of prescribing, and it is not surprising that prescriptions for medicinal powders in cachets are increasing rapidly in favour.

The essential qualities of a perfect cachet are—convenience of form, tastelessness, ready solubility with easy digestibility. The basis from which cachets are stamped is perhaps of the least harmful nature possible, consisting merely of flour and water.

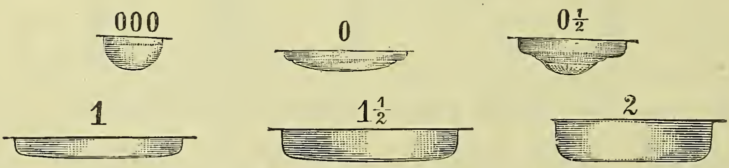


Fig. 180.—Empty cachets.

No.	000	will hold	1½	grains of quinine sulphate.	
,,	0	,,	3 to 4½	,,	,,
,,	0½	,,	6 to 7½	,,	,,
,,	1		9 to 10½	,,	,,
,,	1½		10½ to 15	,,	,,
,,	2	,,	15 to 18	,,	,,

This material after it has been dipped in cold water becomes quite soft and limp, and powders enveloped in it are swallowed without difficulty.

Directions for filling and closing.—In the apparatus shown in Figs. 181 to 184, the cachets are pressed with the fingers into the inner spaces of plates A and B (Fig. 182). The plate C serves to prevent the powder from finding its way between the edges of the cachets. It is laid on plate B and the powder placed in the cachets by means of the funnel, Fig. 182, and pressed down with a thimble. When all the cachets are filled, plate C is lifted up, and the damping roller, which must not be too wet, passed over the edges of the cachets in plate A. The latter, A, Fig. 183, is then closed over B, and a slight pressure closes all the cachets at once, and on opening the apparatus they are found adhering to plate A, complete and ready for sending out. The cachets are pressed out with the pusher, Fig. 184, on to a sheet of paper to allow the edges to dry and harden, before being touched with the fingers.

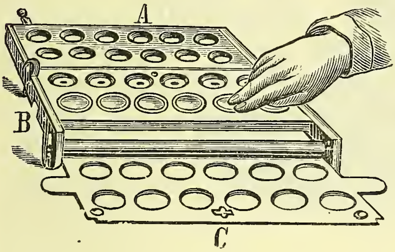


Fig. 181.*

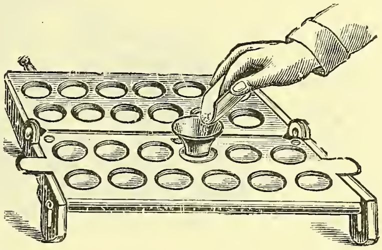


Fig. 182.

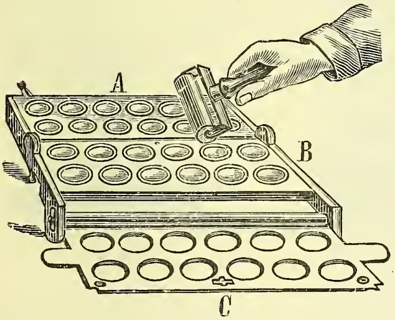


Fig. 183.

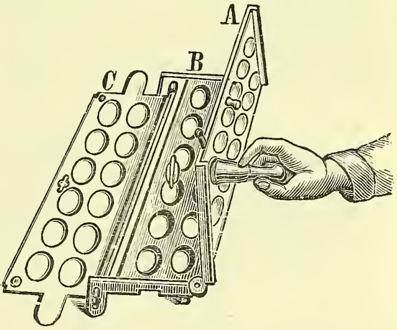


Fig. 184.

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* Figs. 181—184.—The Morstadt cachet apparatus.

Smaller forms of apparatus are worked on the same principle as the larger, the only difference being that only three cachets are closed at a time instead of twelve, Fig. 185.

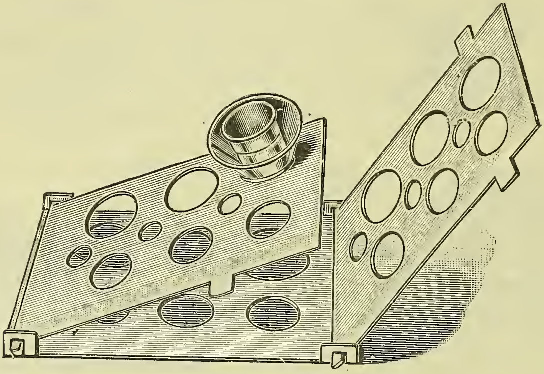

Fig. 185.

Lately an improved form of the Morstadt cachet has been introduced under the name of *Koseal.* In these, the shape of the rim has been altered on the inside by raising a portion, as will be seen in Fig. 186. The object of this is to simplify the process of moistening, as some difficulty is occasionally experienced in obtaining just the right degree of dampness when closing the cachets. If made too wet, ordinary cachets are liable to crinkle and look unsightly at the edges, and if insufficiently moistened they do not cohere. Figs. 187 and 188 show the finished "Koseal." If considerable pressure is used in closing these, their appearance differs in no way from ordinary cachets, but if only slight pressure is employed the two halves only cohere around the raised rim, the finished cachet appearing as the last figure.

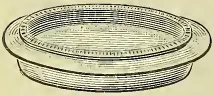

Fig. 186.

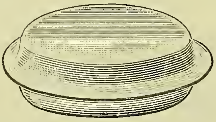

Fig. 187.

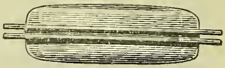

Fig. 188.

CHAPTER XXXII

CAPSULES

Gelatin capsules are now very largely used for the administration of nauseous drugs both in the solid and liquid form. Empty shells of soft gelatin, may be procured from the manufacturers ready for filling, or they may be prepared by dipping slightly oiled olive-shaped moulds as Fig. 189, fixed on a frame, into a solution of gelatin, allowing excess to drain off, and rotating slowly until the gelatin sets in an evenly distributed coat. When cold a knife is passed round the neck of the mould and the shells gently detached and placed in supports to dry.

Fig. 189.—Capsule mould.

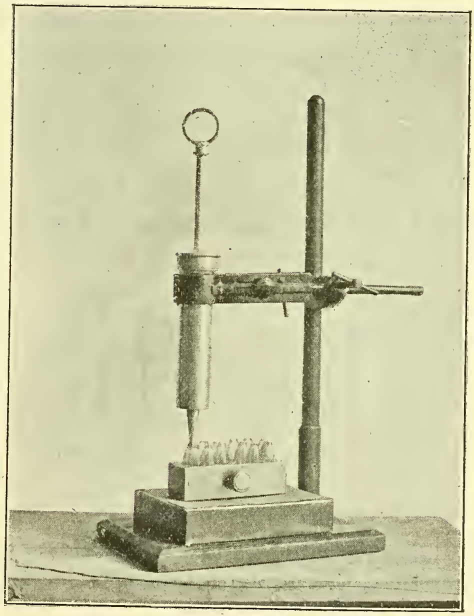

Fig. 190.

When ready for filling the capsules are placed in convenient supports (an ordinary suppository mould answers very well for small quantities) and the liquid introduced by means of a syringe with a long fine nozzle. If any oily fluid touches the neck of the shell it must be carefully wiped off before attempting to seal, or a leaky capsule will be the result. There are several methods in use for closing the capsule; the most convenient is to touch the mould with a camel-hair brush charged with thick gelatin solution. When cold the capsules may be picked up very gently and dipped in the ordinary gelatin solution to finish them off neatly.

It is found by experience that it is almost impossible to keep the hand perfectly steady when filling capsules, and a device similar to Fig. 190 is found useful. A rigid and heavy stand supports the syringe, the capsules being pushed under the nozzle one by one. It is essential that the piston of the syringe should fit the barrel accurately and yet sufficiently easily to move without jerking. The following is a good working formula for the gelatin solution (Forret's).

Gelatin	16 ounces.
Glycerin	12 ,,
Water	22 ,,
Syrup	8 ,,

Hard gelatin capsules (Fig. 191) are occasionally used for dispensing powders and pastes. Those used in this country are generally imported from America. They may be made if desired by dipping cylindrical iron moulds, one mould being slightly larger than the other, into a solution of gelatin made as follows:

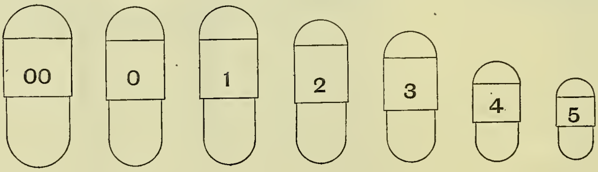

FIG. 191.

Gelatin	6 parts,
Gum acacia	1 part,
Sugar	1 ,,
Water	6 parts;

draining, cooling, and detaching as described for *soft* gelatin capsules. The shells are afterwards dried in a current of warm air until perfectly hard. To use, the smaller half is filled with the medicament and the cap slipped over the end. Various devices have been introduced for the expeditious and cleanly filling of these hard capsules. In no case should the capsule be filled by simply pressing into a heap of the powder. With bitter drugs such as quinine, no matter how carefully the capsule is afterwards wiped, the taste will be perceptible as soon as placed in the mouth.

The most convenient method is to place the empty shells in holes sunk in a block of wood, introducing the medicament by means of a small funnel and plunger (Fig. 192). With ordinary care it is quite possible to fill the capsules without getting even a trace on the outside. If desirable the inner edge of the cap can be painted with a little mucilage before slipping on, and the contents hermetically sealed.

There are several drugs and compounds the capsuling of which presents a little difficulty.

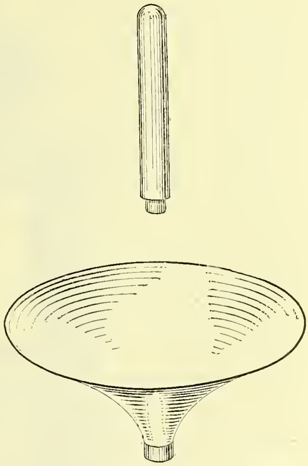
Fig. 192.—Capsule filler.

Creasote and guaiacol.—These are usually prescribed in 1 and 2 minim doses, but are always diluted with almond oil to at least 5 minims.

Fluid extract of cascara.—The ordinary dose in a capsule is 30 minims. If the extract is simply evaporated to a pasty consistence and then filled into soft capsules, the latter soon shrink and become unsightly. To avoid this a proportionate quantity of the dry extract should be reduced to fine powder and mixed with vaseline or liquid paraffin. Capsules filled with this mixture retain their plumpness for a long time.

Blaud's pill capsules.—Dry the alkali perfectly and mix with soft white paraffin. Powder the ferrous sulphate, dry at 212° F. (100° C.), and also mix with soft paraffin; then mix the two together and capsule as rapidly as possible.

Folding capsules consist of very thin sheets of gelatin, and may be used in a similar manner to wafer papers, for covering nauseous powders and confections. They are especially useful when the dose is too great for an ordinary capsule. The powder is placed in the centre of a sheet, the edges turned over and secured by moistening with water or mucilage. The gelatin sheets may be prepared by pouring a very thin film of gelatin solution (gelatin 25, water 60, glycerin 1) into a shallow tray, which is allowed to set and then detached and hung up to dry. The films should be cut into sheets 2½ inches wide and 3 inches long.

Medicinal perles.—This form of capsule can only be prepared on a manufacturing scale, the necessary apparatus being somewhat large and costly. The process consists in spreading sheets of a mixture of gelatin, gum, and sugar, with a machine in which the gelatin solution is kept warm in a jacketed funnel, through the nozzle of which it finds its way on to a perfectly level slab, which travels in grooves beneath an accurately adjusted knife edge, the solution being distributed in a thin film of the required area and thickness. The film, as soon as cold, is detached and partially dried, when it is ready for use. One of the films is then laid on a brass mould, consisting of a block of metal having a shallow rim, with a number of depressions corresponding to the size of perle required. The margin of each depression is also provided with a shallower projecting rim. The liquid medicament is then poured into the centre of the film, the edges being upheld by the outside rim of the mould, and another film laid on. A second mould, exactly similar to the first, is now placed over the films, and the whole placed in a powerful press (Fig. 193), when the liquid, being unable to escape owing to the closely fitting outside rims, forces the gelatin into the depressions, which are instantly filled. At the same time the perles are sealed and separated by the projecting edges of the individual depressions. After removal from the press the perles are washed with alcohol or other solvent to remove any traces of adhering medicament, and left to dry, the gelatin scrap being hung up to drain.

Lenticules.—This name was given by the author to a special class of gelatin capsule having the form of a double convex lens. The following processes, now published for the first time, enable the pharmacist to prepare them extemporaneously with little trouble.

Preparation of the film.—Take of—

Gelatin	25 parts.
Water	60 „
Glycerin	15 „

Allow the gelatin to soak in the distilled water until softened; apply a gentle heat until dissolved; add the glycerin and strain

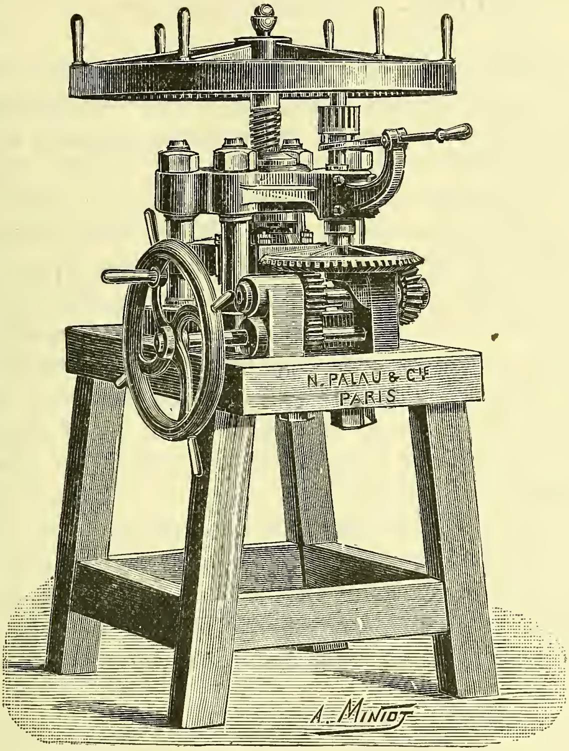

Fig. 193.—Perle press.

through a felt filter that has been previously wrung out in hot water. The product is then made up to weight with distilled water.

Next pour out sufficient of the melted gelatin to cover a shallow tray,—which may be conveniently 8 or 10 inches square and about one sixteenth of an inch deep,—slightly oiled. Allow the film to become perfectly cold, detach and place on a sheet of glazed paper. Now with a cork borer or small punch cut a number of circular discs, each half an inch in diameter, and an equal number of slightly smaller ones.

The mould may be economically made by drilling hemispherical holes of the required size, in brass or iron about one inch thick. It is essential that a metal mould be used, as it conducts the heat away rapidly during the sealing operation, otherwise there would be danger of the gelatin disc running. For discs half an inch in diameter the moulds should be $\frac{3}{8}$ of an inch, or rather less, across and $\frac{1}{4}$ inch deep. A lenticule this size will hold about 5 grains.

Filling.—One of the large-sized discs is placed over each of the depressions in the mould and pressed in with a rounded glass rod. The disc will be found to retain its concave form by its own elasticity, thus forming the lower half of the shell. It is now easy to introduce the medicament, whether solid or liquid, but care must be taken not to allow any to touch the edge of the gelatin, which should project slightly above the top of the mould. The next proceeding is to lay one of the smaller gelatin discs exactly over the lower half so that their edges correspond. The lenticules are now to be sealed by passing a very small gas flame round their edges. The best way of doing this is to connect the mouth-piece of an ordinary blow-pipe by means of rubber tubing to the gas supply. The flame, which should be about $\frac{1}{4}$ inch long, can then be regulated to a nicety, while the angle which the burner forms with the body of the blow-pipe is convenient for sealing the edges of the lenticule furthest away from the operator. It must be particularly noted that a blow-pipe is only made use of for its convenient shape; any other form of burner will do equally well if the flame is small enough and at right angles to the supply tube.

If a stock solution of gelatin is kept ready, a dozen or two lenticules can, with a little practice, be turned out in the same time as a box of pills; while having such wide mouths the shells are filled without any of the trouble experienced in filling the ordinary olive-shaped capsule.

There is another and simpler plan for enclosing solid medicaments in gelatin. Pour a film as directed for lenticules, about $\frac{1}{16}$ of an inch in thickness; when cold remove from the tray and set on one side. Now pour a second film and leave it in the tray. If the medicament is a powder it must be lightly pressed into buttons with any convenient apparatus, and the buttons or tablets placed at intervals of an inch or more, according to their size, on the surface of the film contained in the tray. When the tray is full lay the film that was poured first over the buttons, and having placed the tray on a block of metal or in cold water, to conduct the heat rapidly away, take a large Bunsen burner with a flexible tube and pass the colourless flame rapidly and evenly over the surface of the upper film. It will almost instantly melt, and if the operation has been successfully performed, sink down and completely envelop the tablets of medicament. The tray is now allowed to become perfectly cold, one end of the gelatin raised with a knife, and the whole carefully lifted out. The medicament is cut out of the films with a small lozenge punch; if the edges appear rough they may be smoothened by passing a small flame close to them in the manner described for sealing lenticules.

CHAPTER XXXIII

MEDICATED COTTONS AND GAUZES

Medicated cottons are prepared by impregnating absorbent material with solution of the medicinal agent, afterwards drying in a current of warm air. When the medicinal substance is soluble in water, a solution of definite strength is prepared, and the material allowed to soak in it until thoroughly saturated. It is then withdrawn from the solution and squeezed until only sufficient liquid is retained to correspond with a definite percentage of the active ingredient. The wet material is then laid on nets, and turned over from time to time to ensure even distribution of the liquid during the drying. Examples of this method of procedure are boric acid and sal alembroth. When the active ingredient is highly poisonous, as mercuric chloride, it is customary to colour the solution with methyl blue.

For the medication of cottons, &c., with iodoform, iodol, &c., solutions are prepared in a mixture of alcohol and methylated ether. The solutions are poured into the centre of the material, which is then quickly rolled up into a ball and submitted to hydraulic pressure for a few moments. It is then taken out of the press, shaken slightly, rolled up, and again pressed tightly. By this means a small quantity of liquid can be made to saturate a comparatively large bulk of absorbent material.

The following examples will serve as types of the different methods employed.

Cotton with sal alembroth.—Corresponds to 1 per cent. of mercuric chloride. Take of—

Sal alembroth	2·4	parts.
Soluble water blue	0·05	„
Distilled water	150	„
Absorbent cotton	100	„

Dissolve; soak the cotton in the solution until evenly wetted, and squeeze until it weighs 225 parts; dry. This is commonly known as "blue wool."

Cotton with boric acid, 10 *per cent.* Take of—

Boric acid	12 parts by weight.
Boiling water	138 „ „
Absorbent cotton	100 „ „

Dissolve; soak the cotton in the solution and squeeze until it weighs 225 parts; dry.

Cotton with iodoform, 10 *per cent.* Take of—

Iodoform	10 parts by weight.
Methylated ether, sp. gr. ·717 ...	70 „ „
Absorbent cotton	100 „ „

Distribute the solution evenly through the cotton, press quickly, and spread out to dry, away from any naked lights or fires, and in a dark place.

Medicated Gauzes.

The gauze employed should be of soft texture, and contain about sixty threads to the linear inch; it requires purification by some process similar to the following:—100 parts of gauze are soaked for several hours in warm water, then wrung out and boiled for several hours in water containing soap and common washing soda. The gauze is then well washed in clean water and soaked for several hours in dilute chlorinated lime solution; again washed, soaked in dilute hydrochloric acid, and washed in running water until every trace of chlorine or acid is removed; it is then dried.

To medicate the gauze, either an atomiser is used to spray the solution upon it, or the gauze is immersed in the solution and then dried.

Carbolic gauze, 10 *per cent.* Take of—

Castor oil	5 parts by weight.
Resin	48 „ „
Phenol	12 „ „
90 per cent. alcohol	85 „ „

Dissolve; in the solution impregnate 100 parts of purified gauze, and press until the weight is 225; dry.

Salicylic gauze, 10 *per cent.* Take of—

Salicylic acid	12 parts by weight.
90 per cent. alcohol	68 „ „
Distilled water	70 „ „

Dissolve; impregnate 100 parts by weight of purified gauze in the mixture, and press until the weight is 225; dry. If desired a gauze of the same type as carbolic can be prepared, using an equivalent quantity of the oil, resin, and alcoholic mixture in place of the mixture of alcohol and water.

Boric acid gauze, 10 *per cent.* Take of—

Boric acid	12 parts by weight.
Hot distilled water	138 " "
Gauze	100 " "

Press to obtain 225 parts, and dry.

Thymol gauze, 2 *per cent.* Take of—

Thymol	3 parts by weight.
Resin	5 " "
Spermaceti	50 " "
Alcohol (90 per cent.)	50 " "
Gauze	100 " "

Moisten the gauze with the solution, place in a hot press, squeeze two or three times to evenly distribute the medicament, and dry.

Iodised gauze, 10 *per cent.* Take of—

Purified gauze	90 parts by weight.

Place in a wide-mouthed stoppered bottle, heat to 100° C. Then take of—

Iodine	10 parts by weight.

Wrap in blotting-paper, and drop in the jar; heat until the gauze is uniformly impregnated.

Iodoform gauze, 10 *per cent.* Take of—

Iodoform	10 parts by weight.
Methylated ether	70 " "
Gauze	100 " "

Dissolve the iodoform in the ether, moisten the gauze, wrap in paper, press quickly, and dry in the dark, away from lights or fires.

CHAPTER XXXIV

ZINC GELATINS

The class of preparations to which the above name has come to be commonly applied, was originally introduced by Unna for the cleanly application of medicaments in certain skin diseases.

Zinc gelatin (*plain*). Take of—

Resorcin	½ part.
Gelatin	10 parts.
Glycerin	15 „
Zinc oxide	10 „
Distilled water	30 „

Allow the gelatin to soak in the water until softened, add the resorcin, dissolve by aid of a water-bath, and strain; sift the zinc oxide through a muslin sieve on to the glycerin contained in a mortar, and triturate until perfectly smooth; incorporate with the gelatin solution, continuing the heat until perfectly fluid; add sufficient distilled water to make up for the loss by evaporation, and pour into a level mould 1 inch deep. When perfectly cold, divide into 1-inch cubes by means of ruler and a sharp knife.

A mould for zinc gelatins and similar preparations is conveniently made as follows:—A marble slab with a smooth surface is laid perfectly flat on a level counter. Two metal bars, each 7½ inches long and 1½ inches thick, and two other bars of the same thickness, but 20 inches long, are procured, and arranged on the slab with the ends touching, so as to form a rectangular figure. Any sized mould can now be constructed by simply moving the end bars. If desired the bars may be made of mahogany, but they require weighting to withstand the internal pressure of the liquefied mass. The bars and the slab should be slightly oiled to prevent adhesion of the zinc gelatin.

The following are given as examples of different methods of procedure.

Zinc gelatin with extract of Indian hemp, 2 *per cent.* Take of—

Plain zinc gelatin	98 parts.
Extract of Indian hemp	2 „

Place the extract in a warm mortar and add a little of the previously melted zinc gelatin ; triturate until a perfectly smooth mixture results ; add to the remainder of the zinc gelatin, mix evenly, and strain though muslin into the mould. When cold, divide into 1-inch cubes.

Zinc gelatin with Stockholm tar, 5 *per cent.* Take of—

Stockholm tar (strained)	5 parts.
Plain zinc gelatin	95 „

Proceed as directed for extract of Indian hemp.

Zinc gelatin with iodoform, 5 *per cent.* Take of—

Precipitated iodoform	5 parts.
Zinc gelatin	95 „

Proceed as directed for Extract of Indian hemp.

Zinc gelatin with salicylic acid, 2½ *per cent.* Take of—

Salicylic acid (in very fine powder) ...	2½ parts.
Plain zinc gelatin	97½ „

Proceed as directed for extract of Indian hemp, but great care must be taken during the operation not to heat the mixture more than necessary, or it will not solidify. The use of an iron spatula is to be avoided; also in this case a wooden mould becomes necessary.

Zinc gelatin with ichthyol, 5 *per cent.* Take of—

Ichthyol-ammonia	5 parts.
Plain zinc gelatin	95 „

Melt the zinc gelatin over a water-bath, add the ichthyol, and stir until uniformly mixed; then proceed as directed above.

CHAPTER XXXV

CONFECTIONS

Confections or electuaries are very ancient forms for the administration of medicines, and are made by simply bringing the solid ingredients to a smooth pasty condition by the addition of syrup or honey.

Confection of pepper is known as Ward's paste. Confection of roses is possibly retained for sentimental reasons. Confection of senna (lenitive-electuary) requires great care in manufacture to produce an even product. The ingredients after boiling should be first pulped through a 20-hole sieve to remove the seeds, skins, and fibrous material. While still warm the thin pasty mass should be pulped through a 60-hole sieve, and mixed with the extract of liquorice and 20 ounces of the sugar. By careful evaporation the whole should then be brought to exactly 56 ounces by weight, and allowed to cool. When almost cold the powdered senna and coriander, previously mixed with the remainder of the sugar, is sifted in through a 60-hole sieve and intimately mixed. If the last ingredients are added to the pasty mixture while hot, it will form into little balls, which can only be broken up and evenly distributed, by passing the whole of the confection again through a fine sieve.

The pulping is accomplished by placing a sieve over a wide-mouthed jar or mortar, and forcibly pressing the softer parts of the well-boiled fruits through, by rubbing with the bottom of a gallipot. The operation is somewhat tedious, and cannot be hurried without detriment to the product.

CHAPTER XXXVI

GRANULAR EFFERVESCENT PREPARATIONS

Granular effervescent salts afford a palatable and pleasant form for the administration of many medicinal substances. Although the Pharmacopœia only recognises seven preparations of this class, so many different combinations are in constant demand that the pharmacist should be in a position to manufacture them for himself, particularly as no expensive apparatus is required for their production. Different methods of granulation are employed according to the nature of the medicinal substance, but all require the ingredients in a state of fine powder. Chemical compounds such as sodium sulphate, containing much water of crystallisation, must be desiccated before granulation is attempted, otherwise fusion of the mixed salts would result in loss of carbon dioxide. A working basis for effervescent granules should contain approximately—

Tartaric acid	28 per cent.
Citric acid	19 „
Sodium bicarbonate	53 „

The general rule when experimenting with new formulæ is to calculate for drachm doses, and to add sufficient basis to each dose of the medicament as will bring the total weight up to 70 grains. The loss in granulation is from 10 to 15 per cent. of the weight of basis operated on. If, for example, granular effervescent citrate of caffeine, 2 grains in a teaspoonful, is required, 68 grains of basis should be mixed with 2 grains of caffeine citrate. The basis must be freshly mixed for each operation.

When the medicinal ingredients are sufficiently stable, the mixture of salts may be granulated according to the pharmacopœial process, the ingredients "being heated together until a temperature between 200° F. and 220° F. is reached, stirring assiduously all the while, so as to form granules." Substances likely to be injured by heat, such as pepsin, antipyrin, piperazin, &c.,

should be granulated in the cold. This is best accomplished by mixing the acids and sodium bicarbonate in a mortar with a large flat pestle, using sufficient pressure to cause the powders to become somewhat pasty. The medicament is next incorporated, and the mixture left until sufficiently soft to be pressed through a No. 6 or No. 8 wire sieve, when it is dried at a low temperature. If dried in a mass it requires to be broken up in a mortar, the fine powder being sifted from the granules by a 15- or 20-hole sieve. It is important that the sieves used in granulating operations be thoroughly well tinned.

Alcohol has been suggested, and, indeed, is directed by the United States Pharmacopœia, to be used for moistening the dry powders before granulating through a sieve, but its use hardly recommends itself to the practical pharmacist. The operation is rendered rather more expeditious, but at the same time more costly, as at least 95 per cent. alcohol must be employed while the resulting granules are more friable than those obtained by either of the processes described.

The use of flavouring agents, such as vanilla or lemon, is deprecated, as if the preparations are kept for any length of time a very disagreeable odour and taste is developed.

CHAPTER XXXVII

SCALE PREPARATIONS

Of late years a large number of scale compounds containing iron have been introduced into pharmacy, but as they are all prepared on similar lines, it will be sufficient to describe the three official ones.

Ferri et Ammonii Citras.—Ferric hydroxide is first prepared by pouring solution of ferric sulphate into dilute ammonia, and washing the precipitate until free from sulphates.* After well draining, the magma is squeezed as dry as possible and added to strong solution of citric acid until, after warming to about 120° F. (49° C.), no more iron is taken up. This gives solution of ferric citrate. On the addition of ammonia, double decomposition takes place, according to the equation—

$$FeC_6H_5O_7 + 3NH_4OH = Fe(OH)_3 + (NH_4)_3C_6H_5O_7.$$

Ferric hydroxide, although formed, is not precipitated, but is held in solution by the ammonium citrate, just in the same way as cupric hydrate is held in solution by tartarated soda in Fehling's solution.

The resulting clear mixture is then concentrated to a syrupy consistence, spread in a thin layer on sheets of glass, and allowed to dry in an atmosphere free from dust. In establishments where very large quantities of scale preparations are turned out, the moist air is constantly drawn out of the drying-room by a ventilating fan, warm air that has filtered through cotton wool being admitted to take its place.

Ferri et Quininæ Citras.—In the first stage ferric hydroxide is prepared as in the foregoing, and washed free from sulphate.

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* The washing of such precipitates is best accomplished by stirring up with a large volume of distilled water, allowing to deposit, and decanting by means of a syphon, the process being repeated three or four times.

Quinine alkaloid is next precipitated from the sulphate by ammonia, and also washed until free from sulphate. The squeezed ferric hydroxide is then dissolved in solution of citric acid, the quinine added, and solution of ammonia poured in, a little at a time, so that the precipitate of quinine which at first forms, redissolves in excess of the reagent. It is then filtered and scaled in the usual way.

Ferrum Tartaratum.—This compound differs somewhat from the preparations just described. Ferric hydroxide is precipitated and added to hydrogen-potassium tartrate, when probably the following reaction takes place:

$$KHC_4H_4O_6 + Fe(OH)_3 = K(FeO)C_4H_4O_6 + 2H_2O.$$

The mixture is then warmed and extracted with water, and the filtered solution concentrated and scaled as previously described. Ferrum Tartaratum is extremely rich in iron, containing about 30 per cent. of ferric oxide. It is slowly but perfectly soluble in water. The solubility is greatly increased by the addition of a little ammonia before concentrating.

CHAPTER XXXVIII

ESSENTIAL OILS

All essential oils either pre-exist in plants, or are formed under the action of ferments by the splitting up of resins, glucosides, and similar bodies. The essential oils which occur naturally are sometimes found in distinct cells, as in the glands of the orange and lemon, in tubes, as in the vittæ of umbellifers, or in deep-seated tissue, as in cinnamon bark. In other instances they are found associated with resins, as oleo-resin of copaiba, or with gum-resins, as ammoniacum. Essential oils obtained from different parts of the same plant frequently differ not only in their physical, but their chemical properties,—as, for example, oil of orange flowers and oil from the ripe fruits. Unlike fixed oils, essential oils leave a harsh feeling when rubbed on the fingers; they are all inflammable, burning with a white flame with the production of much carbon.

The greater number of essential oils are obtained by distilling the parts of the plant yielding them with steam; although their boiling-points may be higher than that of water, the oils are carried over in a state of minute division with the aqueous vapour. In this way are obtained the essential oils of chamomile, cloves, cinnamon, cassia, cubebs, eucalyptus, lavender, peppermint, &c., and all the oils obtained from umbelliferous fruits. The oils from the rinds of the fruits of the species *citrus*, as lemon, bergamot, are obtained by expression. The odorous principles of most flowers are so sparingly distributed and of so delicate a character that they cannot be prepared by either distillation or expression. The usual method employed for their extraction consists in placing the flowers between sheets of cotton wool soaked in an odourless hydrocarbon, from which the perfume may be recovered by macerating with purified alcohol (enfleurage). See also Distillation of Volatile Oils and Medicated Waters.

The essential oils of almonds and mustard, differ from the foregoing in that they do not exist ready formed in the plant, but are produced by the reaction of certain principles in the presence of water. When the cake left after the expression of the fixed oil from bitter almonds is macerated with cold water, fermentation sets in; this is caused by a ferment called emulsin, which, in the presence of water, decomposes the amygdalin, a glucosidal body, with formation of benzaldehyde (*oil of bitter almonds*), hydrocyanic acid, and glucose. When reaction is complete the mixture is distilled, and the essential oil collected and dried. On exposure to air benzaldehyde readily undergoes oxidation, yielding benzoic acid:

$$C_6H_5.CHO + O = C_6H_5.COOH.$$

Large quantities of benzaldehyde are now prepared by heating benzal chloride with dilute sulphuric acid under pressure.

Volatile oil of mustard is prepared by macerating the cake left after pressing black mustard seeds, with water. Usually a little white mustard cake is added to supply any possible deficiency of myrosin. The black seeds contain a glucosidal compound, potassium myronate, soluble in water, which in the presence of a ferment, myrosin, gradually undergoes fermentation, allyl isothiocyanate (mustard oil), glucose, and potassium hydrogen sulphate being formed. The products of decomposition are then distilled in steam, and the oil collected and dried.

All volatile oils oxidise in contact with air. They should, therefore, be preserved in well-stoppered bottles away from the light. When essential oils are required to be mixed with spirit, as in the preparation of perfumes, the official spirits, &c., such of them as are soluble should be mixed with an equal bulk of alcohol, it having been found by experience that not only is oxidation retarded, but in most cases the oil actually improves in odour by the treatment, especially on keeping.

In the examination and valuation of essential oils, much may be learned by a careful systematic study of such physical data as are derived from determinations of gravity, rotation, and fractionation; but in such oils as those of lavender and cinnamon, which contain definite odorous principles, it becomes necessary to estimate the percentages of such bodies. Exact processes are generally applicable for the estimation of the following:

Aldehydes.—Citral, cinnamic aldehyde.
Esters.—Bornyl acetate, and other esters of borneol.
Linalyl acetate, and other esters of linalool.
Geranyl acetate, and other esters of geraniol.

Phenols.—Thymol, eugenol, carvacrol.

Alcohols.—Borneol, linalool, geraniol, rose alcohols, santalol.

Estimation of Aldehydes and Ketones

The following process—Schimmel's—is only applicable to those aldehydes and ketones which form with concentrated bisulphite of sodium solution, crystalline compounds soluble in boiling water. Such are cinnamic and benzoic aldehydes, and the ketone carvol contained in the oils of dill, caraway, and green mint.

Necessary apparatus.—1. A glass flask holding about 70 c.c., the neck graduated in c.c. subdivided into tenths. These are known on the market as Hirschsohn's flasks.

2. Solution of sodium bisulphite, 30 per cent.

3. A 10-c.c. pipette.

About 40 c.c. of the bisulphite solution is brought to the boiling-point in the flask, 10 c.c. of the oil immediately added, and thoroughly agitated for half an hour. Any aldehydes or ketones present, combine with the bisulphite to form definite compounds, which dissolve in the hot aqueous solution. The reaction that takes place, is analogous to the behaviour of ordinary aldehyde with sodium bisulphite solution :

$$CH_3.CHO + NaHSO_3 = CH_3.CHO.NaHSO_3\,;$$

or with ordinary acetone—

$$CH_3.CO.CH_3 + NaHSO_3 = CH_3.CO.CH_3.NaHSO_3.$$

As a rule the bisulphite compound completely dissolves, the non-aldehydes forming a clear oily layer on the surface, but to ensure the thorough fixing of the aldehydes the flask should be immersed in a water-bath for a couple of hours or so. The flask should then be allowed to cool, and be filled up with more bisulphite solution, so that the non-aldehydes may be measured in the graduated neck of the flask. The figure obtained multiplied by ten gives the percentage of non-aldehydes, which, again, deducted from 100 indicates the percentage of aldehyde or ketone.

Estimation of Esters (Ethereal Salts)

All ethereal salts undergo hydrolysis (saponification) when they are heated with alkalies ; thus ethyl acetate warmed with solution of potassium hydroxide yields potassium acetate and ethyl alcohol:

$$CH_3.COOC_2H_5 + KOH = CH_3.COOK + C_2H_5.OH.$$

It follows then, that the esters in essential oils may be estimated by saponifying them with a definite volume of alcoholic potash, titrating back the uncombined alkali with sulphuric acid. The only reason for using an alcoholic solution of the alkali, is the insolubility of most ethereal salts in water, and consequent slowness with which the reaction would proceed if aqueous alkali were used.

Take a definite weight of the oil (about 2 grams), mix with 10 c.c. $\frac{N}{2}$KOH (alcoholic), and boil for thirty minutes under a reflux condenser, cool, and titrate the excess of alkali with $\frac{N}{2}$ H_2SO_4, using phenol-phthalein as an indicator. To hasten hydrolysis, the flask should be closed by a cork through which a pressure tube, containing an inch of mercury, is passed.

Example.—Supposing 2 grams of French lavender oil is taken for examination, and it is found that 3 c.c. of $\frac{N}{2}H_2SO_4$ are required to neutralise the excess of alkali. This indicates that 7 c.c. of $\frac{N}{2}$KOH have entered into combination. The principal ester present is linalyl acetate, and the reaction is expressed by the following equation:

$$\underbrace{C_{10}H_{17}O.CH_3.CO}_{\begin{array}{r}2)\underline{194{\cdot}68}\\97{\cdot}34\end{array}} + \underbrace{KOH}_{\begin{array}{r}2)\underline{55{\cdot}71}\\27{\cdot}85\end{array}} = C_{10}H_{17}OH + CH_3.COOK.$$

That is, 1000 c.c. $\frac{N}{2}$KOH = 97·34 grams linalyl acetate.

1 c.c. ,, = ·09734 gram ,,

Therefore 7 c.c. ,, = ·68138 ,, ,,

As ·68138 gram of linalyl acetate are contained in 2 grams, this weight multiplied by 50 gives the percentage (·68138 × 50 = 34·06 per cent.).

Note.—The above equation is applicable in calculating the amount of linalyl acetate in bergamot and lavender, as well as its isomer bornyl acetate in oil of pine.

Estimation of Alcoholic Bodies

In the estimation of alcohols, such as menthol in oil of peppermint, and borneol in oil of rosemary, advantage is taken of the fact that these bodies when boiled with acetic anhydride yield an ethereal salt and acetic acid. The ethereal salt is then saponified and determined in the usual way, when the percentage of alcohols present is obtained by calculation.

Process.—10 grams of the essential oil (peppermint, for instance) is boiled for two hours with 10 c.c. of acetic anhydride and 1 gram of dried sodium acetate, in a flask fitted with a reflux condenser. The reaction that takes place is expressed by the following equation :

$$C_{10}H_{19}.OH + (CH_3.CO)_2O = C_{10}H_{19}O.CH_3.CO + CH_3.COOH.$$

The condenser is next rinsed with a little water, and the whole contents of the flask washed into a stoppered glass separator, and shaken up with water until the washings run free from acidity. The purified menthyl acetate is then saponified with alcoholic potash, as described in Estimation of Esters. Now supposing it is found after 50 c.c. $\frac{N}{2}$KOH have been added, and the reaction is complete, that 14 c.c. $\frac{N}{2}H_2SO_4$ are required to neutralise the excess of alkali, it would indicate that 36 c.c. $\frac{N}{2}$ KOH had been decomposed. The following equation is then applied :

$$\underbrace{C_{10}H_{19}.O.CH_3.CO}_{\begin{array}{r}2)\underline{198{\cdot}68}\\ 99{\cdot}34\end{array}} + \underbrace{KOH}_{\begin{array}{r}2)\underline{55{\cdot}71}\\ 27{\cdot}85\end{array}} = \underbrace{C_{10}H_{19}.OH}_{154{\cdot}98} + CH_3.COOK.$$

That is, 1000 c.c. $\frac{N}{2}$KOH $= 99{\cdot}34$ grams of menthyl acetate.

1 c.c. ,, $=$ ·09934 gram ,, ,,

Therefore 36 c.c. ,, $\doteq$ 3·57 grams ,, ,,

As 3·57 grams of ester are obtained from 10 grams of oil, this weight multiplied by 10 gives the percentage ($3{\cdot}57 \times 10 = 35{\cdot}7$ per cent.).

Now it will be seen from the equation above that the relation between menthyl acetate and menthyl alcohol is as 198·68 is to 154·98, or as 1 is to ·78 ($154{\cdot}98 \div 198{\cdot}68 = {\cdot}78$). If, therefore, the percentage of ester is multiplied by ·78 it will give the percentage of alcohol (35·7 × ·78 = 27·8 per cent.).

Estimation of Phenols

If *exact* results are required, Thom's method must be followed. Briefly this consists in heating benzoyl chloride with a mixture of alkali and essential oil (oil of cloves, for instance). On cooling benzoyl-eugenol separates out, which after purification is weighed and calculated to eugenol.

A much more rapid and simple process, consists in treating a definite volume of oil, in a flask having an elongated graduated neck, with 10 per cent. aqueous solution of caustic potash, warming and allowing the uncombined oil to rise to the surface, where it is measured at a temperature of 15° C. The difference between the reading and the amount of oil taken, is the approximate amount of eugenol present. The process is applicable for the estimation of eugenol in oil of cloves, cinnamon, and cassia.

Estimation of Allyl Isothiocyanate

Shake a mixture of 3 grams of the oil and 3 grams of alcohol with 6 grams of solution of ammonia, 10 per cent., and heat to 50° C. The mixture will first become clear, and then deposit crystal of thiosinamine. Decant the mother liquor from the crystals, and evaporate to dryness in a tared dish over a water-bath. Then add the crystals to those in the dish, rinsing out the last portion with alcohol, and continue the heat until the weight remains constant. The amount of thiosinamine thus obtained from 3 grams of oil should not fall below 3·25 grams, corresponding to 92 per cent. by weight of allyl isothiocyanate :

$$C_3H_5NCS + NH_3 = CS\left\langle\begin{matrix}NH.C_3H_5\\NH_2.\end{matrix}\right.$$

Oleum Anethi

Source.—Distilled from dill fruits (*Anethum graveolens*), cultivated in England, Russia, Germany and Roumania, the yield of oil varying from 2 to 4 per cent.

Characters.—An almost colourless liquid, having an odour strongly resembling that of caraway. *Specific gravity* at 15° C. between ·905 and ·920. A lower specific gravity indicates either adulteration with turpentine or partial decarvolisation. The oil distilled from Indian fruits has a higher specific gravity, usually above ·940. *Optical rotation* should not fall below + 70°.

Fractionation gives a clear indication of the relative proportions present, of low boiling terpenes and carvol. Not more than 15 per cent. should distil below 185° C., and not less than 40 per cent. above 220° C. An oil distilled by J. C. Umney from fruits cultivated in Lincolnshire, gave 50 per cent. boiling between 220° C. and 230° C.

The oil distilled from Indian fruits possesses somewhat different characters, and the presence of a body having a higher density than water, and characteristics similar to apiol, has been established.

Constituents.—Normally this oil has the same constituents as caraway oil, though it contains practically a smaller percentage of carvol; its aroma is, however, somewhat modified by the presence of traces of other bodies.

Oleum Anisi

Source.—Distilled in China from the fruit of the Star-anise (*Illicium verum*), and in Europe from the fruits of *Pimpinella anisum*. Anise is largely cultivated in Italy, Prussia, Russia, and Spain, but by far the larger proportion of oil used in this country is imported from China.

Characters.—Colourless or pale yellow, with the characteristic odour of the fruit. *Specific gravity* at 15° C. ·975 to ·990, the latter figure indicating a high percentage of anethol. *Optical rotation* from − 1° to − 3° at 20° C. It congeals when stirred, between 10° and 15° C.

Constituents.—From 80 to 90 per cent. of anethol, with small quantities of methyl-chavicol and anisic aldehyde.

Anethol, to which the odour and sweet taste of anise oil are due, has a specific gravity of ·985 at 25° C. It boils constantly at 234° C., and melts after solidification, between 21° and 22° C.

Anisic aldehyde has a fragrance resembling hawthorn, and has been used in perfumery under the name of "Aubepine."

Note.—A saturated solution of hydrochloric acid gas in absolute alcohol affords a blue coloration with anise oil, and a yellow with that of star-anise.

Oleum Anthemidis

Source.—Distilled from the flowers of *Anthemis nobilis*, which are largely grown in Belgium, France, and in England (Mitcham).

Characters.—Pale blue or greenish blue when freshly distilled, but gradually becoming yellowish brown. *Specific gravity* at 15° C. from ·905 to ·912. *Optical rotation* from + 1° to + 3°.

Fractionation.—The oil distils between 170° and 220° C., the bluish coloured oil distilling with the higher boiling fractions.

Constituents.—The odour of the oil is due to the presence of angelic and tiglic esters of isobutyl, amyl, and hexyl alcohols.

Oleum Cajuputi

Source.—Distilled from the leaves of *Melaleuca leucadendron.*

Characters.—Bluish green, having an agreeable, penetrating, camphoraceous odour and taste. The green colour is due to copper, and may be removed by shaking with solution of potassium ferrocyanide. *Specific gravity* from ·922 to ·930. *Optical rotation* not exceeding − 5°.

Constituents.—From 50 to 70 per cent. of cineol, which may be estimated by phosphoric or arsenic acid. (See page 260.)

Oleum Carui

Source.—Distilled from caraway fruits (*Carum carui*). The fruits cultivated in different parts show considerable variation in the yield of oil, as well as in the relative proportion of their constituents. Oil distilled from Dutch seeds is the most esteemed, and contains the highest percentage of carvol.

Characters.—Colourless or pale yellow, having the characteristic odour and spicy taste of the fruits. The *specific gravity* of oil distilled from Dutch seeds varies from ·910 to ·920 at 15·5° C., a lower specific gravity frequently indicating abstraction of carvol. *Optical rotation* from + 75° to + 85°.

Fractionation.—The oil distils almost completely between 180° and 230° C. At least 55 per cent. should distil above 200° C. in the finest oils. The boiling-point of carvol is 224° to 225° C.

Constituents.—*Limonene* (carvene), a terpene, almost odourless when pure, having a specific gravity of ·846 at 15° C., and boiling-point 175° C., and put on the market as oil of caraway-chaff.

Carvol, the odorous constituent of the oil, has the following characters :—Pale or colourless ; *specific gravity* at 20° C. ·96058 ; *optical rotation* + 60°.

Oleum Caryophylli

Source.—Distilled from the flower buds of *Eugenia caryophyllata.*

Characters.—Colourless or pale yellow when fresh, but gradually becoming reddish brown, having the strong odour and taste of cloves. *Specific gravity* at 15·5° C. should not fall below 1·05. The following gravities have been found for oils distilled from fruits and stems in different conditions :

Oil from green cloves	1·0648 at 15° C.
„ ripe „	1·0629 „
„ dried „	1·0569 „
„ green stems	1·0608 „
„ dried „	1·0593 „

Constituents.—Caryophyllene, a sesquiterpene, specific gravity at 15° C. ·903 ; the phenol eugenol ($C_{10}H_{12}O_2$), however, constitutes about 80 per cent. of clove oil. Eugenol has a strong aromatic odour, and boils at 247° C. Ferric chloride gives a blue colour to its alcoholic solution. It has all the properties of a phenol, and when oxidised with potassium permanganate yields vanillin. In addition to caryophyllene and eugenol, Schimmels' have recently demonstrated the presence of methylic alcohol and furfurol.

Oleum Cinnamomi

Source.—Distilled from the bark of *Cinnamomum zeylanicum,* but very often adulterated with oil distilled from the leaves.

Characters.—Pale yellow when freshly distilled, but gradually becoming reddish, having the sweet aromatic odour and taste of the bark. *Specific gravity* at 15° C. from 1·025 to 1·035. The addition of leaf oil (specific gravity about 1·055) materially raises this figure. *Optical rotation* inactive or very slightly lævogyre.

Constituents.—Phellandrene ; eugenol in small proportion, generally less than 8 per cent. As leaf oil contains from 80 to 90 per cent., its presence in the official oil may be suspected if the eugenol exceeds 10 per cent. Cinnamic aldehyde (see Cassia oil) ; various cinnamyl esters.

Typical cinnamon oil should contain at least 55 per cent. of cinnamic aldehyde ; 1 c.c. of oil dissolved in 5 c.c. of 90 per cent.

alcohol should only afford a *pale* green coloration with solution of ferric chloride, thus limiting the eugenol.

Oleum Copaibæ

Source.—Distilled from copaiba. That met with in commerce is chiefly derived from Para oleo-resin, which yields from 65 to 80 per cent. of volatile oil. The Maranham and Maracaibo varieties yield from 35 to 40 per cent. (Umney).

Characters.—Colourless or pale yellow, with the odour and taste of copaiba. *Specific gravity* at 15° C. from ·900 to 910, increasing with age. *Optical rotation* lævogyre. Readily soluble in an equal volume of absolute alcohol (distinction from African oil).

Constituent.—Caryophyllene.

Oleum Coriandri

Source.—Distilled from coriander fruits.

Characters.—Colourless or pale yellow, having the odour and flavour of the fruit. *Specific gravity* at 15° C. from ·870 to ·885. Adulteration with oil of orange or turpentine reduces these figures considerably. It is readily soluble in three times its volume of 70 per·cent. alcohol (absence of turpentine or added terpenes).

Fractionation.—The principal fraction is that boiling between 190° and 200° C. This fraction contains the coriandrol (boiling-point 194° to 198° C.). Pure oils yield about 50 per cent. distilling between these temperatures.

Constituents.—Pinene, a terpene boiling about 154° C. This terpene forms the chief constituent of ordinary oil of turpentine, about 5 per cent. being present in coriander oil. Coriandrol, an alcohol isomeric with linalool, and readily estimated by the acetylisation process.

Oleum Cubebæ

Source.—Distilled from cubebs (*Piper cubeba*).

Characters.—Colourless, pale green, or greenish yellow, with the odour and camphoraceous taste of the fruits. *Specific gravity* at 15° C. from ·910 to ·930. *Optical rotation* from − 30° to − 40°.

Fractionation.—In the higher fractions the presence of two sesquiterpenes is indicated, the hydrate of one being known as cubeb-camphor.

Constituents.—Dipentene, cadinene, cubeb-camphor.

Oleum Eucalypti

Source.—Distilled from the fresh leaves of *Eucalyptus globulus* and other species of *Eucalyptus*. The general description of the source of the oil in the British Pharmacopœia for 1885 has been the means of introducing into British pharmacy a great variety of oils, differing very markedly in physical and chemical characters.

The oils principally met with in commerce during the last few years have been the *globulus* oils of Australian, Tasmanian, French, and Spanish origin, and *dumosa, oleosa, amygdalina,* and others distilled in Australia. Amygdalina oil is but little used, owing to its poorness, and is now excluded by the wording of the tests of the B.P. and U.S.P.

Characters.—Colourless or pale yellow, having an aromatic camphoraceous odour and pungent taste, leaving a sensation of coldness in the mouth. *Specific gravity* from ·910 to ·930. *Optical rotation* should not vary more than 10° in either direction. Oil distilled from *E. amygdalina* frequently has a lævogyre direction of − 40° or higher.

Fractionation.—It distils chiefly between 170° and 190° C.

Constituents.—Eucalyptol, or more properly cineol; phellandrene, a terpene boiling about 170° C., found also in fennel oil; cumin aldehyde in oil distilled from *E. oleosa.*

Cineol, which should be present to about 40 per cent., is frequently found associated with terpenes in essential oils; it is a colourless liquid at 15° C., when it has a sp. gr. of about ·926; it is optically inactive, and boils about 176° C. Below the freezing-point of water it solidifies to a crystalline mass. Cineol forms crystalline compounds with several acids, the principal one being that with syrupy phosphoric acid, sp. gr. 1·75. The Pharmacopœia requires that oil of eucalyptus should become semi-solid, on stirring with one half or one third its volume of this acid.

If 1 c.c. of eucalyptus oil be mixed with 2 c.c. of glacial acetic acid and 2 c.c. of a saturated aqueous solution of sodium nitrite, crystalline phellandrene nitrite should not be precipitated (absence of *amygdalina* oil).

Oleum Juniperi

Source.—Distilled from the full-grown, but unripe fruits of *Juniperus communis.*

Characters.—Colourless or pale greenish yellow, with the characteristic odour of the fruit, and a warm, aromatic, bitterish taste. *Specific gravity* from ·865 to ·890. The specific gravity of the oil increases rapidly with age, though without material change in colour. Oil distilled from *ripe* fruit has been found to possess as low a sp. gr. as ·840.

Constituents.—These are chiefly terpenes, although the fragrance is attributed to small quantities of esters. On distilling, the largest portions pass over between 155° and 175° C., corresponding respectively to pinene and the sesquiterpene cadinene.

Oil of juniper is sometimes adulterated with oil of juniper wood and oil of turpentine. Both these yield crystalline compounds with hydrochloric acid. If true oil of juniper is exposed in a thin layer to the atmosphere it is only slowly thickened, and scarcely darkened in colour; while oil of juniper wood thickens rapidly, and becomes a dark-coloured resin. Oil of juniper fruit forms a perfectly clear solution with its own volume of 95 per cent. alcohol.

Oleum Lavandulæ

Source.—Distilled from the flowers of *Lavandula vera*, cultivated in various parts of England and the south of France.

Characters.—The difference between the oils produced in different districts is very marked. The English oil is pungent and camphoraceous, whilst the French oil has a flatter and in some respects sweeter odour, these differences being directly dependent on variations in composition which will be referred to later. The oil should be colourless or pale yellow. *Specific gravity* not below ·885, being slightly higher in English than in French oils. The admixture of the latter with spike oil raises the sp. gr. considerably (see Spike oil). *Optical rotation* from −4° to −10°. Admixture with spike oil reduces the rotation, as it is generally slightly dextrogyre. The oil should form a perfectly clear solution with three times its volume of 70 per cent. alcohol.

Constituents.—The principal point of difference between the English and French oils is the variation of ester and cineol percentages. The finest French oils contain from 30 to 40 per cent. of esters (principally linalyl acetate), and the estimation of the ester content is the most useful means of comparing this class of lavender oils, and ensuring freedom from oil of spike. Spike oils contain practically no esters, and reduce the ester percentage proportionately to their presence.

English oils only contain from 7 to 10 per cent. of esters (also principally linalyl acetate), and attempts to discredit the English oil on this account have been frequently made on the Continent.

Cineol (see Oil of eucalyptus) is also present in very considerable quantity in English oils, and gives to it its characteristic pungency; mere traces are present in the French oils. Other differences in constituents and their proportions doubtless exist, which may help to make up for the great variability in odour.

Oleum Limonis

Source.—Obtained by expression from the fresh peel of the fruit of *Citrus limomum* in Sicily, the two centres for export being Messina and Palermo.

Characters.—Pale yellow, with the fragrant odour of the lemon and a warm, bitterish taste. The odour of the Messina oil is slightly different from that of the Palermo variety; and whilst the latter is preferred in this country, on account of its fine flavour, for aërated water manufacture, the former is principally employed on the Continent, where it is used in perfumery. *Specific gravity* at 15° C. from ·857 to ·860. *Optical rotation* not less than + 60°. Adulteration with turpentine reduces this figure according to the degree of sophistication.

Fractionation.—The oil distils between 160° and 230° C. The chief fraction to observe is that boiling below 173° C., which should not exceed 25 per cent.; any addition of turpentine increases this fraction considerably, since turpentine consists almost wholly of pinene (boiling-point 155° C.). The optical rotation of the first 10 per cent. of the distillate should not differ from that of the whole bulk of oil by more than 2° (for detection of turpentine).

Constituents.—The terpene limonene (see Oil of caraway) constitutes over 90 per cent. of this oil. Lemon oil contains two aldehydes, citral and citronellal. The former predominates, and has a specific gravity of ·895 at 15° C., and boiling-point between 223° and 228° C. It is optically inactive, and is formed by the oxidation of geraniol, a constituent of a great number of essential oils. Citronellal, the aldehyde of the newly discovered alcohol citronellol, has a sp. gr. of ·869 at 15° C., and boils between 203° and 208° C. Several methods have been proposed for the estimation of these aldehydes, but their application to lemon oil has so far, not been altogether successful.

Oleum Menthæ Piperitæ

Source.—Distilled from fresh-flowering peppermint, *Mentha piperita.*

Characters.—Colourless, pale yellow, or greenish yellow when freshly distilled, but gradually becoming darker by age. It has the odour of the herb, and a strong penetrating aromatic taste, followed by a sensation of coldness in the mouth. *Specific gravity* from ·900 to ·920. The sp. gr. of the English oil usually ranges from ·900 to ·910, while the American has a much higher specific gravity. *Optical rotation* from – 20° to – 32°. It is soluble in four parts of 70 per cent. alcohol.

Fractionation.—The principal fraction passes over between 210° and 220° C. The oil should not distil below 200° C. (absence of turpentine). If a portion of the oil be cooled below 17° F. (– 8·3° C.), and a few crystals of menthol be added, a considerable separation of menthol should take place. It should be noted, however, that the English oil crystallises with much more difficulty than the Japanese.

Constituents.—The principal constituent is menthol (menthyl alcohol, $C_{10}H_{19}.OH$). This is crystalline at ordinary temperatures. It is readily estimated by the processes given in the early portion of this chapter dealing with alcohols. Besides menthol, peppermint oil contains small quantities of other bodies, the principal being menthyl acetate. The best English oil contains from 50 to 65 per cent. of menthol. The superior aroma of the English oil does not appear to actually depend upon the percentage of menthol present, but upon the esters and aldehydes, which are present in varying proportions. Undoubtedly the "white mint" furnishes the most delicate oil, and for much useful information dealing with this subject the reader is referred to a paper read by J. C. Umney at an evening meeting of the Pharmaceutical Society, and printed in the 'Pharmaceutical Journal,' series iv, vol. lvi, p. 125.

Japanese peppermint oil is derived from *Mentha arvensis,* var. *piperascens.* The natural oil is not employed in pharmacy, its only value in this country being as a source of menthol, which varies up to 70 per cent.

Oleum Menthæ Viridis

Source.—Distilled from the fresh-flowering spearmint, *Mentha viridis.* Very little oil is now distilled, and its use is so very limited that its inclusion in the new Pharmacopœia is a matter for surprise. English spearmint oil is very costly, and the small quantity that is used in this country is generally imported from America.

Characters.—In appearance this oil is indistinguishable from peppermint oil, but it has a totally different odour and taste. *Specific gravity* from ·920 to ·940. *Optical rotation* – 35° to – 50°. On *fractionation* the greater proportion passes over between 200° and 230° C., the principal fraction, about 35 per cent., boiling between 222° and 226° C., the range of the boiling-point of carvol. The oil dissolves in an equal volume of 95 per cent. alcohol, becoming turbid on further dilution.

Constituents.—Besides terpenes the principal constituent of spearmint oil is carvol (see Oil of caraway).

Oleum Myristicæ

Source.—Distilled from nutmeg; the yield varies from 8 to 10 per cent.

Characters.—Colourless or pale yellow, having the odour and taste of nutmeg. It becomes darker and thicker by age and exposure to air. *Specific gravity* from ·870 to ·910. *Optical rotation* from + 16° in English oils to + 23° in foreign samples.

Constituents.—The principal is the terpene pinene. Owing to presence of fixed oil, however, nutmeg oil frequently contains myristicin and myristic acid. Should these be present they are detected by their insolubility in an equal volume of 95 per cent. alcohol, and by their leaving a crystalline residue when evaporated on a water-bath. The foreign oil rarely complies with the last test.

Oleum Pimentæ

Source.—Distilled from pimento fruits, as well as largely imported from Jamaica. The yield is about 3·5 per cent.

Characters.—Yellow or yellowish red when first distilled, but gradually darkening on exposure to air and light. *Specific gravity* from 1·040 to 1·050, varying according to the proportion of eugenol. *Optical rotation* from – 1° to – 3°.

Fractionation.—The principal fraction distils between 247° and 251° C., the proportion boiling below 247° C. not exceeding 40 per cent.

Constituents.—The chief constituent is the phenol eugenol (see

Oil of cloves). The proportion present, varies from 65 per cent. upwards. The only other constituent is a sesquiterpene of which little is known, but which materially modifies the clove-like odour of the eugenol. The Pharmacopœia roughly estimates the eugenol present, by requiring the oil to form a semi-solid mass when shaken with an equal volume of strong solution of ammonia.

Oleum Pini

Source.—Distilled from the fresh leaves of *Pinus pumilio*.

Characters.—Colourless, or nearly so, with a pleasant aromatic odour and pungent taste. *Specific gravity* from ·865 to ·870. *Optical rotation* from − 5° to − 10°.

Fractionation.—Not more than 10 per cent. should distil below 329° F. (165° C.).

Constituents.—Various terpenes, with some bornyl acetate. The proportion of the latter should not fall below 10 per cent.

The oil distilled from the leaves of *Pinus sylvestris* (official in B.P. 1885) is unobtainable in commerce, and was generally replaced by that of *Abies excelsa, pectinata,* and other species. The oil of *Pinus pumilio* is, however, obtainable in large quantities without difficulty, and is of fairly constant composition. All the pine oils possess a characteristic pine odour, which varies apparently in direct proportion to the amount of bornyl acetate and esters present. As the pine oils are of great interest from a pharmaceutical point of view the following information will be found useful.

	Sp. gravity at 15° C.	Optical rotation.
Ol. Pini Sylvestris	·886	−19°.
Ol. Abies Canadensis	·9026	−25°.
Ol. Abies Excelsæ	·855—·865	−70°.
Ol. Piceæ Vulgaris	·8806	−37°.
Ol. Abies Pectinatæ	·856	−63°.
Ol. Pini Pumilionis	·865—·870	−5° to −10°.

Table showing relative fractionation.

	Below 165° C.	165° to 177° C.	177° to 200° C.	Above 200° C.
Ol. Pini Sylvestris	6 per cent.	29 per cent.	24 per cent.	41 per cent.
Ol. Abies Canadensis	29 ,,	22 ,,	28 ,,	21 ,,
Ol. Abies Excelsæ	2 ,,	77 ,,	13 ,,	8 ,,
Ol. Piceæ Vulgaris	5 ,,	17 ,,	19 ,,	59 ,,
Ol. Abies Pectinatæ	2 ,,	72 ,,	12 ,,	14 ,,
Ol. Pini Pumilionis	2 ,,	52 ,,	24 ,,	22 ,,

These figures are dependent upon the nature and proportion of terpenes present in the oils, but the decomposition of the esters during distillation considerably affects these figures in some instances.

General constituents.—The various oils contain different terpenes, the following being found in one or other specimens:—Pinene, phellandrene, limonene, dipentene, and sesquiterpenes. The acetic ester of borneol seems to be present in all the oils, though there is some doubt as to its presence in *Pinus sylvestris*; the proportion varies from 8 per cent. in *Picea vulgaris* to ·5 per cent. in *Abies excelsa* distilled from young cones. The esters may be estimated by the general process of saponification with alcoholic potash.

Oleum Rosæ

Source.—Distilled from the fresh flowers of *Rosa damascena*, principally in the Balkan districts. The oil is also distilled in Germany and the south of France, but it is found that the proportion of odourless stereoptene in oils made in these countries is very considerably in excess of that from the colder districts of Bulgaria.

Characters.—The oil is a semi-solid crystalline mass at temperatures below 20° C., the range of melting-point being from 19° C. to 22° C. The *specific gravity* of various samples differs according to the percentage of stereoptene and other constituents, varying in the best samples from ·854 to ·860 at 30° C., although below ·856 the proportion of stereoptene is usually somewhat higher. A sp. gr. above ·860 usually indicates adulteration with geranium oil.

The normal percentage of alcohols as determined by the acetylation process is between 70 and 72.

Oleum Rosmarini

Source.—Distilled from the flowering tops of *Rosmarinus officinalis.* Rosemary is but little cultivated in this country, but grows abundantly and is largely distilled in France, Italy, Spain, Dalmatia, &c.

Characters.—Colourless or pale yellow, with the odour of rosemary and a warm camphoraceous taste. *Specific gravity* ·900 to ·915. French and Italian oils vary between ·900 and ·908, whilst the Spanish generally ranges from ·915 to ·920.

Soluble in twice its volume of 90 per cent. alcohol. *Optical rotation* from + 2° to + 8°. The usual adulterant, French turpentine oil, gives a lævogyrate direction in proportion to the percentage added.

Fractionation.—Not more than 25 per cent. should boil below 170° C., and not less than 15 per cent. above 200° C.

Constituents.—Pinene, cineol, borneol. The last is an alcohol isomeric with geraniol, linalool, and menthol; is crystalline at ordinary temperatures; it melts at 204° C., and boils at 212° C.; it is present in pure oils to the extent of 10 to 15 per cent., and may be estimated by the acetylation process. Camphor, the ketone of borneol, may be estimated by the process for menthone in peppermint oil, to which substance it bears an analogous composition.

Oleum Santali

Source.—Distilled from the wood of *Santalum album,* imported from India.

Characters.—Somewhat viscid, pale yellow, and possessing a strongly aromatic odour. *Specific gravity* ·975 to ·980. Soluble in six times its volume of 70 per cent. alcohol (absence of cedar-wood oil). *Optical rotation* not less than − 16°, and not more than − 20°. The Australian and West Indian oils are dextrogyre.

Fractionation is a useful test for cedar-wood oil, as pure sandal oils distil almost completely between 275° and 295° C., whilst the former oil has a much lower boiling-point.

Constituents.—The oil contains not less than 90 per cent. of an alcohol, $C_{15}H_{25}OH$, readily estimated by the acetylation process. The proportion of acetylisable bodies in cedar-wood oil is from 15 to 20 per cent.

Oleum Sinapis Volatile

Source.—Distilled from black mustard seeds after maceration with cold water.

Characters.—Colourless or pale yellow, possessing an intensely penetrating odour. Applied to the skin it produces almost instant vesication. *Specific gravity* 1·018 to 1·030.

Fractionation.—It distils between 147·2° and 152° C., and the first and last portions of the distillate should have the same specific gravity as the original oil (absence of ethylic alcohol and petroleum spirit).

Constituents.—The principal constituent of mustard oil is allyl thiocyanate C_3H_5NCS, present in good oil to 95 per cent. or more. When treated with solution of ammonia the compound thiosinamine is formed, or allyl-thio-urea. This reaction is sufficiently definite to afford a ready means of valuing the oil (see Estimation of allyl thiocyanate).

Oleum Terebinthinæ

Source.—Distilled from oleo-resin obtained from various species of Pinus.

Characters.—Limpid colourless oil, neutral in reaction. It should leave no residue on evaporation. *Specific gravity* ·855 to ·865. American and German oil is dextrogyre while the French oil is lævogyre. The principal constituent is pinene, boiling point 154° C.

The following, although not included in the Pharmacopœia, possess considerable interest for pharmacists.

Oleum Aurantii.—Expressed from the fresh peel of the sweet orange (*Citrus aurantium*) and the bitter orange (*Citrus vulgaris*). In France the oil distilled from the former is known as Essence de Portugal, that from the latter as Essence de bigarade. "Oil of neroli" is distilled from orange flowers, and is practically only used in perfumery.

