THE ANALYSIS OF OILS AND ALLIED SUBSTANCES

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PREFACE

THIS brief account of the methods used in the Analysis of Oils, Fats, and Waxes has been written with the definite aim of presenting the subject in a form suited to the needs of the student and beginner, while at the same time including all recent developments likely to be found of value in practical work. In accordance with these objects, the chemistry of the various processes is explained in some detail, and methods which have been recently proposed are fully explained.

An attempt has been made to indicate the extent to which reliance may be placed on methods for detecting adulteration.

It is hoped that the subject has been treated throughout in such a manner that the book may serve as a laboratory guide for chemists who are not extensively engaged in oil analysis, or who have to deal with only a limited number of oils.

A selection has been made of what may be termed the stock processes for estimating each constant. The

substances dealt with have also been subjected to a process of selection, in which those of no technical interest were excluded.

In indicating original papers, reference is made whenever possible to the *Journal of the Society of Chemical Industry* (*J.S.C.I.*), to which practically all chemists and students have access.

Thanks are due to Messrs. Baird & Tatlock, Limited, who have kindly supplied blocks for several of the illustrations in the volume.

A. C. WRIGHT.

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September 1902.

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THE ANALYSIS OF OILS

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CHAPTER I

THE OCCURRENCE AND COMPOSITION OF OILS, FATS, AND WAXES

General.—Any liquid, insoluble in water and possessing the peculiar slippery feel which cannot be described better than by the adjective 'oily,' may in ordinary parlance be termed an 'oil.' Those oils which are the products of animal and vegetable life may be divided broadly into two classes: volatile oils, which are also termed 'essential' or 'ethereal' oils, and non-volatile or 'fixed' oils, which may be termed 'fatty oils,' in order to distinguish them from the former class and from other oils, which are not (directly at least) produced by living organisms. It is this latter class of fatty oils that we have especially to consider in the course of this book; it is therefore to be understood that the term 'oil,' without further qualification, must be here taken to mean 'fatty oil.' The difference between an oil and a fat is merely one of physical condition, due to

temperature ; a fat melts to an oil, and an oil when frozen forms a fat. Consequently, the same substance may be known as an oil in one country and climate, and as a fat in another and colder country and climate. As will appear later, the oils and fats all exhibit great similarity in chemical composition.

The waxes are also products of animal and vegetable life, they differ from the fatty oils in composition ; in respect of external properties, they range from the very fluid sperm oil to the brittle carnaüba wax.

Sources.—In the vegetable kingdom oils and fats may be found disseminated in minute quantity throughout the entire plant, but the considerable deposits which can be economically extracted are found in the fruits of certain plants, and especially in the seeds, where the fat serves the purpose of a reserve material, which is subsequently converted into carbohydrates, or undergoes other changes, in order to form the material for the tissues of the young plant. Vegetable waxes are excreted on the surface of leaves, probably for protective purposes.

In the bodies of animals fats are found in large deposits and also scattered throughout the tissues ; they apparently serve the purposes of protection and of a reserve material, by which the animal processes may be maintained in the absence of food. Milk fat has, of course, a different function. The function of the animal waxes, with the exception of beeswax, is somewhat uncertain.

Composition.—Oils, fats, and waxes are mainly composed of *esters*—the salt-like compounds of acids with alcohols—in which the alcohol radical plays the part of the

metal in a salt. Let us first consider a simple case: by the action of acetic acid on ethyl alcohol, the ester ethyl acetate is produced:

$$CH_3.CO.O\,H + HO\,C_2H_5 = CH_3.CO.OC_2H_5 + H_2O$$

Contrast this equation with the one which represents the action of acetic acid on caustic potash:

$$CH_3.CO.O\,H + HO\,K = CH_3.CO.OK + H_2O$$

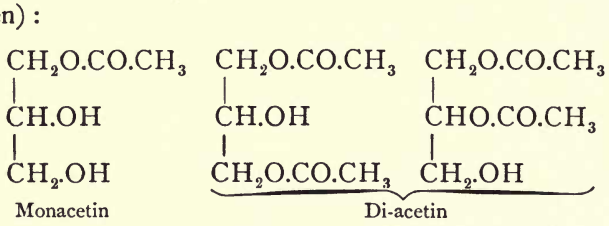
It is seen that water is formed in each case, and that, in the ester resulting from the first reaction, the ethyl radical, C_2H_5, takes the place of the potassium in the potassium acetate formed in the second reaction.

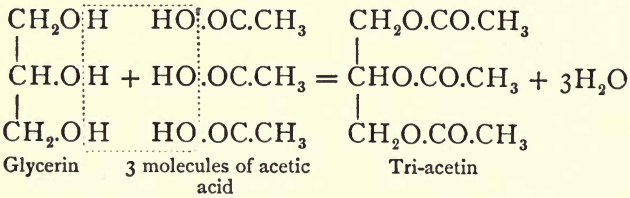
If, now, in place of ethyl alcohol and acetic acid, we consider the compound of myricyl alcohol, $C_{30}H_{62}O_2$, and palmitic acid, $C_{16}H_{32}O_2$ or $C_{15}H_{31}.CO.OH$, we have myricyl palmitate, $C_{30}H_{61}O.CO.C_{15}H_{31}$, which is precisely analogous to ethyl acetate, $C_2H_5O.CO.CH_3$.

Myricyl palmitate, which is the principal constituent of beeswax, contains simply the more complex radicals $C_{30}H_{61}$ and $C_{15}H_{31}$ in place of the simpler radicals C_2H_5 and CH_3 in ethyl acetate. The two compounds being built upon the same plan from similar components exhibit, in the main, similar reactions. Both solid and liquid waxes are essentially composed of the esters of monatomic alcohols with monovalent acids.

The esters, which form the chief constituents of the fatty oils and fats are not derived from a monatomic alcohol such as ethyl or myricyl alcohol, but from the triatomic alcohol glycerin, $C_3H_8O_3$. The constitution of glycerin is expressed by the following formula:

This formula indicates that the molecule of glycerin contains three hydroxyl (OH) groups, each of which has the same alcoholic properties as the single hydroxyl group in the molecule of ethyl alcohol. By the progressive action of acetic acid (or its anhydride) on glycerin each of the three hydroxyl groups reacts in turn with the acid, thus producing compounds of the following formulæ (the equation for the formation of tri-acetin is alone given):





The glycerin esters of acetic acid are known as *acetins*, the terms monacetin, di-acetin, and tri-acetin being applied to the esters produced by the action on one molecule of glycerin of one, two, or three molecules respectively of acetic acid. It is to be noted that there are two possible formulæ for di-acetin, both of which are given above, according to the particular two hydroxyl groups which

react with the acid. It will be apparent that a second formula for monacetin is also possible. No doubt it would be possible to make two isomeric monacetins and two isomeric di-acetins.

If now, in place of the simple acetic acid, we suppose oleïc acid, $C_{17}H_{33}.CO.OH$, to act on glycerin, we shall obtain the three esters corresponding to the three acetins, which are termed monoleïn, di-oleïn, and tri-oleïn respectively.

Neglecting the possibility of the existence of isomeric monoleïns and di-oleïns, the formulæ of the three oleïns may be written as follows

Monoleïn	Di-oleïn	Tri-oleïn
$CH_2O.CO.C_{17}H_{33}$	$CH_2O.CO.C_{17}H_{33}$	$CH_2O.CO.C_{17}H_{33}$
$CH.OH$	$CHO.CO.C_{17}H_{33}$	$CHO.CO.C_{17}H_{33}$
$CH_2.OH$	$CH_2.OH$	$CH_2O.CO.C_{17}H_{33}$

As a matter of fact, Berthelot obtained these three compounds by heating glycerin and oleïc acid together at high temperatures. Tri-oleïn is an important component of a very large number of oils and fats, and all the other chief components of the fatty oils are constituted in the same manner; they simply contain the radicals of other acids in place of that of oleïc acid in the above formula for tri-oleïn.

The esters of glycerin are known generally as *glycerides*; thus monoleïn is spoken of as the monoglyceride of oleïc acid, di-oleïn as the diglyceride, and tri-oleïn as the triglyceride. It is to be noted that diglycerides may contain the radicals of two, and triglycerides of three, different acids; the compounds would then be known as *mixed*

glycerides—for example, the diglyceride of acetic and propionic acids would have the formula $C_3H_5(OH)(C_2H_3O_2)(C_3H_5O_2)$, the triglyceride of acetic, propionic, and butyric acids would be $C_3H_5(C_2H_3O_2)(C_3H_5O_2)(C_4H_7O_2)$ (The formulæ are here written somewhat compressed, instead of in the extended form used above, in order to economise space.) Whilst in all probability the chief constituents of fats and oils are simple triglycerides, the occurrence of diglycerides and mixed glycerides in several oils has now been proved. Thus Reimer and Will found di-erucin in the solid fat deposited by rape oil; Bell and Blyth and Robertson extracted from butter a glyceride which contained oleïc, palmitic, and butyric acids; Heise, and later Henriques and Künne (*J.S.C.I.*, 1899, 377, 590, 693), obtained from Mkani fat a glyceride which is to be regarded as oleodistearin, $C_3H_5(C_{18}H_{33}O_2)(C_{18}H_{35}O_2)_2$; Holde and Stange (*J.S.C.I.*, 1901, 1003; 1902, 126) have separated from olive oil oleodimargarin, $C_3H_5(C_{18}H_{33}O_2)(C_{17}H_{33}O_2)_2$; Klimont (*J.S.C.I.*, 1901, 1121; 1902, 486) regards cocoa butter as consisting mainly of the mixed glyceride of oleïc, palmitic, and stearic acids, $C_3H_5(C_{18}H_{33}O_2)(C_{16}H_{31}O_2)(C_{18}H_{35}O_2)$.

Properties of the Glycerides.—The pure glycerides are colourless, tasteless, and odourless, and solid or liquid according to the acid or acids they contain. The natural oils owe their characteristic colours, flavours, and odours to small quantities of substances other than glycerides. The natural glycerides are practically insoluble in water, but they themselves dissolve a small quantity of water, which is evolved (as steam) on heating. They dissolve in a small volume of ether, petroleum ether, carbon bisul-

phide, chloroform, acetone, &c.; in absolute alcohol they are somewhat soluble, but in more dilute alcohol only to a slight extent. The glycerides of the hydroxy fatty acids (ricinoleïc acid), however, behave in a different manner; they dissolve readily in alcohol, but are insoluble in petroleum ether. On heating, the glycerides generally remain undecomposed below 250° C., above which temperature they soon decompose, producing a variety of volatile substances, among which is acroleïn, a product of the decomposition of glycerin.

By the action of alkalis, esters are decomposed into their constituent alcohols and acids, the latter of course forming salts. In the case of ethyl acetate, the reaction is represented by the equation:

$$C_2H_5.O.CO.CH_3 + K.OH = C_2H_5.OH + KO.CO.CH_3$$

The action of alkalis on the glycerides is quite similar, *e.g.* tristearin is decomposed according to the following equation:

$$\begin{array}{l} CH_2.O.CO.C_{17}H_{35} \\ | \\ CH.O.CO.C_{17}H_{35} \\ | \\ CH_2.O.CO.C_{17}H_{35} \end{array} \begin{array}{l} K.OH \\ \\ K.OH \\ \\ K.OH \end{array} = \begin{array}{l} CH_2.OH \\ | \\ CH.OH \\ | \\ CH_2.OH \end{array} + \begin{array}{l} KO.CO.C_{17}H_{35} \\ \\ KO.CO.C_{17}H_{35} \\ \\ KO.CO.C_{17}H_{35} \end{array}$$

i.e. one molecule of tristearin and three molecules of caustic potash produce one molecule of glycerin and three molecules of potassium stearate. The salts of the acids contained in the fats are commonly known as 'soaps,' and since the production of soap is the most important reaction in which this decomposition of esters is utilised, the term 'saponification' is applied broadly to the resolution of any ester into its

component alcohol and acid by the action of alkalis. We shall see shortly that glycerides may be decomposed into their component alcohols and acids by other means than the action of alkalis. The term 'saponification' is applied broadly, though somewhat loosely, to all these reactions, whether a soap is actually produced or not.

The saponification process has recently been examined on several sides and results have been obtained of great importance both from the analytical and manufacturing points of view. Henriques has found (*J.S.C.I.*, 1896, 299, 476; 1897, 746; 1898, 673, 853) that, in saponifying fats in the cold by alcoholic potash, a quantity of potash as little as 15 per cent. of that theoretically necessary is sufficient to produce almost complete decomposition of the glycerides. The product of the reaction then consists mainly of the ethyl esters of the acids contained in the glycerides, together with the soap obtained from the small quantity of potash. Geitel (*J. prakt. Chem.*, 1897, 429; 1898, 113) and Lewkowitsch (*J.S.C.I.*, 1898, 1107; 1899, 1031; 1900, 254) have shown that the saponification of triglycerides takes place in three stages, in the first of which the triglyceride produces diglyceride and one molecule of soap, the diglyceride then in the same manner forming monoglyceride, and this subsequently free glycerin. Thus there are present at any moment during the process: triglyceride, diglyceride, monoglyceride, glycerin, alkali, and soap.

The process of saponification has so far been represented as due to the action of alkalis, which is not, strictly speaking, the case; it is rather a *hydrolysis*, that is, a decomposition in which the elements of water are taken up.

The esters are in fact decomposed by water at the ordinary temperature, but at an extremely slow rate, the hydrolysis being enormously accelerated by heating under pressure or by the presence of acids or alkalis, which latter serve to neutralise, and remove from the sphere of the reaction, the acid produced. In the candle industry, in the manufacture of stearic acid, fats are entirely hydrolysed by heating them under pressure in an autoclave with water and a quantity of lime or magnesia much below the amount required for complete saponification, or by heating at 120° C. with strong sulphuric acid and then boiling with water. It has also been proposed to hydrolyse fats for this purpose by heating with water only at very high pressures, but the process does not appear to have been extensively adopted. Klimont (*J.S.C.I.*, 1902, 126) has investigated the action of steam on fats at various pressures; at lower pressures (seven atmospheres) the different fats were hydrolysed to different extents, but the differences disappeared at higher pressures. The following figures, calculated from Klimont's results, give the percentages of free acid (as oleïc acid) present in olive oil (originally neutral) after heating for six hours in an autoclave with steam of various pressures.

Pressure, atmos.	Free acid, calculated as oleïc, per cent.	Pressure, atmos.	Free acid, calculated as oleïc, per cent.	Pressure, atmos.	Free acid, calculated as oleïc, per cent.
3	3·2	7	26·7	13	54·6
5	17·8	10	31·4	15	84·2
6	21·0	—	—	—	—

When exposed to the action of air and light, oils and fats, with the exception of the drying oils, become rancid—

i.e. acquire an unpleasant odour and taste. The causes of the change are not yet properly understood ; it is regarded by some as due to bacterial action, by others as an oxidising process which takes place under the influence of light. Whatever the cause of fats turning rancid may be, the change is certainly accompanied by oxidation both of the glycerin and fatty acids of the glycerides, and also probably by hydrolysis of the glycerides, with the production of free acids and lower fatty acids. The amount of free acid, however, bears no relation to the degree of rancidity, as measured by the unpleasantness of taste and odour.

The drying oils, when exposed in a thin layer to the action of air, absorb oxygen, and are converted finally into a more or less hard elastic substance. Other reactions of natural glycerides, which are characteristic of the different acids entering into their composition, will be treated under the heading of the acids.

The following glycerides, which are found in fats or are allied to those found in fats, have been obtained in a more or less pure condition ; they are therefore briefly described.

Tri-acetin, $C_3H_5(C_2H_3O_2)_3$, is obtained by boiling glycerin with glacial acetic acid for a long time. Mon-acetin and di-acetin are formed at the same time ; they are separated from tri-acetin by means of their greater solubility in water. Pure tri-acetin is readily soluble in alcohol, ether, and benzene, and soluble in water to some extent. Its density at 15° C. is 1·1606 ; it boils at 172–172·5° C. under ordinary pressure.

Tributyrin, $C_3H_5(C_4H_7O_2)_3$, is obtained by heating normal butyric acid with glycerin *in vacuo*, the water

formed being carried away by a slight current of air. It is a colourless liquid, which does not solidify at −70° C.; it boils at 186° C., its density is $\frac{20°}{4°}$ 1·0324. (Scheij, *Rec. trav. chim. Pays-bas*, 18, 189.)

Tricaproïn, $C_3H_5(C_6H_{11}O_2)_3$, is formed from normal caproïc acid under the same conditions as tributyrin. It is a colourless, odourless, and tasteless liquid, which solidifies at −60° C., mixes with 85 per cent. alcohol, and has the density $\frac{20°}{4°}$ 0·9867.

Tricaprylin, $C_3H_5(C_8H_{15}O_2)_3$, is also obtained in a similar manner to tributyrin, which it resembles in properties. It melts at 8° C.; its density is $\frac{20°}{4°}$ 0·954.

Tricaprin, $C_3H_5(C_{10}H_{19}O_2)_3$, similarly produced, is a crystalline solid, which melts at 31·1° C.; its density is $\frac{40°}{4°}$ 0·9205. It is readily soluble in warm alcohol, ether, &c.

Trilaurin, $C_3H_5(C_{12}H_{23}O_2)_3$, melts at 46·4° C.; its density is $\frac{60°}{4°}$ 0·8944.

Trimyristin, $C_3H_5(C_{14}H_{27}O_2)_3$, melts at 56·6° C.; its density is $\frac{60°}{4°}$ 0·8848. After heating to 57–58° C., trimyristin melts at 49° C., becomes solid again at 50° C., and then melts at the original melting-point of 56·6° C.

Tripalmitin, $C_3H_5(C_{16}H_{31}O_2)_3$, may be obtained from palm oil by first extracting free acids with alcohol and then repeatedly recrystallising the residue from ether. It may also be obtained by the process of Scheij (see above). Tripalmitin melts at 65·1° C.; after solidifying it melts at

45–46° C.; it again becomes solid, and finally melts at 65·1° C.; its density is $\frac{80°}{4°}$ 0·8657.

Tristearin, $C_3H_5(C_{18}H_{35}O_2)_3$, may be obtained by Scheij's method, or by heating monostearin with stearic acid at 275° C. It melts first at 55° C., then becomes solid, and melts finally at 71·6° C.; the density is $\frac{80°}{4°}$ 0·8621.

Tri-oleïn, $C_3H_5(C_{18}H_{33}O_2)_3$, is obtained by heating glycerin with oleïc acid at 240° C.; it is a liquid which distils *in vacuo* without decomposition.

Di-erucin, $C_3H_5(OH)(C_{22}H_{41}O_2)_2$, separates from rape oil on long standing. It crystallises from a mixture of ether and alcohol in silky needles, which melt at 47° C.

Tri-erucin, $C_3H_5(C_{22}H_{41}O_2)_3$, is obtained by heating di-erucin with erucic acid at 300° C. It melts at 31° C.

Triricinoleïn, $C_3H_5(C_{18}H_{33}O_3)_3$, is obtained by heating glycerin with ricinoleïc acid at 230° C. It is a colourless oil, which mixes with absolute alcohol, but not with petroleum ether. Artificial triricinoleïn differs from castor oil, which is almost entirely composed of triricinoleïn, in not giving a solid ricinelaïdin by the action of nitrous acid.

Oleodimargarin, $C_3H_5(C_{18}H_{33}O_2)(C_{17}H_{33}O_2)_2$, was obtained by Holde and Stange by repeatedly recrystallising olive oil from ether at very low temperatures. It is a white porcelain-like mass, which melts at 29° C. or 31° C., according as it has been previously fused or not.

Oleodistearin, $C_3H_5(C_{18}H_{33}O_2)(C_{18}H_{35}O_2)_2$, is contained in Mkani fat (see p. 6). It melts at 45–46° C., and after previous fusion and solidification at 39–40° C. After rapid cooling of the fused glyceride, Heise obtained the

melting-point of 27° C.; on then heating slowly crystals reformed, which melted at 37–38° C.

Oleopalmitostearin, $C_3H_5(C_{18}H_{33}O_2)(C_{16}H_{31}O_2)(C_{18}H_{35}O_2)$, was obtained from cocoa-butter by Klimont (see p. 6), by fractional crystallisation from acetone, in the form of white plates, which melted at 31·4° C.

After this brief description of the few glycerides, which occur in the fats and have also been prepared in the pure state, we have next to consider the acids and alcohols which are the constituents of the glycerides and other esters.

THE ACIDS

The acids which occur in oils and fats are monobasic; they belong to the acetic acid series of saturated acids, the general formula of which is $C_nH_{2n}O_2$; to the unsaturated series, which have the general formulæ $C_nH_{2n-2}O_2$, $C_nH_{2n-4}O_2$, $C_nH_{2n-6}O_2$, $C_nH_{2n-8}O_2$; and finally to the series of unsaturated hydroxy-acids $C_nH_{2n-2}O_3$.

ACIDS OF THE ACETIC SERIES $C_nH_{2n}O_2$.

The acids of this series which are found in fats are:

Acetic acid, $C_2H_4O_2$.
Butyric acid, $C_4H_8O_2$.
(Iso-)Valeric acid, $C_5H_{10}O_2$.
Caproïc acid, $C_6H_{12}O_2$.
Caprylic acid, $C_8H_{16}O_2$.
Capric acid, $C_{10}H_{20}O_2$.
Umbellulic acid, $C_{11}H_{22}O_2$.
Lauric acid, $C_{12}H_{24}O_2$.

Myristic acid, $C_{14}H_{28}O_2$
Palmitic acid, $C_{16}H_{32}O_2$.
Margaric acid, $C_{17}H_{34}O_2$
Daturic acid, $C_{17}H_{34}O_2$.
Stearic acid, $C_{18}H_{36}O_2$.
Arachidic acid, $C_{20}H_{40}O_2$.
Behenic acid, $C_{22}H_{44}O_2$.
Lignoceric acid, $C_{24}H_{48}O_2$.
Cerotic acid, $C_{27}H_{54}O_2$.
Melissic acid, $C_{30}H_{60}O_2$.

The gradual change in the properties of these acids, as the molecular weight rises, is to be noticed. The melting- and boiling-points rise: the lower members are liquids and can be distilled under ordinary pressure; the higher members of the series are solids, and can only be distilled without decomposition under reduced pressure. At the same time there is a diminution in the solubility both of the acids and their salts to such an extent that the higher members of the series, which are the most important, are only slightly soluble in cold alcohol, and only their alkali salts are soluble in water, whilst, on the other hand, all metallic acetates are soluble.

The acids of this series are saturated—i.e. they contain no ethylene or acetylene linkages, consequently they do not unite with iodine, sulphuric acid, &c.

Acetic acid, $C_2H_4O_2$, is present as the triglyceride in small quantities in various fats. It is not necessary to describe this acid in detail here.

Butyric acid, $C_4H_8O_2$. The triglyceride of normal butyric acid is present in butter to the extent of 6 per

cent. Butyric acid is a liquid with an odour resembling acetic acid, which, however, on dilution becomes unpleasant and resembles that of rancid butter. It solidifies at −19° C., and melts at −2° to +2° C. (Linnemann), −4·5° to −2° (Zander), −7·9° C. (Schneider). The boiling-point is 162–163° C., and the density $\frac{19°}{4°}$ 0·9599, $\frac{81°}{4°}$ 0·8983. Butyric acid mixes with water, alcohol, and ether in all proportions; it is separated from the solution in water on the addition of calcium chloride. It is readily volatile with steam. Butyric acid is oxidised by boiling nitric acid to succinic acid, by chromic and sulphuric acids (under certain conditions only) to carbon dioxide and acetic acid, by alkaline permanganate to carbon dioxide, and by acid permanganate to carbon dioxide and acetic acid.

The salts of butyric acid are generally soluble in water. The calcium salt, $Ca(C_4H_7O_2)_2.H_2O$, crystallises in pearly leaflets, which dissolve in about 3·5 parts of water at the ordinary temperature. The salt is less soluble in warm water, so that a considerable precipitate is obtained on heating the cold saturated solution; the minimum solubility is at about 70° C. The barium salt, $Ba(C_4H_7O_2)_2.4H_2O$, also crystallises in pearly leaflets and has a minimum solubility at about 40° C. Silver butyrate is soluble in about 200 parts of cold water and 70 parts of hot water; the hot solution deposits needles on cooling.

Valeric acid. The modification of valeric acid which occurs in fats is isovaleric (isopropylacetic) acid, $(CH_3)_2.CH.CH_2.CO_2H$. It is a liquid with a clinging odour, which is very unpleasant when dilute. It boils at 173·7°

C. under 760 mm. pressure (Kahlbaum), solidifies at −57° C., and melts at −51° C. It dissolves in 23·6 parts of water at 20° C., and is separated from the solution by soluble salts. Chromic acid oxidises isovaleric acid to carbon dioxide and acetic acid. The calcium and barium salts are readily soluble in water. Silver isovalerate crystallises in leaflets, of which 100 parts of cold water dissolve 0·18 part.

Caproïc acid, $C_6H_{12}O_2$. Isobutylacetic acid, $(CH_3)_2.CH.CH_2.CH_2.CO_2H$, occurs in butter as the glyceride. This caproïc acid is a colourless oil of unpleasant odour, which does not solidify at −18° C., and boils at 207·7° C. (corr.); it is somewhat soluble in water. The calcium and barium salts are soluble.

Caprylic acid, $C_8H_{16}O_2$. The normal acid is contained in butter and cocoanut oil. Caprylic acid crystallises in the cold in leaflets, which melt at 16·5° C.; it boils at 237·5° C (corr.). It dissolves in 400 parts of boiling water, from which it almost completely separates on cooling. The calcium and barium salts are only slightly soluble in water.

Capric acid, $C_{10}H_{20}O_2$, crystallises in fine needles, which melt at 31·3–31·4° C. and boil at 268–270° C. The acid is almost insoluble in cold water and very slightly in boiling. The calcium and barium salts are almost insoluble, only the alkali salts dissolve readily.

Umbellulic acid, $C_{11}H_{22}O_2$, melts at 21–23° C., and boils at 275–280° (corr.) without decomposition.

Lauric acid, $C_{12}H_{24}O_2$, is obtained by saponifying cocoanut oil, liberating the fatty acids, driving off volatile acids in steam, extracting the dry lead salts of the residual acids with ether to remove lead oleate, decomposing with

hydrochloric acid, dissolving the liberated acids in alcohol, and fractionally precipitating by barium acetate. The first precipitate contains the acids of higher molecular weight. According to Caspari (*J.S.C.I.*, 1902, 711), lauric acid is best prepared from the fat in the seeds of *Lindera benzoïn*; the acid melts at 42° C. and boils at 166° C. under 10–11 mm. pressure. Lauric acid crystallises from alcohol in needles, it cannot be distilled under the ordinary pressure without decomposition, but is volatile in steam. The laurates, with the exception of the alkali salts, are practically insoluble in water. Magnesium laurate dissolves freely in hot absolute alcohol. The alkali laurates are tolerably soluble in common salt solutions.

Myristic acid, $C_{14}H_{28}O_2$, is contained in many fats. It forms crystalline leaflets, which melt at 53·8° C. and boil at 196·5° C. under 15 mm. pressure. Myristic acid is insoluble in water and cannot be carried over in a current of steam.

Palmitic acid, $C_{16}H_{32}O_2$, is found in a very large number of animal and vegetable oils. It is obtained from the fatty acids of palm oil or myrtle wax, and is purified by recrystallisation from hot alcohol, from which it separates in bundles of fine needles; the recrystallisation is repeated until the correct melting-point is obtained. The acid melts at 62°C (according to De Visser, the absolutely pure acid melts at 62·618°), and solidifies to pearly scales. It boils at 339–356° C. with some decomposition, and at 215° C. under 15 mm. pressure.

The salts of palmitic acid, with the exception of those of the alkali metals, are insoluble in water and not more than slightly soluble in alcohol. According to Lidow, 50 c.c. of the solution of lead palmitate in ether contain 0·0092

grm. of the salt. The alkali palmitates dissolve in alcohol and a small quantity of water without decomposition, but are decomposed by much water into free alkali and an insoluble acid palmitate. The potassium salt, for example, is decomposed according to the following equation:

$$2C_{16}H_{31}O_2K + H_2O = KOH + KH(C_{16}H_{31}O_2)_2.$$

When soluble salts, caustic alkalis, &c. are added to solutions of the alkali palmitates in water, the solid palmitates are separated; this is the process which is known generally as 'salting out.'

Margaric acid, $C_{17}H_{34}O_2$, is one of the few acids containing an uneven number of carbon atoms found in fats. It melts at 59·8° C. and boils at 227° C. under 100 mm. pressure.

Daturic acid, $C_{17}H_{34}O_2$, is isomeric with margaric acid; it melts at 54·5° C.

Stearic acid, $C_{18}H_{36}O_2$, is found as the triglyceride in most fats. It is produced by reducing oleïc acid, or elaïdic acid, with hydriodic acid and red phosphorus at 200° C. It may be obtained by saponifying mutton tallow or, better, shea butter, separating the fatty acids and recrystallising them from alcohol. It crystallises in leaflets, which melt at 69·320° C. (De Visser) and boil with decomposition at 360–380° C. or at 232° C. under 15 mm. pressure. Stearic acid dissolves readily in hot alcohol and in about 40 parts of cold absolute alcohol (see p. 150).

The salts of stearic acid are similar to those of palmitic acid. The alkali salts are similarly decomposed by much water into free alkali and acid salts. The solution of the lead salt in dry ether contains 0·0074 grm. in 50 c.c. (Lidow).

Arachidic acid, $C_{20}H_{40}O_2$, is obtained when brassidic acid is fused with caustic potash. It is present in earth-nut, rapeseed, and maize oils. It melts at 77° C.

Behenic acid, $C_{22}H_{44}O_2$, crystallises in needles, which melt at 84° C. and solidify at 78° C.

Lignoceric acid, $C_{24}H_{48}O_2$, is found together with arachidic acid in earth-nut oil; it is separated from arachidic acid by repeated recrystallisation from 90 per cent. alcohol (Archbutt, *J.S.C.I.*, 1898, 1124). It then melts at 79·6° C.

Cerotic acid, $C_{26}H_{52}O_2$, is found in beeswax, carnaüba wax, and wool-wax. It crystallises in microscopic needles, which melt at 78° C. (Henriques, 82·5° C.); it dissolves readily in boiling alcohol, but is little soluble in cold alcohol. The alkali salts dissolve in hot water and hot alcohol; the lead salt is insoluble in ether, but dissolves in hot benzene, from which it crystallises on cooling.

Melissic acid, $C_{30}H_{60}O_2$, occurs in beeswax; it crystallises in silky scales, which melt at 91° C. The lead salt crystallises from toluene in needles, which melt at 118–119° C.

ACIDS OF THE OLEÏC ACID SERIES, $C_nH_{2n-2}O_2$

The following acids of this series are to be considered:

Tiglic acid,	$C_5H_8O_2$.
Hypogæic acid Physetoleïc acid	$C_{16}H_{30}O_2$.
Oleïc acid Elaïdic acid Iso-oleïc acid	$C_{18}H_{34}O_2$.

Dœglic acid, $C_{19}H_{36}O_2$.
Erucic acid, Brassidic acid } $C_{22}H_{40}O_2$.

The gradual rise in melting- and boiling-points, the gradual change in the solubility of the salts, and in other properties, noticed in the acetic acid series, are not apparent in the oleïc acid series. The reason, no doubt, is that the known acids of this series differ as regards the position of the double bond; if we could compare a series of acids of similar constitution in this respect, we should in all probability find the same regularity in physical properties.

The acids of the oleïc series, and also their salts and glycerides, have, as a rule, lower melting-points than the acids of the acetic series containing the same number of carbon atoms. Thus these acids occur in the softer fats and more fluid oils, while the higher the proportion of saturated acids in a fat the harder it is. The acids of the oleïc series are unsaturated, they contain one ethylene linkage, and hence can unite with one molecule of bromine, hydrobromic acid, &c., to produce a saturated compound. They also react with sulphuric acid to produce an unstable addition compound. The action of nitrous acid on the liquid glycerides of the higher acids, by which they are converted into solid glycerides of isomeric acids, is characteristic, and serves to distinguish them from the glycerides of the more unsaturated acids. Only the drying oils yield a fluid product in applying this reaction.

Tiglic acid, $CH_3.CH:C(CH_3).CO_2H$, may be made synthetically by various methods. It is found as the triglyceride in croton oil. Tiglic acid crystallises in tables

and columns which have an odour of benzoïc acid, it melts at 64·5° C. and boils at 198·5° C.; it is readily soluble in hot water, but with difficulty in cold. It unites with hydriodic acid, hydrobromic acid, hypochlorous acid, and bromine to form addition compounds. The salts are mainly soluble in water.

Hypogæïc acid $C_{16}H_{30}O_2$, is found as the triglyceride in earth-nut oil; it crystallises in needles, which melt at 33° C. and are readily soluble in alcohol. It oxidises in the air, forming volatile acids of rancid odour. It unites with two atoms of bromine, and is converted by nitrous acid into the isomeric gaïdic acid, which melts at 39° C. This transformation is similar to that of oleïc acid into elaïdic acid.

Physetoleïc acid, $C_{16}H_{30}O_2$, is isomeric with hypogæïc acid; it is found in sperm oil and seal oil. It melts at 30° C., oxidises in the air, and is not altered by nitrous acid.

Oleïc acid, $C_8H_{17}.CH \parallel CH.(CH_2)_7.CO_2H$, occurs as the triglyceride in most oils and fats; it is obtained in large quantities as a by-product in the manufacture of stearin candles; as so prepared it is not quite pure. The pure acid may be made by saponifying lard, converting the fatty acids into lead salts, extracting with ether, evaporating the ethereal solution, liberating oleïc acid from the evaporated residue, converting into the barium salt, recrystallising from alcohol, and decomposing by acid. Oleïc acid crystallises in needles, which melt at 14° C. It can be distilled in superheated steam without decomposition, or at 223° C. under 10 mm. pressure. Oleïc acid slowly oxidises in the air,

forming a variety of volatile and non-volatile acids. Alkaline permanganate produces mainly dihydroxystearic acid. When fused with potash it gives palmitic acid in almost the theoretical yield. It unites with two atoms of bromine, is reduced by hydriodic acid and red phosphorus to stearic acid, and unites with strong sulphuric acid in the cold to form sulphohydroxystearic acid, $C_{17}H_{34}(SO_3H).CO_2H$, which is easily decomposed by water, forming hydroxystearic acid. Zinc chloride at a high temperature converts oleïc acid into a mixture of substances with higher melting-points: β-hydroxystearic acid, stearolactone (which is derived from γ-hydroxystearic acid), and iso-oleïc acid. The mechanism of the reaction resembles that of the change effected by sulphuric acid.

By the action of nitrous acid, oleïc acid is converted into its solid isomeride, elaïdic acid (see p. 96).

The salts of oleïc acid, with the exception of the alkali salts, are insoluble in water, but are more easily soluble in alcohol and ether than the corresponding salts of the saturated acids. The alkali oleates are not so readily decomposed by water as the corresponding stearates and palmitates: the insoluble acid oleates separate from aqueous solutions only at low temperatures.

Elaïdic acid, $C_8H_{17}.CH$
$\|$
$CO_2H.(CH_2)_7CH$. According to Baruch (*Ber.*, 27, 172), oleïc and elaïdic acids differ in constitution in respect simply of the position of the groups which are united to the ethylene carbon atoms; the isomerism is, therefore, stereochemical only. Elaïdic acid is obtained by the action of nitrous acid or sulphurous acid on oleïc acid;

it crystallises from alcohol in leaflets, which melt at 51–52° C. and boil at 225° C. under 10 mm. pressure. According to Edmed (*J.S.C.I.*, 1899, 1031), oleïc acid is quantitatively converted into elaïdic acid by the action of nitric acid of 1·2–1·25 specific gravity in the cold. The reactions of elaïdic acid are practically the same as those of oleïc acid given above.

Iso-oleïc acid, $C_{17}H_{33}.CO_2H$. The exact constitution of this acid is still unknown. Oleïc acid unites with hydriodic acid to form iodostearic acid, which is decomposed by alcoholic potash into iso-oleïc acid. In the sulphuric acid method of saponifying fats, used in the candle industry, a similar (partial) transformation of oleïc acid into iso-oleïc acid takes place. Oleïc acid unites with the sulphuric acid to form sulphohydroxystearic acid, $C_{17}H_{34}(SO_3H).CO_2H$, which is decomposed by water, producing hydroxystearic acid, $C_{17}H_{34}(OH).CO_2H$. When the acids are distilled in superheated steam, the hydroxy-acid loses water, yielding iso-oleïc acid, $C_{17}H_{33}.CO_2H$, which has a much higher melting-point than the original oleïc acid; thus the yield of material available for candle-making is increased. Iso-oleïc acid crystallises from ether in transparent tables, which melt at 44–45° C. It is readily soluble in alcohol, combines with two atoms of bromine, and is decomposed by fusion with potash into acetic and palmitic acids.

Dœglic acid, $C_{19}H_{36}O_2$, is found in Arctic sperm (bottle-nose) oil; it is a yellow oil which solidifies at about 4° C.

Erucic acid, $C_{22}H_{40}O_2$. According to Baruch (*Ber.* 26 1876) the constitution of erucic acid is represented by the

formula $C_8H_{17}.CH$
$\|$
$HC.(CH_2)_{11}.CO_2H$. Erucic acid is found in rape oil and in the fatty oil of black and white mustard seed. It is obtained by repeatedly recrystallising the fatty acids of rape oil from alcohol at 0° C. It crystallises in long thin needles, which melt at 33—34° C. and boil at 264° C. under 15 mm. pressure. It unites with two atoms of bromine, is converted into arachidic and acetic acids by fusion with potash and by nitrous acid into the isomeric brassidic acid. The salts of erucic acid are similar to those of oleïc acid, but lead erucate is little soluble in cold ether.

Brassidic acid, $C_8H_{17}.CH$
$\|$
$CO_2H.(CH_2)_{11}.CH$. As is seen from the formulæ, erucic and brassidic acids differ in constitution in the same way as oleïc and elaïdic acids. Brassidic acid is obtained by the action of nitrous or sulphurous acid on erucic acid. It crystallises from alcohol in leaflets, which melt at 65–66° C. and boil at 256° C. under 10 mm. pressure. Its reactions are similar to those of erucic acid ; the lead salt is, however, very little soluble, even in hot ether.

ACIDS OF THE PROPIOLIC SERIES, $C_nH_{2n-4}O_2$

The only acids of this series to be considered are :

Elaomargaric acid, $C_{17}H_{30}O_2$.
Linolic acid, Tariric acid } $C_{18}H_{32}O_2$.

Of these acids linolic acid is the only one of great importance ; it is characteristic of the drying oils, but is also

found in smaller quantity in other oils and even in solid fats. The acids of this series unite with four atoms of bromine to form tetrabromo addition compounds; they also readily unite with oxygen, and are soon altered by exposure to air, but they do not dry to the same extent as the drying oils from which they are obtained.

Elaomargaric acid, $C_{17}H_{30}O_2$, is found as the glyceride in Chinese wood oil. It crystallises in rhombic tables, which melt at 48° C. It soon becomes resinous on exposure to air, owing to the absorption of oxygen. On exposure to light in alcoholic solution it is transformed into the isomeric elaostearic acid, which melts at 71° C. A similar change takes place in Chinese wood oil itself on exposure to light, the liquid glyceride of the elaomargaric acid being transformed into the solid glyceride of elaostearic acid.

Linolic acid, $C_{18}H_{32}O_2$, occurs principally in the drying oils, especially in linseed oil, from which it can be obtained by precipitating the calcium salts of the fatty acids, extracting calcium linolate with ether, liberating the acid from the extracted salt, preparing the barium salt, and repeatedly recrystallising this from ether. Linolic acid is a yellow oil which does not solidify at −18° C. It readily oxidises in the air, and the salts oxidise more readily still. This rapid alterability of linolic acid and its derivatives is the great obstacle to their investigation. Linolic acid unites with two molecules of bromine to form a tetrabromide, which, according to Hehner and Mitchell (*J.S.C.I.*, 1899, 77), melts at 113·4° C. The tetrabromide is obtained by adding bromine to a solution of the acids of linseed oil (or, better, maize oil) in acetic acid,

washing the mixture of precipitated bromides with ether, when linolenic acid hexabromide remains insoluble, evaporating the ethereal solution, and recrystallising from alcohol (Hehner and Mitchell, *loc. cit.*). Linolic acid is not converted into a solid isomeride by treatment with nitrous acid. It is reduced by hydriodic acid to iodostearic acid; it is oxidised by alkaline permanganate to sativic acid, $C_{18}H_{32}(OH)_4O_2$, linusic acid, $C_{18}H_{30}(OH)_6O_2$, or azelaïc acid, $CH_2(CH_2.CH_2.CH_2.CO_2H)_2$, according to the strength of the solution. The salts of linolic acid are amorphous; the barium and calcium salts are soluble in ether and alcohol, lead linolate is soluble in ether.

Tariric acid, $C_{18}H_{32}O_2$, is oxidised to adipic and lauric acids; its constitution is accordingly given by the formula, $CH_3.(CH_2)_{10}.C:C.(CH_2)_4.CO_2H$. It melts at 50·5° C.

ACIDS OF THE SERIES $C_nH_{2n-6}O_2$

Linolenic acid, $C_{18}H_{30}O_2$.
Jecoric acid, $C_{18}H_{30}O_2$.

The former of these two acids is the only acid of this series the existence of which has been definitely proved. These acids unite with six atoms of bromine and absorb oxygen even more readily than the acids of the propiolic series.

Linolenic acid, $C_{18}H_{30}O_2$, is contained in the drying oils and probably to a small extent in other oils. It is obtained by reducing with zinc and hydrochloric acid the linolenic hexabromide obtained from the liquid acids of linseed oil. The product is a nearly colourless oil which very rapidly absorbs oxygen from the air and darkens. It is

to be noted, however, that the iodine value of the linolenic acid so obtained is lower than the theoretical, and that the quantity of hexabromide it produces is not more than 50 per cent. of the theoretical. Linolenic hexabromide, when freed from linolic tetrabromide by washing with ether, melts at 180–181° C. (Hehner and Mitchell, *loc. cit.*). By oxidation with alkaline permanganate, Hazura obtained from linolenic acid two acids of the formula $C_{18}H_{30}(OH)_6O_2$, linusic and isolinusic acids, and hence concluded that linseed oil contains two acids of the formula $C_{18}H_{30}O_2$—linolenic and isolinolenic acids.

The existence of still less saturated acids—of the series $C_nH_{2n-8}O_2$ and $C_nH_{2n-10}O_2$—is probable in marine animal oils (which see).

ACIDS OF THE SERIES $C_nH_{2n-2}O_3$

Ricinoleïc acid
Rapic acid } $C_{18}H_{34}O_3$.

The acids of this series combine alcoholic properties with acid properties; they contain an alcoholic hydroxyl group, and hence can unite with acetic anhydride to produce acetylated acids. They are also unsaturated acids, and unite with one molecule of bromine, &c., to form addition compounds.

Ricinoleïc acid, $C_6H_{13}.CH(OH).CH_2.CH \parallel CH.C_7H_{14}.CO_2H$. The triglyceride of this acid is almost the only constituent of castor oil. According to Krafft, pure ricinoleïc acid is a hard crystalline mass which melts at 16–17° C.

Juillard, however, gives the melting-point as 4–5° C. The acid mixes with alcohol and ether in every proportion; it spontaneously changes at the ordinary temperature into poly-ricinoleïc acids, from which ricinoleïc acid may be obtained by saponification with alcoholic potash. Ricinoleïc acid combines with two atoms of bromine, but cannot be reduced to stearic acid. It unites with sulphuric acid to form substances soluble in water (see Turkey-red oil). By the action of nitrous acid, ricinoleïc acid is transformed into the isomeric ricinelaïdic acid, which melts at 50° C. By the action of acetic anhydride, ricinoleic acid is converted into acetyl-ricinoleïc acid.

Rapic acid, $C_{18}H_{34}O_3$. According to Reimer and Will (*Ber.* 20, 2387), the liquid acids of rapeseed oil consist mainly of rapic acid and not of oleïc acid. Rapic acid is obtained by saponifying rapeseed oil, liberating the fatty acids, dissolving them in alcohol, precipitating the alcoholic solution of the acids with zinc acetate, and extracting the zinc salts with cold ether. Rapic acid is then liberated from the extracted zinc salt. Rapic acid is a liquid which is not solidified by nitrous acid; the zinc salt is crystalline, and melts at 78° C. According to Zellner, rapic acid is isomeric with oleïc acid and has the formula $C_{18}H_{34}O_2$; it is reduced to stearic acid by hydriodic acid (*J.S.C.I.*, 1896, 661).

THE ALCOHOLS

The alcohols which are found, generally in the form of esters, in fats and waxes, belong both to the fatty and aromatic series. With the exception of glycerin, which is by far the most important, the alcohols are monovalent;

they are among the highest known members of their respective series.

These alcohols (always with the exception of glycerin) are insoluble in water and soluble in ether; hence they appear in the course of analysis as 'unsaponifiable matter.' They have the ordinary alcoholic properties in respect of ester formation.

A characteristic reaction of the higher saturated and unsaturated fatty alcohols is that, on heating with soda lime, they are converted into the corresponding acids, hydrogen being evolved. For example, myricyl alcohol gives melissic acid :

$$C_{29}H_{59}.CH_2.OH + NaOH = C_{29}H_{59}.CO.ONa + 2H_2.$$

The aromatic alcohols (cholesterols, &c.) do not give this reaction, but remain unchanged.

ALCOHOLS OF THE SATURATED SERIES $C_nH_{2n+2}O$

Cetyl alcohol, $C_{16}H_{34}O$.
Octadekyl alcohol, $C_{18}H_{38}O$,
Carnaübyl alcohol, $C_{24}H_{50}O$.
Ceryl alcohol, $C_{27}H_{56}O$,
Myricyl alcohol, $C_{30}H_{62}O$.

Cetyl alcohol, $C_{16}H_{34}O$. Cetyl palmitate is the chief constituent of spermaceti, from which cetyl alcohol is obtained by saponifying, precipitating the lime soaps, washing, drying, and extracting them by alcohol. The alcohol solution is evaporated, the residue repeatedly extracted with water, dissolved in ether, decolourised by animal charcoal, and recrystallised. The cetyl alcohol so obtained may contain

octadekyl alcohol ; it is therefore converted into the acetate and purified by fractionally distilling *in vacuo*. Cetyl alcohol may also be obtained by reducing palmitic aldehyde ; on oxidation it is converted into palmitic acid. Cetyl alcohol crystallises from alcohol in leaflets, which melt at 49–50° C. ; it is insoluble in water, and soluble in alcohol and ether. It distils undecomposed at 344° C., or at 189·5° C. under 15 mm. pressure.

Octadekyl alcohol, $C_{18}H_{38}O$. The occurrence and preparation of this alcohol are sufficiently indicated above. It may be obtained by reducing stearic aldehyde. Octadekyl alcohol crystallises from alcohol in large shining leaflets, which melt at 59° C. It can only be distilled under low pressures ; under 15 mm. pressure it boils at 210·5° C.

Carnaübyl alcohol, $C_{24}H_{50}O$, was isolated from wool-fat by Darmstaedter and Lifschütz (*J.S.C.I.*, 1897, 150 ; *Ber.* 29, 2980 ; 31, 97). It melts at 68–69° C., and is soluble with difficulty in cold alcohol, from which it can be recrystallised.

Ceryl alcohol, $C_{27}H_{56}O$. Ceryl cerotate is the principal constituent of Chinese wax ; ceryl alcohol is also present in wool-fat (Darmstaedter and Lifschütz, *loc. cit.*), carnaüba wax, and beeswax. It melts at 79° C.

Myricyl alcohol, $C_{30}H_{62}O$. Myricyl cerotate is the chief constituent of carnaüba wax, myricyl palmitate of beeswax. Myricyl alcohol crystallises from ether in needles, which melt at 85° C. ; according to Gascard (*Beilstein*, 1 suppl.), the melting-point is 88° C.

UNSATURATED ALCOHOL, $C_nH_{2n}O$

Lanolinic alcohol, $C_{12}H_{24}O$, has been isolated by Marchetti from wool-fat. It is insoluble in ether and soluble with difficulty in cold alcohol and chloroform. It melts at 102–104° C.

Other unsaturated alcohols appear to be present in wool-fat, and Lewkowitsch is of opinion that the alcohols of sperm oil are also unsaturated.

GLYCERIN, $C_3H_5(OH)_3$

The constitution and certain of the chemical properties of glycerin have already been given (p. 3). Glycerin is a very thick water-white liquid, odourless, sweet in taste (hence the name), miscible in all proportions with water and alcohol, but insoluble in ether. When kept at low temperatures glycerin solidifies to deliquescent rhombic crystals, which melt at 17° C. (Henninger). It boils at 290° C. under atmospheric pressure ; *pure* glycerin distils without decomposition, but glycerin containing salts is partially decomposed into acrolein and water. Under lower pressures, especially in a current of steam, glycerin distils undecomposed ; under 13 mm. pressure it boils at 180° C. Glycerin is very hygroscopic, it rapidly absorbs considerable quantities of moisture from the air. According to Hehner, glycerin solutions can be evaporated on the water-bath without loss until the strength reaches 75 per cent. ; and in a flask closed by a paper cap glycerin can be completely dried in the air-bath at 100–110° C. without appreciable loss (Benedikt, *Analyse der Fette*).

The specific gravities of solutions of glycerin at 15° C. are as follows (Gerlach):

Percentage of Glycerin in the Solution	Specific Gravity	Percentage of Glycerin in the Solution	Specific Gravity	Percentage of Glycerin in the Solution	Specific Gravity
100	1·2653	70	1·1850	40	1·020
95	1·2526	65	1·1710	35	1·0885
90	1·2400	60	1·1570	30	1·0750
85	1·2265	55	1·1430	25	1·0620
80	1·2130	50	1·1290	20	1·0490
75	1·1990	45	1·1155	10	1·0245

Glycerin itself has considerable solvent properties, it dissolves certain salts even more readily than water, metallic soaps which are insoluble in water, &c.

Glycerin forms compounds analogous to the alcoholates of the monatomic alcohols, but more stable. Calcium glycerate, $CaC_3H_6O_3$, a crystalline powder, is obtained by the action of quicklime on glycerin. Lead glycerate, $PbC_3H_6O_3$, is obtained by the action of caustic potash on aqueous solution of lead acetate containing glycerin. Litharge and glycerin at once combine when ground together, forming a hard solid glycerate. The action of acids on glycerin, producing three series of esters, has already been mentioned (page 4). Nitroglycerin, which is obtained by the action of nitric and sulphuric acids, is really glycerin trinitrate, $C_3H_5(NO_3)_3$. When heated with dehydrating agents, or when rapidly heated alone glycerin is decomposed into water and acroleïn, $C_3H_8O_3 = 2H_2O + C_3H_4O$.

Manganese dioxide and sulphuric acid oxidise glycerin to carbon dioxide and formic acid; alkaline permanganate produces carbon dioxide, formic, oxalic, acetic, propionic,

and a little tartronic acid, but if the oxidation is conducted in a strongly alkaline solution at the ordinary temperature oxalic and carbonic acids are the only products. This reaction is utilised in estimating glycerin. Potassium bichromate and sulphuric acid oxidise glycerin to carbon dioxide and water.

Glycerin is obtained on the large scale from the aqueous liquid left after saponifying fats for the candle industry and from the residual soap lyes obtained in the manufacture of soap after 'salting out.' The liquid is neutralised and evaporated in a vacuum, when the soluble salts separate to a large extent and are removed. The glycerin is now known as 'crude.' It is then distilled in a vacuum by means of superheated or high-pressure steam, and the distillate concentrated under reduced pressure; it is then known as 'once distilled.' 'Refined' glycerin is obtained by again distilling 'once distilled' under reduced pressure, concentrating, also under reduced pressure, and filtering through animal charcoal.

AROMATIC ALCOHOLS

Cholesterol, $C_{26}H_{44}O$, is found in various parts and secretions of the human body. It is an important constituent of the harder portion of wool-fat (Darmstaedter and Lifschütz), and is present in small quantity in other animal fats and oils, from which it is obtained by saponifying and extracting with ether. Cholesterol crystallises from alcohol or ether in leaflets or rhomboid tables containing one molecule of water of crystallisation, which is lost at 100° C., or over sulphuric

acid. From chloroform it crystallises in anhydrous needles. Cholesterol melts at 145–146° C. and can be distilled *in vacuo* without decomposition. By the action of acid anhydrides it forms esters; it unites with bromine in carbon bisulphide solution to form a dibromide, $C_{26}H_{44}Br_2O$.

Cholesterol gives a number of colour reactions, of which the following are mentioned: concentrated sulphuric acid is added, a drop at a time, to a cooled saturated solution of cholesterol in acetic anhydride: the solution is at first rose-red and then turns to blue (Liebermann). About 0·05 grm. of cholesterol is dissolved in 2 c.c. of chloroform, 2 c.c. of strong sulphuric acid added, and the tube shaken: the chloroform solution becomes blood-red, then cherry-red to purple, whilst the sulphuric acid acquires a green fluorescence (Salkowski); the appearance of a blue colouration on the first addition of sulphuric acid is to be disregarded—it is due to the presence of colouring matters extracted from the fat along with the cholesterol. Cholesterol is evaporated almost to dryness with a little ferric chloride, hydrochloric acid, and chloroform until the edge of the liquid is coloured reddish-violet; the dish is then cooled, more chloroform added and evaporated: the residue becomes purple-violet, bluish-violet, and finally dirty green (Schiff).

Cholesteryl acetate, $C_{26}H_{43}.C_2H_3O_2$, obtained by boiling cholesterol with acetic anhydride, crystallises in needles, which melt at 113° C.

Phytosterol, $C_{26}H_{44}O$, is found in small quantities in vegetable oils. It is isomeric with, and very similar to, cholesterol. Phytosterol forms the solid unsaponifiable

matter extracted by ether from the solutions obtained on saponifying vegetable oils. It crystallises from alcohol with one molecule of water in needles united to stars or bundles, from chloroform and ether in anhydrous needles. It melts at 132–133° C. Phytosterol gives with chloroform and sulphuric acid the same colouration as cholesterol.

Phytosteryl acetate, $C_{26}H_{43}.C_2H_3O_2$, crystallises from alcohol in shining leaflets which melt at 120° C.

Isocholesterol, $C_{26}H_{44}O$, is contained in the softer portion of wool-fat (Schulze, Darmstaeder, and Lifschutz, *J.S.C.I.*, 1898, 773). It crystallises from ether in fine needles, which melt at 137–138° C. The chloroform solution gives no reaction with sulphuric acid. The solution in acetic anhydride gives with sulphuric acid a yellow colouration, turning to reddish-yellow. In applying this reaction to mixtures of cholesterol and isocholesterol, the colouration produced by the latter hides the blue cholesterol reaction (Lewkowitsch).

Paracholesterol, $C_{26}H_{44}O$, has been found in wheat oil by Frankforter and Harding (*J.S.C.I.*, 1899, 1030). It crystallises from ether in silky needles, which melt at 134–134·5° C. From alcohol it crystallises in leaflets containing one molecule of water of crystallisation. Paracholesterol gives the same colouration as cholesterol in Salkowski's reaction, but the chloroform solution on long standing turns blue and then violet (Schulze). Other isomerides of cholesterol are found in cryptogamous plants (Gerard, *J.S.C.I.*, 1898, 587).