Sp. gr. ·848 to ·852. *Optical rotation* from + 92° to + 98°. The addition of oil of turpentine reduces the last figures very considerably. The only identified constituent is the terpene limonene, which distils almost completely between 175° and 180° C.

Oleum Bergamii.—Expressed from the fresh peel of *Citrus bergamia.* Greenish or greenish yellow, having a very fragrant odour. Sp. gr. ·882 to ·886. A lower gravity usually indicates adulteration with lemon oil, and consequent reduction of the ester value. *Optical rotation,* after decolorisation by filtering through animal charcoal, not more than + 15°; adulteration with lemon or orange oil raises this figure. One part of oil should dissolve in 2 parts of 80 per cent. alcohol.

Constituents.—The terpene limonene, and dipentene. Linalool, which is also present in very small quantity, and its acetic ester linalyl acetate, may be estimated by the ordinary process. The finest oil contains from 38 to 42 per cent. of ester. When evaporated on a water-bath the oil should not leave more than 6 per cent. of residue (absence of fixed oils).

Oleum Cassiæ.—Distilled from cassia bark and imported from China. It is yellowish brown in colour when fresh, but gradually becomes darker and thicker by exposure to air and age. *Specific gravity* from 1·055 to 1·065. *Constituents.*—Cinnamyl acetate, which is only present in traces, and cinnamic aldehyde (see also Cinnamon oil). Cinnamic aldehyde is present in proportions varying from 70 to 90 per cent.; it is a light yellow odorous oil, readily estimated by the aldehyde process.

Oleum Eucalypti Citriodoræ.—Distilled from the leaves of *Eucalyptus maculata*, var. *citriodora.* A pale yellow oil having a sweet melissa-like odour. *Specific gravity* ·870 to ·900. The principal constituent is citronellal, $C_{10}H_{20}O$, the aldehyde of the alcohol $C_{10}H_{17}OH$. The percentage of oil boiling between 190° and 210° C. (over 90 per cent.) gives a fair idea of the amount of aldehyde present, but for exact determination it must be tested with acetic anhydride, when the acetate of iso-pulegone is formed, which may be saponified in the usual way, using the same calculation as for menthol in peppermint oil.

Oleum Fœniculi.—Distilled from the fruits of *Fœniculum capillaceum* and other species. *Specific gravity* ·965 to ·980, the highest specific gravities being found in those oils containing the greatest proportion of anethol. *Optical rotation* from + 6° to + 22°. *Fractionation.*—The oil distils almost entirely between 190° and 230° C., the principal fractions being from 190° to 200° C., which includes fenchone, and above 225° C., which consists principally of anethol. *Constituents.*—The terpenes, pinene, phellandrene, and dipentene, although these are not always simultaneously present; anethol (see Anise oil),—the proportion present varies from 60 to 75 per cent.; fenchone, closely resembling its isomeride camphor; it is crystalline below 6° C. The proportion may be determined by reduction with sodium and alcohol to the corresponding fenchyl alcohol, $C_{10}H_{17}.OH$, and estimating by the acetylation process. For further information the reader is referred to a paper read by J. C. Umney before the Pharmaceutical Society, and printed in their 'Journal' for March 13th, 1897.

Oleum Gaultheriæ.—Distilled from the leaves of *Gaultheria procumbens.* This oil is almost identical with that of *Betula alba.* *Specific gravity* from 1·180 to 1·187. *Optical rotation* very slightly lævogyre; optical inactivity indicates that it has either been distilled from *Betula alba* or that it is an artificial oil (methyl salicylate). *Constituents.*—Oil of gaultheria contains about 99 per cent. of methyl salicylate, $C_6H_4(OH).COOCH_3$.

When one drop of the oil is shaken with a little water the mixture should give a deep violet colour with ferric chloride.

Oleum Lavandulæ Spicæ.—Spike oil is largely distilled in France. *Specific gravity* from ·905 to ·915, the average being ·910. *Optical rotation* slightly dextro-rotatory. *Constituents.*—In addition to terpenes the oil contains about 30 per cent. of alcohols—geraniol, linalool, and borneol,—which may be estimated by the acetylation process. Adulteration with cheap rosemary oil reduces the alcohol percentage.

Oleum Origani.—Distilled from the flowering tops of *Thymus vulgaris.* The colour of the oil varies from red to colourless, the former being due to imperfect distillation and condensation ; the herb is distilled on the mountain sides in rough stills, where it is often difficult to obtain water for condensing purposes. The red colour is entirely removed by careful redistillation. *Specific gravity* from ·900 to ·920 ; those samples below ·905 are of poor quality. *Optical rotation.*—Pure oil from *Thymus vulgaris* is only slightly lævogyre, whilst the poorer oils show greater rotation according to their admixture with *Thymus serpyllum,* &c. Turpentine adulterated oil sometimes reaches – 20°. *Constituents.*—Thymol and carvacrol. As these boil at 225° and 232° C. respectively, it is possible by fractionation to ascertain approximately the proportion of these bodies present. Thymol and carvacrol may be estimated by the process for phenols ; the percentage should not fall below 75.

TERPENELESS OILS

Terpeneless oils consist of oxygenated compounds such as alcohols, aldehydes, ethereal salts, &c., which have been separated from the accompanying terpenes by fractional distillation under reduced pressure and by other methods. The terpenes have little or no value, and simply act as diluents of the odoriferous and flavouring principles. They have a lower boiling-point than the more valuable alcohols, aldehydes, &c., and come over first, leaving a residue in the still more powerful in flavour than the original oil, and much more soluble in alcohol. From lemon oil, for instance, there can be separated by careful fractionation about 90 per cent. of the terpene limonene, leaving in the still a residue consisting principally of aldehydes. Their chief use is in the manufacture of aërated water essences, &c.

CHAPTER XXXIX

PREPARATIONS FROM ANIMAL PRODUCTS

Fel Bovinum Purificatum.—This soft extract is prepared by evaporating ox-bile to a quarter of its original volume, and mixing with 90 per cent. alcohol, whereby mucilaginous matters are precipitated. The clear solution is then decanted, and the remainder filtered. On distilling off the alcohol a residue is left, which is concentrated to a thick extract on a water-bath.

The composition is exceedingly complex, the chief constituents being taurocholic, glyocholic, and hyoglycholic acids, and their sodium salts. Purified ox-bile should yield perfectly clear solutions with either alcohol or water.

Pancreatinum.—Pancreatin is a mixture of ferments or enzymes obtained from the pancreas or sweetbread—generally of pigs. The pancreatic juice has an alkaline reaction, and possesses the property of converting starch into sugar, peptonising albuminoids, and emulsifying fats.

Pancreatin may be obtained by macerating finely chopped sweetbreads in water slightly acidulated with hydrochloric acid, for two days. The strained liquor is then neutralised with calcium carbonate, filtered, and mixed with strong alcohol. The precipitated pancreatin is collected, washed, and spread in a thin layer on sheets of glass to dry. Unlike pepsin, it is not precipitated by saturated solution of sodium chloride. The United States Pharmacopœia determines its value as follows:—·28 gram of pancreatin, and 1·5 grams of sodium bicarbonate are dissolved in 100 c.c. of water heated to 100° F., and mixed with 400 c.c. of fresh cow's milk, also heated to 100° F. If the mixture is maintained at the same temperature for thirty minutes, no coagulation should occur when strong nitric acid is added to a small quantity contained in a test-tube.

Pepsinum.—Pepsin is a proteolytic ferment obtained from the glandular layer of fresh stomachs of healthy pigs. When tested by the pharmacopœial process it should be capable of dissolving 2500 times its own weight of freshly coagulated and disintegrated egg-albumen.

Pepsin may be prepared by the following process. Perfectly fresh pigs' stomachs are slit open and freed from adhering food. The stomachs are next scraped, and the pulp macerated in water acidulated with hydrochloric acid for several days, with frequent stirring. The mixture is then strained and allowed to deposit. After decanting, the clear liquid is mixed with sodium chloride, which causes the pepsin to float to the surface. After complete separation has taken place, the pepsin is collected and pressed to remove saline matter. Purification is effected by redissolving in distilled water, again precipitating with salt, collecting and pressing ; the last traces of salt being got rid of by washing with a little distilled water. The moist pepsin is dried as rapidly as possible in thin layers on glass, from which it is afterwards detached in the form of scales.

Dry pepsin is scarcely affected by a temperature of 212° F., but in solution if heated to this temperature it rapidly loses its proteolytic power. *Glycerinum Pepsini* should contain 5 grains of the official pepsin in each fluid drachm. *Vinum Pepsini* is usually made by dissolving 160 grains of pepsin in a pint of sherry, and filtering bright by the aid of talc. As thus made it is, however, very prone to deposit. The author prefers to use fresh pepsin *that has not been dried,* dissolved in the smallest quantity of glycerin slightly acidulated with hydrochloric acid. There is no difficulty in obtaining a solution of almost any strength, but it will suffice if 5 minims of the strong glycerin will dissolve 2500 grains of egg-albumen when tested according to the pharmacopœial directions. After valuing, such a volume as corresponds to 160 grains of official pepsin should be diluted to one pint with sherry ; each drachm will then contain one grain of pepsin. The small amount of alcohol contained in sherry does not appear to affect the value of the pepsin as a digestive agent unless the wine has been kept for a very long time.

Gelatinum.—All animals contain certain tissues which are insoluble in water, but which by continued boiling with water are dissolved, the solution on evaporation to dryness yielding a brittle substance, which in a pure state is transparent and free from smell. This substance is called gelatin; it swells up in cold water, and on heating dissolves, the solution if not too dilute, setting to a jelly on cooling. Among gelatin-yielding substances, are, osseine the organic matter of bones, tendons, cartilage, &c.

The process of gelatin making roughly consists in boiling these materials with water until they become dissolved. The solution is then drawn off, strained, and allowed to cool, the jelly being afterwards cut into strips and placed on netting to dry. It is important to note that gelatin solution, after it has been alternately heated and cooled several times, finally refuses to set.

Liquor Thyroidei. — Thyroid solution is prepared from the fresh and healthy thyroid gland of the sheep. The Pharmacopœia directs the glands, which consist of two lobes, to be thoroughly bruised and macerated in a mixture of glycerin and half per cent. phenol solution, for twenty-four hours; the liquid is then strained through linen, and diluted so that 100 minims represent one entire gland. The product is a pinkish turbid liquid which will only keep good for a short time. If, however, it is filtered under pressure through a sterilised porcelain filter into sterilised bottles and hermetically sealed, it may be kept for a considerable period without losing its virtues.

Thyroideum Siccum.—Thyroid powder is obtained by mincing the perfectly healthy glands, and drying at a temperature between 90° and 100° F., powdering the dry product, and removing fat by percolation with petroleum spirit, and again drying the residue. As long as the powder is kept perfectly dry it does not deteriorate, but if allowed to become damp, putrefaction soon sets in.

Keratinum.—See Pill coating.

PART III

DISPENSING

CHAPTER XL

DISPENSING

The dispensing or compounding of medicines, whether for internal or external use, is one of the most important duties of the practical pharmacist.

The *art of dispensing* may be said to comprise all the methods and details pertaining to the extemporaneous preparation of medicines.

To cope successfully with the difficulties that commonly arise in an ordinary day's work, requires a clear head, scrupulous accuracy, and the possession of a very varied store of general knowledge.

In a well-ordered establishment, when a fresh prescription is handed in to be made up, a fair copy is first made in the prescription book, and the prescription itself marked for future reference. The practice of employing large stamps for this purpose is much to be deprecated, the dispenser having no right to disfigure a prescription with an impression that may possibly obliterate a not too well-defined ingredient or quantity. Indeed, many physicians head their note-paper with a request that the reference number or stamp may be placed on the back of the prescription, and it is to be regretted that the practice has not become general.

The numbering of prescriptions is best performed by an automatic contrivance, as Fig. 194. Such machines print the numbers in clear and legible characters and with absolute accuracy, the changes of figures being entirely automatic, while by a self-acting device the figures are freshly inked after each movement of the machine.

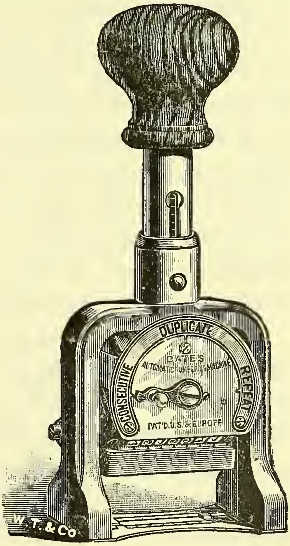

Fig. 194.—Automatic numbering machine for prescriptions.

In addition to marking the prescription with a reference number, it is customary to indicate the price. For this purpose a cipher is usually adopted. Any word of ten letters will do, provided it contains no duplicate letters. Some pharmacists use a word containing nine letters only; in that case the tenth letter is supplied by a sign, as X.

Thus:

F L O R. A N T H E M. C H E M I S T R Y X.
1 2 3 4 5 6 7 8 9 10. 1 2 3 4 5 6 7 8 9 10.

Other words are *volumetric, mind the fox, duplicates.* Adopting Flor. Anthem. as the cipher, a mixture priced 2*s.* 6*d.* would be marked *l/n.*

The letters V. E. occasionally appear on the side of the prescription, and are abbreviations of the Latin *vide exemplum.* This mark is made when the dispensing of the prescription presents some peculiarity, and directs attention to the copy in the prescription book, where an explanation is given.

Physicians sometimes write P. P. at the top of the prescription, thereby indicating that the patient is a "poor person," to whom it would be an act of charity to charge less than the usual rates.

Having copied the prescription and carefully considered the ingredients, their compatibility, dose, &c., the labels should next be written in a neat and legible manner. Due attention must be paid to the spacing, as a label may be written accurately and legibly and yet not present a good appearance, owing to some of the words being immoderately drawn out or crowded together. The labels themselves should be as neat and plain as possible, without ornamentation in the way of pictures; particularly should medicine labels be free from advertisements of specialities.

Whilst on the subject of prescriptions, it may be as well to mention that should any error in dosage or serious incompatibility be detected in a prescription, it becomes the duty of the dispenser to communicate at once with the prescriber. This, if possible, should be done without calling the attention of the patient to the fact. If the prescriber lives within a short distance, it is easy to say that the medicine will take a little time to make up, and induce the patient to wait or to call again; but if this course is not open there may remain no alternative but to explain the matter. With a little tact this may often be done without alarming the patient, but in any case the pharmacist must act with the greatest circumspection.

Bottles and Labels

Mixtures.—Mixtures intended for internal administration may be sent out in green flint *oval* bottles. The upper edge of the label should come to within half an inch of the shoulder. If it is necessary to affix a "shake the bottle" label, it should be placed above the directions label, for the reason that careless people frequently tear back the wrapping paper only just sufficiently to read the directions, so that any label placed near the base of the bottle is not observed. When any of the ingredients happen to be more than usually potent, as solution of strychnia, arsenic, &c., the words "*by measure glass*" may be introduced into the body of the directions, thus:—*One table-spoonful* (*by measure glass*) *every four hours.*

Mixtures of smaller bulk than six ounces are generally sent out in white glass vials. When the dose is less than a tea-spoonful such mixtures are termed "drops." Of late years the practice of sending out mixtures in bottles purporting to be divided into an equal number of parts has become very prevalent. A properly divided bottle saves trouble in measuring the doses, and as long as the practice is confined to the division in ounces, or even table-spoons, of bottles of the capacity of three or four ounces and upwards, no great harm is likely to ensue. It is, however, unreasonable to expect three- or four-ounce bottles to be graduated in teaspoons with anything approaching accuracy.

Gargles.—Gargles, although intended for internal use, may not always be swallowed with impunity, and for distinction may be dispensed in green flint *flat* bottles. The same applies to spray solutions for the throat and nose. The label should be plainly headed "The Gargle," and may be printed on grey or blue-toned paper.

Lotions.—Eye lotions and other moderately harmless external applications are best sent out in light green *fluted* octagonal bottles, although some pharmacists prefer deep green bottles of the ordinary flat pattern. A "not to be taken" label must always be attached. If the lotion is of a distinctly poisonous nature, such as Goulard's extract, a *deep blue fluted* bottle should invariably be selected, and labelled with the words "Poison" and "For external application only."

Liniments.—Liniments and poisonous applications generally should only be dispensed in *deep blue fluted* bottles of octagonal or other distinctive shape. The shape is of almost greater importance than the colour, as the bottles may be readily distinguished in the dark by the sense of touch.

After dispensing the medicines ordered, the prescription should be carefully re-read and the labels checked. When making up old, and perhaps familiar forms, the memory should not be trusted, for, as a notable pharmacist used to tell his dispensers, "memory is treacherous, and medicine is dangerous." The prescription or copy should always be open before the dispenser. Although not trusting to memory when compounding prescriptions and manufacturing preparations, the gift must be carefully cultivated. It is absolutely imperative to familiarise one's self with the ordinary and maximum doses of all important medicines, as well as the proportion of active ingredients in their official preparations. It is of little moment, for instance, in dispensing Pulv. Opii Comp. whether one remembers the amount of ginger contained in five grains of the powder, but it is necessary to realise that one tenth of the weight consists of powdered opium.

The dispenser should cultivate habits of neatness in his work. To this end the littering of the bench with bottles, books, and apparatus should be avoided, each article being replaced or put away as soon as done with. Before removing a bottle from the shelf let the label be carefully read; the habit of taking bottles from accustomed places without looking at the label is a most dangerous one, and has before now led to fatal consequences. Indeed, in more than one well-known pharmacy a rule is made of *slightly* altering the arrangement of the bottles every morning, with the view of discouraging the practice.

Moistening corks with the lips, licking labels, &c., are highly objectionable habits. If too large, the cork should be softened by squeezing in a cork press, as Fig. 195 or Fig. 196.

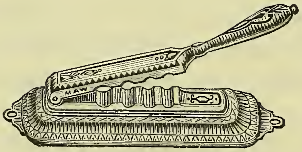

FIG. 195.—Lever cork press.

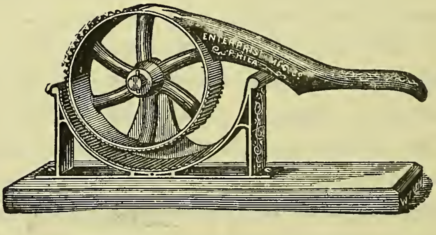

FIG. 196.—Rotary cork press.

The disadvantage of the lever press is that unless used with judgment, it invariably breaks the corks. The rotary cork press, Fig. 196, is a distinct improvement, but is rarely seen in this country. In some of the largest dispensing establishments cork presses are never used, the dispensers preferring to roll the cork beneath a small marble block. Label dampers may take the form shown in Fig. 197. Ready gummed labels, however, are not an unmixed blessing, and many pharmacists still prefer to affix their labels with paste. A good adhesive is prepared as follows :

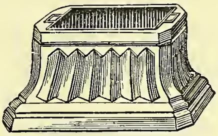
FIG. 197.—Label damper.

℞	Corn flour	6 drachms.
	Distilled water	10 ounces.
	Gelatin	20 grains.

Make a smooth paste with the flour and water, and heat to boiling, stirring constantly. Soak the gelatin in a little water, and when perfectly softened strain off the excess of water and add to the hot mucilage. Stir well, and when cool add 5 minims of oil of cloves.

CHAPTER XLI

PRESCRIPTIONS

Prescription (*præscriptio,* from *præ,* before ; and *scribo,* I write) is the term applied to the written formula and directions of a physician, for the preparation and use of remedies.

A prescription may be divided into four parts ; the heading, designation of the ingredients, directions to the compounder, and the directions to the patient. In addition, every prescription should bear both the name of the patient and the date, and for reference in case of emergency the name and address of the prescriber.

In ancient times every prescription or formula had at its commencement, certain characters, abbreviations or sentences of a pious or superstitious nature, such as + (the sign of the cross) ; *α* and *ω* (the first and last letters of the Greek alphabet), Christ being designated the "Alpha and Omega, the beginning and the ending ;" C.D., *cum Deo ;* J.D., *juvante Deo ;* L.D., *laus Deo ;* N.D., *nomine Dei,* &c. These constituted the *invocation,* or, as it was called, the *inscription.*

The heading.—The symbol ℞, or abbreviated *Rec.* (*recipe,* take thou), usually commences every formula, but in French prescriptions the letter P. or the word *Prenez* (take thou) is usually substituted.

The designation of the ingredients to be employed.—Each ingredient should have a separate line. It is customary to write the names of the ingredients in Latin ; the quantities should be expressed in apothecaries' weights and measures, the numbers being stated in Roman numerals. To minimise the chance of error, every unit should have a dot placed immediately above it, thus : xii, viii. If metrical weights and measures are designated, the greatest care should be observed in making the position of the decimal point plain : thus, 1 gramme, 1·00 ; 1 decigramme, 0·10 ; 1 centigramme, 0·010; milligramme, 0·001. When potent remedies are ordered it is safer to write out the quantity in full, thus:

℞ Strychninæ... ... ... 50 milligrammes.
Sacch. Lact. ... ... q. s.
Make a mass, and divide into 30 pills. One twice a day.

The direction to the compounder as to the manner, form, or method of use of the medicine, constitutes what was originally known as the *subscription*, and is generally written in Latin, as misce, fiat pilula, &c.

The direction to the patient declares the dose and time of administration, if the medicine is intended for internal use, and the method of application if intended for outward use. It is sometimes preceded by the letter S. (*signetur*, let it be entitled). This part of the prescription should invariably be written in English.

There are several reasons for employing Latin in preference to the vernacular in prescriptions, at least for the designation of the ingredients and directions to the compounder. Latin, if not spoken, is written and understood throughout the civilised world; moreover Latin professional terms are concise and definite; and further, the Latin names for drugs and chemicals are nearly the same all over Europe, whereas the vernacular names differ for each nation and sometimes for each province. Lastly, it is sometimes necessary or advisable to conceal from a patient the precise nature of the remedies which are employed. These reasons, however, do not apply to the use of the Latin language for writing the directions to the patient, for as these are intended for the use of the patient, and as sooner or later he must have them in English, there is no advantage to be gained in practising a temporary concealment by writing them in a dead language.

On the contrary, there are several weighty objections to the practice, such as the inability to give in good and intelligible Latin the requisite directions to the patient; the limited acquaintance with Latin unfortunately possessed by some dispensers; and lastly, the difficulty of finding concise and intelligible English words which are the exact equivalent of many professional Latin terms. There is no doubt that by throwing on the compounder the responsibility of expressing in appropriate phraseology, and in the brief compass of a label, the exact intentions of the prescriber expressed other than in English, the risks of error are greatly augmented.

The signs used in prescriptions.—Formerly the signs or symbols employed in chemistry and pharmacy, as substitutes for words, were numerous. At the present time they are very few. The following alone deserve notice:

℞.	*Recipe,* take thou. Ancient authors use the sign ♃, being the old invocation to Jupiter. "It is at present so disguised by the addition of the downstroke, which converts it into the letter ℞, that, were it not for its cloven foot, we might be led to question the fact of its superstitious origin" ('Paris's Pharmacologia').
♏.	*Minimum,* the $\frac{1}{60}$ part of a fluid drachm.
Gtt.	*Gutta,* drop.
℈.	*Scrupulus* vel *scrupulum,* a scruple, equal to 20 grains troy.
ʒ.	*Drachma,* a drachm, equal to three scruples, or in liquids the eighth part of a fluid ounce.
℥.	*Uncia,* an ounce apothecaries', or in liquids the twentieth part of an imperial pint.
lb.	*Libra,* a pound weight avoirdupois.
O.	*Octarius,* a pint.
gr.	*Grana,* a grain.

The rules for the grammatical construction of a prescription would be out of place here, and the reader is referred to the excellent Latin grammar of pharmacy by Mr. Joseph Ince, as well as to the 'Selecta e Præscriptis' of Dr. Pereira, on which the writer has drawn largely.

An explanation of a prescription written entirely in Latin may be useful—from Pereira.

℞ (1) Ferri Carbonatis ... ... ʒiss.
(2) Rhei pulveris ... ... gr. xv.
(3) Conservæ Rosæ, quantum sufficiat, ut fiat massa in pilulas viginti dividenda, quarum sumat æger tres octavis horis.

(1) *Recipe,* verb active, imperative mood, second person singular, agreeing with *tu* understood; governs the accusative case.
drachmam, noun substantive, accusative singular, first declension, governed by *recipe.*
cum, preposition, governing ablative.
semisse, substantive, ablative case, governed by *cum.*
Carbonatis, substantive, genitive singular, governed by *drachmam.*
Ferri, substantive, genitive singular, neuter, second declension, governed by *carbonatis.*

(2) *Recipe* (understood).

grana, substantive, accusative plural, neuter, second declension, governed by *recipe.*

quindecim, adjective, indeclinable.

pulveris, substantive, genitive singular, masculine, third declension, governed by *grana.*

Rhei, substantive, genitive singular, neuter, second declension, governed by *pulveris.*

(3) *Recipe* (understood).

quantum, adverb, governing the genitive.

sufficiat, verb, impersonal, potential mood, present tense, active, third conjugation.

Conservæ, substantive, genitive singular, feminine, first declension, governed by *quantum.*

Rosæ, substantive, genitive singular, feminine, first declension, governed by *conservæ.*

ut, conjunction, governing subjunctive mood.

massa, substantive, nominative case, feminine, first declension.

fiat, verb, subjunctive, present tense, third person singular, governed by *ut,* and agreeing with its nominative *massa.*

dividenda, participle, nominative case, feminine, agreeing with *massa.*

in, preposition.

pilulas, substantive, accusative plural, feminine, first declension, governed by *in.*

viginti, adjective, indeclinable.

quarum, relative pronoun, genitive plural, feminine, agrees with its antecedent *pilulas* in number, gender, and case; governed in the genitive case by *tres.*

æger, adjective, masculine gender, nominative, agreeing with *homo* understood.

sumat, verb, third person singular, imperative, active, third conjugation, agreeing with *homo* (understood).

tres, adjective, accusative plural, feminine, agreeing with *pilulas* (understood).

horis, substantive, ablative plural, feminine, first declension, signifying part of time, and therefore put in the ablative case.

octavis, adjective, ablative plural, feminine, agreeing with *horis.*

CHAPTER XLII

LATIN TERMS USED IN PRESCRIPTIONS, WITH THE MORE COMMONLY OCCURRING ABBREVIATIONS

Latin	*Contracted form*	*English*
A, ab, or abs	a	by or from
Abdomen	abd.	the belly
Absente febre	abs. febr.	fever being absent
Absque	absq.	without
Ac		and
Accidit		it happens
Ad		to, up to
Adde	ad.	add
Addendo	add.	by adding
Addendus	add.	to be added
Ad duas vices		for two times
Adeps suillus		lard
Adhibendus	adhib.	to be administered
Ad libitum	ad lib.	at pleasure
Admove	admov.	apply
Ad secundam vicem	ad sec. vic.	to the second time
Adstrictus	ads.	bound
Ad tertiam vicem	ad. ter. vic.	for three times
Ad tres vices		for three times
Æger, ægra	æg.	the patient
Affectare	aff.	to affect
Agere		to act
Agitato vase	agitat. vas.	the bottle being shaken
Alter	alt.	the other
Alternis horis	alter. horis	every other hour
Alvo adstricta	alv. ads.	when the bowels are confined
Alvus	alv.	the belly (the bowels)
Ambo	amb.	both
Amplus	amp.	full, large
Ampulla	amp.	a large bottle
Ana	a. aa. āā	of each
Ante	ant.	before
Aperiens	aper.	aperient
Apud		at, near
Aqua bulliens	aq. bull.	boiling water

Latin	*Contracted form*	*English*
Aqua communis	aq. com.	common water
Aqua destillata	aq. dest.	distilled water
Aqua fervens	aq. ferv.	warm water
Aqua fluvialis	aq. fluv.	river water
Aqua fontana	aq. font.	spring water
Aqua pluvialis	aq. pluv.	rain water
Aquosus	aq.	watery
Auratus		golden
Auris		the ear
Aut		or
Balneum	baln.	a bath
Balneum tepidum		a warm bath
Bene		well
Bibe	bib.	drink (thou)
Biduum	bid.	two days
Bis		twice
Bis in die	b. i. d.	twice a day
Bolus	bol.	a large pill
Brachio sanguis emittatur		let blood be taken from the arm
Brachium		an arm
Brevis	brev.	short
Bulliens	bull.	boiling
Cæterus ; et cætera	cæt. ; et cæt.	the other ; and so forth
Calefactus	calef.	warmed
Calidus	calid.	warm
Calomelas	calom.	calomel
Calor		heat
Cape	cap.	take (thou)
Capiat	cap.	let him take
Capillus		a hair
Capsula		a capsule
Caput		the head
Cataplasma	catap.	a poultice
Caute		cautiously
Ceratum	cerat.	a cerate
Cibus	cib.	food
Circa	circ.	around
Circiter	circ.	about
Cochleare	coch.	a spoon
Cochleare amplum	coch. amp.	a tablespoon
Cochleare magnum	coch. mag.	a tablespoon
Cochleare minimum	coch. min.	a teaspoon
Cochleare parvum	coch. parv.	a teaspoon
Cochleatim	cochleat.	by spoonfuls
Colatur	colat.	let it be strained
Colatus	colat.	strained
Collutorium	collut.	a mouth wash

Latin	*Contracted form*	*English*
Collyrium	collyr.	an eye wash
Compositus	comp.	compound
Concisus	concis.	sliced
Confectio	conf.	a confection
Congius	cong. C.	a gallon
Contritus	cont.	bruised
Contusus	cont.	bruised
Coque	coq.	boil
Cortex	cort.	the bark
Cras	cr.	to-morrow
Cras mane sumendus	c. m. s.	to be taken to-morrow morning
Cras nocte	c. n.	to-morrow night
Cras vespere sumendus	c. v. s.	to be taken to-morrow evening
Crus		the leg
Cujus	cuj.	of which
Cum	c̄	with
Cyathus	cyath.	a cup, wine-glass
Cyathus vinarius	cy. vinar.	a wine-glass
De		from
Deaurentur pilulæ	deaur. pil.	let the pills be gilded
Decanta	decant.	pour off
Decem		ten
Decoctum	dec.	a decoction
Decubitus	decub.	lying down
Decubitus horâ	decub. hor.	at bedtime
De die in diem	de d. in d.	from day to day
Deinde	deind.	then ; the next
Dens		a tooth
Dexter		the right side
Diebus alternis	dieb. alt.	every other day
Diebus tertiis	dieb. ter.	every third day
Dies	d.	a day
Dilutus	dil.	diluted
Dimidium	dimid.	half
Dividatur in partes æquales	divid. in p. æq.	let it be divided into equal parts
Donec		until
Donec alvus soluta fuerit	don. alv. sol. fuer.	until the bowels are opened
Donec dolor exulaverit	donec dolor ex.	until the pain is removed
Dosis	dos.	a dose
Drachma	dr.	a drachm
Dulcis	dulc.	sweet
Dum		until
Duo		two
Duplex	dup., d–x.	double
E, ex		out of, from
Eadem	ead.	the same
Ejusdem	ejus.	of the same

Latin	*Contracted form*	*English*
Electuarium	elect.	an electuary
Electus	elect.	selected
Emeticum	emet.	an emetic
Emplastrum	emp.	a plaster
E vehiculo crasso	e vehic. crass.	in a thick vehicle
Exhibeatur	exhib.	let it be exhibited
Ex tempore		at the time
Extende	extend.	spread
Extende super alutam mollem	ext. sup. alut. moll.	spread upon soft leather
Extractum	ext.	an extract
Fac	f.	make thou
Facies		the face
Fiat	ft.	let it be made
Fiat haustus	ft. hst.	let a draught be made
Fiat mistura	ft. mist.	let a mixture be made
Fiat pilula	ft. pil.	let a pill be made
Fiat secundem artem	ft. s. a.	let it be made according to art
Febre durante	febr. dur.	during the fever
Febricula	febr.	fever
Febris	febr.	a fever
Fervens	ferv.	boiling
Flatus	flat.	breathing (flatulence)
Folium	fol.	a leaf
Fortis	fort.	stronger
Fotus		a fomentation
Frigidus	frigid.	cold
Frustillatim	frust.	in little pieces
Fuscus	fusc.	brown
Gallicus	Gall.	French
Gargarisma	garg.	a gargle
Genu		the knee
Gradatim	grad.	by degrees
Granum	gran.	a grain
Gummi	gum.	a gum
Gutta	gutt., gtt.	a drop.
Guttatim	guttat.	drop by drop
Guttis quibusdam	gtt. quib.	with a few drops
Haustus	hst.	a draught
Heri		yesterday
Hirudo	hirud.	a leech
Hodie		to-day
Hora	hor.	an hour
Horâ decima matutina	h. dec. matut.	at ten in the morning
Horâ decubitus	hora decub.	at bedtime
Horâ somni	h. s.	at bedtime
Horâ unius spatio		in an hour's time

Latin	*Contracted form*	*English*
Ibi		these
Idem	id.	the same
In		in
Incisus	incis.	cut, sliced
Indies		daily
Infunde	infund.	pour in
Infusum	inf.	an infusion
In pulmento	in pulm.	in gruel
Inspissare	insp.	to thicken, inspissate
Inter		between
Intime	intim.	intimately
Involvere	involv.	to roll in
Jecur		the liver
Jentaculum	jentac.	breakfast
Julepus	julep.	a julep
Juxta		near to
Lac		milk
Latus		the side
Laxativus	laxat.	a laxative
Libra	lb.	a pound
Lignum	lign.	wood
Linimentum	lin.	a liniment
Liquor	liq.	a solution
Lotio	lot.	a lotion
Macerare	mac.	to macerate
Male		ill
Mane	m.	in the morning
Mane primo	m. prim.	very early in the morning
Manipulus	manip.	a handful
Manus		the hand
Massa	m.	a mass
Medicamentum	med.	a drug
Mensurâ	mens.	by measure
Meridies	merid.	midday, noon
Mica panis	mic. pan.	crumb of bread
Minimum	min., m.	a minim
Misce	m.	mix
Misce bene	m. b.	mix well
Mistura	mist.	a mixture
Mitis		mild
Mitte	mit.	send
Modo præscripto	mod. pr.	in the manner prescribed
Mollis	moll.	soft
More dictu	m. dict.	in the way directed
More solito	mor. sol.	in the usual manner

Latin	*Contracted form*	*English*
Nimis		too much
Nisi		unless
Nocte	noct.	at night
Non repetatur	non rep.	do not repeat
Nucha capitis	nuch. cap.	the nape of the neck
Numero	no.	in number
Octarius	oct., O.	a pint
Oculus	oc.	the eye
Omne mane	om. m.	every morning
Omne nocte	om. n.	every night
Omni biduo	om. bid.	every two days
Omni bihorio	om. bihor.	every two hours
Omnibus alternis horis	om. alt. hor.	every other hour
Omni horâ	om. h.	every hour
Omni quadrante horæ	om. quad. hor.	every quarter of an hour
Omnis	om.	all
Pars		a part
Partes æquales	p. æq.	equal parts
Partitis vicibus	part. vic.	in divided doses
Partem affectam	part. aff.	the affected part
Partem dolentem	part. dol.	the painful part
Parvulus	parv.	small
Pastillus	past.	a pastille
Pauxillum	paux.	a little
Per		through, by
Pes, pedis	ped.	a foot
Pharmacopœia Britannica	Ph. B.	The British Pharmacopœia
Pharmacopœia Dublinensis	Ph. D.	The Dublin Pharmacopœia
Pharmacopœia Edinensis	Ph. E.	The Edinburgh Pharmacopœia
Pharmacopœia Londinensis	Ph. L.	The London Pharmacopœia
Pilula	pil.	a pill
Pocillum	pocill.	a little cup
Poculum	poc.	a cup
Pollex	poll.	the thumb
Ponderosus	pond.	heavy
Postea	post.	afterwards
Post jentaculum	p. jent.	after breakfast
Post meridiem	p. mer.	the afternoon
Post prandium	p. prand.	after dinner
Post singulas sedes liquidas	p. sing. sed. liq.	after every liquid stool
Pro ratione ætatis	p. rat. æt.	according to the age of the patient
Pro re natâ	p. r. n.	occasionally
Proximâ luce	prox. luc.	on the following day
Pugillus	pug.	a pinch
Pulvis	pulv.	a powder
Quantum	q., quant.	as much

Latin	*Contracted form*	*English*
Quantum libet	q. lib.	as much as desired
Quantum sufficiat	q. s.	a sufficiency
Quaque	qq.	each, every
Quaque quarta hora	q. q. hora	every four hours
Quartus	quart.	the fourth
Quater	quat.	four times
Quaterve	quat.	four times
Quies		quiet sleep
Quinquies	quinq.	five times
Quotidie	quotid.	daily
Recipe	℞	take thou
Redigatur in pulverem	redig. in pulv.	let it be reduced to powder
Regio		region, part
Repetatur	rep.	let it be repeated
Rursus		again
Scatula	scat.	a box
Scrobiculus cordis	scrob. cord.	the pit of the stomach
Secundem artem	s. a.	according to art
Semel		once
Semidrachma	semidr.	half a drachm
Semihora	semih.	half an hour
Semis	ss.	half
Sequenti luce	seq. luce	the following day
Sesquihora	sesquih.	an hour and a half
Sesuncia	sesunc.	an ounce and a half
Si		if
Signe	sig.	write thou
Signatura	signat.	a label
Simul	sim., s.	together
Sine	s.	without
Singulorum	sing.	of each
Singulus	sing.	single, each
Si non valeat	s. n. valeat	if it does not answer
Si opus sit		if there be occasion
Solve	solv.	dissolve
Somnus	somn.	sleep
Spiritus vini rectificatus	s. v. r.	rectified spirit
Spiritus vini tenuior	s. v. t.	proof spirit
Statim	st.	at once, immediately
Stet	st.	let it stand
Sufficiens	suff.	sufficient
Sumat	sum.	let him take
Sumatur	sum.	let it be taken
Sume	sum.	take thou
Sumendus	sum.	to be taken
Super	sup.	upon

Latin	*Contracted form*	*English*
Tabella	tab.	a lozenge
Talis	tal.	such, like this
Tempori dextro	temp. dext.	to the right temple
Ter	t.	thrice, three times
Tere		rub thou
Tere simul	t. s.	rub together
Ter in die	t. i. d.	thrice daily
Tinctura	tr.	a tincture
Tot		so many
Totus		the whole
Triduum	trid.	three days
Trochiscus	troch.	a lozenge
Tussis	tus.	a cough
Ultimo præscripto	ult. præsc.	the last ordered
Ut dictum	ut dict.	as directed
Uncia	unc., ℥	an ounce
Vas vitreum	vas. vit.	a glass vessel
Vel		or
Venæ		the veins
Vices	vic.	times
Vitellus	vit.	yolk
Venenum	ven.	poison
Vesper	vesp.	the evening
Vomitus	vomit.	vomiting
Vulnus		a wound

CHAPTER XLIII

TERMS LIKELY TO OCCUR IN FRENCH AND GERMAN PRESCRIPTIONS *

À, *Fr.*, to, or. Trois à quatre paquets (three or four powders).

Abendessen, Abend-brod, -mahlzeit, -tisch, *Ger.*, supper. Drei von diesen Pillen vor dem Abendessen (three of these pills before supper).

Acide azotique, *Fr.*, nitric acid.

Aetz, *Ger.*, caustic.

Aetzstein, *Ger.*, caustic potash.

Alcohol sulphuris, *Ger.-Latin*, carbon bisulphide.

Alcool de soufre, *Fr.*, carbon bisulphide.

Aqua calcaria, *Ger.-Latin*, lime water.

Arsenige säure, *Ger.*, arsenious acid.

Azotate, *Fr.*, nitrate.

Barbotine, *Fr.*, santonica.

Baudruche, *Fr.*, goldbeater's skin.

Bisse, *Ger.*, bolus. Sechs bissen im Tage zu nehmen auf drei Gaten vertheilt (six boluses to be taken daily, divided into three doses).

Bleiessig, *Ger.*, Liq. Plumbi Subacetatis.

Bol, *Fr.*, bolus. À prendre sex bols par jour en les partageant en trois doses (six boluses to be taken every day, dividing them into three doses).

Calcaria, *Ger.-Latin*, calx or calcium.

Carboneum, *Ger.-Latin*, carbon.

Carbonicum, *Ger.-Latin*, carbonas or carbonate.

Cautère potentiel, *Fr.*, caustic potash.

Chaux, *Fr.*, lime.

Chinin, *Ger.*, quinine.

Chininum, *Ger.-Latin*, quinine.

Chloratum, *Ger.-Latin*, chloride.

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* Abstracted, by permission, from "The Art of Dispensing," 'C. and D.'

Chlorsäures, *Ger.-Latin*, chlorate.

Citricum, *Ger.-Latin*, citrate.

Coccionella, *Ger.-Latin*, cochineal.

Colla piscium, *Ger.-Latin*, ichthyocolla.

Coton cardé, *Fr.*, cotton wool.

Coucher, *Fr.*, bedtime, going to bed. À pendre deux pilules avant le coucher (two pills to be taken at bedtime).

Cuillerée à café, *Fr.*, teaspoonful. Un cuillerée à café au cas d'une attaque de toux (a teaspoonful to be taken if the cough comes on).

Cuillerée à soupe, *Fr.*, tablespoonful. Prenez une cuillerée à soupe tout les deux heures (one tablespoonful every two hours).

Cyantum, *Ger.-Latin*, cyanide.

Dower'sche Pulver, *Ger.*, Dover's powder.

Eau de rabel, *Fr.*, Mistura Sulphurica Acida.

L'effet voulu, *Fr.*, the desired effect. Une cuillerée à café toutes les demi-heures jusqu'à l'effet voulu (a teaspoonful every hour till it acts).

Einspritzung, *Ger.*, injection.

Eisessig, *Ger.*, glacial acetic acid.

Emplastrum adhæsivum anglicum, *Ger.-Latin*, court plaster.

Emplastrum picatum, *Ger.-Latin*, pitch plaster.

Essen, *Ger.*, meals.

Essig, *Ger.*, vinegar.

Esslöffel, *Ger.*, tablespoon. Alle zwei Stunden einen Esslöffel voll (a tablespoonful every two hours).

Ferrocyanatum, *Ger.-Latin*, ferrocyanide.

Flasche, *Ger.*, bottle. Schütteln sie die flasche (shake the bottle well).

Fois, *Fr.*, time. Prenez en quatre fois à une demi-heure d'intervalle (to be taken in four portions at intervals of half an hour).

Gouttes, *Fr.*, drops. À prendre dix gouttes trois fois par jour (ten drops to be taken thrice daily).

Glas, *Ger.*, glass, tumbler.

Hirschtalg, *Ger.*, mutton suet.

Höllenstein, *Ger.*, silver nitrate, lunar caustic.

Iodure de formyle, *Fr.*, iodoform.

(À) Jeun, *Fr.*, fasting. Prenez deux au trois de ces pilules à jeun (take two or three of these pills fasting).

Kohlensäure, *Ger.*, carbonic acid.

Kümmel, *Ger.*, caraway.

Latwerge, *Ger.*, electuary.

Lavement, *Fr.*, enema.

Limonade sèche, *Fr.*, effervescent saline.

Liqueur de belloste, *Fr.*, Liq. Hydrarg. Nitratis Acidus.

Liquiritia, *Ger.-Latin*, liquorice.

Mal, *Ger.*, time, portion. Auf vier mal in Halbstündigen zwischenräumen zu nehmen (to be taken in four portions at intervals of half an hour).

Mittagsessen, *Ger.*, dinner (properly the midday meal). Dieses Pulver unmittelbar vor dem Mittagsessen zu nehmen (this powder to be taken immediately before dinner).

Natrium, *Ger.-Latin*, sodium, soda.

Nüchtern, *Ger.*, sober, fasting. Vier oder sechs von diesen Pillen nüchtern zu nehmen (four or six of these pills to be taken fasting or before breakfast).

Oblate, *Ger.*, wafer. Ein Pulver vor der Mahlzeit in einer Oblate zu nehmen (a powder to be taken in a wafer before meals).

Ordonnance, *Fr.*, prescription.

Ouate, *Fr.*, cotton wool.

Pain azyme, *Fr.*, wafer. Un de ces paquets à prendre dans du pain azyme avant le repas (one of these powders to be taken in a wafer before meals).

Paquet, *Fr.*, a packet, powder. À prendre un paquet toutes les deux heures (one powder to be taken every two hours). On prendre un de ces paquets peu de temps avant l'attaque de fièvre (one of these powders to be taken shortly before the fever fit).

Pasta gummosa, *Ger.-Latin*, pâte de guimauve.

Pastillen, *Ger.*, lozenges. Man nimmt von diesen Pastillen auf einmal nur eine alle zwei Stunden (one only of these lozenges to be taken every two hours).

Pastilles, *Fr.*, lozenges. À prendre quatre à six pastilles par jour (four to six lozenges to be taken daily).

Pierre à cautère, *Fr.*, caustic potash.

Pillen, *Ger.*, pills. Zwei Pillen jeden Abend vor dem zubettegehen (two pills every evening at bedtime).

Pilules, *Fr.*, pills. Deux pilules chaque soir avant le coucheur (two pills every evening at bedtime).

Pincée, *Fr.*, a pinch. Infusez une pincée de ces herbes avec un demi-litre d'eau bouillante pour faire un tisane (infuse a pinch of these herbs in half a pint of water to make a draught).

Potasse à la chaux, *Fr.*, caustic potash.

Potion, *Fr.*, mixture, potion.

Poudre, *Fr.*, powder. Matin et soir une poudre dix minutes avant le repas (one powder ten minutes before meals every morning and night).

Poudre alexitère, *Fr.*, Pulv. Ipecacuanhæ Comp.

Poudre anodine, *Fr.*, Pulv. Ipecacuanhæ Comp.

Poudre gazeuse, ou gazifère purgative, *Fr.*, seidlitz powder.

Poudre gazogène, *Fr.*, effervescent or gazogene powder.

Poudre gazogene laxative, *Fr.*, seidlitz powder.

Poudre gazogène neutre, *Fr.*, soda powder.

Poudre savory, *Fr.*, seidlitz powder.

Poudre sudorifique, *Fr.*, Pulv. Ipecacuanhæ Comp.

Pulver, *Ger.*, powder. Ein Pulver jeden Morgen und Abend zehn Minuten vor dem Essen (one powder every morning and evening ten minutes before meals).

Pulvis aërophorus, *Ger.-Latin*, effervescent powder, soda powder.

Pulvis aërophorus laxans, *Ger.-Latin*, seidlitz powder.

Pulvis gummosa, *Ger.-Latin*, Pulv. Tragacanth. Comp.

Räucherkezchen, *Ger.*, fumigating pastilles.

Rucheressig, *Ger.*, toilet vinegar.

Remède du capucin, *Fr.*, Liq. Hydrargyri Nitratis Acidus.

Remède du Duc d'Antin, *Fr.*, Liq. Hydrargyri Nitratis Acidus.

Repas, *Fr.*, meals.

Rezept, *Ger.*, prescription.

Rhodomel, *Fr.*, Mel Rosæ.

Riechessig, *Ger.*, aromatic vinegar.

Saindoux, *Fr.*, lard.

Säure, *Ger.*, acid.

Schlafengehen, *Ger.*, bedtime. Vor dem Schlafengehen zwei Pillen zu nehmen (two pills to be taken at bedtime).

Schnupfen, *Ger.*, to snuff. Fünf bis sechs Mal im Tage zu schnupfen (to be snuffed five or six times daily).

Schwarzeswasser, *Ger.*, Lotio Nigra.

Schwefel, *Ger.*, sulphur.

Schwefelsäure, *Ger.*, sulphuric acid.

Schwefligesäure, *Ger.*, sulphurous acid.

Sebum, *Ger.-Latin*, suet.

Sel de lait, *Fr.*, milk-sugar.

Semen ciné, *Fr.*, santonica.

Soufre végétal, *Fr.*, lycopodium.

Stibium, *Ger.-Latin*, antimony.

Suère de saturne, *Fr.*, lead acetate.

Sulfuratum, *Ger.-Latin*, sulphide.

Sulfuricum, *Ger.-Latin*, sulphate.

Table, *Fr.*, a table. Se mettre à table (to dine). À prendre deux de ces pilules en se mettant à table (two pills to be taken before dining).

Taffetas d'angleterre, *Fr.*, court plaster.

Tartarus depuratus, *Ger.-Latin*, cream of tartar.

Tartarus natronatus, *Ger.-Latin*, Rochelle salt.

Tartarus stibiatus, *Ger.-Latin*, tartar emetic.

Theelöffel, *Ger.*, teaspoon. Ein Theelöffelvoll (a teaspoonful).

Tisane, *Fr.*, draught, medicated drink.

Tische, *Ger.*, table. Zu Tische gehen (to dine). Man nehme zwei von diesen Pillen wen man zu Tische geht (take two pills before dining).

Tropfen, *Ger.*, a drop. Drei Mal des Tages zehn Tropfen zu nehmen (ten drops to be taken three times a day).

Verordnung, *Ger.*, prescription.

Verre, *Fr.*, glass tumbler. Un verre d'eau sucrée (a tumbler of sugar and water).

Weinsteinsäure, *Ger.*, tartaric acid.

Wirkung, *Ger.*, action, effects. Ein Theelöffelvoll alle halbe stunden bis zur Wirkung zu nehmen (take a teaspoonful every half-hour till it acts).

Zubettegehen, *Ger.*, bedtime.

CHAPTER XLIV

INCOMPATIBLES

Incompatible substances cannot exist together in solution without mutual decomposition, nevertheless prescriptions containing two or more incompatibles are commonly met with. Sometimes this is intentional, as in the following :

℞	Zinci Sulphatis	gr. ij.
	Plumbi Acetatis	gr. iv.
	Vini Opii	ʒj.
	Aquæ, ad	℥iij.

In this lotion the zinc sulphate decomposes part of the lead acetate, the remainder being precipitated as lead meconate, acetate of zinc and acetates of the alkaloids remaining in solution. The following prescription of a well-known London physician is an excellent example of a mixture containing incompatibles, but it is always dispensed as written :

℞	Quininæ Salicylatis	gr. viij.
	Potassii Iodidi	℈j.
	Potassii Bicarbonatis	ʒij.
	Vini Colchici	ʒj.
	Pulv. Tragacanthæ Comp.	ʒj.
	Aquæ, ad	℥viij.

The next prescription is mentioned by Professor Remington in his 'Practice of Pharmacy :'

℞	Strychninæ Sulphatis	gr. j.
	Potassii Bromidi	ʒvij.
	Aquæ, ad	℥viij.

This mixture remains perfectly bright for some time after mixing, but eventually deposits crystals of strychnia bromide, and unless the bottle is well shaken before each dose, the probability, is that the patient would receive a poisonous quantity in the last.

It is difficult to lay down any general rule for the treatment of incompatibles in prescription, but if any substances are contained which are likely to interact and produce dangerous decomposition products, it becomes the duty of the pharmacist to communicate with the prescriber; in no case should medicine be sent out which in the judgment of the dispenser is likely to endanger the life of the patient. If, on the other hand, no harm is likely to ensue from mixing incompatible substances, the dispenser should, unless he has good reason to believe it to be the result of an oversight, compound the medicine as elegantly as circumstances permit.

The table given below summarises the principal incompatible combinations likely to occur in prescriptions, and which may form dangerous mixtures.

Alkaloidal salts, with alkalies, alkaline or earthy carbonates, Donovan's solution, tannic acid, iodides or bromides.

Antipyrin, with spirit of nitrous ether, nitrites, per-salts of iron, sodium salicylate, chloral hydrate, or tannic acid.

Arsenic, with salts of iron, magnesia, or astringents.

Bromides, as iodides.

Chloral hydrate, with alkalies, alkaline carbonates, or antipyrin.

Chloralamide, with hot water or alkalies; these split the compound up into chloral and formamide.

Donovan's solution, with alkaloidal salts.

Iodides, with spirit of nitrous ether (unless containing free alkali), potassium chlorate.

The 'Chemist and Druggist' has recorded an apparently harmless mixture containing ferrous iodide and potassium chlorate, which caused death:

℞	Potassii Chloratis	ʒij.
	Syrupi Ferri Iodidi	ʒvj.
	Vini Antimonialis	ʒss.
	Spiritûs Chloroformi	ʒij.
	Aquæ, ad	℥viij.

This mixture is almost colourless when first prepared, but rapidly turns reddish brown, and after a few days deposits crystals of iodine. This is no doubt due to the action of the chlorate on the ferrous iodide, potassium chloride being formed and iodine set free, ferric hydrate finally precipitating.

Nitro-glycerin solution, with aqueous menstrua, unless the mixture contains sufficient alcohol to keep the nitro-glycerin in solution.

Strychnia (see Alkaloidal salts), *with* iodides, bromides, alkalies, or alkaline carbonates.

Mr. Campbell ('Chemist and Druggist,' April 28th, 1888) has shown that although sodium bicarbonate is incompatible with *solution of strychnia*, it does not follow that the alkaloid will *always* be precipitated. The following mixture, for example, remains clear:

℞	Liquor Strychninæ	♏v.
	Sodii Bicarbonatis	gr. xv.
	Aquæ, ad	℥j.

Each dose of the mixture contains about $\frac{1}{22}$ grain of alkaloid, but as strychnia is soluble 1 in 5700, and the solution under consideration is weaker than this, being 1 in about 9600, no deposition of crystals takes place.

Spirit of nitrous ether, as stated, is incompatible with antipyrin, iodides, bromides, and sodium salicylate.

The following mixture, noted in the 'Chemist and Druggist,' November 6th, 1897, is colourless when freshly made, but rapidly acquires a red colour, due to the formation of nitro- and diazo-oxybenzoate of sodium:

℞	Sp. Ætheris Nitrosi	♏v.
	Sodii Salicylatis	gr. v.
	Syrupi	ʒss.
	Aq. Menthæ Pip., ad	℥j.

If it is specially desired to order antipyrin with spirit of nitrous ether, bicarbonate of soda should also be prescribed in order to avoid formation of isonitroso-antipyrin, which gives a green colour to the mixture. In the presence of water the ethyl nitrite is then gradually decomposed, with formation of sodium nitrate and ethyl alcohol.

Note.—It is not generally known that solution of perchloride of iron is incompatible with glycerin and alcohol, especially when kept in strong sunlight. Tincture of perchloride of iron, when freshly made, is of a dark sherry colour, but after keeping some time becomes of a much lighter tint. The same phenomenon is noted with glycerin. In both cases a considerable portion of the iron is reduced to the ferrous condition, the preparation becoming much less astringent. Knowing this, one is at no loss to understand the complaint sometimes made by patients taking this class of medicine, that the last dose is not nearly so nasty as the first.

CHAPTER XLV

EXPLOSIVE COMBINATIONS

It occasionally happens that mixtures of chemical compounds are ordered in prescriptions, which, if brought together in the ordinary way, combine with explosive violence.

Chlorates should never be mixed with, and particularly never rubbed in a mortar with—

Acid, carbolic.
,, gallic.
,, oxalic.
,, salicylic.
,, tannic.
Catechu.
Creasote.
Glycerin.
Hypophosphites.
Iodine.
Shellac.*
Lycopodium.
Sugar.
Sulphur.
The sulphides of antimony.

Fulminates.—All fulminates are exceedingly explosive.

Nitrogen iodide, formed by the action of iodine on ammonia, decomposes with great violence when dry, even if touched with a feather. A case of explosion has been reported caused by mixing iodine with ammoniated liniment of camphor.

Silver oxide explodes when mixed with creasote. If, however, the oxide be first diluted with some inert powder, as kaolin, and then mixed with paraffin, it may be made into pills without much risk. Pills containing oxide of silver, if massed with syrup or other reducing agent, often swell up and crumble, but rarely explode with any violence.

Potassium dichromate and chromic acid must not be mixed with glycerin.

Permanganates, owing to the readiness with which they part with oxygen, give rise to an explosion when mixed with many organic substances; in particular, they should never be mixed with glycerin, alcohol, sulphides, fats, oils, or sugar.

Picric acid and picrates, especially the picrates of ammonium and

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* The preparation of coloured fires is illegal except on licensed premises, and is attended with considerable danger.

calcium, must be handled with caution, as they explode even when moderately heated.

Iodine must not be added to oil of turpentine.

Phosphorus inflames spontaneously, and should always be kept under water. Care should also be taken in the handling of *sodium* and *potassium*. Both should be stored under mineral naphtha or other hydrocarbon, and when weighing or handling, must be kept free from water. Ethyl and methyl compounds, inflame under certain conditions in the presence of some metals, such as aluminium, zinc, arsenic, &c.

Hypophosphites sometimes decompose violently. They should never be heated, even in solution.

Explosions sometimes occur in mixtures containing bismuth oxynitrate and sodium bicarbonate owing to liberation of CO_2; also in mixtures containing nitric and hydrochloric acids, unless the acids after mixing have been left to interact for some time before corking; hypochloride of sulphur occasionally explodes if the bottle is handled at all carelessly. If the stopper sticks, the bottle should be enveloped in a cloth before attempting to loosen.

Erythrol tetranitrate.—Care should be exercised in dispensing this new remedy, as a fatal accident recently occurred during its preparation for tablet making. It should not be diluted with any organic or readily oxidisable substance, and if required in powder, it should be triturated in a perfectly clean mortar with wooden pestle.

CHAPTER XLVI

EMULSIONS

Emulsions are milky liquids produced by the suspension of oil or resin in water, by the intervention of some mucilaginous or alkaline substance. Milk, which consists of minute globules of fat suspended in water by casein, may be regarded as a type of a perfect emulsion; in it the oil globules are invisible to the naked eye, appearing even under the microscope only as very small spheres.

In making artificial emulsions, the object is to so break up an oil or resin, that each particle will become enveloped in a thin mucilaginous coat, and thus be prevented from reuniting with its fellows. If a fixed oil—olive oil, for instance—is shaken vigorously with five or six times its bulk of distilled water, a milky-looking liquid is formed, which, on standing, separates into two distinct layers. If mucilage of acacia be now added to the mixture and the whole well agitated again, an emulsion, imperfect, it is true, but still an emulsion, is formed, for even if allowed to rest for several days the oil and water will not separate into two clear layers as before. The globules of oil have each become enveloped in a film of gum, and the smaller the globules and the more perfectly they are coated with such a film, the longer will such an emulsion persist.

Although many emulsifying agents have been put forward from time to time, acacia powder still holds an unrivalled position. To obtain the best results the acacia should not be in fine powder, as it is liable to form clots which are dissolved only with great difficulty; if the powder will pass through a forty-hole sieve it may be considered sufficiently fine for the purpose. There are two distinct methods of emulsifying oils with acacia, and each should be thoroughly mastered.

(*a*) The first method aims at the preparation of a concentrated emulsion, which may be diluted afterwards to almost any desired extent. Take of—

Any fixed oil	8 fluid parts.
Powdered acacia	2 parts.
Distilled water	6 fluid parts.

Place the gum in a clean and perfectly dry Wedgwood mortar, add the oil, and triturate with a flat-bottomed pestle until evenly mixed; then add the water all at once and triturate briskly, until the mixture thickens and appears almost white. Scrape down any portions of unmixed gum that may adhere to the mortar or pestle, and continue the trituration for a short time. The operation as described, should not take more than five minutes; when finished, the concentrated emulsion may be gradually diluted until of the required consistence. If salts, glycerin, or syrup are to be incorporated with the emulsion, they should first be diluted with a portion of the water and added towards the end of the process; on no account are they to be added to the incipient emulsion direct.

The above proportions may be varied slightly according to the viscidity of the oil under treatment. Thus castor oil may be made with rather less gum than almond oil, which is fairly limpid; essential oils, on the other hand, require more gum, the best proportions being—

Essential oil	4 fluid parts.
Powdered acacia	2 parts.
Distilled water	3 fluid parts.

This form yields an excellent emulsion, which bears dilution remarkably well.

(*b*) By the second method a stiff mucilage is prepared with gum and water in the same proportions, and the oil added by degrees, each portion being thoroughly incorporated before the addition of the next. This process requires much longer for its completion than that first described, and is much more liable to go wrong. If the oil be added too quickly, in nine cases out of ten it ruins the operation, and the spoiled materials can only be used up by starting afresh, and when the familiar crackling sound is heard, adding them very cautiously to the concentrated emulsion.

A modification of the above process consists in adding the oil and water in equal proportions, but alternately, to the thick mucilage. The result is practically the same as the above, but requires even longer for its accomplishment.

In the hands of the author the best and most uniform results have been obtained by method (*a*). Beginners generally make the mistake of triturating the oil and gum too long before adding the water. As a matter of fact, the less the oil is rubbed with the gum, so long as it is properly mixed, the better will be the result.

Tragacanth.—Tragacanth has been recommended by some, as superior to all other emulsifying agents, but by the majority of practical pharmacists it is rarely used. Emulsions prepared with this gum, have perhaps less tendency to separate owing to their viscidity, but under the microscope the globules of oil are seen to be much larger than they would be if emulsified with acacia. The following proportions produce moderate results, but the author does not recommend the process:

Fixed oil	10 parts.
Tragacanth...	½ part.
Rectified spirit	½ ,,
Water	10 parts.

Place the gum and spirit in a perfectly dry bottle of about twice the capacity of the emulsion, and mix by shaking; add half the water all at once and shake briskly; finally add the oil, and after further agitation, any flavouring agent. Volatile oils require double the quantity of tragacanth.

Linseed-gum.—As is well known, linseed contains a large amount of gum, which may be obtained by precipitating a strong decoction with 90 per cent. alcohol. A flocculent white precipitate falls, which may be obtained in a fairly pure state by washing with alcohol. It dries to a light grey mass, soluble in water, two grains of which are sufficient to emulsify 30 grains of fixed oil. Emulsions prepared with it are said to be similar to those prepared with gum arabic.*

Irish moss gelatin.—Irish moss contains about 90 per cent. of mucilage, which when evaporated, is identical with Carrageen Gelatin of the German Pharmacopœia. It is obtained by treating the plant in a steam-jacketed percolator with water, and evaporating the clear mucilaginous liquid to dryness at a low temperature. Excellent emulsions can be prepared with this mucilage, the method employed being in no way different from that followed when gum arabic is used. The gelatin must, however,

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* 'C. and D.,' Sept., 1888.

be used sparingly, as one part dissolved in sixty parts of water gelatinises on cooling.

For emulsifying fixed oils and oleo-resins the following proportions may be taken:

Irish moss gelatin	1 part.
Water	400 parts.
Oil...	400 „

Casein.—The use of casein as an emulsifying agent has been strongly recommended by Leger, but has not received much application in this country. When milk is exposed to the atmosphere it becomes acid and separates casein (curds). The same result is obtained by the addition of rennet or dilute acids. For pharmaceutical purposes casein may be obtained from poor cheese or milk. Leger's process is practically as follows:—One gallon of skim-milk is warmed to 100° F., two fluid ounces of solution of ammonia added, and the whole, after well mixing, set aside to separate. The lower layer is drawn off and acidified with acetic acid, when the casein separates. After well washing with tepid water it is collected on a calico strainer and strongly pressed to get rid of moisture. The casein is next mixed with 10 per cent. of sodium bicarbonate, and the whole made up with sugar to 100 parts. The resulting pasty mass is spread out in a thin layer, and dried at a temperature not exceeding 90° F.

Emulsions of the fixed oils are made by rubbing 10 parts of the saccharated casein with 3 parts of water, adding by degrees 10 parts of oil. After thorough trituration the mixture is diluted to the required extent with water. Resinous substances are not so readily emulsified by casein as by gum arabic, but fairly stable preparations may be turned out by dissolving the resin in a little alcohol before adding to the casein mucilage. M. Leger claims that casein emulsions are more readily digested than the corresponding ones prepared with gum, and indeed this seems only reasonable.

Yolk of egg.—The use of yolk of egg for emulsifying oils, is of great antiquity, and for certain purposes cannot be superseded. The yolk of an ordinary egg is equal in emulsifying power to about 2 drachms of gum arabic or 15 grains of tragacanth.

To obtain the yolk, the shell is broken by a smart blow on the edge of a tumbler or measure, and by careful manipulation the albumen separated as nearly as possible. After rubbing the yolk smooth, the oil is added by degrees, stirring constantly, a little water being added from time to time as the emulsion thickens. Well-made egg emulsions bear the addition of acids exceedingly well, an excellent example being the "white oils" so popular in certain rural districts.

Extract of malt.—Perhaps one of the most important pharmaceutical preparations of the present day is emulsion of cod-liver oil with malt extract. The operation of mixing, however, rarely succeeds in the hands of the retail pharmacist on account of the tedium of the operation. For this reason some have endeavoured to shorten the process by adding a strong tincture of quillaia to the malt, or even powdered gum arabic. These additions are not necessary and are not to be recommended. The most successful results are obtained by mixing the oil and malt in the proportion 1 to 4; the ingredients are placed in a mixing machine and the beaters revolved at a speed of about twenty revolutions a minute for six hours, during which time, air is excluded as much as possible.

Quillaia.—The inner bark of *Quillaia saponaria* contains a glucosidal principle (saponin) soluble in alcohol and hot water, which possesses remarkable emulsifying properties. The most useful form for employing this agent is a strong decoction, prepared by exhausting the coarsely bruised bark with boiling water, evaporating over a water-bath, and adding 25 per cent. of alcohol when cold. One fluid part of the finished preparation should represent one part by weight of bark.

It is not known whether preparations of quillaia possess the same medicinal properties as other drugs containing saponin—senega, for instance,—and for this reason, although its physical properties are so well known, it is but rarely used. As regards its strength, one part of strong decoction made as above, is sufficient to emulsify ten parts of fixed oil or three parts of volatile oil; emulsions prepared with this agent remain permanent on the addition of either alkalies or acids.

Gelatin.—Gelatin is not infrequently used for emulsions. The most convenient form for keeping it is glyco-gelatin:

Glycerin	5
Gelatin	10
Water	50

Allow the gelatin to soften in the water, dissolve by heat, add the glycerin, and strain through muslin.

One part of this mixture will emulsify its own weight of fixed oil.

The great drawback to gelatin, Irish moss, and linseed-gum emulsions, lies in their proneness to decomposition. Chloroform is probably the best preservative agent, but of course it must only be used with the sanction of the prescriber.

Emulsification of special drugs.—The following formulæ have been well tested and can be thoroughly relied upon.

Fixed oils.—Take of—

Castor oil	1 fluid ounce.
Powdered gum arabic	2 ~~fluid~~ drachms.
Syrup	4 fl ,,
Water	sufficient to produce 3 fl. ounces.

Place the gum in a dry mortar, triturate with the oil until *just* mixed; add 6 drachms of distilled water, and stir until a thick creamy emulsion is formed; dilute gradually to 2 ounces, and add the syrup previously mixed with the remainder of the water.

Essential oils.—Take of—

Turpentine	1½ fluid drachms.
Powdered tragacanth	10 grains.
Syrup	4 fluid drachms.
Water	sufficient to produce 3 fluid ounces.

Introduce the tragacanth into a *perfectly dry* bottle, add the oil and shake gently; add all at once half the water; shake vigorously, and when thoroughly emulsified, the syrup mixed with the remainder of the water.

Chloroform or bromoform.—Take of—

Chloroform	30 minims.
Almond oil	4 fluid drachms.
Powdered gum arabic	1 drachm.
Powdered tragacanth	10 grains.
Water	sufficient to produce 4 fluid ounces.

Mix the gums in a dry mortar; add the chloroform and oil previously mixed, and triturate gently; add all at once an ounce of water and stir rapidly; gradually add the remainder of the water, and finish off as quickly as possible.

Chloroform (alternative formula).—Take of—

Chloroform	30 minims.
Strong Dec. Quillaia	1 fluid drachm.
Rectified spirit	2 fluid drachms.
Water	sufficient to produce 4 fl. ounces.

Mix the chloroform with the spirit and decoction, add half the water and shake; afterwards add the remainder of the water.

Salol.—Take of—

Salol	2 drachms.
Almond oil	4 fluid drachms.
Powdered acacia	2 „
Syrup	4 „
Water	sufficient to produce 6 fluid ounces.

Melt the salol in a porcelain dish with the oil; pour on to the gum contained in a warm mortar; add one ounce of water at 150° F., and triturate until a creamy emulsion is produced; dilute gradually to 4 ounces, and add the syrup mixed with the remainder of the water.

Balsam of Tolu.—Take of—

Balsam of tolu	1 drachm.
Rectified spirit	4 fluid drachms.
Powdered gum arabic	3 drachms.
Syrup	4 fluid drachms.
Water	sufficient to produce 4 fluid ounces.

Heat the tolu balsam in a porcelain capsule, add the spirit and dissolve; place in a mortar with the gum, and add all at once half an ounce of water; stir briskly until a good emulsion is formed, when it may be gradually diluted to 2 fluid ounces. Add the syrup dissolved in the remainder of the water.

Lanolin.—Take of—

Lanolin	2 drachms.
Powdered curd soap	1 drachm.
Water	sufficient to produce 4 fluid ounces.

Place the lanolin in a warm mortar, and when melted add the powdered soap; mix and add at once 2 fluid drachms of *hot* water; triturate briskly, and add the remainder of the water by degrees.

Spermaceti.—Take of—

Spermaceti	2 drachms.
Yolk of one egg.	
Syrup	4 fluid drachms.
Water	sufficient to produce 4 fluid ounces.

Powder the spermaceti with a drop of almond oil, add the syrup, and rub into a perfectly smooth paste. Now triturate with the yolk of egg and dilute gradually with the water.

Cocoa butter, waxes, spermaceti.—Take of—

Cocoa butter	1 drachm.
Almond oil	1 fluid drachm.
Powdered gum arabic	1 drachm.
Distilled water	2 fluid ounces. Mix.

Make a mortar hot with boiling water. Wipe perfectly dry and introduce the fat; as soon as it melts add the oil and gum, and triturate gently. Now add at once half an ounce of boiling water and stir briskly. Gradually add the remainder of the water (warm).

Extract of male fern.—Take of—

Liq. extract of male fern ...	20 minims.
Fresh milk	1½ fluid ounces. Mix.

Gum-resins, such as ammoniacum and myrrh, require powdering and moistening with water. Brisk trituration then yields a very good emulsion, which only requires straining. In this case, the natural gum present, is sufficient to emulsify the resin without further addition.

Resinous tinctures are readily emulsified with a little fresh mucilage, equal parts being sufficient as a general rule. Mix the mucilage with a little water, pour into the bottle, and rotate so as to coat the inside and prevent the resin adhering to the glass. Now by degrees add the tincture, agitating *gently* after each addition.

Seeds.—Certain seeds, such as almonds, form excellent emulsions. The almonds are first blanched by means of hot water, and powdered very finely. A *little* water is next added and the whole beaten into a stiff paste. After well working, water is added by degrees, and the finished emulsion strained through muslin.

General remarks.—No mention has been made on the use of alkalies in the preparation of emulsions. The omission is intended, as the indiscriminate use of solution of potash for making emulsions, frequently defeats the intentions of the prescriber. When fats and oils are brought into contact with solutions of the alkali metals, *soaps* are formed, which may or may not present the appearance of milky emulsions. Soap itself is frequently made use of to suspend tarry, and other matters, for outward application. Thus—

Soft soap	4 drachms.
Stockholm tar...	4 ,,
Rectified spirit	8 fluid drachms.

Dissolve the soap in the spirit and mix with the tar. On pouring the clear mixture into water, an excellent emulsion is formed, which persists for a considerable time.

In the preparation of emulsions, a good-sized mortar, provided with a large flat-bottomed pestle, should be selected. As little pressure as possible should be used, a light hand always producing the better emulsion, other things being equal.

CHAPTER XLVII

PILLS

Pills are small ovoid or spherical masses of medicament, intended to be swallowed whole, without previous mastication. Their weight may vary from one to five grains, but occasionally, when the medicament is very dense, they may weigh as much as ten grains without being abnormally large. Very small pills are sometimes called *granules,* but it is a misuse of the word, and is to be deprecated as likely to lead to misconception.

A practical acquaintance with the art of pill-making, both extemporaneously and in bulk, is of the first importance to the pharmacist, as the preparation of a good pill mass requires considerable judgment, as well as manipulative dexterity. It is a matter of regret, that owing to the strenuous endeavours of manufacturers of factory-made products to press their wares on physicians and public alike, the legitimate opportunity for practising the art of pill-making appears in danger of extinction.

Pill apparatus.—For the preparation of small quantities of pills at the dispensing counter, the mortar and pestle used for massing the ingredients should be composed of Wedgwood ware, somewhat shallow, with well-rounded edges. Many pill mortars are provided with a spout, a useless and inconvenient addition. The pestles supplied with the mortars are as a rule much too short, besides having tapering handles ; it is found by experience that the handle should not be less than 8 inches in length, almost cylindrical, with a well-rounded end so as to fit comfortably into the palm of the hand when kneading, as Fig. 198. Larger mortars are frequently made of bell-metal or iron, as Fig. 199 ; some of the older patterns are often found beautifully moulded. To assist in kneading the mass, as well as to prevent loss of material when pounding a dry substance, these metal mortars are provided with stout wooden covers, as Fig. 200.

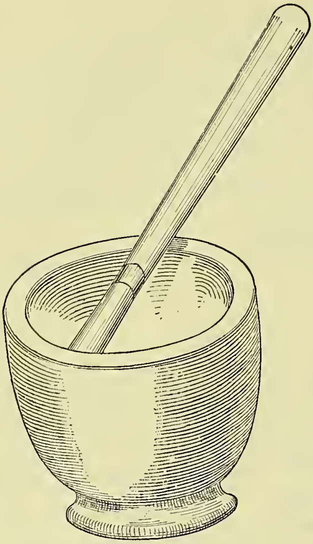

Fig. 198.—Pill mortar.

Fig. 199.—Iron mortar.

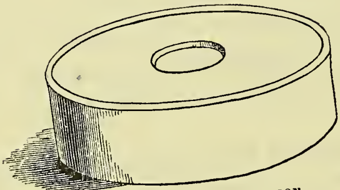

Fig. 200.—Mortar cover.

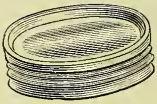

Fig. 201.—Pill rounder.

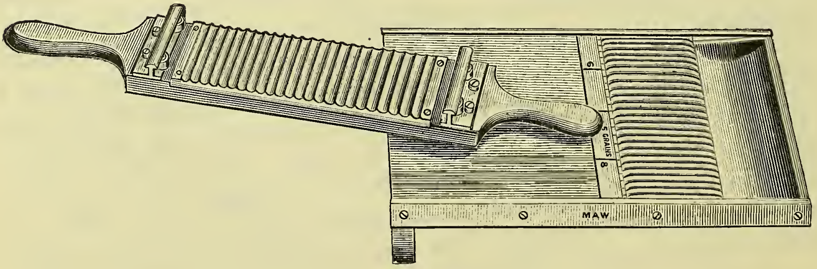

Fig. 202.—Pill machine.

A good operator can frequently manipulate a pill mass so deftly that scarcely any of the material will finally adhere to the bottom of the mortar ; but as *during* the operation the mass generally requires loosening, special knives are employed, having short stiff blades. These, again, should have well-rounded handles, to enable them to be used with comfort. The machine for piping and dividing the mass into pills is usually made in mahogany, with brass runners and cutting surfaces, as Fig. 202. In cutting pills by hand, it is necessary that the machine should rest on a firm bench or counter, as considerable force is exerted in rolling hard masses.

The pills as a rule are not perfectly round when they leave the grooves, and to finish them off they are rolled in an eccentric manner beneath a rounder, Fig. 201, made in either boxwood or metal, a little starch being used to prevent them sticking.

The advantage of using a metal pill finisher is that it can be warmed over a Bunsen burner for very hard pills. Many pharmacists make use of a thick slab of iron, heated on a gas flame, for softening very hard pill masses. It should not be resorted to where heat is likely to injure the ingredients. Fig. 203 shows Pindar's kneading machine for small masses. In the figure there is a device for keeping the bed-plate warm, which can also be utilised for rounding the pills.

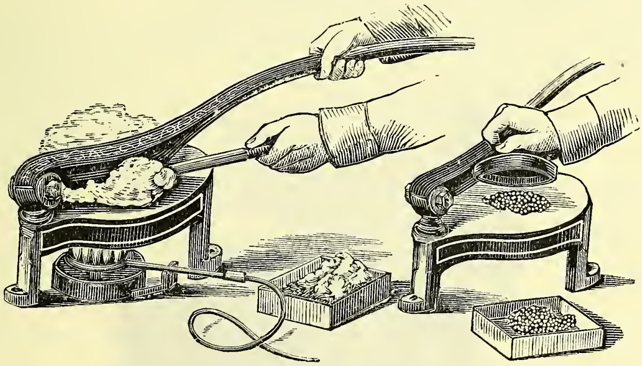

Fig. 203.—Kneading machine.

For the production of pills on a larger scale than that hitherto considered, the ingredients are mixed and massed in kneading machines actuated by hand or power, similar to Figs. 204, 205. When sufficiently worked, the mass is placed in the cylinder of the piping machine and pressed through a

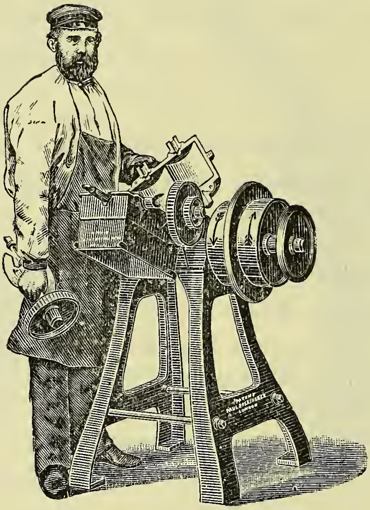

FIG. 204.—Werner & Pfleiderer's patent kneading machine.

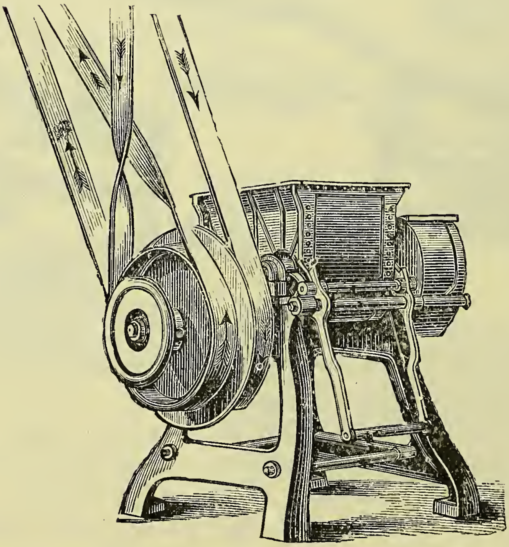

FIG. 205.—Enlarged view showing the machine closed ready for use.

series of holes in the bed-plate, which is made very thick and massive. Great pressure is brought to bear on the plunger,

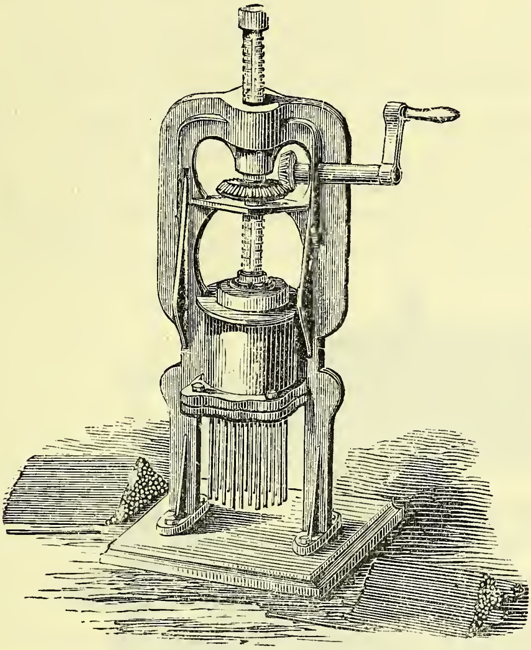

FIG. 206.—Double-action piping press.

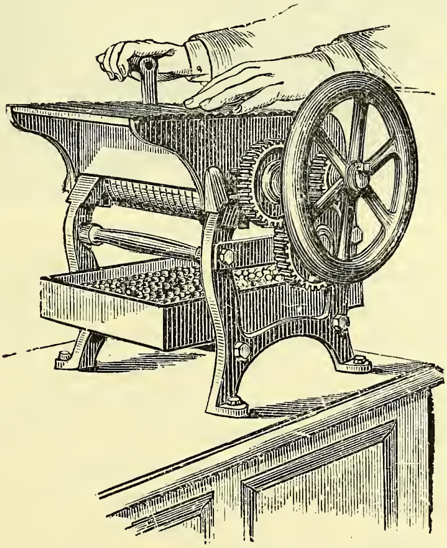

FIG. 207.—Pindar's rotary pill-cutting machine.

by a worm and wheel action similar to that made use of in the construction of double-action tincture presses, Fig. 206. The piped mass is then divided into pills by a rotary machine as Fig. 207. The cutting parts consist of a pair of grooved metal rollers, revolving towards one another at different speeds. When the pipe is laid between the rollers it is drawn in and instantly divided into pills, the weight or size varying with the size of the pipe and the cutting grooves. As the pills leave the rollers they drop into a tray fixed on the frame of the machine. The final rounding is also accomplished by machinery (Fig. 208). The rounder illustrated, takes four ounces of pills at one time and rounds them perfectly in about a minute. The motion is a most ingenious imitation of the manual operation of rounding, the pills being placed between two metal plates, one of which travels to and fro, while the other is endowed with an eccentric motion, the pressure being adjusted according to requirement. When the pills leave the machine, the imperfect ones are separated from the rest by shaking in a zinc tray perforated with holes, which allows all the pills to fall through, except the large ones; they next fall on to a tray with smaller holes, which retains all the pills of the desired size, but allows

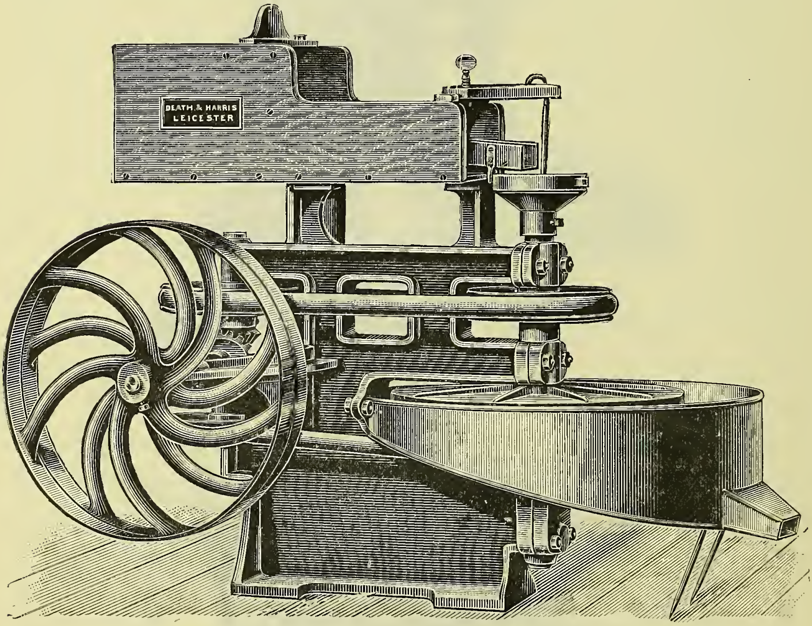

FIG. 208.—Pill-rounding machine.

the small ones to drop through into another tray placed underneath. The larger and the smaller pills are then returned to the kneading machine, re-piped and cut.

Preparation of the mass.—The pill mass may be divided into two parts,—the active ingredients, and the excipient or material used to bind the mass and give it proper consistence. The chief characteristics of well-made pills are their uniformity of dose and size, as well as their ready solubility when introduced into the system. For the production of pills complying with the above conditions the ingredients must be thoroughly mixed before addition of any excipient. Potent remedies, such as the toxic alkaloids, should be carefully diluted with a little milk-sugar before adding to the bulk of the material. It occasionally happens that a fraction of a grain of some energetic remedy is ordered in a pill, which it is impossible to weigh directly. When such quantities occur, the substance may be mixed with milk-sugar in such proportions that a convenient quantity can be weighed off. Thus if 24 pills are required, each containing $\frac{1}{30}$ of a grain of strychnine, one grain of the alkaloid should be intimately mixed with 9 grains of diluent, and 8 grains of the mixture taken, representing $\frac{8}{10}$ or $\frac{24}{30}$ of a grain. Or it may happen that 24 pills are ordered, each containing $\frac{1}{25}$ grain of the active ingredient. In this case it may be more convenient to weigh one grain, afterwards dividing the mass into 25 pills, one of which may be thrown away. One of the objects in pill making should be to keep the pill as small as possible, but no pills should ever be sent out weighing *less* than one grain.

To avoid the danger of adding too much excipient at one time, making the pills either too large or too soft, liquid excipients should be kept in 1-ounce bottles, into which a small pipette attached to a rubber ball just dips ; then by simply squeezing, as small a quantity as desired may be dropped on to the ingredients. Powders may be kept in wide-mouthed bottles provided with a perforated cork, in which a short piece of quill is inserted.

After each addition of excipient the mixture is detached from the side of the mortar, and well kneaded until an uniform plastic mass results. Before rolling, the gross weight of the mass should be entered in the prescription book. This rule should never be omitted, as errors in weighing the ingredients may sometimes be detected, while it ensures that the pills are always sent out of the same size and weight.

Pill excipients. — Under this name are comprised all the materials used to bind the ingredients into a mass. In selecting liquid excipients, care must be exercised that none are used that will act as complete solvents of the materials, nor any powders likely to form insoluble compounds with the ingredients. Although special excipients, have been recommended from time to time for almost every medicinal substance used in medicine, it is generally found that practised dispensers rely on one or two only, with the behaviour of which they are well acquainted.

White powders generally, may be made into a mass with compound powder of tragacanth and a little syrup, with glycerin of tragacanth, or with glucanth. Glycerin by itself should be avoided (except for pepsin), as pills containing it are always liable to lose their shape and become sticky through absorption of moisture.

Coloured powders may be massed with any physiologically inert excipient that appears appropriate.

Acid, benzoic.—Glucanth, or a little tragacanth and simple syrup.

Acid, carbolic.—Carbolic acid in pills may be treated in several ways, one of which is to add $\frac{1}{20}$ the weight of glycerin, afterwards massing with a mixture of wheaten flour 8 parts, and tragacanth 2 parts. Another and far better method, is to mass with wheaten flour and hard paraffin. If desired the following mass, containing 50 per cent. of phenol, may be kept ready made :

Absolute phenol	60 parts.
Hard paraffin	12 „
Wheaten flour	45 „
Glucanth	3 „

Pills made from this formula readily disintegrate in lukewarm water, and no objection is likely to be raised to the presence of 10 per cent. of paraffin.

Aloes.—The best excipient for pills containing aloes in combination with extract of colocynth and similar drugs, is the official compound decoction of the same name. Its usefulness is chiefly due to the presence of the alkaline carbonate, as proof spirit is of much less value as an excipient. Owing to its solvent action, Dec. Aloës Co. must be used very sparingly, and if iron salts enter into the ingredients its use is contra-indicated. Pills composed of extract of aloes alone, should be mixed with $\frac{1}{10}$ their weight of powdered althæa and massed with rectified spirit.

Antipyrin.—With glucanth.

Bismuth carbonate and oxynitrate.—With glucanth, or compound tragacanth powder and syrup.

Calcium sulphide is usually ordered in small quantities, and should be first mixed with milk-sugar, and then massed with tragacanth and syrup, or glucanth.

Calomel.—Confection of roses has been recommended for massing calomel, but it is better to follow the rule of only using white excipients for white pills. Strained manna, or tragacanth and syrup, answer every purpose.

Camphor is often stated to give more trouble than any other medicinal agent when ordered in pilular form. This is true for certain *combinations*, as camphor with carbolic acid, &c., but camphor *per se* is not really difficult to manipulate. The secret is to powder the camphor very finely; if then, it is mixed with a little starch or wheaten flour, it may be massed with glucanth without trouble. It has been suggested to powder the camphor with the help of a little fixed oil, adding one third of its weight of powdered curd soap, finally massing with a few drops of rectified spirit. When ordered with extract of henbane or other "green" extract, the camphor if powdered finely, frequently forms a very good pill without further addition, but with equal parts of camphor and extract of henbane, the mass is too soft, and should be stiffened with equal parts of tragacanth and wheat flour.

Camphor, monobromated.—Powder finely, add one tenth the weight of tragacanth, and mass with glucanth.

Cerium oxalate.—As Camphor, monobromated

Creasote.—In the author's opinion, creasote occasions more trouble when ordered in pills than does camphor. The best plan is to keep a 50 per cent. creasote mass ready made, which may be dispensed from as required. The following modification of Martindale's formula gives very good results. To 50 parts of creasote in a wide-mouthed bottle add 15 parts of melted yellow wax, and 35 parts of powdered *curd* soap. Cork, and heat in a water-bath, shaking occasionally until combination takes place. This, when cold, gives a plastic and fairly firm mass, which only requires the addition of a little powder when dispensing. The use of light magnesia for massing creasote pills is much to be condemned, the pills becoming as hard as marbles and about as insoluble.

Crystalline salts, such as potassium and ammonium bromide, potassium iodide, &c., should after powdering very finely, be mixed with one tenth their weight of tragacanth, and massed with syrupy glucose. Glycerin in any form should never be used.

Ergotin.—See Extracts.

Extracts, such as ergotin and the "green" extracts generally, are not sufficiently firm for making into pills. It is customary, therefore, to evaporate them over a water-bath until sufficiently hardened, making a note on the label of the jar that six parts represent eight parts of the soft extract, or whatever the case may be. Purified ox-bile is best evaporated to complete dryness, and either a note of the loss registered, or else brought up to the original weight with finely powdered althæa.

Iron phosphate.—This is frequently ordered in combination with quinine and strychnine. It is customary to mass with syrupy phosphoric acid (sp. gr. 1·5). The mass should be worked and rolled rapidly, as it sets almost at once.

Iron, reduced.—Powder very finely, mix with one tenth weight of tragacanth, and mass with "glucanth."

Iron sulphate, dried.—The dried ferrous sulphate of the Pharmacopœia is of uncertain composition. It is best massed with syrup, taking care to add enough at once, or a crumbly mass results.

Lithium guaiacate.—Powder finely and make into a rather soft mass with rectified spirit. Work up and roll as quickly as possible, as the mass hardens very rapidly.

Mercury with chalk.—Hydrarg. c Cretâ requires very careful manipulation in kneading. If worked too vigorously, or if the mass is made too hard, the mercury separates. Extract of gentian is an excellent excipient. Wheat flour and glucanth also make a good pill.

Oil, croton.—As essential oils.

Oils, essential.—When considerable quantities of essential oils are ordered in pills, the best results are obtained by massing with a mixture of equal parts of powdered curd soap, calcium phosphate, and wheaten flour. If absolutely necessary, a small proportion of yellow wax may be melted with the oil, but must not exceed one tenth the weight, or probably the pills will not disintegrate.

Pepsin is readily massed with glycerin and a trace of tragacanth. If the pills are made with what is known as soluble pepsin they have a tendency to absorb moisture and become sticky; in that case glycerin must not be used, but syrup.

Phosphorus.—When small doses of phosphorus are ordered, it may be possible to use an equivalent quantity of the official mass; but if this would unduly increase the bulk, an equivalent quantity of 10 per cent. phosphorated stearin. Prepare some fresh mutton suet according to the pharmacopœial directions, and allow to cool. Now wrap the fat in several folds of white blotting-paper and then in calico, place in a press, and express strongly. Repeat the operation with fresh paper until the stearin is obtained fairly pure. Its melting-point should be about 122° F. Take of—

Phosphorus	10 grains.
Carbon bisulphide	1 fluid drachm.
Stearin	90 grains.

Dissolve the phosphorus in the carbon bisulphide in a bottle, add a little of the stearin and shake well; then add the remainder, mix thoroughly, allow the bisulphide to evaporate, and preserve under water.

This basis contains 10 per cent. of phosphorus. It is more plastic than phosphorated suet, and makes firmer pills.

Amorphous phosphorus is a red powder insoluble in bisulphide of carbon. It is unsafe to use for medicinal purposes unless absence of crystalline phosphorus is ensured by previous extraction with the above-named solvent. After extracting and drying, it may be made into pills with compound tragacanth powder and syrup.

Potassium permanganate for obvious reasons must not be massed with any readily oxidisable excipient. Very good pills may be made with Martindale's kaolin ointment, or if not at hand with what amounts to the same thing, Unguentum Paraffini, B.P., and fuller's-earth. Lanolin has also been recommended, but the author does not find it possesses any advantage over the excipients mentioned, or even over resin ointment.

Quinine and iron citrate.—Powder finely, add one twentieth of powdered tragacanth, and mass with syrup, or with the official Unguentum Paraffini. Many dispensers prefer to moisten the powdered scales with proof spirit, in which case the mass must be kneaded and rolled out as quickly as possible. Under no circumstances must any preparation of glycerin be used.

Quinine sulphate may be massed with glucanth or tragacanth and syrup.

Salol is more frequently prescribed in tablets or cachets than in pilular form. It may, however, be made into pills with glucanth. The mass should not be worked too vigorously, as salol melts about 108° F., and is then somewhat intractable.

Silver nitrate should be finely powdered with white fuller's-earth and massed with Ung. Paraffini, B.P. Bread crumb should never be used, as it not only yields unsatisfactory pills, but contains sodium chloride, with which silver nitrate is incompatible.

Silver oxide.—As for silver nitrate.

Sulphonal, tetronal, trional.—As for salol.

Sulphur.—When sulphur is ordered in pills, either alone or with other ingredients, only the "washed" variety should be used. This, if finely powdered, gives no trouble with the ordinary excipients.

Tar pills are now seldom prescribed, but may be prepared by adding one fifth the weight of melted yellow wax to the tar in a warm mortar; a mixture of equal parts of lycopodium and wheaten flour may be then incorporated until the mass is sufficiently firm for rolling into pills.

Zinc oxide.—As cerium oxalate.

Zinc valerianate, if ordered alone in pills, should be reduced to fine powder, one twentieth of powdered acacia added, and massed with glucanth. It is, however, more generally prescribed with asafœtida and other resins, when the mixed powders only need slightly moistening with rectified spirit to produce an excellent mass.

Classified List of Excipients.

Acacia powder and mucilage. Avoid as much as possible, as pills containing gum become exceedingly hard.

Alcohol for resinous pills.

Althæa in fine powder. Useful as an absorbent; has tendency to make pills somewhat spongy.

Bread crumb.—Useless.

Calcium phosphate.—Either alone, or mixed with wheaten flour, for essential oils.

Confection of roses has nothing to recommend its use over many other more reliable excipients.

Decoction of aloes, compound.—Useful on account of the carbonate of potassium it contains. May be used for most pills containing aloes. Incompatible with iron salts.

Elm bark in fine powder is used for the same purposes as powdered althæa.

Fuller's-earth.—Useful to give firmness to such pills as potassium permanganate, &c.

Glucanth.—A mixture of—

Tragacanth	½ ounce.
Glycerin	1½ ounces.
Water	½ ounce.
Syrupy glucose	3½ ounces.

This excipient is useful in many instances where glycerin of tragacanth is unsuitable on account of the large proportion of glycerin it contains.

Glycerin should be avoided as far as possible, especially for pills intended to be silvered or coated with either gelatin or chalk.

Liquorice powder is a most useful general absorbent.

Curd soap.—Useful in massing oils, creasote, &c.

Tragacanth.—Both the simple and compound powders are invaluable for binding friable, and giving firmness to soft masses.

Tragacanth, glycerin of.—One of the most generally useful excipients.

Water.—Use sparingly.

Wax.—Use sparingly. See Creasote and Tar pills.

Before sending out pills it is customary to either coat them or add a little dry powder, such as liquorice, lycopodium, starch, or French chalk. By far the most elegant method is to cover the pills with some innocuous and soluble coating, such as sugar or gelatin.

Sugar Coating.

Sugar coating is an operation which, if the best results are looked for, can only be conducted on a large scale, partly because the art requires considerable skill and experience, and partly because the deposit of sugar only becomes uniform from the mutual attrition of a large bulk of pills in constant motion. The pills, in the first instance, should be varnished with a *very thin* coating of tolu or sandarach, and allowed to become perfectly dry. A convenient number are then placed in the pan, a few drops of mucilage added, and the machine started. When quite dry a little warm syrup of the sp. gr. 1·40 is added, taking care not to add too much at one time, or the pills will aggregate into a mass and be difficult to separate again afterwards. Give four or five charges of syrup, increasing the quantity slightly each time, but let each charge become quite dry before adding another ;

finally give a dust with wheaten flour and take out of the pan. Separate the pills from the powder in a hair sieve and wash out the pan. Put the pills back in the pan and repeat the addition of syrup for five or six more charges, then give a dust of flour and sift as before. Repeat, cleaning out the pan every five or six charges until a fairly uniform coating has been attained. The pills should then be placed in a drying cupboard at a temperature of not more than 80° F. for a day, after which the operation is repeated until the coating has become sufficiently thick. At the last the pills are put into a perfectly clean pan with a piece of hard paraffin, and rotated briskly to give them a final polish. It will be found that the syrup does not dry very quickly unless means are taken to warm the pan; this may be done by placing a spirit lamp or Bunsen burner in such a position that at each swing of the pan it passes over the flame. When coating pills on a large scale the operation is performed in a pan provided with a steam jacket, a current of dry air being at the same time projected into the pan by a mechanical blower (Fig. 209).

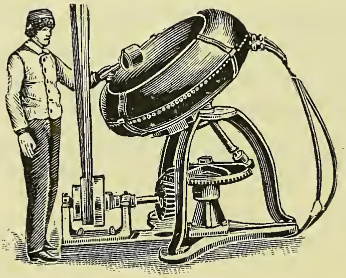

FIG. 209.—Brierley's sugar-coating pan with steam coil.

For extemporaneously coating small quantities of pills with sugar, such as two or three dozen, the following process may be adopted. Moisten the pills slightly but evenly with this mixture:

Sandarach pill varnish	1 part.
Mucilage of acacia	2 parts.
Simple syrup	4 ,,

and throw into a covered pot containing a mixture of finely powdered sugar 7 parts, starch 1 part. Rotate rapidly for a few seconds, and turn out on to a hair sieve; separate the powder and rotate gently on the sieve until excess of powder has been rubbed off. Now place in another clean warm pot and rotate until perfectly dry. Repeat the operations until the pills have acquired three or four coatings, when they may be left on the sieve to dry. Finally they may be polished with paraffin as before described.

Gelatin coating is perhaps one of the best methods yet discovered for masking the taste and odour of nauseous pills. No expensive apparatus is necessary for its application, while the pills are exposed to undesirable conditions of temperature and moisture, for the minimum of time. The coating is, moreover, both soluble and transparent.

The selection of the excipients for massing pills intended to be gelatin-coated requires considerable experience, as it is important that no hygroscopic substance such as glycerin be used. The pills are first shaken in a bag of soft material to free them from adhering powder; they are then impaled on long slender needles, which may be fixed in corks, and dipped in a solution of gelatin kept at a temperature of about 80° F. After withdrawal they are rotated gently while the gelatin sets, and left to dry. The gelatin solution may be prepared as follows:

Gelatin	2 parts.
Water	15 ,,
Mucilage of acacia	1 part.

Allow the gelatin to soak in the water until softened; dissolve by heat of a water-bath; add the mucilage, strain, and make up the weight to 18 ounces. When nearly cool stir in 10 drops of chloroform.

For white pills the solution must be as nearly colourless as possible, but for dark pills a little caramel may be added with advantage.

Figs. 210, 211, and 212 illustrate Niblett's unique pill-coating apparatus. The first figure shows the dipper with

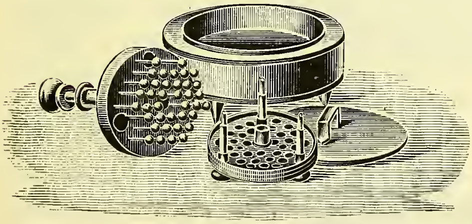

FIG. 210.

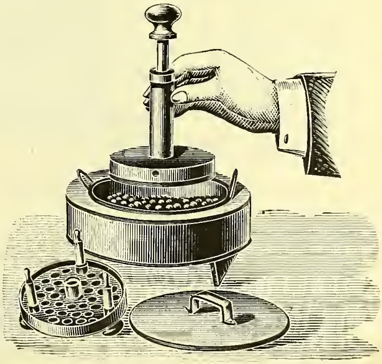

FIG. 211.

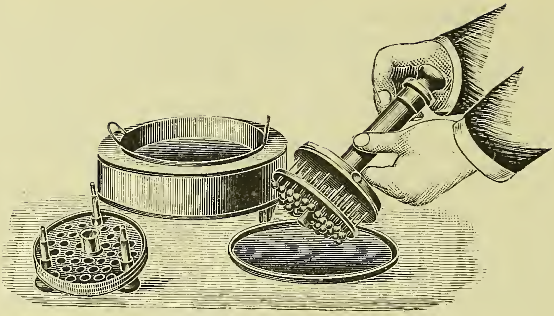

Fig. 212.

pills fixed on the needles. This is accomplished by means of the base or pill receiver. Each recess accommodates a pill, and as the needles on the dipper correspond in position with these recesses, the pills are taken up simultaneously and perfectly true. Fig. 212 shows the process of dipping. The superfluous gelatin is got rid of after withdrawing the dipper, by lowering it again until the lower side of the pills merely touches the surface of the liquid, after which the dipper is lifted out and rotated gently for a few seconds, when it is placed on its side until the pills are dry. Fig. 212 represents the action of expelling the pills from the needles. This is done with a sliding plate actuated by a spring, the whole of the pills being detached at once. After removal of the pills from the needles, it is sometimes found, when the pills have been dipped too deeply, that a little gelatin tube projects from the side of each. This should be cut off with a pair of sharp scissors before sending out.

Pearl coating is largely made use of in this country, and if properly done imparts a very pleasing appearance to the pills. The disadvantage is that the coating is quite insoluble, and cases are not rare where pills so coated have passed unchanged through the system. For coating small quantities extemporaneously at the dispensing counter, the perfectly round and hard pills are varnished with a very thin covering of sandarach or tolu. They are then damped evenly with a mixture of—

Mucilage of acacia	2 parts,
Syrup	2 ,,
Sandarach varnish	½ part,

and thrown into finely powdered talc and shaken vigorously for a few moments. After sifting out the excess of powder, the pills are transferred to a perfectly smooth and clean covered pot, or a metal coater similar to Figs. 213, 214, or 215, and rotated until

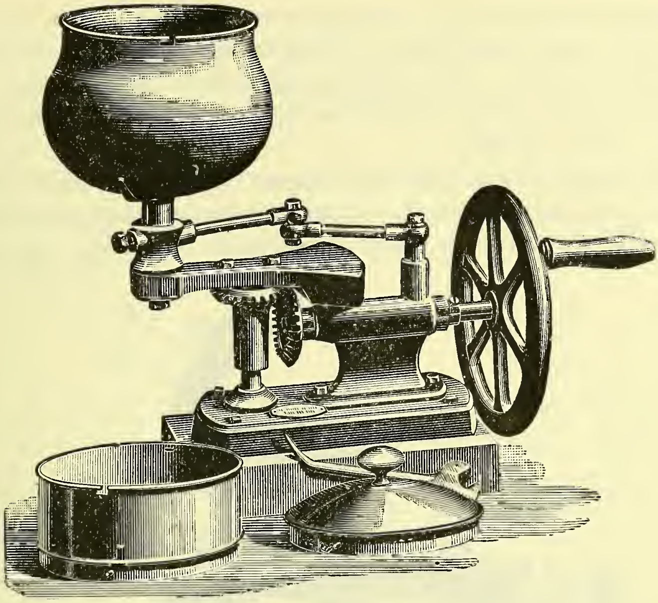

Fig. 213.—Pindar's eccentric pill coater.

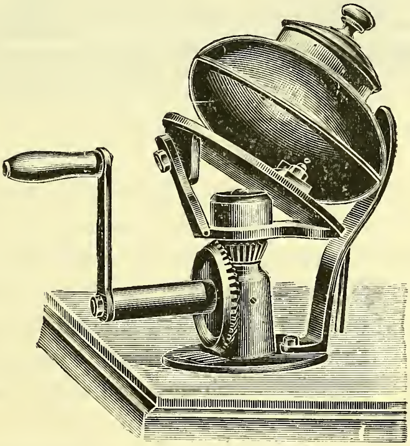

Fig. 214.—Niblett's oscillating pill coater.

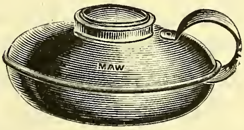

Fig. 215.—Maw's hand-coater.

smooth and fairly dry. After exposure to the air for a short time a second coat is applied in a manner similar to the first, and if necessary a third. The pills should then be allowed to dry perfectly, when they may be polished by rotating in a clean pan with a ball of hard paraffin.

For coating stock pills on a larger scale, the method of procedure is slightly different. After varnishing, the pills are introduced into the pan of a machine similar to Fig. 216. A mixture of sugar and mucilage is then sprinkled on and the pills rotated until evenly moistened; white talc is then added, and the motion continued until uniformly coated. Alternate additions of solution and powder are then made until a sufficiently thick coating has been put on, when after drying the pills are polished in a clean pan with hard paraffin. In the machine illustrated, an oscillating and eccentric motion is given to the central shaft carrying the pan. The effect of this motion causes the pills to revolve without jerking in a continuous stream round the sides of the pan, thereby ensuring the maximum amount of attrition.

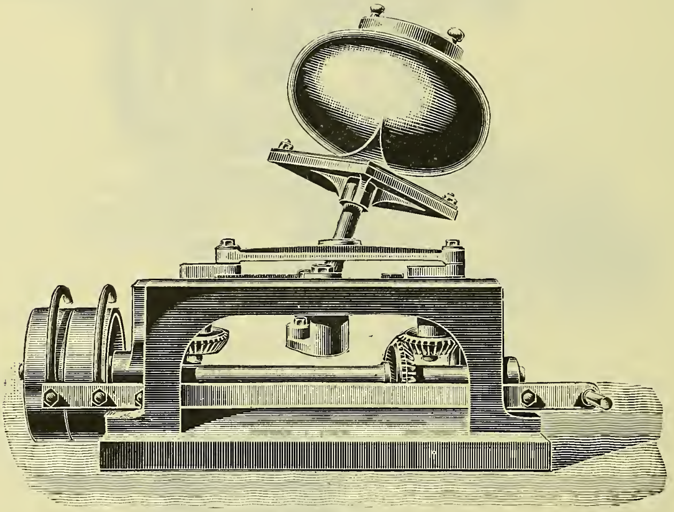

FIG. 216.—Niblett's pill coater.

Keratin coating is made use of, not so much to give the pills a finished appearance, as to protect them during their passage through the stomach,—keratin being insoluble in the gastric juice, so that solution of the pill will not take place until the intestine is reached. The process for keratin-coating is very tedious, but otherwise presents no special difficulty. The pills, which should be massed with Ung. Petrolei or Ung. Resinæ, are placed in a dish with a little of the keratin solution, and rolled about until evenly coated, when they are turned on to a porcelain tray and allowed to dry. At least three coatings should be given to be of any service.

The keratin employed for the solution may be bought in the market and dissolved as directed, or it may be made by digesting horn shavings which have been freed from fat by ether, in a mixture of—

Pepsin	4 parts,
Hydrochloric acid	1 part,
Water	95 parts,

for several days. The residue is then washed with water and dissolved in either solution of ammonia or acetic acid. In Dieterich's modified formula, goose quills, chopped in small pieces, are first treated with ether to remove fat, and then dissolved by boiling with strong acetic acid in a flask fitted with a reflux condenser. The solution is then evaporated and spread on glass plates to dry.

Salol has been proposed as a substitute for keratin coating when the pills are not intended to dissolve in the stomach, but its utility has been questioned. It may be applied by dipping the pills, fixed on needles, into melted salol, allowing to cool, detaching, and closing the needle-holes with a camel-hair pencil dipped in the melted substance.

Varnishing.—For the extemporaneous coating of pills no method has yet been introduced to oust varnishing, and with a little care, excellent results are attained with the least expenditure of time. The pills, as for all coating operations, should be round and firm, and free from any adhering powder; they are first placed in a covered pot, a few drops of the varnish added, well shaken for a few moments, and rapidly turned out into a porcelain tray to dry. The most convenient trays for the purpose are the developing dishes used by photographers. Either of the following varnishes may be used; both remain bright when dry, but the second dries the quicker:

Martindale's form.—

Sandarach	1 part.	
Absolute alcohol	1 „	Dissolve.

John Bell and Co.'s form.—

Sandarach ... ... ··· ...	4½ parts.	
Methylated chloroform...	4 „	
„ ether, sp. gr. ·717... ...	10 „	Dissolve.

For dark-coloured pills a varnish made by dissolving the residue left in the manufacture of syrup of Tolu may be used. The proportions are—

Tolu residue	6 ounces.
Methylated ether, sp. gr. ·717	8 fluid ounces.
„ chloroform	4 „

Silvering.—Covering pills with gold or silver leaf was one of the earliest methods of coating adopted, and silver leaf is still employed for the same purpose. The pills are slightly moistened with very thin acacia mucilage, and thrown into a covered pot or wooden coater (Fig. 217) containing silver leaf and rotated vigorously for a minute or so. They are then exposed to the air for a little time to dry, and afterwards burnished by rotating in a second clean pot. As a rule, one leaf of silver suffices for covering one dozen 5-grain pills. Pills containing sulphur or sulphides should not be silvered.

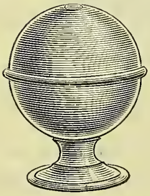

FIG. 217.—Boxwood pill silverer.

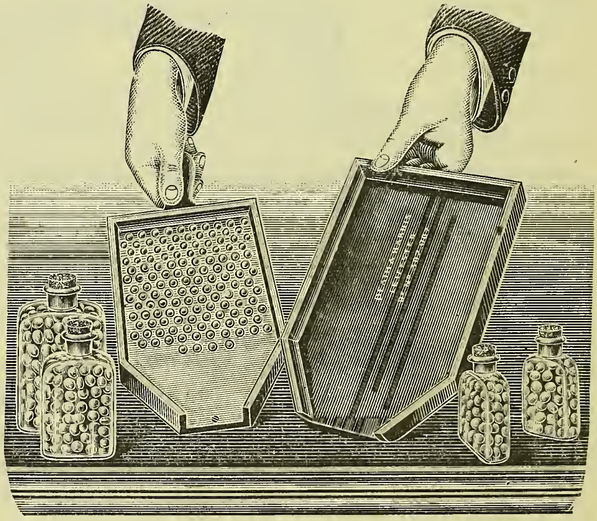

FIG. 218.

Pill counting.—When pills are required to be counted in considerable quantities, the device shown in Fig. 218 is resorted to. An excess of pills are thrown on to the counter, the apparatus shaken so as to get one pill in each hole; the remainder are allowed to run off. This leaves a gross of pills ready for emptying on to the scoop, by means of which they are transferred to bottles.

The Pharmacopœial Pill Masses

The practising pharmacist, in addition to keeping a stock of the official pills already rolled and rounded, generally finds it necessary to keep stock masses to fill the requirements of special prescriptions in which only small quantities are ordered. Thus the following prescription contains two pharmacopœial masses, with other ingredients :

℞ Pil. Coloc. Co.	gr. ij.
Pil. Rhei Co.	gr. ij.
Ext. Bellad.	gr. ¼.
Ol. Carui	♏ ½.

Now if this is dispensed as written, the mass would weigh nearly 5 grains, and be too soft to roll into pills. If sufficient excipient is added to make them hard enough to retain their shape, then the pills would be inconveniently large. To meet this difficulty it is customary to prepare "pill powders"; that is ingredients for pills, *minus* the excipients. With a stock of the following "pill powders" the dispenser is then able to cope with all ordinary prescriptions in which pharmacopœial pill masses are ordered, without difficulty :

Pulv. pro Pil. Aloës Barb. (1 = 2).—

℞ Pulv. Aloës Barb.	2 ounces.
Ol. Carui	1 fl. dr.

Misce.

Pulv. pro Pil. Aloës et Asafœtidæ (1 = 2).—

℞ Pulv. Aloës Socot.	1 ounce.
„ Asafœtidæ	1 „

Misce.

Pulv. pro Pil. Aloës et Ferri (2 = 3).—

℞ Ferri Sulph. Exsicc.	1 ounce.
Pulv. Aloës Barb.	2 ounces.
„ Cinnam. Co.	3 „

Misce.

Pulv. pro Pil. Aloës et Myrrhæ (2 = 3).—

℞ Pulv. Aloës Socot.	2 ounces.
„ Myrrhæ	1 ounce.

Misce.

Pulv. pro Pil. Aloës Socot. (1 = 2).—

℞ Pulv. Aloës Socot.	2 ounces.
Ol. Myristicæ	1 fl. dr.

Misce.

Pulv. pro Pil. Cambogiæ Co. (1 = 2).—

℞ Pulv. Cambogiæ	1 ounce.
„ Aloës Barb.	1 „
„ Cinnam. Co.	1 „

Misce.

Pulv. pro Pil. Coloc. Co. (1 = 1).—

℞ Pulv. Colocynth.	1 ounce.
„ Aloës Barb.	2 ounces.
„ Resin. Scammon.	2 „
„ Potass. Sulph.	¼ ounce.
Ol. Caryoph.	2 fl. dr.

Misce.

Pulv. pro Pil. Galbani Co. (1 = 1).—

℞ Pulv. Asafœtidæ	2 ounces.
„ Galbani	2 „
„ Myrrhæ	2 „
„ Sacchari	1 ounce.

Misce.

Pulv. pro Pil. Hydrarg. Subchlor. Co. (1 = 1).

℞ Hydrarg. Subchlor.	1 ounce.
Antim. Sulphurat.	1 „
Pulv. Resin. Guaiaci	2 ounces.
„ Sacch. Lact.	½ ounce.

Misce.

Pulv. pro Pil. Plumbi cum Opio. (1 = 1).—

℞ Pulv. Plumbi Acet.	12 drachms.
„ Opii	2 „
„ Sacch. Lact.	80 grains.

Misce.

Pulv. pro Pil. Rhei Comp. (5 = 8).—

℞ Pulv. Rhei	3 ounces.
„ Aloës Socot.	2¼ „
„ Myrrhæ	1½ „
Ol. Menth. Pip.	1½ drachms.

Misce.

Pulv. pro Pil. Scammonii Co. (1 = 1).—

℞ Pulv. Resin. Scammonii	1 ounce.
„ Resin. Jalapæ	1 „
„ Zingiberis	2½ drachms.
„ Saponis Animal.	1 ounce.

Misce.

Pulv. pro Pil. Scillæ Comp. (2 = 3).—

℞ Pulv. Scillæ	10 drachms.
„ Zingiberis	8 „
„ Ammoniaci	8 „
„ Saponis	2 „

Misce.

CHAPTER XLVIII

SUPPOSITORIES

The name suppository was originally given to solid preparations of conical shape intended for the introduction of medicaments into the rectum. Similar preparations for the vagina are now known as *pessaries*, while those for the application of remedial agents to the nose and male urethra, are termed *bougies*. As the usefulness of this class of preparations is entirely dependent upon their melting completely at the temperature of the body, great care must be exercised in the selection of the basis with which they are to be compounded. The bases in most general use are oil of theobroma, and a combination of gelatin and glycerin; others have been proposed, such as stearin, and a mixture of stearic and oleic acids; while in the 1885 edition of the Pharmacopœia, curd soap massed with glycerin of starch was employed. Of all these, oil of theobroma is by far the most useful; although melting below 94° F. it is sufficiently firm to be handled at the ordinary temperature, while its non-irritating properties pre-eminently adapt it for internal application.

The usual shape of rectal and vaginal suppositories, is that of
220 a cone with a rounded apex, as Fig. 219. Besides this shape,
221 another form has been lately introduced, Fig. 220, which is
supposed to be more readily retained in the rectum. The weight
of rectal suppositories is officially fixed at 15 grains, although
in practice they are often made to exceed it, whilst pessaries vary
from 30 to 90 grains. Pencil-shaped bougies for the nose may
weigh about 30 grains, and are usually 4 inches in length, those
219 for the male urethra being a little longer, Fig. 221.

In the preparation of suppositories, crystalline and other solids must be brought to a state of the minutest subdivision before admixture with the basis; vegetable and other extracts may be rubbed into a smooth paste with water. When oil of theobroma is used, it is cut up into small pieces and melted at as low a

Fig. 220.—Rectal suppository.

Fig. 222.

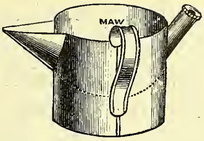

Fig. 223.—Tin suppository bath.

Fig. 219. Nasal bougie.

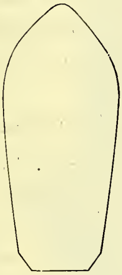

Fig. 221.—Rectal suppository.

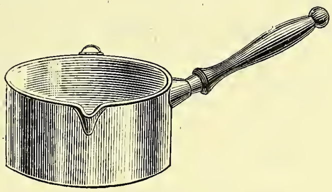

Fig. 224.

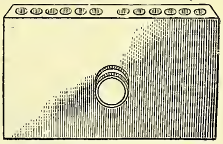

Fig. 225.—Bougie mould.

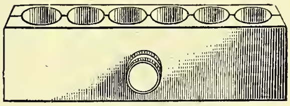

Fig. 226.—Pessary mould.

temperature as possible, a little added to the medicament and triturated until perfectly smooth. The mixture is then added to the remainder of the still liquid, but nearly cool, oil of theobroma, and thoroughly mixed, when it is poured into the moulds.

The successful preparation of suppositories is not by any means so simple an operation as appears at first sight. In the first place, no more heat should be used in the liquefaction of the oil of theobroma than is absolutely necessary; and secondly, when the mixture of medicament and basis is added to the bulk, the heat should be only just sufficient to bring the whole to the consistence of a thick cream. If this is carefully attended to, extracts and dense powders will remain in suspension until the fat solidifies; but if the temperature is allowed to rise ever so little, the medicament will, in many cases, separate and be found in a hard lump in the apex of the suppositories when they are removed from the mould. To ensure rapid solidification, the moulds should be placed on a block of ice or in cold water. To prevent adhesion of the suppositories it is customary to wipe the interior of the moulds with a plug of cotton dipped in soap liniment, the merest trace being used. If the moulds are absolutely cold before pouring there is no need for any lubricant at all, as it suffices to breathe into the moulds just prior to pouring, when the condensation of a film of moisture on the surface of the cold metal effectually prevents the suppositories sticking. The most convenient dishes for melting the ingredients are the porcelain capsules with handles, sold by the chemical apparatus makers (Fig. 222), although small water-baths of the shapes shown in Figs. 223 and 224 are sometimes preferred. Metal moulds holding 15, 20, 30, and 60 grains of water are in common use ; these open in different ways, but preference should be given to those opening vertically, so as to expose the whole length of one half of the suppositories (Figs. 225, 226).

Large quantities of suppositories are made now-a-days by cold compression. Although the finish is not quite so good as when made by casting in moulds, there is great saving of time and an entire absence of all danger from overheating. In addition, by incorporation in the cold it is possible to exhibit certain medicaments, such as chloral hydrate, which cannot be prepared by melting in the ordinary way. The suppository mass is prepared for compression, by careful admixture in a mortar. For this purpose all solid substances must be reduced to a state of impalpable powder before adding to the oil of theobroma, which is generally used in a coarsely grated condition. The machine

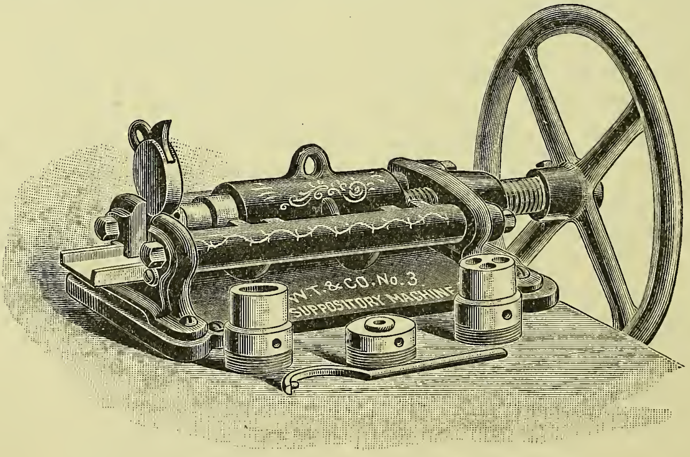

Fig. 227.—Whitall, Tatum's No. 3 suppository machine.

shown in Fig. 227 is furnished with moulds for making both suppositories and bougies. The medicated mass is placed in the cylinder, a mould attached, and the cylinder fixed in position with the end of the mould resting against the moveable plate at the end of the machine. The mass is then compressed by turning the wheel, which screws the plunger into the cylinder. After forming the suppositories the end plate is removed, when a further turn of the wheel forces them out upon the tray, the operation being repeated until all the mass has been used. Fig. 228 shows a smaller machine. The mould is adapted for making three sizes of suppositories, two rectal and one vaginal.

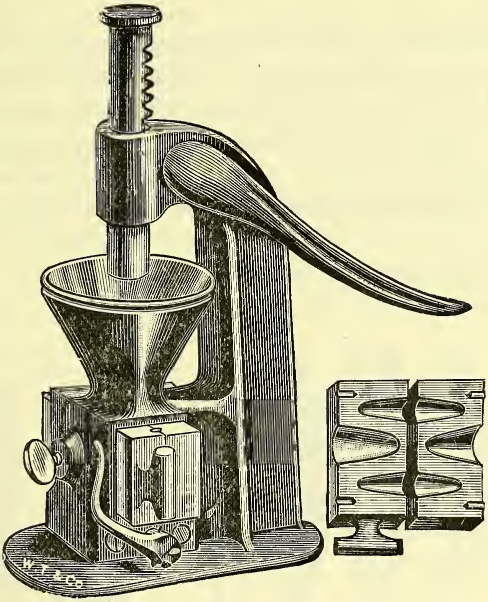

Fig. 228.—Whitall, Tatum's No. 1 suppository machine.

Bougies, pessaries.—The preparation of these differs in no way from that of suppositories, except in regard to the shape finally given to the mass.

Gelatin basis.—Although some time ago a mixture of gelatin and glycerin was strongly advocated as a basis for suppositories and bougies, it has almost dropped out of use, except for the administration of glycerin. The official quantities for the preparation of glycerin suppositories are as follows. Take of—

Gelatin	14·2 grams.
Glycerin	71·0 grams.
Distilled water	sufficient to produce 102·0 grams.

For the preparation of bougies and suppositories generally, a good basis is prepared with—

Gelatin	5 parts.
Glycerin	7 „
Distilled water	15 „

Soak the gelatin in the water until softened, dissolve by aid of gentle heat, add the glycerin and evaporate to 20 parts. For hygroscopic substances this basis answers as it is, but for insoluble powders it may be made to contain 2 parts more of water.

Stearin.—Cocoa-nut oil stearin has been suggested as a basis for suppositories; its melting-point is considerably lower than oil of theobroma, rendering it all the more inconvenient to handle in hot weather; otherwise it is used in a precisely similar manner.

Hollow suppositories made of oil of theobroma are sometimes made use of for the introduction of certain medicaments into the rectum. The drug should, however, be diluted with grated theobroma before packing into the shells, so as to insure slow and even melting of the mass (Fig. 229).

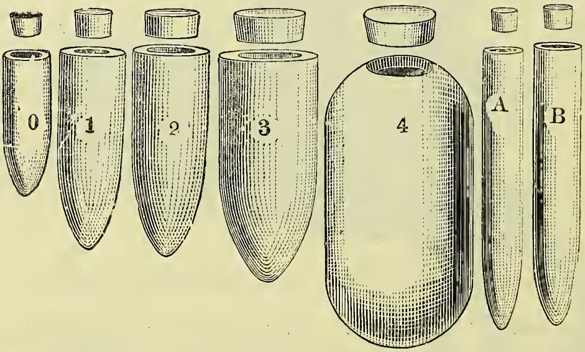

FIG. 229.—Hollow cacao suppositories.

Remarks.—Suppositories should be sent out in partitioned boxes; failing this they may be sent out in shallow boxes lined with cotton wool. The practice of wrapping them singly in tinfoil is not recommended, as ignorant patients might omit to remove the covering, which, moreover, often adheres in an annoying manner. For tropical climates, suppositories may be hardened by the addition of not more than 10 per cent. of white wax.

Alkaloids should be dissolved in a little oleic acid before adding to the fat. Salts must as far as possible be dissolved in water; if this is not practicable, they must be reduced to impalpable powder.

For the preparation of suppositories containing green or resinous extracts, the author suggests the following. Take of—

Ext. Hyoscyami ... ... gr. iij.
Ol. Theobromæ ... ... q. s.
For one suppository. Send xij.

Place the 36 grains of extract in a porcelain capsule with 12 grains of glyco-gelatin (such as used for Suppos. Glycerini, B.P.), and warm. When softened, mix intimately, gradually adding the previously melted theobroma. If the proper degree of temperature has been employed there is no need to use a slab or mortar for mixing in the extract, as a perfect emulsion is formed, which persists quite long enough to enable the operator to pour a dozen suppositories in almost as many minutes without fear of separating.

CHAPTER XLIX

LABELS

The labels on stock bottles, whether for the pharmacy or the store-room, should be as plain as possible. For general effect and utility, gold-labelled bottles are still preferred by many pharmacists, although sunk glass labels find numerous applications, especially for corrosive liquids, &c. In any case, whatever the material of the label, the lettering cannot be too distinct, and should be so arranged that the whole of the label is visible from the front. Labels that extend three-quarters of the way around the bottle, are dangerous in the extreme, as fatal results have, in more than one instance, been traceable to their use. Fig. 230 shows a label which cannot possibly be misinterpreted ; but that on the bottle Fig. 231 might easily be taken to indicate the *simple*

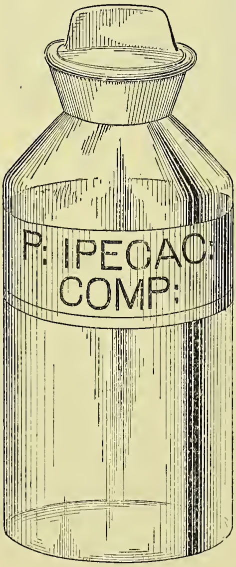

Fig. 230.

Fig. 231.

powder of ipecacuanha instead of the *compound*, which it purports to. No essential part of the label should be hidden from view, and no abbreviations should be used which permit of more than one rendering.

The labels on store-room bottles, while not necessarily of the same expensive or ornamental character as those on the pharmacy shelves, should be equally distinct. If it is not found convenient to employ printed labels, it is at any rate possible for all to stencil them for themselves; and if the blank spaces left by the stencil plates are filled in with Chinese ink, the labels leave nothing to be desired as regards legibility. Labels prepared in this way should be left for one or two days to dry, when they may be affixed by flour paste, and then sized with dilute gelatin solution. After another interval of a day or two, they should have a coat of pale paper varnish, care being taken to extend the varnish well over the edges of the paper to prevent moisture running under. Paper labels treated in this manner will wear for a very long time, but are not suited for bottles containing corrosive liquids. For the last mentioned, labels etched on the glass itself are usually employed. The following process involves very little trouble, and, moreover, requires no out-of-the-way appliances.

Cover one side of the bottle to be etched, with smooth wrapping paper, out of which an oblong space has been cut where the label is destined to appear. When the paper is dry, print the name of the intended contents as neatly as possible on the bare glass, and set aside until perfectly hard. The paint should be made by thinning white-lead with boiled linseed oil; if turpentine is used, the writing is liable to crumble under the subsequent treatment.

A small tin funnel, having a nozzle of about $\frac{1}{16}$ inch diameter at its orifice, is next filled with a short length of glass tubing, which has been drawn out as shown in Fig. 232. The supports may be conveniently made out of a cork cut in the form of a cross. The glass tube is connected by a rubber pipe with a foot blower, Fig. 233, and the funnel filled with sharp sand. The bottle having been placed label uppermost on a sheet of paper, a fine stream of sand is violently projected on its surface by means of the blower. The depth of the etching is regulated by the force, and the length of time to which it is subjected to the action, of the blast. When in the opinion of the operator the etching appears distinct enough, the paper is

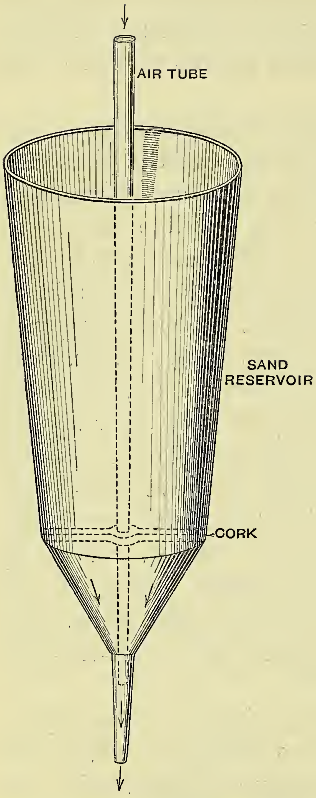

Fig. 232.—Sand-blast apparatus.

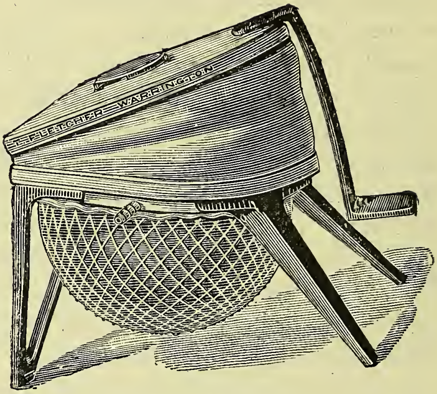

Fig. 233.—Foot blower.

soaked off, and the paint removed by oil of turpentine. If the operation has been properly carried out, a very neat and indestructible label will be the result.

CHAPTER L

STOPPERING OF GLASS BOTTLES

It occasionally happens that glass stoppers do not fit perfectly into the mouth of the bottles to which they belong, or the proper stopper may have become lost or broken, in which case new ones have to be fitted. If the stopper can be moved laterally it may be considered as ill-fitting, and unsuited for the preservation of volatile substances, or such as are readily oxidised or injured by exposure to air.

To render such stoppers air-tight they must be re-ground. This is effected by dipping the stopper into water, and then into coarse emery or sand, and placing it in the neck of the bottle, twisting it to and fro until the emery has been ground smooth, when more is applied. After grinding for some time the stopper and the neck of the bottle should be rinsed with water and the fitting examined. If fairly firm the grinding may be finished with fine emery powder and oil. On a commercial scale this grinding is accomplished by means of a lathe, but with a little care equally good, if not better results may be obtained by hand.

Removal of fixed stoppers.—The removal of stoppers that have become firmly fixed in the necks of bottles is an operation with which the practical pharmacist has frequently to deal. There are several causes which give rise to the fixing of glass stoppers, the most frequent being the deposition of resinous or other adhesive matter between the stopper and the bottle neck. Sometimes salts become crystallised there, either from the sublimation of the salt itself, or in consequence of the evaporation of the solvent. In some cases the pressure of the atmosphere, occasioned by contraction of the gaseous contents of the bottle, is the cause of the stopper becoming fixed.

The means of effecting the removal of the stopper must depend upon the cause of the adhesion, and on the size, form, and thickness of the bottle and its stopper. If the bottle and its stopper be of sufficient capacity and strength, it is customary in the first instance to attempt the extrication by pushing the stopper in a lateral direction, and at the same time pulling it upwards. Should this method fail, the stopper may be gently tapped in a lateral and slightly ascending direction with the wooden handle of a palette knife. Great care must be taken to regulate the weight of the blows to the size and apparent strength of the bottle. Instead of tapping the stopper, a lever may be used, which fitting closely to the stopper enables more force to be applied than would be otherwise possible. The lever consists of a piece of hard wood (Fig. 234), in which are oblong holes of different sizes, into one of which the stopper of the bottle is placed. As in the use of the palette knife, great care and judgment is required or the stopper may be easily broken off.

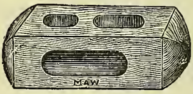

Fig. 234.—Stopper extractor.

If none of the foregoing prove effectual, other means must be adopted. Thus, if resinous or other matter soluble in spirit is the cause of the adhesion, a little alcohol may be placed in the groove between the stopper and the lip of the bottle, and after allowing some time to elapse, the methods previously described may be repeated. Should the deposition be soluble in water or oil, these liquids may be substituted for the alcohol. Should none of these methods prove successful, it may be necessary to warm the neck of the bottle over a *small* Bunsen flame. The immediate result of warming is expansion of the bottle neck, thus relaxing the grasp on the stopper, which may often be readily withdrawn. Care is needed in the application of the heat, as if not warmed uniformly the bottle neck may split; while if the heat is applied too long, it becomes communicated to the stopper, which may or may not expand uniformly with the bottle. Should the bottle contain a liquid of an inflammable nature the application of a flame may be unsafe, in which case a strip of cloth dipped in hot water may be wrapped round the part to be heated; or a piece of stout string may be passed around the bottle neck, one end being tied to some stationary object, the other held firmly in the left hand. If now the bottle is grasped in the right hand and rapidly moved backwards and forwards, the friction will generate sufficient heat for the purpose. If the stopper cannot be removed by the hand after warming, tapping and levering may be tried before the glass cools.