Free Fatty Acids.—All the fats and oils contain some quantity of free fatty acid—a quantity which increases

with the age of the fat and the degree of its exposure to air and light. The amount of free acid is generally small, but may rise to 50 per cent. or more, as for example, in palm oil and linseed oil made from damaged seed. Refined cottonseed oil, which is refined by means of alkalis, contains when fresh not more than a trace of free fatty acid.

Other Constituents.—Most oils and fats contain a small quantity of colouring matters in solution, which may be more or less completely removed by the various processes of refining and bleaching to which an oil is subjected before it reaches the consumer. These colouring matters, together with other constituents, some of which contain nitrogen, and all of which occur in very small quantity and hence have usually escaped complete investigation, give rise to the various colour reactions by means of which certain oils can be distinguished. It may be pointed out at once that, since most colour reactions are due to traces of unnecessary constituents, a method of refining may at any time be discovered which will produce an oil that cannot be detected by a reaction hitherto regarded as reliable. Improvements in refining have furnished one cause for the disuse of many of the older colour reactions.

TO STUDENTS.—The methods of analysis described in the following chapters should be practised on samples of known purity, until the manipulations become easy and correct and concordant results can be obtained.

CHAPTER II

THE PHYSICAL PROPERTIES OF OILS, FATS, AND WAXES, AND THEIR DETERMINATION

BOTH the physical and chemical properties of oils and fats are used to establish their identity or purity. Among the physical properties it may be necessary to examine the specific gravity, melting or solidifying point, viscosity, flash point, solubility, refractive index, optical rotation, and absorption spectrum.

Before proceeding to determine the physical properties of an oil or fat, it is necessary to ascertain whether it is free from water, dirt, and similar accidental impurities, and, if not, to purify it before it is used (see Chapter IV., p. 105). The methods given in the present chapter are also applied to the free fatty acids of the oils, which are prepared for the purpose as described at the commencement of Chapter III., p. 70.

The specific gravity frequently affords important evidence as to the purity of an oil, it is the physical property most frequently determined. There are several methods in general use for determining the specific gravities of oils, to all of which reference must be made.

The Hydrometer consists of a closed weighted pear-shaped or cylindrical glass vessel, to the upper end of

which is attached a narrow glass tube containing a scale. When this instrument is floated in a liquid, the specific gravity of the liquid is given by that graduation on the scale which lies at the level of the surface of the liquid. The hydrometer is used in a narrow cylinder, in order to economise the liquid, but the cylinder must be so wide that the instrument floats freely in the middle of the liquid without adhering to the walls. The temperature of the oil must be taken either while the hydrometer is being floated in it, or just previously (see below, *Correction for temperature*). The indications of the hydrometer are not very exact, and it necessitates the use of a relatively large quantity of liquid, so that for many purposes some other process of determining the specific gravity is used.

It may not be out of place to state that glass apparatus, in which oil has been used, is conveniently cleaned by washing out in turn with turpentine (twice), methylated spirit, water, strong sulphuric acid (to which a little potassium bichromate may be added), and water.

The specific gravity of oils is now almost universally stated as such, and not with reference to the scale of any particular instrument. Fischer's *oleometer* is a hydrometer with a scale divided in degrees in a particular manner and intended to be used for determining the specific gravities of oils. The specific gravity, s, of an oil, the density of which is $n°$ by Fischer's oleometer, is given by the formula:

$$s = \frac{400}{400 + n}$$

Westphal's Balance utilises directly the principle that the apparent loss in weight of a solid substance, when

immersed in a liquid, is equal to the weight of liquid displaced. A balance beam has suspended from one end a glass sinker containing a thermometer, the other end is counterpoised, so that when the whole apparatus is in air the beam is exactly horizontal and a point on the end of the counterpoised arm is exactly opposite a fixed point (see fig. 1). The volume of the sinker is exactly 10 c.c., the balance arm from which it is suspended is graduated. The instrument is used by immersing the sinker in the oil, and placing riders on the beam until it is horizontal. The weight of the riders on the beam, having regard to their position, at once gives the specific gravity when divided by 10. The balance may be used to take specific gravities at any required temperature by placing the oil in a wide test-tube supported in a water-bath, which is kept at a constant temperature. In this case the whole arrangement must be such that the steam from the bath does not condense on the balance.

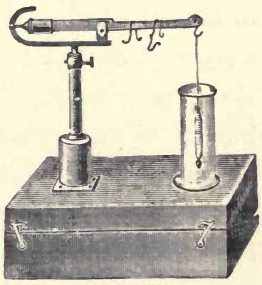


FIG. 1

The Specific Gravity Bottle is the most convenient instrument to use for determining the specific gravities of liquid oils at the ordinary temperature. There are several forms of specific gravity bottle, of which perhaps the most suitable is that which has a capillary tube through the stopper. The bottle is made to contain, when quite full to the top of this tube, a definite weight of water at the standard temperature of 60° F. A 10 c.c. bottle is quite large enough to give sufficiently accurate determinations for all ordinary purposes ; it

has the advantage that very much smaller quantities of oil are required than are necessary for the two methods previously described. The bottle is filled with the oil, put in the balance-case together with the stopper and a thermometer, and allowed to remain until all air bubbles have risen, and until bottle, oil, and thermometer have all had time to come to the temperature of the balance-case, for which twenty minutes is generally ample. The bottle is then taken out of the balance-case, the stopper inserted and pushed home, taking care that no bubble of air remains round the bottom. The bottle is immediately wiped clean, returned to the case, and weighed. In performing these operations contact with the hand must be avoided, since the oil would thereby be warmed ; the insertion of the stopper must therefore be rapidly performed, and the bottle protected from the heat of the hand by a thick cloth. The weight of the oil, divided by the weight of water contained by the bottle at the standard temperature, gives the specific gravity at the temperature indicated by the thermometer, which has then to be corrected to the standard temperature of 60° F. (=15·5° C.).

Sprengel's Tube.—Several forms of the Sprengel tube are made and have been described ; the only correct form, which possesses all the advantages of the instrument, consists (fig. 2) of a wide tube, to each end of which is sealed a narrow capillary tube, of which the shorter is bent at right angles and the longer three times at right angles. It is important that one of these capillary tubes should be wider than the other. The instrument can readily be made in the laboratory by sealing pieces of wide soda-glass capillary tube to each end of a piece of ordinary

glass tubing (the dimensions of which depend on the required capacity) and then drawing out the capillary tubes to the proper thinness. An instrument with a total capacity of 1–2 c.c. can be made from ordinary glass tubing of 5–7 mm. bore, drawn out to a thick-walled capillary at

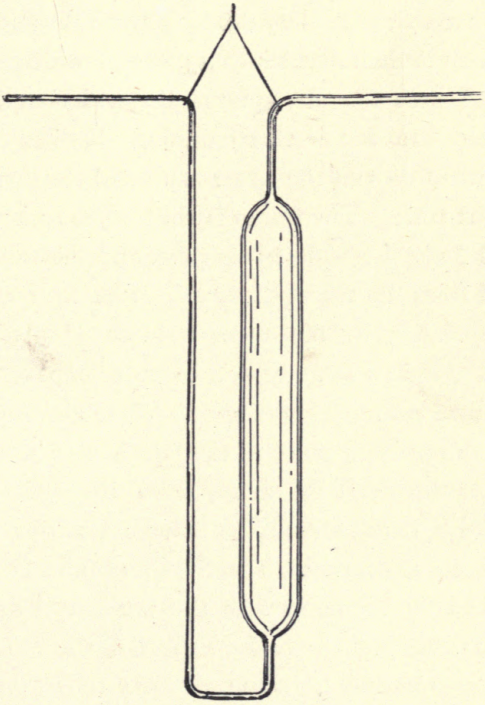


FIG. 2

each end. By means of a Sprengel tube of this capacity, or even one holding only 0·5 c.c., the specific gravity of a very small quantity of oil—such, for example, as the mineral oil isolated from an adulterated fatty oil—can readily be determined with an accuracy unattainable by

other means. In view of the difficulty of cleaning the apparatus and the ease with which a new one is made and standardised, the writer prefers to use a fresh apparatus for each determination. The whole process is then as follows (if an apparatus is used of which the capacity is known, that part of the process is naturally omitted):

The narrower capillary tube is passed through a rubber cork, which is then fitted into a piece of wide glass tubing connected with the filter-pump, the wider capillary tube is dipped into a vessel containing distilled water, the pump turned on and the water aspirated through the tube for a short time. The pump is shut off, the rubber stopper removed from the wide glass tube, and the capillary then removed from the vessel of water. The Sprengel tube is now suspended, by means of a piece of platinum wire fastened to it, in a large beaker of water kept at a constant temperature some degrees above the laboratory temperature (or the melting-point of the fat, in case a solid fat is under examination) by means of a tiny gas flame. A temperature regulator and mechanical stirrer are advisable, but not necessary. The Sprengel tube is suspended alongside a thermometer with the horizontal arms of the capillary tubes just above the surface of the water. When water ceases to flow from the capillary tubes, the excess is removed from the arms by means of filter paper, the tube is removed from the bath, allowed to cool a little, then wiped, placed in the balance-case, left for a short time, and weighed. The water is then aspirated out by means of the filter-pump, the tube placed in the air-bath at 110° C., and a current of air aspirated through for a minute or two. The tube is now dry, and can be allowed to cool and then be

weighed. It is next filled with the oil in the same manner as with water, placed in the water-bath, and weighed exactly as before. In the case of very thick and viscid oils the short capillary tube should be the wider of the two, and the oil (which may be heated) should pass through it into the instrument, otherwise the operation of filling may be very protracted. The following example indicates the method of calculating the desired specific gravity:

Weight of Sprengel tube, empty	2·7738 grms.
„ „ „ „ filled with water at 36° C.	3·4670 „
„ „ „ „ „ „ oil at 37° C. .	3·4273 „

The tube contains 0·6932 grm. of water at 36° C. Now the density of water at 36° C. is 0·9938 and the density at 15·5° C. is 0·9991 (from the tables, water at 4° C. = 1) Therefore the tube would contain at 15·5° C.

$$0{\cdot}6932\,\frac{0{\cdot}9991}{0{\cdot}9938}\text{ grm. of water.}$$

The tube contains 0·6535 grm. of oil at 38·5° C.; thus the specific gravity of the oil at 38·5° C. compared with water at 15·5° C. as unity is

$$\frac{0{\cdot}6535}{0{\cdot}6932}\times\frac{0{\cdot}9938}{0{\cdot}9991}=0{\cdot}9378$$

38·5° C. = 101·3° F.; 15·5° C. = 60° F.; 101·3 − 60 = 41·3. The difference in specific gravity of the oil for 1° F. being taken as 0·00037 (the difference for lithographic varnish), the required specific gravity is (see p. 45)

$$0{\cdot}9378+41{\cdot}3\times0{\cdot}00037=0{\cdot}9530.$$

It is obvious that the Sprengel tube may be used at any temperature above that of the atmosphere—*e.g.* at

100° C., by placing the tube in the neck of a flask in which water is being boiled—and that, when the tube is once filled, a series of determinations may be made at different temperatures, and thus the coefficient of expansion of the oil can be found, or, what comes to the same thing, the decrease in specific gravity for every 1° rise in temperature.

The greater capillary attraction in the narrower capillary tube causes this tube always to remain full to the end; contraction in cooling causes the liquid to leave the end of the wider capillary. If, while the tube is in the water-bath, the liquid for any reason does not fill the end of the wider capillary, the instrument will at once take up liquid, presented on a glass rod to the narrower capillary, until the wider is full.

Suspension Methods.—The specific gravities of liquid and solid fats may be determined by suspending a drop of the oil or a small portion of the solid fat in dilute spirit of ammonia, &c., the strength of which is increased or diminished until the drop or lump remains exactly suspended in the liquid, without rising or sinking. The specific gravity of the liquid is then taken by means of the hydrometer. The method is troublesome, because the drops of oil frequently include bubbles of air. The following process may be used: Make mixtures of six volumes of water with one volume of methylated spirit (free from petroleum), and of one volume of water with two volumes of spirit; let them stand until clear. In the case of an oil, let a drop fall into the lighter liquid from a glass rod held near the surface. In the case of a solid fat or wax, cut out a small lump and carefully brush it with a wet brush so that it will not take bubbles of air with

it. Then add the heavier liquid, a little at a time, stirring after each addition and allowing to come to rest, until the point is reached at which the drop or lump just remains suspended in the middle of the liquid.

Correction of Results.—These methods of determination give as a rule the specific gravity at some temperature which is not the standard temperature. In England it is usual to take 60° F. (15·5° C.) as the standard temperature for this purpose. Consequently the specific gravity of an oil is generally to be understood to mean its density at 60° F. compared with water at 60° F. Now the methods which have been described give as the result the specific gravity at some other temperature compared with water at 60° F. Allen has determined the coefficients of expansion of a large number of oils and found them to be identical for all practical purposes. The correction deduced by Allen from his results is 0·00035 for 1° F. or 0·00064 for 1° C., which quantity, multiplied by the difference between the temperature of observation and the standard temperature of 60° F. or 15·5° C., is to be added to or subtracted from the specific gravity at the temperature of observation, according as the latter is above or below the standard temperature. For example, if a 10 c.c. specific gravity bottle holds 9·295 grms. of linseed oil at 70° F., the specific gravity at 60° F. is $0{\cdot}9295 + 0{\cdot}00035 \times 10 = 0{\cdot}9330$.

The specific gravities of the more important oils, fats, and waxes are given, together with other physical properties, in the table on p. 62.

Melting and Solidifying Points.—Many methods have been proposed for determining the melting and solidifying

points of fats—methods differing not only in regard to details of process but in regard to the exact condition of the fat, which is to be considered as just melted or just solidified. When a solid fat is heated, it first becomes fluid, though it is still turbid ; then the liquid more or less quickly becomes transparent and free from solid particles. According as the temperature is taken at which the solid just becomes liquid, or the turbid liquid just becomes clear, a different melting-point is obtained. In consequence of the divergence of opinion and practice as to the exact point to be taken as the melting-point, the statements of different observers show some divergence. A very simple and satisfactory method, which enables a definite temperature to be recorded, is as follows: a piece of fairly thin-walled glass tubing, just softened in the flame, is drawn out to a thin-walled and wide capillary. A piece of the capillary 7–8 cms. long is cut off and one end dipped in the melted fat, which is drawn up (if necessary) to form a column 1–1·5 cms. long at the bottom of the capillary. It is now advisable to allow this tube to remain at rest for twenty-four hours, since fats do not at once regain their proper melting-points after being fused. The tube is attached to a thermometer by means of a rubber ring cut from a piece of tubing, in such a manner that the column of fat is exactly on a level with the bulb of the thermometer, which it should about equal, but not exceed, in length. The thermometer is suspended in a beaker of water, which is then very slowly heated by means of a small flame, while the water is stirred up and down with a glass rod bent at one end to a ring which fits comfortably into the beaker. The water should be 7–10 cms. deep,

with the bulb of the thermometer about halfway down. The temperature is observed at which the fat is sufficiently melted to be forced up the tube by the pressure of the water. The fat will generally not then form a clear liquid ; the higher temperature is next to be observed at which it just becomes clear. These two temperatures are to be regarded as the beginning and end of melting.

A modification of this method, which removes the chief drawback—the interval required for the fat to attain its proper solidifying point after having been melted to fill the capillary—has been described by Le Sueur and Crossley (*J.S.C.I.*, 1898, 988). A thin-walled tube about 75 mm. long and 7 mm. wide contains a fine capillary tube open at both ends. The solid fat is placed on the bottom of the wide tube round the lower end of the capillary. The tube is then attached to the thermometer by rubber bands, the bottom being on a level with the bulb of the thermometer. The whole is then suspended in water, which is slowly heated and stirred continuously. The melting-point is the temperature at which the fat is sufficiently melted to rise in the fine tube by capillary attraction. The rise takes place suddenly, and indicates a perfectly definite temperature, which is to be taken as the melting-point. This method is perfectly simple and reliable.

The solidifying points, which are of great importance in connection with the fats and fatty acids used in the candle industry, are determined by a different method. When a considerable quantity of melted fat is allowed to cool, protected from too rapid loss of heat, whilst it is stirred with a thermometer, the temperature is observed to sink,

first rapidly, then slowly, next to remain constant for some time (or to rise), and, finally, to fall. The fat solidifies during the time when the thermometer remains constant or rises. The constant temperature is then the solidifying point. In the case of the temperature rising, the highest point reached is taken as the solidifying point, which, with the fatty acids, is termed the 'titer.' As usual, many modifications in the details of the 'titer test' have been proposed. The following is Dalican's method, which has been generally adopted: About 80 grms. of the dried filtered fatty acids are melted on the water-bath and poured into a 6 × 1 inch test-tube, which is fastened by means of a ring of cork or rubber in the (short) neck of an empty flask. A thermometer, divided in fifths of a degree, is then suspended with its bulb at about the middle of the column of fat, which should half fill the test-tube. It is well to warm both test-tube and thermometer before use. The fat is then observed from time to time; when it begins to solidify at the bottom it is stirred round three times with the thermometer in one direction and then three times in the opposite. The thermometer is then closely watched; it first falls slightly, owing to the admixture of the cooler fat from the bottom, then rises and remains stationary for some two minutes. The highest temperature recorded during this rise is the solidifying point of the fatty acids or 'titer' of the fat. After the temperature has reached the stationary point, or 'titer,' it often afterwards rises; the temperature reached in the second rise must then also be noted. Lewkowitsch recommends that the temperature noted at the beginning of the rise and also the stationary temperature reached in

the rise should be recorded; the latter is of course what is generally understood by the 'titer.'

In determining the 'titer' of the fatty acids of a fat, the use of larger quantities of the acids and more efficient means for preventing the escape of heat give rise to a higher 'titer' and possibly to more concordant results, but the determinations take a longer time. To obtain comparable results it is therefore necessary to work always under substantially the same conditions (see also under *Tallow*).

Cold Test.—When lubricating oils are to be used in cold situations, it is frequently specified that they shall not lose their fluidity, or cease to flow, at a certain low temperature. They must then be tested by a 'cold test.' A uniform method of performing this test has not yet been adopted in this country and is perhaps not very necessary. Sufficiently reliable results may be obtained (when the exact method of conducting the test is not specified by the consumer) by placing the oil in a $6 \times \frac{3}{4}$ inch test-tube, cooling for thirty minutes by a freezing mixture of ice and salt maintained at $-5°$ or $-10°$ C., as may be required, and then noticing whether the oil still flows when the tube is inclined. For the methods and apparatus employed in Germany see *J.S.C.I.*, 1889, 423; 1890, 772.

Viscosity.—The viscosity of an oil represents its degree of *non-fluidity*. It is occasionally determined for purely analytical purposes, but generally in order to estimate the value as a lubricant. It is very doubtful whether the viscosity of an oil, as generally determined, does give a real estimate of its lubricating power, but it affords the only convenient method for obtaining an estimate of the lubricating value.

Many instruments have been devised for determining the viscosity of oils, nearly all of which are based on the principle of estimating the time required for a definite volume of the oil to flow through a narrow orifice. Redwood's *Viscosimeter* is the instrument almost universally employed for the purpose in this country. Other instruments, based on the same principle, are employed on the Continent and in America. Redwood's viscosimeter consists (fig. 3) of a silver-plated brass cylinder about 2 inches in diameter and 3·5 inches deep. The bottom of the cylinder is concave; at the centre there is inserted a piece of agate, through which is drilled a hole of accurate dimensions. The upper surface of the agate contains a hemispherical cup, into which may be fitted the bulb of the thermometer, or the ball on the end of the wire, in order to close the orifice. Inside the cylinder near the top is a piece of thick wire bent upwards at right angles and terminating in a point, which serves to indicate the position of the surface of the oil when the instrument is filled. The instrument is standardised by adjusting the position of this point until the time of

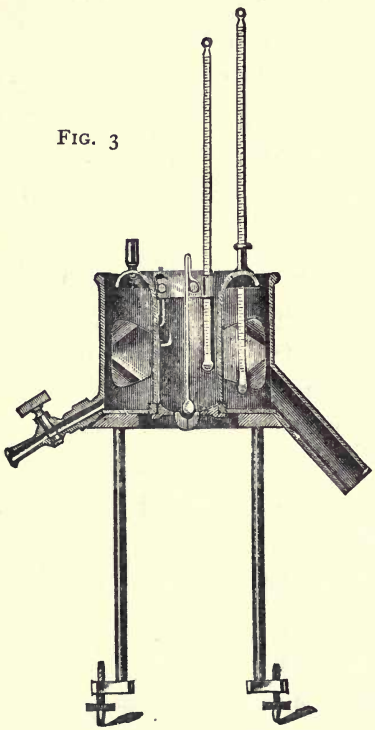


FIG. 3

outflow of 50 c.c. of (standard) rape oil is exactly 535 secs. It is of course always desirable to ascertain whether the instrument has been properly standardised by the makers; if not, the results will require correction before they can be compared with those of other instruments. The results obtained from the same instrument will always be comparable one with another. The cylinder is contained in an outer bath supported on a tripod provided with levelling screws, which should be so adjusted before use that the top of the cylinder, as shown by a spirit-level placed thereon, is exactly horizontal. The bath has at one side a tube, which can be heated by a burner, and at the other a tap, through which the water or other liquid can be run off. The stirrer is a cylinder of sheet copper bent outwards at the top, closely surrounding the oil-cylinder, and provided with wings which effectually agitate the liquid in the bath when the stirrer is revolved by means of the handle at the top. The stirrer also carries the thermometer by means of which the temperature of the bath is shown. A rod attached to the top of the oil-cylinder carries a clamp, which supports the thermometer in the oil. The apparatus is used as follows:

The oil, which must be free from dirt and water, is filtered, if necessary, through fine wire gauze. The oil-cylinder being clean and dry, and the orifice closed by the ball on the rod, the liquid in the bath is heated to the desired temperature, and the oil itself is brought to a temperature about 5° F. above that at which its viscosity is to be taken; it is then poured into the instrument until the point of the wire is just in the surface. The stirrer

is then kept in motion until the temperature of the oil is constant and at the desired point. If the oil is previously heated, as mentioned, this is a matter of only two or three minutes. A 50 c.c. graduated flask is placed with the neck just below the orifice of the agate, the stopper is then lifted from the agate and hung on the thermometer clamp, while at the same moment a stop-watch is started or the time taken by a watch with a seconds hand. The stop-watch is stopped, or the time taken, when the outflowing oil has just filled the flask up to the 50 c.c. mark. The production of froth in the flask may be to some extent avoided by running the oil down the side of the neck, or by using a flask with the graduation high up in the neck, when the froth formed in the flask is destroyed as it rises in the neck. The stirrer should be kept in motion and the temperature of the bath maintained at the right point during the whole time of outflow of the oil. The oil-cylinder is best cleaned out with filter paper; cloth or other material, which might leave fibres, should be avoided.

It would probably be better to leave the viscosities, as expressed in the times of outflow of 50 c.c., without further calculation. It is, however, not unusual to express the results in comparison with the viscosity of rape oil and to make a correction for the specific gravity of the oil. Thus if the viscosity of standard rape oil be 100 and its specific gravity 0·915, the viscosity of another oil, the time of outflow of 50 c.c. of which is t seconds (see p. 51), and the specific gravity s, is

$$\frac{100\,t\,s}{535 \times 0{\cdot}915}.$$

Viscosities are generally determined at the temperatures of 70°, 140°, and 210° F., and occasionally at higher temperatures. The viscosities at 70° F. give little information as to the behaviour of oils at the higher temperatures which they will probably have to experience in practice. For ordinary machinery and engine oils it is generally sufficient to take the viscosity at 140° F. The viscosities of cylinder oils should be taken at 210° F. or at higher temperatures. The viscosities decrease rapidly with rise in temperature, and the decrease is greater with some oils than others; but the viscosities at higher temperatures are generally in the same order, if not in the same ratio, as the viscosities at 140° F.

The viscosity, expressed as the time of outflow of a given volume, of a mixture of oils is far from being the mean of the viscosities of its constituents. It may, however, be calculated roughly from the formula given below.

Let t_1 and t_2 denote the times of outflow of 50 c.c. of each of two oils,

s_1 and s_2 be the specific gravities of the oils,

T and S be the time of outflow and specific gravity respectively of a mixture of a_1 volumes of the first oil with a_2 volumes of the second;

then
$$\frac{a_1+a_2}{\mathrm{T\,S}}=\frac{a_1}{t_1s_1}+\frac{a_2}{t_2s_2}$$

from which the time of outflow of the mixture, T, can be calculated, all the other values being known.

If the influence of the specific gravity be neglected, the above equation may be reduced to the form

$$a_1=a_2\frac{\mathrm{T}t_1-t_1t_2}{t_1t_2-\mathrm{T}t_2}$$

from which the proportions of a mixture required to have a definite viscosity can be roughly calculated.

In the following table are given the viscosities of a number of mineral oils at different temperatures as determined by Redwood's instrument (the temperature of 200° F. is the highest it is easy to maintain in the oil-cylinder when water is the liquid used in the jacket):

Description of Oil	Specific Gravity	Viscosity (time of outflow of 50 c.c. from Redwood's Viscosimeter)		
		70° F.	140° F.	200° F.
		secs.	secs.	secs.
No. 1 Russian Engine Oil	0·907	1110	122–130	53
American Engine Oil (·900–·907)	0·902	—	72	—
Bayonne Engine Oil (American)	0·912	637	101	50
Extra Filtered Cylinder Oil (American)	0·889	—	440	129
Kosmos Cylinder Oil (American)	0·888	—	315	100
Dark Cylinder Oil N. (American)	0·897	—	622	162
Victoria Cylinder Oil (Russian)	0·919	—	510	115

Flash Point.—The flash point of a combustible substance is that temperature to which it must be heated in order that the vapour it gives off may just take fire when a light is applied to it. The fatty oils and fats do not emit vapour at the ordinary temperatures; they require to be heated to temperatures of 250° C. and beyond before the vapours which are evolved form a combustible mixture with air.

It is frequently found necessary for fire insurance and other purposes to determine the flash points of fatty oils and mixtures of fatty oils with mineral oils. For this purpose the flash-point apparatus of Abel, Gray, and others, which is mainly used for substances producing an inflam-

mable vapour at temperatures little above the ordinary, is not suitable. It is better to determine the *open* flash point, which is obtained when the oil is heated in direct contact with the air and not in a closed vessel, as in the forms of apparatus mentioned above. The flash point by the open test is always a few degrees higher than the flash point taken by the closed test. The former is easily obtained by heating the oil in a beaker, with a thermometer suspended so that the bulb is at about the middle of the column of oil. When vapours begin to be evolved a small flame is passed just over the surface of the oil at intervals of every 2° C. rise in temperature, until the point is reached at which a blue flame momentarily passes over the surface. The thermometer then gives the open flash point. It is of course important that the temperature of the oil should rise slowly when it approaches the neighbourhood of the anticipated flash point.

A simple apparatus for the determination of open flash points has been described by Kissling (*J.S.C.I.*, 1899, 1114): the oil is placed in a porcelain crucible, 45 mm. deep and 40 mm. in diameter at the top, which is filled to within 10 mm. of the top. The crucible rests in a sand-bath and the whole arrangement is surrounded by a glass cylinder to exclude draughts. The temperature is permitted to rise through 2° C. every thirty seconds, which is the interval between successive applications of the test-flame.

Burning oils and paint oils are occasionally met with which contain petroleum oils of low flash point. The determination is in this case best made in the apparatus of Abel or Gray. Pure fatty oils should not give a flash point below 250° C. Anything much below this is to be

regarded as indicating the presence of mineral oils. The mineral oils generally mixed with fatty oils flash at temperatures between 190° and 220° C.

Solubility.—With certain few exceptions, the solubilities of oils and fats in different solvents give little indication of value as to their identity. All the oils and fats are soluble in ether, carbon bisulphide, chloroform, and benzene, and all, with the exception of castor oil, in petroleum ether. None of the oils dissolve to any considerable extent in cold alcohol except castor oil, which is soluble in every proportion, and croton oil. Palm-kernel oil, cocoanut oil, and oils containing a high proportion of free fatty acids, are comparatively soluble in alcohol. The varying proportion of free acids contained in any individual oil explains the divergent results obtained for the solubilities in alcohol, since the free acids are very soluble, but the glycerides only slightly soluble.

In an equal volume of glacial acetic acid (Valenta), castor oil and olive-kernel oil are completely soluble at the ordinary temperature, whilst rapeseed oil is insoluble in an equal volume of boiling acetic acid. The remaining oils dissolve at some temperature below the boiling-point and separate again on cooling. Valenta proposed to utilise the temperature at which the turbidity appeared as a means of recognising these oils, but Allen obtained temperatures generally very different from those noticed by Valenta. Cocoa butter was also found by Allen to be insoluble in the boiling acid. The divergent results in this case, too, are probably to be attributed to the varying amounts of free fatty acids.

The method of determining the solubilities of oils and

fats is quite simple: the oil and solvent are measured in a graduated stoppered cylinder, which is then well shaken. The mixture may then be transferred to a test-tube to heat to any required temperature.

Crismer (*J.S.C.I.*, 1895, 1069) has proposed to determine the critical temperature of solution of oils and fats in 90 per cent. alcohol. The oil is heated with alcohol in a sealed tube until complete solution occurs, and the temperature noted at which, on cooling, the solution becomes turbid. In view of the divergent results obtained by other solubility methods, this process is not to be recommended until Crismer's results have been confirmed by the examination of a large number of samples of each oil.

Refractive Index.—In certain cases, as in the examination of butter fat, a determination of the refractive index affords a valuable indication of purity, and in this and other cases it enables a distinction to be drawn between samples, which are certainly pure, and those which must be subjected to a chemical examination. This application to the purpose of a preliminary examination is very useful, since, when once the apparatus is in working order, a large number of samples can be rapidly tested. The instruments used for this purpose are next described.

The *Butyro-refractometer.* This instrument consists essentially of two prisms, placed together, with an air-space between, to form a rectangular block. The material and angles of the prisms are such that all the light passing into the first prism is totally reflected at its second internal face, which is in contact with the air-space, and also that the dispersion of the light, which passes through when

the space between the prisms is filled with pure butter-fat, is compensated. In the focus of the eyepiece is a scale, divided into one hundred parts; the tenth parts of each scale division can be estimated. When the space between the prisms is filled with oil, a portion only (the left) of the field is illuminated; the position of the line on the scale separating the bright from the dark side gives the reading of the instrument in degrees, which may be converted into actual refractive indices by means of a table. This dividing line is distinct and colourless when pure butter fat is being examined, by reason of the compensation for dispersion mentioned above. When other fats and oils are under examination the dividing line is broader and coloured, so that in order to obtain an exact reading it may be necessary to use monochromatic (sodium [1]) light and thus obtain the refractive index for the D line.

The portion of the instrument containing the prisms is surrounded by a hot-water jacket, so that it may be used for fats which are solid at the ordinary temperature. The hot water enters at D (see fig. 4) and leaves at E; it may be supplied from a large vessel by siphon action, but for continuous working the makers of the instrument supply a special apparatus, in which the water circulates under a constant pressure and is heated in a copper spiral.

To use the instrument, turn the pin F and move the portion B aside, thus exposing the prism face, which must be cleaned by a soft cloth moistened with ether. Move the instrument so that the prism B is horizontal, apply to it two or three drops of clear oil (or melted fat), place it against A and secure it by means of F. Adjust the mirror J

[1] Sodium chloride in a small platinum spoon is heated in the Bunsen flame.

so that the dividing line on the scale is distinct, pass a current of water of constant temperature (25° or 40° C.) through the apparatus, and, when the whole is thoroughly warmed and the line has assumed a fixed position, take the reading on the scale, note the colour (if any) of the line,

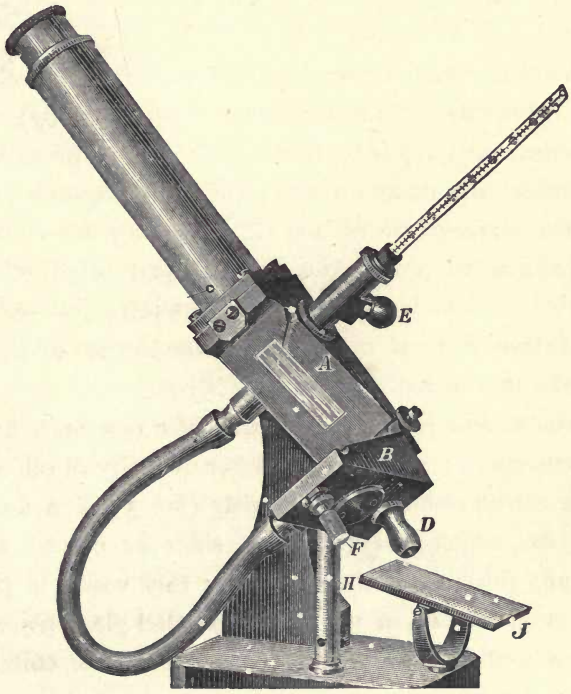


FIG. 4

and the temperature as shown by the thermometer. The reading is then to be corrected to the standard temperature, which is either 25° or 40° C. The decrease in the reading for every 1° C. rise in temperature is about 0.55 scale division. The refractive index may be calculated from the scale reading by means of the following table :

Degrees	Index of Refraction, n_D	Diff.	Degrees	Index of Refraction, n_D	Diff.
0	1·4220	0·0080	50	1·4593	0·0066
10	1·4300	0·0077	60	1·4659	0·0064
20	1·4377	0·0075	70	1·4723	0·0060
30	1·4452	0·0072	80	1·4783	0·0057
40	1·4524	0·0069	90	1·4840	0·0055
50	1·4593		100	1·4895	

The refractive indices of a number of fats and oils are given in the table of physical properties (pp. 62–67).

Marpmann (*J.S.C.I.*, 1901, 509) makes possible the examination of fats and waxes of high melting-point at the standard temperature of 40° C., by taking the refractive index of a solution of the wax in an equal weight of clove oil or terpeneless peppermint oil. The refractive index of the mixture is then the mean of the indices of its constituents, all expressed in scale divisions.

Amagat and Jean's *Oleorefractometer* is a more expensive instrument; it requires a larger quantity of oil, and is not so easily cleaned. It consists (fig. 5) of a metallic vessel C*y*, which has two glass sides *c c* placed at an angle and thus constituting a prism; this vessel is placed in an outer one, *t t*, in which are parallel glass plates *g g*.

The light from a gas flame passes down the collimator through the central part of the apparatus, and thence to the telescope L, which has a scale in the focus of the eye-piece. The central portion is surrounded by a water-jacket R R, which can be kept at a constant temperature by means of the lamp L P. The instrument is adjusted by moving the shutter so that, when the prism and outer vessel C*y* are both filled with the oil supplied as the standard, the edge of the shadow falls on the zero of the scale (fig. 5A). The

standard oil is then run out by the tap *r''*, and replaced by the oil to be examined, which has generally a different refractive power; consequently the edge of the shadow moves on the scale, and its position is noted. The scale is arbitrary.

Optical Rotation.—The majority of the fatty oils have little action on polarised light, consequently the

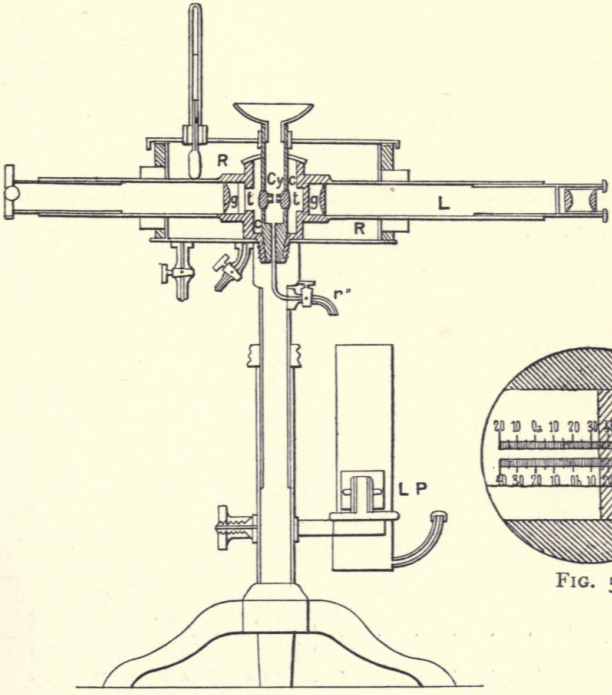


FIG. 5

FIG. 5A

determination of this constant is rarely of value. Croton oil, castor oil, and rosin oil are, however, strongly dextro-rotatory. Olive oil, from which camphorated oil is made, having a low rotatory power, the percentage of camphor may be estimated by means thereof (*J.S.C.I.* 1900, 861).

THE PHYSICAL PROPERTIES OF

——	Specific Gravity	Melting Point	Solidifying Point
DRYING OILS:			
Linseed	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·930—0·937; $\frac{99}{15}$ 0·8809	−16° to −20° C	−16°, −27° C.
Hempseed oil	$\frac{15}{15}$ 0·925—0·933	—	−27·5°
Wood oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·9343—0·9431	—	Below −17° (?)
Candle-nut oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·9256; $\frac{15}{15}$ 0·920	—	—
Walnut oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·9212—0·9259	—	—
Poppyseed oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·9235—0·9268 (0·937)	—	−18°
Nigerseed oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·9248—0·9270	—	Below −9°
SEMI-DRYING OILS:			
Sunflower-seed oil	$\frac{15}{15}$ 0·924—0·926	—	—
Maize oil	$\frac{15}{15}$ 0·9216; $\frac{15{\cdot}5}{15{\cdot}5}$ 0·9244—0·9284	—	−36°
Pumpkin-seed oil	$\frac{15}{15}$ 0·917—0·925	—	−15°
Sesamé oil	$\frac{15}{15}$ 0·921—0·926	—	—
Cottonseed oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·920—0·928; ($\frac{15}{15}$ 0·930)	—	—
Rapeseed oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·912—0·917	—	−1° to −10°
Black mustard oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·9155—0·919	—	−17·5°
White mustard oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·912—0·915	—	−8° to −16°
NON-DRYING OILS AND SOLID FATS			
Earthnut oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·916—0·9256	—	−3° to −7°
Almond oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·917—0·9195	—	−10° to −21°
Croton oil	15° C. 0·940—0·960; 100° C. 0·8874	—	−16°
Grapeseed oil	15° C. 0·920—0·956	—	−10° to −17°
Castor oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·960—0·967	—	−10° to −18°
Olive-kernel oil	$\frac{15}{15}$ 0·918—0·920	—	—

THE OILS, FATS, AND WAXES

Refractive Index	Optical Rotation in 200 mm. tube	The Insoluble Fatty Acids		
		Specific Gravity	Melting Point	Solidifying Point
15° C. 1·481; 40° C. 1·4740–1·4750; 60° C. 1·4674	+ 6′, − 18′	$\frac{15·5}{15·5}$ 0·923; $\frac{100}{100}$ 0·8925	17°–24° C.	13°–17° C.
—	—	—	17°–19°	16°
—	—	—	30°–49°	17°–34°
15° C. 1·4756–1·4759; 25° C. 1·4759	—	—	20°–21°	13°
40° C. 1·4689–1·4710	+ 16′	—	16°–20°	14·3°
40° C. 1·4680	0, + 4′	—	20°–21°	16°–19°
40° C. 1·4678	0, + 18′	—	—	—
—	—	—	22°–24°	17°–19·8°
15° C. 1·4765–1·4767 60° C. 1·4605	—	100° C. 0·8529	17°–22·4°	—
25° C. 1·4723–1·4738	—	—	26·5°–28·5°	—
15° C. 1·4746; 40° C. 1·4647–1·4656; 60° C. 1·4580	0·8 – 1·6°	—	24·2°–31°	18°–28°
15° C. 1·475; 40° C. 1·4647; 60° C. 1·4586	—	$\frac{100}{100}$ 0·8816	35°–40°	32°–38°
15° C. 1·4745; 40° C. 1·4653; 60° C. 1·4584	− 5′ − 10′	$\frac{100}{100}$ 0·8758	17°–21°	12°–16·5°
40° C. 1·4655	−17′ to −30′	—	16°–17°	15°
40° C. 1·4649	− 9′	—	15°–16°	—
15° C. 1·4731; 40° C. 1·4642; 60° C. 1·4564	− 7′ to + 24′	$\frac{100}{100}$ 0·8475–0·864	27°–32°	22°–30°
25° C. 1·4684–1·4672; 60° C. 1·4555	—	—	14°	5°–14°
27° C. 1·4768	—	—	—	16·4°–19°
—	—	—	23°–25°	18°–20°
15° C. 1·4803; 40° C. 1·4720; 60° C. 1·4647	+8° + 9°	—	13°	3°
1·4682–1·4688	—	—	—	—

THE PHYSICAL PROPERTIES OF

——	Specific Gravity	Melting Point	Solidifying Point
NON-DRYING OILS AND SOLID FATS (*cont.*)			
Olive oil	$\frac{15}{15}$ 0·914 $\frac{15·5}{15·5}$ 0·917—0·920 $\frac{100}{100}$ 0·862—0·864	—	—
Ben oil	$\frac{15}{15}$ 0·9120	—	0°
Neatsfoot oil	$\frac{15}{15}$ 0·914—0·919	—	—
Lard	$\frac{15}{15}$ 0·931—0·938 $\frac{60}{15·5}$ 0·886 $\frac{100}{15}$ 0·860—0·862	40°—45°	27°—30°
Lard oil	$\frac{15·5}{15·5}$ 0·917 100° 0·8626	—	—
Mahwa butter (Bassia latifolia)	$\frac{100}{100}$ 0·8943—0·8975	23·0°—31·0°	19°—22°
Mowrah seed oil (Bassia longifolia)	100° C. 0·8854	35·5°—42°	25°—36°
Palm oil	$\frac{60}{15·5}$ 0·8837 $\frac{100}{100}$ 0·858	—	—
Bone fat	$\frac{15·5}{15·5}$ 0·914—0·916 $\frac{60}{15·5}$ 0·894	21°—22°	—
Tallow (beef)	$\frac{60}{15·5}$ 0·901, $\frac{100}{100}$ 0·860	42°–48°	—
Tallow (mutton)	$\frac{60}{15·5}$ 0·907	47°–51°	—
Tallow oil	$\frac{100}{100}$ 0·794	—	—
Cocoa (cacao) butter	$\frac{100}{100}$ 0·903—0·904	23·5°–33°	—
Vegetable tallow	—	37°–46°	24°–31°
Butter-fat	$\frac{40}{15·5}$ 0·904—0·9102 $\frac{100}{15}$ 0·865—0·870 $\frac{100}{100}$ 0·909—0·914	29°–34·6°	19°–20°
Palm-kernel oil	$\frac{60}{15·5}$ 0·896, $\frac{99}{15}$ 0·873	23°–28°	—
Japan wax	$\frac{60}{15·5}$ 0·902—0·907 $\frac{98}{15·5}$ 0·875	42°–55°	—
Myrtle wax	$\frac{98}{15·5}$ 0·875	40°–44°	39·5°–43°
Cocoa-nut oil	$\frac{60}{15·5}$ 0·897, $\frac{99}{15·5}$ 0·874	—	—

THE OILS, FATS, AND WAXES

Refractive Index	Optical Rotation in 200 mm. tube	The Insoluble Fatty Acid		
		Specific Gravity	Melting Point	Solidifying Point
15° C. 1·4718; 40° C. 1·4602–1·4635; 60° C. 1·4546	+0·13°	$\frac{60}{15}$ 0·878 $\frac{100}{100}$ 0·8749	22°–26°	21°–24°
—	—	—	—	—
15° C. 1·473; 60° C. 1·4559	—	$\frac{100}{100}$ 0·8742–0·8800	28°–30°	16°–26·5°
40° C. 1·4586–1·4602; 60° C. 1·452	—	—	35°–45°	37°–39°
—	—	0·885	35°	31°
40° C. 1·4605–1·4609	—	—	45°	40°
—	—		54·5°–55·5°	52·5°
60° C. 1·451	—	$\frac{100}{100}$ 0·870	47°–50°	40°–45°
60° C. 1·451	—	—	30°–42°	28°–32°
40° C. 1·4987; 60° C. 1·442	—	—	45°–47°	42·5°–45°
60° C. 1·4531	—	—	45°–50°	43°–46°
—	—	—	—	35°–37·5°
40° C. 1·4482–1·4580; 60° C. 1·450–1·458	—	—	45°–51°	47·2°–49·2°
—	—	—	40°–57°	34°–48°
25° C. 1·4590–1·4620; 40° C. 1·4532–1·4565; 60° C. 1·445–1·450	—	$\frac{20}{20}$ 0·9106–0·9242	38°–43°	—
40° C. 1·450; 60° C. 1·443	—	—	21°–28°	—
60° C. 1·450	—	—	56°–57°	53°–56·5°
—	—	$\frac{99}{15·5}$ 0·837	47·5°	46·0°
40° C. 1·445–1·450; 60° C. 1·442	—	—	24°–27°	19°–23°

THE PHYSICAL PROPERTIES OF

——	Specific Gravity	Melting Point	Solidifying Point
MARINE ANIMAL OILS:			
Menhaden oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·932–0·935	—	−4°
Sardine oil	$\frac{15}{15}$ 0·925–0·928 ($\frac{15}{15}$ 0·916–0·933)	20°–22°	—
Cod-liver oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·922–0·935	—	—
Seal oil	$\frac{15}{15}$ 0·924–0·927	—	—
Whale oil	$\frac{15}{15}$ 0·920–0·927	—	—
Porpoise oil	$\frac{15}{15}$ 0·926–0·927	—	−16°
Dolphin oil	$\frac{15}{15}$ 0·918	—	—
LIQUID WAXES:			
Sperm oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·875–0·886	—	—
Arctic sperm (bottlenose) oil	$\frac{15{\cdot}5}{15{\cdot}5}$ 0·880	—	—
SOLID WAXES:			
Spermaceti	$\frac{98}{15{\cdot}5}$ 0·808–0·816	43°–49°	42°–45°
Beeswax	$\frac{15}{15}$ 0·961–0·970	62°–65°	—
Carnaüba wax	$\frac{15}{15}$ 0·99–1·0	83°–86°	—
Woolwax	$\frac{15}{15}$ 0·973; $\frac{60}{15{\cdot}5}$ 0·885	36°–43°	—

NOTES.—It has not been thought necessary to quote the authorities for the figures in this table. Figures in brackets represent abnormal results, possibly due to the examination of a genuine oil of

THE OILS, FATS, AND WAXES

Refractive Index	Optical Rotation in 200 mm. tube	The Insoluble Fatty Acids		
		Specific Gravity	Melting Point	Solidifying Point
—	—	—	—	—
15° C. 1·479	—	—	30°–31°	—
15° C. 1·482; 40° C. 1·4729; 60° C. 1·462–1·466	—	$\frac{60}{60}$ 0·882	21°–25°	—
15° C. 1·4784; 60° C. 1·4619	—	—	—	—
15° C. 1·476; 60° C. 1·4603	—	—	—	—
—	—	—	—	—
—	—	—	—	—
15° C. 1·4675; 40° C. 1·4567; 60° C. 1·4508	—	$\frac{15}{15}$ 0·899	13·3°	—
—	—	—	10·3°–10·8°	—
—	—	—	—	—
—	—	—	—	—
—	—	—	—	—
60° C. 1·465	—	—	41·8°	40°

unusual character, or of a sample which has undergone modification through keeping.

CHAPTER III

THE CHEMICAL PROPERTIES OF OILS, FATS, AND WAXES FROM THE ANALYTICAL STANDPOINT.

General.—Oils, fats, and waxes, as already stated, are composed mainly of esters. The fatty oils are generally complex mixtures of the glycerides of several acids; the waxes (liquid and solid) contain several esters derived from different alcohols and acids. In the present state of our knowledge it would generally be most difficult and tedious, and frequently impossible, to determine the exact quantity of each constituent of a fat or wax. In default of methods by which the percentage of each constituent can be determined, certain chemical properties of the entire oil—which, it is needless to say, depend on the properties of each constituent and the proportions of the several constituents present—are estimated. To take an example: the glycerides of the different acids require to saponify them different amounts of caustic potash, which can be calculated. Any particular oil, which we must regard as containing always the same glycerides in practically the same proportion, requires for its saponification always the same proportion of caustic potash, which can be accurately determined. Although the quantity of potash required to saponify an oil does not give in most cases an indication of the actual glycerides present in the oil, it however affords a property of the oil capable

of numerical expression and varying for the same oil within narrow limits. Thus, if an unknown oil is found to require a certain proportion of caustic potash for its saponification, we can say, knowing the proportions of potash required by all the ordinary oils, that the unknown oil may be one of a certain class, that it cannot be one of certain other classes. Other chemical or physical properties might then be employed to pick out, from the class to which the oil evidently belonged, the particular oil with which, in the end, it would be found to be identical. The method here sketched indicates very roughly the principle upon which the identification of an unknown oil is conducted. In order to determine whether a particular sample of oil is pure, certain of the chemical and physical properties are examined. If the results all fall within the limits of the properties of the particular oil, it is assumed to be pure, otherwise probable adulterants must be looked for. Examples to illustrate the methods will be given in future chapters.

When a fatty oil contains mineral oil, rosin oil, or other similar unsaponifiable matter, certain of the chemical properties of the fatty oil itself cannot be directly determined, since the behaviour of the unsaponifiable oil in the reactions involved is unknown or variable. It is then necessary to apply the reactions to the free fatty acids of the oil, which can be obtained free from the unsaponifiable matter. When the chemical properties of the fatty oils are determined, the glycerin of the glycerides may have a disturbing effect on the reactions, preventing the behaviour of the fatty acids from being clearly expressed. In such cases the reactions are therefore best applied to the free

fatty acids. For these reasons the application of many of the reactions to the free acids is of equal importance with the application to the oils themselves. The method by which the free fatty acids can be isolated from the fats is here given:

Put 25 c.c. of water in a 6- or 7-inch evaporating basin on the water-bath, add 20 grms. of caustic potash sticks, and when dissolved add 50 c.c. of absolute alcohol or purified methylated spirit.[1] Then pour in slowly about 50 grms. of the oil, stirring well all the time with a glass rod, and continue to stir until the contents of the dish form a clear pasty mass. Care must be taken that no oil on the sides of the dish escapes saponification. It is generally well to add more alcohol and stir until the whole is dissolved, in order that no oil may remain undecomposed. The stirring is continued until most of the alcohol has evaporated, when the mass is transferred to a 1,000 c.c. beaker, dissolved in much water, and the solution boiled until all the alcohol is driven off. Dilute sulphuric acid is then cautiously added, continuing the boiling until all the soap is decomposed and excess of acid is present. Boiling is still continued until the fatty acids have separated at the surface as a clear oil, and the aqueous liquid beneath is

[1] Methylated spirit may be made sufficiently pure for this and many other purposes in the following manner: Weigh into a 1 gallon tin bottle about 6 lb. of methylated spirit (free from petroleum; the ordinary methylated spirit which is sold retail will not do), add 10 oz. of stick caustic potash, and boil gently under an inverted condenser for two hours. Then distil under a distilling column of glass beads, or other ordinary form. Take samples of the distillate from time to time, and test by boiling with a small fragment of caustic potash; when the spirit remains quite colourless, the distillate may be collected for use. Probably some 200–300 c.c. will be rejected. The distillation is continued so long as spirit comes over freely; the temperature of the vapour will rise to about 85° C.

almost clear. The latter is then siphoned off as completely as possible, and the acids boiled repeatedly with distilled water until the aqueous liquid siphoned off is free from mineral acid, as shown by its neutral reaction to litmus paper. It will be found expedient to boil the liquids by means of a current of steam generated in another vessel, rather than over an open flame. If lead is present in the oil, as in boiled oil, hydrochloric acid should be used in place of sulphuric acid. The clear oil, consisting of the free fatty acids, is finally drawn off free from globules of water, placed for a short time in the drying oven at 105° C., and filtered (also in the oven) through a dry paper. If the oil contained unsaponifiable oil, the latter would have to be extracted by ether after saponifying (see p. 109) and before acidifying.

The acid value of an oil gives the number of milligrammes of caustic potash, KOH, required to neutralise the free acids contained in 1 grm. of the oil. The free acids in an oil are also often calculated and expressed as the percentage of oleïc acid; now 1 mgrm. of caustic potash neutralises 5·04 mgrms. of oleïc acid, consequently an acid value of 1 corresponds to 0·504 per cent. of oleïc acid. Koettstorfer gives the acidity of an oil as the number of c.c. of normal potash solution required to neutralise the free acid in 100 grms. of oil; this method of stating results is, however, very seldom employed.

For the estimation there is required a standard caustic potash solution, which may be either semi- or fifth-normal, the latter being preferable. The solution may be in water or alcohol. It is frequently stated that there is no advantage in using alcoholic potash, but the writer prefers it on

account of the greater ease with which the titration is effected. Dilute 19 c.c. of strong hydrochloric acid to 1 litre, and estimate the exact strength by precipitating 10 c.c. with excess of silver nitrate in the ordinary manner. Dissolve 15 grms. of pure caustic potash in the smallest possible quantity of water, pour into a large volume of purified methylated spirit, shake well, fill up to 1 litre, let stand twenty-four hours, and filter. Heat 1 grm. of phenolphthalein with 100 c.c. of purified methylated spirit on the water-bath, let cool, and filter.

The strength of the potash solution is found by titrating with it 25 c.c. of the standard hydrochloric acid, using two or three drops of the phenolphthalein solution as indicator. It is necessary to repeat this standardisation at short intervals (every week) in the case of alcoholic potash; aqueous potash retains its strength unaltered for much longer periods. The estimation is conducted as follows: Measure into a flask or small bottle about 30 c.c. of benzene (or ether) and 60 c.c. of alcohol, add a few drops of the phenolphthalein solution, and then the alcoholic potash, a drop at a time, until the liquid, after well shaking, is just coloured a rose tint. Then weigh[1] into the vessel about 10 grms. of the oil or fat, heat on the water-bath to melt and dissolve a solid fat, shake well and run in the alcoholic potash, with continuous shaking, until the liquid is just coloured red. The volume of potash used then gives the

[1] Liquid oils are best weighed from an ordinary weighing bottle standing on a watch-glass. In the case of solid fats, the bottle containing the fat may be weighed, then the fat melted by gently warming, a portion of the melted fat poured out for the operation, the bottle allowed to cool, and again weighed when quite cold. Hard fats may be cut up and weighed from a bottle or watch-glass in the same manner as any other solid.

acid value. If the oil contains much free acid, a smaller quantity than 10 grms. should be taken. One grm. is sufficient for the free fatty acids themselves, or for a fat which is nearly all free acid. If the oil is almost neutral a larger quantity than 10 grms. should be used—it is not convenient or necessary to exceed 20 grms.

Example: 9·786 grms. of oil required 5·2 c.c. of fifth-normal alcoholic potash for neutralisation. 1 c.c. of $\frac{1}{5}$ N potash contains 11·2 mgrms. of KOH, therefore the acid value is $\frac{5{\cdot}2 \times 11{\cdot}2}{9{\cdot}786} = 5{\cdot}9$. This result corresponds to 2·97 per cent. of free acids, calculated as oleïc acid (see p. 71).

When phenolphthaleïn is used as the indicator in titrating dark or reddish oils, the exact point, at which the red colouration due to the indicator appears, is obscured by the colouring matter of the oil. In such cases an alcoholic solution of the dyestuff known as Alkali Blue is a very efficient substitute for the phenolphthaleïn solution; it is made by boiling 1 grm. of the dyestuff with 100 c.c. of alcohol, allowing to stand some hours, and filtering. Some 2–5 c.c. of this solution are used, according to the depth of colour of the oil. The standard potash is added until the bluish or greenish colour changes to red. According to Freundlich (*J.S.C.I.*, 1902, 75), the commercial form of the dyestuff which should be used is Alkali Blue II OLA (Meister, Lucius, & Brüning); 10 c.c. of a 2 per cent. solution are prescribed. This quantity appears somewhat excessive.