If after trying all these devices the stopper still appears immoveable, the following plan may be resorted to as a last expedient. In the first place, a strong cord three or four feet in length, is selected, and knotted tightly around the stopper. The cord being thus secured, it is suspended from any convenient hook. The operator, having wrapped a cloth around the bottle in case of accident, should grasp the bottle firmly with both hands, and endeavour to loosen the stopper by a series of jerks. The jerks, which should be slight at first, are gradually increased, until as much force is applied as the bottle will stand. If care be taken to maintain the vertical position of the bottle and direction of the jerk, there is little danger of breaking the stopper.

As prevention is better than cure, it is a wise plan to smear the stoppers of all bottles that are only occasionally opened, with soft paraffin ; a few twists of the stopper will then effectually distribute the lubricant. The paraffin serves another purpose than preventing sticking of the stopper, for it makes a tighter seal and prevents evaporation. If it is desired to hermetically seal the bottle, the neck and the stopper should be gently warmed, and a little hard paraffin, which has been previously melted in a small capsule, poured carefully into the groove. Ethereal and other liquids having a solvent action on paraffin are best sealed with a mixture made by dissolving one part of gelatin in five parts of water, adding just before use, half per cent. of potassium dichromate. After exposure the water evaporates, leaving a film of insoluble gelatin on the stopper or cork.

PART IV

PHARMACOPŒIAL TESTS—VOLUMETRIC AND GRAVIMETRIC ANALYSIS

CHAPTER LI

VOLUMETRIC ANALYSIS

Volumetric analysis determines quantities of substances by measuring volumes, instead of determining weights of masses. Volumetric analysis, or quantitative analysis by measure, can only be applied to fluids; therefore it is necessary that one, if not both of the substances should be in a liquid condition.

The chief advantage in determining the quantity of substances by measure instead of by weight, lies in the great saving of time that is effected. Suppose, for instance, that one decigram (0·1) of sodium chloride be dissolved in sufficient distilled water to produce 100 cubic centimetres (100 c.c.), it would obviously be more expeditious to measure 100 volumes of one cubic centimetre, each containing one milligram (0·001) of salt, than it would be to divide the salt into 100 equal parts by weight. Another and not less important advantage, is that it is perfectly easy to operate with quantities of substances that could not possibly be estimated by weighing under ordinary conditions. Thus, instead of measuring off the salt solution in quantities of 1 cubic centimetre at a time, it would be equally easy to measure it off by one-tenths of a cubic centimetre, equivalent in this case to $\frac{1}{10}$ of a milligram (·0001) of the salt originally taken.

Apparatus.—In carrying out analyses depending on volumetric methods, the following special apparatus is employed.

1. A glass flask which, when filled to a mark on the neck, contains 1000 grams of distilled water at 60° F. (15·5° C.), Fig. 235. This flask is used to represent 1 litre (1000 c.c.), as it is necessary for the sake of convenience to make the measurement of liquids with metric apparatus which has been graduated at 60° F. (15·15° C.).*

2. A graduated cylindrical jar, preferably stoppered, which

---

* Properly, the litre contains a thousand grams of distilled water at its maximum density, 4° C.

when filled at 60° F. (15·5° C.) contains either 500 c.c. or 1 litre (1000 c.c.), (Fig. 236).

3. A burette. This is a cylindrical graduated glass tube, which when filled to 0 at 60° F. holds within the graduated portion 50 cubic centimetres, each centimetre being divided into ten equal parts. The lower end of the burette should be provided with a glass tap for delivering the test solution (Fig. 237). For use with certain solutions, the burette may be provided with a short length of rubber tubing, terminated by a glass nozzle having a very fine opening. The rubber tube is closed by a metal pinchcock. Fig. 238 illustrates the ordinary kind devised by Mohr, and Fig. 239 a modification of Bunsen's pinchcock, which is so made that one side is always open, enabling it to be removed without being passed over the end of the tube.

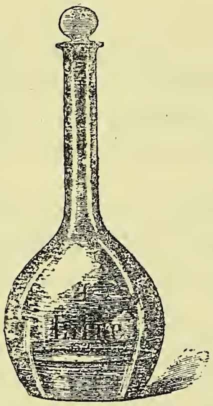

Fig. 235.

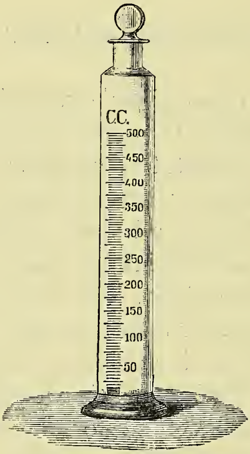

Fig. 236.

Fig. 237.

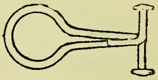

Fig. 238.

Fig. 239.

4. A burette stand. This is simply a contrivance for holding the burette in an upright position during the process of titration. Fig. 240 illustrates a wooden stand with two arms for holding a pair of burettes. Of late years metal burette clamps have come into use. These may be fixed to the rod of any retort stand. Fig. 241 is an enlarged view of one of the most useful of these clamps; a fixed arm carries four rubber rings, and the burette is pressed against them by a fifth ring carried on a small moveable arm. This enables the burette to be held tightly without chance of breaking, but yet in such a position that no part of the scale is hidden from view.

5. A weighing bottle. This should be made of thin blown

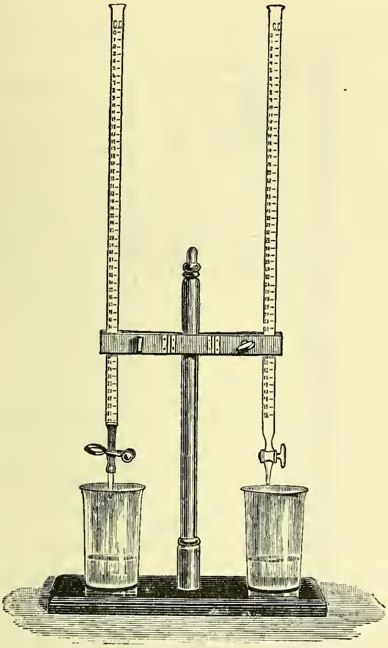

Fig. 240.

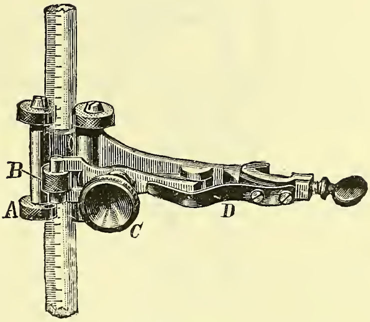

Fig. 241.

glass, and have a capacity of about 25 or 30 c.c. The neck should be very wide, and be fitted with a well-ground stopper.

When volumetric operations are of frequent occurrence, it is often found convenient to keep one or more burettes permanently fitted up, as shown in Fig. 242. This arrangement consists of a burette with glass tap connected to a reservoir, which is placed on a shelf a little above the burette, by a glass tube having a stopcock in the middle. The lower end of this tube is bent in such a manner that the test solution is directed against the inner side of the burette, whence it flows gently down without causing froth or bubbles. A flexible rubber tube that does not come in contact with the solution conveys air from the burette to the reservoir. The burette is filled without contact with the external air by simply opening the stopcock in the bent glass tube. As the reservoir empties, it may be necessary to remove the cork for an instant to permit entrance of air, or to completely empty the burette and allow air to enter by the tap.

Fig. 243 shows another very good method of mounting a burette so that it can be easily filled with test solution. *d* is the

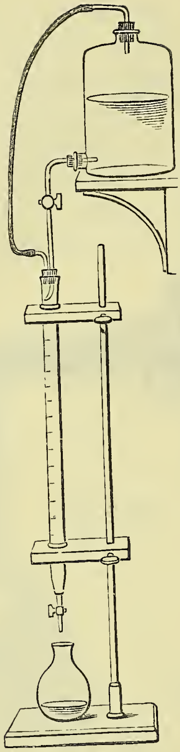

FIG. 242.

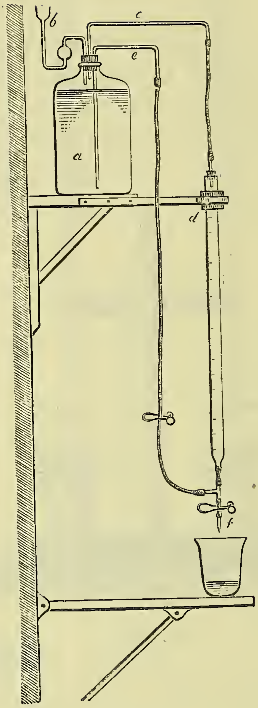

FIG. 243.

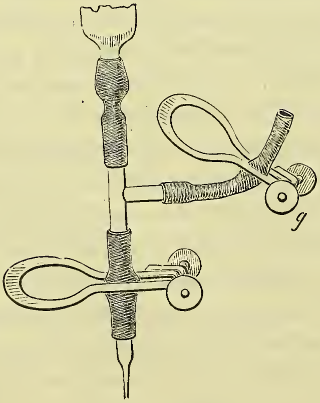

FIG. 244.

FIG. 245.

FIG. 246.

burette; *f*, the filling arrangement shown in detail at Fig. 244; *a* is a bottle of test solution placed on a shelf; *b*, a safety funnel to regulate entrance of air; *e*, a syphon by which the solution descends to the burette; *c*, a tube by which air passes from the burette to the bottle. The burette is filled by opening the pinchcock *g*. When the test solution runs out of the burette during titration, air enters through the tube *c*. By this device the test solution is kept out of contact with air, and the burette being filled from the bottom, the liquid enters without formation of bubbles.

Floats.—In reading the volume of liquid in a burette, the lowest point of the curve of the meniscus, Fig. 245, is always made to coincide with the graduation marks; but as, owing to capillarity, a certain difficulty is experienced in getting true readings, some persons employ an Erdmann's float. This consists of a cylindrical glass bulb, rather smaller in diameter than the burette itself, and weighted at its lower end with a globule of mercury, like a hydrometer (Fig. 246). A thin line is made around the middle of the bulb, and the coincidence of this line, when the bulb floats freely in the liquid, with a graduation mark is taken as the true reading.

Indicators.

An indicator is a substance added to the solution to be estimated, which, by a change of colour or appearance, indicates the exact point at which a given reaction is complete.

Mucilage of starch.—Gives an intensely blue colour with iodine, at ordinary temperatures. The Pharmacopœia directs the mucilage to be prepared by triturating 1 gram of starch with water to form a smooth paste, adding more water and boiling, so as to produce 50 c.c. when cold. A better plan consists in boiling a few grams of starch with water, cooling and allowing to stand until the gelatinised starch has precipitated. The *clear* liquor is then strained through flannel, and preserved for use. Mucilage of starch should be fairly recently prepared, as old specimens react comparatively slowly with iodine.

Solution of potassium ferricyanide.—Gives an intensely blue precipitate or coloration with ferrous salts, but none with ferric salts. The solution must be freshly prepared, and should contain about 5 per cent. of clear crystals free from powder.

Solution of litmus.—Gives a red colour with acids, blue with alkalies. It is not distinctly reddened by boric acid, but it is reddened by moist carbonic anhydride; hence the necessity for boiling when estimating carbonates. It is prepared as follows:

Litmus powder	20 grams.
Alcohol, 90 per cent.	200 c.c.
Distilled water	200 c.c.

Boil the litmus with 80 c.c. of the alcohol for an hour, using an upright condenser to prevent evaporation; allow to settle, and pour off the clear liquid, which reject; repeat the operation with 60 c.c. of alcohol, and again with the remainder. Digest the now purified litmus in the distilled water at a temperature of about 120° F. for twenty-four hours, and filter.

If properly made and in good condition, litmus solution should be of a purple colour. After long keeping the colour slowly disappears, but may be restored by exposing the solution to the air in an open dish for a few days.

Solution of methyl orange.—Gives a pink coloration with mineral acids, and a faint yellow colour with alkalies. It is not reddened by moist carbonic anhydride or by boric acid. It is the most reliable indicator for excess of ammonia.

Methyl orange	0·4 gram.	
Alcohol, 90 per cent.	50 c.c.	
Distilled water	150 c.c.	Dissolve.

Solution of phenol-phthalein.—Gives a red colour with alkalies, which is discharged by acids. It is the most trustworthy indicator of excess of organic acids. It does not accurately indicate the point of neutralisation of ammonia with an acid. The solution should be quite colourless.

Phenol-phthalein	0·4 gram.	
Alcohol, 90 per cent.	120 c.c.	
Distilled water	80 c.c.	Dissolve.

Solution of neutral potassium chromate.—Gives a permanent red-coloured precipitate with silver nitrate, but not while any soluble bromide, chloride, or iodide is present.

Note on the use of indicators

Phenol-phthalein, methyl orange, and litmus, are only reliable when moderately strong solutions (normal or decinormal) are employed; *they are not to be depended upon in the titration of centinormal solutions or weak alkaloids.* For these latter, either hæmatoxylin, brazil-wood, or cochineal should be used, the first two being the most delicate. For general acidimetry and alkalimetry the author prefers purified litmus to the coal-tar products; for alkaloidal alkalimetry, hæmatoxylin or brazil-wood. In any case it is far better to accustom one's self to one indicator for general work, and one for alkaloidal titrations; the eye then becomes educated to the tints, and the end reactions perceived with certainty.

Brazil-wood.—Boil 50 grams in 150 c.c. distilled water for half an hour in a porcelain dish, cool, strain, wash until 100 c.c. of decoction are obtained, add 25 c.c. 90 per cent. alcohol; keep for a week and filter.

Hæmatoxylin.—Dissolve 1 gram of Merck's crystalline hæmatoxylin in 100 c.c. of alcohol.

Cochineal.—Macerate 20 grams of whole cochineal in 100 c.c. of dilute alcohol (about 30 per cent.) for a fortnight. Strain and filter.

In the preparation of the foregoing be careful to avoid any metallic contamination. Hæmatoxylin is especially sensitive; in fact, in titrating alkaloids it is important to use water which has been distilled and collected in glass vessels.

Volumetric solutions

Volumetric solutions are called *normal* $\left(\frac{N}{1}\right)$ when they contain in 1 litre the molecular weight of the active reagent expressed in grams, and reduced to the valency corresponding to one atom of replaceable hydrogen, *or its equivalent.* In other words, 1 litre of a normal volumetric solution, contains as many grams of active reagent as are equivalent to 1 gram of replaceable hydrogen.

Thus the molecule of sodium hydroxide, NaOH (molecular weight 39·76), has but one sodium atom to replace one hydrogen atom in an acid; hence its normal solution contains 39·76 grams in 1 litre.

Again, the molecule of sulphuric acid H_2SO_4, (molecular weight 97·34), contains 2 atoms of hydrogen replaceable by a metal; its normal solution therefore contains 48·67 grams in 1 litre, or half its molecular weight in grams.

Decinormal solutions $\left(\frac{N}{10}\right)$ are one-tenth the strength of normal solutions. Solutions containing quantities of the active reagent having no simple relation to the molecular weight, are termed "empirical."

Note on the quantities of substances to be taken for determination

Although under each heading it is stated that a definite weight of substance should require a definite volume of a volumetric solution for complete decomposition, it does not follow that the student is to actually operate on that amount. Indeed, such a proceeding would involve great loss of time, for it is extremely tedious business to *exactly* counterpoise the weights *with the substance,* on a sensitive balance, especially with liquids. Practically, an analyst rarely, if ever, weighs out a definite quantity. The general rule is to first weigh out a bulk in a weighing bottle, operate on a quantity selected by judgment, then weigh what is left of the bulk, the difference being the quantity actually taken. Thus:

Weight of bottle and bulk	=	28·915	grams.
Weight after removal of a portion	=	28·723	,,
Quantity taken	=	0·192	,,

As a rule the quantity selected should not require more than 50 c.c. of a volumetric solution to complete the reaction, as, apart from waste of solution, there is more liability to error if the burette has to be refilled.

Volumetric solution of iodine (I = 125·9)

Indicator, mucilage of starch.

This is a decinormal solution $\left(\frac{N}{10}\right)$. It contains 12·59 grams in 1 litre.

Preparation.—

Purified iodine	... ...	12·59 grams.
Pure potassium iodide	...	18·00 ,,
Distilled water	... ...	a sufficient quantity.

Purified iodine may be obtained by mixing the official "iodum" with one fourth of its weight of dry potassium iodide, re-subliming, and drying the new crystals over sulphuric acid in an apparatus similar to Fig. 77. By re-sublimation, compounds of iodine with cyanogen, bromine, and chlorine are got rid of. Put the purified iodine and the potassium iodide with about 20 c.c. of distilled water into the litre flask, and agitate gently until solution is complete, then dilute with water until it measures exactly 1000 c.c. at 60° F.

Verification.—The strength of the solution is most conveniently verified by the aid of pure arsenious anhydride, and the solution either strengthened or diluted, so that 1000 c.c. contain exactly 12·59 grams of iodine; or its strength may be noted and calculations made accordingly, when the solution is used.

Volumetric solution of iodine $\left(\frac{N}{10}\right)$ is used in testing the following:

Acidum Arseniosum.—0·25 gram of arsenious anhydride is dissolved in 100 c.c. of boiling water, with about four times its weight of pure sodium bicarbonate, and allowed to cool; a little starch mucilage is added, and the iodine solution run in from a burette until a faint permanent blue colour appears. This should take 50·8 c.c., corresponding to nearly 100 per cent. of arsenious anhydride.

$$\underbrace{2I_2} + \underbrace{As_2O_3} + 5H_2O = 2H_3AsO_4 + 4HI.$$

$$\begin{array}{ll} 4)\overline{503{\cdot}6} & 4)\overline{196{\cdot}64} \\ 10)\overline{125{\cdot}9} & 10)\overline{49{\cdot}16} \\ \quad\ \overline{12{\cdot}59} & \quad\ \overline{4{\cdot}91} \end{array}$$

That is, 1000 c.c. $\frac{N}{10}$ I = 4·91 grams arsenious anhydride.

1 c.c. ,, = 0·00491 ,, ,,

Therefore 0·25 gram arsenious anhydride = 50·9 c.c. $\frac{N}{10}$ I (1000 × ·25 ÷ 4·91 = 50·9).

As seen by the above equation, iodine in presence of water, converts arsenious anhydride into arsenic acid, the iodine being converted into hydriodic acid. As soon as the last trace of arsenious anhydride is oxidised, free iodine appears in the solution, and at once strikes a blue colour with starch mucilage.

Acidum Sulphurosum.—Sp. gr. 1·025. One gram weighed in a stoppered bottle, mixed with 100 c.c. of recently boiled and cooled distilled water, and a little mucilage of starch added, should not acquire a permanent blue tint until 15·7 c.c. $\frac{N}{10}$ I have been added, corresponding to nearly 5 per cent. by weight of sulphur dioxide.

$$H_2O + \underbrace{I_2}_{} + \underbrace{SO_2}_{}.\ H_2O = 2HI + H_2SO_4.$$

$$\begin{array}{ll} 2)\overline{251{\cdot}8} & 2)\overline{63{\cdot}58} \\ 10)\overline{125{\cdot}9} & 10)\overline{31{\cdot}79} \\ \overline{\quad 12{\cdot}59} & \overline{\quad 3{\cdot}179} \end{array}$$

That is, 1000 c.c. $\frac{N}{10}$ I = 3·179 grams of sulphur dioxide.

1 c.c. „ = 0·003179 „ „

Therefore 1 gram sulphur dioxide = 314·56 c.c. $\frac{N}{10}$ I.

But as Acidum Sulphurosum is but a 5 per cent. solution of sulphur dioxide, 1 gram would only require 314·56 × ·05 = 15·728 c.c. $\frac{N}{10}$ I for oxidation.

Sodii Sulphis.—One gram dissolved in 50 c.c. of water should decolourise not less than 77·7 c.c. nor more than 81·7 c.c. $\frac{N}{10}$ I, corresponding to not less than 97·30 per cent., nor more than 102·3 per cent. if the salt is slightly effloresced. Indicator, starch mucilage.

$$\underbrace{I_2}_{} + \underbrace{Na_2SO_3.7H_2O}_{} + H_2O = 2HI + Na_2SO_4 + 7H_2O.$$

$$\begin{array}{ll} 2)\overline{251{\cdot}8} & 2)\overline{250{\cdot}38} \\ 10)\overline{125{\cdot}9} & 10)\overline{125{\cdot}19} \\ \overline{\quad 12{\cdot}59} & \overline{\quad 12{\cdot}519} \end{array}$$

That is—

1000 c.c. $\frac{N}{10}$ I = 12·519 grams crystallised sodium sulphite.

1 c.c. = ·012519 „ „

Therefore 1 gram crystallised sodium sulphite = 79·8 c.c. $\frac{N}{10}$ I (1000 ÷ 12·519 = 79·8).

Antimonii Oxidum.—·5 gram dissolved in a little boiling water containing 1 gram of hydrogen-potassium tartrate, and the solution made alkaline with 3 grams of sodium bicarbonate, the resulting liquid should discharge the colour of 70 c.c. $\frac{N}{10}$ I, corresponding to 100 per cent. of antimony trioxide. Indicator, starch mucilage.

$$\underbrace{Sb_2O_3}_{285\cdot64} + 2KHC_4H_4O_6 = \underbrace{[K(SbO)C_4H_4O_6]_2.H_2O}_{659\cdot14}.$$

Then $\underbrace{[K(SbO)C_4H_4O_6]_2.H_2O}_{659\cdot14} + 2NaHCO_3 + 4H_2O + \underbrace{2I_2}_{500\cdot76}$

$$= 2KNaC_4H_4O_6 + 2CO_2 + 4HI + 2H_3SbO_4 + H_2O.$$

Therefore $\underbrace{Sb_2O_3}$ $=$ $\underbrace{2I_2}$

$$\begin{array}{ll} 4)\underline{285\cdot64} & 4)\underline{500\cdot76} \\ 10)\underline{71\cdot41} & 10)\underline{125\cdot19} \\ \quad 7\cdot141 & \quad 12\cdot519 \end{array}$$

That is, 1000 c.c. $\frac{N}{10}$ I = 7·141 grams antimony trioxide.

1 c.c. ,, = 0·00741 ,, ,,

Therefore 0·5 gram antimony trioxide = 70 c.c. $\frac{N}{10}$ I (1000 × ·5 ÷ 7·141 = 70).

Antimonium Tartaratum.—One gram dissolved in water with 2 grams of sodium bicarbonate should discharge the colour of not more than 60·7 nor less than 60·2 c.c. $\frac{N}{10}$ I, corresponding to at least 99 per cent. of tartarated antimony. See equation under Antimony trioxide. Indicator, starch mucilage.

$$\underbrace{[K(SbO)C_4H_4O_6]_2.H_2O} = \underbrace{2I_2}.$$

$$\begin{array}{ll} 4)\underline{659\cdot14} & 4)\underline{500\cdot76} \\ 10)\underline{164\cdot785} & 10)\underline{125\cdot19} \\ \quad 16\cdot478 & \quad 12\cdot519 \end{array}$$

That is, 1000 c.c. $\frac{N}{10}$ I = 16·478 grams tartarated antimony.

1 c.c. ,, = 0·016478 ,, ,,

Therefore 1 gram tartarated antimony = 60·68 c.c. $\frac{N}{10}$ I (1000 ÷ 16·478 = 60·68).

Liquor Arsenicalis.—Twenty-five c.c. boiled with 1 gram of pure sodium bicarbonate for five minutes, cooled, and diluted with water to which a few drops of starch mucilage have been added, should discharge the colour of exactly 50·8 c.c. $\frac{N}{10}$ I. For equation and working see Acid. Arseniosum.

Liquor Arsenici Hydrochloricus.—The same as for Liq. Arsenicalis, but using 2 grams sodium bicarbonate.

Volumetric solution of potassium dichromate ($K_2Cr_2O_7 = 292{\cdot}3$).

One molecule of potassium dichromate heated with sulphuric acid yields, in the presence of a deoxidising agent, 3 atoms of oxygen, *which are equivalent to 6 atoms of hydrogen.* These 3 atoms of oxygen are capable of raising 6 atoms of iron from the ferrous to the ferric state. Thus—

$$K_2Cr_2O_7 + 4H_2SO_4 = K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + O_3.$$
$$6FeO + O_3 = 3Fe_2O_3.$$

Therefore a *normal* solution of potassium dichromate would contain 48·7 grams of the salt in 1 litre ($292{\cdot}3 \div 6 = 48{\cdot}7$). As the pharmacopœial volumetric solution only contains 4·87 grams in 1 litre, it is decinormal $\left(\frac{N}{10}\right)$.

Preparation.—

Potassium dichromate ...	4·87 grams.
Distilled water	sufficient to produce 1000 c.c. at 60° F.

100 c.c. of this solution yield 0·0794 gram of oxygen, which is capable of converting 0·556 gram of iron from the ferrous to the ferric state.

Verification.—The strength of the solution should be verified by the aid of pure ferrous ammonium sulphate, or other reliable substance, and the solution either strengthened or diluted so that 1000 c.c. shall contain exactly 4·87 grams of potassium dichromate, or its strength noted and calculations made accordingly when the solution is used.

Place 0·7 gram pure green ferrous ammonium sulphate in a white porcelain dish with 100 c.c. distilled water and 1 c.c. sulphuric acid. Dissolve. Take a clean white porcelain slab, and with a glass rod cover it with spots of *recently* prepared solution of potassium ferricyanide. Run in the dichromate solution from a burette until a trace taken from the basin on a stirring rod just ceases to give a blue colour with one of the spots of ferricyanide solution on the slab. This should require 17·9 c.c. of the dichromate solution. The reactions that take place are explained by the following equations:

$$\mathrm{K_2Cr_2O_7 + 4H_2SO_4 = K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + O_3.}$$

$$\underbrace{\mathrm{6FeSO_4.(NH_4)_2SO_4.6H_2O}}_{\begin{array}{r}6)\underline{2336{\cdot}64}\\ 10)\underline{389{\cdot}44}\\ 38{\cdot}94\end{array}} + \underbrace{\mathrm{K_2Cr_2O_7}}_{\begin{array}{r}6)\underline{292{\cdot}3}\\ 10)\underline{48{\cdot}7}\\ 4{\cdot}87\end{array}} + \mathrm{7H_2SO_4 = 3Fe_2(SO_4)_3}$$

$$\mathrm{+\ 6(NH_4)_2SO_4 + K_2SO_4 + Cr_2(SO_4)_3 + 43H_2O.}$$

That is—

1000 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ = 38·94 grams ferrous ammonium sulphate.

1 c.c. ,, = 0·03894 ,, ,, ,,

Therefore 0·7 gram ferrous ammonium sulphate = 17·9 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ (1000 × ·7 ÷ 38·94 = 17·9).

Ferri Arsenas.—One gram of the official salt dissolved in excess of sulphuric acid diluted with water should not cease to give a blue precipitate with sol. of potassium ferricyanide until at least 6·7 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ have been added, corresponding to 12·28 per cent. of *hydrous* ferrous arsenate, or reckoned as *anhydrous* salt, 9·89 per cent.

$$\underbrace{\mathrm{2Fe_3(AsO_4)_2.6H_2O}}_{\begin{array}{r}6)\underline{1100{\cdot}24}\\ 10)\underline{183{\cdot}373}\\ 18{\cdot}3373\end{array}} + \underbrace{\mathrm{K_2Cr_2O_7}}_{\begin{array}{r}6)\underline{292{\cdot}3}\\ 10)\underline{48{\cdot}7}\\ 4{\cdot}87\end{array}} + \mathrm{7H_2SO_4 = Fe_2(SO_4)_3}$$

$$\mathrm{+\ 2Fe_2(AsO_4)_2 + Cr_2(SO_4)_3 + 19H_2O.}$$

That is—

1000 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ = 18·3373 grams of *hydrous* ferrous arsenate.

1 ,, ,, = 0·0183373 ,, ,, ,,

6·7 ,, ,, = 0·12285991 ,, of ditto in 1 gram of the official compound, or 12·28 grams in 100 grams.

But the weight of 2 molecules of *anhydrous* ferrous arsenate is only 885·68, and the co-efficient for 1 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$, derived in the same way as above, is ·0147613.

That is—

1 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ = 0·0147613 gram of *anhydrous* ferrous arsenate.

6·7 c.c. ,, = 0·0989 ,, of ditto in one gram of the official compound, or 9·89 grams in 100 grams.

Ferri Carb. Sacch.—One gram dissolved in excess of warm phosphoric acid and diluted with water should not cease to give a precipitate with solution of potassium ferricyanide until at least 29 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ have been added, corresponding to at least 33 per cent. of ferrous carbonate.

$$\underbrace{6FeCO_3}_{\begin{array}{r}6)\underline{690{\cdot}9}\\10)\underline{115{\cdot}15}\\11{\cdot}515\end{array}} + \underbrace{K_2Cr_2O_7}_{\begin{array}{r}6)\underline{292{\cdot}3}\\10)\underline{48{\cdot}7}\\4{\cdot}87\end{array}} + 9H_3PO_4 = K_2HPO_4 + 2CrPO_4 + 3Fe_2(PO_4)_2 + 6CO_2 + 13H_2O.$$

That is—

1000 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ = 11·515 grams ferrous carbonate.

1 c.c. ,, = 0·011515 ,, ,,

Therefore 1 gram ferrous carbonate = 86·84 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$.

As the pharmacopœial preparation only requires 29 c.c. for oxidation, it fixes 33 per cent. as the minimum of ferrous salt required to be present (100 × 29 ÷ 86·84 = 33·3 per cent.).

Ferri Phosphas.—One gram dissolved in hydrochloric acid should not cease to give a blue precipitate with potassium ferricyanide solution until at least 28·2 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ have been added, corresponding to 47 per cent. of ferrous phosphate.

$$\underbrace{2Fe_3(PO_4)_2.8H_2O}_{\begin{array}{r}6)\underline{996{\cdot}96}\\10)\underline{166{\cdot}16}\\16{\cdot}616\end{array}} + \underbrace{K_2Cr_2O_7}_{\begin{array}{r}6)\underline{292{\cdot}3}\\10)\underline{48{\cdot}7}\\4{\cdot}87\end{array}} + 14HCl = Fe_2Cl_6 + 2Fe_2(PO_4)_2 + 2KCl + Cr_2Cl_6 + 15H_2O.$$

That is—

1000 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ = 16·616 grams hydrated ferrous phosphate.

1 c.c. ,, = 0·016616 ,, ,, ,,

Therefore 1 gram ferrous phosphate = 60·18 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ (1000 ÷ 16·616 = 60·18).

But as a pharmacopœial salt cannot be prepared without oxidation, it will fulfil official requirements if it contains 47 per cent. of ferrous phosphate (60·18 × 47 ÷ 100 = 28·284).

Ferri Sulphas.—One gram dissolved in water acidulated with sulphuric acid should not cease to give a blue precipitate with potassium ferricyanide solution until at least 36 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ have been added, corresponding to 99·4 per cent. crystallised ferrous sulphate.

$$\underbrace{6FeSO_4.7H_2O} + \underbrace{K_2Cr_2O_7} + 7H_2SO_4 = 3Fe_2(SO_4)_3 + K_2SO_4$$

$$\begin{array}{ll} 6)\underline{1656{\cdot}6} & 6)\underline{292{\cdot}3} \\ 10)\underline{276{\cdot}1} & 10)\underline{48{\cdot}7} \\ \quad 27{\cdot}61 & \quad 4{\cdot}87 \end{array}$$

$$+ Cr_2(SO_4)_3 + 14H_2O.$$

That is—

1000 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ = 27·61 grams crystalline ferrous sulphate.

1 c.c. ,, = 0·02761 gram ,, ,,

Therefore 1 gram crystalline ferrous sulphate = 36·2 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ (1000 ÷ 27·61 = 36·2).

If 36·2 c.c. = 100 per cent., 36 c.c. = 99·4 per cent. (36 × 100 ÷ 36·2 = 99·4).

Ferri Sulph. Exsicc.—One gram treated as above should not cease to yield a precipitate with potassium ferricyanide until at least 54·6 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ have been added, corresponding to 92·2 per cent. dried ferrous sulphate.

$$\underbrace{6FeSO_4.H_2O} + \underbrace{K_2Cr_2O_7} + 7H_2SO_4 = 3Fe_2(SO_4)_3 + K_2SO_4 +$$

$$\begin{array}{ll} 6)\underline{1012{\cdot}92} & 6)\underline{292{\cdot}3} \\ 10)\underline{168{\cdot}82} & 10)\underline{48{\cdot}7} \\ \quad 16{\cdot}882 & \quad 4{\cdot}87 \end{array}$$

$$Cr_2(SO_4)_3 + 7H_2O.$$

That is—

1000 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ = 16·882 grams dried ferrous sulphate.

1 c.c. ,, = 0·016882 ,, ,,

Therefore 1 gram dried ferrous sulphate = 59·2 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ (1000 ÷ 16·882 = 59·2).

If 59·2 c.c. = 100 per cent., 54·6 c.c. = 92·2 per cent. (54·6 × 100 ÷ 59·2 = 92·2).

Ferrum Redactum.—If ·25 gram be added to 1 gram of cupric sulphate in 15 c.c. of hot water in a flask that can be immediately well corked, and the whole occasionally shaken during ten minutes, the liquid, after being rapidly filtered with the minimum of exposure to air and acidified with sulphuric acid, should not cease to yield a blue precipitate with solution of potassium ferricyanide, until at least 33·7 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ have been added, corresponding to 75 per cent. of metallic iron.

$$3Fe_2 + 6CuSO_4 = 6FeSO_4 + 3Cu_2$$

$$6FeSO_4 + K_2Cr_2O_7 + 7H_2SO_4 = 3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O.$$

$$\text{Therefore } \underbrace{3\,Fe_2} = \underbrace{K_2Cr_2O_7}.$$

$$\begin{array}{ll} 6)\underline{333{\cdot}6} & 6)\underline{292{\cdot}3} \\ 10)\underline{55{\cdot}6} & 10)\underline{48{\cdot}7} \\ \quad\;\; 5{\cdot}56 & \quad\;\; 4{\cdot}87 \end{array}$$

That is, 1000 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$ = 5·56 grams metallic iron.

1 c.c. ,, = 0·00556 ,, ,,

Therefore 1 gram metallic iron = 179·8 c.c. $\frac{N}{10}$ $K_2Cr_2O_7$.

·25 ,, ,, ,, = 44·95 c.c. ,,

But Ferrum Redactum is only required to contain 75 per cent. of metallic iron, therefore 44·95 × 75 ÷ 100 = 33·7 c.c.

Volumetric solution of silver nitrate ($AgNO_3$ = 168·69)

This is a decinormal solution. It contains 16·869 grams of $AgNO_3$ in 1 litre.

Preparation.—Dissolve 16·869 grams of pure re-crystallised silver nitrate in sufficient distilled water to produce exactly 1 litre of solution, and preserve the product in an opaque stoppered bottle.

Verification.—The strength of the solution should be verified by the aid of pure sodium chloride, or solution of pure hydrochloric acid of known strength, and the solution either strengthened or diluted, so that 1000 c.c. contain exactly 16·869 grams in 1000 c.c., or its strength may be noted and calculations made accordingly when the solution is used.

Dissolve ·1 gram of pure dry sodium chloride in a little water in a small porcelain dish, and add sufficient neutral potassium chromate solution to impart a faintly yellow tint. Run in the silver solution, stirring constantly until the last drop changes the colour from yellow to orange. This should require 17·22 c.c.

$$\underbrace{AgNO_3}_{\substack{10)168{\cdot}69 \\ \overline{\quad 16{\cdot}869}}} + \underbrace{NaCl}_{\substack{10)58{\cdot}07 \\ \overline{\quad 5{\cdot}807}}} = AgCl + NaNO_3.$$

That is, 1000 c.c. $\frac{N}{10}$ $AgNO_3$ = 5·807 grams sodium chloride.

1 c.c. „ = 0·005807 „ „

Therefore 0·1 gram sodium chloride = 17·22 c.c. $\frac{N}{10}$ $AgNO_3$ (1000 × ·1 ÷ 5·807 = 17·22).

Acid. Hydrocyan. Dil.—Sp. gr. ·997. Contains 2 per cent. by weight of real hydrogen cyanide, HCN.

One gram rendered distinctly but not strongly alkaline by addition of sodium hydroxide, as indicated by a few drops of litmus solution, and *maintained fairly alkaline throughout the operation,* should require 3·7 c.c. $\frac{N}{10}$ $AgNO_3$ to be added before a permanent precipitate begins to form. If during the operation the litmus becomes red, a few drops more of solution of sodium hydroxide must be added. It will be seen by the equation that titration is carried on only so long as the double salt AgCN.NaCN forms; directly the sodium cyanide has all been used up, a *permanent* precipitate of silver cyanide (AgCN) falls, which denotes the end of the reaction (thus: $AgCN.NaCN + AgNO_3 = 2AgCN + NaNO_3$).

$$2HCN + 2NaOH = 2NaCN + 2H_2O\,;$$
$$\text{and } 2NaCN + AgNO_3 = AgCN.NaCN + NaNO_3.$$

$$\text{Therefore } \underbrace{2HCN}_{\substack{10)53{\cdot}7 \\ \overline{\quad 5{\cdot}37}}} = \underbrace{AgNO_3}_{\substack{10)168{\cdot}69 \\ \overline{\quad 16{\cdot}869}}}.$$

That is, 1000 c.c. $\frac{N}{10}$ $AgNO_3$ = 5·37 grams hydrogen cyanide.

1 c.c. „ = 0·00537 „ „

Therefore 1 gram hydrogen cyanide = 186·2 c.c. $\frac{N}{10}$ $AgNO_3$ (1000 ÷ 5·370 = 186·2).

But as Acid. Hydrocyan. Dil. is only a 2 per cent. solution, 1 gram would only require 3·72 c.c. for precipitation (186·2 × ·02 = 3·72).

Ammonii Bromidum.—0·5 gram, dissolved in about 25 c.c. distilled water to which a few drops of neutral chromate of potassium have been added, should require not more than 51·8 c.c., and not less than 51·1 c.c. $\frac{N}{10}$ $AgNO_3$ to have been added before a permanent red precipitate begins to form, corresponding to 99·4 per cent. of ammonium bromide.

$$\underbrace{AgNO_3}_{\begin{array}{r}10)\underline{168{\cdot}69}\\16{\cdot}869\end{array}} + \underbrace{NH_4Br}_{\begin{array}{r}10)\underline{97{\cdot}29}\\9{\cdot}729\end{array}} = AgBr + NH_4.NO_3.$$

That is, 1000 c.c. $\frac{N}{10}$ $AgNO_3$ = 9·729 grams ammonium bromide.

1 c.c. „ = 0·009729 „ „

Therefore 0·5 gram ammonium bromide = 51·39 c.c. $\frac{N}{10}$ $AgNO_3$ (1000 × ·5 ÷ 9·729 = 51·39).

Potass. Bromid.—One gram of the salt treated as for Ammon. Bromid. should require not less than 83·7, and not more than 85·4 c.c. $\frac{N}{10}$ $AgNO_3$ for complete precipitation, corresponding to at least 98·9 per cent. of potassium bromide.

$$\underbrace{AgNO_3}_{\begin{array}{r}10)\underline{168{\cdot}69}\\16{\cdot}869\end{array}} + \underbrace{KBr}_{\begin{array}{r}10)\underline{118{\cdot}18}\\11{\cdot}818\end{array}} = AgBr + KNO_3.$$

That is, 1000 c.c. $\frac{N}{10}$ $AgNO_3$ = 11·818 grams potassium bromide.

1 c.c. „ = 0·011818 „ „

Therefore 1 gram potassium bromide = 84·6 c.c. $\frac{N}{10}$ $AgNO_3$.

Potass. Iodid.—One gram of the salt treated as for Ammon. Bromid. should require for complete precipitation not less than 59·5, and not more than 61·9 c.c. $\frac{N}{10}$ $AgNO_3$, corresponding to 98·9 per cent. potassium iodide.

$$\underbrace{AgNO_3} + \underbrace{KI} = AgI \times KNO_3.$$
$$10)\underline{168{\cdot}69} \qquad 10)\underline{164{\cdot}73}$$
$$16{\cdot}869 \qquad\quad 16{\cdot}473$$

That is 1000 c.c. $\frac{N}{10}$ $AgNO_3$ = 16·473 grams potassium iodide.

1 c.c. „ = 0·016473 „ „

Therefore 1 gram potassium iodide = 60·7 c.c. $\frac{N}{10}$ $AgNO_3$ (1000 ÷ 16·473 = 60·7).

Note.—Bromides containing iodide take *less* silver nitrate for complete precipitation than the equation shows, but if the impurity is chloride they take *more.* It is for this reason that a definite weight of any soluble bromide must not take less than the B.P. minimum, and not more than the B.P. maximum.

Sodii Bromid.—One gram of dry salt treated as for Ammon. Bromid. should require for complete precipitation not less than 95·8, nor more than 97·8 c.c. $\frac{N}{10}$ $AgNO_3$, corresponding to at least 96·9 per cent. of sodium bromide.

$$\underbrace{AgNO_3} + \underbrace{NaBr} = AgBr + NaNO_3.$$
$$10)\underline{168{\cdot}69} \qquad 10)\underline{102{\cdot}23}$$
$$16{\cdot}869 \qquad\quad 10{\cdot}223$$

That is, 1000 c.c. $\frac{N}{10}$ $AgNO_3$ = 10·223 grams sodium bromide.

1 c.c. „ = 0·010223 „ „

Therefore 1 gram sodium bromide = 97·8 c.c. $\frac{N}{10}$ $AgNO_3$ (1000 ÷ 10·223 = 97·8).

Sodii Iodid.—One gram treated as for ammonium bromide should require for complete precipitation not less than 66·5 c.c. $\frac{N}{10}$ $AgNO_3$, corresponding to 98·9 per cent. by weight of sodium iodide.

$$\underbrace{AgNO_3} + \underbrace{NaI} = AgI + NaNO_3.$$
$$10)\underline{168{\cdot}69} \qquad 10)\underline{148{\cdot}78}$$
$$16{\cdot}869 \qquad\quad 14{\cdot}878$$

That is, 1000 c.c. $\frac{N}{10}$ $AgNO_3$ = 14·878 grams sodium iodide.

1 c.c. „ = 0·014878 „ „

Therefore 1 gram sodium iodide = 67·2 c.c. $\frac{N}{10}\,AgNO_3$ (1000 ÷ 14·878 = 67·2).

Syrup. Ferri Iodid.—Dissolve 1 gram dried sodium carbonate in 10 c.c. distilled water in a flask, of which the capacity to a mark on the neck is 100 c.c.; weigh into the flask 13·87 grams (= 10 c.c.) of the syrup, and agitate the mixture occasionally until the precipitation of the iron as ferrous carbonate is complete, then add more water to make the whole measure 100 c.c.; mix and filter. 25 c.c. of the filtrate (= 2·5 c.c. syrup), exactly neutralised with dilute nitric acid, should require not less than 16, and not more than 16·5 c.c. of $\frac{N}{10}\,AgNO_3$ for complete precipitation of the iodine, corresponding to 10 grams of ferrous iodide in 100 c.c. Use neutral potassium chromate solution as an indicator.

$$FeI_2 + Na_2CO_3 = FeCO_3 + 2NaI.$$
$$2NaI + 2AgNO_3 = 2AgI + 2NaNO_3.$$

$$\text{Therefore}\quad \underbrace{FeI_2}_{} \;=\; \underbrace{2\;AgNO_3}_{}.$$
$$\begin{array}{rr} 2)\overline{307{\cdot}4} & 2)\overline{337{\cdot}38} \\ 10)\overline{153{\cdot}7} & 10)\overline{168{\cdot}69} \\ \overline{15{\cdot}37} & \overline{16{\cdot}869} \end{array}$$

That is, 1000 c.c. $\frac{N}{10}\,AgNO_3$ = 15·37 grams ferrous iodide.

1 c.c. „ = 0·01537 „ „

Therefore 1 gram ferrous iodide = 65 c.c. $\frac{N}{10}\,AgNO_3$ (1000 ÷ 15·37 = 65·06).

But 100 c.c. of the syrup are only required to contain 10 grams of ferrous iodide, so that 2·5 c.c., the quantity of syrup really operated on, should contain ·25 gram. As 1 gram of ferrous iodide requires 65 c.c. $\frac{N}{10}\,AgNO_3$ for complete precipitation, ·25 gram will require 16·2 c.c. (65 × ·25 = 16·25 c.c.).

Volumetric solution Sodium Thiosulphate
($Na_2S_2O_3.5H_2O$ = 246·44).

This is a decinormal solution. It contains 24·644 grams in 1 litre.

Preparation.—

Crystalline sodium thiosulphate	28 grams.
Distilled water	a sufficient quantity.

Dissolve the salt in 1000 c.c. of the water, and adjust by titrating against $\frac{N}{10}$ I solution as follows:

Fill a burette with the "hypo" solution, and drop it cautiously into 100 c.c. $\frac{N}{10}$ I solution until only a faint yellow colour remains. Now add a few drops of starch mucilage, and continue the addition of the "hypo" until the blue colour is just discharged. Note the number of c.c. used to produce this effect, then put ten times this quantity of the same solution into the litre flask, and add sufficient distilled water to fill it up to the mark on the neck. One litre will then contain 24·644 grams sodium thiosulphate.

$$\underbrace{2\ Na_2S_2O_3.5H_2O}_{\begin{array}{r}2)\overline{492{\cdot}88}\\ 10)\overline{246{\cdot}44}\\ \hline 24{\cdot}644\end{array}} + \underbrace{I_2}_{\begin{array}{r}2)\overline{251{\cdot}8}\\ 10)\overline{125{\cdot}9}\\ \hline 12{\cdot}59\end{array}} = 2NaI + Na_2S_4O_6 + 10H_2O.$$

Note.—A *normal* solution of sodium thiosulphate would contain one molecular weight of the active reagent in grams in a litre, because according to the above equation 2 molecules of the salt have 2 atoms of available Na, which are equivalent to 2 atoms of H (or 2 atoms of I). Therefore 1 molecule is equivalent to 1 atom of H. As the pharmacopœial volumetric solution only contains 24·644 grams in 1 litre, it is decinormal.

Instead of using sodium thiosulphate, other reducing agents, such as arsenious acid, sulphurous acid, or ferrous sulphate, might be used for quantitatively determining substances which either yield a definite amount of oxygen, or which, from their great affinity for hydrogen, act as indirect oxidisers in the presence of water, as iodine and chlorine.

Calx Chlorinata.—·5 gram mixed with 1·5 grams potassium iodide dissolved in 200 c.c. water produces, when acidulated with 6 c.c. hydrochloric acid, a reddish solution, which requires for the discharge of its colour at least 46·8 c.c. $\frac{N}{10}$ $Na_2S_2O_3.5H_2O$, corresponding to 33 per cent. available chlorine. Use a few drops of fresh starch mucilage as an indicator towards the close of the reaction.

1. $CaOCl_2 + 2HCl = CaCl_2 + H_2O + Cl_2$.
2. $Cl_2 + 2KI = 2KCl + I_2$.
3. $2Na_2S_2O_3.5H_2O + I_2 = 2NaI + Na_2S_4O_6 + 10H_2O$.

$$\text{Therefore } \underbrace{2Na_2S_2O_3.5H_2O}_{\begin{array}{r}2)\underline{492{\cdot}88}\\10)\underline{246{\cdot}44}\\24{\cdot}644\end{array}} = I_2 = \underbrace{Cl_2}_{\begin{array}{r}2)\underline{70{\cdot}38}\\10)\underline{35{\cdot}19}\\3{\cdot}519\end{array}} = \underbrace{CaOCl_2}_{125{\cdot}97}.$$

That is, 1000 c.c. $\frac{N}{10}$ $Na_2S_2O_3.5H_2O$ = 3·519 grams chlorine.

$$\text{According to the above equations } \underbrace{CaOCl_2}_{125{\cdot}97} \text{ yields } \underbrace{Cl_2}_{70{\cdot}38}.$$

As 125·97 grams $CaOCl_2$ yield 70·38 grams chlorine, 100 grams will yield 55·87 $\left(\frac{70{\cdot}38 \times 100}{125{\cdot}97}\right)$. As 100 grams $CaOCl_2$ theoretically yield 55·87 grams of chlorine, ·5 gram should yield ·27935 gram (55·87 × ·5 ÷ 100).

Now if 3·519 grams of chlorine (see equation above) are equivalent to 1000 c.c. $\frac{N}{10}$ sol. $Na_2S_2O_3.5H_2O$, it follows that ·27935 gram chlorine is equivalent to 79·38 c.c. (·27935 × 1000 ÷ 3·519).

But this is supposing that the theoretical yield of 55·87 per cent. of chlorine is obtained. As only 33 per cent. is evolved, ·5 gram $CaOCl_2$ should require 46·8 c.c. (79·38 × 33 ÷ 55·87).

Liquor Calcis Chlorinatæ.—Sp. gr. 1·055. One gram mixed with ·5 gram potassium iodide dissolved in water, when acidulated with 3 c.c. hydrochloric acid, yields a reddish solution which requires for the discharge of its colour not less than 5·6 c.c. $\frac{N}{10}$ $Na_2S_2O_3.5H_2O$, corresponding to 2 per cent. of available chlorine. When *freshly* made, the solution should yield about 3 per cent. available chlorine. For reactions and calculations see Calx Chlorinata. (Indicator, starch mucilage.)

Liquor Sodæ Chlorinatæ.—Sp. gr. 1·054. 3·5 grams mixed with 1 gram potassium iodide dissolved in 100 c.c. water, and acidulated with 6 c.c. hydrochloric acid, yield a reddish solution, which requires for the discharge of its colour at least 25 c.c. $\frac{N}{10}$ $Na_2S_2O_3.5H_2O$, corresponding to about 2½ per cent. available chlorine. For reactions and calculations see Calx Chlorinata. (Indicator, starch mucilage.)

Iodum.—One gram dissolved in 50 c.c. distilled water containing 2 grams of potassium iodide should require for complete decoloration at least 78·4 c.c. $\frac{N}{10}$ $Na_2S_2O_3.5H_2O$, corresponding to nearly 99 per cent. of actual iodine. (Indicator—*towards the end,*—starch mucilage.)

$$\underbrace{2Na_2S_2O_3.5H_2O}_{\begin{array}{r} 2)\overline{492{\cdot}88} \\ 10)\overline{246{\cdot}44} \\ \overline{24{\cdot}644} \end{array}} + \underbrace{I_2}_{\begin{array}{r} 2)\overline{251{\cdot}8} \\ 10)\overline{125{\cdot}9} \\ \overline{12{\cdot}59} \end{array}} = 2NaI + Na_2S_4O_6 + 10H_2O.$$

That is—

$$1000 \text{ c.c. } \frac{N}{10}\ Na_2S_2O_3.5H_2O = 12{\cdot}59 \text{ grams of iodine.}$$

$$1 \text{ c.c. } \frac{N}{10} \quad \text{,,} \quad = 0{\cdot}01259 \quad \text{,,} \quad \text{,,}$$

Therefore 1 gram of iodine = 79·4 c.c. $\frac{N}{10}$ $Na_2S_2O_3.5H_2O$ (1000 ÷ 12·59 = 79·4).

Tinct. Iodi.—Ten c.c. diluted with 20 c.c. of water should require for complete decoloration 19·6 c.c. $\frac{N}{10}$ $Na_2S_2O_3.5H_2O$, corresponding to 2·46 per cent. of iodine.

Volumetric solution of Sulphuric Acid
(H_2SO_4 = 97·34).

This is a normal solution. It contains 48·67 grams in 1 litre. The molecule of sulphuric acid, H_2SO_4, contains 2 atoms of hydrogen replaceable by a base; its normal solution therefore, contains *half* the molecular weight in grams in 1 litre of solution.

Preparation.—

Sulphuric acid	50 grams.
Distilled water	a sufficient quantity.

Dilute the sulphuric acid with 900 c.c. of distilled water and cool. Prepare a small quantity of sodium carbonate by heating the pure bicarbonate to redness in a platinum crucible for a quarter of an hour, afterwards cooling in a desiccator. Make a solution of 1·053 grams of the sodium carbonate, and add to it from a burette the solution of sulphuric acid, until exact neutrality is obtained, care being taken to boil off the carbonic anhydride. Note the number of c.c. used, then put 50 times this quantity of the same solution into the litre flask, and add sufficient distilled water to fill it up to the mark on the neck. (Indicator, litmus.)

One thousand c.c. then contain exactly 48·67 grams sulphuric acid. *A decinormal volumetric solution of sulphuric acid* may be prepared by adding to 100 c.c. of the above solution sufficient distilled water to produce 1000 c.c. ($\frac{N}{10}$ H_2SO_4 = 4·867 grams in 1000 c.c.).

$$\underbrace{H_2SO_4}_{\substack{2)\underline{97{\cdot}34}\\ 48{\cdot}67}} + \underbrace{Na_2CO_3}_{\substack{2)\underline{105{\cdot}31}\\ 52{\cdot}655}} = Na_2SO_4 + H_2O + CO_2.$$

Ammonii Carbonas.—The official salt is a mixture of ammonium bicarbonate and carbamate, the latter forming normal carbonate when dissolved in water; in reality, therefore, a mixture of neutral and acid carbonates are titrated.

One gram dissolved in 40 c.c. distilled water should require for neutralisation at least 18·7 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 97·3 per cent. of the official salt. (Indicator, methyl orange.)

$$2H_2O + 2NH_4HCO_3.NH_4NH_2CO_2 = 2NH_4HCO_3.(NH_4)_2CO_3.$$

Then—

$$\underbrace{2NH_4HCO_3.(NH_4)_2CO_3}_{\substack{3)\underline{312{\cdot}08}\\ 2)\underline{104{\cdot}02}\\ 52{\cdot}01}} + \underbrace{3H_2SO_4}_{\substack{3)\underline{292{\cdot}02}\\ 2)\underline{97{\cdot}34}\\ 48{\cdot}67}} = 3(NH_4)_2SO_4 + 4CO_2 + 4H_2O.$$

That is—

1000 c.c. $\frac{N}{1}$ H_2SO_4 = 52·01 grams of the pharmacopœial salt.

1 c.c. ,, = 0·05201 ,, ,, ,,

Therefore 1 gram of the pharmacopœial salt = 19·2 c.c. $\frac{N}{1}$

H_2SO_4 (1000 ÷ 52·01 = 19·2 c.c.). As 19·2 c.c. = 100 per cent. 18·7 c.c. = 97·3 per cent.

Liquor Ammoniæ Fortior (sp. gr. ·891).—One gram mixed with a little distilled water should require for neutralisation, 19·1 c.c. $\frac{N}{1}$ H_2SO_4 corresponding to 32·5 per cent. by weight of ammonia gas. (Indicator, methyl orange.)

$$\underbrace{2NH_3.OH_2}_{\substack{2)33\cdot88 \\ 16\cdot94}} + \underbrace{H_2SO_4}_{\substack{2)97\cdot34 \\ 48\cdot67}} = (NH_4)_2SO_4 + 2H_2O.$$

That is—

1000 c.c. $\frac{N}{1}$ H_2SO_4 = 16·94 grams ammonia gas, NH_3.
1 c.c. ,, = 0·01694 ,, ,,

Therefore 1 gram of ammonia gas = 59·03 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 16·94 = 59·03).

But the Pharmacopœia only requires the solution to contain 32·5 per cent. by weight of ammonia gas, therefore 59·03 × 32·5 ÷ 100 = 19·18 c.c.

Liquor Ammoniæ (sp. gr. ·959).—One gram treated as for Liquor Ammoniæ Fortior should require for neutralisation at least 5·9 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 10 per cent. by weight of ammonia gas (59·03 × 10 ÷ 100 = 5·9 c.c.).

Sp. Ammoniæ Aromaticus (sp. gr. ·888—·893).—Twenty cubic centimetres require for neutralisation 25·5 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to about 2·4 per cent. of ammonia gas, NH_3, or 2·16 grams in 100 c.c. (Indicator, methyl orange.)

The co-efficient of ammonia gas for 1 c.c. of $\frac{N}{1}$ H_2SO_4 is ·01694. Therefore 25·5 c.c. $\frac{N}{1}$ H_2SO_4 correspond to ·4319 gram of NH_3, and as this weight is contained in 20 c.c., 2·159 grams must be contained in 100 c.c.

Sp. Ammoniæ Fœtidus.—Twenty-five cubic centimetres should require for neutralisation at least 42·5 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to nearly 2·88 grams of ammonia gas, NH_3, in 100 c.c. (Indicator, methyl orange.)

The co-efficient of NH_3 for 1 c.c. $\frac{N}{1}$ H_2SO_4 is ·01694. Therefore 42·5 c.c. $\frac{N}{1}$ H_2SO_4 correspond to ·7199 gram of NH_3, and as this weight is contained in 20 c.c., 2·879 grams must be contained in 100 c.c.

Borax.—One gram dissolved in 200 c.c. distilled water should require for neutralisation 5·2 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 98·6 per cent. of crystallised borax. (Indicator, methyl orange.)

$$\underbrace{Na_2B_4O_7.10H_2O}_{\substack{2)\overline{379{\cdot}12} \\ 189{\cdot}56}} + \underbrace{H_2SO_4}_{\substack{2)\overline{97{\cdot}34} \\ 48{\cdot}67}} = Na_2SO_4 + H_2B_4O_7 + 10H_2O.$$

That is—

1000 c.c. $\frac{N}{1}$ H_2SO_4 = 189·56 grams crystallised borax.

1 c.c. " = 0·18956 " "

Therefore 1 gram crystallised borax = 5·27 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 189·56 = 5·27).

Liquor Calcis Saccharatus.—The lime exists as calcium saccharosate, $C_{12}H_{20}(CaOH)_2O_{11}$. Ten grams should require for neutralisation 6·3 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 1·75 per cent. of calcium oxide. (Indicator, litmus.)

$$C_{12}H_{20}{\underbrace{(CaOH)}_{\substack{4)\overline{111{\cdot}18} \\ 27{\cdot}79}}}_2O_{11} + \underbrace{2H_2SO_4}_{\substack{4)\overline{194{\cdot}68} \\ 48{\cdot}67}} = C_{12}H_{22}O_{11} + 2CaSO_4 + 2H_2O.$$

That is—

1000 c.c. $\frac{N}{1}$ H_2SO_4 = 27·79 grams of calcium oxide.

1 c.c. " = 0·2779 " "

6·3 c.c. " = ·175 " "

Therefore 10 grams of Liq. Calcis Saccharatus contain ·175 gram of calcium oxide, equivalent to 1·75 per cent.

Liquor Calcis.—Twenty-four cubic centimetres should require for neutralisation 10 c.c. $\frac{N}{10}$ H_2SO_4, corresponding to about ·1 per cent. of calcium oxide. (Indicator, litmus.)

$$\underbrace{CaO.H_2O}_{\begin{array}{r}2)\overline{55{\cdot}59}\\10)\overline{27{\cdot}79}\\2{\cdot}779\end{array}} + \underbrace{H_2SO_4}_{\begin{array}{r}2)\overline{97{\cdot}34}\\10)\overline{48{\cdot}67}\\4{\cdot}867\end{array}} = CaSO_4 + 2H_2O.$$

That is—

1000 c.c. $\frac{N}{10}$ H_2SO_4 = 2·779 grams of calcium oxide.

1 c.c. ,, = ·002779 ,, ,,

10 c.c. ,, = ·02779 ,, ,,

Therefore 24 c.c. Liquor Calcis = ·02779 gram of calcium oxide, equivalent to ·115 gram of oxide in 100 c.c.

Liq. Plumbi Fort (sp. gr. 1·275).—One gram diluted with a little distilled water should require for complete precipitation 17 c.c. $\frac{N}{10}$ H_2SO_4, corresponding to about 23 per cent. of lead oxyacetate.

$$\underbrace{Pb_2O(C_2H_3O_2)_2}_{\begin{array}{r}4)\overline{543{\cdot}74}\\10)\overline{135{\cdot}93}\\13{\cdot}59\end{array}} + \underbrace{2H_2SO_4}_{\begin{array}{r}4)\overline{194{\cdot}68}\\10)\overline{48{\cdot}67}\\4.867\end{array}} = 2PbSO_4 + 2HC_2H_3O_2 + H_2O.$$

That is—

1000 c.c. $\frac{N}{10}$ H_2SO_4 = 13·59 grams lead oxyacetate.

1 c.c. ,, = 0·01359 ,, ,,

Therefore 1 gram lead oxyacetate = 73·5 c.c. $\frac{N}{10}$ H_2SO_4 (1000 ÷ 13·59 = 73·5).

But the pharmacopœial solution is only required to contain 23 per cent. of lead oxyacetate, therefore 73·5 × 23 ÷ 100 = 17 c.c.

Plumbi Acetas.—One gram dissolved in a little distilled water should require for complete precipitation 53·1 c.c. $\frac{N}{10}$ H_2SO_4, corresponding to nearly 100 per cent. of lead acetate.

$$\underbrace{Pb(C_2H_3O_2)_2.3H_2O}_{\begin{array}{r} 2)\overline{376{\cdot}15} \\ 10)\overline{188{\cdot}07} \\ \hline 18{\cdot}807 \end{array}} + \underbrace{H_2SO_4}_{\begin{array}{r} 2)\overline{97{\cdot}34} \\ 10)\overline{48{\cdot}67} \\ \hline 4{\cdot}867 \end{array}} = PbSO_4 + 2HC_2H_3O_2 + 3H_2O.$$

That is—

1000 c.c. $\frac{N}{10}$ H_2SO_4 = 18·807 grams crystallised lead acetate.
1 c.c. ,, = 0·018807 ,, ,, ,,

Therefore 1 gram crystallised lead acetate = 53·17 c.c. $\frac{N}{10}$ H_2SO_4 (1000 ÷ 18·807 = 53·17).

Liquor Potassæ (sp. gr. 1·058).—Nine c.c. should require for neutralisation 10 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 6·19 grams of potassium hydroxide in 100 c.c. (Indicator, litmus.)

$$\underbrace{2KOH}_{\begin{array}{r} 2)\overline{111{\cdot}42} \\ \hline 55{\cdot}71 \end{array}} + \underbrace{H_2SO_4}_{\begin{array}{r} 2)\overline{97{\cdot}34} \\ \hline 48{\cdot}67 \end{array}} = K_2SO_4 + 2H_2O.$$

That is, 1000 c.c. $\frac{N}{1}$ H_2SO_4 = 55·71 grams potassium hydroxide.
1 c.c. ,, = 0·05571 ,, ,,
10 c.c. ,, = 0·5571 ,, ,,

Therefore 9 c.c. Liq. Potas. = 0·5571 ,, ,,
and 100 c.c. ,, = 6·19 ,, ,,

Potassa Caustica.—One gram dissolved in water to which a little litmus solution has been added, and well boiled during the reaction to expel carbonic anhydride (carbonate being an impurity commonly present in the commercial salt), should require for neutralisation at least 16·1 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 89·9 per cent. potassium hydroxide. For equation, see above (Liq. Potassæ).

That is—

1000 c.c. $\frac{N}{1}$ H_2SO_4 = 55·71 grams potassium hydroxide.

1 c.c. ,, = 0·05571 ,, ,,

Therefore 1 gram potassium hydroxide = 17·9 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 55·71 = 17·9).

Then if 17·9 c.c. = 100 per cent., 16·1 c.c. = 89·9 per cent.

Potassii Bicarbonas.—One gram exposed to a low red heat for a few minutes should leave ·69 gram normal carbonate, which when added to water containing a few drops of litmus solution, and well boiled during the operation to expel carbonic anhydride, requires exactly 10 c.c. $\frac{N}{1}$ H_2SO_4 for neutralisation, corresponding to 99·3 per cent. potassium bicarbonate.

$$\underbrace{2KHCO_3}_{\begin{array}{r}2)\underline{198{\cdot}76}\\ 99{\cdot}38\end{array}} + \underbrace{H_2SO_4}_{\begin{array}{r}2)\underline{97{\cdot}34}\\ 48{\cdot}67\end{array}} = K_2SO_4 + 2CO_2 + 2H_2O.$$

That is—

1000 c.c. $\frac{N}{1}$ H_2SO_4 = 99·38 grams potassium bicarbonate.

1 c.c. ,, = 0·09938 ,, ,,

Therefore 1 gram potassium bicarbonate = 10·06 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 99·38 = 10·06).

Potassii Carbonas—One gram, dissolved in water containing a few drops of litmus solution, and well boiled during the operation to expel carbonic anhydride, should require for neutralisation at least 11·9 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 82·06 per cent. of potassium carbonate.

$$\underbrace{K_2CO_3}_{\begin{array}{r}2)\underline{137{\cdot}21}\\ 68{\cdot}60\end{array}} + \underbrace{H_2SO_4}_{\begin{array}{r}2)\underline{97{\cdot}34}\\ 48{\cdot}67\end{array}} = K_2SO_4 + H_2O + CO_2.$$

That is, 1000 c.c. $\frac{N}{1}$ H_2SO_4 = 68·6 grams potassium carbonate.

1 c.c. ,, = 0·0686 ,, ,,

Therefore 1 gram potassium carbonate = 14·5 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 68·6 = 14·5). As 14·5 c.c. = 100 per cent., 11·9 c.c. = 82·06 per cent.

Potassii Citras.—One gram heated to redness until gases cease to be evolved should leave an alkaline residue (K_2CO_3), which when boiled with distilled water, filtered, and the filter well washed, should yield a clear solution, requiring for neutralisation at least 9·7 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 99·2 per cent. potassium citrate. Use litmus indicator, and boil during the operation to expel carbonic anhydride.

$$2K_3C_6H_5O_7 + 9O_2 = 3K_2CO_3 + 9CO_2 + 5H_2O.$$

$$\text{Therefore } \underbrace{2K_3C_6H_5O_7}_{607{\cdot}38} = \underbrace{3K_2CO_3}_{411{\cdot}63}.$$

$$\text{Then } \underbrace{3K_2CO_3}_{411{\cdot}63} + \underbrace{3H_2SO_4}_{292{\cdot}02} = 3K_2SO_4 + 3H_2O + 3CO_2.$$

$$\text{Therefore } \underbrace{2K_3C_6H_5O_7}_{\begin{array}{r}3)\underline{607{\cdot}38}\\2)\underline{202{\cdot}46}\\101{\cdot}23\end{array}} = \underbrace{3H_2SO_4}_{\begin{array}{r}3)\underline{292{\cdot}02}\\2)\underline{97{\cdot}34}\\48{\cdot}67\end{array}}.$$

That is, 1000 c.c. $\frac{N}{1}$ H_2SO_4 = 101·23 grams potassium citrate.

1 c.c. ,, = 0·10123 ,, ,,

Therefore 1 gram potassium citrate = 9·77 c.c. $\frac{N}{1}$ H_2SO_4 $\left(\frac{1000}{101{\cdot}23} = 9{\cdot}77\right)$.