This method determines the total quantity of free acid in the oil. Now certain oils may contain free mineral acid

introduced in the refining process. If it be required to determine the mineral acid (when present) and the free fatty acids separately, the total acid may be determined as above, and then the mineral acid (determined as in Chapter IV., p. 106) deducted, in order to give the free fatty acids.

The quantity of free acid in any particular oil may vary greatly. It depends mainly upon the conditions to which the raw material has been subjected in the process of extracting the oil or before the extraction. Certain fats contain very large proportions of free acids. Palm oil is commonly found to contain as much as 50 per cent. of free acid, while samples have been known to consist almost entirely of free acid. Linseed oil expressed from damaged seed also contains very large proportions of free acids. The animal oils, as a rule, contain little free acid. In view of the varying quantity of free acid in an oil, this property cannot be used as a means of identification.

Edible oils must contain not more than a small percentage of free fatty acids. They should, however, not be quite neutral, when the flavour would be insipid. It is not possible to state any particular proportion of free acids as the best for an edible oil, since the requirements of different markets vary. Two per cent. may be given as an approximate limit. It is stated that butyric acid is added to oil which contains too little free acid. Lubricating oils should be as far as possible free from free fatty acids, which corrode the brasses. It is not unusual for owners of machinery to stipulate that their lubricating oils shall not contain more than a certain small proportion of free acids.

The Saponification Value.—The saponification value of an oil or fat is the number of milligrams of caustic potash, KOH, required to saponify one gram of the oil. This property is frequently calculated and expressed in a different manner: The *saponification equivalent* is the weight of fat saponified by one equivalent—*i.e.* by 56 parts —of caustic potash.

For the purpose of determining the saponification value we require semi-normal solutions of hydrochloric acid and alcoholic potash. Dilute 47 c.c. of hydrochloric acid to one litre. The solution is approximately semi-normal. Determine its exact strength by precipitating 5 c.c. with excess of silver nitrate in the well-known manner. Dissolve 35 grms. of pure caustic potash in the smallest quantity of water (about 30 c.c.), pour into 500 c.c. of purified methylated spirit (see p. 70), shake, fill up to 1 litre, let stand twenty-four hours, and filter. The strength of this solution will diminish slightly on keeping, in proportion as it is exposed to the air; it is therefore necessary to determine the strength whenever it is used.

The estimation may be conducted as follows: Weigh 1·5 to 2 grms. of the oil into a dry 4-oz. conical flask. Measure into the flask 25 c.c. of the semi-normal alcoholic potash, and at the same time measure a further volume of 25 c.c. into a similar flask. Attach the flask containing the oil to an inverted condenser, place it on the boiling water-bath, and boil gently for thirty minutes, shaking the flask from time to time until all the oil has dissolved. At the same time place the second flask on the water-bath; when it is heated to boiling add 1 c.c. of the phenolphthalein solution (page 72) and titrate with the semi-normal hydro-

chloric acid. The strength of the alcoholic potash is thus found under conditions parallel with those of the estimation itself. Saponification as a rule is complete in thirty minutes; remove the flask from the water-bath, add 1 c.c. of phenolphthaleïn, and titrate with the semi-normal hydrochloric acid as before. The difference between the volumes of hydrochloric acid used in the two titrations is equivalent to the caustic potash consumed in saponifying the oil. The saponification value can thus be calculated.

Example.—1·961 grms. of oil were saponified by boiling with 25 c.c. of alcoholic potash. In the titration 5·1 c.c. of semi-normal hydrochloric acid were required; 25 c.c. of the alcoholic potash in the blank experiment required 19·05 c.c. of hydrochloric acid. Thus the potash consumed in saponifying the oil is able to neutralise $19{\cdot}05 - 5{\cdot}1$ c.c. $= 13{\cdot}95$ c.c. of semi-normal hydrochloric acid. The saponification value is therefore $\frac{13{\cdot}95 \times 28}{1{\cdot}961} = 199{\cdot}2$ mgrms. of KOH.

In the case of dark-coloured oils Alkali Blue (see page 73) can be used in place of phenolphthaleïn. It is necessary always to use some considerable excess of caustic potash, otherwise the saponification may not be complete; should the titration with hydrochloric acid only require 1 to 2 c.c., the experiment must be repeated with a smaller weight of oil. The waxes are saponified with much greater difficulty than the oils and fats; in their case the operation should be conducted in a bottle, with the stopper securely wired in, heated on the water-bath.

The determination of the saponification value affords a simple and rapid method of estimating the proportion of

unsaponifiable oil in an animal or vegetable oil, when the nature of the latter is known and the proportion of unsaponifiable matter is not too small. It also affords a rapid method of determining small quantities of animal or vegetable oils present in mineral oils. These uses will be readily understood from the example given below. In either case it is advisable to take a larger quantity of oil than 2 grms., say 4–5 grms. (there must always be a considerable excess of caustic potash), and to boil with frequent shaking for one hour in order that saponification may be complete.

Example.—1·9777 grm. of a lubricating oil containing rapeseed oil was saponified by means of 25 c.c. of alcoholic potash; in titrating back 13·75 c.c. of hydrochloric acid were used. In a blank experiment 25 c.c. of potash required 20·6 c.c. of hydrochloric acid; 1 c.c. of hydrochloric acid was equivalent to 0·0271 grm. of KOH; 20·6 − 13·75 = 6·85. Therefore the saponification value of the oil was $\frac{27{\cdot}1 \times 6{\cdot}85}{1{\cdot}977} = 94$. Taking the saponification value of rape oil as 178, the percentage of that oil in the mixture was $\frac{94 \times 100}{178} = 52{\cdot}8$.

Ester Value.—The saponification value as determined as above gives the weight of caustic potash required to neutralise the free acids *plus* the weight of potash required to saponify the neutral esters in one grm. of the fat. The latter quantity is known as the true saponification value, or ester value. The ester value is, of course, obtained by subtracting the acid value from the saponification value. It is evident that the acid value and ester

value may both be determined on the same quantity of oil by weighing 1·5–2 grms., titrating in alcoholic solution with fifth-normal alcoholic potash, adding excess of semi-normal potash, boiling for thirty minutes, and titrating the excess with semi-normal hydrochloric acid.

The Hehner Value gives the percentage of insoluble acids contained in a fat. A small quantity of the oil is saponified, the acids liberated, washed, and weighed directly. Dry in the oven at 100° C. a close-textured filter paper in a funnel with a short neck standing in a beaker; allow to cool in the balance-case, and weigh. Put 2–3 grms. of caustic potash in a 6-inch basin on the water-bath, add 2 c.c. of water, and when the potash is dissolved add 25 c.c. of alcohol; then weigh accurately into the dish about 4 grms. of the oil, stir and boil gently until a clear liquid is obtained; more alcohol may be added if required. When a clear solution is obtained and saponification is complete, evaporate down until the alcohol is expelled, dissolve the residue in hot water, and add to the clear solution dilute sulphuric acid in some excess. Heat until the fatty acids separate at the surface as a clear layer, then transfer to the dried filter paper, which remains in the funnel in which it was dried. The paper must first be filled with water, and must remain partially filled with water during the whole time the acids are being washed in it. There is no difficulty in removing the whole of the acids from the dish by means of a jet of hot water. The acids in the funnel are to be washed with hot water until the washings no longer have an acid reaction to blue litmus paper, for which purpose a large volume of water may be required (one or two litres) if the fat contains one

of the lower fatty acids which, though soluble in water, is only slightly soluble. When the washings are neutral, the filter is immersed in a vessel of cold water up to the level of the acids inside the funnel, which then usually solidify, or at least thicken. The funnel is then lifted out, the water allowed to drain off, the funnel returned to the beaker in which it was first dried, and the whole dried in the oven at 100° C. for two hours, when it is allowed to cool in the balance-case and weighed. It is again dried for thirty minutes and weighed ; the difference should be small, but a constant weight cannot be obtained, though the slight volatility of the fatty acids is to some extent counteracted by their oxidation.

The Hehner value of most oils and fats is about 96; only in the case of butter-fat, cocoanut oil, and one or two other fats, all of which contain a considerable quantity of the lower and soluble fatty acids, is the Hehner value appreciably smaller.

The process by which the fatty acids are isolated and weighed in determining the Hehner value may be adapted to other purposes—*e.g.* to the determination of the fatty acids in soap and in Turkey-red oil.

The Reichert-Meissl Value gives a measure of the volatile (soluble) fatty acids contained in a fat. For reasons which will appear later it is difficult to estimate the total amount of volatile acids, consequently the Reichert-Meissl value gives the amount of caustic potash (expressed in c.c. of a deci-normal solution) required to neutralise the volatile fatty acids in the distillate obtained by treating 5 grms. of the fat in a particular manner. (In the original process of Reichert, which differed otherwise only in small

details from the amended method due to Meissl, only 2·5 grms. of fat were taken.)

The process of Meissl is substantially as follows: Weigh 5 grms. of the oil or purified fat into a 200 c.c. flask, add 36 c.c. of absolute alcohol (purified by distillation with caustic potash, see p. 70), 14 c.c. of water, and about 2 grms. of caustic potash (sticks). Saponify by heating on the water-bath with frequent shaking, distribute the soap round the lower part of the flask, and continue to heat until all the alcohol is driven off. Blow in a current of air, filtered through cotton-wool, for a few minutes to remove the last traces of alcohol. Add 100 c.c. of water and dissolve the soap in it, then add 40 c.c. of dilute sulphuric acid (1 volume of acid to 10 volumes of water), and drop in a few fragments of ignited pumice to prevent bumping. Fit the flask with a cork carrying a short straight adaptor, the narrower end of which, inside the cork, is ground off at an angle. Connect the upper end of the adaptor [1] with a condenser in the ordinary manner by means of a tube bent at an acute angle and ground off at the ends. Distil slowly and regularly, at such a rate that 110 c.c. pass over in about sixty minutes. Collect the distillate in a graduated flask or cylinder, filter through a small paper, take 100 c.c. of the filtrate, add a little phenolphthaleïn, and titrate by deci-normal caustic potash. Then conduct a blank experiment with the same alcohol, caustic potash, and sulphuric acid, in exactly the same manner and the same apparatus. Deduct the volume of deci-normal potash required from that used in the first experiment, add 10 per cent.

[1] Any other modification of apparatus designed to prevent spirting over, may of course be used.

(*i.e.* multiply by $\frac{110}{100}$), and the result is the Reichert-Meissl value of the fat. In distilling under these conditions by no means the whole of the volatile acids passes into the distillate ; thus it is requisite to work under precisely the same conditions in order to obtain comparable results. A quantitative estimation of the volatile acids may be obtained by distilling in steam, but the process is too lengthy for ordinary use.

The volatile fatty acids are also soluble in water ; the volatility and solubility decrease to much the same extent with increasing molecular weight. The results of a process for titrating the soluble acids would thus be more or less comparable with the Reichert-Meissl values obtained by distillation. Several processes with this object have been proposed ; they consist essentially in saponifying, adding a quantity of acid exactly equivalent to the potash used in saponification, filtering off the acids, washing, and titrating the filtrate with deci-normal potash. These processes have no advantage over the distillation methods : it is very difficult to wash out the whole of the less soluble acids (see *Hehner value*), and if the total soluble acids are not determined it is not possible to devise a method of washing to give constant results. A saving of time would result from combining the determination of the soluble acids in this manner with estimations of the Hehner and saponification values.

The Reichert-Meissl value depends on the quantity of volatile acids and on their mean molecular weight. The variation is well shown by the following figures : Blumenfeld and Seidel (*J.S.C.I.*, 1900, 914), by saponifying,

acidifying, distilling in steam, and extracting with ether, isolated 4·53 and 15·1 per cent. of volatile acids from palm-kernel and cocoanut oils respectively; the Reichert-Meissl values of these oils are about 5 and 7. It is evident that the mean molecular weight (and also the volatility) of the volatile acids of cocoanut oil must be much higher than the mean molecular weight of the palm-kernel oil acids.

The great majority of the fats and oils contain very small quantities of the glycerides of volatile acids. Only butter-fat, palm-kernel, maize, croton, and cocoanut oils, and certain fish oils, possess high Reichert-Meissl values. In the examination of most of these oils a determination of the R.-M. value is naturally of great importance.

The Acetyl Value.—When an organic compound containing hydroxyl groups of alcoholic function is heated with acetic anhydride, acetic esters are generally produced. If the acetylated compound be isolated and its ester value (see p. 77) determined, this value is a measure of the hydroxyl groups in the original substance. Benedikt and Ulzer applied this method to the estimation of the acetyl value of fatty acids in the following manner: 25 grms. of the isolated acids (p. 70) were boiled with an equal weight of acetic anhydride under a return condenser for two hours; the product was poured into a large volume of water, and the mixture boiled for some hours. The aqueous solution was then siphoned off, and the oil boiled with fresh quantities of water until the latter remained neutral. The acetylated acids were then dried and filtered, and their acid and saponification values determined. The difference between the two gave the acetyl value, *i.e.* the

number of milligrammes of caustic potash, KOH, required to saponify the acetyl esters in 1 grm. of the acetylated fatty acids. This process, by which most of the acetyl values given in the literature were determined, has been shown by Lewkowitsch to be untrustworthy ; the reasons are given in the following paragraph.

When a fatty acid of the acetic series, such as stearic acid, is boiled with acetic anhydride, it is partially converted into its anhydride:

$$(CH_3.CO)_2\,O + 2C_{17}H_{35}.CO.OH = 2CH_3.CO.OH + (C_{17}H_{35}.CO)_2O.$$

These fatty acid anhydrides are very stable, and are not entirely decomposed in the boiling with water to which the acetylated acids are subjected in the above process of Benedikt and Ulzer. They are, however, saponified readily by potash, and thus the acetylated oil acquires a higher saponification value, leading to the deduction of a fictitious acetyl value, which is actually obtained when the higher acids of the acetic series are subjected to Benedikt and Ulzer's process. The pure anhydrides of stearic, palmitic, oleïc, and erucic acids have been made by Albitzky (*J.S.C.I.*, 1900, 358) by heating the acids with acetic anhydride in a sealed tube.

To replace Benedikt and Ulzer's method, Lewkowitsch (*J.S.C.I.*, 1890, 847 ; 1897, 503 ; 1900, 74) has devised the following process, in which is determined the number of milligrammes of caustic potash, KOH, required to neutralise the acetic acid formed on saponifying 1 grm. of the acetylated oil or fat : 10 grms. of the *original fat* (not the fatty acids) are boiled with an equal volume of acetic

anhydride for two hours under a return condenser. The product is then transferred to a large beaker and boiled with 400–500 c.c. of water for thirty minutes, the aqueous liquid is siphoned off and the boiling with water twice repeated. In order to prevent bumping a slow current of carbon dioxide is passed into the liquid, or the heating may be accomplished by blowing in steam. Three washings with water suffice to remove all the acetic acid; too prolonged washing decomposes the acetyl compounds and leads to low acetyl values. The acetylated oil is finally freed from water and filtered through a dry paper in an oven at 100–105° C. The acetylated oil may be quantitatively collected and weighed, as in the determination of the Hehner value (p. 78); an excess of weight over that of the original oil then indicates the introduction of acetyl groups. The actual separation of the acetic acid may be conducted by a process involving either distillation or filtration:

(a) *The distillation process.* Weigh accurately about 5 grms. of the acetylated oil into a flask, and saponify with alcoholic potash exactly as described for the determination of the Reichert-Meissl value (p. 80). After removing the alcohol and acidifying, blow in a current of steam until 600–700 c.c. of distillate have collected. The water from which the steam is generated must be previously well boiled to expel carbonic acid, and the caustic potash should be as far as possible free from carbonate. This operation differs from the determination of the Reichert-Meissl value in that the total volatile acids are distilled over. This will be the case, in treating fats (cocoanut oil) containing the less volatile of the lower

fatty acids, when 600–700 c.c. of distillate have been collected; in the case of fats practically free from the lower acids a smaller volume of distillate may be sufficient, but the acidity of the later portions should be tested. The distillate is filtered and titrated with deci-normal caustic potash, using phenolphthalein as indicator. This distillation is more tedious than the filtration process, but is better suited to the fats which contain the difficultly volatile and soluble lower fatty acids.

(b) *The filtration process.* Saponify 5 grms. of the acetylated fat in an evaporating basin with a measured volume (50 c.c.) of semi-normal alcoholic potash, and evaporate off the alcohol (see p. 70). Measure from a burette a volume of standardised sulphuric acid *exactly* equivalent to the caustic potash used in saponifying, warm on the water-bath until the fatty acids collect in an oily layer, then filter as in the determination of the Hehner value (p. 78), and wash until the filtrate amounts to 600–700 c.c., or is neutral. Filter and titrate with deci-normal potash as above.

The amount of caustic potash used in either method of determination is equivalent to the liberated acetic acid *plus* the volatile (or soluble) acids of the fat. The latter must be determined by performing a process of distillation or filtration exactly as above, employing the original fat; the result, deducted from the total caustic potash required in the case of the acetylated fat, gives the caustic potash used to neutralise the acetic acid, which quantity is then calculated to milligrams of KOH per gram of acetylated fat. In the case of fats with a very low Reichert-Meissl value, this correction may be omitted if very exact results are not required. A determination of the saponification value of

the acetylated fat may be made in the course of either of the above processes.

The alcoholic hydroxyl groups, of which the acetyl value affords a determination, may be present in fats in mono- and diglycerides, higher alcohols (cholesterol, &c.), hydroxy fatty acids (ricinoleïc acid), and oxidised fatty acids (such as occur in blown oils), the constitution of which is yet unknown (see p. 143). Thus in the present state of our knowledge it is not always possible to draw definite conclusions from a determination of the acetyl value.

The Iodine Value gives the percentage of iodine absorbed by an oil under particular conditions. In recent years much work has been done, and considerable light shed on the mechanism of the reaction upon which the determination of the iodine value depends. It will be necessary to discuss this work in some detail, but before doing so we shall give two of the processes taken from the many which have been proposed for this determination.

Hübl's method is based on the absorption of iodine by fats in alcoholic solution in the presence of mercuric chloride. The following reagents are required :

(1) Chloroform. This must be pure ; methylated chloroform cannot be used The suitability of the chloroform must be tested by performing a blank experiment, in which it should absorb no iodine.

(2) Alcohol. Absolute alcohol must be used.

(3) Potassium iodide solution. This is a solution of 1 part of pure potassium iodide in 10 parts of water. It should give no blue colouration when it is slightly acidified, and a little starch paste is added.

(4) Starch paste is made by grinding 1 grm. of starch

with a little water and pouring into 100 c.c. of boiling water. It should then be filtered, and the filtrate alone used, though this is not absolutely essential. Starch paste must be made fresh every day.

(5) Sodium thiosulphate solution. 25 grms. of pure crystallised sodium thiosulphate are dissolved in water and the solution made up to 1 litre. The exact strength of this solution is now to be determined, and since the strength of the solution diminishes gradually in keeping the following process is found the most convenient: Dissolve 5·908 grms. of fused recrystallised potassium bichromate to 1 litre. Take 50 c.c of this solution, add a little hydrochloric acid and 20 c.c. of the potassium iodide solution, and then run in the sodium thiosulphate from a burette until the yellow tint has almost entirely disappeared, then add starch paste, and continue the titration until the blue colour due to the iodine just disappears. The strength of the thiosulphate can then be calculated as follows: Suppose that 30·5 c.c. of thiosulphate were required in the titration. Now $K_2Cr_2O_7$ liberates 6 I,

$\therefore$ 50 c.c. of the bichromate solution $= \frac{5{\cdot}908}{20} \times \frac{6 \times 127}{590{\cdot}8}$

$= 0{\cdot}381$ grm. iodine, $\therefore$ 1 c.c. of thiosulphate $= \frac{0{\cdot}380}{30{\cdot}5}$

$= 0{\cdot}01249$ grm. of iodine.

(6) The iodine solution. (*a*) Dissolve 25 grms. of iodine in 500 c.c. of absolute alcohol. (*b*) Dissolve 30 grms. of mercuric chloride in 500 c.c. of absolute alcohol, let cool, and filter. Mix these two solutions, let stand twenty-four hours, and determine the strength of the mixture by adding 25 c.c. to 300–400 c.c. of water, then 25 c.c. of

the potassium iodide solution, and titrating with the standard thiosulphate, adding starch paste towards the end of the titration. The strength of the mixture gradually diminishes on keeping ; it must be determined every time the solution is used. According to Lewkowitsch, the solutions (*a*) and (*b*) should be kept separate, when little change occurs in the iodine solution ; equal volumes are to be mixed from time to time as required, and the strength of the mixture determined after standing twenty-four hours, when it may be used (see, however, p. 89). The iodine solution, thiosulphate, potassium iodide, and chloroform must all be kept in the dark.

The determination of the iodine value is conducted as follows : Weigh accurately 0·15–0·2 grm. of a drying oil, 0·3–0·5 grm. of a non-drying oil, or 0·8–1 grm. of a solid fat into a dry bottle of 500–600 c.c. capacity, with an accurately fitting stopper. Add 10 c.c. of chloroform and dissolve the fat by rotating the liquid. Then measure off into the bottle 25 c.c. of the iodine solution, taking care to deliver always the same number of drops from the pipette, after the continuous flow of the liquid has ceased and while the pipette is draining. Wet the stopper of the bottle with a drop of the potassium iodide solution and insert it securely. Shake the bottle round gently to mix the two liquids, and in case a clear solution is not obtained add 10 c.c. more chloroform. The bottle is then to be put aside in the dark for two hours ; if the liquid is not still dark brown in colour, a further 25 c.c. of the iodine solution is to be added, and the bottle left overnight. It is now recognised that the reaction is not complete in the four hours formerly thought to be sufficient. The mixture

should stand for sixteen to twenty-four hours, and such an excess of iodine must be used that one half remains unabsorbed. On the following day about 400 c.c. of water are added, and 20 c.c. of the potassium iodide solution ; the bottle is well shaken, and more potassium iodide is added if it is required to dissolve the scarlet mercuric iodide. The sodium thiosulphate solution is then run in, shaking continually, until the liquid is barely yellow ; starch paste is now added, and the titration completed. Either immediately before or after the titration 25 c.c. of the iodine solution are mixed with 10 c.c. of potassium iodide and a large volume of water, and the iodine titrated by thiosulphate as before. The difference between the two results gives the iodine absorbed by the oil, which is calculated to a percentage.

Ingle (*J.S.C.I.*, 1902, 588) modifies Hübl's method by adding 15 c.c. of solution (*b*), then 15 c.c. of solution (*a*), allowing to stand sixteen hours and then titrating; 15 c.c. of solution (*a*) are then mixed with water and potassium iodide and titrated by thiosulphate.

Wijs's method depends on the use of a solution of iodine monochloride, ICl, in glacial acetic acid. Dissolve 12·5 grms. of powdered iodine in 1 litre of glacial acetic acid by warming gently. When quite cold measure off 25 c.c.,[1] dilute largely, add potassium iodide (10 c.c.), and titrate by the standard thiosulphate solution, adding starch paste when the yellow colouration has almost disappeared. Arrange an apparatus consisting of a flask, with a long thistle funnel, or mercury safety-funnel, connected in turn to wash-bottles containing water and sulphuric acid.

[1] A soda-lime tube should be fused to the top of the pipette in order to retain the vapours, or a burette may be used.

From the last wash-bottle a delivery tube leads into the acetic acid solution of iodine. In the flask put a mixture of 20 grms. of manganese dioxide with 20 grms. of common salt, pour down the funnel a *cold* mixture of 28 c.c. of strong sulphuric acid with 24 c.c. of water. Warm gently, and regulate the reaction so that a steady current of chlorine passes through the iodine solution, which must be gently shaken at intervals. The deep colour due to the iodine becomes gradually paler, but the final change to yellow which marks the completion of the formation of iodine monochloride takes place quite suddenly, so that there is no difficulty in stopping the operation at almost the correct point. Then measure 25 c.c. of the acetic acid solution (with the same pipette and allowing the same number of drops to drain out as before), add 20 c.c. of potassium iodide solution, 400 c.c. of water, and titrate with sodium thiosulphate. The volume of thiosulphate used should be exactly double the volume required by the iodine solution at first. If less than double the volume is required, the current of chlorine must be continued until the correct point is reached. If more than double the volume is required, measure the solution and dissolve in it the weight of iodine equivalent to the excess over the double volume of thiosulphate. For example: 25 c.c. I solution required 24·8 c.c. thiosulphate, 25 c.c. ICl solution required 50 c.c. thiosulphate, 1 c.c. thiosulphate = 0·0127 grm. iodine. Then to 900 c.c. of the ICl solution would have to be added $\frac{900}{25}(50-2\times24{\cdot}8)\ 0{\cdot}0127 = 0{\cdot}18$ grm. of iodine.

The estimation of the iodine value by Wijs's method is

conducted exactly as in the Hübl process, except that 25 c.c. of the iodine chloride solution are used, and that the reaction only requires twenty minutes for completion, or thirty minutes in the case of linseed oil. It is a most important advantage of Wijs's solution that its strength diminishes very little with keeping.

The above method for the preparation of Wijs's solution may be modified by warming on the water-bath after passing the current of chlorine, in order to remove excess of chlorine by formation of chloracetic acid (Ingle, *loc. cit.*). It is stated (*J.S.C.I.*, 1902, 454) that Wijs himself, following Lewkowitsch, now prepares the solution by dissolving 5 grms. of iodine trichloride and 5·4 grms. of iodine in 500 c.c. of glacial acetic acid. Wijs now allows for non-drying oils fifteen minutes; for semi-drying oils, thirty minutes; and for drying oils, sixty minutes. The writer's experience is that a period of reaction of thirty minutes is sufficient to give constant results with linseed oil.

Many modifications of Hübl's method, which was in general use until recently, have been proposed. The modified processes generally give somewhat different results to the original Hübl method. The results obtained by Wijs's method are, as a rule, slightly higher than those of Hübl's method, as may be seen from the table below. In view of the divergent results which follow on deviations from the details of the methods, it is necessary to adhere strictly to one method, which may be either Hübl's or Wijs's, but in view of the much greater permanence of Wijs's solution and the shorter time required for the reaction, the latter is to be strongly recommended and will no doubt shortly be in general use. No practical difficulty

will arise in changing from Hübl's to Wijs's process if it is recollected that the latter gives somewhat higher figures.

IODINE VALUES OBTAINED BY HÜBL'S AND WIJS'S PROCESSES

Oil	Hübl	Wijs	Observer	
Linseed, Russian seed	180·9	182·2	Wijs	
Liver	160·6	166·5	,,	
Maize, commercial	124·9	128·6	,,	Hübl solution acted four hours, Wijs's solution made in 95 per cent. acetic acid acted not more than ten minutes.
Poppy seed, Dutch seed, old	119·3	119·7	,,	
Sunflower, Dutch seed	117·8	119·0	,,	
Sesamé, Indian seed	110·3	111·9	,,	
Cotton seed, Egyptian seed	108·8	110·1	,,	
Rape, commercial	103·0	103·3	,,	
Earthnut	87·3	87·3	,,	
Olive, commercial	83·3	84·4	,,	
Seal, pale	117·5	124·8	Hunt	
Whale	120·2	123·7	,,	
Olive	82·3	83·4	,,	
Castor, cold-drawn	82·6	85·6	,,	The period of reaction was for Hübl's solution five hours, for Wijs's solution one hour.
Dogfish liver	134·7	143·2	,,	
Earthnut	91·8	93·4	,,	
Arctic shark	89·0	98·9	,,	
Rape, pale	103·0	102·1	,,	
Cod, coast	148·5	154·6	,,	
,, Newfoundland	144·8	154·7	,,	
Linseed	174·8	177·3	,,	
Cotton seed	108·5	110	,,	
Linseed, Baltic	197·5	198	Ingle	Hübl method applied as above (see p. 89); Wijs's solution: thirty minutes.
,, Bombay	184	185	,,	
,, La Plata	180	180	,,	
Wood Oil	167	169·5	,,	

The unsaturated acids of the oleïc, linolic, and linolenic series have the property, as has been stated before (pp. 20, 25, 26), of absorbing 1, 2, or 3 molecules of bromine respectively, in order to form saturated compounds. The reaction, upon which the determination of the iodine value is based, depends on some similar absorption of halogen, and therefore affords a measure of the unsaturated acids in the glycerides of an oil. Now iodine itself is only very

slowly absorbed by unsaturated compounds: some different reaction must occur. Hübl supposed that iodine chloride was formed by the action of iodine on the mercuric chloride of his solution, and then combined with the unsaturated acids to give chloro-iodo compounds of the type $\begin{matrix}-CHCl\\|\\-CHI\end{matrix}$. Wijs, on the other hand, assigned the chief part in the reaction to hypo-iodous acid, produced by the action of water on the iodine chloride, $ICl + H_2O = HIO + HCl$, the hypo-iodous acid then giving compounds of the type $\begin{matrix}-CH.OH\\|\\-CHI\end{matrix}$. Marshall has, however, shown that the reaction takes place with iodine chloride in carbon tetrachloride solution in the entire absence of water (*J.S.C.I.*, 1900, 213); and Ingle (*loc. cit.*), who has examined the reaction and isolated the products of the action of Wijs's solution on stilbene, $C_6H_5.CH:CH.C_6H_5$, and styrolene, $C_6H_5.CH:CH_2$, has arrived at certain conclusions, of which the most important are summarised in the following paragraph:

Iodine chloride is the active substance, not hypo-iodous acid. Free acid is formed in the action of Hübl's reagent on oils. This was supposed to be due to substitution taking place alongside the action of addition; substitution, however, does not take place, and the free acid is produced by a reaction between the addition compounds and water:

$$\begin{matrix}-CHI\\|\\-CHCl\end{matrix} + 2H_2O = \begin{matrix}-CH.OH\\|\\-CH.OH\end{matrix} + HI + HCl.$$

The results of the determination of the iodine value

are always expressed in percentages of iodine, as given directly from the titration, on the hypothesis that iodine alone is absorbed. The iodine values of the saturated fatty acids and of their glycerides are, of course, practically *nil*; the iodine values found by experiment for those unsaturated acids, which it is possible to prepare in a state of comparative purity, agree with the values calculated from theory. Thus, if we had to deal with a mixture of saturated acids and one unsaturated acid, it would be possible, by means of the iodine value, to calculate the percentage of the latter in the mixture.

The *bromine value*—i.e. the percentage of bromine absorbed by an oil, and the *bromine thermal value*, or heat of bromination, *i.e.* the quantity of heat developed in the reaction between bromine and oils, have also been proposed for use in oil analysis. These values, however, vary directly in proportion to the iodine values, and thus there is no particular reason for their employment since the introduction of the speedy Wijs's method.

The *absolute iodine value* gives the percentage of iodine absorbed by the liquid fatty acids of a fat, and hence affords a measure of the proportions in which oleïc and linolic or linolenic acids are present (see p. 132).

Apart from the ordinary analytical use of the iodine value to determine the purity of oils of known iodine value, this constant affords a means of ascertaining to some extent the constitution of the liquid oils and also gives the principle of a rough method of classification. The iodine value of pure tri-oleïn is 86·2, consequently if an oil absorbs more than 86·2 per cent. of iodine it must (neglecting the possible occurrence of lower acids of the oleïc

series) contain the glyceride of an acid of the linolic or linolenic series. Now linolic and linolenic acids and their glycerides rapidly absorb oxygen from the air, which property is not possessed by oleïc acid and tri-oleïn, and it is to the presence of trilinolin and trilinolenin that the drying oils owe their property of drying by absorption of oxygen on exposure to air. The iodine value of pure trilinolin is 173·6. We might therefore classify oils with iodine values in the neighbourhood of 170 as drying oils, those with iodine values of 90 and below as non-drying oils, and designate the intermediate oils as semi-drying oils, since they contain some proportion of the glycerides which are characteristic of drying oils. The oils possessing drying properties, of which practical advantage can be taken, have iodine values of 140 or more; oils with iodine values of less than 100 are practically without the drying power. The oils with intermediate iodine values of 100–140 are able to absorb a certain quantity of oxygen, and may be designated as semi-drying, but they will not dry when exposed to air in a thin layer at the ordinary temperature.

Certain of the fish oils (see table, p. 103) have iodine values as high as linseed oil and are also able to absorb large quantities of oxygen; they do not, however, dry to a hard layer. These oils consequently form an exception in any system of classification based on iodine values alone. It is evident that their unsaturated acids are of a different constitution from linolic and linolenic acids.

The oxygen absorption of drying oils may be measured by spreading a thin layer on a plate of glass or metal and weighing at intervals until the maximum is attained. In order that the absorption may be rapidly finished, it is

necessary to dissolve 'driers' in the oil or to expose it on finely divided lead. It is doubtful whether the various methods proposed for this purpose are of practical value, and it is certain that no conclusions could be drawn from the times required to attain the maximum weights, unless all the tests were made at the same time, in order to obtain exactly the same conditions of light, temperature, atmospheric moisture, &c., upon which the rate of drying depends. The percentage of oxygen absorbed by an oil is of course in close relation to the iodine value, though the two values are not found to be in exact proportion. In examining the drying power of an oil it is probably better, and certainly simpler, in place of determining the percentage of oxygen absorbed, to compare its drying with that of a standard sample (see under *Linseed oil*).

The Elaïdin Reaction.—The action of nitrous acid on the liquid glycerides of the acids of the oleïc series has already been mentioned. The basis of the elaïdin reaction is the transformation of liquid tri-oleïn into solid tri-elaïdin. The degree of solidification, and the time within which solidification occurs, vary with the nature of the different oils. The elaïdin test cannot be used as a quantitative reaction; it serves generally to indicate the character of an unknown oil, and in certain cases may give evidence of gross adulterations. The solution of nitrous acid required in the test may be prepared and used in a variety of ways, which give divergent results. Poutet, who devised the test, shook 12 grms. of oil with 1 grm. of the solution obtained by the action of 6 grms. of mercury on 7·5 grms. of nitric acid (sp. gr. 1·35). The shaking was repeated at intervals of ten minutes during two hours, when the

mixture was left in a cool place for twelve hours. Archbutt prepares the reagent by shaking 12 grms. of mercury with 15·6 c.c. of nitric acid (sp. gr. 1·42). The solution of nitrous acid which is obtained is green, and may be used while it is that colour. The test is performed, according to Archbutt (*J.S.C.I.*, 1886, 303), by shaking 96 grms. of oil with 3·25 c.c. of the reagent in a wide-mouthed bottle, placing in water at 25° C., and shaking at intervals of ten minutes during two hours. The time is noted at which the mixture can no longer be shaken up; 10 per cent. of walnut oil retarded the solidification of olive oil from 230 to 300 minutes, and 10 per cent. of rape or cotton oil gave rise to a buttery product. However the test be performed, a parallel experiment with an oil known to be genuine should be performed, since the results vary in a manner which cannot be foreseen. It may be stated that olive oil, lard oil, and almond oil always give a hard solid mass, that the drying oils always yield a liquid product, and that the semi-drying and other oils vary between these extremes.

With a view to devising a quantitative process for the determination of oleïc acid in mixtures with the more unsaturated acids, Farnsteiner (*J.S.C.I.*, 1899, 500) endeavoured to convert oleïc acid quantitatively into elaïdic acid. This endeavour was not realised, but the results (80–85 per cent. of the oleïc acid was transformed) were such that a qualitative process for the detection of oleïc acid in the liquid acids of a fat was obtained. The acids are placed in a small flask provided with a tube carrying a tap, the air is partially exhausted by means of a pump, a measured volume of nitric oxide is admitted from

a gas burette (20–25 c.c. to 1 grm. of oleïc acid), then half the volume of oxygen. The temperature is maintained at 10–20° C., when solidification rapidly takes place. The formation of solid acid indicates the presence of oleïc acid.

The Maumené test measures the amount of heat evolved in the reaction of the oil with strong sulphuric acid. Certain conditions must be observed if comparative results are to be obtained: the same apparatus and acid of the same strength must be used, the oil and acid must be at the same temperature before the reaction, the acid must be added in the same manner. Many modifications of the process have been described; the following is essentially that of Archbutt: Fit a tall beaker of about 200 c.c. capacity into a slightly larger beaker by means of an indiarubber ring. Adjust the orifice of a 10 c.c. pipette so that it delivers 10 c.c. of strong sulphuric acid in about one minute. Weigh 50 grms. of the oil into the smaller beaker, put a thermometer in it, and place it, together with the bottle of acid, in a large vessel of water at 20° C. When the oil and acid have had time to reach the temperature of the water, fix the beaker in the larger beaker by means of the rubber ring, and pack the whole in a third beaker (or a box) with cotton-wool. Measure 10 c.c. of acid by means of the pipette and run it into the oil, stirring with the thermometer until the temperature ceases to rise. Note the highest temperature the thermometer reaches, and deduct the original temperature of the oil. The rise in temperature increases with the strength of the acid; differences due to this cause and to differences in the apparatus are to some extent removed by adopting the proposal of Thomson

and Ballantyne (*J.S.C.I.*, 1891, 233): determine the rise of temperature when 10 c.c. of acid are mixed with 50 c.c. of water, exactly in the same manner as in the experiment with oil, divide the rise in temperature given by the oil by the rise given by the water, and multiply by 100. The result is known as the 'specific temperature reaction.' The acid used must be of 95 to 99 per cent. strength.

In the case of linseed and other oils, which produce a considerable rise of temperature, charring occurs, and it is necessary to moderate the violence of the reaction by mixing 25 grms. of the oil with 25 grms. of olive oil, which gives a smaller rise, or with mineral oil, which gives only a slight rise in temperature. The rise in temperature due to the olive oil or mineral oil is then found by a separate experiment. Twice the difference between the rises given by the mixture and by the olive or mineral oil is the rise which would be caused by the oil under investigation. Sherman, Danziger, and Kohnstamm have, however, found that considerably higher results are obtained by calculating from the rise produced by a mixture of equal parts of an oil and mineral oil than are given by the oil alone (*J.S.C.I.*, 1902, 564). The differences were smaller when carbon tetrachloride was used in place of mineral oil. These chemists propose to use acid of 89 to 90 per cent. strength, with which more uniform results are obtained, and it is not necessary to dilute drying oils with mineral oil or olive oil. This modification is to be recommended.

In view of the want of an authoritative method for the performance of the Maumené reaction, some one process, such as that given above, must be adopted and retained, and it must be remembered that the results obtained by dif-

ferent operators may differ. Consequently, in using the reaction as a test of the purity of an oil, it is requisite to execute a parallel experiment with an oil of undoubted purity.

As may be seen from the values given in the table, the rise in temperature obtained in the Maumené test is roughly proportional to the iodine value, except in the case of certain fish oils, which exhibit much higher rises than are proportional to the iodine values. With these exceptions, a high Maumené reaction indicates a drying oil, and the extent of the rise in temperature is proportional to the drying power of the oil. Oils which have been exposed to the action of air and light give abnormally high Maumené figures, while, as a consequence of the exposure, the iodine value is reduced. In this case, therefore, the indications of the Maumené test are of little value.

THE MAUMENÉ TEST.

Oil	Specific Temperature Reaction		Specific Temp. Reaction. Strong acid		Actual rise, °C.	Iodine value
	Strong acid	89-90 per cent. acid	Acting on the pure oil	Calculated from result with oil and mineral oil		
Linseed	315	299	—	—	103-126	170-200
Poppy seed	—	212	—	—	74-88	128-137
Maize	176-178	163	—	—	82	118-128
Sesamé	—	143	158	200	65	105-110
Cotton seed	167	152	167	200	70-76	102-110
Rape seed	133	150	—	—	55-64	98-105
Earthnut	121	161	176	194	47-60	92-105
Almond	—	95	114	154	52-54	93-102
Castor	91	84	—	—	46	83-87
Olive	92	90	103	138	41-45	79-84
Neatsfoot	88-103	—	—	—	—	80-82
Lard oil	—	85	101	126	41	73-77
Tallow oil	73	—	—	—	42	71-76
Menhaden	306	333	—	—	123-128	175-183
Cod-liver	254	270	—	—	113-116	140-177
Seal	222	255	—	—	92	125-146
Whale	157	—	—	—	85-92	106-130
Sperm	100	102	—	—	51	79-84
Bottlenose	93	—	—	—	—	80-82

The foregoing table gives (1) the specific temperature reactions obtained with strong acid and acid of 89–90 per cent. strength, (2) a comparison of results obtained directly on the pure oil and calculated from the rise obtained with equal parts of the oil and mineral oil (p. 99), (3) the actual rise in temperature observed, and (4) the iodine values of the oils for purposes of comparison. The different results obtained by different observers are illustrated in the table.

Classification.—The solid waxes, of animal and vegetable origin, and the liquid waxes (sperm oil and bottle-nose oil) are at once marked off as separate classes by their high content of higher alcohols (unsaponifiable matter). The oils derived from marine animals are characterised by peculiar odours and analytically by a high Maumené value in conjunction with a high iodine value. The remaining oils and fats may be divided, according to their iodine values, into drying oils, semi-drying oils, and non-drying oils and solid fats (see p. 95). No strict line can be drawn between these three classes: each merges into the other. There are points of difference between animal and vegetable oils, as will be seen later, but for the present purpose it is more convenient to treat the two together. In the tables of properties (pp. 62, 102) and in Chapter V. the drying oils, semi-drying oils, non-drying oils, and solid fats are arranged, as far as possible, in the order of their iodine values. Certain subdivisions may be made; thus rapeseed and mustard oils are allied together and distinguished from other oils; castor oil, croton oil, and grapeseed oil also differ from the other oils.

THE CHEMICAL PROPERTIES OF

—	Acid Value.[1]	Saponification Value.	Ester Value.[2]	Hehner Value
DRYING OILS.				
Linseed oil	1–8	187–196	186·7	94·8
Hempseed oil	1	190–195	—	—
Wood oil	0·7–10·7	(155–)190–197	—	96·0–96·6
Candle-nut oil	—	184–192·6	—	95·5
Walnut oil	10	192–197	182·4	95·4
Poppyseed oil	4–11	189–198	180–186	95·0
Nigerseed oil	5–12	189–192	177–186	94·1
SEMI-DRYING OILS.				
Sunflowerseed oil	0·2–6	190–193	—	—
Maize oil	2·2–20	187–190	—	88·2–93·6
Pumpkinseed oil	3·5–19	188–195	—	96·2
Sesamé oil	0·2–8	188–192	—	95·6–95·8
Cottonseed oil	0·4–2·2	190·4–197	—	94·2
Rapeseed oil	1·4–4·0	(169·4–)173–178(–181)	168·5–171	94·5–96·3
Black mustard oil	5·7–7·4	173–175	166–167·3	96·0
White mustard oil	5·4	170–171	165·8	95·8
NON-DRYING OILS AND SOLID FATS.				
Earthnut oil	0·3–33	185·6–196	162–186·4	95·6
Almond oil	10	188–195	—	96·6
Croton oil	—	210·3–215·6	—	—
Grapeseed oil	16	178–179	—	—
Castor oil	0·4–0·8	173–183	—	—
Olive-kernel oil	2·0–3·5	182·3–188	—	—
Olive oil	0·6–5·0(–25)	185–195	95·1–95·3	0·6
Ben oil	—	—	—	—
Neatsfoot oil	0–0·7	194–197·4	195–196·7	95·3–95·5
Lard	—	195·3–196·6	—	95·8–96·15
Lard oil	0·4	193	—	97·4
Mahwa butter (*Bassia latifolia*)	4·8–70·8	187–194	122·4–186·1	94·7, 95·0
Mowrah seed oil (*Bassia longifolia*)	—	188·4	—	—
Palm oil	—	196–202	—	—
Bone fat	—	172–194	—	86–94
Tallow (beef)	—	193–198	—	95·6
,, (mutton)	—	195	—	95·5
Tallow oil	—	197	—	—
Cocoa (Cacao) butter	1·1–4·5	191·8–194·5 (–200)	—	—
Vegetable tallow	—	198·5–203·6	—	—
Butter-fat	0·2–0·6	221–235	223–235	86·0–88·8
Palm-kernel oil	—	246–250	—	—
Japan wax	11–32	(206–212–) 217–222 (–237·5)	207·7–208·5	90·6
Myrtle wax	3	205·7–211·5	—	—
Cocoanut oil	10·0–35·2	255–263	223–245·5	82·3
MARINE ANIMAL OILS.				
Menhaden oil	0·2	189–192	—	—
Sardine oil	2·2–21·7	189·8–193·8	—	94·5–97·1
Cod-liver oil	0·3–31·9	(171–) 175–188	—	96·5
Seal oil	0·8–43·1	(178–) 189–193	—	95·4–96·0
Whale oil	0·3–51·4	184–200 (–224)	—	—
Porpoise oil	0·8	203·4–218·8	—	—
Dolphin oil	—	197–290	—	66·3–93
LIQUID WAXES.				
Sperm oil	0·4	(117–) 125–133 (–147)	—	—
Arctic sperm (bottlenose) oil	—	123–133	—	—
SOLID WAXES.				
Spermaceti	0·1–5	(108) 122·7–136	—	—
Beeswax	17·5–21	87·5–99	70–78	—
Carnaüba wax	4–8	79–95	75–76	—
Wool wax	0·6–1·8	83–102·4	—	—

Notes (see also the notes appended to the Table of Physical Properties, p. 62).

[1] The acid values frequently vary greatly; in most cases the higher value given is not to be regarded as a superior limit.

[2] The ester value, which depends on the variable acid value, is not of much use. The figures given in the table represent the examination of only a small number of samples.

[3] The *total* volatile (soluble) acids may be calculated from the difference between the apparent and true acetyl values determined by Lewkowitsch's method, given in the next column. This

THE OILS, FATS, AND WAXES

Reichert-Meissl Value.[2]	Acetyl Value[4] (Lewkowitsch).		Iodine Value.[3]	Insoluble Fatty Acids.	
	Apparent.	True.		Saponification Value.	Iodine Value.[6]
0 (?)	12·5	11·7	(160–)170–202	182–199	170–180 (?)
—	—	—	(140–)155–166	—	141 (?)
—	—	—	150–170	(168)–189	(144–)150–160
—	—	9·8	(136–140) 163·7	—	142·7–144
0	—	—	143–148	—	—
0	—	—	(120–)128–137	—	139
0·1–0·6	—	—	126·6–134	—	—
—	—	—	118–132(–136)	201·5	124–134
4·2–9·9	—	—	(113–)118–128·6	200·0	121–126·4
—	—	—	113–130·7	—	—
0·35	—	—	104·8–110·4	195–199	109–112
—	7·7	7·6	(97·5–)102·5–110	201–208	102·4–115
0·0–0·8	—	—	(94–)98–105(–110)	174–182	98–105
0	—	—	98·8–106(–110·5)	—	110
0	—	—	92–96·7	—	96
0	—	—	(82–)92–105	198	96–103·4
0	—	—	93–102	—	—
12–14	38·6–40·8	—	101–104·7	201	111·2–111·8
0·46	—	—	94–96·2	187	98–99
—	—	146·9	82·6–87	182–193	86·6–94
—	—	—	81·8–87·8	—	—
—	—	—	78–85(–88)	196–200	86–90
—	—	—	80–84	—	—
—	—	—	67–72·9	200·6–201·2	(63·6–69·5)74·5–75·8
—	—	—	55–63(–68·8)	—	59·0–63·5
0	—	—	73–77·3	—	—
0·4–0·9(1·23)	—	—	(29·9?) 53·4–67·8	206·0	(31·6?)
—	—	—	50·1	—	—
0·5	—	—	50–53·5	205–213	53
—	—	—	46–56 (–62)	201–206	55–57
0·5	6·6	4·6	average 41–46	196–207	41
—	—	—		—	37·8
—	—	—	71·0–75·7	—	54·6–57
0·2–0·8 (–1·6)	2·8	2·0	32–37·0 (–41)	198	39·1
—	—	—	28–38	202–209·5	30·3–39·5
(11·2–) 24–33 (–41)	—	—	29–37	212·5–217	28–31
5	—	—	10–17	258–265	(3·6–) 12–13·6
(4·7–6 per cent. of soluble acids)	—	—	8·3–12·8	211–216	10·6
—	—	—	10·7	230	—
6·6–8·5	—	—	8·2–9·6	—	8·4–9·3
2·4	—	—	(148–) 175·6–183·1	—	—
—	—	—	134·1–191·7	—	—
0·2	4·75	1·15	(123–) 138·8–177	204	165–170
—	—	—	(91–94–) 125–146	—	—
Reichert value 3·7–12·5	—	—	(81–) 106–131	—	130–132
22–24	—	—	126·9	—	—
Reichert value 5·6–66	—	—	33–99	—	—
Reichert value 1·3	7·1	5·4	78·7–84	—	—
—	6·7	5·4	80·4–82·1	—	—
—	4·6	2·6	4	—	—
—	17·4	15·2	8·8–10·7	—	—
—	57·5	55·2	13·5	—	—
—	32·6	23·3	(10–) 20–21 (–28)	—	—

difference, multiplied by $\frac{5}{5·6}$, gives (very nearly) the number of c.c. of deci-normal potash required to neutralise the volatile acids contained in 1 grm. of the fat.

[4] Only acetyl values determined by Lewkowitsch's method are included (see p. 83).

[3] Iodine value. Low results were obtained in the older determinations owing to the use of an insufficient excess of the reagent. Low results in the case of the drying oils may also be due to the sample having undergone oxidation. Thus the lower figures given in the table represent values which may not now be expected.

CHAPTER IV

DETECTION AND DETERMINATION OF NON-FATTY CONSTITUENTS

Water, Dirt, &c.—The presence of more than a trace of water in an oil or melted fat gives rise to a turbidity or to the separation of drops of water. A turbidity, which disappears on heating in an open dish to above 100° C. and does not reappear on standing in the cold, may be ascribed to the presence of water. Oils are able to dissolve a small proportion of water and remain clear, which water is given off on heating to above 100° C.; the quantity is, however, too small to be of practical importance.

The quantity of water in an oil or fat is determined by weighing 5–10 grms. into a small evaporating basin, which has been tared together with a glass rod, and heating in an air-bath at 105° C. until the weight, determined after cooling in a desiccator at intervals of thirty minutes, is approximately constant. If the weight first decreases and then increases, owing to oxidation, the lowest recorded weight is to be taken. Stirring the heated oil by means of the glass rod much facilitates the evolution of the water. In order to avoid loss due to spirting, C. B. Davis (*J.S.C.I.*, 1901, 941) introduces a coil of thick filter paper into a large weighing bottle, dries at 110° C. to constant weight, adds sufficient fat or wax to saturate the

filter paper, and again dries at 110° C. to constant weight. By exposing a large surface to the action of the air, this method increases the error due to oxidation. Hence in the case of oils and fats liable to oxidation, the weighing bottle should be fitted, while in the air bath, with a cork, through which pass two tubes, one to introduce, the other to carry away, carbon dioxide or coal gas, a current of which should be sent through the bottle during the whole operation of drying.

The water may be removed from a larger quantity of oil or fat, required for further examination, by heating to about 100° C. and blowing a current of air through for a few minutes.

The presence of 'dirt' — portions of the plant or animal tissues, accidental impurities, &c.—is at once evident to the sight in the dried oil or fat. These insoluble impurities are determined by dissolving a weighed quantity of the oil or melted fat, contained in a small beaker, in ether or petroleum ether,[1] filter the solution through a dried and weighed paper, transfer the residue to the filter, and continue the washing until a drop of the filtrate evaporates entirely without residue. The filter paper is then dried, weighed, ignited, and the ash weighed. The first weighing gives the total insoluble matter, the second the inorganic insoluble matter, or ash. The ash may also be determined directly by heating 1–2 grms. gently in a platinum crucible, igniting the vapours, and finally heating

[1] For this and other purposes commercial petroleum ether must be fractionated by distilling with a column. The residue boiling above 80° C. is rejected; it is also convenient to reject the portion which distils below 40° C. and to divide the intermediate part into fractions boiling at 40–50° C., 50–65° C., and 65–80° C., and to use each fraction separately.

the residue until all the carbon is burnt. The ash of pure oils and fats is very small in quantity. In order to avoid loss by spirting and to facilitate the combustion of oils and fats in determining the ash, Delecœuillerie (*J.S.C.I.*, 1898, 958) melts the fat in a platinum dish and places in it a small folded filter paper with the point upwards. The paper is lighted, and serves the purpose of a wick.

For the purposes of further examination, oils may be freed from dirt and other suspended matter by filtering at the ordinary temperature through paper of close texture, or through hardened paper by means of the filter pump. Oils filter much more rapidly in the oven at 100° C.; melted fats are filtered either in the oven or in a hot-water jacket by means of the filter pump.

Metals and Inorganic Acids.—Oils may contain in a soluble form metals—lead, copper, iron—derived from the vessels in which they have been refined or stored; drying oils (boiled oil) may contain compounds of lead, manganese, &c., added as 'driers.' The metals may be detected and estimated by dissolving a weighed quantity of the oil in ether or petroleum ether, shaking well in a separating funnel with a little nitric acid (one part of strong acid to nine parts of water), drawing off the acid solution, washing the ethereal solution with water until the washings are neutral, evaporating the aqueous liquids to dryness, igniting to destroy organic matter, dissolving the residue in acid, and then treating by the ordinary analytical methods.

As already stated (p. 73), oils and fats may contain inorganic acids (sulphuric) introduced in the refining process and not removed by thorough washing. This acid is

extracted by dissolving the oil in ether or petroleum ether, and shaking with small quantities of water until the wash waters are neutral. The washings are then united and the ether driven off by boiling, a little hydrochloric acid is added, the liquid is filtered through a wet paper to remove traces of oil, and precipitated by barium chloride. This is the method as usually given, but the writer has reason to believe that washing with water fails to remove the last traces of sulphuric acid. It is preferable to saponify the fat with pure caustic potash (see p. 70), separate the fatty acids by pure hydrochloric acid, and determine the sulphuric acid in the aqueous liquid.

Colouring Matters are present in many oils and fats. The quantity is always very small, but it is sufficient to give rise to the colour-reactions characteristic of certain oils, and may cause the oils to have absorption spectra. Foreign colouring matters, natural and artificial, are added to certain fats. In cases where this addition is suspected the examination of the colouring matters may be necessary (see p. 178), in other cases such examination would have little object or result.

Albumin.—Albuminous substances are stated to be present in animal fats, and nitrogenous compounds appear to exist in traces in certain seed oils. The occurrence of these compounds is, however, at present without analytical significance.

Unsaponifiable Matter.—This term is applied to any substance which is extracted by ether or similar solvents from the solution obtained by saponifying an oil or fat with excess of caustic soda or potash, and dissolving in water. 'Unsaponifiable matter,' therefore, does not

include glycerin, but does include the higher monatomic alcohols. If the liquid and solid waxes, which contain large quantities of the higher alcohols, be excluded, it is probably correct to say that all oils and fats contain naturally some small quantity (generally less than 1 per cent.) of unsaponifiable matter. The isolation and investigation of this natural unsaponifiable matter are often of great importance.

In addition to the normal unsaponifiable matter just mentioned, oils and fats may contain abnormal unsaponifiable matter—petroleum or shale oils, coal-tar oils, rosin oil, &c.—the presence and exact determination of which are always of great importance, since these oils are very much cheaper than the animal and vegetable oils. The presence of these unsaponifiable oils may readily be detected in the following manner: Boil a fragment of stick potash about the size of a pea with about 5 c.c. of absolute alcohol in a test-tube, and, without waiting for the potash to dissolve, add three drops of the oil or melted fat, boil for not less than two minutes, and add 5 c.c. of hot distilled water. If the oil is pure a perfectly clear solution should be obtained ; if the oil contains 1 per cent. of unsaponifiable oil a distinct cloudiness is produced—2 per cent. of unsaponifiable oil gives a turbidity. It should be remarked that this simple test is reliable if carefully conducted, but not unless.