Potassii Tartras.—One gram, treated as for Potassii Citras, should yield a clear solution, requiring for neutralisation at least 8·4 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 98 per cent. of potassium tartrate.

$$\underbrace{(K_2C_4H_4O_6)_2.H_2O}_{467{\cdot}04} + 5O_2 = \underbrace{2K_2CO_3}_{274{\cdot}42} + 6CO_2 + 5H_2O.$$

$$\text{Then } \underbrace{2K_2CO_3}_{274{\cdot}42} + \underbrace{2H_2SO_4}_{194{\cdot}68} = 2K_2SO_4 + 2CO_2 + 2H_2O.$$

$$\text{Therefore } \underbrace{2H_2SO_4}_{\begin{array}{r}4)\underline{194{\cdot}68}\\48{\cdot}67\end{array}} = \underbrace{(K_2C_4H_4O_6)_2H_2O}_{\begin{array}{r}4)\underline{467{\cdot}04}\\116{\cdot}76\end{array}}.$$

That is—

1000 c.c. $\frac{N}{1}$ H_2SO_4 = 116·76 grams neutral potassium tartrate.

1 c.c. ,, = 0·11676 ,, ,,

Therefore 1 gram neutral potassium tartrate = 8·56 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 116·76 = 8·56). As 8·56 c.c. = 100 per cent., 8·4 c.c. = 98 per cent.

Soda Tartarata.—One gram, treated as for Potassii Citras, should yield a clear solution, requiring for neutralisation at least 7 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 98·1 per cent. of tartarated soda.

$$\underbrace{2KNaC_4H_4O_6.4H_2O}_{560{\cdot}30} + 5O_2 = \underbrace{2KNaCO_3}_{242{\cdot}52} + 6CO_2 + 12H_2O.$$

$$\text{*Then } \underbrace{2KNaCO_3}_{242{\cdot}52} + \underbrace{2H_2SO_4}_{194{\cdot}68} = 2KNaSO_4 + 2CO_2 + 2H_2O.$$

$$\text{Therefore } \underbrace{2KNaC_4H_4O_6.4H_2O}_{\begin{array}{r}4)\underline{560{\cdot}30}\\ 140{\cdot}07\end{array}} = \underbrace{2H_2SO_4}_{\begin{array}{r}4)\underline{194{\cdot}68}\\ 48{\cdot}67\end{array}}.$$

That is, 1000 c.c. $\frac{N}{1}$ H_2SO_4 = 140·07 grams Soda Tartarata.

1 c.c. ,, = 0·14007 ,, ,,

Therefore 1 gram Soda Tartarata = 7·13 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 140·07 = 7·13).

Sodii Benzoas.—One gram of the salt, when heated, melts, emitting an odour of benzoin, then chars, and finally leaves a residue which, when treated as for Potassii Citras, should yield a clear solution, requiring for neutralisation from 6·8 c.c. to 6·9 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to nearly 100 per cent. sodium benzoate.

$$\underbrace{2NaC_7H_5O_2}_{286{\cdot}02} + 15O_2 = \underbrace{Na_2CO_3}_{105{\cdot}31} + 13CO_2 + 5H_2O.$$

$$\text{Then } \underbrace{Na_2CO_3}_{105{\cdot}31} + \underbrace{H_2SO_4}_{97{\cdot}34} = Na_2SO_4 + H_2O + CO_2.$$

---

* Equations written as above to save space. Probably sodium and potassium carbonates, and sulphates, are actually formed.

$$\text{Therefore } \underbrace{2NaC_7H_5O_2}_{\substack{2)\underline{286{\cdot}02}\\ 143{\cdot}01}} = \underbrace{H_2SO_4}_{\substack{2)\underline{97{\cdot}34}\\ 48{\cdot}67}}$$

That is, 1000 c.c. $\frac{N}{1}$ H_2SO = 143·01 grams sodium benzoate.

1 c.c. „ = 0·14301 „ „

Therefore 1 gram sodium benzoate = 6·9 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 143·01 = 6·9).

Sodii Bicarbonas.—One gram, dissolved in distilled water, should require for neutralisation 11·8 to 11·9 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to nearly 99 per cent. sodium bicarbonate.

Use litmus indicator, and boil to expel carbonic anhydride.

$$\underbrace{2NaHCO_3}_{\substack{2)\underline{166{\cdot}86}\\ 83{\cdot}43}} + \underbrace{H_2SO_4}_{\substack{2)\underline{97{\cdot}34}\\ 48{\cdot}67}} = Na_2SO_4 + 2H_2O + 2CO_2.$$

That is, 1000 c.c. $\frac{N}{1}$ H_2SO_4 = 83·43 grams sodium bicarbonate.

1 c.c „ = 0·08343 „ „

Therefore 1 gram sodium bicarbonate = 11·98 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 83·43 = 11·98).

Sodii Carbonas.—One gram dissolved in distilled water should require for neutralisation at least 6·9 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 98·1 per cent. sodium carbonate.

Use litmus indicator, and boil to expel carbonic anhydride.

$$\underbrace{Na_2CO_3.10H_2O}_{\substack{2)\underline{284{\cdot}11}\\ 142{\cdot}05}} + \underbrace{H_2SO_4}_{\substack{2)\underline{97{\cdot}34}\\ 48{\cdot}67}} = Na_2SO_4 + 11H_2O + CO_2.$$

That is, 1000 c.c. $\frac{N}{1}$ H_2SO_4 = 142·05 grams sodium carbonate.

1 c.c. „ = 0·14205 „ „

Therefore 1 gram sodium carbonate = 7·03 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 142·05 = 7·03). As 7·03 c.c. = 100 per cent., 6·9 c.c. = 98·1 per cent.

Sodium.—One gram very cautiously added to water affords a solution which should require for neutralisation at least 42·6 c.c. $\frac{N}{1}$ H_2SO_4, corresponding to 97·4 per cent. of sodium.

$$\underbrace{Na_2(OH)_2}_{\substack{2)45{\cdot}76 \\ 22{\cdot}88}} + \underbrace{H_2SO_4}_{\substack{2)97{\cdot}34 \\ 48{\cdot}67}} = Na_2SO_4 + 2H_2O.$$

That is, 1000 c.c. $\frac{N}{1}$ H_2SO_4 = 22·88 grams metallic sodium.

1 c.c. „ = 0·02288 „ „

Therefore 1 gram metallic sodium = 43·7 c.c. $\frac{N}{1}$ H_2SO_4 (1000 ÷ 22·88 = 43·7).

Volumetric solution of Sodium Hydroxide
(NaOH = 39·76).

This is a normal solution. It contains 39·76 grams in 1 litre. The molecule of sodium hydroxide has but one sodium atom to replace one hydrogen atom in an acid, hence its normal solution contains the molecular weight in grams in 1 litre.

Preparation.—

Sodium hydroxide	45 grams.
Distilled water	a sufficient quantity.

Dissolve the sodium hydroxide in 1000 c.c. distilled water; fill a burette with the solution, and cautiously run into a vessel containing 100 c.c. $\frac{N}{1}$ H_2SO_4, until the acid is exactly neutralised. (Use litmus indicator, and boil to expel carbonic anhydride, carbonate being a common impurity in the commercial salt.)

Note the number of c.c. used, then put ten times this quantity of the same solution into the litre flask, and add sufficient distilled water to fill it up to the mark on the neck. 1000 c.c. then contain exactly 39·76 grams sodium hydroxide.

A decinormal $\left(\frac{N}{10}\right)$ solution may be prepared by adding 100 c.c. of the above to sufficient distilled water to produce 1000 c.c.

Ninety per cent. alcohol may when necessary be used as the solvent for the alkali. An equivalent proportion of potassium hydroxide (KOH 55·71) may in certain cases be employed in place of sodium hydroxide.

Acetic acid (sp. gr. 1·044).—One gram should require for neutralisation 5·5 c.c. $\frac{N}{1}$ NaOH, corresponding to 33 per cent. by weight of hydrogen acetate. (Indicator, phenol-phthalein.)

$$\underbrace{HC_2H_3O_2}_{59\cdot58} + \underbrace{NaOH}_{39\cdot76} = NaC_2H_3O_2 + H_2O.$$

That is, 1000 c.c. $\frac{N}{1}$ NaOH = 59·58 grams hydrogen acetate.
1 c.c. ,, = 0·05958 ,, ,,

Therefore 1 gram hydrogen acetate = 16·78 c.c. $\frac{N}{1}$ NaOH (1000 ÷ 59·58 = 16·78).

But Acid. Acetic., P.B., is only required to contain 33 per cent. of hydrogen acetate, therefore 16·7 × 33 ÷ 100 = 5·51 c.c.

Acid. Acetic. Dilutum (sp. gr. 1·006).—One gram should require for neutralisation 7·1 c.c. $\frac{N}{10}$ NaOH, corresponding to about 4·27 per cent. by weight hydrogen acetate. (Indicator, phenol-phthalein.)

1000 c.c. $\frac{N}{10}$ NaOH = 5·958 grams hydrogen acetate.
1 c.c. ,, = 0·005958 ,, ,,

One gram hydrogen acetate = 167·8 c.c. $\frac{N}{10}$ NaOH.

But Acid. Acetic. Dil. is only required to contain 4·27 per cent. by weight of hydrogen acetate, therefore 167·8 × 4·27 ÷ 100 = 7·16.

Acid. Acetic. Glacial (sp. gr. 1·058).—One gram diluted with 50 c.c. distilled water should require for neutralisation 16·6 c.c. $\frac{N}{1}$ NaOH, corresponding to 99 per cent. by weight of hydrogen acetate. (Indicator, phenol-phthalein.)

One gram hydrogen acetate = 16·78 c.c. $\frac{N}{1}$ NaOH (see Acid. Acetic.).

Acid. Citricum.—One gram dissolved in distilled water should require for neutralisation 14·3 c.c. $\frac{N}{1}$ NaOH, corresponding to 100 per cent. by weight of hydrogen citrate. (Indicator, phenol-phthalein.)

$$\underbrace{H_3C_6H_5O_7.H_2O}_{\substack{3)\underline{208{\cdot}5}\\ 69{\cdot}5}} + \underbrace{3NaOH}_{\substack{3)\underline{119{\cdot}28}\\ 39{\cdot}76}} = Na_3C_6H_5O_7 + 10H_2O.$$

That is, 1000 c.c. $\frac{N}{1}$ NaOH = 69·5 grams hydrogen citrate.

1 c.c. ,, = 0·0695 ,, ,,

Therefore 1 gram hydrogen citrate = 14·3 c.c. $\frac{N}{1}$ NaOH (1000 ÷ 69·5 = 14·3).

Acid. Hydrobromic. Dil.—Sp. gr. 1·077. Four grams require for neutralisation 5 c.c. $\frac{N}{1}$ NaOH, corresponding to about 10 per cent. by weight of hydrogen bromide. Litmus indicator, and boil.

$$\underbrace{HBr}_{80{\cdot}35} + \underbrace{NaOH}_{39{\cdot}76} = NaBr + H_2O.$$

That is, 1000 c.c. $\frac{N}{1}$ NaOH = 80·35 grams hydrogen bromide.

1 c.c. ,, = 0·08035 ,, ,,

Therefore—

1 gram hydrogen bromide = 12·4 c.c. $\frac{N}{1}$ NaOH (1000 ÷ 80·35 = 12·4).

4 grams ,, = 49·6 c.c. ,,

But the pharmacopœial solution is only required to contain 10 per cent. by weight of hydrogen bromide, therefore 49·6 × 10 ÷ 100 = 4·96 c.c.

Acid. Hydrochloric.—Sp. gr. 1·16. One gram diluted with water should require for neutralisation 8·7 c.c. $\frac{N}{1}$ NaOH, corresponding to 31·79 per cent. by weight of hydrogen chloride. Litmus indicator, and boil.

$$\underbrace{HCl}_{36{\cdot}19} + \underbrace{NaOH}_{39{\cdot}76} = NaCl + H_2O.$$

That is, 1000 c.c. $\frac{N}{1}$ NaOH = 36·19 grams hydrogen chloride.

1 c.c. ,, = 0·03619 ,, ,,

Therefore 1 gram hydrogen chloride = 27·6 c.c. $\frac{N}{1}$ NaOH (1000 ÷ 36·19 = 27·6).

But the pharmacopœial solution is only required to contain 31·79 per cent. by weight of hydrogen chloride, therefore 31·79 × 27·6 ÷ 100 = 8·77 c.c.

Acid. Hydrochlor. Dil.—Sp. gr. 1·052. One gram should require for neutralisation 2·9 c.c. $\frac{N}{1}$ NaOH, corresponding to 10·58 per cent. by weight of hydrogen chloride (27·6 × 10·58 ÷ 100 = 2·92 c.c.).

Acid. Lacticum.—Sp. gr. 1·21. One gram diluted with water should require for neutralisation 8·3 c.c. $\frac{N}{1}$ NaOH, corresponding to 75 per cent. hydrogen lactate. Indicator, phenol-phthalein.

$$\underbrace{HC_3H_5O_3}_{89\cdot37} + \underbrace{NaOH}_{39\cdot76} = NaC_3H_5O_3 + H_2O.$$

That is, 1000 c.c. $\frac{N}{1}$ NaOH = 89·37 grams hydrogen lactate.

1 c.c. " = 0·08937 " "

Therefore 1 gram hydrogen lactate = 11·1 c.c. (1000 ÷ 89·37 = 11·1 c.c.).

But the pharmacopœial solution is only required to contain 75 per cent. by weight, therefore 11·1 × 75 ÷ 100 = 8·3.

Acid. Nitricum.—Sp. gr. 1·42. One gram diluted with water should require for neutralisation 11·1 c.c. $\frac{N}{1}$ NaOH, corresponding to 70 per cent. by weight hydrogen nitrate. Litmus indicator, and boil.

$$\underbrace{HNO_3}_{62\cdot58} + \underbrace{NaOH}_{39\cdot76} = NaNO_3 + H_2O.$$

That is, 1000 c.c. $\frac{N}{1}$ NaOH = 62·58 grams hydrogen nitrate.

1 c.c. " = ·06258 " "

Therefore 1 gram hydrogen nitrate = 15·9 c.c. $\frac{N}{1}$ NaOH (1000 ÷ 62·58 = 15·9 c.c.).

But the pharmacopœial solution is only required to contain 70 per cent. by weight of hydrogen nitrate, therefore 15·9 × 70 ÷ 100 = 11·13 c.c.

Acid. Nitric. Dilutum.—Sp. gr. 1·101. One gram should require for neutralisation 2·7 c.c. $\frac{N}{1}$ NaOH, corresponding to 17·44 per cent. by weight of hydrogen nitrate. Therefore 15·9 × 17·74 ÷ 100 = 2·77 c.c.

Acid. Nitro-hydrochlor. Dilutum.—Sp. gr. 1·07. Four grams should require for neutralisation 10 c.c. $\frac{N}{1}$ NaOH. Litmus indicator, and boil.

Acid. Sulphuric.—Sp. gr. 1·843. One gram diluted with water should require for neutralisation 20·1 c.c. $\frac{N}{1}$ NaOH, corresponding to 98 per cent. by weight of hydrogen sulphate. Use litmus indicator, and boil.

$$\underbrace{H_2SO_4}_{\begin{array}{r}2)\underline{97{\cdot}34}\\48{\cdot}67\end{array}} + \underbrace{2NaOH}_{\begin{array}{r}2)\underline{79{\cdot}52}\\39{\cdot}76\end{array}} = Na_2SO_4 + 2H_2O.$$

That is—

1000 c.c. $\frac{N}{1}$ NaOH = 48·67 grams hydrogen sulphate.

1 c.c. ,, = 0·04867 ,, ,,

Therefore 1 gram hydrogen sulphate = 20·54 c.c. $\frac{N}{1}$ NaOH (1000 ÷ 48·67 = 20·54 c.c.).

But the pharmacopœial solution is only required to contain 98 per cent. by weight of hydrogen sulphate, therefore 20·54 × 98 ÷ 100 = 20·1.

Acid. Sulph. Dilutum.—Sp. gr. 1·094. One gram should require for neutralisation 2·8 c.c. $\frac{N}{1}$ NaOH, corresponding to 13·65 per cent. by weight of hydrogen sulphate.

If 20·54 c.c. $\frac{N}{1}$ NaOH = 100 per cent. hydrogen sulphate, 2·8 c.c. = 13·65 per cent.

Acid. Tartaric.—One gram dissolved in water should require for neutralisation 13·3 c.c. $\frac{N}{1}$ NaOH, corresponding to nearly 100 per cent. of hydrogen tartrate. Indicator, phenol-phthalein.

$$\underbrace{H_2C_4H_4O_6}_{\substack{2)\underline{148{\cdot}92} \\ 74{\cdot}46}} + \underbrace{2NaOH}_{\substack{2)\underline{79{\cdot}52} \\ 39{\cdot}76}} = Na_2C_4H_4O_6 + 2H_2O.$$

That is—

1000 c.c. $\frac{N}{1}$ NaOH = 74·46 grams hydrogen tartrate.

1 c.c. ,, = 0·07446 ,, ,,

Therefore 1 gram hydrogen tartrate = 13·4 c.c. $\frac{N}{1}$ NaOH (1000 ÷ 74·46 = 13·4).

Potassii Tartras Acida.—Indicator, phenol-phthalein. One gram of the *dry* salt should require for neutralisation at least 5·2 c.c. $\frac{N}{1}$ NaOH, corresponding to 97·1 per cent. hydrogen-potassium tartrate.

$$\underbrace{KHC_4H_4O_6}_{186{\cdot}75} + \underbrace{NaOH}_{39{\cdot}76} = KNaC_4H_4O_6 + H_2O.$$

That is—

1000 c.c. $\frac{N}{1}$ NaOH = 186·75 grams hydrogen-potassium tartrate.

1 c.c. ,, = 0·18675 ,, ,,

Therefore 1 gram hydrogen-potassium tartrate = 5·35 c.c. $\frac{N}{1}$ NaOH (1000 ÷ 186·75 = 5·35 c.c.).

Quininæ Hydrochloridum Acidum.—One gram when dissolved in 20 c.c. of water should require for neutralisation not more than 2·5 c.c. $\frac{N}{1}$ NaOH. (Indicator, cochineal or brazil-wood solution.)

$$\underbrace{C_{20}H_{24}N_2O_2.2HCl.3H_2O}_{447{\cdot}86} + \underbrace{NaOH}_{39{\cdot}76} = C_{20}H_{24}N_2O_2.HCl + NaCl + 4H_2O.$$

That is—

1000 c.c. $\frac{N}{1}$ NaOH = 447·86 grams acid quinine hydrochloride.

1 c.c. ,, = 0·44786 ,, ,,

Therefore 1 gram pure acid quinine hydrochloride = 2·2 c.c. $\frac{N}{1}$ NaOH (1000 ÷ 447·86 = 2·23).

Miscellaneous Estimations

Potassii Bichromas.—The Pharmacopœia requires this compound to be standardised by means of ferrous sulphate; as, however, the latter salt is very variable, it is better to employ the double iron and ammonium sulphate in its place.

Prepare decinormal standard solution of ferrous ammonium sulphate by dissolving 38·94 grams of the pure green crystallised salt in sufficient distilled water to produce 1000 c.c. Place ·25 gram of potassium dichromate in a porcelain dish with 100 c.c. of water and 2 c.c. of sulphuric acid; dissolve; take a clean white porcelain slab, and with a glass rod cover it with spots of recently prepared solution of potassium ferricyanide. Run in the iron solution from a burette until a trace taken from the dish on the stirring rod just ceases to give a blue coloration with one of the ferricyanide spots. This should require 51·3 c.c., corresponding to 100 per cent. potassium dichromate.

$$\underbrace{6\mathrm{FeSO_4(NH_4)_2SO_4.6H_2O}}_{\begin{array}{r}6)2336{\cdot}64\\10)389{\cdot}44\\38{\cdot}94\end{array}} + \underbrace{\mathrm{K_2Cr_2O_7}}_{\begin{array}{r}6)292{\cdot}3\\10)48{\cdot}7\\4{\cdot}87\end{array}} + 7\mathrm{H_2SO_4} =$$

$$3\mathrm{Fe_2(SO_4)_3} + 6\mathrm{(NH_4)_2SO_4} + \mathrm{K_2SO_4} + \mathrm{Cr_2(SO_4)_3} + 43\mathrm{H_2O}.$$

That is—

1000 c.c. $\frac{N}{10}$ “iron solution” = 4·87 grams potassium dichromate.
1 c.c. ” = ·00487 ” ”

Therefore ·25 gram potassium dichromate = 51·3 c.c. $\frac{N}{10}$ ferrous ammonium sulphate (1000 × ·25 ÷ 4·87 = 51·3).

Calx Sulphurata.—If ·8 gram be mixed with a cold solution of 1·4 grams of copper sulphate in 50 c.c. water, and after the addition of a little hydrochloric acid the mixture be well stirred and heated to about 200° F. and then filtered, the filtrate should give no red coloration with solution of potassium ferrocyanide, indicating complete precipitation of the copper as sulphide. This corresponds to about 50 per cent. of calcium sulphide. The reaction may be assumed to take place in two stages:

1. $\mathrm{CaS} + 2\mathrm{HCl} = \mathrm{CaCl_2} + \mathrm{H_2S}$.
2. $\mathrm{CuSO_4.5H_2O} + \mathrm{H_2S} = \mathrm{CuS} + \mathrm{H_2SO_4} + 5\mathrm{H_2O}$.

Or in one equation—

$$\underbrace{\mathrm{CaS}}_{71\cdot53} + \underbrace{\mathrm{CuSO_4.5H_2O}}_{247\cdot86} + \mathrm{2HCl} = \mathrm{CuS} + \mathrm{CaCl_2} + \mathrm{H_2SO_4} + \mathrm{5H_2O}.$$

That is—

71·53 grams of calcium sulphide = 247·86 grams copper sulphate
·8 ,, ,, = 2·772 ,, ,,
(247·86 × ·8 ÷ 71·53 = 2·772).

Therefore 1·4 grams of copper sulphate = ·4 gram real calcium sulphide (·8 × 1·4 ÷ 2·772 = ·4).

As ·4 gram of real calcium sulphide is contained in ·8 gram of Calx Sulphurata, it corresponds to 50 per cent.

Chloral Hydras.—If 4 grams be boiled with 30 c.c. normal sodium hydroxide solution, not more than 6 c.c. normal sulphuric acid should be required to neutralise the soda remaining free on the completion of the reaction, corresponding to at least 98·1 per cent. of chloral hydrate.

$$\underbrace{\mathrm{CCl_3.CHO.H_2O}}_{164\cdot15} + \underbrace{\mathrm{NaOH}}_{39\cdot76} = \mathrm{CHCl_3} + \mathrm{H.COONa} + \mathrm{H_2O}.$$

That is, 1000 c.c. $\frac{N}{1}$ NaOH = 164·15 grams chloral hydrate.
1 c.c. ,, = ·16415 ,, ,,

Therefore 4 grams of chloral hydrate require 24·36 c.c. $\frac{N}{1}$ NaOH (1000 × 4 ÷ 164·15 = 24·36).

If 24·36 c.c. $\frac{N}{1}$ NaOH = 100 per cent., 24 c.c. = 98·1 per cent.

As will be seen by the above equation, chloral hydrate yields chloroform and sodium formate when boiled with sodium hydroxide solution.

Calcii Hypophosphis.—·25 gram boiled for ten minutes with a solution of ·6 gram of potassium permanganate should yield a colourless solution on filtration, corresponding to about 100 per cent. purity.

$$\underbrace{\mathrm{3Ca(H_2PO_2)_2}}_{506\cdot49} + \underbrace{\mathrm{4K_2Mn_2O_8}}_{1254\cdot96} =$$
$$\mathrm{Ca_3(PO_4)_2} + \mathrm{4K_2HPO_4} + \mathrm{8MnO_2} + \mathrm{4H_2O}.$$

That is, 506·49 grams of calcium hypophosphite require 1254·96 grams of potassium permanganate for complete oxidation to orthophosphate.

Therefore ·25 gram would require ·6 gram of permanganate (1255·36 × ·25 ÷ 506·49 = ·61).

Sodii Hypophosphis.—·5 gram boiled for ten minutes with 25 c.c. of water and 1·15 grams of potassium permanganate should yield a nearly colourless solution on filtration.

$$\underbrace{3NaH_2PO_2}_{262\cdot32} + \underbrace{2K_2Mn_2O_8}_{627\cdot48} = 2K_2HPO_4 + Na_3PO_4 + 4MnO_2 + 2H_2O.$$

That is, 262·32 grams of sodium hypophosphite require 627·68 grams of potassium permanganate for complete oxidation to orthophosphate.

Therefore ·5 gram, if pure, would require 1·19 grams of permanganate (627·48 × ·5 ÷ 262·32 = 1·19).

Potassii Permanganas.—One gram dissolved in 100 c.c. distilled water, and acidulated with 5 c.c. diluted sulphuric acid, should require for complete decoloration 31·8 c.c. normal solution of oxalic acid. The standard solution may be prepared by dissolving 62·55 grams of pure recrystallised oxalic acid in sufficient distilled water to produce 1000 c.c.

$$\underbrace{K_2Mn_2O_8}_{\begin{array}{r}5)\underline{313\cdot74}\\2)\underline{62\cdot74}\\31\cdot37\end{array}} + \underbrace{5H_2C_2O_4.2H_2O}_{\begin{array}{r}5)\underline{625\cdot5}\\2)\underline{125\cdot1}\\62\cdot55\end{array}} + 3H_2SO_4 = K_2SO_4 + 2MnSO_4 + 18H_2O + 10CO_2.$$

That is—

1000 c.c. $\frac{N}{1}$ $H_2C_2O_4.2H_2O$ = 31·37 grams potassium permanganate.

1 c.c. ,, = ·03137 ,, ,,

Therefore 1 gram potassium permanganate = 31·8 c.c. $\frac{N}{1}$ $H_2C_2O_4.2H_2O$ (1000 ÷ 31·37 = 31·8).

Volumetric analysis involving the use of a nitrometer

In the last edition of the Pharmacopœia four substances are determined quantitatively by the volume of gas liberated during a specified reaction; these are Amyl Nitris, Liquor Ethyl Nitritis, Sodii Nitris, and Spiritus Ætheris Nitrosi. When any one of these substances is treated with potassium iodide and sulphuric acid, nitric oxide is given off, according to the general equation.

$$R'NO_2 + KI + H_2SO_4 = NO + I + KHSO_4 + R'OH.$$

The decomposition is made to take place in a specially constructed instrument called a nitrometer (Fig. 247). This consists essentially of a cylindrical glass measuring tube, to which is attached a small funnel by a "three-way" stopcock. According to the direction in which this stopcock is turned it can either make connection with the funnel, or the waste, or completely shut off all connection with the outer air. Connected to the measuring tube by a flexible pipe is a second tube, which serves as a reservoir for the displaced water and for equalising the pressure on the liberated gas.

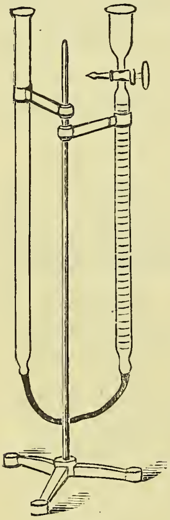
Fig. 247.

To prepare the nitrometer for use, the graduated tube is filled with strong brine, the second tube being raised at the same time, so that while the level of the liquid in the two tubes is identical, it shall not be more than a third full. The requisite quantity of the sample to be tested is placed in the cup of the nitrometer, and the leveling tube being lowered the sample is allowed to enter, care being taken that no air gets in at the same time. The potassium iodide solution is next added, and this in its turn is followed by the sulphuric acid. Decomposition at once takes place, and if the tube is gently agitated the reaction is complete in about ten minutes. The level of the liquid in the plain tube is next made to correspond with that in the measuring tube, and the volume of nitric oxide read off and corrected to normal temperature and pressure.

Amyl Nitris.—Amyl nitrite is too strong for determination in its pure state, so it is diluted with 90 per cent. alcohol to form a 5 per cent. solution. Introduce 5 c.c. of this solution (= ·25 c.c.

amyl nitrite) into the nitrometer charged with strong brine as directed in the previous paragraph, followed by 5 c.c. potassium iodide test solution, and 5 c.c. dilute sulphuric acid. When reaction is complete it should yield at the ordinary temperature, 60° F. (15·5° C.), and pressure, 30 inches (760 mm.) of mercury, not less than 30 c.c. of nitric oxide, corresponding to about 56 per cent. of amyl nitrite.

$$\underbrace{C_5H_{11}NO_2}_{116\cdot25} + KI + H_2SO_4 = \underbrace{NO}_{2)\overline{29\cdot82}} + KHSO_4 + C_5H_{11}OH + I.$$

$$14\cdot91 = \text{density.}$$

The percentage by weight of real amyl nitrite present may be calculated as follows:

$$30 \text{ c.c. NO at N.T.P. occupy } \frac{30 \times 273}{15\cdot5 + 273} = 28\cdot38 \text{ c.c.}^*$$

The density of a compound gas is *half* its molecular weight.

1 c.c. H at N.T.P. weighs ·0000896 gram.

1 c.c. NO „ „ (·0000896 × 14·91) = ·001336 gram.

Therefore—

28·38 c.c. NO at N.T.P. weigh (·001336 × 28·38) = ·038 gram.

Now, according to the equation above, 29·82 grams of NO represent 116·25 grams of amyl nitrite; ·038 gram of NO therefore represents (116·25 × ·038 ÷ 29·82) = ·14 gram.

Then if ·25 c.c. of amyl nitrite, B.P. (= 5 c.c. of the diluted solution), contains ·14 gram real $C_5H_{11}NO_2$, 100 c.c. should contain 56 grams (·14 × 100 ÷ ·25 = 56).

Sodii Nitris.—0·1 gram dissolved in 5 c.c. of water should, when treated in a nitrometer as described under "Amyl Nitris," yield at the ordinary temperature and pressure (15·5° C. and 760 mm.) not less than 32·5 c.c. nitric oxide, corresponding to not less than 95 per cent. by weight of sodium nitrite.

$$\underbrace{NaNO_2}_{68\cdot58} \quad \text{yields} \quad \underbrace{NO}_{2)\overline{29\cdot82}}$$

$$14\cdot91 = \text{density.}$$

$$32\cdot5 \text{ c.c. NO at N.T.P. occupy } \frac{32\cdot5 \times 273}{15\cdot5 + 273} = 30\cdot75 \text{ c.c.}$$

1 c.c. NO at N.T.P. weighs ·001336 gram.†

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* N.T.P.—Normal temperature and pressure = 760 mm. and 0° C.

† For calculations see Amyl Nitris.

Therefore 30·75 c.c. NO at N.T.P. weigh ·041 gram (·001336 × 30·75).

According to the equation above, 29·82 grams NO represent 68·58 grams of sodium nitrite; ·041 gram of NO therefore represents ·095 gram (68·58 × ·041 ÷ 29·82).

Then if ·1 gram of sodium nitrite, B.P., contains ·095 gram real $NaNO_2$, 1 gram should contain ·95, corresponding to 95 per cent.

Spirit. Ætheris Nitrosi.—Sp. gr. ·838—·842.

Five c.c., when treated in a nitrometer as described under "Amyl Nitris," should yield at the ordinary temperature and pressure (15·5° C. and 760 mm.), *when freshly prepared*, at least 31·25 c.c., but not more than 35 c.c. of nitric oxide gas. Even after it has been kept for some time, and the vessel containing it has been occasionally opened, it should yield not much less than 25 c.c. of nitric oxide, but never less than would correspond to a minimum of 1·75 per cent. by weight of ethyl nitrite.

$$\underbrace{C_2H_5NO_2}_{74{\cdot}52} + KI + H_2SO_4 = \underbrace{NO}_{2)\,\underline{29{\cdot}82}} + KHSO_4 + C_2H_5.OH + I.$$

$$14{\cdot}91 = \text{density.}$$

Supposing 5 c.c. of spirit of nitrous ether yielded 30 c.c. of nitric oxide gas at the ordinary temperature and pressure (15·5° C. and 760 mm.), the percentage of ethyl nitrite may be calculated as follows:

$$30 \text{ c.c. NO at N.T.P. occupy } \frac{30 \times 273}{15{\cdot}5 + 273} = 28{\cdot}38 \text{ c.c.}$$

The density of a compound gas is *half* its molecular weight.

1 c.c. H weighs ·0000896 gram at N.T.P.

1 c.c. NO ,, ·0000896 × 14·91 = ·001336 gram.

Therefore 28·38 c.c. NO at N.T.P. weigh ·038 gram (·001336 × 28·38).

Now, according to the equation above, 29·82 grams NO represent 74·52 grams of ethyl nitrite; ·038 gram NO, therefore, represents ·0949 gram (74·52 × ·038 ÷ 29·82).

Then if 5 c.c. spirit of nitrous ether contain ·0949 gram of ethyl nitrite, 100 c.c. should contain ·0949 × 100 ÷ 5 = 1·898 grams.

Supposing spirit of nitrous ether had the same specific gravity as water, viz. 1·000, the sample under consideration would contain 1·898 per cent. by weight of ethyl nitrite; but for all practical purposes the specific gravity may be taken as ·838, therefore 100 grams would contain 2·26 grams (1·898 × 1·000 ÷ ·838).

Liquor Ethyl Nitritis.—Sp. gr. ·823—·826. Five c.c., when treated in a nitrometer as described under "Amyl Nitris," should, *when freshly prepared,* yield at the ordinary temperature and pressure (15·5° C. and 760 mm.) at least 38 c.c. nitric oxide gas, and even after the solution has been kept some time, and the bottle containing it occasionally opened, it should not yield less than 31·6 c.c., corresponding to 2·41 per cent. by weight of ethyl nitrite.

For equation and calculations see Spirit. Ætheris Nitrosi.

Liquor Hydrogenii Peroxidi.—One c.c. introduced into a nitrometer charged with nearly saturated solution of sodium sulphate followed by 10 c.c. of a mixture consisting of—

Strong sulphuric acid	1 c.c.,
Solution of potassium permanganate (5 per cent.) ...	2 c.c.,
Distilled water	7 c.c.,

should afford at normal temperature and pressure, not less than 18 c.c., and not more than 22 c.c. of oxygen, indicating a yield of 9 to 11 volumes from the solution of hydrogen peroxide. The reaction that takes place is expressed by the equation—

$$2KMnO_4 + 5H_2O_2 + 3H_2SO_4 = 8H_2O + K_2SO_4 + 2MnSO_4 + 5O_2,$$

in which it will be seen that half the oxygen only, comes from the peroxide.

Volumetric Tables and Co-efficients

Weight taken.	Substance.	Number of c.c. volumetric solution required.	Corresponding to percentages of pure substance.	Co-efficient.
0·25 gram	Acid, arsenious	50·9 c.c. $\frac{N}{10}$ I	100·00	·00491
1·00 „	„ sulphurous	15·7 „ „ „	5·00	·003179
1·00 „	Sodium sulphite	77·7 „ „ „	97·30	·012519
0·50 „	Antimony trioxide	70·0 „ „ „	100·00	·00741
1·00 „	„ tartarated	60·2 „ „ „	99·60	·016748
1·00 „	Iron arsenate	6·7 „ $\frac{N}{10}$ $K_2Cr_2O_7$	9·89	·0147613
1·00 „	Saccharated iron carbonate	29·0 „ „ „	33·00	·011515
1·00 „	Iron phosphate	28·3 „ „ „	47·00	·016616
1·00 „	Crystallised ferrous sulphate	36·0 „ „ „	99·40	·02761
1·00 „	Dried ferrous sulphate	54·6 „ „ „	92·20	·016882
0·25 „	Reduced iron	33·7 „ „ „	75·00	·00556
1·00 „	Diluted hydrocyanic acid	3·7 „ $\frac{N}{10}$ $AgNO_3$	2·00	·00537
0·50 „	Ammonium bromide	51·1 „ „ „	99·40	·009729
1·00 „	Potassium bromide	83·7 „ „ „	98·90	·011818
1·00 „	„ iodide	59·5 „ „ „	98·90	·016473
1·00 „	Sodium bromide	95·8 „ „ „	96·90	·010223
1·00 „	„ iodide	66·5 „ „ „	98·90	·014878
2·50 c.c.	Syrup of ferrous iodide	16·2 „ „ „	10·00	·01537
0·50 gram	Chlorinated lime	46·8 „ $\frac{N}{10}$ $Na_2S_2O_3,5H_2O$	33·00	—
1·00 „	Solution of chlorinated lime	5·6 „ „ „	2·00	—
3·50 „	„ „ soda	25·0 „ „ „	2·50	—
1·00 „	Iodine	78·4 „ „ „	99·00	·01259
1·00 „	Ammonium carbonate, B.P.	18·7 „ $\frac{N}{1}$ H_2SO_4	97·30	·05201
1·00 „	Stronger solution of ammonia	19·1 „ „ „	32·50	·01694
1·00 „	Solution of ammonia	5·9 „ „ „	10·00	·01694
1·00 „	Crystallised borax	5·2 „ „ „	98·60	·18956
10·00 „	Saccharated solution of lime	6·3 „ „ „	1·75	·02779

24·00 c.c.	Lime water	10·0 ,,	$\frac{N}{10}$	,,	0·11	·002779
1·00 gram	Stronger solution of lead oxyacetate	17·0 ,,	,,	,,	23·00	·01359
1·00 ,,	Lead acetate	53·1 ,,	,,	,,	100·00	·018807
9·00 c.c.	Solution of potash	10·0 ,,	$\frac{N}{1}$	,,	6·19	·05571
1·00 gram	Potassium hydroxide	16·1 ,,	,,	,,	89·90	·05571
1·00 ,,	,, bicarbonate	10·0 ,,	,,	,,	99·30	·09938
1·00 ,,	,, carbonate	11·9 ,,	,,	,,	82·06	·06860
1·00 ,,	,, citrate	9·7 ,,	,,	,,	99·20	·10123
1·00 ,,	,, tartrate	8·4 ,,	,,	,,	98·00	·11676
1·00 ,,	Tartarated soda	7·0 ,,	,,	,,	98·10	·14007
1·00 ,,	Sodium	42·6 ,,	,,	,,	97·40	·02288
1·00 ,,	,, benzoate	6·9 ,,	,,	,,	100·00	·14301
1·00 ,,	,, bicarbonate	11·9 ,,	,,	,,	99·30	·08343
1·00 ,,	,, carbonate	6·9 ,,	,,	,,	98·10	·14205
1·00 ,,	Acid, acetic	5·5 ,,	$\frac{N}{1}$	NaOH	33·00	·05958
1·00 ,,	,, ,, (dilute)	7·2 ,,	$\frac{N}{10}$	,,	4·27	·005958
1·00 ,,	,, ,, glacial	16·6 ,,	$\frac{N}{1}$	,,	98·80	·05958
1·00 ,,	,, citric	14·3 ,,	,,	,,	100·00	·0695
4·00 ,,	,, hydrobromic (dilute)	5·0 ,,	,,	,,	10·00	·08035
1·00 ,,	,, hydrochloric	8·7 ,,	,,	,,	31·79	·03619
1·00 ,,	,, ,, (dilute)	2·9 ,,	,,	,,	10·58	·03619
1·00 ,,	,, lactic	8·3 ,,	,,	,,	75·00	·08937
1·00 ,,	,, nitric	11·1 ,,	,,	,,	70·00	·06258
1·00 ,,	,, ,, (dilute)	2·7 ,,	,,	,,	17·44	·06258
4·00 ,,	,, nitro-hydrochloric (dilute)	10·0 ,,	,,	,,	—	—
1·00 ,,	,, sulphuric	20·1 ,,	,,	,,	98·00	·04867
1·00 ,,	,, ,, (dilute)	2·8 ,,	,,	,,	13·65	·04867
1·00 ,,	,, tartaric	13·3 ,,	,,	,,	100·00	·07446
1·00 ,,	Acid tartrate of potassium	5·2 ,,	,,	,,	97·10	·18675
4·00 ,,	Chloral hydrate	24·0 ,,	,,	,,	98·10	·16145
1·00 ,,	Potassium permanganate	31·8 ,,	$\frac{N}{1}$	$H_2C_2O_4.2H_2O$	100·00	·03137
·25 ,,	Potassium dichromate	51·3 ,,	$\frac{N}{10}$	$(NH_4)_2SO_4.FeSO_4.6H_2O$	100·00	·00487

Gasometric co-efficients

1 c.c. of NO at N.T.P. = ·00333 gram real Ethyl nitrite.
1 „ „ „ = ·0052 „ „ Amyl „
1 „ „ „ = ·00307 „ „ Sodium „

Alkaloidal co-efficients for 1 c.c. $\frac{N}{10}$ *Sulphuric Acid*

Strychnine	($C_{21}H_{22}N_2O_2$)	...	·0331
Morphine	($C_{17}H_{19}NO_3$)	...	·0283
Atropine	($C_{17}H_{23}NO_3$)	...	·0287
Caffeine	($C_8H_{10}N_4O_2$)	...	·0192
Cocaine	($C_{17}H_{21}NO_4$)	...	·0300
Codeine	($C_{18}H_{21}NO_3$)	...	·0296
Physostigmine	($C_{15}H_{21}N_3O_2$)	...	·0546
Pilocarpine	($C_{11}H_{16}N_2O_2$)	...	·0199
Quinine	($C_{20}H_{24}N_2O_2$)	...	·0321
Emetine	( )	...	·0248
Cephaeline	( )	...	·0234

Determination of the haloids

Although the Pharmacopœia relies upon nitrate of silver for determining the amount of iodine and bromine in the respective salts of these elements, it is well known that only approximate results are obtained. It is, in fact, quite possible to manufacture potassium iodide, that by the nitrate of silver method shows 99 per cent. of iodide, without *actually* containing more than 93 or 94 per cent. For the exact determination of the pharmacopœial salts of the halogens, therefore, the reader cannot do better than follow the method devised by Swinton, and published in the ‘Pharmaceutical Journal’ for December 25th, 1897. As the process is of great interest to pharmacists the paper is given at length.

1. *Determination of iodide in any mixture of haloids*

Having an approximate idea of the percentage of iodide present, a quantity of the substance corresponding to about 0·2 gram of iodine should be taken (in the absence of such knowledge a rough estimation should be made by the process subsequently detailed) and dissolved in 10 c.c. of water in a separator, about

5 c.c. of sulphuric acid slowly added, and the separator kept cool meanwhile by allowing a stream of cold water to flow over it. Then thoroughly agitate, first with 5 c.c. liquefied phenol (90 per cent.), next with 20 c.c. saturated bromine water. After agitating with the bromine water there should be no trace of bromine left uncombined, the excess combining with the phenol to form tribromophenol :

$$C_6H_5.OH + 3Br_2 = C_6H_2Br_3.OH + 3HBr.$$

At the same time most of the iodine is replaced by the bromine. The iodine is washed out with successive portions of 10 c.c. of chloroform till the chloroform is colourless, two washings usually sufficing. The solution is again agitated with liquefied phenol and bromine water as before, when it will probably be found that more iodine has separated. This is again washed out, the process being continued as long as any trace of iodine is set free, two phenol and bromine treatments usually being sufficient.

The mixed chloroformic solutions of iodine are now covered with a layer of water and titrated with $\frac{N}{10}$ sodium thiosulphate in the usual way, using starch paste as an indicator.

1 c.c. $\frac{N}{10}$ $Na_2S_2O_3,5H_2O$ = ·01259 gram iodine.

1 c.c. „ = ·016473 gram potassium iodide.

It may be thought that a better method for effecting the bromine absorption would be setting free the iodine with excess of bromine, no phenol being present, washing out the iodine and excess of bromine together, then adding phenol to the chloroformic solution to absorb the bromine ; but this is impracticable owing to the formation of iodine bromide.

The process is found to answer well for the assay of syrup of iodide of iron, the syrup being previously diluted with three volumes of water.

The following table shows some of the results obtained by the pharmacopœial tests in comparison with this process.

	Percentage by B.P. test.	Percentage by this process.
1. Potassium iodide (specially purified)	99·5	99·1
2. „	102·2	89·8
3. „	98·6	95·7
4. Sodium iodide (anhydrous)	98·8	94·5
5. „ ($2H_2O$)	83·2	79·8
6. Ammonium iodide	101·5	97·8

2. *Determination of bromides in the presence of both iodides and chlorides*

For the determination of bromides, if the reaction for iodine be obtained, the salt is examined by the process given subsequently for the determination of halogens in mixtures of all three.

From ·2 to ·3 gram of the salt is dissolved in a separator in 10 c.c. water, acidified with 2 c.c. of a cooled mixture of equal volumes of sulphuric acid and water. About 20 c.c. chloroform is then introduced, then excess of saturated aqueous solution of potassium permanganate, and the separator violently agitated.

The chloroform, which dissolves the liberated bromine, is now run into a 20-ounce stoppered bottle containing 20 c.c. alcoholic solution of potassium iodide, and the treatment with chloroform repeated until the washings are colourless. The bromine replaces an equivalent amount of iodine from the potassium iodide, and this solution is now largely diluted with water and titrated with $\frac{N}{10}$ $Na_2S_2O_3.5H_2O$.

1 c.c. $\frac{N}{10}$ $Na_2S_2O_3.5H_2O$ = ·007935 gram bromine.

1 c.c. „ = ·011818 „ potassium bromide.

The precautions absolutely necessary in this process are—

First, to add the chloroform before the permanganate solution, so that the bulk of the bromine set free may be immediately absorbed by the chloroform, in order to minimise the risk of loss of bromine vapour.

Second, to make sure that excess of potassium permanganate solution is present, this being easily ascertained by the persistence of the violet colour.

Third, to largely dilute with water before the titration with thiosulphate, in order to obtain the blue colour of the iodide of starch, which does not develop in the alcoholic solution.

The following table gives the comparative results obtained in the examination of various bromides by this process as compared with the B.P.:

	Percentage by B.P. test.	Percentage by this test.
1. Ammonium bromide ...	100·6 ...	99·2
2. Potassium „ ...	101·5 ...	95·5
3. „ „ ...	101·1 ...	98·6
4. Lithium „ ...	91·5 ...	89·4
5. Strontium „ ...	72·1 ...	70·3

3. *Determination of iodides, bromides, and chlorides in the presence of each other*

The following process is employed, quite irrespective of the relative proportions of the halogens present.

1. *Determination of the iodine.*—Ten grams of the mixture of salts is dissolved in 250 c.c. water, and the iodine in 10 c.c., determined as previously described.

2. *Determination of the iodine and bromine together.*—Another 10 c.c. is acidified with 2 c.c. of sulphuric acid (1 in 2); the iodine first removed by adding the theoretical volume of $\frac{N}{2}$ potassium permanganate solution, (the quantity being arrived at by dividing the number of c.c. of $\frac{N}{10}$ thiosulphate used in titrating the iodine from 10 c.c. of the solution, by 5), and thoroughly washing out with chloroform. This chloroform is run into a stoppered bottle containing 20 c.c. alcoholic solution of potassium iodide.

The bromine in the residue is next set free with excess of saturated solution of potassium permanganate, the same precautions being observed as in the determination of a simple bromide. The bromine is now thoroughly washed out with chloroform, and also run into the alcoholic solution of potassium iodide, where it replaces an equivalent quantity of iodine.

After diluting with a large volume of water, the total amount of free iodine is determined by titration with $\frac{N}{10}$ thiosulphate.

To calculate the amount of bromine—

Let A = number of c.c. of $\frac{N}{10}$ solution of thiosulphate of sodium required for the iodine in 10 c.c. of the solution (paragraph 1).

Let B = number of c.c. of ditto required by the iodine and the iodine equivalent of the bromine in 10 c.c. of the solution (paragraph 2).

Then B − A = number of c.c. of thiosulphate required by the iodine equivalent of the bromine, which, multiplied by ·007935, gives the weight of bromine in 10 c.c. of the solution.

3. *Determination of the chlorine.*—Ten c.c. of the original solution is titrated with $\frac{N}{10}$ silver nitrate in the usual manner, using potassium chromate as an indicator.

To calculate the chlorine present—

Let X = number of c.c. of $\frac{N}{10}$ $AgNO_3$ solution required to precipitate the chlorides, bromides, and iodides in 10 c.c. of the solution.

Let Y = number of c.c. ditto required by the known quantity of bromides and iodides in 10 c.c. of the solution.

Then $X - Y$ = number of c.c. of $\frac{N}{10}$ $AgNO_3$ solution used by the chlorides, which multiplied by ·003519, gives the weight of chlorine in 10 c.c. of the solution.

The following results are from experiments made on mixtures, he composition of which was unknown to the operator :

		Percentage calculated.	Percentage found.
1	NaCl	98·11	98·10
	AmBr	1·07	1·09
	KI	·82	·81
2	NaCl	41·73	41·34
	KBr	30·07	30·31
	KI	28·19	28·38

Syrup of ferrous iodide

Dilute 10 c.c. of the syrup with 90 c.c. water; slowly mix 10 c.c. of this with 5 c.c. strong sulphuric acid, taking care to prevent rise of temperature. Next, agitate with 5 c.c. 90 per cent. phenol, then with 25 c.c. water, freshly saturated with bromine. Shake out the displaced iodine with water; two shakings are usually sufficient. Repeat the treatment with phenol, bromine, and chloroform, and add the chloroformic washings to the first. Cover the chloroformic layer with water, and titrate with $\frac{N}{10}$ thiosulphate solution, using starch mucilage as indicator.

$$1 \text{ c.c. } \frac{N}{10}\ Na_2S_2O_3.5H_2O = ·0155 \text{ gram } FeI_2.$$

CHAPTER LII

ASSAY OF ALKALOIDAL EXTRACTS

The processes employed in the valuation of these preparations depend upon two facts: first, that the alkaloids constituting their active principles are soluble in certain liquids in which their salts are insoluble; secondly, that the respective solvents will not mix with one another, but readily separate into distinct layers.

When an aqueous solution of an extract is made alkaline with soda or ammonia, any alkaloids present are precipitated; if, for instance chloroform, or other suitable solvent is now added, and the mixture shaken in a stoppered glass separator, the alkaloids will be dissolved out, and on allowing the layers to separate, their solution may be drawn off. If next the chloroformic solution is shaken with dilute acid, the alkaloids will be abstracted in the form of salts. On again making alkaline and extracting with chloroform, the alkaloids may be often obtained in a sufficiently pure state to be weighed, but only rarely are they sufficiently pure to crystallise.

The processes appear somewhat complicated at first sight, but are really very simple, consisting practically in—

1. Precipitating the alkaloids and shaking out with chloroform or other suitable solvent.
2. Abstracting the alkaloids from their solution by shaking with a dilute acid.
3. Re-precipitating the alkaloids from this solution of their salts by addition of an alkali, shaking out with chloroform, and evaporating.

To obtain anything like concordant results, the following points must be carefully attended to.

The separator should not be too large, and is preferably made in the shape of an elongated pear.

The shaking out must not be accomplished with a violent or jerky motion, or an emulsion will form which only separates with difficulty. The intermixing is best performed by imparting a rotary movement to the liquids, accompanied by frequent inversion of the separator. Emulsions are more liable to occur with concentrated extracts, especially when much fat is present. In this latter case, it is often found advantageous to make the extract distinctly acid so as to keep the alkaloids in solution, and extract the oily matter first, by shaking out very gently with a small quantity of chloroform, or preferably, a mixture of chloroform and ether. Afterwards the extract may be alkalised and extracted in the usual way without trouble. The formation of emulsions is also in part prevented by operating only with *warm* solutions.

If, notwithstanding all precautions, an emulsion has formed, it should be transferred to a small pressure filter connected with a water-pump, as described under Filtration, the filter being finally washed with a little fresh solvent. After passing through the filter the two liquids will be found in distinct layers.

During the shaking out, especially when operating with warm solutions, considerable pressure is often generated in the separator. This must be relieved from time to time, not by removing the stopper, as it involves loss by spirting, but by inverting the separator, and *very slowly opening the stopcock.*

After withdrawing a solution of the alkaloids or salts the outlet tube must be rinsed by pouring a little fresh solvent into the separator, and allowing it to trickle through.

Liquid extract of belladonna

This liquid extract is obtained by exhausting English belladonna root by the process of *re-percolation.* It should contain ·75 gram of the alkaloids of belladonna in 100 c.c. The proportion of alkaloids (of which hyoscyamine constitutes nearly 90 per cent.) is determined by shaking out with chloroform after precipitation by ammonia, extracting the chloroformic solution by dilute acid, again precipitating with ammonia, and redissolving the purified alkaloids in chloroform, which is evaporated on a tared dish.

Details of process of manufacture.—Take at least 48 ounces of English belladonna root in No. 20 powder and divide into four equal portions, and exhaust with 78 per cent. alcohol by the process of *re-percolation* described in the chapter on Extraction (p. 80), until the percolate from the last portion measures 12 fluid ounces. The liquid remaining in the marc may be recovered by pressure, and used to exhaust the next batch, instead of starting with fresh menstruum.

Details of assay process.—1. Carefully measure 10 c.c. of the strong liquid extract obtained by the above process, into a small porcelain capsule, and allow the spirit to evaporate at a temperature not exceeding 100° F. Add 10 c.c. of distilled water, and transfer to a stoppered glass separator. Wash out the dish with 40 c.c. of distilled water made slightly acid with dilute sulphuric acid. Now add 10 c.c. of chloroform and rotate gently. Allow to separate *completely,* draw off and reject the chloroformic layer, which will contain the fat. Then add—

Chloroform	10 c.c.
Solution of ammonia	a decided excess.

Agitate and set aside to separate. Draw off the chloroform, which will contain the greater part of the alkaloids.

2. Repeat the agitation with another 10 c.c. of chloroform, separate and draw off as before.

3. Repeat No. 2, so as to extract last trace of alkaloid.

4. Introduce the mixed chloroformic solutions into a separator, and shake with—

Dilute sulphuric acid	5 c.c.
Warm distilled water	10 „

Allow the chloroform to separate. Draw off and reserve. Now draw off the acid solution of the alkaloids and set on one side.

5. Re-introduce the reserved chloroformic solution and agitate with a further quantity of diluted acid, separate and draw off as before.

6. Mix the acid liquids, add excess of ammonia to precipitate the alkaloids, and 10 c.c. chloroform. Shake, separate, and draw off the chloroformic solution. Twice repeat the agitation with chloroform, so as to extract the last traces of alkaloids.

7. Mix the three chloroformic solutions with 5 c.c. distilled water containing one drop of solution of ammonia. Shake and separate.

8. Draw off the purified chloroformic solution and evaporate in a counterpoised dish at the temperature of a water-bath; finally dry the residue at a temperature not exceeding 200° F., and weigh.

Check.—Dissolve the alkaloidal residue obtained as above in exactly 10 c.c. decinormal hydrochloric acid $\left(\frac{N}{10}\text{ HCl} = 3{\cdot}619\right.$ grams in 1 litre) and add centinormal solution of soda $\left(\frac{N}{10}\right.$

Na OH = 0·3976 gram in 1 litre), until the liquid is neutral, using a few drops of hæmatoxylin or Brazil-wood as an indicator.

Deduct the measure of soda solution thus required from 100, and multiply the remainder by ·00287; the product will be the weight in grams of alkaloids present in the quantity of liquid operated upon. From this weight calculate the amount of alkaloids in the bulk of strong percolate, and add to the latter sufficient of the alcoholic menstruum to produce liquid extract of belladonna, containing 0·75 gram of alkaloids in each 100 c.c.

$$\underset{\substack{10)\underline{287{\cdot}05}\\ 28{\cdot}7}}{\underbrace{C_{17}H_{23}NO_3}} \quad + \quad \underset{\substack{10)\underline{36{\cdot}19}\\ 3{\cdot}619}}{\underbrace{HCl}} \quad = \quad C_{17}H_{23}NO_3.HCl.$$

That is, 1000 c.c. $\frac{N}{10}$ HCl = 28·7 grams atropine (or hyoscyamine).
1 c.c. „ „ = ·0287 „ „
Therefore 1 c.c. $\frac{N}{100}$ „ = ·00287 „ (co-efficient).

Example:

Supposing the alkaloids from 10 c.c. of strong percolate, after acidifying with 10 c.c. $\frac{N}{10}$ HCl, required 70 c.c. $\frac{N}{100}$ NaOH for neutralisation, it follows that 30 c.c. represents the amount of alkaloid in combination with the acid; and as the co-efficient for atropine is ·00287, then ·00287 × 30 = ·0861 gram of alkaloid in 10 c.c., or ·861 gram in 100 c.c.

Every 87·1 c.c., therefore, must be diluted with 78 per cent. alcohol to 100 c.c. $\left(\frac{100 \times {\cdot}75}{{\cdot}861} = 87{\cdot}1\right)$.

Red cinchona bark

When used for purposes other than obtaining the alkaloids or their salts, cinchona bark should yield between 5 and 6 per cent. of total alkaloids, of which not less than half must consist of quinine and cinchonidine.

Details of assay process.—

1.	Red cinchona bark in 60 powder	20 grams.
	Calcium hydroxide (fresh)	6 „
	Distilled water	20 c.c.

Triturate very thoroughly in a porcelain mortar and allow to stand for two hours, when it should present the appearance of a moist dark brown powder in which no lumps are visible.

2. Transfer the powder to a suitable flask fitted with a reflux condenser, and add 130 c.c. benzolated amylic alcohol. Boil for half an hour, allow to settle, and decant the clear liquid on to a filter, leaving the partially exhausted powder in the flask.

Note.—A convenient condenser may be made by fitting the flask with a good cork, through which a glass tube of fairly wide bore and about six feet in length is passed. The large surface exposed to the air is quite sufficient to condense the greater part of the vapour. Another plan consists in placing a good-sized funnel in the flask, and then standing another flask containing cold water in the mouth of the funnel.

3. Add more benzolated amylic alcohol to the powder, boil as before, decant and filter.

4. Repeat this operation a third time, then turn the contents of the flask on to a filter, washing out the flask with a little more benzolated amylic alcohol. The washings added to the previous filtrates should amount to about 200 c.c.

5. Introduce the mixed filtrates, which now contain the whole of the alkaloid, while still warm, into a stoppered glass separator, and add—

Dilute hydrochloric acid	2 c.c.
Distilled water (warm)	12 ,,

Shake well together, allow the acid liquid to separate, and draw off.

6. Repeat the last operation three times, finally mixing the acid liquids, which will contain the alkaloids as hydrochlorides. While still warm *exactly* neutralise with solution of ammonia, and concentrate to 16 c.c. Cool and add 1·5 grams of tartarated soda ($KNaC_4H_4O_6$) dissolved in 3 c.c. distilled water. Stir well with a glass rod and set aside. At the end of three hours the precipitate, which consists of the mixed tartrates of quinine and cinchonidine, is collected on a filter, washed with distilled water, and dried at 212° F. (The mother-liquor and washings are preserved for further treatment.) These tartrates contain practically $\frac{8}{10}$ of their weight of alkaloid, so that if the total weight is multiplied by 8, and divided by 10, it gives the weight of quinine and cinchonidine in 20 grams of bark, and this weight multiplied by 5, the amount in 100 grams.

7. To the mother-liquor from above add solution of ammonia in slight excess. Collect, wash, and dry the precipitate, which consists of quinidine and cinchonine with other alkaloids of cinchona.

The weight of these multiplied by 5, and added to the percentage of the quinine and cinchonidine, gives the percentage weight of total alkaloids contained in the bark.

Liquid extract of cinchona

A liquid extract obtained by exhausting powdered cinchona bark with *very dilute* solution of hydrochloric acid and glycerin, by the process of maceration and percolation, the percolate being subsequently evaporated to a low bulk and standardised. It should contain 5 grams of total alkaloids in every 100 c.c. of liquid extract.

The proportion of alkaloids present is determined by alkalising the liquid and extracting with benzolated amylic alcohol, which in its turn is deprived of the alkaloids by shaking with acid. From the latter the alkaloids are again precipitated, and dissolved out by chloroform, which on evaporation leaves them sufficiently pure for weighing.

Details of the process of manufacture.—

1.	Red cinchona bark in 60 powder ...	20 ounces.
	Hydrochloric acid	5 fluid drachms.
	Glycerin	2½ fluid ounces.
	Distilled water	100 ,,

Mix, and macerate together for forty-eight hours, stirring frequently. Transfer to a percolator, and when the fluid ceases to pass, and the contents of the percolator have become properly packed, continue the percolation with distilled water until the percolate no longer gives a precipitate of alkaloids on the addition of solution of potash. Evaporate the percolate in a porcelain or glazed earthenware vessel at a temperature not exceeding 180° F. (82·2° C.) until it is reduced to 20 fluid ounces; determine the proportion of alkaloids in the liquid by the following process of assay.

Details of assay process.—1. Introduce into a stoppered glass separator—

Liquid percolate from above	5 c.c.
Distilled water	25 ,,
Benzolated amylic alcohol	25 ,,
Solution of potash	15 ,,

Shake together during twenty minutes; allow to rest until the fluids separate. Draw off the alkaline layer into a beaker and preserve; draw off the alcoholic layer into a flask and cork.

2. Re-introduce the alkaline solution into the separator and agitate with a further 25 c.c. of benzolated amylic alcohol; separate and draw off as before.

3. Place the mixed alcoholic solutions of the alkaloids again in the separator and wash with a little distilled water; separate and draw off the water.

4. Add to the contents of the separator a warm mixture of—

Dilute hydrochloric acid	5 c.c.
Distilled water	25 ,,

Agitate thoroughly; separate and draw off the acid liquid, which will contain most of the alkaloids as hydrochlorides.

5. Repeat the treatment with acid and water, agitating thoroughly; separate and draw off the acid liquid, which add to the first.

6. To the mixed acid solutions add 10 c.c. of chloroform, and sufficient solution of ammonia to impart a distinct alkaline reaction, shake thoroughly and allow to separate; draw off the chloroform, which will contain the greater part of the alkaloids, into a counterpoised dish.

7. Repeat the agitation and separation with two successive quantities of 10 c.c. of chloroform, and add to that already in the counterpoised dish. Allow the chloroform to evaporate slowly, and finally dry the alkaloids at a temperature of about 230° F. (110° C.). The weight of the dish and its contents, after deducting the known weight of the dish, will give that of the alkaloids in 5 c.c., and this weight multiplied by 20, the weight of alkaloids in 100 c.c. of the strong percolate.

8. Having thus ascertained the alkaloidal strength of the strong percolate, every measure of it containing 5 grams of total alkaloids is first to be brought to 85 c.c. by evaporation, or if necessary by dilution with distilled water; then 12·5 c.c. of 90 per cent. alcohol is to be added, and sufficient distilled water to produce 100 c.c. The finished liquid extract will then contain 5 grams of the alkaloids of cinchona in every 100 c.c. As this liquid extract continues to deposit for some time after manufacture, it should not be filtered for at least fourteen days.

Example.—Supposing it was found in an experiment that 5 c.c. of the strong percolate yielded ·244 gram of alkaloid, then 102·4 c.c. would contain 5 grams; every 102·4 c.c., therefore, must be reduced by evaporation to 85 c.c., then 12·5 c.c. of 90 per cent. alcohol added, and sufficient distilled water to produce 100 c.c.

Liquid extract of ipecacuanha

A liquid extract obtained by exhausting ipecacuanha root with 90 per cent. alcohol by the process of percolation. It should contain between 2 and 2·25 grams of alkaloids in 100 c.c. The proportion of alkaloids present is estimated after precipitation of the colouring matter with solution of lead oxyacetate, by alkalising with solution of ammonia, dissolving out with chloroform, evaporating and weighing.

Details of process of manufacture.— Percolate 1½ pounds of coarsely powdered ipecacuanha root with 90 per cent. alcohol until 13½ fluid ounces of percolate have collected. Reserve this first portion. Remove the marc from the percolator and mix it intimately with 1½ ounces of freshly slaked and sifted lime, allowing them to remain in contact for twenty-four hours; then continue the percolation with more alcohol until exhaustion is complete. Distil off the alcohol, and dissolve the residual soft extract in the reserved portion of percolate. Determine the proportion of alkaloids in this strong liquid extract by the following process of assay.

Details of assay process.—1. Dilute 20 c.c. with an equal bulk of water, and evaporate on a water-bath to remove the alcohol; add excess of strong solution of lead oxyacetate (about 3 c.c.).

2. Filter; wash the precipitate with distilled water, and add the washings to the filtrate. Now add slight excess of normal sulphuric acid to remove the lead still in solution (about 5 c.c.); again filter, washing the precipitate as before.

3. Transfer the mixed filtrates, which now contain the alkaloids as sulphates, to a stoppered glass separator, add excess solution of ammonia, agitate with 25 c.c. chloroform, and set on one side.

4. Separate the chloroform and twice repeat the extraction with two other 25 c.c. of chloroform. Mix the chloroformic solutions of the alkaloids thus obtained, evaporate, dry at a temperature below 176° F. (80° C.), and weigh.

5. From this weight calculate the amount of alkaloids in the strong liquid extract, and add to the latter sufficient 90 per cent. alcohol to produce a liquid extract of ipecacuanha containing not less than 2 and not more than 2·25 grams of the alkaloids in 100 c.c.

Example.—Supposing 14 fluid ounces of the strong liquid extract is obtained by the process of percolation, 20 c.c. of which, when assayed as above, yield ·489 gram of alkaloids; then 100 c.c. will contain 2·445 grams. Therefore every 81·7 c.c. must be diluted with 90 per cent. alcohol to 100 c.c. $\left(\frac{100 \times 2}{2{\cdot}445} = 81{\cdot}7\right)$ to produce a liquid extract containing 2 grams of alkaloids in 100 c.c.

Liquid extract of nux vomica

A liquid extract obtained by exhausting nux vomica with 70 per cent. alcohol by the process of percolation. It should contain 1·5 grams of strychnine in 100 c.c.

The proportion of alkaloids present is determined by precipitating the mixed alkaloids, extracting with chloroform, converting into hydrochlorides, from which the strychnine is precipitated as ferrocyanide. The ferrocyanide compound is then decomposed by ammonia solution, the alkaloid extracted with chloroform, which, on evaporation, leaves it in a weighable condition.

Details of process of manufacture.—Percolate 1 pound of fresh nux vomica in 20 powder with 70 per cent. alcohol until 12 fluid ounces of percolate has collected. Reserve this portion. Continue the percolation with the alcohol until exhausted. Distil off the alcohol and evaporate the residue to 1 fluid ounce; dissolve in the reserved portion, and add sufficient 90 per cent. alcohol to produce 16 fluid ounces. Set aside for twenty-four hours and filter; determine the proportion of strychnine in this strong percolate by the following assay process.

Details of assay process.—1. Evaporate 10 c.c. of the above strong percolate on a water-bath to a syrupy consistence, so as to drive off all the alcohol. Dissolve the residue in 10 c.c. of warm water slightly acidulated with sulphuric acid, and transfer to a glass separator. Rinse out the dish with 10 c.c. more warm water and add 10 c.c. of chloroform. Rotate gently for five minutes and allow to separate *completely*, draw off and reject the chloroformic layer, which will contain the fat. Then add—

Sodium carbonate	5 grams.
Dissolved in warm water	25 c.c.

Agitate and add—

Chloroform	10 c.c.

Rotate gently for five minutes, and set aside for the chloroform, which contains most of the alkaloids, to separate.

2. Draw off the chloroformic solution, and twice repeat the agitation with additional quantities of 10 c.c. each of chloroform, separating as before.

3. Make a mixture of—

Diluted sulphuric acid	6 c.c.
Distilled water (warm)...	24 ,,

Divide into three equal parts and wash the mixed chloroformic solution with each in turn, so as to obtain the alkaloids in solution as sulphates.