The *determination* [1] of the unsaponifiable matter may be conducted by extracting either the solution of the soap

[1] A considerable proportion of mineral oil in a saponifiable oil may be estimated somewhat roughly by means of a determination of the saponification value of the oil (see p. 77).

obtained by saponifying with caustic soda or potash, or by extracting the dry soap. (*a*) The extraction of the soap solution. Numerous modifications of this process have been recommended, for which the larger text-books should be consulted; the following will be found of general application. Dissolve 5 grms. of caustic potash in 6 c.c. of water in a 5-inch dish on the water-bath, add 40 c.c. of alcohol, and then weigh into the dish about 10 grms. of the oil or fat. Keep the alcohol boiling gently and stir well until all the saponifiable oil has dissolved. If a large proportion of unsaponifiable oil is present, some will remain undissolved. Evaporate down to drive off the alcohol, stirring occasionally. In order to ensure complete saponification it is well to dissolve the soap again in alcohol to a clear solution and again evaporate. After heating until the smell of alcohol is no longer evident, dissolve the soap in a little hot water, and transfer to a cylindrical separating funnel of 200 c.c. capacity. Wash the dish out with boiling water, so that the total volume of soap solution in the funnel is about 75 c.c., cool well, add about 40 c.c. of ether or petroleum ether, shake, and leave at rest to separate. If in a short time the liquid does not begin to separate a clear layer of ether at the top, add 1–2 c.c. of alcohol, shake the liquid gently round, and again leave at rest. The formation of emulsions which separate with difficulty is the great bugbear of this process. If separation should not commence and show a slow progress after the addition of alcohol, a further addition may be made, but the total quantity added must not be large, otherwise the ethereal solution will dissolve a large quantity of soap. When the mixture in the funnel has

separated into a clear ether solution above and a clear aqueous solution below (the presence of a few flocks is of no moment), the latter is drawn off, and the ether solution is washed two or three times with small quantities of about 5–10 c.c. of water, which are added to the main aqueous liquid. The ether solution is then transferred to a tared flask, and the soap solution is again twice extracted with smaller quantities of ether in the same manner; the ethereal solutions are washed as before, and then transferred to the flask. The ether is now evaporated on the water-bath, the last portions of the vapour are driven out of the flask by blowing in a current of air filtered through cotton-wool, and when the ether appears to be all expelled the flask is allowed to cool in the balance-case and weighed. It must be again heated on the water-bath, the current of air blown in for a minute or two and the flask reweighed; this process should be repeated until successive weighings do not show a decrease of more than 0·5 per cent. of the total quantity of extracted matter. In case the oil contained one of the more volatile unsaponifiable oils, such as burning kerosene—a contingency which probably rarely occurs—this method would give too low results, since unsaponifiable matter would be lost both during the saponification on the water-bath and while the ether was being driven off. In such a case the most satisfactory course would be to distil off the more volatile portions of the unsaponifiable oil in a current of steam, separate them from the distillate, and then to determine, as above, the less volatile unsaponifiable matter remaining in the oil. The presence of volatile unsaponifiable matter of this nature would be indicated by a low flash point.

If the unsaponifiable oil left after evaporation of the ether is gelatinous, it probably still contains soap, which has been simultaneously extracted by the ether. If it is found, after weighing, to have an alkaline reaction when mixed with hot water and tested with phenolphthaleïn, it should be heated with water and a few drops of caustic potash, and the liquid again extracted by ether as before. Petroleum ether dissolves less soap than ordinary ether, and does not give rise to such obstinate emulsions; petroleum ether, however, occasionally does not extract unsaponifiable oils from soap solutions. The cause of this behaviour, which was noted by Lewkowitsch and which the writer can confirm, does not appear to be known.

(*b*) Extraction of the dry soap. This method is useful for the extraction of small quantities of unsaponifiable matter, especially of the higher alcohols and such constituents as do not dissolve readily in petroleum ether. In view of its certainty and the absence of difficulty owing to the formation of emulsions, which occasionally renders the method (*a*) most tedious, the writer, in fact, prefers to employ this method in all cases. The time occupied in manipulation is no greater than in extracting the soap solution. The process may be conducted as follows: Saponify 10 grms. of the oil with caustic potash in a 6–7 inch dish as directed above (p. 109); after evaporating off the alcohol, add 8 grms. of sodium bicarbonate and 20 c.c. of methyl alcohol,[1] stir the bicarbonate well in and evaporate down, add 10 c.c. of methyl alcohol and 25 grms.

[1] Ordinary methyl alcohol should be purified by boiling with solid caustic potash on the water-bath and then distilling.

of precipitated chalk[1] all at once, mix well together by means of a porcelain pestle, dry first on the water-bath, stirring well and removing the soap caked on the sides of the dish, then transfer to an oven and dry at 110° C. for a few minutes. The methyl alcohol is used to facilitate the removal of water, which it effects much more readily than ethyl alcohol. Next transfer the mixture, which must not be exposed to the air for any length of time after leaving the oven, to a large Soxhlet extractor, the bottom of which is provided with a plug of glass-wool or cotton-wool (fat-extracted). Extract with petroleum ether for some hours, evaporate the ether, and weigh the residue, adopting the precautions given above (p. 110). The mixture of calcium carbonate and soap, from which the unsaponifiable matter has been extracted, may be dissolved in hydrochloric acid and the fatty acids separated for further examination (p. 70).

The unsaponifiable matter extracted from unadulterated oils and fats (the solid and liquid waxes are exceptions) is solid, and rarely amounts to more than 1 per cent. Liquid unsaponifiable matter is generally due to the admixture of mineral (or similar) oils. Bone-fat may contain 2 per cent. of unsaponifiable matter, linseed oil and maize oil as much as 1·5 per cent., and lithographic varnish 2·5 per cent.

Examination of Unsaponifiable Matter.—(*a*) The unsaponifiable matter is liquid and large in quantity. It may contain (1) the various oils produced in the distillation

[1] Sand, which has been washed with hydrochloric acid and ignited, is generally used for this purpose; some 75 grms. are required. The writer has found that precipitated chalk, which is cheap and requires no preparation, very well answers the purpose of dividing up the soap. No calcium is found in the extract.

of petroleum and shale oil, which range in specific gravity from 0·800 to 0·920. These oils generally show a green or blue fluorescence or 'bloom,' though 'debloomed' oils are largely used for mixing with animal and vegetable oils. (2) Heavy coal-tar oils, which are heavier than water and contain the more complex aromatic hydrocarbons; (3) rosin oil. The specific gravities of the oils produced in the distillation of rosin range from 0·966 to 1·00. The crude oils contain considerable quantities of rosin acids (20 per cent. is quite common, and 50 per cent. is sometimes reached); consequently, if rosin oil be found in the unsaponifiable matter, the rosin acids in the saponifiable oil should be determined by Twitchell's process (p. 125). The simultaneous presence of rosin oil in the unsaponifiable matter and rosin acids in the saponifiable might be due to the use of refined (neutral) rosin oil together with rosin itself.

In the examination of unsaponifiable oils valuable indications are afforded by the colour, fluorescence, and odour. The specific gravity is then to be taken; according to the available quantity, one of the methods previously described (pp. 39–45) may be used. A specific gravity of 0·914 or below indicates a petroleum or shale oil; the oils generally used for *adulteration* range from 0·885 to 0·905. A specific gravity greater than unity indicates the presence of coal-tar oils, which are rarely used except in lubricating greases. Specific gravities between 0·900 and 1·000 might be due to mixtures of coal-tar oils or rosin oils with petroleum oils. Rosin oil can always be detected by its peculiar odour, which becomes more evident on warming. The neutral rosin oil isolated as un-

saponifiable matter may be expected to vary in specific gravity from 0·965 to 0·980. Rosin oil also gives the Liebermann-Storch reaction (p. 124).

(*b*) The unsaponifiable matter is solid at the ordinary temperature, and does not dissolve when warmed with five times its volume of alcohol. It consists of paraffin or ceresin (refined ozokerite). The melting-point indicates the quality of the paraffin or ceresin. To confirm the result treat as in (*c*).

(*c*) The unsaponifiable matter is solid at the ordinary temperature and dissolves in five times its volume of warm alcohol. It may contain cetyl, octadekyl, carnaübyl, ceryl, and myricyl alcohols, unsaturated alcohols from the liquid waxes, also cholesterol, phytosterol, isocholesterol, and paracholesterol. A mixture of paraffin or ceresin with these alcohols would partially dissolve in alcohol.

Transfer the unsaponifiable matter to a test-tube, add an equal quantity of acetic anhydride, and boil under an inverted condenser for an hour, allow to cool, and then add about five volumes of water. If the unsaponifiable matter does not dissolve in the hot acetic anhydride, but remains as an oily layer on the surface and solidifies on cooling, it is paraffin or ceresin. If, on the other hand, the unsaponifiable matter dissolves entirely, and on cooling separates a quantity of crystals, it may contain the alcohols mentioned above, the acetates of which crystallise out on cooling or on the addition of water.

The total unsaponifiable matter extracted from a vegetable oil containing a small proportion (1–2 per cent.) of mineral oil, may dissolve entirely in acetic anhydride. On cooling, or on adding water, the mineral oil separates as a

layer at the surface, while the phytosterol acetate crystallises from the liquid.

Although the number of alcohols which may occur in the unsaponifiable matter is somewhat considerable, very rarely are more than one or two found together: the separation and detection of cholesterol and phytosterol are frequently necessary, and it may occasionally be desirable to determine the quantities of ceryl and myricyl alcohols in a mixture of the two. The latter determination would be made by converting the alcohols into the acetates by boiling with acetic anhydride, taking the saponification value of the mixture of acetates, and thence calculating the proportion of each (also see p. 131). The isolation of phytosterol and cholesterol, and the detection of each in the presence of the other, are of great importance, and will be treated in some detail.

Cholesterol is characteristic of animal fats, phytosterol of vegetable fats; thus in cases of suspected adulteration of an animal fat by a vegetable fat, or *vice versa*, in which the ordinary analytical methods are not decisive, owing to the similarity of the oils or the small proportion of the adulterant, an examination of the unsaponifiable matter gives a definite result. Examples of such cases are: the addition of rape oil, &c., to neatsfoot oil, cottonseed oil to lard, lard oil to olive oil, tallow to cacao butter.

The oils contain such small quantities of phytosterol or cholesterol that a considerable weight of the oil has to be treated in order to obtain a sufficient amount of the alcohol. If the aqueous solution of the soap is extracted with ether, the large volume of the soap solution necessitates an inconveniently large quantity of the solvent, and

the manipulation is not easy. Von Raumer recommends the saponification of 50 grms. of the oil by means of alcoholic potash, the evaporation of the solution, and the extraction of the powdered dry soap in a Soxhlet apparatus. The drying of large quantities of soap is very difficult, and a second saponification of the extracted matter may be necessary (*J.S.C.I.*, 1898, 774). Bömer, who relies mainly on the examination of the crystalline form of the cholesterol or phytosterol, considers that 10 grms. of oil are sufficient; the manipulation is then easy (*J.S.C.I.*, 1898, 90). Kreis and Rudin (*J.S.C.I.*, 1899, 1158) saponify 50 grms. of oil with strong caustic soda and alcohol, evaporate off the alcohol, dissolve in water, and precipitate the calcium soaps by the addition of calcium chloride. The precipitate is then dried, mixed with 100 c.c. of a mixture of equal parts of alcohol and ether, transferred to a filter, and washed with 50 c.c. of alcohol and ether. The filtrate is evaporated, the residue mixed with 3 c.c. of caustic soda (40 per cent.), evaporated to dryness, mixed with 20 grms. of sand, thoroughly dried, and extracted with ether in a Soxhlet. The yields of unsaponifiable matter obtained by this process are lower than those given by other methods.

Ritter saponifies by means of sodium ethylate (*J.S.C.I.*, 1902, 643); 50 grms. of the oil are heated on the water-bath with 100 c.c. of alcohol, and a hot solution of 8 grms. of sodium in 160 c.c. of 99 per cent. alcohol is then added. The alcohol is evaporated off, 75 grms. of common salt added, together with sufficient water to dissolve the whole, the solution is evaporated, dried in the oven at 80° C., powdered, and left in the exsiccator. The dry powder

is then extracted in a Soxhlet apparatus, the operation lasting about nine hours. The ether is poured off from the glycerin, which collects at the bottom of the flask; the latter is washed out with ether, and the solvent evaporated. The residue is dissolved in a little alcohol and precipitated by adding water in small quantities at a time. The precipitate is washed with water on a filter, dried at 60° C., dissolved in ether, the solution transferred to a weighed flask, evaporated, and the residue dried at 100–120° C. Ritter has made parallel experiments on the same sample of fat by various methods; he states that the alcohols separated by the process just described are the most pure, but still not quite pure; also the yields are higher.

Marcusson has described several methods for obtaining cholesterol and phytosterol from fats which contain large quantities of mineral oil (*J.S.C.I.*, 1901, 484; 1902, 725). In the case of a lamp oil containing 60 per cent. of a thin mineral oil, the total unsaponifiable matter was extracted from the aqueous solution of the soap obtained from 200 grms. of the oil, and was then distilled in superheated steam, the temperature of the oil not being allowed to rise above 200° C. The residual oil (3 c.c.) was repeatedly extracted by boiling 70 per cent. alcohol; the crystals which separated on cooling were then recrystallised. In the case of a mixed oil containing a heavy cylinder oil, the oil was dissolved in ether and precipitated by adding alcohol, the upper layer was removed, evaporated, again separated from the deposit, and finally evaporated. The residue then contained very little mineral oil, it was saponified, extracted by ether in the

ordinary manner, and the ethereal residue recrystallised from 70 per cent. alcohol. Marcusson further points out that the mineral oil can be completely extracted from a soap solution by means of petroleum ether, whilst a portion of the cholesterol or phytosterol is left, and can then be extracted by ordinary ether.

When a vegetable oil is suspected of containing animal oil—i.e. when phytosterol is to be examined for traces of cholesterol—the crude unsaponifiable matter is repeatedly recrystallised from the smallest possible quantity of hot 70 per cent. alcohol, and each crop of crystals is examined under the microscope. The last crop which it is practicable to obtain should show the correct form and have the correct melting-point of phytosterol (see p. 35 and table below).

The presence of a trace of phytosterol in cholesterol materially alters the crystalline form, even when the quantity is not sufficient materially to lower the melting-point. Thus examination of the crystalline form is more reliable than determination of the melting-point. (Bömer, *J.S.C.I.*, 1898, 954.)

The following method, given by Bömer (*J.S.C.I.*, 1902, 192), is particularly applicable to the detection of phytosterol in cholesterol, and is more reliable than methods based on examination of the alcohols themselves. The crude phytosterol or cholesterol obtained from 100 grms. of the oil is resaponified (if necessary), and the ethereal extract dissolved in the least possible volume of hot absolute alcohol. The crystals which first separate are examined under the microscope; phytosterol will only be found if present in some quantity.

The alcohol is then evaporated on the water-bath, 2–3 c.c. of acetic anhydride are added, the dish is covered, heated over a naked flame, and the excess of acetic anhydride is finally driven off on the water-bath. The residue is dissolved in hot absolute alcohol by heating over the open flame, a little alcohol is added to prevent immediate crystallisation, and the clear liquid is allowed to evaporate at the ordinary temperature. When nearly two-thirds of the liquid have evaporated, the crystals are transferred to a small filter by means of a spatula and 2–3 c.c. of 95 per cent. alcohol. The residue on the filter is dissolved in 5–10 c.c. of hot absolute alcohol, and the recrystallisation continued as long as is practicable. The melting-point of the acetate is determined after the third and following recrystallisations. If the acetate does not melt completely at 116° C., the presence of phytosterol (vegetable oil) is probable ; and if the melting-point is 117° C. or higher, Bömer regards its presence as certain. Those vegetable oils which contain comparatively large amounts of phytosterol—*e.g.* cottonseed, earth-nut, sesamé, rapeseed, hempseed, poppy, and linseed oils—can be detected in this manner when present to the extent of 1–2 per cent. in animal fats. Their detection in animal fats containing abnormally large quantities of unsaponifiable matter would be less certain. Olive, palm, palm-kernel, and cocoanut oils, which contain less phytosterol, can only be detected if present to the extent of 3–5 per cent.

The following are the (corrected) melting-points of phytosterol and cholesterol and their acetic and benzoïc esters ; the temperature at which complete fusion occurs is taken as the melting-point.

	Cholesterol	Phytosterol
	°C.	°C.
The alcohols	148·4–150·8	138·0–143·8
Acetates	114·3–114·8	125·6–137·0
Benzoates	148·4	145·3–148·4

Unsaponifiable Matter from Resins.—Ordinary rosin (colophony) may contain as much as 15 per cent. of unsaponifiable matter. Rosin dissolves in caustic potash to a clear solution, from which ether extracts the unsaponifiable matter. When rosin is found in an oil or fat, unsaponifiable matter will consequently be obtained. The different resins (copals, &c.) contain different proportions of unsaponifiable matter, most of which separates in a solid lump when the resins themselves, or mixtures containing them, are saponified.

Saponifiable Matter in Mineral Oils.—The detection and determination of small quantities of saponifiable oils in mineral oils are often of importance—lubricating oils for steam-engine cylinders frequently contain some 5 per cent of a saponifiable oil. A simple qualitative test for a saponifiable oil consists in boiling a few c.c. of the oil in a test-tube with an equal volume of strong (40 per cent.) caustic soda solution. Pure mineral oils at once separate to a clear layer, whilst in the presence of small quantities (2–5 per cent.) of saponifiable oils more or less complete and persistent emulsions are formed.

The best method for detecting the presence of saponifiable oil in mineral oils is afforded by a determination of the saponification value; if this value is found to be as great as 1, saponifiable matter is certainly present (see example on p. 77). It is by no means unusual to receive

samples of practically pure mineral oil with a distinct odour of animal or vegetable oil, possibly due to the oil having been put in a vessel which had previously contained the saponifiable oil and had not been cleaned.

Resins.—The resins are vegetable products, which exude from the bark of trees or collect in cavities beneath it. They vary greatly in composition, but they generally contain some quantity of volatile oil and more or less large quantities of acids. When the resin which exudes from pines is heated, or distilled with steam, (spirits of) turpentine distil over and the residue constitutes ordinary rosin or colophony. In the ordinary course of oil analysis it is probable that no other resin than colophony will be encountered. A considerable number of resins are, however, used in the manufacture of varnishes; unfortunately, in the present state of knowledge it is not possible to identify or estimate the resins in most varnishes. The analytical constants of certain varnish resins, determined by Lewkowitsch by means of the ordinary methods used in oil analysis (*J.S.C.I.*, 1901, 372), are given in the appended table (p. 122). Some idea of the composition of the resins is afforded by these figures.

Rosin.—American rosin is obtained in the manner mentioned above from *Pinus australis*; in this country it is used in far the greatest quantity. In France, rosin and turpentine are obtained from *P. maritima* and in Russia from the Scotch fir, *P. sylvestris*. The commercial qualities of rosin are designated by letters, from A to N, the first letters of the alphabet representing the common very dark qualities. W and WW are specially refined pale qualities. Rosin is a brittle friable solid, ranging in colour

—	The original Resins						Resins after heating at 300° C.				
	Acid value	Saponification value	Unsaponifiable matter, per cent.	Iodine value		Soluble in alcohol, per cent.	Acid value	Saponification value	Unsaponifiable matter, per cent.	Iodine value	
				Wijs's method	Calculated from Br value					Wijs's method	Calculated from Br value
Copal, commercial .	109·8	143·1	7·96	135·5	183·6	93·06	26·49	85·4	14·73	124·3	181·3
,, ,, .	42·43	66·82	14·99	191·2	114·6	54·82	24·94	61·04	46·40	143·7	233·5
,, Sierra Leone	72·83	119·03	18·81	105·7	96·05	—	12·89	114·9	17·22	125·5	173·6
,, Manila .	127·6	175·17	15·98	137·9	188·2	—	68·21	136·3	22·99	133·3	186·4
,, Brazil . .	108·99	171·4	7·66	127·7	72·66	—	46·25	113·8	38·74	136·7	225·9
,, Sierra Leone	65·7	110·5	16·27	94·55	117·0	—	15·32	123·8	22·31	95·2	135·4
Kauri . . .	37·39	53·84	20·07	90·99	—	—	17·14	61·07	10·39	67·7	74·7
Mastic . . .	52·73	81·79	51·13	175·7	185·0	84·41	23·23	50·24	49·28	165·0	217·8
Shellac, dark . .	61·13	203·0	3·56	36·57	13·25	—	—	—	—	—	—
Sandarac, Mogador .	134·39	143·42	13·2	112·2	99·98	—	64·84	136·14	14·28	126·4	69·1
,, Austral .	131·15	134·32	17·44	125·4	63·83	—	106·6	137·26	9·93	—	—
Animi, No. 1 . .	18·69	73·15	6·3	105·3	182·7	—	9·25	58·73	—	106·1	207·4
,, No. 2 . .	30·22	93·5	6·85	96·21	95·85	—	—	—	—	—	—
Damar . . .	35·22	32·73	75·96	127·5	169·4	—	10·85	60·44	86·57	127·0	196·8
Amber . . .	16·7	121·27	18·86	58·98	82·82	—	—	—	—	—	—
Succinit (species of amber) . .	11·24	113·68	7·06	55·05	78·4	—	10·60	99·19	16·25	74·3	147·8
Colophony . .	—	—	—	—	—	—	146·46	153·01	15·99	133·7	303·1

from the almost colourless W W (water-white) to a very dark brown ; it has a pleasant and characteristic odour. It is heavier than water, the specific gravity ranging from 1·05 to 1·1 at 15° C. On heating it softens at about 75° C., and is completely melted to a viscid liquid at 130° C. When subjected to dry distillation, rosin yields water, acetic acid, rosin spirit, and rosin oil. Rosin dissolves in ether, benzene, chloroform, oils, &c., in strong alcohol, and in alcohol of 70 per cent. strength, in which latter respect it differs from the fats. In petroleum ether it gives a turbid solution.

Rosin is mainly composed of abietic acid, $C_{19}H_{28}O_2$, with a small quantity of sylvic acid, $C_{20}H_{30}O_2$. The fact that rosin has a higher saponification value than acid value indicates that some portion of the acids is present as anhydride. Rosin also contains unsaponifiable matter soluble in the solution obtained on saponification. Rosin dissolves on boiling with caustic soda or sodium carbonate ; the strong solutions so obtained are able to dissolve considerable quantities of rosin, which separates on dilution. The salts of the rosin acids are known as *resinates* ; the resinates of the earth metals and heavy metals are insoluble in water, but soluble to a greater or less extent in oils, benzene, turpentine, &c.

Sodium and potassium resinates are very similar to the corresponding salts of the fatty acids, and are therefore known as rosin soaps ; they cannot, however, be completely separated from their solutions by the addition of common salt. Sodium resinate is soluble in alcohol, and in ether containing alcohol.

The acid value of rosin ranges from 152 to 167, and

may generally be taken for the ordinary pale grades (F, G) at about 160. The saponification value ranges from 170 to 190. Rosin does not give a definite iodine value: the amount of iodine absorbed generally increases with the period of reaction and with the excess of reagent used. When rosin is dissolved in alkalis, the solution acidified, extracted by ether, and the extract evaporated, the re-obtained rosin has not quite the same analytical constants as the original substance; the acid value is somewhat increased, and the saponification value decreased, but the values fall within the above limits.

Detection of Rosin.—Rosin is not generally added to any of the liquid oils, with the exception of boiled linseed oil. It is used in enormous quantities in the manufacture of soap, and may also be found in the solid waxes, in lubricating greases, linoleum, &c.

Rosin may be detected by means of the Liebermann-Storch colour reaction, which is best applied to the fatty acids isolated from the fat: 0·2 grm. of the fatty acids is dissolved in 5 c.c. of acetic anhydride, and 1 to 2 drops of cold dilute sulphuric acid are added. A deep reddish-violet colouration is produced in the presence of 1 to 2 per cent. of rosin. The reaction is also given by rosin oil. The dilute sulphuric acid is obtained by mixing equal volumes of strong acid and water. It may be mentioned here that a small quantity of rosin may legitimately be present in boiled oil; it is added to the oil in the form of resinates of lead and manganese—the 'driers.' This quantity of rosin is sufficient to give the above reaction.

The solubility of rosin in 70 per cent. alcohol may be employed to extract rosin from fats for purposes of

qualitative examination. The oil is well stirred on the water-bath with the hot alcohol, the mixture allowed to cool, the alcoholic layer withdrawn and evaporated. The residue, after heating in the oven at 105° C., exhibits the appearance and properties of rosin if that substance is present in any quantity. It must be remembered that the fatty acids, especially oleïc, are also soluble in alcohol; extracted matter consisting essentially of fatty acids is, however, more fluid, and does not give the characteristic odour of rosin on heating.

The high acid value of rosin also affords a means for its detection and approximate determination in oils which have normally a low acid value, *e.g.* in boiled oil. If a high acid value is found, rosin is probably present (see Chapter VII., *boiled oil*).

Determination of Rosin.—Of the various methods which have been proposed for the estimation of rosin in fats, Twitchell's (*J.S.C.I.*, 1891, 804) is undoubtedly the best. It is based on the fact that the fatty acids, when dissolved in absolute alcohol and subjected to the action of dry hydrochloric acid gas, are converted into esters, whilst the rosin acids remain unaltered, and can subsequently be withdrawn by the action of alkalis. The apparatus and process are as follows: A round-bottom flask, provided with a safety-funnel, in which is a mercury trap, is connected to two small wash-bottles containing strong sulphuric acid, from the last of which a delivery tube leads to the bottom of a 250 c.c. flask cooled in water. The first flask contains 96 grms. of common salt, upon which is poured a cooled mixture of 88 c.c. of strong sulphuric acid with 64 c.c. of water. These materials evolve hydro-

chloric acid gas on warming, and the evolution ceases when the source of heat is removed. The quantities given are sufficient for two or three determinations; if the method is in regular use, a larger evolution apparatus might be arranged. From 2 to 3 grms. of the mixture of fatty and rosin acids, isolated and dried in the usual manner, are weighed into the 250 c.c. flask, ten times the volume (i.e. 20–30 c.c.) of absolute alcohol is added, and the acids are dissolved, if necessary by warming. The flask containing the *cold* solution of the acids in alcohol is then placed in a vessel of cold water, to which ice should be added in warm weather. The temperature must not exceed 20° C. The flask containing the salt and sulphuric acid is then heated by a small flame, so that a fairly rapid current of gas passes through the wash-bottles and absorption flask. When the air has been expelled, the hydrochloric acid gas is seen to be absorbed by the alcoholic solution. The flask should be occasionally shaken in order to keep the temperature low. The absorption of gas generally ceases in thirty to forty minutes, but the current should be continued for about one hour, after which the flask is allowed to stand for another hour. The esters of the fatty acids generally, but not always, separate in a layer at the surface of the alcoholic solution towards the end of the operation. After standing for one hour, 100–150 c.c. of water are added, a few scraps of platinum foil are dropped in to prevent bumping, the contents of the flask are boiled slowly until they become clear, and then allowed to cool.