4. Dilute the mixed acid liquids to 175 c.c., transfer to a stoppered flask, adding 25 c.c. of a 5 per cent. solution of potassium ferrocyanide. Shake well and frequently during half an hour, and then stand for six hours. This precipitates the strychnine as ferrocyanide, but does not affect the brucine.

5. Transfer the contents of the flask to a small filter, rinsing out the last portions with dilute sulphuric acid (1 in 250), and wash until the washings are free from bitterness (indicating removal of last trace of brucine).

6. Very carefully rinse the strychnine ferrocyanide into a separator, add 5 c.c. solution of ammonia, and shake well to set free the alkaloid; then add 10 c.c. of chloroform and shake well; allow the chloroformic solution of the alkaloid to separate, and draw off. Repeat the extraction with 5 c.c. of chloroform; separate and draw off as before. Mix the chloroformic solutions, and evaporate spontaneously in a counterpoised dish in a current of warm air; dry the residue for one hour on a water-bath, inverting a funnel over the dish to prevent loss of strychnine from spirting, and weigh.

7. From this weight calculate the amount of strychnine in the strong liquid extract, to which add sufficient 90 per cent. alcohol to produce a liquid extract of nux vomica containing 1·5 grams of strychnine in 100 c.c.

Example.—Supposing 16 fluid ounces of strong (filtered) percolate is obtained by the process of percolation, and 10 c.c., when assayed as above, yields ·176 gram of strychnine, then 100 c.c. will yield 1·76 grams. Therefore every 85·2 parts must be diluted to 100 parts with 90 per cent. alcohol $\left(\frac{100 \times 1{\cdot}5}{1{\cdot}76} = 85{\cdot}2\right)$.

Note 1.—In the preparation of liquid extract of nux vomica it is very important to see that the seeds are not rancid, as extract prepared from seeds in which the fat has become oxidised acquires a most disagreeable odour.

Note 2.—Although the Pharmacopœia does not require the above preparation to be standardised by titration, it is strongly recommended that the results be *checked* by this method. 10 c.c. $\frac{N}{10}$ H_2SO_4 should be run on to the alkaloid, and then be titrated back with $\frac{N}{100}$ KOH. Each c.c. $\frac{N}{10}$ H_2SO_4 in combination represents ·0331 gram strychnine. (Indicator, Brazil-wood solution.)

Opium

Any variety of opium may be used for tincture of opium, provided that when dry it contains not less than 7·5 per cent. of anhydrous morphine. For every other official purpose, however, it must contain when quite dry not less than 9·5 per cent., nor more than 10·5 per cent. of anhydrous morphine.

Opium yielding when dry more than 10 per cent. of anhydrous morphine may be diluted to that percentage with opium of weaker percentage, or with milk-sugar.

To dilute opium rich in morphia with opium of poorer quality, the percentage strength of the two samples having been ascertained by direct assay, the correct proportions are arrived at by a simple calculation.

Thus, supposing one sample of opium contained 14 per cent. of morphia and another 8 per cent.; then—

14 per cent. = 4 over 10.
8 ,, = 2 under 10.

Therefore proportionately 2 parts of the stronger opium (14 per cent.) must be mixed with 4 parts of the weaker (8 per cent.) to produce 6 parts of 10 per cent. opium.

Details of assay process.—1. Triturate together in a small mortar the following:

Opium, dried at 212° F., in 50 powder ...	14 grams.
Lime, freshly slaked	6 ,,
Distilled water	40 c.c.

When a uniform mixture results add 100 c.c. more water, and stir occasionally during half an hour to promote decomposition of the natural alkaloidal salts.

2. Filter the mixture into a wide-mouthed stoppered bottle having a capacity of 300 c.c. The filtrate will contain, besides the morphine in combination with the lime, a little extractive, and traces of other alkaloids. Filtration is continued until exactly 104 c.c. have collected, corresponding to 10 grams of the powdered opium.

3. To the 104 c.c. of filtered liquid add—

Alcohol, 90 per cent.	10 c.c.
Ether	50 „

Shake vigorously and add—

Ammonium chloride	4 grams.

Shake well and frequently during half an hour; set aside for twelve hours for the morphine to crystallise out. The precipitation of the alkaloid is brought about by the ammonium chloride, which converts the calcium hydroxide into chloride, at the same time liberating ammonia.

4. Now counterbalance two small filters; place one within the other in a small glass funnel in such a way that the triple fold of the inner filter shall rest upon the single fold of the outer filter; moisten the filters with ether. Remove the ethereal layer of the liquid in the bottle as completely as possible by means of a pipette, and transfer to the filter; rinse out the bottle with 20 c.c. of ether, allow to separate, and remove by means of a pipette as before; now wash the filter with 10 c.c. of ether, added slowly and in portions.

Note.—The foregoing treatment with ether practically removes all alkaloids except morphine; a little morphine dissolves in the ether and is lost, but as this loss is constant for the definite quantity of ether used, it is very properly allowed for in the final calculations.

5. Let the filter dry in the air, and pour upon it the liquid in the bottle in such a way as to transfer the crystallised morphine as completely as possible. When all the liquid has passed through, wash the remainder of the morphine from the bottle with *morphinated water* until the whole has been removed.

6. Wash the crystals with *morphinated water* until they are practically free from colour; allow the filter to drain, and dry it first by pressing between sheets of bibulous paper, afterwards at a temperature between 131° and 140° F. (55° and 60° C.), and finally at 230° F. (110° C.) for two hours; weigh the crystals in the inner filter, counterbalancing by the outer filter.

Check.—Dissolve ·5 gram of the crystals obtained as above in 20 c.c. $\frac{N}{10}$ H_2SO_4, and titrate back with $\frac{N}{100}$ KOH, using brazil-wood or hæmatoxylin as an indicator. Deduct the measure of potash solution required from 200 c.c. The remainder, divided by 10, indicates the number of c.c. of $\frac{N}{10}$ H_2SO_4 neutralised by the alkaloid. One c.c. $\frac{N}{10}$ H_2SO_4 = ·0283 gram anhydrous morphine.

$$\begin{array}{lll} \underbrace{2C_{17}H_{19}NO_3} & + \underbrace{H_2SO_4} & = (C_{17}H_{19}NO_3)_2H_2SO_4. \\ \quad 2)\overline{566{\cdot}1} & \quad 2)\overline{97{\cdot}34} & \\ 10)\overline{283{\cdot}05} & 10)\overline{48{\cdot}67} & \\ \quad \overline{28{\cdot}305} & \quad \overline{4{\cdot}867} & \end{array}$$

That is—

1000 c.c. $\frac{N}{10}$ H_2SO_4 = 28·305 grams pure anhydrous morphine.

1 c.c. ,, = ·0283 gram ,, ,,

The weight of pure anhydrous morphine in the *total weight* of impure crystals, as indicated by titration, *plus* ·104 (correction for loss of alkaloid during process) should not be less than ·95, and not more than 1·05 gram, corresponding to about 10 per cent. of anhydrous morphine in the powdered opium.

Example.—Supposing the weight of the crystals in the filter before titration was 1·10 gram, and that ·5 gram of these crystals titrated as above, indicated ·48 gram of *pure* anhydrous morphine. Then 1·1 gram would contain 1·056 grams of *pure* anhydrous morphine, and 1·056 + ·104 (the correction for loss of morphine during the process) = 1·160 gram in 10 grams of the dried opium; therefore, unless required for making tincture of opium, every 86·2 parts of the dry powder of the strength indicated above, must be diluted to 100 parts with sugar of milk $\left(\frac{100 \times 10}{11{\cdot}6} = 86{\cdot}2\right)$.

Morphinated water is prepared by digesting pure morphine in chloroform water until the latter is saturated, and filtering off.

Tincture of opium

Process of manufacture.—Digest 3 ounces of opium in 10 fluid ounces of distilled water at a temperature of 200° F., stirring occasionally, until reduced to a perfectly smooth paste. Allow to cool, and add 10 fluid ounces of 90 per cent. alcohol. Strain, press, set aside for several days and filter. Determine the proportion of morphine in the resulting strong tincture by the following process of assay.

Details of assay process.—1. Pour 80 c.c. into a porcelain dish, and evaporate over a water-bath until reduced to 30 c.c. Mix the residual liquid, now freed from alcohol, with 3 grams of freshly slaked lime, and dilute with water to exactly 85 c.c.; set aside for half an hour, stirring occasionally.

2. Filter off 50 c.c. of the liquid (representing 50 c.c. of the strong tincture) through a small paper filter into a wide-mouthed stoppered bottle, having a capacity of about 200 c.c., and add—

90 per cent. alcohol	5 c.c.
Ether	30 „

Agitate, and add—

Ammonium chloride	2 grams.

Shake well and frequently during half an hour, and stand for twelve hours for the morphine to crystallise out. The precipitation of the alkaloid is brought about by the interaction of the ammonium chloride with the calcium hydroxide, calcium chloride and ammonium hydroxide being formed. The alcohol facilitates the separation of morphine in crystals.

3. Counterbalance two small filters, place one within the other in such a way that the triple fold of the inner filter rests upon the single fold of the outer filter, and moisten them with ether. Remove the ethereal layer of the liquid in the bottle as completely as possible by means of a small pipette, and transfer it to the filter; pour into the bottle 15 c.c. ether, agitate gently, and set aside. When a complete separation has again taken place, transfer the ethereal layer to the filter by means of a pipette as before, and when all has passed through, wash the filter with a total amount of 10 c.c. of ether, added slowly and in portions; allow the filter to dry in air.

The treatment with ether practically removes all alkaloids except morphine; a little morphine does, however, dissolve in the ether and is lost, but as this loss is constant for a *definite* volume of ether, it is allowed for in the final calculation.

4. Now pour the contents of the bottle upon the filter in such a way as to transfer most of the crystals, washing the remainder of the morphine from the bottle with *morphinated* water (*vide ante*).

Continue the washing with the morphinated water until the washings are free from colour.

5. Allow the filter to drain; dry it first by gentle pressure between sheets of filter or blotting paper, and afterwards at a temperature between 131° and 140° F., and finally at 230° F. (110° C.), for two hours. Weigh the crystals in the inner filter, counterbalancing by the outer.

Check.—Take ·3 gram of the crystals dried as above, and titrate as described under Opium. According to the equation given under Opium 1 c.c. $\frac{N}{10}$ H_2SO_4 is equivalent to ·0283 gram of pure anhydrous morphine. Add to the weight of anhydrous morphine represented by the titration ·05 gram, a proportion representing the average loss of morphine during the process.

Having ascertained the proportion of anhydrous morphine present in the 50 c.c. of strong tincture, the remainder is to be diluted with 45 per cent. alcohol (equal parts of 90 per cent. alcohol and water) to produce a tincture of opium containing ·75 gram of morphine, calculated as anhydrous, in 100 c.c.

Example.—Suppose, for example, the weight of the crystals on the filter was ·47 gram, and that ·3 gram of these crystals titrated as before indicated ·28 gram of pure anhydrous morphine; then ·47 gram would contain ·438 gram of pure anhydrous morphine, and ·438 + ·05 (correction for loss) = ·443 gram of alkaloid in 50 c.c. of strong tincture, or ·886 gram in 100 c.c.

Therefore every 84·6 volumes of the strong tincture must be diluted with 45 per cent. alcohol to 100 volumes $\left(\frac{100 \times \cdot 75}{\cdot 886} = 84{\cdot}6\right)$.

CHAPTER LIII

GRAVIMETRIC ANALYSIS

In carrying out the gravimetric estimations of the Pharmacopœia, no especial apparatus is required other than that already mentioned in the earlier chapters of this work. Next in importance to the balance, which should turn with ·0005 gram (½ milligram), come filtering papers. These should be circular and of the best Swedish or Rhenish make, capable of retaining the finest precipitates. To such a pitch of excellence has the manufacture of these filters been brought, that no difficulty is experienced in obtaining paper that will keep back even recently precipitated barium sulphate, and yet not filter inordinately slowly.

When every fresh packet of filter-paper is opened, ten sheets should be dried, ignited, and weighed. This is most conveniently performed by folding the papers into a little bundle, held by a platinum wire. The paper is next set on fire, and held over a weighed platinum dish. As soon as the flame goes out, the charred residue is carefully shaken into the dish and ignited at a low red heat until perfectly white, when the dish is again weighed. The increase in weight indicates the amount of ash in the ten filters, so that dividing by ten gives the ash of one filter. With good paper that has been washed with hydrofluoric acid a filter of nine centimetres in diameter should not yield more than ·002 gram of ash. When an estimation has been made involving the ignition of precipitate and filter, the weight of the ash of the latter should always be deducted from the total weight.

Acid. Phosphoricum Concentratum.—One gram mixed with 2·5 grams pure lead oxide in fine powder should leave on evaporation a residue, which, after it has been heated to dull redness, weighs 2·98 grams.

$$\underbrace{3\mathrm{PbO}}_{663{\cdot}69} + \underbrace{2\mathrm{H_3PO_4}}_{194{\cdot}64} = \underbrace{\mathrm{Pb_3 2PO_4}}_{804{\cdot}69} + 3\mathrm{H_2O}.$$

As excess of lead oxide is used, the first calculation must be to find how much would be required to combine with 1 gram of 100 per cent. phosphoric acid.

According to the above equation 194·64 grams H_3PO_4 require 663·69 grams PbO, therefore 1 gram H_3PO_4 will require $\frac{663 \cdot 69}{194 \cdot 64} = 3 \cdot 4$ grams.

But as the pharmacopœial acid only contains 66·3 per cent. of real hydrogen orthophosphate, the figure becomes $\frac{3 \cdot 4 \times 66 \cdot 3}{100} =$ 2·25 grams of PbO required. Then, as 194·64 grams of hydrogen orthophosphate yield 804·69 grams of lead phosphate, 1 gram will yield $\frac{804 \cdot 69}{194 \cdot 64} = 4 \cdot 13$ grams if of 100 per cent.; but as the pharmacopœial acid is only 66·3 per cent., the figures become $\frac{4 \cdot 13 \times 66 \cdot 3}{100} = 2 \cdot 73$ grams.

It has been previously found that the theoretical amount of lead oxide for the decomposition of 1 gram of 66·3 per cent. acid is 2·25 grams; and as 2·5 grams were actually taken, the difference must be added to the weight of lead orthophosphate obtained, thus: 2·73 + ·25 = 2·98.

Acid. Phosphoricum Dilutum.—One gram mixed with ·5 gram pure lead oxide in fine powder should leave on evaporation a residue, which, after it has been heated to dull redness, weighs ·6 gram, corresponding to 13·8 per cent. by weight of hydrogen orthophosphate.

Ammonii Phosphas.—When 2 grams are dissolved in water, and ammonio-magnesium sulphate solution added in excess, a crystalline precipitate of ammonio-magnesium phosphate, $MgNH_4PO_4.6H_2O$, is formed, which when well washed on a filter with 5 per cent. solution of ammonia gas, dried, and heated to redness, should leave a residue of magnesium pyrophosphate, $Mg_2P_2O_7$, weighing 1·68 grams, corresponding to 100 per cent. of ammonium phosphate. The reactions that take place are expressed by the following equations:

$$(1)\quad (NH_4Mg)_2(SO_4)_3 + 2(NH_4)_2HPO_4 + 12H_20. = 2NH_4MgPO_4.6H_2O + (NH_4)_2SO_4 + 2NH_4HSO_4.$$

$$(2)\quad 2NH_4MgPO_4.6H_2O = Mg_2P_2O_7 + 13H_2O + 2NH_3.$$

Therefore $\underbrace{2(NH_4)_2HPO_4}_{262\cdot4} = \underbrace{Mg_2P_2O_7}_{221\cdot12}$.

As according to the last equation 221·12 grams of magnesium pyrophosphate are equivalent to 262·4 grams of ammonium phosphate, 1·68 grams will be equivalent to 2 grams of ammonium phosphate, equivalent to 100 per cent.

In performing the test great care must be taken that the stirring rod does not touch the side of the beaker, or it will lead to a deposition of crystals on the side of the glass. To obviate the danger the rod may be tipped with rubber; but a better plan consists in precipitating in a stoppered flask and agitating violently for ten minutes, at the end of which time precipitation is practically complete.

Antimonium Sulphuratum.—Three grams when warmed with successive portions of nitric acid until red fumes cease to be evolved, and then dried and heated to redness, should leave a white residue of antimony trioxide, weighing about 2 grams.

Sulphurated antimony consists principally of antimony pentasulphide, Sb_2S_5, with a little oxide. When the former is heated with nitric acid it is oxidised to antimonic acid (H_3SbO_4); this on gently heating yields antimony pentoxide (Sb_2O_5), which on further heating gives off oxygen and leaves a white residue of trioxide (Sb_2O_3). Thus—

(1) $Sb_2S_5 + 10HNO_3 = 2H_3SbO_4 + 5SO_2 + 10NO + 2H_2O$.
(2) $2H_3SbO_4 = Sb_2O_5 + 3H_2O$.
(3) $Sb_2O_5 = Sb_2O_3 + O_2$.

Therefore $\underbrace{Sb_2S_5}_{397\cdot1} = \underbrace{Sb_2O_3}_{285\cdot64}$.

As, according to the above equation, 397·1 grams of antimony pentasulphide are equivalent to 285·64 grams of antimony trioxide, 3 grams of the former should be equivalent to 2·15 grams of the trioxide. The Pharmacopœia requires the trioxide to weigh *about* 2 grams, which is equivalent to *about* 100 per cent.

Argenti Nitras.—One gram dissolved in 10 c.c. distilled water affords with hydrochloric acid a precipitate of silver chloride, which, when thoroughly washed and dried at 100° C. should weigh

·843 gram; while the filtrate when evaporated to dryness should not leave any residue.

$$\underbrace{AgNO_3}_{168·69} + HCl = \underbrace{AgCl}_{142·30} + HNO_3.$$

According to the above equation, 168·69 grams of silver nitrate yield 142·30 grams of silver chloride; 1 gram, therefore, yields ·843 gram (142·30 ÷ 168·69 = ·843), corresponding to 100 per cent.

Argenti Nitras Induratus.—One gram treated as for Argenti Nitras yields ·8 gram of silver chloride, corresponding to nearly 95 per cent. of silver nitrate.

Argenti Nitras Mitigatus.—Three grams treated as for silver nitrate yield ·843 gram, corresponding to about 33·3 per cent. of silver nitrate.

Argenti Oxidum.—One gram dissolved in nitric acid should yield with hydrochloric acid a precipitate of silver chloride, which when well washed and dried at 100° C. should weigh 1·237 grams.

$$Ag_2O + 2HNO_3 = 2AgNO_3 + H_2O.$$
$$2AgNO_3 + 2HCl = 2AgCl + 2HNO_3.$$

$$\text{Therefore } \underbrace{Ag_2O}_{230·1} = \underbrace{2AgCl}_{284·6}.$$

As 230·1 grams of silver oxide yield, when treated as above, 284·6 grams of chloride, 1 gram will yield 1·237 grams (284·6 ÷ 230·1 = 1·237), corresponding to 100 per cent.

Bismuthi Carbonas.—One gram dissolved in slight excess of weak hydrochloric acid and saturated with hydrogen sulphide gives a precipitate of bismuth sulphide, which, when very rapidly washed, and dried at 100° C., weighs ·99 gram.

$$(Bi_2O_2CO_3)_2H_2O + 12HCl = 4BiCl_3 + 2CO_2 + 7H_2O.$$
$$4BiCl_3 + 6H_2S = 2Bi_2S_3 + 12HCl.$$

$$\text{Therefore } \underbrace{(Bi_2O_2CO_3)_2H_2O}_{1029·7} = \underbrace{2Bi_2S_3}_{1020·12}.$$

As 1029·7 grams of bismuth oxycarbonate yield 1020·12 grams of sulphide, 1 gram will yield ·99 gram (1020·12 ÷ 1029·7 = ·99), corresponding to 100 per cent. of bismuth oxycarbonate.

Bismuthi Oxidum.—One gram dissolved in weak hydrochloric acid should yield with hydrogen sulphide a precipitate of bismuth sulphide, which, when well washed, and rapidly dried at 100° C., weighs 1·1 grams.

$$Bi_2O_3 + 6HCl = 2BiCl_3 + 3H_2O.$$
$$2BiCl_3 + 3H_2S = Bi_2S_3 + 6HCl.$$

$$\text{Therefore } \underbrace{Bi_2O_3}_{462{\cdot}24} = \underbrace{Bi_2S_3}_{510{\cdot}06}.$$

As 462·24 grams of bismuth oxide yield 510·06 grams of sulphide, 1 gram yields 1·1 grams (510·06 ÷ 462·24 = 1·1), corresponding to nearly 100 per cent. of oxide.

Bismuthi Salicylas.—One gram treated as for bismuth oxycarbonate should yield ·7 gram of bismuth sulphide.

$$(1)\ 2C_6H_4.OH.COO.BiO + 6HCl = 2C_6H_4.OH.COOH + 2BiCl_3 + 2H_2O.$$
$$(2)\ 2BiCl_3 + 3H_2S = Bi_2S_3 + 6HCl.$$

$$\text{Therefore } \underbrace{2C_6H_4.OH.COO.BiO}_{718{\cdot}38} = \underbrace{Bi_2S_3}_{510{\cdot}06}.$$

As 718·38 grams of bismuth oxysalicylate yield 510·06 grams of sulphide, 1 gram will yield ·7 gram (510·06 ÷ 718·38 = ·7) of sulphide, corresponding to very nearly 100 per cent. of bismuth oxysalicylate.

Bismuthi Subnitras.—One gram treated as for bismuth carbonate should yield ·84 gram of bismuth sulphide.

$$2BiO.NO_3.H_2O + 6HCl = 2BiCl_3 + 2HNO_3 + 4H_2O.$$
$$2BiCl_3 + 3H_2S = Bi_2S_3 + 6HCl.$$

$$\text{Therefore } \underbrace{2BiONO_3H_2O}_{605{\cdot}28} = \underbrace{Bi_2S_3}_{510{\cdot}06}$$

As 605·28 grams of bismuth oxynitrate yield 510·06 grams of sulphide, 1 gram will yield ·84 gram (510·06 ÷ 605·28 = ·84) of sulphide, corresponding to very nearly 100 per cent. of oxynitrate of bismuth. It is, however, unobtainable of this purity.

Calcii Phosphas.—When calcium orthophosphate is dissolved in excess of hydrochloric acid, calcium chloride and orthophosphoric acid are formed; on the addition of ammonia the excess of acid is neutralised and calcium phosphate reprecipitated, leaving any impurity behind. One gram treated in this manner should yield a precipitate of *pure* phosphate, which after washing, and drying at 100° C. should weigh ·95 gram, corresponding to 95 per cent. of calcium orthophosphate.

$$Ca_3(PO_4)_2 + 6HCl = 2H_3PO_4 + 3CaCl_2.$$
$$2H_3PO_4 + 3CaCl_2 + 6NH_4OH = Ca_3(PO_4)_2 + 6NH_4Cl + 6H_2O.$$

Ferri et Quininæ Citras.—Five grams, dissolved in 50 c.c. of water with excess of solution of ammonia, should yield a white precipitate of quinine alkaloid, which, dissolved out by repeated agitation with ether in a separator, evaporated, and dried at a temperature of 120° C., weighs ·75 gram, corresponding to 15 per cent. of alkaloid.

Note.—Ferric hydrate is formed at the same time, but is not precipitated, being held in solution by the ammonium citrate. (Compare preparation of Fehling's solution, in which cupric hydrate is held in solution by a tartrate.) If, however, caustic soda is added and the liquid well boiled, ferric hydrate is partly thrown out as a brownish-red precipitate.

Ferri et Ammonii Citras.—Here again ferric hydrate cannot be precipitated by solution of ammonia; 1 gram incinerated in an open crucible should leave ·31 gram of residue, corresponding to 31 per cent. of ferric oxide.

Ferrum Tartaratum.—Ten grams, when incinerated at a red heat, cooled, and washed with warm water to remove the potassium salt, dried, and again incinerated, should leave a residue weighing not less than 3 grams, corresponding to about 30 per cent. of ferric oxide.

Hydrargyri Iodidum Rubrum.—This is estimated by heating with copper filings, collecting and weighing the distilled mercury, which should amount to 44 per cent.

Take a piece of combustion tubing about 16 inches long, and seal one end in the blowpipe flame; allow to cool and introduce a few grams of magnesite, carefully avoiding tight packing. Next add the mercuric iodide which has been well mixed with excess of copper filings, then pour in small copper turnings until the tube is about three quarters full. Now draw out the open end of the tube in a blowpipe flame, and bend it at an angle of about 60 degrees. The prepared tube is then laid on asbestos cloth in a combustion furnace, and the burners immediately under the *turnings* lighted. As soon as it will bear it this part of the tube is heated to redness, when the burners under the iodide may be lighted. Decomposition soon commences, the mercury distilling over and condensing in the narrow portion of the tube, which should dip beneath the surface of a little water contained in a beaker. The temperature is maintained until mercury ceases to distil, when the burners under that portion of the tube containing the magnesite are lighted, with the effect that as the heat increases carbonic anhydride is given off, sweeping the last portions of mercury out of the tube. While still red-hot the bent portion of the tube containing a portion of the condensed mercury is broken off by a file cut, and the mercury globules shaken into the tared beaker. By rotating these globules are made to cohere, the water poured off, and the last traces of moisture got rid of, first by drying with blotting-paper, afterwards over sulphuric acid in a desiccator; finally weigh.

$$\underbrace{HgI_2}_{450{\cdot}60} = \underbrace{Hg.}_{198{\cdot}8}$$

As 450·60 grams of mercuric iodide yield 198·8 grams of mercury, 100 grams should yield 44·1 grams, corresponding to 100 per cent. of mercuric iodide.

Hydrargyri Oxidum Rubrum and *Hydrargyri Oxidum Flavum.*—When these oxides, which are identical in composition, are heated to incipient redness, they yield 92·5 per cent. of mercury. A combustion tube similar to that described under Hydrarg. Iodid. Rub. is used, magnesium carbonate being first introduced, to generate carbonic anhydride towards the close of the process. The mercuric oxide is then added so as to form a thin layer on the side of the tube. The prepared tube is then laid in the combustion furnace, with the bent end dipping into a beaker of water. As the temperature rises the oxide is decomposed, oxygen and mercury being given off. At the end of the process the last traces of mercury are swept out by carbonic anhydride as before, the mercury collected, dried, and weighed.

$$\underbrace{2HgO}_{429{\cdot}36} = \underbrace{Hg_2}_{397{\cdot}6} + O_2.$$

As 429·36 grams of mercuric oxide yield 397·6 grams of mercury, 100 grams should yield 92·5 grams of metallic mercury, corresponding to 100 per cent. of mercuric oxide.

Hydrargyri Perchloridum, Hydrargyri Subchloridum, and *Hydrargyrum Ammoniatum.*—These three compounds are estimated in a manner precisely similar to mercuric iodide, *with the exception* that lime is used in place of copper. The percentages of mercury obtained are relatively 73·8, 84·9, and 79, corresponding in each case to 100 per cent. purity of the various salts.

Liquor Bismuthi.—Ten c.c. diluted with 40 c.c. of water, and treated with excess of hydrogen sulphide, yield a precipitate of bismuth sulphide, which, when rapidly washed and dried, weighs ·55 gram. (See Bismuthi Carbonas.)

Liquor Ferri Perchloridi Fortior.—Five c.c. diluted with 80 c.c. of water yield, on the addition of solution of ammonia, a reddish-brown precipitate of ferric hydrate, which, when washed, dried, and incinerated, gives a residue of ferric oxide weighing 1·6 grams.

$$Fe_2Cl_6 + 6NH_4OH = 2Fe(OH)_3 + 6NH_4Cl.$$
$$2Fe(OH)_3 = Fe_2O_3 + 3H_2O.$$

$$\text{Therefore } \underbrace{Fe_2Cl_6}_{322{\cdot}34} = \underbrace{Fe_2O_3}_{158{\cdot}84}.$$

As 158·84 grams of ferric oxide represent 322·34 grams of ferric chloride, 1·6 grams represent 3·24 grams of chloride, equivalent to 64·8 grams of ferric chloride in 100 c.c. $\left(\frac{322{\cdot}34 \times 1{\cdot}6 \times 20}{158{\cdot}64} = 64{\cdot}8\right)$.

Liquor Ferri Pernitratis.—Five c.c. diluted and treated as above yield ·23 gram of ferric oxide.

$$Fe_2(NO_3)_6 + 6NH_4OH = 2Fe(OH)_3 + 6NH_4.NO_3.$$
$$2Fe(OH)_3 = Fe_2O_3 + 3H_2O.$$

Therefore $\underbrace{Fe_2(NO_3)_6}_{480·68} = \underbrace{Fe_2O_3}_{158·84}$.

As 158·84 grams of ferric oxide represent 480·68 grams of ferric nitrate, ·23 gram represents ·696 gram of nitrate, corresponding to 13·92 grams of ferric nitrate in 100 c.c.

$$\left(\frac{480·68 \times ·23 \times 20}{158·84} = 13·92\right).$$

Liquor Ferri Persulph.—Five c.c. treated as for Liquor Ferri Perchloridi Fortior yield 1·04 grams of ferric oxide.

$$Fe_2(SO_4)_3 + 6NH_4OH = 2Fe(OH)_3 + 3(NH_4)_2SO_4.$$
$$2Fe(OH)_3 = Fe_2O_3 + 3H_2O.$$

Therefore $\underbrace{Fe_2(SO_4)_3}_{397·22} = \underbrace{Fe_2O_3}_{158·84}$.

As 158·84 grams of ferric oxide represent 397·22 grams of ferric sulphate, 1·04 grams will represent 2·6 grams of sulphate, corresponding to 52 grams of ferric sulphate in 100 c.c. of solution.

Liquor Magnesii Carbonatis.—Twenty c.c. evaporated to dryness leaves a residue of pure hydrous carbonate ($MgCO_3.3H_2O$). This on calcining loses water and carbonic anhydride, a white residue of magnesium oxide being left, weighing between ·16 and ·19 gram, corresponding to *about* 2 per cent. of the *official* magnesium carbonate.

Lithii Carbonas.—One gram carefully neutralised with dilute sulphuric acid so as to avoid loss by spurting, dried, and heated to redness, should leave a residue of lithium sulphate weighing 1·479 grams, corresponding to 99·5 per cent. of lithium carbonate. The Pharmacopœia figures are incorrect.

$$\underbrace{Li_2CO_3}_{73·49} + H_2SO_4 = \underbrace{Li_2SO_4}_{109·28} + H_2O + CO_2.$$

Lithii Citras.—Two grams when ignited in a crucible with free access of air should leave a white residue of carbonate weighing ·77 gram, corresponding to 97·9 per cent. of lithium citrate.

$$\underbrace{2C_3H_4.OH(COOLi)_3.4H_2O}_{560\cdot10} + 5O_2 = \underbrace{3Li_2CO_3}_{220\cdot47} + 9CO_2 + 13H_2O.$$

Magnesia Levis and *Magnesia Ponderosa.*—These powders differ only in density, and consist of pure magnesium oxide, which should not lose weight after igniting at a red heat.

Magnesii Carbonas Levis et Ponderosus.—Five grams calcined at a low red heat should lose carbonic anhydride, and leave a residue of magnesium oxide weighing 2·1 grams.

$$\underbrace{(MgCO_3)_3Mg(OH)_2.4H_2O}_{380\cdot65} = \underbrace{4MgO}_{160\cdot24} + 3CO_2 + 5H_2O.$$

As 380·65 grams of oxycarbonate yield 160·24 grams of oxide, 5 grams should yield 2·1 grams of oxide, corresponding to 100 per cent. of official salt.

Magnesii Sulphas.—·5 gram dissolved in 250 c.c. of distilled water, when violently shaken for fifteen minutes in clean stoppered flask (*free from scratches inside*) with a mixture of ammonium hydroxide, ammonium chloride, and sodium phosphate, yields a precipitate of ammonio-magnesium phosphate, $MgNH_4PO_4.6H_2O$, which, when thoroughly washed with dilute ammonia, dried, and heated to redness, leaves a white residue of magnesium pyrophosphate, weighing ·22 gram.

$$2MgSO_4.7H_2O + 2Na_2HPO_4 + 2NH_4OH =$$
$$2MgNH_4PO_4 + 2Na_2SO_4 + 16H_2O.$$
$$2MgNH_4PO_4 = Mg_2P_2O_7 + 2NH_3 + H_2O.$$

$$\text{Therefore } \underbrace{2MgSO_4.7H_2O}_{489\cdot36} = \underbrace{Mg_2P_2O_7}_{221\cdot12}.$$

As 489·36 grams of magnesium sulphate yield 221·12 grams of pyrophosphate, ·5 gram sulphate should yield ·22 gram of pyrophosphate, corresponding to 100 per cent. of crystallised magnesium sulphate.

Morphinæ Acetas.—Two grams dissolved in 6 c.c. of warmed morphinated water and 1 drop of acetic acid give, with a *slight* excess of solution of ammonia, a white precipitate of alkaloid, which, when washed and dried as described under Morphinæ Hydrochloridum, weighs 1·42 grams.

$$\underbrace{C_{17}H_{19}NO_3.C_2H_4O_2.3H_2O}_{396·27} = \underbrace{C_{17}H_{19}NO_3}_{283·05}.$$

As 396·27 grams of the pure salt yield 283·05 grams of anhydrous alkaloid, 2 grams should yield 1·42 grams, corresponding to nearly 100 per cent. of morphine acetate.

Morphinæ Hydrochloridum.—Two grams dissolved in 250 c.c. of warmed morphinated water give, with *slight* excess of solution of ammonia, a precipitate of alkaloid which, when washed with a little cold morphinated water and dried, weighs 1·51 grams. The drying should be accomplished first by pressing the filter containing the precipitate between sheets of blotting-paper, and then at a temperature of about 55° C., finally at 110° C. for twenty minutes.

$$\underbrace{C_{17}H_{19}NO_3.HCl.3H_2O}_{372·88} = \underbrace{C_{17}H_{19}NO_3}_{283·05}.$$

As 372·88 grams of the salt yield 283·05 grams of alkaloid, 2 grams should yield 1·51 grams of alkaloid, corresponding to nearly 100 per cent. of morphine hydrochloride.

Morphinæ Tartras.—Two grams dissolved in 20 c.c. of warmed morphinated water give, with *slight* excess of solution of ammonia, a precipitate of alkaloid which, when washed and dried as described above, weighs 1·47 grams, corresponding to 100 per cent.

$$\underbrace{(C_{17}H_{19}NO_3)_2H_2C_4H_4O_6.3H_2O}_{768·66} = \underbrace{C_{17}H_{19}NO_3}_{566·1}.$$

Potassii Sulphas.—One gram dissolved in distilled water acidified with hydrochloric acid gives, with solution of barium chloride, a precipitate of barium sulphate, which, when washed and dried at 100° C., weighs 1·339 grams.

$$\underbrace{K_2SO_4}_{173·00} + BaCl_2 = \underbrace{BaSO_4}_{231·74} + 2KCl.$$

As 173 grams of potassium sulphate yield 231·74 grams of barium sulphate, 1 gram will yield 1·339 grams of sulphate, corresponding to 100 per cent. of potassium sulphate.

Sodii Arsenas (anhydrous).—The Pharmacopœia directs that 1 gram dissolved in distilled water acidified with acetic acid should require not less than 2·03 grams of lead acetate for complete precipitation, thus:

$$2Na_2HAsO_4 + 3Pb(C_2H_3O_2)_2 =$$
$$Pb_3(AsO_4)_2 + 4NaC_2H_3O_2 + 2HC_2H_3O_2.$$

The author recommends the following process:—Dissolve 2 grams sodium arsenate in water, add excess of ammonio-magnesium sulphate solution, and shake vigorously in a stoppered flask for five minutes. Transfer the precipitate (ammonio-magnesium arsenate) to a filter, and wash with dilute solution of ammonia (5 per cent.). Dry, heat to redness. The residue of magnesium pyroarsenate, $Mg_2As_2O_7$, should weigh 1·19 grams.

$$(1)\ 2Na_2HAsO_4 + (NH_4Mg)_2(SO_4)_3 =$$
$$2NH_4MgAsO_4 + 2NaHSO_4 + Na_2SO_4.$$
$$(2)\ 2NH_4MgAsO_4.6H_2O = Mg_2P_2O_7 + 13H_2O + 2NH_3.$$

$$\text{Therefore } \underbrace{2Na_2HAsO_4}_{369·56} = \underbrace{Mg_2P_2O_7.}_{221·12}$$

Since 221·12 parts magnesium pyroarsenate are equivalent to 369·56 of anhydrous sodium arsenate, a residue of 1·19 grams represents 2 grams, or 100 per cent. purity.

Sodii Sulphas.—One gram dissolved in distilled water acidified with hydrochloric acid gives, with solution of barium chloride, a precipitate of barium sulphate, which, when washed and dried at 100° C., weighs ·725 gram, corresponding to about 100 per cent.

$$\underbrace{Na_2SO_4.10H_2O}_{319·9} + BaCl_2 = \underbrace{BaSO_4}_{231·74} + 2NaCl + 10H_2O.$$

Zinci Valerianas.—One gram heated to redness after moistening with nitric acid should leave not less than ·26, and not more than ·304 gram of zinc oxide.

$$\underbrace{Zn(C_5H_9O_2)_2}_{265{\cdot}53} = \underbrace{ZnO}_{80{\cdot}79}.$$

(Then 80·79 ÷ 265·53 = ·304.)

CHAPTER LIV

TESTS FOR SUBSTANCES MENTIONED IN THE TEXT OF THE PHARMACOPŒIA, WITH EXPLANATIONS OF THE MORE IMPORTANT REACTIONS

Acetates.—1. Acetic acid and acetic ether possess characteristic odours.

2. All the acetates are soluble in water; the neutral or faintly acid solution affords a deep red coloration (ferric acetate) with ferric chloride, which on boiling deposits a reddish-brown precipitate (ferric oxyacetate). The red colour due to the formation of ferric acetate is not discharged by mercuric chloride solution; it turns yellow on the addition of hydrochloric acid.

3. Acetates warmed with sulphuric acid evolve the odour of acetic acid; warmed with sulphuric acid and a little ethylic alcohol, ethyl acetate is formed.

4. When dry acetates are heated gradually, they yield acetone:

$$(CH_3.COO)_2Ca = CH_3.CO.CH_3 + CaCO_3.$$

5. When heated with a caustic alkali they yield methane:

$$CH_3.COOK + KOH = CH_4 + K_2CO_3.$$

6. When heated with arsenious anhydride they yield cacodyl oxide (dimethylarsine oxide):

$$4CH_3.COOK + As_2O_3 = As_2(CH_3)_4O + 2K_2CO_3 + 2CO_2.$$

Cacodyl oxide has an intensely obnoxious smell, and is exceedingly poisonous, consequently very minute quantities should be used in performing this test.

Aluminium.—1. Ammonium hydrate solution and ammonium hydrosulphide solution afford a white gelatinous precipitate of aluminium hydrate:

$$Al_2(SO_4)_3 + 6NH_4OH = Al_2(OH)_6 + 3(NH_4)_2SO_4.$$

Freshly precipitated aluminium hydrate is soluble in hydrochloric acid, acetic acid, and in solution of potassium hydroxide, but nearly insoluble in solution of ammonia, and quite insoluble if the solution is boiled.

Ammonium salts.—1. Ammonium salts volatilise when strongly heated. Benzoate, bromide, carbonate, and chloride leave no residue, but *sublime* unchanged. Ammonium phosphate when strongly heated leaves a residue of meta-phosphoric acid. The nitrate decomposes into nitrous oxide and water:

$$NH_4NO_3 = N_2O + 2H_2O.$$

Ammonium nitrite yields nitrogen and water:

$$NH_4NO_2 = N_2 + 2H_2O.$$

2. When heated with solution of potassium or sodium hydroxide, all salts of ammonia yield ammonia gas, recognised by its odour, and a glass rod dipped in hydrochloric acid held over the test-tube in which the operation is performed produces white fumes of ammonium chloride.

3. Solution of platinic chloride affords with ammonium salts acidified with hydrochloric acid a yellow crystalline precipitate of the double chloride of platinum and ammonium, $PtCl_4.2NH_4Cl$. If the solution is very dilute, a little alcohol should be added. The double salt on ignition leaves a residue of metallic platinum.

4. A concentrated solution of tartaric acid produces in concentrated solutions of ammonium salts a white crystalline precipitate of acid tartrate of ammonium, especially in the presence of alcohol:

$$NH_4Cl + H_2C_4H_4O_6 = NH_4HC_4H_4O_6 + HCl.$$

5. Solution of potassio-mercuric iodide (Nessler's solution) affords a brown precipitate of oxy-dimercuric ammonium iodide:

$$4NH_3 + 2HgI_2 + H_2O = (NH_2Hg)IHgO + 3NH_4I.$$

In *very* dilute solutions a coloration ranging from reddish brown to yellow is produced.

Antimony.—1. Hydrogen sulphide in slightly acid solutions yields an orange-coloured precipitate of amorphous antimonious sulphide, Sb_2S_3. This precipitate has the same composition as the crystalline black sulphide, into which it is converted on drying and exposing to heat. The moist precipitate dissolves in solution of potassium hydroxide, forming potassium antimonite, K_3SbO_3, and thioantimonite; in solution of ammonium hydrosulphide forming ammonium thioantimonite $(NH_4)_3SbS_3$. It also dissolves in warm strong hydrochloric acid, evolving hydrogen sulphide, leaving antimonious chloride, $SbCl_3$, in solution (distinction from arsenic). Antimonious sulphide is almost insoluble in solution of the official ammonium carbonate, and in solution of acid potassium sulphite.

2. Antimony compounds, when placed in a Marsh's apparatus (Fig. 248) with dilute hydrochloric acid and some fragments of zinc, suffer decomposition, the antimony coming off as hydrogen antimonide. If the evolved gases are burnt at the jet, antimony oxide, Sb_2O_3, is formed; but if a cold porcelain tile be held in the flame a stain of metallic antimony forms, which is *not* appreciably dissolved by solution of chlorinated soda (distinction from arsenic).

3. When hydrogen antimonide, SbH_3, is led into solution of silver nitrate, a black precipitate of metallic silver and antimony is formed, and on the cautious addition of ammonium hydroxide to the supernatant liquid it causes no yellow precipitate (distinction from arsenic).

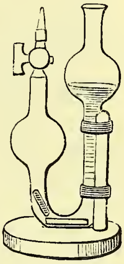

FIG. 248.—Marsh's apparatus.

$$SbH_3 + 3AgNO_3 = 3HNO_3 + SbAg_3.$$

4. If one end of a strip of pure zinc be allowed to rest on a bright platinum capsule, containing an acidified antimony solution, the other end being in the liquid, hydrogen antimonide is *not* evolved, but the antimony is precipitated on the platinum as a black, adherent, non-granular stain, insoluble in hydrochloric acid (distinction from tin, reduced in the same manner).

5. Reinch's test (see Arsenic) produces a black coating of metallic antimony on bright copper-foil, which, if gently dried and heated in a glass tube, sublimes and condenses *close* to the copper as the white *amorphous* trioxide, Sb_2O_3. This sublimate is insoluble in water, but readily dissolves in a boiling solution of acid tartrate of potassium.

Arsenic.—1. Hydrogen sulphide, after acidulation with hydrochloric acid, affords a yellow precipitate of arsenious sulphide, As_2S_3; arsenious sulphide may be regarded as a thioanhydride, as it gives rise to a series of salts known as thioarsenites, or sulpharsenites. Thus when arsenic trisulphide is added to a solution of a caustic alkali such as potassium hydroxide, the sulphide readily dissolves with the formation of arsenite and thioarsenite:

$$As_2S_3 + 4KOH = HK_2AsO_3 + HK_2AsS_3 + H_2O.$$

2. The yellow precipitate is also soluble in solution of the official ammonium carbonate, forming ammonium thioarsenite, $(NH_4)_3AsS_3$; in a hot solution of hydrogen potassium sulphite (distinction from the sulphides of Sb, Sn, Au, and Pt) forming metarsenite and thiosulphate, thus:

$$2As_2S_3 + 16KHSO_3 = 4KAsO_2 + 6K_2S_2O_3 + 7SO_2 + S_3 + 8H_2O.$$

From these solutions arsenic trisulphide is again precipitated on heating with hydrochloric acid. In contradistinction to antimony, arsenic trisulphide is insoluble in strong hydrochloric acid.

3. Arsenic compounds, when introduced into a Marsh's apparatus (see Antimony) with dilute hydrochloric acid and some fragments of zinc, suffer decomposition, the arsenic coming off as hydrogen arsenide. If the evolved gases are ignited at the jet As_2O_3 is formed, but if a cold porcelain tile be held in the flame a stain of metallic arsenic forms, readily dissolved in solution of chlorinated soda (distinction from antimony).

4. When hydrogen arsenide, AsH_3, is led into solution of silver nitrate a black precipitate of metallic silver is produced, and the cautious addition of ammonium hydroxide to the supernatant liquid causes a yellow precipitate of silver arsenite, Ag_3AsO_3.

5. If a few fragments of zinc are boiled with solution of potassium hydroxide and an arsenical compound added, hydrogen arsenide is given off, and is detected by the black stain produced on a cap of white filtering-paper, moistened with silver nitrate solution, placed over the mouth of the test-tube (Fleitmann's test).

In this reaction hydrogen arsenide is formed, which reduces the silver nitrate, forming arsenious and nitric acids and liberating metallic silver.

(*a*) $Zn + 2NaOH = H_2 + Na_2ZnO_2.$
(*b*) $As_2O_3 + 6H_2 = 2AsH_3 + 3H_2O.$
(*c*) $AsH_3 + 3H_2O + 6AgNO_3 = H_3AsO_3 + 6HNO_3 + 3Ag_2.$

Note.—Fleitmann's test does not react with antimony compounds at all.

6. Stannous chloride dissolved in a large excess of hydrochloric acid gives, on boiling with arsenical compounds, a

brownish-black precipitate of metallic arsenic, the stannous chloride being oxidised to the stannic salt at the same time. This test (Bettendorff's) cannot be applied in the presence of much water, or of nitrates. It is very useful for detecting arsenic in hydrochloric or sulphuric acid, or in tartar emetic.

7. If an arsenical solution is acidulated with hydrochloric acid and boiled with a strip of bright copper-foil, a grey coating of cupric arsenide is deposited. On drying the copper and cutting into fragments and heating in a wide glass tube, a *crystalline* sublimate of As_2O_3 is formed *at some distance* from the heated portion. On examining under a lens the sublimate is seen to consist of characteristic octahedral crystals (compare Antimony).

8. *Arsenites.*—These yield a yellow precipitate of argentic arsenite, Ag_3AsO_3, with solution of silver ammonio-nitrate.

Arsenates.—These yield a chocolate-coloured precipitate of silver arsenate, Ag_3AsO_4, with solution of silver ammonio-nitrate.

Solution of ammonio-magnesium sulphate yields a white crystalline precipitate.

Bismuth.—1. Hydrogen sulphide affords a brownish-black precipitate of bismuth sulphide, Bi_2S_3. This, the trisulphide, is the only sulphide of bismuth that is known with certainty; it differs from the sulphides of arsenic and antimony in not being soluble in the alkaline hydrates or sulphides. It is also insoluble in solution of potassium cyanide and in dilute hydrochloric acid. It is decomposed and dissolved by boiling nitric acid.

2. Solution of potassium hydroxide, sodium hydroxide, or ammonia (except in the presence of citrates, &c.), yields a white precipitate of hydrated bismuthous oxide, $Bi_2O_3.3H_2O$, insoluble in excess, and becoming converted into the monohydrate on boiling, $Bi_2O_3.H_2O$.

3. When a neutral, or nearly neutral solution of a bismuth salt is poured into a large excess of dilute solution of sodium chloride a white precipitate of bismuth oxychloride, $BiOCl$, is formed, which is insoluble in tartaric acid (distinction from antimony oxychloride).

4. Solution of neutral potassium chromate gives a yellow precipitate of bismuth oxychromate, $Bi_2O_2.CrO_4$, soluble in dilute nitric acid, insoluble in solution of potassium hydroxide (distinction from lead chromate).

Note.—Bismuth salts are not precipitated by dilute sulphuric acid.

Bromates.—1. Sulphurous acid liberates bromine from bromates, recognised by its odour and appearance; on adding a few drops of chloroform it dissolves the liberated bromine, and sinks to the bottom of the test-tube as a brown globule.

2. After ignition with charcoal, bromates are converted into bromides, and the latter yield the characteristic reactions. (See Bromides.)

$$2KBrO_3 + 3C_2 = 2KBr + 3CO_2.$$

Bromides or hydrobromides.—1. All the bromides are soluble in water with the exception of the silver, lead, and mercurous salts.

2. Solution of silver nitrate gives a yellowish curdy precipitate of silver bromide, $AgBr$, readily soluble in solution of potassium cyanide to form a double cyanide of silver and potassium. Silver bromide is slightly soluble in strong, but almost insoluble in weak solution of ammonia, and quite insoluble in nitric acid.

3. Solution of sodium nitrite acidified with dilute hydrochloric acid does not liberate bromine from a bromide (distinction from iodides).

4. Solution of chlorine liberates bromine from bromides, and if a few drops of chloroform are shaken up with the mixture, a brownish-coloured globule containing the bromine settles at the bottom of the tube when allowed to rest. Care must be taken not to have the chlorine in great excess, or colourless bromine trichloride may form.

5. Bromine is liberated when a bromide is heated with sulphuric acid and an oxidiser, such as manganese peroxide, lead peroxide, or potassium dichromate, and the liberated vapour gives an orange-yellow colour to white filter-paper soaked in mucilage of starch. The sulphuric acid first forms hydrobromic acid, and on the addition of an oxidising agent bromine is set free, thus:

$$2KBr + MnO_2 + 2H_2SO_4 = MnSO_4 + K_2SO_4 + 2H_2O + Br_2.$$

Note.—When testing for bromides in iodides, all iodine should first be removed by boiling the aqueous solution with excess of lead peroxide.

Cadmium.—1. Hydrogen sulphide yields a yellow precipitate of cadmium sulphide, CdS, insoluble in weak cold dilute hydrochloric acid, insoluble in solution of ammonium hydrosulphide or potassium hydroxide (distinction from the somewhat similar precipitates of arsenic, antimony, or stannic sulphides); insoluble in solution of potassium cyanide (distinction from copper); soluble in nitric acid, hot diluted sulphuric acid, and hot diluted hydrochloric acid.

2. Solution of potassium or sodium hydroxide affords a white precipitate of cadmium hydrate, $Cd(OH)_2$, insoluble in excess (distinction from zinc).

3. Solution of ammonia gives a white precipitate of cadmium hydrate, $Cd(OH)_2$, readily soluble in excess.

Calcium.—1. Solution of ammonium carbonate yields a white precipitate of calcium carbonate, $CaCO_3$, insoluble in solution of ammonium chloride (distinction from magnesium).

2. Solution of ammonium oxalate gives a white precipitate of calcium oxalate, CaC_2O_4, soluble in hydrochloric acid, but insoluble in acetic acid.

3. Solution of potassium chromate ($K_2Cr_2O_4$) gives no precipitate (distinction from barium).

Carbonates and Bicarbonates.—1. Dilute acids cause an effervescence of carbonic anhydride, which is *odourless*, and passed into lime water gives a white precipitate of $CaCO_3$.

2. Soluble carbonates afford a brownish-red precipitate of mercuric oxide, with mercuric chloride. Bicarbonates (when pure) yield a white opalescence, but if the test-tube is violently shaken and slightly warmed the precipitate becomes more decided and of a brownish tinge. This is caused by loss of carbonic anhydride, the normal carbonate then precipitating mercuric oxide.

3. Soluble carbonates yield a white precipitate with solution of magnesium sulphate in the cold; bicarbonates do not. That is to say, magnesium carbonate is an insoluble, magnesium bicarbonate a soluble salt.

Chlorides or hydrochlorides.—1. Solution of silver nitrate affords a white curdy precipitate of silver chloride, AgCl, soluble in solution of ammonia, but insoluble in nitric acid. Silver chloride dissolves in potassium cyanide solution to form a double cyanide of potassium and silver.

2. A solid chloride when subjected to distillation with sulphuric acid and potassium dichromate yields a reddish-brown distillate of chromyl chloride, CrO_2Cl_2, which is decomposed by water into

chromium trioxide and hydrochloric acid. The resulting solution when nearly neutralised with solution of ammonia gives a yellow precipitate of lead chromate, $PbCrO_4$, with solution of lead acetate; and a mixed red and white precipitate of silver chromate, Ag_2CrO_4, and silver chloride, AgCl, with solution of silver nitrate. The red precipitate is dissolved by nitric acid, and both precipitates by solution of ammonia.

3. Heated with manganese dioxide and sulphuric acid chlorides yield chlorine, recognised by giving a blue colour with solution of potassium iodide and mucilage of starch.

Citrates.—1. Heated alone, citrates slowly become charred, evolving at the same time a faint odour of burnt sugar. At a dull red heat the citrates of potassium, sodium, and lithium become converted into carbonates.

2. Solution of calcium chloride added in excess affords, *when boiled* with a neutral solution of a citrate, a white precipitate of calcium citrate, $Ca_3(C_6H_5O_7)_2$, insoluble in solution of potassium hydroxide, but soluble in solution of ammonium chloride and in solution of alkaline citrates.

3. Solution of nitrate of silver, causes in solutions of neutral citrates, a white precipitate soluble in solution of ammonia. A mirror is not formed on the sides of the tube when the ammoniacal solution is heated (distinction from tartrates).

Copper.—1. Hydrogen sulphide or ammonium hydrosulphide yield in solutions which are not strongly acid, a brownish-black precipitate of cupric sulphide, CuS; cupric sulphide is insoluble in dilute hydrochloric acid and in solution of potassium hydroxide, *almost* insoluble in ammonium hydrosulphide; it is dissolved by boiling nitric acid, and when *freshly precipitated*, by solution of potassium cyanide.

2. Solution of potassium hydroxide gives a bulky light-blue precipitate of cupric hydrate, $Cu(OH)_2$, which is decomposed upon boiling into brownish-black cupric oxyhydrate, $(CuO)_2Cu(OH)_2$. Cupric hydrate is soluble in a very large excess of a concentrated solution of potassium hydroxide, forming a blue solution.

3. In the presence of neutral tartrates and citrates the light blue precipitate of cupric hydrate redissolves almost as soon as formed, yielding a deep blue solution, which is not affected by heat.

4. If a little dextrose be mixed with solution of potassium hydroxide and then with a soluble cupric salt, a similar deep blue

solution is obtained, but when warmed it deposits a bright red precipitate of cuprous oxide, Cu_2O, the solution becoming colourless if sufficient dextrose has been added.

5. Solution of ammonia or ammonium carbonate added in small quantity to a neutral solution of a copper salt gives a greenish-blue precipitate, which readily dissolves in excess of solution of ammonia, forming a deep blue solution, containing—in the case of cupric sulphate—tetrammonio-cupric sulphate, $(NH_3)_4CuSO_4.H_2O$. This blue coloration is perceptible in exceedingly dilute solutions.

6. Solution of potassium ferrocyanide gives a reddish-brown precipitate of cupric ferrocyanide, $Cu_2FeC_6N_6$, or in very dilute solutions a reddish-brown coloration, unaffected by dilute acids, but decomposed by alkaline liquids.

7. Bright steel, such as the blade of a knife, receives a reddish coating of metallic copper when placed in a solution of a copper salt.

Cyanides.—1. Solution of silver nitrate affords a curdy white precipitate of silver cyanide, AgCN, soluble in solution of potassium cyanide, forming the double salt, KCN.AgCN. Silver cyanide is also soluble in boiling concentrated nitric acid, and moderately but slowly soluble in solution of ammonia.

2. If to a soluble cyanide be added a few drops of a mixed solution of ferrous and ferric salts, then of solution of potassium hydroxide, and lastly excess of hydrochloric acid, a precipitate of Prussian blue results, $Fe_4(Fe[C_6N_6])_3$. The explanation of this test is as follows:

(*a*) The addition of the ferrous salt produces ferrous cyanide.

(*b*) This reacting with excess of alkali produces potassium ferrocyanide.

(*c*) On the addition of the ferric salt it is first precipitated by the excess of alkali as ferric hydrate, which on acidulation dissolves to ferric chloride, forming ferric ferrocyanide or Prussian blue, thus:

(*a*) $6KCN + 3FeCl_2 = 3Fe(CN)_2 + KCl.$
(*b*) $3Fe(CN)_2 + 4KOH = K_4FeC_6N_6 + 2Fe(OH)_2.$
(*c*) $3K_4FeC_6N_6 + 2Fe_2Cl_6 = Fe_4(Fe[C_6N_6])_3 + 12KCl.$

3. When insoluble cyanides are heated in a dry tube they decompose and yield cyanogen, C_2N_2, which burns with a characteristic peach-coloured flame.

Hydrobromides.—See Bromides.

Hydrochlorides.—See Chlorides.

Iodates.—1. Solution of silver nitrate gives a white crystalline precipitate of silver iodate, sparingly soluble in water and in dilute nitric acid, but readily dissolved by solution of ammonia. Sulphurous acid, when added to the ammoniacal solution, gives a pale yellow precipitate of silver iodide.

$$(a)\ AgNO_3 + KIO_3 = AgIO_3 + KNO_3.$$
$$(b)\ AgIO_3 + 3H_2SO_3 = AgI + 3H_2SO_4.$$

2. A mixed solution of potassium iodide and a soluble iodate with tartaric acid liberates iodine, which yields a blue colour with mucilage of starch. When excess of tartaric acid is added to an iodate, iodic acid is set free ; and when the same acid is added to an iodide, hydriodic acid is formed.

$$KIO_3 + 5KI + 6H_2C_4H_4O_6 = 5HI + HIO_3 + 6KHC_4H_4O_6.$$

The hydriodic acid being a reducing agent at once acts on the iodic acid, iodine being set free and water formed.

$$5HI + HIO_3 = 3I_2 + 3H_2O.$$

3. Solution of barium chloride gives a white precipitate, nearly insoluble in water and soluble with difficulty in diluted nitric acid.

4. On the addition of mucilage of starch and sulphurous acid a blue colour is produced.

$$2KIO_3 + 5H_2SO_3 = I_2 + 3H_2SO_4 + 2KHSO_4 + H_2O.$$

Iodides.—1. Solution of silver nitrate affords a curdy yellow precipitate of silver iodide, insoluble in nitric acid, almost insoluble in solution of ammonia, but dissolving in solution of potassium cyanide to form a double cyanide of potassium and silver.

2. Solution of mercurous nitrate produces a green precipitate of mercurous iodide, HgI, insoluble in diluted nitric acid, but soluble in solution of potassium iodide.

3. Solution of mercuric chloride yields a scarlet precipitate of mercuric iodide, HgI_2, slightly soluble in excess of the mercuric chloride, and very soluble in iodide of potassium solution.

4. Solution of lead acetate causes a yellow precipitate of lead

iodide, PbI_2, soluble in diluted nitric acid, and soluble in boiling water. From the latter solution the precipitate separates in golden scales as the solution cools.

5. Solution of copper sulphate mixed with a solution of ferrous sulphate, or with sulphurous acid, affords with neutral solutions a whitish precipitate of cuprous iodide, Cu_2I_2, soluble in solution of ammonia and slightly soluble in hydrochloric acid.

$$(a)\ 2KI + 2CuSO_4 + 2FeSO_4 = Cu_2I_2 + K_2SO_4 + Fe_2.3SO_4.$$
$$(b)\ 2KI + 2CuSO_4 + H_2SO_3 + H_2O = Cu_2I_2 + 2KHSO_4 + H_2SO_4.$$

This test is especially valuable, as it distinguishes iodides from both chlorides and bromides, neither of which are precipitated by this treatment.

6. A small quantity of chlorine or bromine, preferably added in aqueous solution, liberates iodine from iodides.

Solution of sodium nitrite and dilute hydrochloric acid also acts in a similar manner. If only a very small amount of iodine is set free it may either be detected by the intense blue colour it produces with starch mucilage in the cold, or the iodine may be dissolved out by shaking with carbon disulphide, to which it communicates a characteristic violet colour.

Iron.—*Reactions common to both ferrous and ferric salts.*—1. Solution of ammonium hydrosulphide yields in neutral solutions a black precipitate of ferrous sulphide, FeS, soluble in cold diluted hydrochloric acid with evolution of hydrogen sulphide.

2. Solution of potassium ferrocyanide gives with ferric salts a precipitate of Prussian blue, or with ferrous salts a white precipitate rapidly turning blue, of Everett's salt—potassium ferrous ferrocyanide, $K_2FeFeC_6N_6$. Either precipitate is insoluble in dilute hydrochloric acid, but is decomposed by solution of potassium hydroxide.

Reactions characteristic of ferrous salts.—1. Hydrogen sulphide does not cause any precipitate in slightly acid solutions.

2. Solution of potassium ferricyanide affords a dark blue precipitate of Turnbull's blue, ferrous ferricyanide, $Fe_3Fe_2C_{12}N_{12}$, insoluble in dilute hydrochloric acid, but decomposed by solution of potassium hydroxide. (Ferric salts give a reddish coloration with this reagent, but no precipitate.)

3. Ferrous salts mixed with solution of potassium hydroxide give a dull green precipitate of ferrous hydrate, $Fe(OH)_2$.

Alkalies are incomplete precipitants of ferrous salts, as the hydrate is slightly soluble in excess of the reagent.

Reactions characteristic of ferric salts.—1. Ferric salts are not precipitated by hydrogen sulphide, but if the gas be passed for some time through a solution containing a ferric salt, a white precipitate of sulphur is thrown down, due to the following reaction taking place :

$$2Fe_2Cl_6 + 2H_2S = 4FeCl_2 + 4HCl + S_2.$$

The iron is reduced to the ferrous state, and remains in solution in the hydrochloric acid, while the sulphur is precipitated.

2. Solution of ammonium thiocyanate, $(NH_4)SCN$, produces a blood-red coloration due to the formation of ferric thiocyanate, $2Fe(CNS)_3.3H_2O$. The red colour is discharged on addition of solution of mercuric chloride (distinction from ferric meconate), but it is not discharged by hydrochloric acid (distinction from ferric acetate).

3. Solution of tannic acid yields a bluish-black coloration or precipitate of tannate with ferric salts, and more slowly with ferrous salts.

4. Solutions of potassium, sodium, or ammonium hydroxide cause a reddish-brown precipitate of ferric hydrate, soluble in solution of citric or tartaric acid. Ferric hydrate is not precipitated in presence of citrates or tartrates (see Ferri et Quininæ Citras).

Lead.—1. Hydrochloric acid affords, except in very weak solutions, a white precipitate of lead chloride, soluble in boiling water. The aqueous solution as it cools deposits the salt in crystalline forms.

2. Hydrogen sulphide yields a black precipitate of lead sulphide in not very strongly acid solutions, insoluble in solution of potassium hydroxide and in solution of ammonium hydrosulphide. Lead sulphide is decomposed by boiling with diluted nitric acid, being partly converted into soluble lead nitrate, and partly into insoluble lead sulphate and free sulphur.

3. Diluted sulphuric acid causes a white precipitate of lead sulphate, $PbSO_4$, almost insoluble in water, and rendered still less soluble by the addition of dilute sulphuric acid or alcohol, but readily soluble in solutions of ammoniacal salts, especially the acetate.

4. Solution of potassium chromate produces a dense orange-

coloured precipitate of lead chromate, $PbCrO_4$, which changes to bright red basic chromate, Pb_2CrO_5, on boiling with solution of potassium hydroxide, and redissolving, when heated with excess of the latter reagent, to form a pale lemon-coloured solution.

5. Solution of potassium hydroxide causes a white precipitate of lead hydrate, dissolving in excess of the reagent to form potassium plumbite, K_2PbO_2. This compound shows that lead possesses, although to a feeble extent, the acidic properties exhibited by other members of the same group of elements.

Magnesium.—1. Solution of ammonium carbonate produces a white precipitate of magnesium carbonate, dissolving in strong solution of ammonium chloride to form a double chloride of ammonium and magnesium. If the ammonium chloride solution is added first, no precipitate is thrown down.

2. Solution of sodium phosphate, Na_2HPO_4, in the presence of ammonium chloride and solution of ammonia, yields a white crystalline precipitate of ammonio-magnesium phosphate, $MgNH_4PO_4$. In very dilute solutions the precipitate only forms after violent shaking, or rubbing the inside of the test-tube with a glass rod.

3. It is well known that the arsenates are isomorphous with the phosphates, and if ammonium arsenate, $(NH_4)_2HAsO_4$, is employed in the reaction described in No. 2 in place of sodium phosphate, a crystalline precipitate of ammonio-magnesium arsenate forms ($MgNH_4AsO_4$).

4. Solutions of the hydroxides of potassium, sodium, ammonium, barium, or calcium, produce a white precipitate of magnesium hydrate, $Mg(OH)_2$, insoluble in excess of the reagent, but readily soluble in solution of ammonium chloride.

Mercury.—Reactions common to mercurous and mercuric salts.—1. Hydrogen sulphide yields a black precipitate of mercury sulphide, insoluble in ammonium hydrosulphide or in hot diluted nitric acid.

2. Copper-foil immersed in a solution free from *excess* of nitric acid becomes coated with a deposit of mercury, which on rubbing becomes bright, and from which the mercury may be volatilised and obtained in globules by heating in a dry test-tube.

3. Solution of stannous chloride, $SnCl_2$, reduces mercury salts to metallic mercury. Mercuric salts are first reduced to the mercurous state, and these reacting with more of the reagent cause a metallic deposit.

Reactions characteristic of mercurous salts.—1. Hydrochloric acid affords a white precipitate of mercurous chloride, Hg_2Cl_2, turning to black mercurous ammonium chloride, NH_2Hg_2Cl, on the addition of solution of ammonia.

2. Solution of potassium hydroxide produces a black precipitate of mercurous oxide, Hg_2O, and solution of ammonia a black precipitate of a mercurous amido-salt similar in constitution to that formed in test No. 1.

3. Solution of potassium iodide gives a green precipitate of mercurous iodide, Hg_2I_2, soluble in excess of the reagent.

Reactions characteristic of mercuric salts.—1. Solution of ammonia affords a white precipitate of a mercuric amido-salt, of which the official "white precipitate" is an example, NH_2HgCl.

2. Solution of potassium hydroxide yields a yellow precipitate of mercuric oxide, HgO.

3. Solution of potassium iodide produces a scarlet precipitate of mercuric iodide, HgI_2, soluble in excess of either the reagent or the mercuric salt. When the precipitate of mercuric iodide first forms it has a distinct yellowish tinge, but rapidly turns scarlet.

Nitrates.—1. If a few drops of a solution containing a nitrate be mixed with a solution of ferrous sulphate, and then strong sulphuric acid be cautiously poured down the side of the test-tube, held in a sloping position so as to fall to the bottom without mixing with the solution, a brown-coloured ring is formed at the junction of the two liquids. The sulphuric acid acting upon the nitrate liberates nitric acid ; this is reduced by the ferrous sulphate to nitric oxide, NO, which dissolving in the ferrous sulphate forms an unstable compound of ferrous sulphate and nitric oxide, $2FeSO_4.NO$. On warming the colour disappears, nitric oxide being evolved, thus :

$$2KNO_3 + 5H_2SO_4 + 6FeSO_4 =$$
$$2KHSO_4 + 3Fe_23SO_4 + 4H_2O + 2NO$$

2. Nitrates liberate red fumes when warmed with sulphuric acid and metallic copper, thus :

$$3Cu + 8HNO_3 = 3Cu(NO_3)_2 + 4H_2O + 2NO.$$
$$\text{Then } 2NO + O_2 = 2NO_2.$$

3. The following is not included in the pharmacopœial tests for the presence of nitrates, but it is nevertheless one of the

most delicate, and distinguishes, moreover, between nitrates and nitrites:

Prepare some phenol-sulphonic acid, $C_6H_4(OH).SO_3H$, by dissolving one part of crystallised phenol in four parts of strong sulphuric acid, and dilute with two parts of distilled water. A small quantity of a solution of a nitrate is evaporated to dryness in a clean porcelain capsule over a water-bath, two or three drops of phenol-sulphonic acid added, well stirred, and heated again for five minutes. On cooling dilute solution of ammonia is added until faintly alkaline, when a bright yellow solution results.

The excess of sulphuric acid in the reagent liberates nitric acid, which reacts with the phenol-sulphonic acid to form picric acid, $C_6H_2(NO_2)_3.OH$, and this on the addition of solution of ammonia yields yellow ammonium picrate, $C_6H_2(NO_2)_3.ONH_4$.

$$C_6H_4(OH).SO_3H + 3HO.NO_2 =$$
$$C_6H_2(NO_2)_3.OH + H_2SO_4 + 2H_2O.$$
$$C_6H_2(NO_2)_3.OH + NH_4OH = C_6H_2(NO_2)_3.ONH_4 + H_2O.$$

Nitrites.—1. On the addition of a few drops of diluted sulphuric acid with potassium iodide and mucilage of starch a deep blue colour is at once formed. The sulphuric acid liberates nitrous acid from the nitrite, and hydriodic acid from the iodide, and these two reacting yield nitric oxide and free iodine, thus:

$$2HNO_2 + 2HI = I_2 + 2H_2O + 2NO.$$

2. Dilute sulphuric acid liberates red fumes without the presence of metallic copper.

3. Nitrites give a dark brown coloration with ferrous sulphate on the addition of a *weak* acid. (Compare Nitrates.)

Oxalates.—1. Solution of calcium chloride affords a white precipitate of calcium oxalate, CaC_2O_4, soluble in hydrochloric, but insoluble in acetic acid.

2. Solution of silver nitrate yields a white precipitate, soluble in solution of ammonia and in dilute nitric acid.

3. Most oxalates are converted into carbonates on igniting. Oxalates do not char at all when heated with strong sulphuric acid, but decompose with evolution of carbonic oxide, CO, and carbonic anhydride, CO_2.

4. Oxalic acid discharges the colour of potassium permanganate

acidulated with dilute sulphuric acid. Oxalic acid also decomposes cupric sulphate, liberating sulphuric acid. (The instances in which a sulphate is decomposed by another acidulous radical are extremely rare.)

Phosphates.—1. Solution of ammonio-nitrate of silver yields a light yellow precipitate of silver phosphate, Ag_3PO_4, soluble both in solution of ammonia and in cold dilute nitric acid.

2. Solution of ferric chloride in the presence of ammonium acetate or other neutral acetate yields a whitish precipitate of ferric phosphate, $FePO_4$, which is insoluble in acetic acid.

3. "Magnesia mixture" (ammonio-magnesian sulphate) affords a white crystalline precipitate of ammonio-magnesian phosphate, $MgNH_4PO_4$, soluble in dilute acids. In very dilute solutions the precipitate appears very slowly, or only after vigorous shaking. Rubbing the sides of the test-tube with a glass rod very often induces precipitation when other methods fail.

4. A *large excess* of solution of ammonium molybdate in strong nitric acid produces on warming a yellow precipitate of ammonium phospho-molybdate, $2(NH_3)_3PO_4.22MoO_3.12H_2O$. This compound is insoluble in dilute mineral acids, but dissolves in excess of alkalies, phosphates, or phosphoric acid.

Potassium.—1. Solution of "platinic chloride," $PtCl_4$, affords with moderately strong solutions of potassium chloride (or with other potassium salts if free hydrochloric acid be present) a yellow crystalline precipitate of potassium-platinic chloride, insoluble in alcohol, $PtCl_4.2KCl$, which upon ignition leaves a residue of potassium chloride and platinum. The formation of this double chloride is understood when it is remembered that in solution of hydrochloric acid, platinum forms a double compound of platinum and hydrochloric acid, $PtCl_4.2HCl$, to which the name of chloroplatinic acid has been given, and the double salts of platinic chloride and various chlorides are regarded as salts of this acid.

2. Potassium compounds when moistened with hydrochloric acid communicate a violet colour to a colourless Bunsen flame when introduced on a loop of perfectly clean platinum wire.

Selenium and Tellurium.—1. Compounds of selenium and tellurium are only to be looked for in bismuth or its preparations.

2. To detect either element in bismuth, dissolve the metal in nitric acid, add to the liquid solution of ammonium chloride, and

dilute freely with water. This precipitates bismuth oxychloride. The filtrate from the above reaction is then mixed with excess of sodium sulphite, when, if even a trace of either element is present, a reddish or grey-coloured powder is precipitated after standing for about twelve hours.

Both selenium and tellurium form highly objectionable compounds with bismuth, and for medicinal use it is important that the latter should be absolutely free from any trace of either element.

Silica.—1. Silica, SiO_2, after exposure to a red heat is insoluble in all acids with the exception of hydrofluoric. It is not dissolved in a bead of "microcosmic salt" when heated to fusion in a blowpipe flame.

Microcosmic salt, hydrogen - sodium - ammonium phosphate, $HNa(NH_4)PO_4$, is obtained by adding a strong solution of common sodium phosphate, Na_2HPO_4, to solution of ammonium chloride.

$$Na_2HPO_4 + NH_4Cl = NaCl + HNa(NH_4)PO_4.$$

2. Silicon dioxide is soluble in a hot solution of the alkalies, and in boiling solution of sodium carbonate. Fused with sodium carbonate, silicon dioxide is converted into soluble sodium silicate.

$$SiO_2 + 2Na_2CO_3 = 2CO_2 + Si(NaO)_4.$$

Silver.—1. Hydrochloric acid and other soluble chlorides afford a white curdy precipitate of silver chloride, AgCl, instantly soluble in solution of ammonia, but insoluble in nitric acid. Silver chloride is also soluble in solution of potassium cyanide.

Sodium.—1. Sodium compounds when moistened with hydrochloric acid communicate a persistent yellow coloration when introduced on platinum wire into the colourless flame of a Bunsen burner.

Note.—Platinic chloride forms a double chloride of platinum and sodium, but the compound is soluble in both alcohol and water (distinction from potassium).

There are no characteristic reactions which can be applied for the detection of sodium, but if a solution gives no precipitate with any of the group reagents, but leaves on evaporation a fixed residue capable of imparting a strong yellow colour to a Bunsen flame, it is almost certain that sodium is present.

Starch.—1. When starch is boiled with water a mucilaginous liquid results, which *on cooling* strikes a deep blue colour with solution of iodine.

2. Boiled for some time with dilute hydrochloric acid and then neutralised with sodium hydroxide, a red precipitate of cuprous oxide is formed on warming with solution of potassio-cupric tartrate (Fehling's solution).

On boiling with dilute mineral acids, starch is first converted into soluble starch or dextrine, the empirical formula of which is $C_6H_{12}O_6$. On further boiling this is converted into dextrose, thus:

$$C_6H_{10}O_5 + H_2O = C_6H_{12}O_6.$$

Sulphates.—1. Solution of barium chloride affords a white precipitate of barium sulphate, insoluble in hydrochloric acid.

2. Heated with a little sodium carbonate on charcoal, in the inner (reducing) blowpipe flame sulphates are reduced to sulphides; and the residue, placed on a clear silver coin and moistened with water, leaves a black stain of silver sulphide.

Sulphides.—1. The official sulphides, hydrosulphides, and sulphurated compounds evolve hydrogen sulphide when boiled with strong hydrochloric acid. Hydrogen sulphide is recognised both by its disagreeable smell, and the fact that a slip of white filter-paper moistened with solution of lead acetate is rapidly blackened by the formation of lead sulphide.

Note.—Sulphonal and thiocyanates do not evolve hydrogen sulphide when heated with an acid. If fused with sodium carbonate mixed with a small proportion of potassium nitrate they afford a mass which, when extracted with water, answers to the tests for sulphates.

Sulphites.—1. Hydrochloric acid liberates sulphurous anhydride, SO_2, a colourless gas with a pungent smell, without deposition of sulphur (distinction from thiosulphates).

2. When sulphites are acted on by zinc and hydrochloric acid they evolve hydrogen sulphide. In this reaction the excess of hydrochloric acid liberates sulphurous acid from sulphites, which

is then attacked by the nascent hydrogen, forming hydrogen sulphide and water, thus :

$$Na_2SO_3 + 2HCl = 2NaCl + H_2SO_3.$$
$$H_2SO_3 + 3H_2 = H_2S + 3H_2O.$$

3. Sulphites decolourise solution of iodine according to the equation—

$$Na_2SO_3 + I_2 + H_2O = Na_2SO_4 + 2HI.$$

For an application of this reaction see Volumetric Estimation of Sulphites.

Tartrates.—1. Tartrates on the application of heat char very readily, giving off the odour of burnt sugar, generally leaving the metal as either carbonate or oxide.

2. Solution of calcium chloride added in excess to a *neutral* tartrate affords a white granular precipitate of calcium tartrate, soluble when freshly precipitated in cold moderately concentrated solution of potassium hydroxide, from which solution it is again precipitated on boiling.

Note.—If the precipitate of calcium tartrate is separated by filtration and washed with a little water, it dissolves quite readily in solution of potassium hydroxide. Calcium tartrate is also soluble in tartaric acid.

3. Solution of silver nitrate affords a white precipitate of silver tartrate, soluble in solution of ammonia and in nitric acid. If the ammoniacal solution is gently heated in a perfectly clean test-tube, a mirror of metallic silver is formed on the sides of the tube (distinction from citrates). Silver tartrate is soluble in solution of ammonia, but on heating the mixture blackens, owing to the formation of silver oxide; and this under the influence of heat gives off oxygen, leaving metallic silver, which deposits as a mirror on the glass.

4. A concentrated solution of potassium acetate gives a white precipitate of hydrogen-potassium tartrate in moderately concentrated solutions when acidulated with acetic acid and well stirred. If the solutions are very dilute a large proportion of 90 per cent. alcohol should be added :

$$KC_2H_3O_2 + Na_2C_4H_4O_6 + HC_2H_3O_2$$
$$= KHC_4H_4O_6 + 2NaC_2H_3O_2.$$

Tellurium.—See Selenium.

Thiosulphates.—1. Hydrochloric acid decomposes thiosulphates, precipitating sulphur and liberating sulphurous anhydride.

$$Na_2S_2O_3.5H_2O + 2HCl = 2NaCl + 6H_2O + SO_2 + S.$$

2. When thiosulphates are acted upon by hydrochloric acid and zinc, hydrogen sulphide is liberated.

3. Thiosulphates decolourise solution of iodine, sodium tetrathionate and sodium iodide being formed. For an application of the reaction, see Volumetric Analysis, Estimation of Iodine.

Tin.—1. Metallic zinc placed in a solution of a salt of tin acidified with hydrochloric acid precipitates the whole of the tin in metallic scales, or in the form of a grey sponge.

2. Metallic tin separated in the preceding is soluble in boiling concentrated hydrochloric acid, and the solution, which contains stannous chloride, $SnCl_2$, gives with solution of mercuric chloride a white precipitate of calomel, Hg_2Cl_2, which becomes grey from separation of globules of mercury if excess of tin salt is present, thus :

$$2HgCl_2 + SnCl_2 = Hg_2Cl_2 + SnCl_4.$$
$$Hg_2Cl_2 + SnCl_2 = 2Hg + SnCl_4.$$

Zinc.—1. Solution of ammonium hydrosulphide gives a white precipitate of zinc sulphide, ZnS, soluble in dilute hydrochloric acid, but insoluble in acetic acid.

2. In alkaline solutions the same result is produced by hydrogen sulphide.

3. Solution of potassium hydroxide affords a white precipitate of zinc hydrate, $Zn(OH)_2$, soluble in excess to form potassium zincate (distinction from aluminium).

4. Solution of potassium ferrocyanide produces a white precipitate of zinc ferrocyanide, $Zn_2FeC_6N_6$, insoluble in dilute hydrochloric acid.

Note.—Magnesium salts are not precipitated either by ammonium hydrosulphide or by potassium ferrocyanide, so that either of these latter serve to distinguish zinc sulphate from magnesium sulphate, with which it is isomorphous.

PART V

TABLES

CHAPTER LV

TABLES

Table of Atomic Weights of Elements

Name.	Symbol.	Atomic weight.	Name.	Symbol.	Atomic weight.
Aluminium	Al	26·90	Iron	Fe	55·60
Antimony	Sb	119·00	Lead	Pb	205·35
Arsenium	As	74·50	Lithium	Li	6·97
Barium	Ba	136·40	Magnesium	Mg	24·18
Bismuth	Bi	207·30	Manganese	Mn	54·52
Boron	B	10·85	Mercury	Hg	198·80
Bromine	Br	79·35	Nitrogen	N	13·94
Calcium	Ca	39·71	Oxygen	O	15·88
Carbon	C	11·91	Phosphorus	P	30·80
Cerium	Ce	139·20	Platinum	Pt	193·30
Chlorine	Cl	35·19	Potassium	K	38·83
Chromium	Cr	51·74	Silver	Ag	107·11
Copper	Cu	63·12	Sodium	Na	22·88
Gold	Au	195·70	Sulphur	S	31·82
Hydrogen	H	1·00	Tin	Sn	118·20
Iodine	I	125·90	Zinc	Zn	64·91

Table of Molecular Weights and Formulæ of the Principal Chemicals and Reagents used in Pharmacy

Name.	Formula.	Molecular weight.
Acetanilid	$CH_3.CO.NH.C_6H_5$	134·10
Acid, acetic	$CH_3.COOH$	59·58
,, arsenious	As_2O_3	196·64
,, benzoic	$C_6H_5.COOH$	121·13
,, boric	H_3BO_3	61·49
,, carbolic	$C_6H_5.OH$	93·34
,, chromic	CrO_3	99·38
,, citric	$C_3H_4.OH(COOH)_3.H_2O$	208·50
,, gallic	$C_6H_2(OH)_3.COOH.H_2O$	186·65
,, hydriodic	HI	126·90
,, hydrobromic	HBr	80·35
,, hydrochloric	HCl	36·19
,, hydrocyanic	HCN	26·85
,, hypophosphorous	HPH_2O_2	65·56
,, lactic	$CH_3.CH(OH).COOH$	89·37

Name.	Formula.	Molecular weight.
Acid, nitric	HNO_3	62·58
,, oleic	$HC_{18}H_{33}O_2$	280·14
,, oxalic	$COOH.COOH.2H_2O$	125·10
phosphoric	H_3PO_4	97·32
,, picric	$C_6H_2(NO_2)_3OH$	227·44
,, salicylic	$C_6H_4.OH.COOH$	137·01
,, sulphuric	H_2SO_4	97·34
,, sulphurous	H_2SO_3	81·46
,, tannic	$HC_{14}H_9O_9,2H_2O$	355·42
,, tartaric	$COOH.CH(OH).CH(OH).COOH$	148·92
Alcohol, ethylic	$CH_3.CH_2.OH$	45·70
,, methylic	$CH_3.OH$	31·79
Aldehyde, ethylic	$CH_3.CHO$	43·70
Alum, ammonia	$Al_2(NH_4)_2.(SO_4)_4.24H_2O$	900·16
,, potash	$Al_2K_2(SO_4)_4.24H_2O$	941·94
Ammonia	NH_3	16·94
Ammonium acetate	$NH_4C_2H_3O_2$	76·52
,, benzoate	$NH_4C_7H_5O_2$	138·07
,, bromide	NH_4Br	97·29
,, carbonate, normal	$(NH_4)_2CO_3$	95·43
,, ,, B.P.	$NH_4HCO_3,NH_4.NH_2CO_2$	156·04
,, chloride	NH_4Cl	53·13
,, citrate	$(NH_4)_3C_6H_5O_7$	241·44
,, iodide	NH_4I	143·84
,, nitrate	NH_4NO_3	79·52
,, oxalate	$(NH_4)_2C_2O_4.H_2O$	141·10
,, phosphate	$(NH_4)_2HPO_4$	131·20
,, sulphate	$(NH_4)_2SO_4$	131·22
,, sulphydrate	NH_4HS	50·76
Amyl nitrite	$C_5H_{11}NO_2$	116·25
Antimony and potassium tartrate	$[K(SbO)C_4H_4O_6]_2.H_2O$	659·14
,, sulphide	Sb_2S_3	333·46
,, trioxide	Sb_2O_3	285·64
Apomorphine hydrochloride	$C_{17}H_{17}NO_2.HCl$	301·36
Arsenium iodide	AsI_3	452·20
Atropine	$C_{17}H_{23}NO_3$	287·05
,, sulphate	$(C_{17}H_{23}NO_3)_2H_2SO_4$	671·44
Barium carbonate	$BaCO_3$	195·95
,, chloride	$BaCl_2.2H_2O$	242·54
,, dioxide	BaO_2	168·16
,, hydroxide	$Ba(OH)_2$	170·16
,, nitrate	$Ba(NO_3)_2$	259·56
Benzol (benzene)	C_6H_6	77·46
Bismuth citrate	$BiC_6H_5O_7$	394·92
,, oxide	Bi_2O_3	462·64
,, oxycarbonate	$(Bi_2O_2CO_3)_2H_2O$	1029·70
,, oxynitrate	$BiONO_3.H_2O$	302·64
,, oxysalicylate	$C_6H_4.OH.COO.BiO$	359·19
Borax	$Na_2B_4O_7.10H_2O$	379·12
Butyl-chloral hydrate	$CH_3.CHCl.CCl_2.CH(OH)_2$	191·97

Name.	Formula.	Molecular weight.
Caffeine	$C_8H_{10}N_4O_2.H_2O$	210·68
Calcium carbonate	$CaCO_3$	99·26
,, chloride	$CaCl_2.2H_2O$	145·85
,, hydroxide	$Ca(OH)_2$	73·47
,, hypophosphite	$Ca(H_2PO_2)_2$	168·83
,, oxide	CaO	55·59
,, phosphate	$Ca_3(PO_4)_2$	307·77
,, sulphate	$CaSO_4.4H_2O$	206·57
,, sulphide	CaS	71·53
Camphor	$C_{10}H_{16}O$	150·98
Carbon disulphide	CS_2	75·55
Cerium oxalate	$Ce_2(C_2O_4)_3.9H_2O$	701·34
Chloral hydrate	$CCl_3.CH(OH)_2$	164·15
Chloroform	$CHCl_3$	118·48
Cocaine	$C_{17}H_{21}NO_4$	300·93
,, hydrochloride	$C_{17}H_{21}NO_4.HCl$	337·12
Codeine	$C_{18}H_{21}NO_3.H_2O$	314·84
,, phosphate	$(C_{18}H_{21}NO_3.H_3PO_4)_2.3H_2O$	842·20
Copper sulphate	$CuSO_4.5H_2O$	247·86
Ethyl acetate	$C_2H_5C_2H_3O_2$	87·40
,, nitrite	$C_2H_5NO_2$	74·52
,, oxide	$(C_2H_5)_2O$	73·52
Eucalyptol	$C_{10}H_{18}O$	152·98
Ferric acetate	$Fe_2(C_2H_3O_2)_6$	462·68
,, chloride	$Fe_2Cl_6.12H_2O$	536·90
,, hydroxide	$Fe_2(OH)_6$	212·48
,, hypophosphite	$Fe_2(PH_2O_2)_6$	498·56
,, nitrate	$Fe_2(NO_3)_6$	480·68
,, oxide	Fe_2O_3	158·84
,, sulphate	$Fe_2(SO_4)_3$	397·22
Ferrous bromide	$FeBr_2$	214·30
,, carbonate	$FeCO_3$	115·15
,, iodide	FeI_2	307·40
,, sulphate	$FeSO_4.7H_2O$	276·10
,, sulphide	FeS	87·42
Glycerin	$C_3H_5(OH)_3$	91·37
Glyceryl trinitrate	$C_3H_5(NO_3)_3$	225·47
Gold chloride	$AuCl_3$	301·27
Hydrogen dioxide	H_2O_2	33·76
,, sulphide	H_2S	33·82
Iodoform	CHI_3	390·61
Lead acetate	$Pb(C_2H_3O_2)_2.3H_2O$	376·15
,, nitrate	$Pb(NO_3)_2$	328·51
,, oxide	PbO	221·23
,, oxyacetate (approx.)	$Pb_2O(C_2H_3O_2)_2$	543·74
,, oxycarbonate	$(PbCO_3)_2.Pb(OH)_2$	768·91
Lithium benzoate	$LiC_7H_5O_2$	127·10
,, bromide	$LiBr$	86·32
,, carbonate	Li_2CO_3	73·49
,, citrate	$Li_3C_6H_5O_7.4H_2O$	280·05

Name.	Formula.	Molecular weight.
Lithium salicylate	$LiC_7H_5O_3$	141·98
Magnesium oxide	MgO	40·06
,, oxycarbonate (approx.)	$(MgCO_3)_3.Mg(OH)_2.4H_2O$	380·65
,, sulphate	$MgSO_4.7H_2O$	244·68
Manganese dioxide	MnO_2	86·28
Menthol	$C_{10}H_{19}.OH$	154·98
Mercuric ammonium chloride	NH_2HgCl_2	249·93
,, chloride	$HgCl_2$	269·18
,, iodide	HgI_2	450·60
,, nitrate	$Hg(NO_3)_2$	321·96
,, oxide	HgO	214·68
Mercurous chloride	Hg_2Cl_2	467·98
,, iodide	Hg_2I_2	649·40
,, nitrate	$Hg_2(NO_3).2H_2O$	556·52
Morphine	$C_{17}H_{19}NO_3.H_2O$	300·93
,, acetate	$C_{17}H_{19}NO_3.C_2H_3O_2.3H_2O$	396·27
,, hydrochloride	$C_{17}H_{19}NO_3.HCl.3H_2O$	372·88
,, sulphate	$(C_{17}H_{19}NO_3)_2H_2SO_4.5H_2O$	752·84
,, tartrate	$(C_{17}H_{19}NO_3)_2H_2C_4H_4O_6.3H_2O$	768·66
Naphthalin	$C_{10}H_8$	127·10
Paraldehyde	$C_6H_{12}O_3$	131·10
Physostigmine sulphate	$(C_{15}H_{21}N_3O_2)_2H_2SO_4$	643·80
Pilocarpine hydrochloride	$C_{11}H_{16}N_2O_2.HCl$	235·74
Platinic chloride	$PtCl_4$	334·06
Potassium acetate	$KC_2H_3O_2$	97·41
,, benzoate	$KC_7H_5O_2.3H_2O$	212·60
,, bicarbonate	$KHCO_3$	99·38
,, bitartrate	$KHC_4H_4O_6$	186·75
,, bromide	KBr	118·18
,, carbonate	K_2CO_3	137·21
,, chlorate	$KClO_3$	121·66
,, chloride	KCl	74·02
,, chromate	K_2CrO_4	192·92
,, citrate	$K_3C_6H_5O_7.H_2O$	321·99
,, cyanide	KCN	64·68
,, dichromate	$K_2Cr_2O_7$	292·30
,, ferricyanide	$K_6Fe_2(CN)_{12}$	654·18
,, ferrocyanide	$K_4Fe(CN)_6.3H_2O$	416·66
,, hydroxide	KOH	55·71
,, iodide	KI	164·73
,, nitrate	KNO_3	100·41
,, permanganate	$KMnO_4$	156·87
,, phosphate	K_2HPO_4	173·98
,, salicylate	$KC_7H_5O_3$	174·84
,, and sodium tartrate	$KNaC_4H_4O_6.4H_2O$	280·15
,, sulphate	K_2SO_4	173·00
,, tartrate	$(K_2C_4H_4O_6)_2.H_2O$	467·04
Quinine	$C_{20}H_{24}N_2O_2.3H_2O$	375·48
,, acid hydrochloride	$C_{20}H_{24}N_2O_2.2HCl.3H_2O$	411·67
,, bisulphate	$C_{20}H_{24}N_2O_2.H_2SO_4.7H_2O$	544·34

Name.	Formula.	Molecular weight.
Quinine hydrobromide	$C_{20}H_{24}N_2O_2.HBr.H_2O$	420·07
,, hydrochloride	$C_{20}H_{24}N_2O_2.HCl.2H_2O$	393·79
,, sulphate	$(C_{20}H_{24}N_2O_2)_2H_2SO_4.7H_2O$	875·12
Resorcin	$C_6H_4(OH)_2$	109·22
Salicin	$C_{13}H_{18}O_7$	283·99
Santonin	$C_{15}H_{18}O_3$	244·29
Silver cyanide	$AgCN$	132·96
,, nitrate	$AgNO_3$	168·69
,, oxide	Ag_2O	130·10
,, sulphate	Ag_2SO_4	309·56
Sodium acetate	$NaC_2H_3O_2.3H_2O$	135·10
,, arsenate	$Na_2HAsO_4.7H_2O$	309·94
,, arsenite (meta)	$NaAsO_2$	129·14
,, benzoate	$NaC_7H_5O_2$	143·01
,, bicarbonate	$NaHCO_3$	83·43
,, bisulphite	$NaHSO_3$	101·34
,, bromide	$NaBr$	102·23
,, carbonate	$Na_2CO_3.10H_2O$	284·11
,, chloride	$NaCl$	58·07
,, citrate	$(Na_3C_6H_5O_7)_2.11H_2O$	709·20
,, hydroxide	$NaOH$	39·76
,, hypophosphite	$NaPH_2O_2$	87·44
,, hyposulphite	$Na_2S_2O_3.5H_2O$	246·44
,, iodide	NaI	148·78
,, nitrate	$NaNO_3$	84·46
,, nitrite	$NaNO_2$	68·58
,, phosphate	$Na_2HPO_4.12H_2O$	355·64
,, salicylate	$NaC_7H_5O_3$	158·89
,, sulphate	$Na_2SO_4.10H_2O$	319·90
,, sulphite	$Na_2SO_3.7H_2O$	250·38
,, sulphocarbolate	$NaSO_3C_6H_4.OH.2H_2O$	230·44
Stannous chloride	$SnCl_2.2H_2O$	224·34
Strychnine	$C_{21}H_{22}N_2O_2$	331·75
Sugar, cane	$C_{12}H_{22}O_{11}$	339·60
,, grape	$C_6H_{12}O_6$	178·74
,, milk	$C_{12}H_{22}O_{11}.H_2O$	357·48
Sulphonal	$(CH_3)_2.C.(SO_2C_2H_5)_2$	226·53
Terebene	$C_{10}H_{16}$	135·10
Thymol	$C_{10}H_{14}O$	148·98
Zinc acetate	$Zn(C_2H_3O_2)_2.2H_2O$	217·83
,, bromide	$ZnBr_2$	223·61
,, carbonate	$ZnCO_3$	124·46
,, chloride	$ZnCl_2$	135·29
,, iodide	ZnI_2	316·71
,, oxide	ZnO	80·79
,, phosphide	Zn_3P_2	256·33
,, sulphate	$ZnSO_4.7H_2O$	285·41
,, valerianate	$Zn(C_5H_9O_2)_2$	265·53

*Table of the more Potent Chemical Substances, with their Preparations, Strengths, and Doses.**

Pharmacopœial name.	Dose.		Preparations.	Strength.	Dose.	
	Apothecaries' denomination.	Metric denomination (approximate).		Metric denomination.	Apothecaries' denomination.	Metric denomination.
Acetanilid	1 — 3 gr.	·06 — ·2 gram				
Acid. Arseniosum	$\frac{1}{60}$ — $\frac{1}{15}$,,	·001 — ·004 ,,	Liquor Arsenicalis	1 part in 100 fl. pts.	2—8 ♏	·1 — ·5 c.c.
			,, Arsenici Hydrochloricus ...	1 ,, in 100 ,,	2—8 ♏	·1 — ·5 ,,
,, Carbolicum	1 — 3 ,,	·06 — ·2 ,,	Acid. Carbolicum Liquidum ...	90·9 pts. in 100 ,,	1—3 ♏	·06— ·2 ,,
			Glycerinum Acidi Carbolici ...	20 ,, in 100 ,,		
			Suppositoria ,, ,, ...	1 grain in each		
			Unguentum ,, ,, ...	4 pts. in 100 pts.		
,, Hydrocyanic. Dil. ...	2 — 6 ♏	·1 — ·4 c.c.	Tr. Chloroformi et Morphinæ ...	5 ,, in 100 fl. pts. of the diluted acid	5—15 ♏	·3 — 1·0 c.c.
Amyl Nitris	2 — 5 ♏	·1 — ·3 ,,	by inhalation.			
Antimonii Oxidum ...	1 — 2 gr.	·06 — ·1 gram	Pulvis Antimonialis	33·$\dot{3}$ pts. in 100 pts.	3—6 gr.	·2 — ·4 gram
,, Tartaratum ...	$\frac{1}{24}$ — $\frac{1}{8}$,,	·002 — ·008 ,,	Vinum Antimoniale	·45 ,, 100 fl. pts.	10—30 ♏	·6 — 2·0 c.c.
,, ,, ...	1 — 2 ,, as an emetic	·06 — ·1 ,,	,, ,, ...	—	2—4 fl. dr. as an emetic	7·0 —14·0 ,,
Apomorphinæ Hydrochlor.	$\frac{1}{10}$ — $\frac{1}{4}$ gr.	·005 — ·002 ,,	Injectio Apomorphinæ Hydrochloridi	1 pt. in 100 fl. pts.	5—10 ♏ by injection	·3 — ·6 ,,
Argenti Nitras	$\frac{1}{4}$ — $\frac{1}{2}$,,	·015 — ·03 ,,	Argenti Nitras Induratus	95 pts. in 100 pts.		
			,, ,, Mitigatus	33$\frac{1}{3}$,, in 100 ,,		
,, Oxidum	1 — 2 ,,	·06 — ·1 ,,				
Arsenii Iodidum	$\frac{1}{20}$ — $\frac{1}{5}$,,	·003 — ·01 ,,	Liq. Arsenii et Hydrargyri Iodidi ...	1 pt. in 100 fl. pts.	5—20 ♏	·3 — 1·2 c.c.
Atropina	$\frac{1}{200}$—$\frac{1}{100}$,,	·0003— ·0006 ,,	Unguentum Atropinæ	2 pts. in 100 pts.		
Atropinæ Sulphas... ...	$\frac{1}{200}$—$\frac{1}{100}$,,	·0003— ·0006 ,,	Liquor Atropinæ Sulphatis	1 pt. in 100 fl. pts.	$\frac{1}{2}$—1 ♏	·03— ·06 ,,
			Lamellæ Atropinæ	$\frac{1}{5000}$ grain in each.		
Butyl Chloral Hydras ...	5 —20 ,,	·3 —1·2 ,,				
Chloral Hydras	5 —20 ,,	·3 —1·2 ,,	Syrupus Chloral	18·25 pts. in 100 fl. pts.	1—2 fl. dr.	3·5 — 7·0 ,,
Chloroformum	1 — 5 ♏	·06 — ·3 c.c.	Aqua Chloroformi	·25 vol. in 100 vols.		
			Spiritus ,, ...	5 ,, in 100 ,,	5—20 ♏	·3 — 1·2 ,,
			Linimentum ,, ...	50 ,, in 100 ,,		
			Tinct. Chlorof. et Morphinæ ...	7·5 ,, in 100 ,,	5—15 ♏	·3 — 1·0 ,,
Cocainæ Hydrochloridum ...	$\frac{1}{5}$ — $\frac{1}{2}$ gr.	·01 — ·03 gram	Injectio Cocainæ Hypodermica ...	10 pts. in 100 fl. pts.	2—5 ♏ by injection	·1 — ·3 ,,
			Lamellæ ,, (Hydrochloridi) ...	$\frac{1}{50}$ grain in each.		
Codeina	$\frac{1}{4}$ — 2 ,,	·01 — ·1 ,,				
Codeinæ Phosphas ...	$\frac{1}{4}$ — 2 ,,	·01 — ·1 ,,	Syrupus Codeinæ	$\frac{1}{4}$ grain in 1 fl. dr.	$\frac{1}{2}$ –2 fl. dr.	2·0 — 8·0 ,,

Cupri Sulphas	$\frac{1}{4}$ — 2 gr.	·01 —·1 gram	5—10 grs. are given as an emetic.			
Elaterium	$\frac{1}{10}$ — $\frac{1}{2}$,,	006 —·03 ,,	Elaterinum	—	$\frac{1}{40}$—$\frac{1}{10}$ gr.	·0015—·006 gm.
Ferri Arsenas	$\frac{1}{16}$ — $\frac{1}{4}$,,	·004 —·015 ,,				
Homatropinæ Hydrobrom.	$\frac{1}{80}$ — $\frac{1}{20}$,,	·001 —·003 ,,	Lamellæ Homatropinæ	$\frac{1}{100}$ grain in each.		
Hydrargyri Iodid. Rubrum	$\frac{1}{32}$ — $\frac{1}{16}$,,	·002 —·004 ,,	Liq. Arsenii et Hydrargyri Iodidum	1 pt. in 100 fl. pts.	10—30 ♏	·6 —2·0 c.c.
			Ung. Hydrargyri Iodidi	4 pts. in 100 pts.		
,, Perchloridum .	$\frac{1}{32}$ — $\frac{1}{16}$,,	·002 —·004 ,,	Liquor ,, Perchloridi ...	Rather more than $\frac{1}{10}$ per cent.	$\frac{1}{2}$—1 fl. dr.	2·0 —4·0 ,,
			Lotio ,, Flava	·46 in 100.		
,, Subchloridum .	$\frac{1}{2}$ — 5 ,,	·03 —·3 ,,	Pilula ,, Subchlor. Comp....	About 25 pts. in 100 pts.	4—8 gr.	·25— ·5 gram.
Hyoscinæ Hydrobromas ...	$\frac{1}{200}$—$\frac{1}{100}$,,	·0003—·0006 ,,				
Hyoscyaminæ Sulphas ...	$\frac{1}{200}$—$\frac{1}{100}$,,	·0003—·0006 ,,				
Iodoformum	$\frac{1}{2}$ — 3 ,,	·03 —·2 ,,	Unguentum Iodoformi	10 pts. in 100 pts.		
			Suppositoria ,, ...	3 grains in each.		
Iodum	—	—	Tinctura Iodi	2·5 pts. in 100 fl. pts.	2—5 ♏	·1 — ·3 c.c.
			Liquor Iodi Fortis	About 10 pts. in 100 fl. pts.		
			Unguentum Iodi	4 pts. in 100 pts.		
Morphinæ Acetatis ...	$\frac{1}{8}$ — $\frac{1}{2}$,,	·01 —·03 ,,	Liquor Morphinæ Acetatis	1 pt. in 100 fl. pts.	10—60 ♏	·6 —3·5 ,,
,, Hydrochloridum	$\frac{1}{8}$ — $\frac{1}{2}$,,	·01 —·03 ,,	,, ,, Hydrochloridi ...	1 ,, in 100 ,,	10—60 ♏	·6 —3·5 ,,
			Troch. ,, ...	$\frac{1}{36}$ grain in each.		
			,, ,, et Ipecacuanhæ ...	$\frac{1}{36}$ grain in each.		
			Tinct. Chlorof. et Morphinæ ...	About 1 pt. in 100 fl. pts.	5—15 ♏	·3 —1·0 ,,
			Suppositoria Morphinæ	$\frac{1}{4}$ grain in each.		
, Tartras ...	$\frac{1}{8}$ — $\frac{1}{2}$,,	·01 —·03 ,,	Liquor ,, Tartratis ...	1 pt. in 100 fl. pts.	10—60 ♏	·6 —3·5 ,,
			Injectio ,, Hypodermica	5 pts. in 100 ,,	2—5 ♏ by subcut.	·1 — ·3 ,,
Phosphorus	$\frac{1}{100}$—$\frac{1}{20}$,,	·0006—·003 ,,	Pilula Phosphori	2 ,, in 100 pts.	1—2 gr.	·06— ·1 gram.
			Oleum Phosphoratum	1 pt. in 100 ,,	1—5 ♏	·06— ·3 c.c.
Physostigminæ Sulphas ...	$\frac{1}{60}$ — $\frac{1}{20}$,,	·001 —·003 ,,	Lamellæ Physostigminæ	$\frac{1}{1000}$ grain in each.		
Picrotoxinum	$\frac{1}{100}$—$\frac{1}{25}$,,	·0006—·0025 ,,				
Pilocarpinæ Nitras ...	$\frac{1}{20}$ — $\frac{1}{2}$,,	·003 —·03 ,,				
Plumbi Acetas	1 — 5 ,,	·06 —·3 ,,	Unguentum Plumbi Acetas... ...	4 pts. in 100 pts.		
			Suppositoria ,, c̄ Opio... ...	3 grains in each.		
			Pilula ,, ,, ...	3 grains in 4.		
Sodii Arsenas	$\frac{1}{40}$ — $\frac{1}{10}$,,	·001 —·006 ,,	Liquor Sodii Arsenatis	1 pt. in 100 fl. pts.	2—8 ♏	·1 — ·5 ,,
Strychnina	$\frac{1}{60}$ — $\frac{1}{15}$,,	·001 —·004 ,,				
,, Hydrochloridum	$\frac{1}{60}$ — $\frac{1}{15}$,,	·001 —·004 ,,	Liquor Strychninæ Hydrochloridi...	1 ,, in 100 ,,	2—8 ♏	·1 — ·5 ,,
Zinci Acetas	1 — 2 ,,	·06 —·1 ,,				
,, Sulphas	1 — 3 ,,	·06 —·2 ,,	as an emetic 10—30 grs.			

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* 1 grain equals approximately 64 milligrams (·064 gram), or 1 milligram equals $\frac{1}{64}$ grain.

Table of the more Potent Vegetable Substances, with their Preparations, Strengths, and Doses *

Name.	Preparations.	Strength.	Dose.	
			Apothecaries' denomination.	Metric denomination (approximate).
Aconiti Radix	Tinctura Aconiti	5 parts in 100 fl. parts	5—15 ♏	·3 — 1·0 c.c.
	Linimentum Aconiti	2 ,, 3 ,,		
Belladonnæ Folia	Extractum Belladonnæ Viride	...	¼— 1 grain	·015— ·06 gram
	Succus ,,	...	5—15 ♏	·3 — 1·0 c.c.
,, Radix	Extractum ,, Liquidum	0·75 part total alkaloid in 100 fl. parts.		
	,, ,, Alcoholicum	1·00 ,, ,, 100 parts	¼— 1 grain	·015— ·06 gram
	Linimentum ,,	0·385 ,, ,, 100 fl. parts.		
	Tinctura ,,	0·1 ,, ,, 100 ,,	5—15 ♏	·3 — 1·0 c.c.
	Emplastrum ,,	0·5 ,, ,, 100 ,,		
Cannabis Indica	Extractum Cannabis Indicæ	...	¼— 1 grain	·015— ·06 gram
	Tinctura ,, ,,	5 parts of the extract in 100 fl. parts	5—15 ♏	·3 — 1·0 c.c.
Colchici Cormus	Extractum Colchici	...	¼— 1 grain	·015— ·06 gram
	Vinum ,,	20 parts in 100 fl. parts	10—30 ♏	·6 — 2·0 c.c.
,, Semina	Tinctura ,,	20 ,, 100 ,,	5—15 ♏	·3 — 1·0 ,,
Digitalis Folia	Infusum Digitalis	3 grains in 1 fl. ounce	2— 4 fl. drachms	7·0 —14·0 ,,
	Tinctura ,,	12·5 parts in 100 fl. parts	5—15 ♏	·3 — 1·0 ,,
Ergota	Extractum Ergotæ	...	2— 8 grains	·1 — ·5 gram
	,, ,, Liquidum	1 part in 1 fl. part	10—30 ♏	·6 — 2·0 c.c.
	Infusum ,,	5 parts in 100 fl. parts	1— 2 fl. ounces	25·0 —50·0 ,,
	Tinctura ,, Ammoniata	25 ,, 100 ,,	½— 1 fl. drachm	2·0 — 3·5 ,,
Gelsemii Radix	,, Gelsemii	10 ,, 100 ,,	5—15 ♏	·3 — 1·0 ,,

Ipecacuanhæ Radix	Extractum Ipecacuanhæ Liquidum	2 parts total alkaloids in 100 fl. parts	½— 2 ♏	·03 — ·1 „
	Acetum „	0·1 part „ 100 „	10—30 ♏	·6 — 2·0 „
Lobelia	Tinctura Lobeliæ Ætherea	20 parts in 100 fl. parts	5—15 ♏	·3 — 1·0 „
Nucis Vomicæ Semina	Extractum Nucis Vomicæ	5 parts of strychnia in 100 parts	¼— 1 grain	·015— ·06 gram
	„ „ Liquidum	1·5 „ „ 100 fl. parts	1— 3 ♏	·06 — ·2 c.c.
	Tinctura „	0·25 part „ 100 „	5—15 ♏	·3 — 1·0 „
Opium, in powder	...	Contains when dry 10 per cent. of anhydrous morphia	½— 2 grains	·03 — ·1 gram
	Emplastrum Opii	Contains 1 part of morphia in 100 parts.		
	Extractum „	„ 20 parts „ 100 „	¼— 1 grain	·015— ·06 „
	„ „ Liquidum	„ 0·75 part „ 100 fl. parts	5—30 ♏	·3 — 2·0 c.c.
	Linimentum „	„ 0·375 „ „ 100 „		
	Tinctura „	„ 0·75 „ „ 100 „	5—15 ♏	·3 — 1·0 „
	„ „ Ammoniata	„ 0·10 „ „ 100 „	½— 1 fl. drachm	2·0 — 3·5 „
	„ Camphoræ Compositus	„ 0·05 „ „ 100 „	½— 1 „	2·0 — 3·5 „
	Pilula Ipecacuanhæ cum Scillâ	„ 0·50 „ „ 100 parts	4— 8 grains	·25 — ·5 gram
	„ Plumbi cum Opio	„ 1·25 „ „ 100 „	2— 4 „	·1 — ·25 „
	„ Saponis Compositus	„ 2·0 „ „ 100 „	2— 4 „	·1 — ·25 „
	Pulvis Cretæ Aromaticus cum Opio	„ 0 25 „ „ 100 „	10—40 „	·6 — 2·5 „
	„ Ipecacuanhæ Compositus	„ 1·00 „ „ 100 „	5—15 „	·3 — 1·0 „
	„ Kino „	„ 0·50 „ „ 100 „	5—20 „	·3 — 1·2 „
	„ Opii „	„ 0·10 „ „ 100 „	2—10 „	·1 — ·6 „
	Unguentum Gallæ cum Opio	„ 0·75 „ „ 100 „		
	Suppositoria Plumbi Composita	1 grain of opium in each.		
Physostigmatis Semina	Extractum Physostigmatis	...	¼— 1 grain	·015— ·06 „
Podophylli Resina	...	...	¼— 1 „	·015— ·06 „
	Tinctura Podophylli	2 grains in 1 fl. drachm	5—15 ♏	·3 — 1·0 c.c.
Stramonii Folia	„ Stramonii	20 parts in 100 fl. parts	5—15 ♏	·3 — 1·0 „
„ Semina	Extractum „	...	¼— 1 grain	·015— ·06 gram
Strophanthus Semina	„ Strophanthi	...	¼— 1 „	·015— ·06 „
	Tinctura „	2·5 parts in 100 fl. parts	5—15 ♏	·3 — 1·0 c.c.

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* 1 grain equals approximately 64 milligrams (·064 gram), or 1 milligram equals $\frac{1}{64}$ grain.

Synonyms

Acid, borussic	Acid. Hydrocyan. Dil.
„ carbazotic	Picric acid
„ of sugar	Oxalic acid
Æthiops Absorbens	Hyd. c̄ Cretâ
„ mineral	Hydrarg. Sulph. c̄ Sulph.
Alcoolat (Fr.)	Alcohol
Alegar	Vinegar
Algaroth's powder	Antimony oxychloride
Alum Rupeum	Alum with ferric oxide
Antimony, diaphoretic	Antimony oxide
Aqua Ammoniæ	Liq. Ammoniæ
„ Coloniensis	Eau de Cologne
„ Flava	Lotio Hydrarg. Flav.
„ Fortis	Acid, nitric
„ Naphæ	Aq. Flor. Aurant.
„ Nigra	Lotio Hydrarg. Nig.
„ Regia	Strong nitro-hydrochloric acid
Aquila alba	Calomel
Argentum vivum	Quicksilver Mercury
Argol	Crude cream of tartar
Aromatic confection	P. Cretæ Aromat.
Atramentum nigrum	Black ink
Aurum musivum	Tin sulphide Mosaic gold
Axungia	Lard
Balsam, friar's	Tr. Benzoin. Comp.
„ sulphur	Sulphurated oil
„ traumatic	Tr. Benzoin. Comp.
Basilicon ointment	Ung. Resinæ
Baume de vie	Dec. Aloës Comp.
Benzene	Coal tar derivative (C_6H_6)
Benzin	Petroleum ether
Bitter apple	Colocynth
„ chips	Quassia
Black draught	Mist. Sennæ Comp.
„ drop	A strong sol. of opium (U.S.P. 1851)
Blaud's pill	Pil. Ferri
Blue ointment	Ung. Hydrarg.
Blue pill	Pil. Hydrarg.
„ stone	Cupric sulphate
„ unction	Ung. Hydrarg. Mite.
Bole, Armenian	Ferric oxide
British gum	Dextrin
Butter of antimony	Liq. Antim. Chlor.
„ zinc	Zinc chloride
Cacao butter	Oil of theobroma
Calisaya bark	Yellow cinchona
Calomelas	Mercurous chloride
Camphor julep	Camphor water
Caramel	Burnt sugar
Carbasus	Gauze
Carron oil	Linseed oil and lime water
Ceratum Galeni	Cold cream
Ceresin	A kind of hard paraffin
Charpie	Lint
Chemical food	Syr. Ferri Phosph. Co.
Chinese white	Zinc oxide
Chinium	Quinine
Cinnabar	Vermilion
„ of antimony	„
Citrine ointment	Ung. Hydrarg. Nitratis
Colcothar	Purple ferric oxide
Colophony	Resin
Commander's balsam	Tr. Benzoin Comp.
Conf. Aromat.	P. Cretæ Aromat.
Copperas	Ferrous sulphate (impure)
Corrosive sublimate	Mercuric chloride
Cosmetic mercury	Hyd. Ammon. Chlor.
Crab's eyes	Calcium carbonate Prepared chalk
Cream of tartar	Potass. Tart. Acidus
Crocus martis	See Colcothar
Crow-fig	Nux vomica
Deadly nightshade	Belladonna
Diachylon plaster	Emp. Plumbi
Donovan's solution	Liq. Arsenii et Hydrargyri Iodidi
Dover's powder	Pulv. Ipecac. Comp.
Dragon's blood	Calamus Draco
Easton's syrup	Syr. Ferri, Quin. et Strych. Phosph.

Eau de Javelle	Sol. Potass. Hypochlorite
„ luce	Tr.Ammon.Co.(P.L.)
„ naphre	Aq. Flor. Aurant.
„ vie	Brandy
Elixir of paregoric	Tr. Camph. Co.
„ vitriol	Acid. Sulph. Aromat.
Epsom salts	Magnes. Sulph.
Ferrugo	Iron rust
Ferrum Limatum	Iron filings
Flake white	Lead carbonate
Flowers of sulphur	Sulphur, sublimed
„ zinc	Zinc oxide
Fluid magnesia	Liq. Magnes. Carb.
Fowler's solution	„ Arsenicalis
Friar's balsam	Tr. Benzoin. Comp.
Glauber's salt	Sodium sulphate
Glonoin, solution	Liq. Trinitrinæ
Goulard's extract	„ Plumbi Fort.
„ water	„ „ Dil.
Grains of paradise	Melagueta pepper
Green ointment	Ung. Sambuci Virid.
„ vitriol	Ferrous sulphate
Gregory's powder	Pulv. Rhei Comp.
Griffith's mixture	Mist. Ferri Comp.
Gum dragon	Tragacanth
Heberden's ink	Mist. Ferri Aromat.
Hepar sulphur	Sulphurated potash
Hiera picra	P. Aloës c̄ Canella
Hoffmann's anodyne	Sp. Ætheris Comp.
Homburg's sedative salt	Acid, boric
Huile de cade	Juniper tar oil
Huxham's tinct. of bark	Tr. Cinchonæ Co.
Hydrarg. Biniodid.	Mercuric iodide
Ichthyocolla	Isinglass
James' powder	Said to be P. Antimonialis
Jesuit's bark	Cinchona
„ drops	Tr. Benz. Comp.
Kaolin	China clay
Kermes mineral	Sulphurated antimony

King's yellow	Arsenic sulphide
Labarraque's liquid	Sol. chlorinated soda
Lapis infernalis	Silver nitrate
Laudanum	Tinct. Opii
Lavender drops	Tr. Lavand. Comp.
Lenitive electuary	Confect. Sennæ
Lignum vitæ	Guaiacum wood
Liq. Cornu Cervi	Liq. Ammoniæ
„ Donovani	See Donovan
„ Potassæ Arsenitis	Liq. Arsenicalis
Litharge	Lead oxide
Liver of sulphur	Sulphurated potash
Lunar caustic	Nitrate of silver (sticks)
Magister of bismuth	Bismuth oxynitrate
„ sulphur	Washed sulphur
Magnesia alba	Mag. Carb. Pond.
Massicot	Lead oxide; litharge
Maw seed	Poppy seeds
Medulla bovis	Beef marrow
Mica panis	Bread crumb
Microcosmic salt	$Na(NH_4)HPO_4$
Milk of sulphur	Precipitated sulphur
Mindererus spirit	Liq. Ammon. Acet.
Mineral, Æthiops	See Æthiops
„ kermes	See Kermes
„ turpeth	See Turpeth
Minium	Red-lead
Mistura Acaciæ	Mucilage of acacia
„ Griffith's	Mist. Ferri Comp.
Mosaic gold	See Aurum
Muriatic acid	Hydrochloric acid
Natrium	Sodium
Nitre	Potassium nitrate
Oil, carron	See Carron
„ Dippel's	Animal oil distilled from bones
„ Lund's	A mixture of almond and castor oil, with phenol, 5 per cent.
„ of kermes	Syr. Rhæados
„ of mace	Expressed oil of nutmeg
„ of swallows	Ol. Sambuci
„ of tar	Creasote

Oil of vitriol	Strong sulphuric acid
Ointment, basilicon	See Basilicon
,, citrine	See Citrine
,, green	See Green
Oleum Bubulum	Neatsfoot oil
,, Nervinum	,, ,,
,, Rusci	Oil of birch tar
,, Viride	Ol. Sambuci
Opodeldoc	Lin. Saponis Comp.
Orpiment	Native yellow arsenic sulphide
Paregoric	Tr. Camph. Comp.
Pearl white	Bismuth oxynitrate
Pill, Blaud's	Pil. Ferri
,, Christison's	,, Coloc. c̄ Hyoscy.
,, Marshall Hall's	,, Aloës Dil.
,, Plummeri	,, Hydrarg. Subchlor. Co.
,, Rufi	,, Aloës et Myrrhæ
Plasma	Glycerin of starch
Pomelian ointment	Ung. Sambuci Virid.
Precipitate, red	Hyd. Oxid. Rub.
,, white	,, Ammon. Chlor.
Preston salts	Normal ammon. carbonate
Pulvis Aërophorus	Seidlitz powders
,, Basilicus	Calomel and sugar, of each ½, Scammony 1
,, Jacobi Fict.	P. Antimonialis
Putty powder	Tin oxide
Realgar	Native red arsenic sulphide
Red dominion plaster	Emp. Ferri
,, lavender drops	Tr. Lavand. Comp.
Roche alum	Crystals of ordinary alum coloured with ferric oxide
Rochelle salt	Soda Tartarata
Roman alum	Crystals of ordinary alum coloured with ferric oxide
,, vitriol	Cupric sulphate
Rufus pill	Pil. Aloës et Myrrhæ
Rupel alum	Crystals of ordinary alum coloured with ferric oxide

Sal acetosella	Potassium oxalate
,, alembroth	Supposed to be a double chloride of ammonium and mercury
,, ammoniac	Ammonium chloride
,, enixum	Potass. hydrogen sulphate
,, prunella	Potassium nitrate (glob.)
,, volatile	Ammonium carbonate
Salt of lemon	Potassium oxalate
,, tartar	,, carbonate
,, wormwood	,, ,,
Saltpetre	,, nitrate
Scott's dressing	Ung. Hydrarg. Co.
Solution, Donovan's	See Donovan's
,, Fowler's	Liq. Arsenicalis
Spirit. Frumenti	Whisky
,, Lavand. Co.	Tr. Lavand. Co.
,, Mindererus	Liq. Ammon. Acet.
,, of hartshorn	Liq. Ammoniæ
,, Sacchari	Rum
Steel drops	Tr. Ferri Perchlor.
,, mixture	Mist. Ferri Comp.
Sugar of lead	Lead acetate
Sulphur, milk of	Precipitated sulphur
,, nigrum	Impure sulphur, black
,, vivum	,, ,,
Tartar emetic	Antim. Tart. (B.P.)
Tinct. Meconii	Tr. Opii
,, of steel	,, Ferri Perchlor.
,, Opii Camph.	,, Camph. Co.
,, Thebaici	,, Opii
Toothache seeds	Sem. Hyoscyami
Torrington's drops	Tr. Benz. Co.
Trooper's ointment	Ung. Hydrarg. Mite.
Turner's cerate	,, Calaminæ
Turpeth mineral	Yellow mercury oxysulphate
Tutty powder	Impure zinc oxide
Vegetable salt	Potassium oxalate
Verdigris	Cupric oxyacetate
Vermilion	Mercury sulphide
Vitriol, blue	Cupric sulphate
,, green	Ferrous ,,

Vitriol, oil of	Sulphuric acid
„ white	Zinc sulphate
Wade's drops	Tr. Benz. Comp.
Ward's paste	Conf. Piper. Nig.
White copperas	Zinc sulphate
„ lead	Lead oxycarbonate
White precipitate	Ammoniated mercury
„ Spanish	Bismuth oxynitrate
„ vitriol	Zinc sulphate
Yellow basilicon ointment	Ung. Resinæ

CHAPTER LVI

POISONS

Precautions required by the Pharmacy Act, 1868, to be observed in selling by retail, and in dispensing poisons

All the articles named or referred to in the list, both in Part I and Part II, are *poisons* within the meaning of the Pharmacy Act, 1868.

Sale by Retail

I. *Relating to Part I and Part II of the list.*—It is unlawful to sell any "poison" by retail unless the vessel, wrapper, or cover in which it is contained be distinctly labelled with *the name of the article,* the word *Poison,* and *the name and address of the seller.* This applies to all the articles in both parts of the list.

II. *Relating to Part I only.*—It is unlawful to sell by retail any poison included in Part I of the list to any person unknown to the seller, unless introduced by some person known to the seller; and on every sale of any such article the seller shall, before delivery, make or cause to be made an entry in a book, to be kept for that purpose, of—

1. The date of sale;
2. The name and address of the purchaser;
3. The name and quantity of the article sold; and
4. The purpose for which it is stated to be required; to which the signature of the purchaser, and of the person, if any, who introduced him, shall be affixed. *The article must also be labelled with the name of the article, the word "Poison," and the name and address of the seller.*

III. *Relating only to arsenic and its preparations.*—It is unlawful to sell arsenic or any of its preparations unless, in addition to all the foregoing regulations, the following provisions of the Arsenic Act be also observed:

1. That the poison, if colourless, be mixed with soot or indigo, so as to colour it;
2. That the person to whom the poison is sold or delivered be of mature age;
3. That the occupation as well as the name and address of the purchaser be entered in the poison book.

Dispensing.—None of the foregoing regulations apply to any article when forming part of the ingredients of any medicine dispensed by a registered chemist and druggist; but it is necessary, if a medicine contain a poison included in Part I or Part II of the list, that the ingredients of the medicine, together with the name of the person to whom it is sold or delivered, be entered in a book kept for that purpose (*Prescription Book*), and that the name and address of the seller be attached to the medicine.

Re-copying of prescriptions containing scheduled poisons.—It is not considered to be absolutely necessary to re-copy such prescriptions each time they are dispensed, *but it is necessary* to enter the number of the prescription, name, date, and address of the patient each time, in order to comply with the foregoing, and for this purpose a special register should be kept.

List of Poisons within the meaning of the Act

PART I.

Not to be sold unless *the purchaser is known to, or is introduced by some person known to, the seller;*
also
Entry to be made in
POISON BOOK
of

1. *Date of sale.*
2. *Name and address of purchaser.*
3. *Name and quantity of article.*
4. *Purpose for which it is wanted; attested by signature,*

and
Must be labelled with—

1. Name of article.
2. The word "POISON."
3. Name and address of seller.

ACONITE and its preparations.
ALKALOIDS: all poisonous vegetable alkaloids and their salts.
ARSENIC and its preparations.
ATROPINE and its preparations.
CANTHARIDES.
CORROSIVE SUBLIMATE.
EMETIC TARTAR.
ERGOT OF RYE and its preparations.
POTASSIUM CYANIDE and all metallic cyanides and their preparations.
PRUSSIC ACID and its preparations.
SAVIN and its oil.
STRYCHNINE and its preparations.
VERMIN KILLERS, if preparations of poisons in Part I of this schedule.

Picrotoxin
Cocaine Salts

Strophanthus & Preps

Carbolic acid over 3%.
Digitalis Preps.
Mercuric Iodide
Merc Sulphocyan

PART II.

Must be labelled with—
1. *Name of article.*
2. *The word "POISON."*
3. *Name and address of seller.*

Almonds, essential oil of (unless deprived of prussic acid).
Belladonna and its preparations.
Cantharides, tincture and all vesicating liquid preparations of.
Chloral hydrate and its preparations.
Chloroform.
Corrosive sublimate, preparations of.
Morphine, preparations of.
Nux vomica and its preparations.
Opium and its preparations, and preparations of poppies.
Oxalic acid.
Precipitate, red (red oxide of mercury).
Precipitate, white (ammoniated mercury).
Vermin killers (*see* Part I).
[Compounds containing "Poisons" prepared for the destruction of vermin, if not subject to the provisions of Part I, are in Part II.]

The dispensing and selling of poisons is one of the most important functions of the legally qualified pharmacist. By the Pharmacy Act of 1868 it is unlawful for any person not being a pharmaceutical chemist, or chemist and druggist within the meaning of the Act, to sell or keep open shop for the retailing, dispensing, or compounding of poisons described in the schedule. As the law has placed pharmacists in such a responsible position, it behoves them to see not only that all the provisions of the Act are duly observed, but that in all dealings of which poisons form a part the greatest care is exercised.

At a general meeting of the Pharmaceutical Society of Great Britain on the 17th of May, 1871, the following recommendations for the keeping, dispensing, and selling of poisons were adopted:

1. That in the keeping of poisons, each bottle, vessel, box, or package containing a poison be labelled with the name of the article, and also some distinctive mark that it contains poison.

2. Also, that in the keeping of poisons, each poison be kept on one or other of the following systems, viz.:

(*a*) In a bottle or vessel tied over, capped, locked, or otherwise secured in a manner different from that in which bottles or vessels containing ordinary articles are secured in the same warehouse, shop, or dispensary; or

(*b*) In a bottle or vessel rendered distinguishable by touch, from the bottles or vessels in which ordinary articles are kept in the same warehouse, shop, or dispensary; or

(*c*) In a bottle, vessel, box, or package kept in a room or cupboard set apart for dangerous articles.

3. That in the dispensing and selling of poisons, all liniments, embrocations, and lotions containing posion be sent out in bottles rendered distinguishable by touch from ordinary medicine bottles, and that there also be affixed to each such bottle (in addition to the name of the article, and to any particular instructions for its use) a label giving notice that the contents of the bottle are not to be taken internally.

Experience has taught that the greatest safety is obtained by keeping all poisons and potent remedies in a cupboard by themselves. As an extra safeguard this cupboard may be kept locked, and a bell so arranged that an alarm is given each time of opening. In establishments where two or more dispensers are kept, no poisons should ever be dispensed or sold, without the presence of a qualified witness, whose duty it is to make a note in a book kept for the purpose, of the name and amount of medicament, the number of the prescription, with both his own and the dispenser's initials. It is impossible to bestow too much care on the keeping and dispensing of poisons, and in every case of doubt the dispenser should, no matter at what cost of time and trouble, assure himself that he is carrying out the *intentions* of the prescriber.

A copy of the poison schedule should be hung in the immediate vicinity of the poison cupboard, as well as a table of antidotes for the more commonly occurring poisons, so as to be provided for all cases of emergency.

In retailing poisons it is always better to err on the side of caution, as although all the poisons in the schedule may be sold if the legal requirements are fulfilled, yet it is the duty of every pharmacist to supply such poisons only when he can reasonably assure himself that they are to be used for legitimate purposes.

Poisons and their Antidotes

In case of poisoning, first send for a medical man; then administer an emetic, unless corrosive acids or strong alkalies are known to have been swallowed. The safest emetic is thirty grains of zinc sulphate in water, preferably slightly warm. If this is not at hand a table-spoonful of mustard in half a pint of tepid water may be administered.

The following antidotes may be given by any person of intelligence if there is reasonable ground for believing a particular poison to have been taken:

ACID, CARBOLIC.—Emetic; large doses of almond or olive oil; the whites of several fresh eggs; brandy.

ACID, PRUSSIC, and CYANIDES.—A mixture of ferric chloride, ammonia, and water; brandy.

ACIDS, CORROSIVE MINERAL.—Chalk or magnesia in water; demulcent drinks.

ACONITE.—Emetic; animal charcoal in water; hot water bottles to feet and chest.

ALKALIES.—Dilute acetic acid; vinegar; olive oil.

AMMONIA.—The same as strong alkalies.

ANTIMONY TARTARATED.—White of egg; tannic acid; strong coffee.

ARSENIC.—Ten grains of copper sulphate in water as an emetic; freshly precipitated ferric hydrate; white of egg and brandy.

BARIUM.—Dilute sulphuric acid; Epsom salts; emetic.

BELLADONNA.—Emetic.

CANTHARIDES.—Emetic; *no* oil of any kind.

CHLOROFORM and CHLORAL.—Fresh air; brandy; induce artificial respiration.

COLCHICUM.—Emetic; egg-albumen; strong coffee.

CONIUM.—Emetic; tannic acid.

COPPER SALTS.—Emetic; twenty grains of potassium ferrocyanide in water; hot fomentation to abdomen.

CROTON OIL.—Emetic; camphor.

DIGITALIS.—Emetic; tannic acid; animal charcoal.

ELATERIUM.—Emetic; hot fomentation to abdomen.

ESERIN.—Emetic; tannic acid.

FUNGI.—Emetic.

HENBANE.—As for belladonna.

INDIAN HEMP.—Emetic.

IODINE.—Mucilage of starch or arrowroot; thirty grains of sodium hyposulphite in water.

LEAD SALTS.—Emetic; dilute sulphuric acid.

MERCURY SALTS.—Emetic; whites of fresh eggs.

OPIUM and MORPHIA.—Emetic; copious draughts of a solution of potassium permanganate, thirty grains to the pint; strong coffee; fresh air; keep patient awake.

Oxalates.—Emetic; chalk and water; calcium chloride.

Phosphorus.—Emetic; milk, and brandy, *no oil.*

Silver Salts.—Common salt in water; egg-albumen.

Strychnia.—Emetic; afterwards twenty grains of chloral and forty grains of potassium bromide in one ounce of water.

Zinc Salts.—Carbonate of soda; tannic acid; milk; strong coffee.

Alcohol Table

Percentage of Absolute Alcohol by Weight corresponding to the Specific Gravity taken at 60° F. (15·5° C.); Water = 1.

Specific gravity.	Percentage.	Specific gravity.	Percentage.	Specific gravity.	Percentage.
0·9981	1	0·9490	35	0·8772	68
0·9963	2	0·9472	36	0·8748	69
0·9944	3	0·9453	37	0·8724	70
0·9928	4	0·9433	38	0·8700	71
0·9912	5	0·9413	39	0·8676	72
0·9896	6	0·9394	40	0·8652	73
0·9880	7	0·9374	41	0·8629	74
0·9866	8	0·9353	42	0·8605	75
0·9852	9	0·9332	43	0·8581	76
0·9839	10	0·9311	44	0·8557	77
0·9826	11	0·9291	45	0·8533	78
0·9813	12	0·9269	46	0·8509	79
0·9800	13	0·9249	47	0·8484	80
0·9788	14	0·9227	48	0·8459	81
0·9775	15	0·9204	49	0·8435	82
0·9763	16	0·9183	50	0·8409	83
0·9751	17	0·9160	51	0·8385	84
0·9739	18	0·9138	52	0·8359	85
0·9727	19	0·9116	53	0·8333	86
0·9714	20	0·9090	54	0·8307	87
0·9702	21	0·9072	55	0·8282	88
0·9690	22	0·9049	56	0·8256	89
0·9677	23	0·9027	57	0·8229	90
0·9664	24	0·9004	58	0·8203	91
0·9651	25	0·8981	59	0·8176	92
0·9637	26	0·8958	60	0·8149	93
0·9622	27	0·8935	61	0·8122	94
0·9607	28	0·8911	62	0·8094	95
0·9592	29	0·8888	63	0·8065	96
0·9577	30	0·8865	64	0·8036	97
0·9560	31	0·8842	65	0·8006	98
0·9544	32	0·8818	66	0·7976	99
0·9526	33	0·8795	67	0·7446	100
0·9508	34				

ALCOHOL TABLE

Percentage of Absolute Alcohol by Volume corresponding to the Specific Gravity taken at 60° F. (15·5°); Water = 1.

Specific gravity.	Percentage.	Specific gravity.	Percentage.	Specific gravity.	Percentage.
0·9985	1	0·9592	35	0·8949	68
0·9970	2	0·9579	36	0·8925	69
0·9956	3	0·9565	37	0·8900	70
0·9942	4	0·9550	38	0·8875	71
0·9928	5	0·9535	39	0·8850	72
0·9915	6	0·9519	40	0·8825	73
0·9902	7	0·9503	41	0·8799	74
0·9890	8	0·9487	42	0·8773	75
0·9878	9	0·9470	43	0·8747	76
0·9866	10	0·9452	44	0·8720	77
0·9854	11	0·9435	45	0·8693	78
0·9843	12	0·9417	46	0·8666	79
0·9832	13	0·9399	47	0·8639	80
0·9821	14	0·9381	48	0·8611	81
0·9811	15	0·9362	49	0·8583	82
0·9800	16	0·9343	50	0·8555	83
0·9790	17	0·9323	51	0·8526	84
0·9780	18	0·9303	52	0·8496	85
0·9770	19	0·9283	53	0·8466	86
0·9760	20	0·9263	54	0·8436	87
0·9750	21	0·9242	55	0·8405	88
0·9740	22	0·9221	56	0·8373	89
0·9729	23	0·9200	57	0·8339	90
0·9719	24	0·9178	58	0·8306	91
0·9709	25	0·9156	59	0·8272	92
0·9698	26	0·9134	60	0·8237	93
0·9688	27	0·9112	61	0·8201	94
0·9677	28	0·9090	62	0·8164	95
0·9666	29	0·9067	63	0·8125	96
0·9655	30	0·9044	64	0·8084	97
0·9643	31	0·9021	65	0·8047	98
0·9631	32	0·8997	66	0·7995	99
0·9618	33	0·8973	67	0·7946	100
0·9605	34				

TABLE OF SOLUBILITIES

One part is soluble in	Distilled water at 60° F.	90 per cent. alcohol at 60° F.	Comments.
Acetanilid	200	4	Freely in ether, benzol, chloroform.
Acid. Arseniosum	100	...	1 in 5 of glycerin; freely in alkaline solutions.
,, Benzoicum	400	3	In fixed and volatile oils; freely in ether.
,, Boricum	25	30	1 in 4 of glycerin.
,, Carbolicum	...	Freely	Freely in glycerin, ether, chloroform, and fixed oils.
,, Chromicum	Freely	Incompatible	
,, Citricum	0·75	Freely	
,, Gallicum	100	5	1 in 40 of ether.
,, Oleicum	...	Freely	Freely in chloroform, ether, and fixed oils.
,, Salicylicum	500	3	Freely in ether, also in sol. of acetates, citrates, and phosphates.
,, Tannicum	1	1	
,, Tartaricum	0·75	3	
Aconitina	...	Freely	Freely in chloroform.
Æther	12	,,	Freely in chloroform, fixed and volatile oils.
,, Aceticus	10	,,	Freely in chloroform.
Alumen	10	...	Freely in glycerin.
Ammonii Benzoas	6	30	
,, Bromidum	Freely	15	
,, Carbonas	4	200	1 in 5 of glycerin.
,, Chloridum	3	60	
,, Iodidum	1	3	3 in 4 of glycerin.
,, Phosphas	4	...	
Amyl Nitris	...	Freely	
Analgen	...	Sparingly	
Antimon. Tartarat.	18	...	
Apomorph. Hydroch.	70	50	
Argenti Nitras	0·75	30	Soluble in ether and glycerin.
Aristol	...	...	Soluble in ether and fixed oils.
Arsenii Iodidum	11	42	
Atropina	300	Freely	Freely in chloroform and ether.
,, Sulphas	0·5	3	
Borax	25	...	In 1 of glycerin.
Butyl-Chloral Hydras	50	1	In 1 of glycerin, in 20 of chloroform.
Caffeina	80	40	Freely in chloroform.
,, Citras	30	22	
Calcii Chloridum	1	3	
,, Hypophosphis	8	...	
Camphora	700	1	1 in 0·25 chloroform, 1 in 4 olive oil.
,, Monobromata	...	...	Freely in glycerin.
Chloral Hydras	1	1	1 in 4 chloroform, 1 in 1 of ether.
Chloralamid	10	Freely	Decomposes in hot water.
Chloroform	200	,,	
Cocaina	...	10	1 in 4 ether, 1 in 0·5 chloroform, 1 in 12 olive oil.
,, Hydrochloridum	0·5	4	1 in 4 glycerin.

One part is soluble in	Distilled water at 60° F.	90 per cent. alcohol at 60° F.	Comments.
Codeina	80	Freely	Freely in chloroform, 1 in 80 sol. ammonia.
„ Phosphas ...	2	...	
Creosotum	150	Freely	Freely in ether and glacial acetic acid.
Cupri Sulphas	4	...	Freely in glycerin.
Eucainæ Hydrochloridum	Freely		
Exalgin	60	Freely	
Ferri et Ammonii Citras ...	0·5	...	
„ et Quininæ Citras ...	0·5	...	
„ Lactas	40	...	
„ Sulphas	2	...	
Ferrum Tartaratum ...	1	...	
Glusidum	400	25	Soluble in dilute alkalies.
Homatropin. Hydrobromid.	6	135	
Hydrarg. Cyanidum ...	13	15	
„ Perchloridum ...	16	3	1 in 4 ether; very freely in glycerin.
Hydrastinin. Hydrochlorid.	Freely	3	
Hyoscine Hydrobromid. ...	1	13	
Hyoscyamine Sulphas ...	0·5	3	
Ichthyol-Ammonium ...	...	...	Miscible with water, alcohol, and fixed oils.
Iodoform	...	80	1 in 5 ether; moderately in volatile and fixed oils and fats.
Iodol	...	25	Moderately soluble in chloroform and fixed oils.
Iodum	5000	Freely	Freely in ether, solution of potassium iodide.
Lactophenin	500	10	
Lithii Benzoas	4	2	
„ Bromidum ...	0·5	Freely	
„ Citras	2	...	
„ Salicylas	Freely	Freely	
Lysidinum...	Freely		
Magnesii Sulphas... ...	1	...	
Menthol	...	Freely	Freely in chloroform; soluble in oils and fats.
Morphinæ Acetas... ...	3	100	
„ Hydrochloridum	24	50	
„ Sulphas ...	22	700	
„ Tartras ...	11	...	
Naphthalinum	...	15	
Naphthol, Beta	1000	2	
Orthoform	...	...	Soluble in water, alcohol, ether, fats, and oils.
Paraldehydum	10	Freely	
Phenacetin	...	20	
Phenazonum	1	1½	
Phenocoll. Hydrochlorid.	16		
Phosphorus	...	...	1 in 350 absol. alcohol; 1 in 80 ether; 1 in 80 olive oil; 1 in 25 chloroform; 1 in ·5 carbon disulphide.
Physostigminæ Salicylas...	150	12	
„ Sulphas ...	Freely	Freely	
Picrotoxinum	350	13	
Pilocarpinæ Nitras ...	8	50	
„ Hydrochloridum	Freely	Freely	
Piperazinum	Freely		

One part is soluble in	Distilled water at 60° F.	90 per cent. alcohol at 60° F.	Comments.
Plumbi Acetas	3	30	
,, Iodidum	2000		
Potassii Acetas	0·5	2	
,, Bicarbonas ...	4	...	
,, Bromidum ...	2	95	
,, Chloras	16	...	
,, Citras	Freely	...	
,, Cyanidum ...	2	100	
,, Dichromas ...	10	...	
,, Ferrocyanidum ...	4	...	
,, Hydroxidum ...	0·5	4	
,, Iodidum	1	12	
,, Nitras	4	...	
,, Permanganas ...	20	...	
,, Sulphas	10	...	
,, Tartras	1	...	
,, ,, Acidus ...	200	...	
Pyrogallol...	2	1	
Quininæ Hydrochloridum	35	3	
,, ,, Acidum	0·5	...	
,, Sulphas	800	65	Freely in dilute mineral acids.
,, Bisulphas ...	10	32	
,, Valerianas ...	100	5	
Resorcinum	0·5	0·5	
Saccharum Lactis ...	7	...	
,, Purificatum ...	0·5	...	
Salicinum	28	60	
Salol	...	10	Freely in ether, chloroform, fixed and volatile oils.
Santoninum	...	40	1 in 4 chloroform.
Soda Tartarata	2	...	
Sodii Arsenas	3	...	
,, Benzoas	2	24	
,, Bicarbonas	11	...	
,, Bromidum	2	16	
,, Carbonas	2	...	
,, Chloridum	3	200	
,, Hypophosphis ...	1	30	
,, Hyposulphis ...	0·75	...	
,, Iodidum	1	3	
,, Nitris	Freely	50	
,, Phosphas	6	...	
,, Salicylas	1	6	
,, Sulphas	6	...	
,, Sulphis	Freely	...	
,, Sulphocarbolas ...	6	150	
Sparteinæ Sulphas ...	Freely	Freely	
Strontii Bromidum ...	Freely	Freely	
,, Iodidum... ...	1	Freely	
,, Lactas	314	Freely	
Strychnina	...	150	
,, Hydrochloridum	35	60	
,, Sulphas ...	50	109	
Sulphonal	450	50	Soluble in ether.
Tetronal	500	15	
Thymol	...	Freely	Freely in ether; 1 in 8 Liq. Potassæ.
Trional	300	Freely	

One part is soluble in	Distilled water at 60° F.	90 per cent. alcohol at 60° F.	Comments.
Urethanum	Freely	Freely	
Urotropinum	...	Freely	
Veratrina	...	3	
Zinci Acetas	3	36	
„ Bromidum	Freely	Freely	
„ Chloridum	Freely	Freely	
„ Iodidum	Freely	Freely	
„ Sulphas	1	...	
„ Sulphocarbolas ...	2	3	
„ Valerianas	100	40	

Table of Thermometric Equivalents according to the Centigrade and Fahrenheit Scale

C.°	F.°	C.°	F.°	C.°	F.°
0	32	43	109·4	86	186·8
1	33·8	44	111·2	87	188·6
2	35·6	45	113·0	88	190·4
3	37·4	46	114·8	89	192·2
4	39·2	47	116·6	90	194·0
5	41·0	48	118·4	91	195·8
6	42·8	49	120·2	92	197·6
7	44·6	50	122·0	93	199·4
8	46·4	51	123·8	94	201·2
9	48·2	52	125·6	95	203·0
10	50·0	53	127·4	96	204·8
11	51·8	54	129·2	97	206·6
12	53·6	55	131·0	98	208·4
13	55·4	56	132·8	99	210·2
14	57·2	57	134·6	100	212·0
15	59·0	58	136·4	101	213·8
16	60·8	59	138·2	102	215·6
17	62·6	60	140·0	103	217·4
18	64·4	61	141·8	104	219·2
19	66·2	62	143·6	105	221·0
20	68·0	63	145·4	106	222·8
21	69·8	64	147·2	107	224·6
22	71·6	65	149·0	108	226·4
23	73·4	66	150·8	109	228·2
24	75·2	67	152·6	110	230·0
25	77·0	68	154·4	111	231·8
26	78·8	69	156·2	112	233·6
27	80·6	70	158·0	113	235·4
28	82·4	71	159·8	114	237·2
29	84·2	72	161·6	115	239·0
30	86·0	73	163·4	116	240·8
31	87·8	74	165·2	117	242·6
32	89·6	75	167·0	118	244·4
33	91·4	76	168·8	119	246·2
34	93·2	77	170·6	120	248 0
35	95·0	78	172·4	121	249·8
36	96·8	79	174·2	122	251·6
37	98·6	80	176·0	123	253·4
38	100·4	81	177·8	124	255·2
39	102·2	82	179·6	125	257·0
40	104·0	83	181·4	126	258·8
41	105·8	84	183·2	127	260·6
42	107·6	85	185·0	128	262·4

C.°		F.°	C.°		F.°	C.°		F.°
129	...	264·2	153	...	307·4	177	...	350·6
130	...	266·0	154	...	309·2	178	...	352·4
131	...	267·8	155	...	311·0	179	...	354·2
132	...	269·6	156	...	312·8	180	...	356·0
133	...	271·4	157	...	314·6	181	...	357·8
134	...	273·2	158	...	316·4	182	...	359·6
135	...	275·0	159	...	318·2	183	...	361·4
136	...	276·8	160	...	320·0	184	...	363·2
137	...	278·6	161	...	321·8	185	...	365·0
138	...	280·4	162	...	323·6	186	...	366·8
139	...	282·2	163	...	325·4	187	...	368·6
140	...	284·0	164	...	327·2	188	...	370·4
141	...	285·8	165	...	329·0	189	...	372·2
142	...	287·6	166	...	330·8	190	...	374·0
143	...	289·4	167	...	332·6	191	...	375·8
144	...	291·2	168	...	334·4	192	...	377·6
145	...	293·0	169	...	336·2	193	...	379·4
146	...	294·8	170	...	338·0	194	...	381·2
147	...	296·6	171	...	339·8	195	...	383·0
148	...	298·4	172	...	341·6	196	...	384·8
149	...	300·2	173	...	343·4	197	...	386·6
150	...	302·0	174	...	345·2	198	...	388·4
151	...	303·8	175	...	347·0	199	...	390·2
152	...	305·6	176	...	348·8	200	...	392·0

Seasons at which the following Plants should be collected

Aconite	June.
Belladonna	June to August.
Black currants	—
Broom	June and July.
Colchicum (flowers)	September and October.
,, (corms)	—
Coltsfoot	July and September.
Cotyledon	April and May.
Dandelion (root)	October to March.
,, (flowers)	April and May.
Elder (leaves)	May to September.
,, (flowers)	June and July.
Foxglove	May and June.
Goose-grass	June.
Hemlock	May and June.
Henbane	May and June.
Laurel (leaves)	October to May.
Lettuce (*L. virosa*)	May and June.
Lily of the Valley (flowers)	April and May.
,, (root)	February to May.
Mulberries	August.
Pansies	May to August.
Raspberries	July.
Red poppy (flowers)	June and July.
Roses	June and July.
Savin	October to May.
Stramonium	September.

Vegetables intended for pharmaceutical operations should be gathered in dry weather. They should, also, be collected annually, and not kept more than a year.

The following rules for the collection and preservation of vegetable substances may be generally followed :

Roots and rhizomes should be dug up when the old leaves and stalks have fallen off, and before the new ones have shot out.

Herbs and leaves should be gathered after the flowers have opened, but before the seeds have ripened.

Flowers only when freshly blown.

Fruits and seeds as soon as they are fully ripe.

All parts of vegetables, unless otherwise directed, should be kept in a dry state. The drying may be effected either by exposing them in shallow baskets to the heat of the sun, or they may be placed in the drying cupboard, which should be kept at a temperature of 95° to 100° F.

PART VI

URINE ANALYSIS

CHAPTER LVII

URINE ANALYSIS

Normal urine is clear, and is of a pale reddish-yellow tinge; on standing, however, a slight cloud of mucus or a deposit of urates may form. The specific gravity ranges from 1·015 to 1·025. All examinations should be made on a sample taken from the whole bulk excreted in twenty-four hours. A healthy adult excretes about fifty ounces in this period, 4 per cent. of which consists of solid matter.

1. The appearance should be carefully noted. Any turbidity may be due to urates (*dissolving on warming*), phosphates (*soluble in acetic acid*), mucus, blood, or pus. When blood is present the urine has a brownish-red smoky appearance. Bile salts tinge urine greenish brown. Uric acid sometimes precipitates in considerable quantity; it is only slightly soluble in boiling water, but readily dissolves on warming with solution of potash. Urine containing much pus is generally turbid from the presence of suspended pus corpuscles; the greater number, however, sink to the bottom, where they form an opaque milky deposit which is readily diffused by agitation. The most reliable reagent for pus is solution of potash, which renders the deposit glairy and gelatinous, so that it will not drop, but may be poured from one tube to another as a viscid mass.

2. The specific gravity is taken at 60° F. In albuminuria the specific gravity falls as low as 1·005, while in diabetes it may rise to 1·050. The determination may be made with a Regnault's bottle, but in ordinary cases it is sufficiently accurate if made with the small form of hydrometer known as an urinometer, Figs. 249 and 250.

3. Reaction.—The behaviour to litmus-paper is noted. Normal urine is faintly acid, but in warm weather it soon becomes alkaline, owing to decomposition of the urea into ammonium carbonate.

4. Detection of Albumen.—Make a few cubic centimetres slightly

alkaline with solution of ammonia, and filter. Divide the filtrate into two equal portions, to one of which add a few drops of acetic acid, and boil. Flocks and coagula separate if albumen is present.

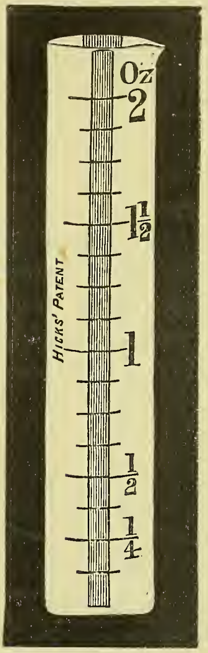

Fig. 249.—Urinometer glass.

Fig. 250.

Fig. 251.—Esbach's albuminometer.

Esbach's test.—Filter about 10 c.c. of the urine and add an equal bulk of the following:

Picric acid	10 parts.
Citric acid	20 ,,
Distilled water	1000 ,,

This produces an immediate precipitate if albumen is present. Peptones which *may* be precipitated, re-dissolve on heating, albumen does not. An *approximate determination* of the amount may be made in Esbach's albuminometer (Fig. 251), but the results are very inaccurate. Comparative examinations may, however, be made by following these directions.

Pour the urine into the tube until the mark U is reached. Fill up to the mark R with the test solution. Close the end of the tube with a rubber stopper and invert twice or three times to ensure intermixture, but avoid violent agitation. Set aside the closed tube for twenty-four hours in an upright position, and at the end of this time read off on the scale the height of the precipitate. The figure noted represents the amount of albumen in grams per litre of urine. Should the urine contain albumen to an extent of over 4 or 5 grams per litre, accuracy is promoted by diluting it with one or two volumes of water and making the requisite allowance for the dilution. When an *exact determination* is required, 100 c.c. of the urine are rendered slightly acid with acetic acid, 2 c.c. of strong nitric acid added, and the whole well shaken; 10 c.c. of a mixture of one part by weight of crystallised phenol, one part by weight of acetic acid, and two parts of rectified spirit are next added and well shaken. Stand for ten minutes and filter, wash with cold 4 per cent. phenol solution, dry, and weigh. The advantage of this process is that the reaction is not interfered with by sugar or salts.

5. DETECTION OF SUGAR: *Fehling's test.*—The urine is rendered alkaline with a few drops of caustic potash, and filtered. Fehling's solution is then added to the filtrate, and the whole slowly raised to boiling. If glucose is present a yellowish-red or red precipitate of cuprous oxide is formed.

Nylander's test.—The reagent is prepared as follows:

Bismuth oxynitrate (pure)	2·50	
Rochelle salt (crystals)	8·00	
Sodium hydrate solution (8 per cent.) ...	100·00.	Dissolve.

1 c.c. of the urine is boiled with 10 c.c. of the reagent, when if glucose is present the solution turns black.

Johnson's test.—5 c.c. of urine are mixed with an equal bulk of saturated picric acid solution, and 2 c.c. of solution of potash added. An orange-red coloration at once appears, due to the reducing action of the creatinine present in all normal urines. If the mixture in the tube is heated to boiling the colour deepens, but is always perfectly transparent when held to the light; but if sugar is present the colour becomes so intense as to render the mixture quite opaque.

DETERMINATION OF SUGAR.—This may be performed by Fehling's method or by Pavy's modification of Fehling's process, or by Sir George Johnson's method. Only the two latter will be described here, as Fehling's method is given in most text-books on chemistry.

Dr. Pavy's modification.—This differs from Fehling's original method, where the copper is weighed as cuprous oxide, in that advantage is taken of the solubility of cuprous oxide in ammonia, so that when the saccharine urine is run into the boiling test solution, instead of a bulky red precipitate falling, which obscures the end reaction, the liquid simply loses its blue colour. The reagent is conveniently prepared as follows:

To 120 c.c. of ordinary Fehling's solution are added 300 c.c. of solution of ammonia, ·891 sp. gr., and 400 c.c. of 12 per cent. caustic soda solution (sp. gr. 1·137). The mixed solutions are then made up to 1 litre with distilled water. 100 c.c. of this solution = 10 c.c. of Fehling's solution. Allen has shown that Pavy's solution differs from Fehling's solution in its oxidising power on maltose and lactose. Its action on invert-sugar under the conditions named is only five sixths of that exerted by Fehling's solution. Therefore 120 c.c. of the latter are employed in making the ammoniacal solution instead of 100 c.c., as would be the case if they were strictly equivalent.

The process is as follows :—Dilute 10 c.c. of urine with 90 c.c. of 10 per cent. ammonia solution. Mix 20 c.c. Pavy's solution (= ·01 gram glucose) with an equal volume of water, and place in a 250-c.c. flask (Fig. 252) fitted with a rubber stopper pierced with two holes. The flask is then fixed to the nozzle of a tapped burette filled with the diluted ammoniacal urine. Through the second hole in the stopper is passed a bent glass tube, to which is attached a length of rubber tubing to convey the ammoniacal fumes given off during the titration, out of a window. The diluted Pavy solution in the flask is gently boiled, and kept boiling during the titration by a spirit lamp or Bunsen burner. The diluted urine is allowed to run cautiously in, a little at a time, until the blue colour in the flask is just discharged. Three titrations are made, and the mean result taken, when $\frac{100 \times \cdot 01}{\text{c.c. used}} =$ percentage of glucose in the sample. An allowance of 0·2 per cent., equal to three quarters of a grain per ounce, may be made for the reducing action of the creatine, creatinine, and other bodies normally present in urine.

Sir George Johnson's method.—The following solutions are required:

Standard solution ferric acetate, equal in tint to that yielded by a solution of glucose containing one grain per fluid ounce. This standard solution is prepared by mixing in the order named—

Solution of ferric chloride, sp. gr. 1·42	ʒj.
Glacial acetic acid, sp. gr. 1·058	ʒiv.
Solution of ammonia, sp. gr. ·959	ʒij.
Distilled water, sufficient to produce	℥iv.

Saturated solution of picric acid, prepared by boiling the crystals with distilled water in the proportion of six grains to one fluid ounce, and allowing the excess to crystallise out on cooling.

Solution of potash, B.P., sp. gr. 1·058. The apparatus is of

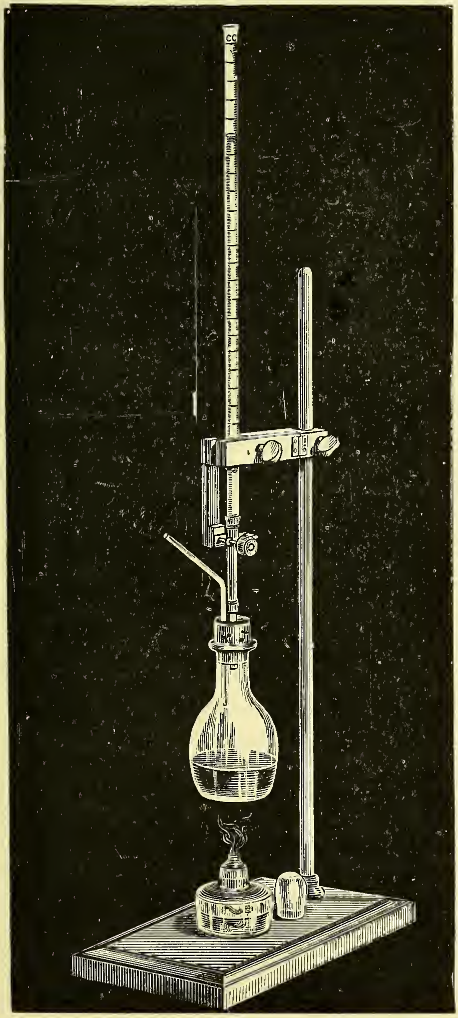

Fig. 252.—Apparatus arranged for sugar estimation by Pavy's method.

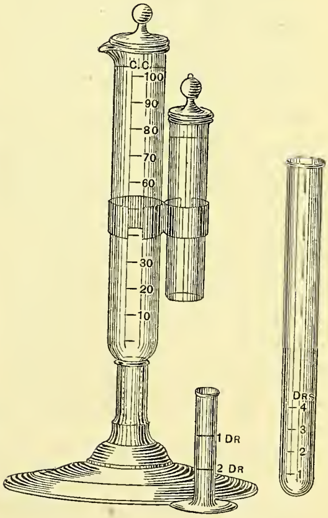

Fig. 253.—Johnson's picro-saccharometer.

a simple character and not costly. A tube about 12 inches in length, graduated into 100 c.c., with longer divisions at each 10 c.c., accurately stoppered and lipped. Another tube, half the above length, accurately stoppered, to hold the standard solution. A boiling tube, 10 inches long, $\frac{3}{4}$ inch diameter, lipped, and graduated at 4 fl. drs. (Fig. 253).

Method of performing the determination.—Measure 1 fl. dr. of urine into the boiling tube, add 1 fl. dr. of the picric acid solution and $\frac{1}{2}$ fl. dr. of solution of potash, and make up to the 4-dr. mark with distilled water. Heat over a small Bunsen flame, and keep the liquid boiling for about one minute. Cool by dipping the tube into cold water, and ascertain that the mixture measures exactly 4 drs.; if less make up with distilled water.

If the colour of the boiled urine is the same as that of the ferric acetate standard or paler, the urine is either free from sugar or contains less than one grain per fluid ounce.

It should be borne in mind that all normal urines reduce picric acid to an extent equivalent to from half to one grain of glucose per fluid ounce. This reduction is due to creatinine, and must be allowed for.

If the colour of the boiled liquid is darker than the standard, introduce it into the graduated tube until it stands at ten divisions, while the stoppered tube at the side is filled with the ferric acetate standard. Now dilute the dark red liquid in the graduated tube with distilled water till the colour is the same as the standard. Each division above ten represents 0·1 grain per fluid ounce. Thus thirteen divisions equal 1·3 grains, thirty divisions 3 grains, &c. If more than 6 grains per ounce are indicated, dilute the urine ten times, then analyse as before; but in this case each division on the saccharometer indicates 1 grain of sugar per fluid ounce. Thus, diluting from ten up to forty-eight divisions shows that the urine contains 48 grains of sugar per fluid ounce.

If the urine when ten times diluted gives a colour paler than the standard, it contains *less* than 10 grains of sugar per fluid ounce. Another portion should then be diluted five times by filling the graduated tube up to ten divisions with urine, and then up to fifty divisions with distilled water. The value of the divisions now will be half that with a ten times diluted sample. Thus eighteen divisions would indicate 9 grains per fluid ounce. If the urine has a specific gravity of 1035 or more, it should be at once diluted five or ten times before commencing an analysis.

The percentage of sugar in the urine may be ascertained by dividing the number of grains per fluid ounce by 4·375.

6. Determination of urea.—This is best performed by the hypobromite of soda method, which depends on the fact that when urea is treated with a solution of hypobromite of sodium, it is decomposed with liberation of nitrogen, according to the following equation :

$$CH_4N_2O + 3NaBrO = 3NaBr + CO_2 + 2H_2O + N_2.$$

It has been found by experiment that the whole of the nitrogen is not evolved from the urea, while some of that evolved may come from other nitrogenous constituents of urine, as creatinine, &c. This, however, is of little consequence, as a given specimen always evolves the same quantity of gas. The most convenient apparatus is that devised by Gerrard (Fig. 254). The small bottle shown on the right of the illustration contains a test-tube marked at 5 c.c. ; it is connected with the graduated vessel by means of a rubber cork and tubing. The two glass vessels are held together by a sliding spring clamp which allows the reservoir to be moved freely up and down, when finally adjusting the water level.

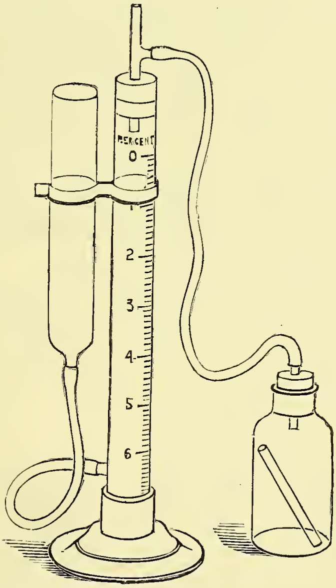

Fig. 254.—Gerrard's ureameter.

Water is poured into the reservoir until it reaches the zero-line in the measuring tube ; then inside the bottle is placed 25 c.c. of a freshly-made solution of sodium hypobromite, and inside this bottle a test-tube containing 5 c.c. of the urine to be tested. Fix the rubber corks securely so that no air can enter at any of the joints, and see that the water is at zero ; if not, open the small pinchcock at the top, and the water will at once return to zero ; then close the pinch-cock. Take the bottle in the hand, and incline it so that the urine upsets into the hypobromite ; effervescence at once follows, and the evolved nitrogen forces the water from the measuring vessel into the reservoir. The end of the determination is known by cessation of effervescence, when the bottle is placed in cold water to reduce the expansion caused by the heat evolved during the reaction. When all is cool, as seen by the steadiness of the water, the reservoir is lowered by means of the sliding clamp until the water-level corresponds, thus equalising the pressure.

The operation is now finished, and the figure at which the water rests is noted ; this represents percentage of urea. The instrument is graduated in one tenths, so that if the water stood two lines below 3, the quantity of urea indicated would be 3·20 per cent. Normal urine contains from 2 to 3 per cent. of urea.

To prepare the sodium hypobromite solution, 100 grams of caustic soda are dissolved in 250 c.c. of water, and to 23 c.c. of this solution 2·2 c.c. of bromine are added, giving the exact quantity for the analysis of 5 c.c. of urine.

To avoid the unpleasantness caused by the escape of bromine vapours, the latter may be used in sealed tubes ; one of these containing 2·2 c.c. is placed in a strong bottle with 23 c.c. of the caustic soda solution, when a sharp shake breaks the tube, and the test solution is ready for use.

Instead of using Gerrard's apparatus, the operation may be conducted in an ordinary nitrometer. Using the above quantities, each c.c. of nitrogen at normal temperature and pressure, equals ·0029 gram of urea in the 5 c.c. of urine tested.

7. DETECTION OF BILE : *Pettenkofer's test.*—To about 5 c.c. of urine in a test-tube add cautiously, so as to avoid much rise of temperature, about 2 c.c. of strong sulphuric acid. If necessary immerse the tube in ice-cold water during the operation. A pinch of powdered cane-sugar is then added and well mixed with the acid liquid, and another 2 c.c. of sulphuric acid poured in. As the temperature rises a reddish-violet coloration is produced if

bile is present, due to the cholalic acid liberated during the reaction.

Gmelin's test.—Filter the urine and place a few c.c. on a clean white plate. Then add a few drops of strong nitric acid. If bile is present, a play of colours varying from green to blue and red is seen on cautiously stirring with a glass rod.

Oliver's test.—

Beef peptone	2·00
Salicylic acid	0·25
Acetic acid (33 per cent.)	2·00
Distilled water, sufficient to produce	200·00

Dissolve, add a gram of clear kieselguhr, and filter through a well-wetted paper filter. The test solution must be perfectly brilliant. Dilute the urine until it has a sp. gr. of 1008, filter bright with kieselguhr, and add 1 c.c. to 3 c.c. of Oliver's reagent. If bile salts are present an opalescence appears, which is more or less distinct according to the quantity present.

8. DETECTION OF BLOOD.—Urine containing blood has a peculiar reddish smoky appearance, and always gives the reaction for albumen after filtration. *Dr. Day's* test gives a fine blue colour with blood, but the test is not a reliable one, as other organic bodies yield a similar reaction. The test consists in adding a little tincture of guaiacum (made freshly and from unoxidised resin) and an equal bulk of ethereal solution of hydrogen peroxide to the suspected urine. The blood-discs should always be looked for, and in hæmaturia are easily discernible; but in hæmoglobinuria only the red colouring matter of the blood is present, and no corpuscles can be recognised. In this latter case *Heller's test* may be made use of. Make the urine strongly alkaline with caustic soda solution, and heat to boiling. If blood is present a bottle-green coloration is produced, and a brownish-red precipitate consisting of earthy phosphates coloured with blood-discs thrown down. The *hæmin* test is also very reliable when no blood-discs are observable. Evaporate the deposit, which separates on standing, to dryness over a water-bath with a few grains of sodium chloride. Place a little of the residue on a microscopical slide, add a drop of glacial acetic acid, and warm. If blood pigments are present, microscopic crystals, of reddish-brown plates, of hæmin separate on cooling.

The diazo test for typhoid or acute tuberculosis consists in dissolving one gram of sulphanilic acid in 10 c.c. of hydrochloric acid, and diluting to 200 c.c. with water. To 50 c.c. of this reagent add 5 c.c. potassium nitrite solution (1 in 200) and 50 c.c. of urine. Make strongly alkaline with ammonium hydrate and shake. A bright red coloration is produced by urines above mentioned, and on standing for twenty-four hours a deposit forms, the upper part of which is green or black. Healthy urine gives no reaction.

9. Determination of uric acid.—Concentrate the urine, add a few drops of hydrochloric acid, and stand for twelve hours. Collect the deposit, wash first with distilled water, then with 90 per cent. alcohol, and weigh. *This gives low results.* Allen gives a more accurate process as follows. Nearly saturate 200 c.c. of urine with finely powdered ammonium chloride (about 30 grams). Allow to stand for two hours with occasional stirring, filter, and wash twice with a saturated solution of ammonium chloride. The precipitate, which consists of acid ammonium urate, $C_5H_3(NH_4)N_4O_3$, is treated with 10 c.c. of $\frac{N}{10}$ sulphuric acid, the liquid boiled for five minutes, cooled, and diluted to 200 c.c. with water. A few drops of a one per cent. solution of methyl orange are added, and $\frac{N}{20}$ caustic soda run in until the orange colour changes to yellow, the point of neutrality. Uric acid does not affect methyl orange. The difference between the volume of acid employed and the alkali required to neutralise it, represents the ammonia of the precipitate. Each c.c. of $\frac{N}{20}$ caustic soda solution used represents 0·0084 gram uric acid.

The acidity of urine may be determined by titrating 100 c.c. with $\frac{N}{10}$ caustic soda solution, using phenol-phthalein as an indicator. Each c.c. of $\frac{N}{10}$ solution represents 0·012 gram of sodium dihydrogen phosphate, to which salt the acidity of normal urine is assumed to be due.

10. Chemical examination of the deposit.—Warm a portion of the deposit with a little urine. Urates dissolve entirely. Deposits of urates are frequently coloured pink from the presence of the red pigment purpurin. If the deposit is not dissolved on heating, add a little acetic acid. This dissolves phosphates, which may be reprecipitated as triple phosphate by the addition of ammonia. If the deposit is still insoluble, it probably consists of either

oxalates which are soluble in hydrochloric acid, or of uric acid. In the latter case the crystals are often distinctly reddish, "cayenne pepper crystals," and dissolve on the addition of a caustic alkali. Uric acid may be confirmed by the "murexid" test. Place the crystals on a clean white dish, remove excess of moisture by means of blotting-paper, add a drop or two of strong nitric acid, and evaporate to dryness at a gentle heat. After cooling add a drop of ammonia, when a purple coloration will be produced.

11. Microscopical examination of the deposit.—Urinary deposits may be divided into two classes—anatomical and chemical. The former consist of such structures as blood, pus, epithelium, renal casts, spermatozoa, fungi, &c.; while included in the latter are uric acid, urates, phosphates, calcium oxalate, leucin, cystin, &c.

Even normal urine sometimes deposits phosphates and urates on standing, and it is not uncommon to find a few epithelial cells. Deposits for microscopical examination are obtained by allowing the urine to rest for a few hours in a conical test-glass; the method is not only very tedious, but the samples undergo such rapid changes on keeping that the results are untrustworthy. For rapid and accurate work a centrifuge is always employed.

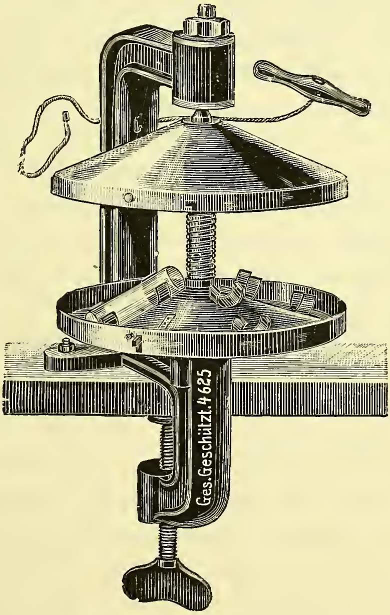

Fig. 255.—Urinary centrifuge.

The machine shown in Fig 255 is one of the simplest, and at the same time one of the most generally useful. A test-tube is completely filled with the urine, which has been previously gently agitated, and placed in the clip, a second tube containing water or another sample of urine being placed on the opposite side to balance the drum. The cover having been secured, a stout cord is wound tightly and evenly round the spindle, when by a steady and vigorous pull the machine is made to revolve at a rapid rate.

This centrifuge, which is nothing more than a large spinning top with a hollow body, revolving in cone bearings, separates the deposit very rapidly, while the most delicate organised structures are not injured by the high rate of speed employed. As soon as the machine comes to a standstill the test-tube is carefully removed, and the supernatant liquid poured off until only a drop or so remains. The tube is then gently shaken, and the deposit transferred to a microscopic slide, covered with a thin slip, and submitted to examination.

Note.—The following illustrations were photographed by the author from ordinary deposits, and as the same objective eyepiece and camera-stretch were used in each instance, they are strictly comparative as regards size.

Uric acid (Figs. 256—260).—Uric acid is one of the most common crystalline deposits in urine. The crystals, which are frequently of a reddish-brown colour, are readily seen by the naked eye. They are almost insoluble in boiling water, but dissolve directly on warming with caustic potash. Uric acid crystallises in a great variety of forms, the rhombic being the most frequent, with two of its angles rounded. At other times rhombic crystals may be found mixed with acicular prisms, tables, squares, and lozenges. One of the most peculiar forms is that known as the millstone, the crystals appearing exactly like a pair of millstones connected by a shaft or spindle (Fig. 259).

Urates.—The urates of sodium, potassium, and ammonium generally occur together as a brick-red precipitate, consisting of amorphous granules. These urates are almost insoluble in cold, but are fairly soluble in boiling water, and are generally associated with a deposit of triple phosphates. Ammonium urate occasionally crystallises in small spheres studded with spicules, to which the name of "thorn-apple" crystals is applied (Fig. 259).

Calcium carbonate spherules (Fig. 263) are sometimes observed in alkaline urine. These spherules are somewhat similar to those found in the urine of the horse and other herbivorous animals, but are much smaller. Under the microscope they are easily recognised by their effervescence with dilute acids.

Calcium oxalate (Fig. 262) crystallises in two distinct forms—octahedra and dumb-bells. The former consists of two four-sided pyramids, and when lying on their side are readily recognised, but when seen in the opposite diameter they appear as squares with a very bright cross in the centre. The dumb-bell form is not so frequently met with, but its very peculiar shape at once distinguishes it from any other salt. Oxalates may occur in either alkaline or acid urine. The crystals are insoluble in acetic acid, but are dissolved by hydrochloric acid.

Earthy phosphates.—Deposits of earthy phosphates only occur in alkaline urine. Crystalline phosphate of lime is rarely seen, but the ammonio-magnesian phosphate ($MgNH_4PO_4.6H_2O$) or triple phosphate (Figs. 264—266) is of common occurrence. The deposit appears as a bulky white precipitate, insoluble on warming, but readily soluble on addition of acetic acid. The regular form of the crystals is prismatic, but under certain conditions arborescent crystals form. The latter variety can always be reproduced by adding solution of ammonia to an acidified mixture of Epsom salts and sodium phosphate.

Leucin and *tyrosin* are crystalline bodies derived from the decomposition of proteids. The former occurs in spherical crystals which appear somewhat like oil globules, the latter always in tufts of needles that bear a striking resemblance to thistle-down (Fig. 267).

Cystin (Fig. 268) is very rarely seen. It crystallises in six-sided tablets, insoluble in boiling water, alcohol, or acetic acid, but soluble in caustic alkalies and mineral acids.

Blood-corpuscles (Fig. 269) are never found in normal urine. As previously stated, urine containing blood always has a smoky appearance. Under the microscope the corpuscles, if present in any quantity, are readily detected by their biconcave shape and yellowish tint, which distinguish them from pus corpuscles. Blood-corpuscles in liquid are generally separate, but occasionally they will be found cohering to one another like piles of coins.

Pus corpuscles (Fig. 270) are slightly larger than red blood-corpuscles, and present the appearance of granular discs, each containing one or more nuclei. When treated with acetic acid the nuclei become more distinct, but the granular appearance vanishes. Urine containing pus is turbid when freshly voided, and gives the reaction for albumen. On the addition of caustic potash the corpuscles swell up, forming a sticky mass which cannot be poured in drops. A deposit of pus somewhat resembles a mixed sediment of phosphates and urates. It will be remembered, however, that urates dissolve on warming, and phosphates on the addition of acetic acid, while a deposit of pus is only rendered more evident by this treatment.

Epithelial cells, derived from the mucous surface of the urinary tracts, may be found in any sample of urine, but under certain conditions the cells are thrown off in such quantities as to form a distinct deposit. It is extremely difficult to distinguish with certainty between epithelial cells. Fig. 271 illustrates the more commonly occurring forms.

Urinary casts are supposed to be moulds of the uriniferous tubules, resulting from the coagulation of some constituent of the blood, around which loose cells and epithelial débris have congregated. The diameter and general characters of the cast will be determined by the state of the uriniferous tubes at the time of formation. If the epithelium be abnormally adherent, the cast will be very narrow ; if, on the other hand, the epithelium is removed, it will be of the width of the tube. Should the epithelium be disintegrating, the cast will afford evidence of the change. If in a state of fatty degeneration, fat cells will be entangled in the cast. In hæmorrhage from any part of the secreting structure blood-corpuscles are present, and when suppuration occurs the cast contains pus corpuscles. When the transudation of the coagulable material occurs in a tube to which the epithelium is firmly adherent, or in a tube whose walls are smooth, the cast will be clear and perfectly transparent. It is supposed that these moulds contract slightly after formation, and in consequence readily find their way into the pelvis of the kidney, and are finally voided with the urine.

Casts of the renal tubes are rarely found unmixed with other deposits. Frequently they are accompanied with much epithelium, and in many cases blood-corpuscles are present in considerable number.

Epithelial casts (Figs. 272, 273).—These casts are highly refractive bodies. They are found in the most varied forms, some consisting of a hyaline body covered with epithelial cells, while occasionally the epithelium of the tubules may be thrown off intact. Very rarely casts are met with consisting entirely of aggregated epithelial cells.

Blood casts are sometimes met with in the deposit of the urine in acute nephritis. They consist of short cylinders forming more or less perfect moulds of the tubules, covered with large numbers of blood-corpuscles associated with epithelial cells.

Casts consisting entirely of *pus* are also very uncommon, although pus is occasionally seen associated with epithelium and granular matter.

Granular casts (Fig. 275) result from the degeneration of cell structures, and are recognised as finely granular, coarsely granular, pale or dark coloured, &c. The ends are generally broken, and fragments of epithelium and oil globules are found embedded in the body of the casts. In some instances a deposit of urates is formed on such casts.

Fatty casts (Fig. 276) are thrown off in cases of fatty degeneration of the kidney, but it must be borne in mind that cells containing oil globules are not invariably derived from tubes in a condition of fatty degeneration, and are not necessarily indicative of diseased kidney.

Many of these casts consist of altered epithelial cells enlarged and gorged with oil; in other instances the globules are very minute, and so crowded together that the mass appears by transmitted light to be perfectly opaque and dark, but if examined by reflected light it appears almost white.

Hyaline casts (Fig. 277).—These casts are very difficult to detect in urine on account of their great transparency. Little is known of their origin, although they are supposed to be derived from tubes in which the lining is entire. They are generally of small diameter, but often of considerable length. Under the microscope these casts present a smooth and glistening surface, studded at rare intervals with a few minute oil globules or small epithelial cells. It is possible that some of these very fine casts have been formed in tubes which have not fully developed.

Spermatozoa are frequently noticed in urinary deposits. When freshly ejected they exhibit a vibratile movement, but as seen in urine they are usually quiescent. A magnifying power of about 400 diameters is required to show them properly, although by the trained eye they may be recognised with a much smaller magnification.

12. METHOD OF DRAWING UP THE REPORT.—It now only remains to consider the best method of recording the results of an examination of a sample of morbid urine. This should be as brief as possible, and should be restricted to a plain statement of the observations made. The following may serve as a guide—

Urine Analysis

Sample marked. H. Smith. Nov. 25, '97.

Appearance.—Pale, slightly cloudy.

Reaction.—Almost neutral.

Specific gravity.—1024.

Albumen.—Trace.

Sugar.—Absent. (If any, state percentage as well as grains per ounce.)

Bile.—Absent.

Urea.—2·25 per cent.

A microscopical examination of the deposit showed crystals of triple phosphate, associated with epithelial cells of the bladder. Casts were not noticed, but a small number of pus corpuscles were present.

(Signed) John Robinson.

Extraneous matters met with in urinary deposits

The recognition of extraneous matters found in urinary deposits is of great importance, as unless the eye is familiar with the characters of such substances, it will be impossible to derive such information from a microscopical examination as will enable the observer to distinguish between bodies denoting the presence of morbid conditions, and matters which have accidentally found access to the urine.

Flax fibres (Fig. 278) and *cotton fibres* (Fig. 279) are very frequently met with in urine. It is hardly possible that either of these could be mistaken for casts, but short pieces, especially when covered with a crust of small crystals, occasionally present a peculiar appearance. The round, somewhat striated fibres of the former, and the twisted flattened ribbons of the latter, should be sufficiently characteristic for recognition.

Hairs.—Hair of various kinds is also of common occurrence. Human hair (Fig. 280) is very characteristic, and should be easily identified. *Cat's hairs* are somewhat similar to the foregoing, but have curious transverse markings. Blanket hairs (wool) may also be present, as well as fragments of dyed wool from rugs, &c. Fig. 281 shows a fragment of feather. A small fragment of a feather is also shown in Fig. 274.

Sputum.—Epithelial cells from the mouth are often found in the specimens of urine sent for examination, and must not be confounded with those from the urinary tracts.

Urine that has been passed some time, especially if much sugar is present, often contains fungoid growths. Fig. 282 shows the spores, and Fig. 283 the mycelium and sporangia of a quick-growing mould that had developed in a specimen of diabetic urine.

Urinary calculi.—Healthy urine contains several constituents which are only slightly soluble in water, and it not infrequently happens that some of these substances are slowly deposited in an insoluble form while yet in the bladder, or even before reaching that organ. The deposit may take the form of small granules and aggregations, or calculi of considerable size may be built up by regular depositions around a central nucleus.

Uric acid calculi.—From 80 to 90 per cent. of calculi found in human urine consists of uric acid. These consist of concentric layers of uric acid, generally deposited upon a nucleus of calcium oxalate. These calculi may be quite smooth, or covered with minute projections. The colour varies, the most frequent being a brownish red, although flesh-coloured calculi are often found. Uric acid calculi are insoluble in water, but if broken up, readily dissolve in solution of potash. Heated in a flame these calculi decompose with a disagreeable ammoniacal odour; the residue consists chiefly of calcium carbonate and phosphate. Confirm by applying the murexid test. Mix a little of the powdered calculus with nitric acid and evaporate to dryness. When cool add a drop of solution of ammonia; a beautiful purple colour at once appears.

Calculi composed of urates.—These calculi are much smaller and softer than the foregoing, and are of a lighter colour. When powdered they dissolve almost entirely in boiling water, the residue generally consisting of organic matter and calcium oxalate. Filter the solution and cool, when the urates will be again deposited. If urate of ammonium is present, it may be recognised by boiling the deposit with solution of potash, when ammonia is given off.

The only other forms of calculus that occur with any frequency are concretions of *calcium oxalate*. These from their colour and general appearance are known as "mulberry calculi," and are insoluble in boiling water or acetic acid, but readily dissolve in mineral acids.

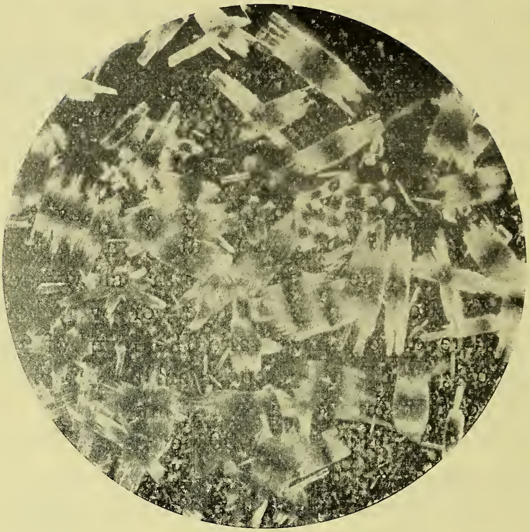

FIG. 256.—Uric acid.

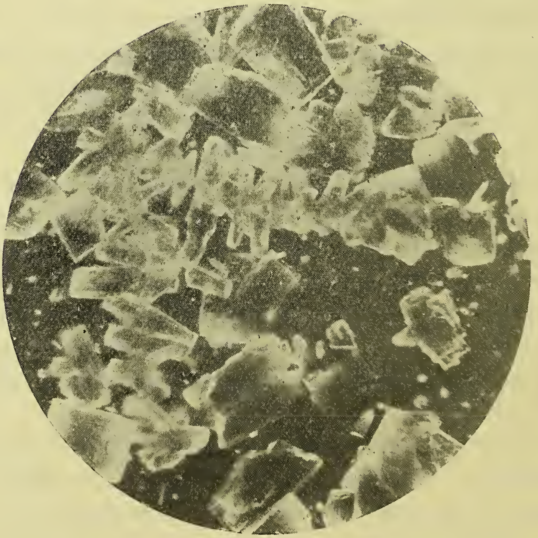

FIG. 257.—Uric acid.

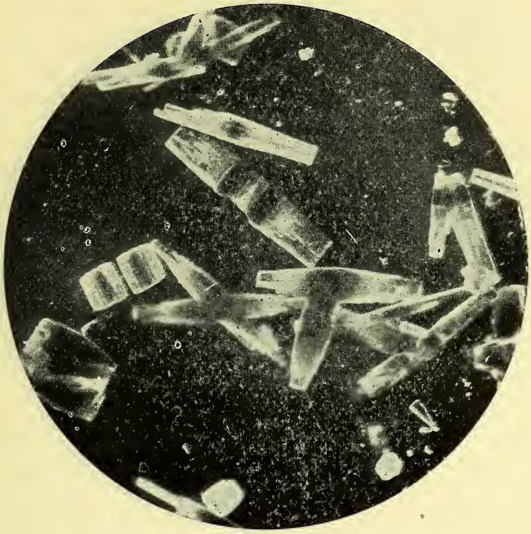

FIG. 258.—Uric acid.

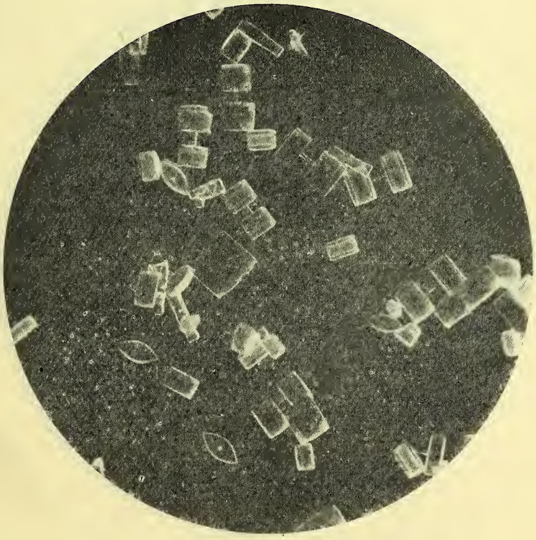

FIG. 259.—Uric acid.

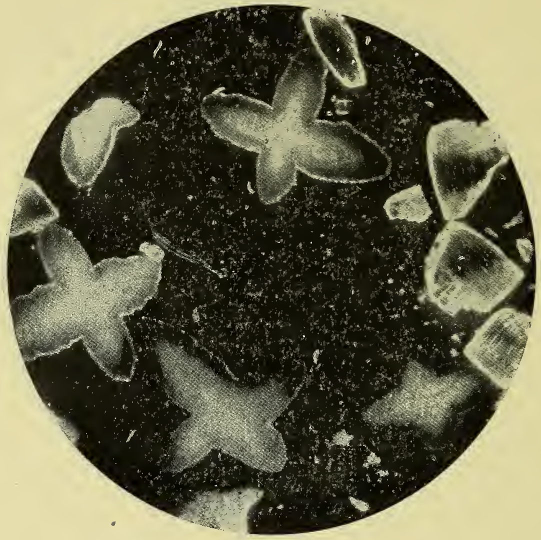

Fig. 260.—Uric acid.

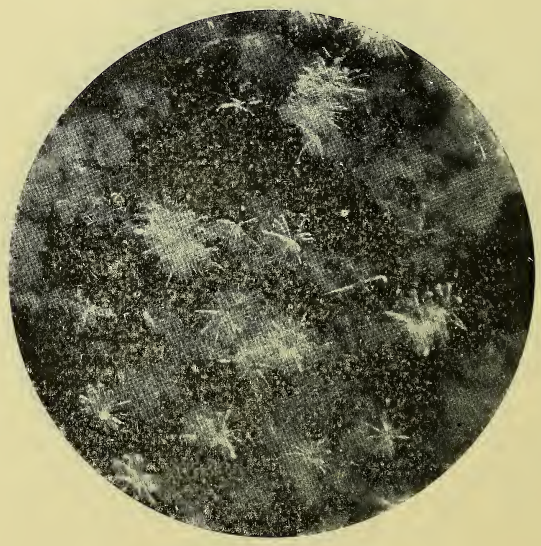

Fig. 261.—Ammonium urate.

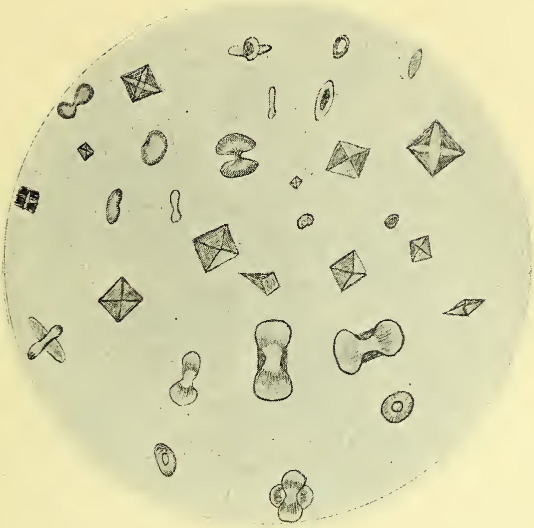

FIG. 262.—Calcium oxalate.

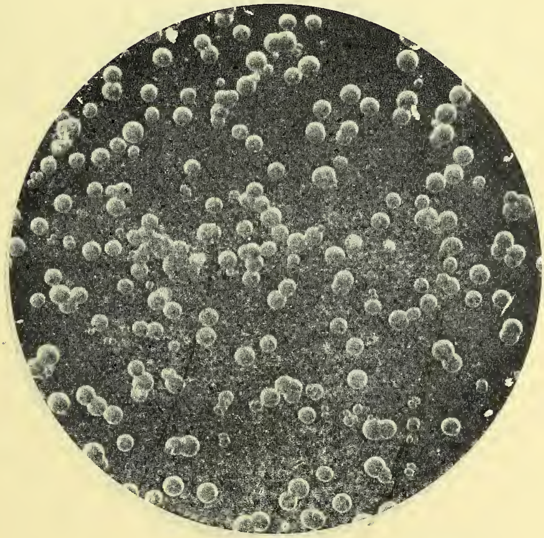

FIG. 263.—Calcium carbonate spherules.

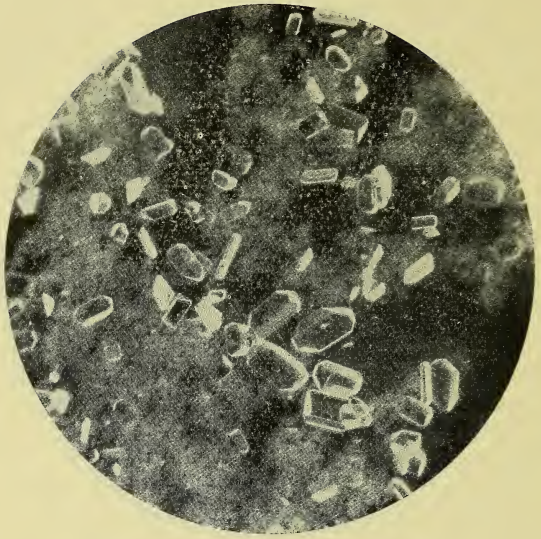

FIG. 264.—Triple phosphate.

FIG. 265.—Triple phosphate.

FIG. 266.—Triple phosphate.

FIG. 267.—Tyrosin.

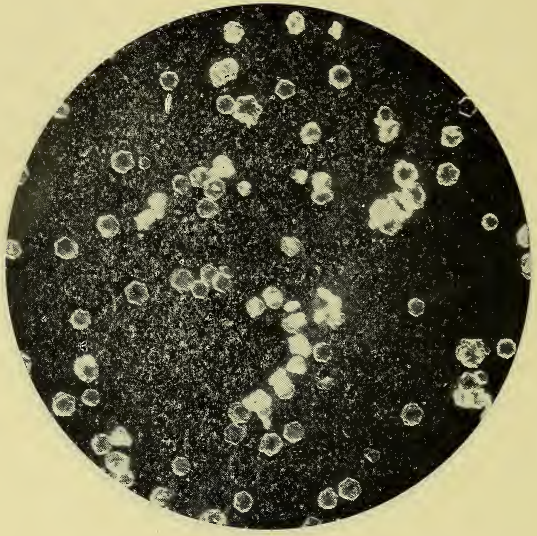

Fig. 268.—Cystin.

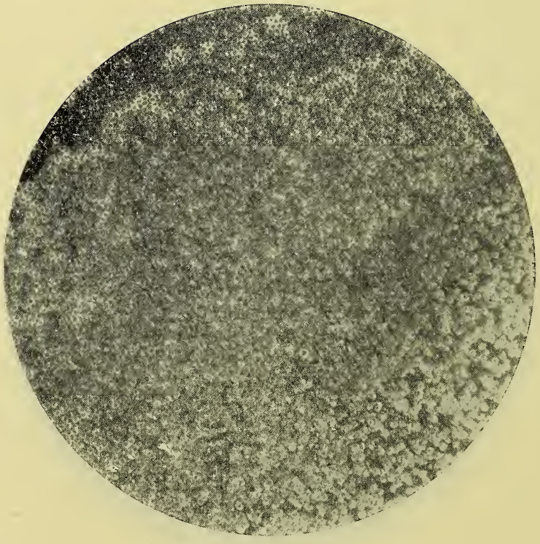

Fig. 269.—Blood-corpuscles.

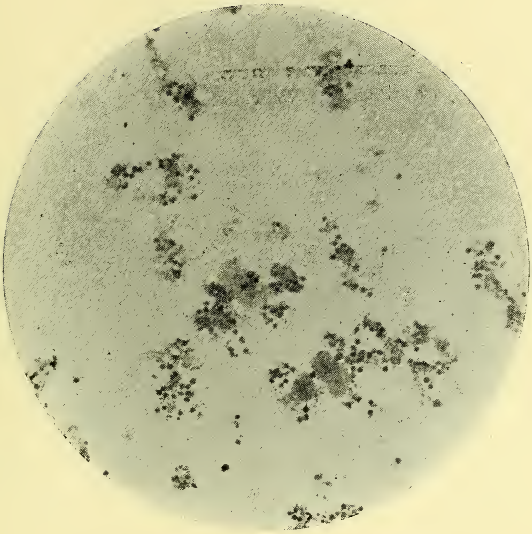

FIG. 270.—Pus corpuscles and epithelial cells from the bladder.

FIG. 271.—Epithelial cells.

A.	From male urethra.	D.	From female urethra.
B.	„ vagina.	E.	„ bladder.
C.	„ prostate.		

FIG. 272.—Epithelial casts.

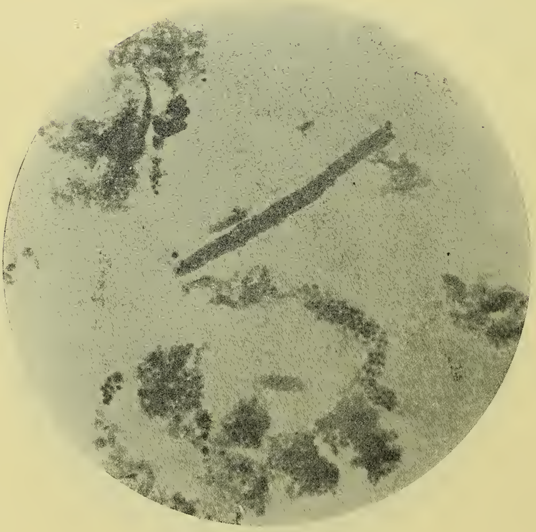

FIG. 273.—Epithelial casts.

FIG. 274.—Large casts containing oil.
Small epithelial cast.
Fatty cast.
Fragment of feather.

FIG. 275.—Granular casts.

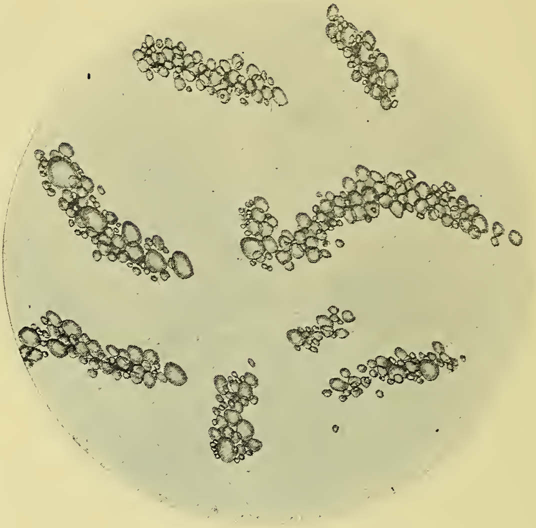

Fig. 276.—Fatty casts.

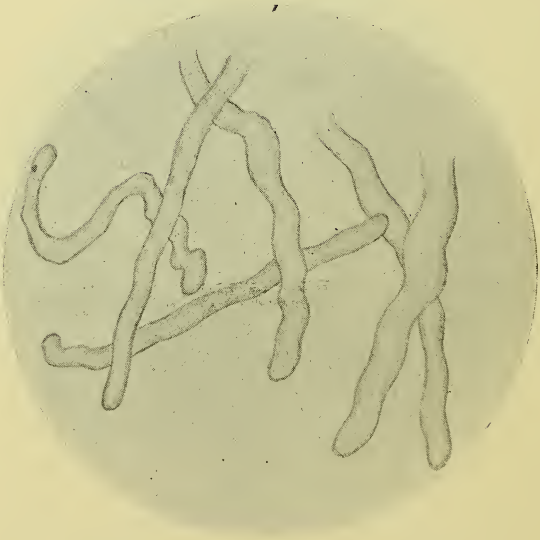

Fig. 277.—Hyaline casts.

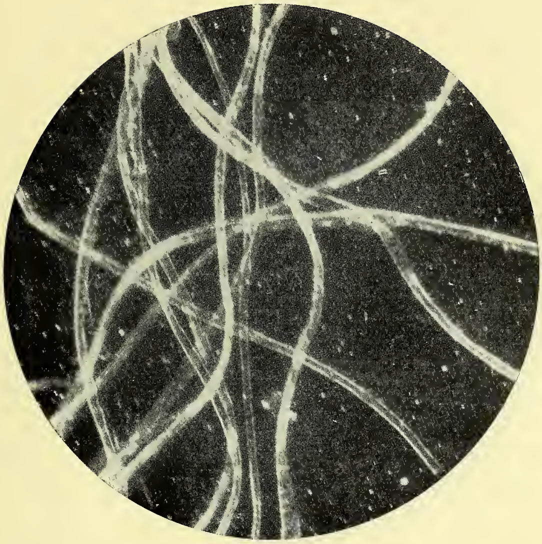

Fig. 278.—Flax fibres.

Fig. 279.—Cotton fibres.

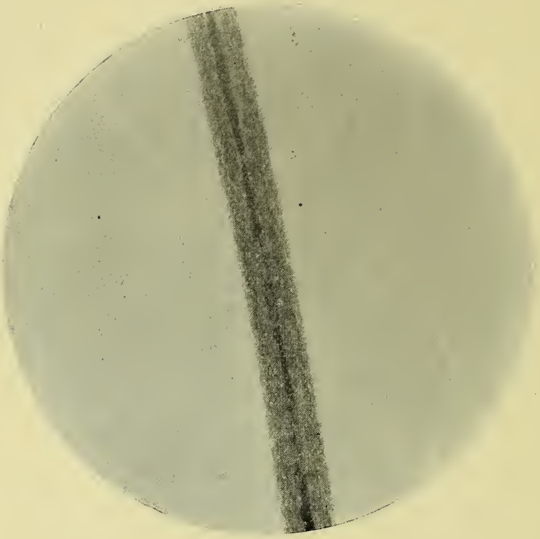

Fig. 280.—Human hair.

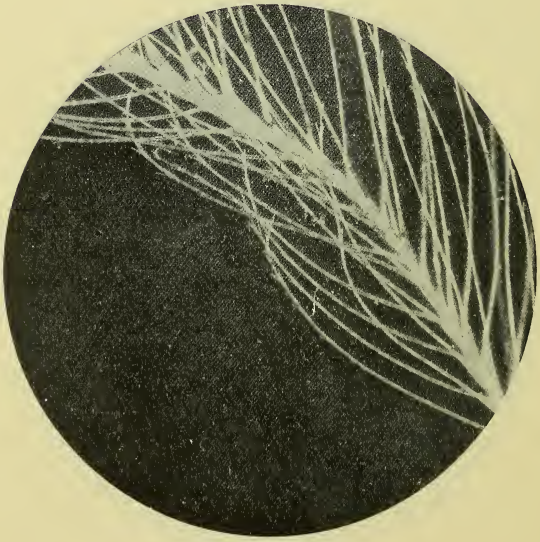

Fig. 281.—Feather.

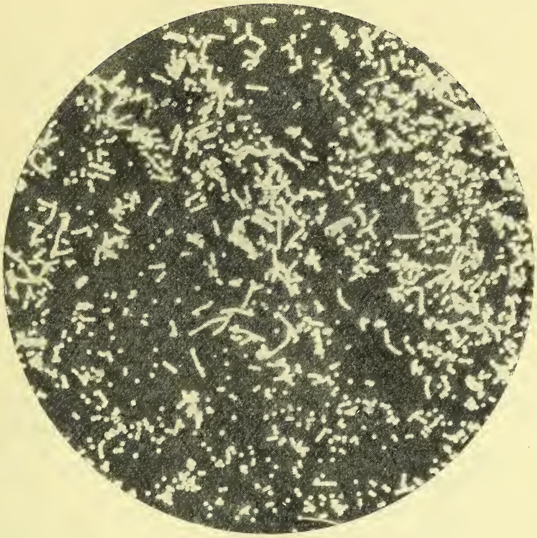

FIG. 282.—Spores of mould.

FIG. 283.—Mycelium and sporangia of mould.

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