The mixture in the flask is next transferred to a 200 c.c. separating funnel, the flask is washed out with ether (in all

about 50 c.c.), and the washings are added to the liquid in the funnel, which is shaken and the aqueous liquid drawn off. The ethereal solution is washed with water and quickly shaken with a solution of 0·5 grm. of caustic potash in 50 c.c. of water and 5 c.c. of alcohol. The caustic potash abstracts the rosin acids. The aqueous solution is drawn off, and the ether solution again shaken with caustic potash. The alkaline liquids are united, made acid with hydrochloric acid, the separated rosin acids extracted by ether, the solution washed with water, the ether partially evaporated, the solution transferred to a tared dish, the evaporation completed on the water-bath, and the residue finally dried in the oven at 105° C. Petroleum ether may be used in place of ordinary ether in this process, except in the presence of oxidised acids.

Instead of weighing the rosin acids they may be volumetrically estimated. In this case the ethereal solution (ordinary ether) of fatty acid esters and rosin acids, obtained as above, is washed with water in a separating funnel until the washings are no longer acid to litmus paper. To 100 c.c. of alcohol contained in a beaker or flask phenolphthaleïn is added, and then semi-normal alcoholic potash until the pink colouration appears; the ether solution is then run in, and the alcoholic potash added until the colouration reappears. One cubic centimetre of semi-normal potash equals 0·173 grm. of rosin acids; this is only an average figure, hence the results are uncertain. The results obtained by the volumetric process are generally slightly too high; those of the gravimetric process slightly too low. Though Twitchell's process can be applied to boiled oil, which contains oxidised acids, the

gravimetric method cannot be used to examine the acids of dried boiled oil, which give very high and variable results.

The degree of accuracy of Twitchell's processes may be gathered from the following table, the figures in which are due to Lewkowitsch (*J.S.C.I.*, 1893, 507).

Rosin acids actually present, per cent.	Rosin acids found.	
	Volumetric process, per cent.	Gravimetric process, per cent.
9·79	9·34 – 9·98	9·38– 9·97
19·69	22·93 –24·55	19·33–20·55
21·45	23·63 –24·96	18·27–19·54
24·66	24·23 –25·12	16·65–21·76
30·31	28·18 –30·12	23·66–26·1
39·81	40·05 –44·82	32·51–38·86
45·05	44·24 –49·61	35·32–40·06
Pure stearic acid . . .	1·85 – 2·4	1·02– 1·07
,, oleïc ,, . . .	5·037– 5·25	3·65– 3·67
Acids of tallow and cocoanut oil	4·9 – 6·35	2·56– 2·78

CHAPTER V

METHODS FOR ESTIMATING THE CONSTITUENTS OF OILS AND FATS

IN the first place we have to consider how the methods described in Chapter III., for determining the analytical constants, may be employed in estimating the proportions in which the various constituents of a fat are present. The methods for estimating the unsaponifiable matter have already been given.

The Free Acids.—In determining the acid value, the fat is shaken in a flask on the water-bath with neutralised absolute alcohol and ether, phenolphthalein is added, and the liquid titrated with semi-normal alcoholic potash. The neutral liquid is diluted, the ethereal layer washed with water, and the washings added to the aqueous liquid, from which the alcohol is then driven off on the water-bath. The residue is then treated with water, and the solution twice extracted with ether; it then contains only the soaps derived from the free acids. On acidifying, these acids are liberated; they are extracted with a little ether, the solution evaporated in a tared dish, and the residue weighed. The ether used to wash the aqueous soap solution is united with the main ethereal solution, which is then washed with water and the ether evaporated, when the neutral fat is obtained.

If a be the acid value expressed in milligrams of KOH, and w the percentage of the free acids extracted from the fat, then the mean molecular weight of these acids is $\frac{560\,w}{a}$. The mean molecular weight of the acids indicates their approximate composition.

The Neutral Fat.—The ester value of an oil or fat is the saponification value of the neutral fat it contains. Consequently if e be the ester value and n the percentage of neutral fat, the mean molecular weight of the esters, M, in the neutral fat is given by the expression

$$M = \frac{3 \times 56 \times 10n}{e}.$$

Now the formula of the triglyceride of the acid $C_nH_mO_2$ is $C_3H_5(C_nH_{m-1}O_2)_3$; hence if A be the molecular weight of the acid,

$$M = 3(A - 1) + 41 = 3A + 38,$$

$$\text{therefore} \quad \frac{1680\,n}{e} = 3A + 38$$

$$\text{or} \qquad A = \frac{560\,n}{e} - \frac{38}{3}.$$

Thus the mean molecular weight of the acids in the neutral fat may be calculated.

Since the ester value is the saponification value of the neutral fat, it is, in the absence of mono- and diglycerides, a measure of the glycerin produced in the saponification. Now 3KOH produces $C_3H_5(OH)_3$, hence the percentage of glycerin yielded by a fat is $\frac{92}{168} \cdot \frac{e}{10}$.

The Insoluble Acids.—The Hehner value gives the percentage of insoluble acids in a fat. In the absence of

soluble acids, the mean molecular weight of the acids in the fat may be calculated from the saponification value, S, and Hehner value, H :

$$A = \frac{560\,H}{S}.$$

The mean molecular weight of the insoluble acids in a fat may of course always be directly determined, after their isolation, from the amount of potash they require for neutralisation. In the absence of any large quantity of free acids, the mean molecular weight of the total acids in a fat is given approximately by the saponification value. The formula follows from the explanation of the last paragraph :

$$A = \frac{560 \times 100}{S} - \frac{38}{3}.$$

If two acids differ appreciably in respect of one analytical constant, the proportion of each acid in a mixture of the two can be determined. In the same manner *three* acids occurring together must differ in respect of *two* constants if the proportion of each acid can be determined in this manner. To take the case of a mixture of palmitic and stearic acids, the calculated molecular weights of these acids are 256 and 284 respectively ; if the saponification value of a mixture of these two acids be found to be S, then the percentage of palmitic acid, p, in the mixture may be calculated as follows :

$$p\frac{56}{256} + (100 - p)\frac{56}{284} = \frac{S}{10}.$$

From this equation p can be found. Since the difference between the molecular weights of palmitic and stearic acids is small, the saponification value of the mixture

would have to be very exactly determined in order to obtain an accurate result. In practice the determination would be made by taking the melting-point or 'titer' of the mixture, and comparing the result with the figures contained in a table of melting-points or 'titers' of mixtures of known composition determined by the same method (also see p. 149 *et seq.*).

Unsaturated Acids.—In mixtures of acids containing only one unsaturated acid—which, strictly speaking, are perhaps not often found—the proportion of the latter may be determined by means of the iodine value. Thus, in the mixtures of palmitic, stearic, and oleïc acids obtained in candle-making, the percentage, o, of oleïc acid (including iso-oleïc acid) is given by the iodine value, I, of the mixture. The iodine value calculated for oleïc acid is 90·1, and experiment gives practically the same figure. Consequently

$$o = \frac{100\,I}{90{\cdot}1}.$$

By combining the results given by the iodine value and saponification value (see p. 131) it is possible to calculate the percentages of all three acids, p, s, o, in the above mixture. The three following equations, which need no further explanation, suffice to determine the three unknown quantities:

$$p + s + o = 100,$$

$$p\frac{56}{256} + s\frac{56}{284} + o\frac{56}{282} = \frac{S}{10},$$

$$o = \frac{100\,I}{90{\cdot}1}.$$

It is obvious that the method just given may be

extended to mixtures of the triglycerides of oleïc, palmitic, and stearic acids. It would be a useful exercise for the student to deduce the equations necessary for the solution of the problem.

The unsaturated acids of a fat are rarely composed of oleïc acid alone. Various methods are known by which the unsaturated acids can be separated from the saturated, and then further examined. The results are not strictly accurate, but any method by which the actual composition of a fat can be ascertained is worthy of attention. The chief reason for separating the unsaturated fatty acids is in order that their iodine value may be determined.

The lead salts of the unsaturated acids are soluble in ether, while those of the saturated acids are only slightly soluble; on this difference are based several processes, of which Wallenstein and Finck's modification is here described (*J.S.C.I.*, 1895, 79). Three grms. of the fat are saponified by boiling on the water-bath with 30 c.c. of semi-normal alcoholic potash, phenolphthaleïn is added, then 10 per cent. acetic acid until the liquid is acid, and finally alcoholic potash to the point of exact neutralisation. The liquid is then slowly poured into a clear boiling solution of 3 grms. of lead acetate in about 200 c.c. of water, stirring continuously. The vessel is placed in cold water, the contents stirred for some minutes, and then left to settle for some hours until the liquid becomes almost clear; it is then poured off from the lead salts, which adhere firmly to the sides of the beaker and can be thoroughly washed with hot water. There is no necessity to filter the washings, which do not carry away appreciable quantities of the lead soaps. When lead is no longer found in the wash-waters, the soaps remaining

in the beaker are dried by means of filter paper, treated with 80 c.c. of ether, the whole transferred by means of further small quantities of ether (30 c.c. in all) to a Drechsel gas wash-bottle, the inside tube of which has been cut off. The air in the bottle is displaced by hydrogen, the tubes are closed by rubber caps, and the bottle is allowed to stand for twelve hours, when the ethereal solution will be clear. It is filtered through a folded filter into a separating funnel, shaken with 40 c.c. of dilute hydrochloric acid (1 : 4), then with more dilute acid, and finally with water. The ether solution of the free unsaturated acids thus obtained is run off into a flask to separate drops of water, and transferred to another flask, which is heated on the water-bath, while a rapid current of carbon dioxide is passed through, until all the ether is removed. The liquid acids may then be weighed off (the proper quantity according to the nature of the fat), and the iodine value determined in the ordinary manner. The iodine value of the unsaturated acids, being the iodine absorbed by the only constituents of the fat which are capable of combining with it, is known as the 'absolute' iodine value. The percentage of unsaturated acids in the total acids, or, what is almost the same thing, of unsaturated glycerides in the fat, is then $100 \times \frac{\text{iodine value of total acids}}{\text{absolute iodine value}}$. This method of calculating the percentage of unsaturated acids is preferable to a direct determination by weighing the acids themselves. Tortelli and Ruggeri (*J.S.C.I.*, 1900, 1043) make this method more reliable by using 20 grms. of the fat and extracting the lead salts with ether at the constant temperature of 8–10° C. The lower the temperature the less the quantity

of the salts of the saturated acids dissolved. A direct determination may be made by extracting the lead salts with ether as described, filtering the solution into a graduated 250 c.c. flask, and extracting with further quantities of ether until the flask is full. An aliquot portion of the ether solution is then evaporated in a tared flask in a current of carbon dioxide and the quantity of acids calculated from the weight of the lead salts on the assumption that oleïc acid is the only unsaturated acid in the fat.

In these processes the protection of the liquid acids and their lead salts from the action of air by means of carbon dioxide or hydrogen, as described, is important because of the readiness with which they oxidise.

In the case of fats containing a large preponderance of solid (saturated) acids, the isolation of the liquid acids may be made much more easy and accurate by dissolving the total acids in hot alcohol, allowing the solid acids to crystallise out, and converting the acids obtained from the solution into lead salts.

Farnsteiner (*J.S.C.I.*, 1898, 804, 958) has described a more rapid method in which benzene is employed to dissolve the lead salts of the unsaturated acids. The salts of the saturated acids are less soluble in benzene than in ether. One to two grms. of the fat are saponified by alcoholic potash, the solution is made neutral to phenolphthaleïn by adding acetic acid, it is transferred to a dish, the alcohol evaporated, the residue washed into a flask by means of about 200 c.c. of boiling water, and the solution precipitated by a boiling solution of 2 grms. of lead acetate in about 60 c.c. of water. When cold the liquid is filtered, the residue on the filter and in the flask washed with cold

water, and the water removed as completely as possible. The lead soaps are next dissolved in 100 c.c. of hot benzene.[1] On standing, crystallisation soon commences; the flask is then kept for two hours at 8–12° C., after which the liquid is filtered. The residue is not washed. The filtrate is shaken with 100 c.c. of 10 per cent. hydrochloric acid, then twice washed with water, and filtered. Three portions of 25 c.c. are taken, in two of which the iodine value is determined by adding Hübl solution and treating in the usual manner; the third portion is evaporated in a tared flask in a current of hydrogen—the residue gives the weight of liquid acids used in the estimations of the iodine value.

If it be desired to weigh the saturated and unsaturated acids, 0·6–1 grm. of the fat is employed, and the lead salts are dissolved in 50 c.c. of benzene. After cooling as described, a cork, carrying a short straight tube and a long tube reaching to the bottom of the flask and bent downwards outside, is inserted. The end of the long interior tube is covered with a plug of cotton wool, which serves as a filter. The benzene solution is expelled by forcing in air, then 10 c.c. of benzene are added and expelled. The residual lead salts are dissolved in 25 c.c. of hot benzene, the solution cooled for one hour at 8–12° C., and then expelled through the cotton-wool filter. This process of solution, cooling, and filtering is repeated a third time. The united benzene solutions are then shaken with 10 per

[1] The benzene must be free from thiophene, otherwise it will absorb iodine from the Hübl solution. Benzene, from which thiophene has been commercially removed by treatment with sulphuric acid, is finally freed from thiophene by boiling with about 5 per cent. of aluminium chloride under a reflux condenser, moisture being excluded, and then distilling (Heusler, *J.S.C.I.*, 1897, 131).

cent. hydrochloric acid, washed, filtered through cotton-wool, and evaporated in hydrogen. The lead salts of the saturated acids are dissolved in 25–30 c c. of benzene, the solution boiled under a reflux condenser with 10 per cent. hydrochloric acid, washed, and evaporated. Farnsteiner states that the liquid acids are found 1–3 per cent. too low and the solid acids 1·65 per cent. (as a maximum) too high.

In the preceding pages the terms 'unsaturated acids' and 'liquid acids' have been used somewhat indiscriminately. In separating the unsaturated acids by means of the solubilities of the lead salts, it must be understood that the solubilities of the lead salts of the solid unsaturated acids (elaïdic, iso-oleïc, and erucic) approximate to the solubilities of the corresponding salts of the *saturated* acids.

The iodine value of the liquid fatty acids affords a means of determining roughly the proportions in which oleïc, linolic, and linolenic acids are present. The analytical use of the absolute iodine value is illustrated in the table given on p. 138, the figures in the first part of which are due to Wallenstein and Finck, who employ this value for the determination of cottonseed oil in lard or mixtures of lard and tallow (oleomargarine). The figures in the second portion of the table are given by Tortelli and Ruggeri.

Detection and Separation of the Unsaturated Acids.—By dilute alkaline permanganate oleïc acid is oxidised to dihydroxystearic acid, linolic acid to sativic acid, $C_{18}H_{32}O_2(OH)_4$, and linolenic acid to linusic acid, $C_{18}H_{30}O_2(OH)_6$. From the product of the oxidation of a mixture of oleïc, linolic, and linolenic acids the three hydroxy-acids can be isolated and identified. The acids present in

'ABSOLUTE' IODINE VALUES

—	Absolute Iodine Value	Iodine Value of Fat
Berlin ox tallow	92·2	38·3
Australian tallow	92·4	45·2
Hungarian mutton tallow	92·7	38·6
American 'Western Steam Lard'	104·5	65·4
Berlin hogs' fat	96·6	52·7
Hungarian hogs' fat	96·2	60·4
Viennese ,,	95·2	60·9
Roumanian ,,	96·0	59·5
American white cotton-seed oil	147·5	108·0
N. American yellow ,, ,,	147·3	107·8
English white ,, ,,	146·8	106·5
Egyptian yellow ,, ,,	148·2	108·0
German white ,, ,,	147·1	107·7
Peruvian yellow ,, ,,	147·8	106·8
Rapeseed oil	120·7	101·1
Earthnut oil	128·5	98·9
Nigerseed oil	147·5	133·5
Maize oil	140·7	122·0
Cocoanut oil	54·0	8·4
Olive oil, Italian, 12 samples	97·5	83·6
,, Spanish, 7 ,,	100·4	85·5
,, Grecian	103·5	84·0
,, Turkish	96·3	79·1
Linseed oil, cold pressed	201·4	179·4
,, Italian, 1898	193·0	176·8
,, ,, 1897	190·1	173·4
Walnut oil	166·8	148·9
Sunflower-seed oil	154·3	137·0
Poppy-seed oil	149·6	137·2
Maize oil, 1899	143·7	124·6
Cotton-seed oil, commercial, 1897	148·9	107·9
,, ,, crude	147·3	105·3
Sesamé oil, Italian, 1898	134·4	108·7
Colza oil, 1898	125·5	101·7
Castor oil, 1898	106·9	87·4
Earthnut oil, commercial	123·4	93·5
Lard (Genoa)	97·0	57·5
Lard, American, average	99·8	63·2

many of the fatty oils have been examined in this manner. A process of this nature is naturally not suitable for, and does not give results applicable in, ordinary analytical work. Methods of value in analytical practice are based on the formation of bromine addition compounds by the direct action of bromine on the acids or the glycerides.

Oleïc acid forms a dibromide, $C_{18}H_{34}Br_2O_2$, which is readily soluble in ether, linolic acid gives a tetrabromide, $C_{18}H_{32}Br_4O_2$, which dissolves with much more difficulty in ether, and linolenic acid yields a hexabromide, $C_{18}H_{30}Br_6O_2$, almost insoluble in ether. Thus when bromine is added to a cooled solution of the mixed fatty acids of linseed oil in acetic acid, a precipitate at once forms, which, when thoroughly washed with ether, melts at 180–181° C. and is almost pure linolenic hexabromide. The weight of the hexabromide is 20–26 per cent. of the total fatty acids (Hehner and Mitchell, *J.S.C.I.*, 1899, 77). The filtrate and washings of the hexabromide yield on evaporation a precipitate of linolic tetrabromide, which melts at 113·4° C. Mixtures of linseed-oil acids with the acids of other oils, which give no precipitate with bromine, yielded weights of precipitate corresponding very nearly to the proportion of linseed oil present. According to Hehner and Mitchell (*loc. cit.*), it is more reliable to wash the precipitate slightly, in order to remove only the very soluble oleïc dibromide, then dry and weigh, and determine the percentage of bromine in the product. From this percentage the weight of linolenic hexabromide in the mixture can be calculated. Thus if m = the percentage of bromine found, and x = the percentage of hexabromide:

$$m = \frac{63{\cdot}3x}{100} + \frac{(100 - x)\,53{\cdot}3}{100};$$

in which equation 53·3 and 63·3 are the calculated percentages of bromine in linolic tetrabromide and linolenic hexabromide. The percentage of linseed-oil acids in the mixture is then roughly $\frac{100\,x}{23}$.

It is much simpler, and apparently equally reliable (Hehner and Mitchell), to act on the oils themselves with bromine. The oils which contain linolenic acid give an immediate precipitate. One to two grms. of the oil are dissolved in 40 c.c. of ether and 5 c.c. of glacial acetic acid, the flask is cooled in ice, and 1–1·5 c.c. of bromine added. It is advisable in accurate work to leave the mixture in ice overnight, to draw off the liquid by means of a thistle-funnel covered with chamois leather, and to wash the deposit in the flask by means of ether cooled to 0° C., four quantities of 10 c.c. being used. The deposit is then dried in the flask. Under ordinary circumstances ether may be used at the room temperature and the precipitate collected and dried on a weighed filter-paper. Five genuine samples of linseed oil examined in this manner gave 23·8–25·0 of the bromide, which is of complex constitution. It contained 55·55-56·38 per cent. of bromine : the theoretical quantities of bromine in the hexabromide of trilinolenin and the tetrabromide of trilinolin are 62·2 and 52·2 respectively.

When treated in the same manner, walnut oil gave 1·4–1·9 per cent. of the insoluble bromide compound, rape-seed oil gave 0·9 per cent., and mustard-husk oil 1·5 per cent. Poppy, cotton-seed, olive, Japanese wood, and almond oils gave none. The oils obtained from marine animals at once gave a precipitate with bromine ; it was, however, a mixture of a solid substance and a heavy oil, which latter was washed out with difficulty. Cod-liver oil gave 42·9 per cent., cod oil 35·5 per cent., shark oil 22 per cent., and whale oil 25 per cent. The bromine compounds obtained from these oils contained about the same percentage of bromine as the linseed-oil bromine compound,

but they decomposed without giving a melting-point. The glycerides from which they are derived must differ in constitution from the linseed-oil glycerides.

Farnsteiner (*J.S.C.I.*, 1899, 501) has employed a similar method in the examination of the liquid fatty acids of a number of oils. Linolic acid tetrabromide is almost insoluble in cold petroleum ether: 100 c.c. dissolve 0·014 grm. at 12·5° C. and 0·008 grm. at about 0° C. Linolenic acid hexabromide is insoluble even in hot petroleum ether. By utilising these solubilities linolic and linolenic acids were detected in fats in which their existence was previously doubtful or unknown. In the case of fats (lard) which contain a large proportion of oleïc acid, it is necessary to concentrate the more unsaturated acids by removing the bulk of the oleïc acid. For this purpose[1] Farnsteiner employed the comparative insolubility of barium oleate in benzene containing 5 per cent. of alcohol (95 per cent.); the liquid acids obtained from the barium salts soluble in this mixture of alcohol and benzene were then treated by the same process as the total fatty acids, or the total liquid fatty acids, of fats containing a larger proportion of linolic and linolenic acids. The acids were dissolved in petroleum ether, the solution cooled, and excess of bromine added. After standing some time, the excess of bromine and the solvent were driven off on the water-bath and the residue heated with petroleum ether, when linolenic hexabromide was left. The solution, after filtering and cooling to 12° C., deposited linolic acid tetrabromide, which was filtered off, washed, redissolved in

[1] This process cannot be used to separate oleïc acid quantitatively (Lewkowitsch, *J.S.C.I.*, 1900, 381).

petroleum ether, crystallised out, washed, dried, and weighed.

By working in this manner Farnsteiner found in cotton-seed oil about 18 per cent. of linolic acid (see p. 180), in sesamé oil 15·5 per cent., earthnut oil 6 per cent., almond oil 5·97 per cent., mustard-seed oil 4·5 per cent., and in the liquid acids of horse-fat 9·9 per cent. Olive oil was found to contain small quantities of linolic acid and a trace of linolenic acid, rapeseed oil and mustard-seed oil both contained linolenic acid, the latter 4 per cent. Traces of linolenic acid were found in butter-fat, tallow, and lard, and linolic acid also in lard.

Halphen has given further modifications of the test based on the formation of insoluble bromine compounds by the drying and marine animal oils (*J.S.C.I.*, 1901, 1244; 1902, 74). The brominating reagent consists of 28 c.c. of glacial acetic acid, 4 c.c. of nitrobenzene, and 1 c.c. of bromine; 10 c.c. are shaken with 0·5 c.c. of the oil. Walnut, hempseed, and linseed oils, also whale, seal, cod-liver, and Japanese fish oils, give heavy precipitates. The non-drying and semi-drying oils produced no precipitate, though certain samples of sesamé oil and horse-foot oil gave slight turbidities, and colza oil gave a turbidity which was removed on the addition of ether. The presence of 10 per cent. of a drying oil (except walnut oil) or marine animal oil in a non-drying oil can be detected in this manner. (See also p. 217.)

To differentiate between drying oils and marine animal oils, the precipitate is washed with ether, dried in the air, extracted by carbon tetrachloride, the solution evaporated, the residue heated with carbon tetrachloride

(2·5 c.c. per 0·1 grm. of residue), and the solution allowed to stand three to four hours. From drying oils a gelatinous deposit is then obtained, whilst marine animal oils give an opalescent solution from which traces of crystals separate.

Hydroxy-acids, Free Alcohols, and Oxidised Acids. The acetyl value of fat or wax is due, as already explained (p. 82), to the presence of free hydroxyl groups of alcohol function in a constituent or constituents. These free hydroxyl groups may be contained in hydroxy-acids, such as ricinoleïc acid; in free alcohols, such as may occur in small quantities in the fats and in larger quantities in the waxes; in diglycerides and monoglycerides, which may be present naturally, or may be formed in fats; and, finally, in what are termed oxidised acids—*i.e.* the acids formed in the oxidation of the drying and semi-drying oils, and which, as will be shown, differ essentially in constitution and properties from the simple hydroxy-acids of the nature of ricinoleïc acid.

Hydroxy-acids.—The oils and fats contain only small quantities of alcohols, so that an acetyl value obtained from an oil or fat cannot be due to that cause. Mono- and diglycerides are not generally present in fresh fats, in which the occurrence of an acetyl value is to be ascribed to the presence of a hydroxy-acid. The acetyl value of castor oil is about 150, and the theoretical acetyl value calculated for triricinoleïn is 158·8; thus the percentage of triricinoleïn in castor oil is $\frac{150 \times 100}{158{\cdot}8} = 94{\cdot}5$.

Free Alcohols.—The acetyl values given by the waxes are certainly due, in the case of beeswax, carnaüba wax, and woolwax, to the presence of free alcohols, which have

been found by other methods, but Lewkowitsch, to whom the figures given in the following table are due (*J.S.C.I.*, 1900, 75), does not regard this as certain in the case of the liquid waxes and spermaceti, the acetyl values of which might be due to the presence of oxidised acids. (All the figures in the table represent mgrms. of KOH per grm. of substance.)

—	Original Wax, Total Volatile Acids	Acetylated Wax, Apparent Acetyl Value		True Acetyl Value
		Distillation Process	Filtration Process	
LIQUID WAXES:				
Sperm oil, Northern . .	2·41	6·95	6·85	4·49
,, ,, (best) .	1·36	8·56	7·03	6·43
,, Southern . .	1·48	7·63	5·83	5·25
Arctic sperm (bottlenose) oil	1·29	8·00	7·28	6·35
,, ,, . . .	1·40	6·16	4·88	4·12
SOLID WAXES:				
Carnaüba	2·26	57·6	57·4	55·24
Beeswax	2·16	17·4	—	15·24
Woolwax	9·30	33·7	31·5	23·3
Spermaceti . . .	1·97	4·7	4·5	2·63
Woolwax alcohols . .	2·2	141·2	143·8	140·3

Diglycerides.—The natural occurrence of diglycerides in certain fats has already been mentioned (p. 6). Now it has been shown that the process of saponifying a triglyceride takes place by partial stages, in which di- and monoglycerides are intermediate products (p. 8); it is therefore to be expected that those fats, which are partially hydrolysed under natural conditions, by a process which accompanies, or is part of, the change to which rancidity is due, will contain mono- or diglycerides. The diglyceride of any acid has lower saponification and Hehner values than the corresponding triglyceride; the acid value, saponification value, and total fatty acids of

fats which have been exposed to the air are higher than the corresponding values of the original fat. Thus rancidity cannot be due simply to partial hydrolysis, which may, however, occur simultaneously with a process of oxidation. The proportion of a diglyceride (or monoglyceride) in a fat is determined by the acetyl value given by the acetylated fat, according to the process described on p. 84. This determination is not sufficient even in the absence of hydroxy-acids such as ricinoleïc acid : the fat may contain oxidised acids. It is therefore necessary also to take the acetyl value of the fatty acids, treating the acids in exactly the same manner as the original fat. If the acetyl value of the fat is appreciably greater than that of the fatty acids, the presence of diglycerides is rendered probable. Lewkowitsch has obtained results which appear to indicate the presence of mono- or diglycerides in rancid fats, and has obtained higher acetyl values for certain fats after exposure to the atmosphere (*J.S.C.I.*, 1900, 76), which may of course be due to the presence of oxidised acids. The changes which occur in fats on exposure to air are still far from fully explained.

Oxidised Acids.—This term is applied to certain acids contained in the products of the oxidation of the drying and semi-drying oils by exposure to air in a thin layer, or by means of a current of air blown through the oil. The oxidised acids are to be regarded as produced from the linolic and linolenic acids contained in the glycerides. The changes which take place in the oils during oxidation are represented analytically by increases in the specific gravity, the acid and saponification values, and in the soluble acids, the acquisition of a considerable acetyl value,

and decreases in the Hehner and iodine values. Such far-reaching changes in the character of the oil are evidently not due to any simple reaction. The mere formation of hydroxy-acids would only account for some of the alterations in the constants. The production of a substance with a considerable acetyl value indicates, however, the formation of a compound which is essentially a hydroxy-acid. The oxidised acids differ from the other fatty acids and from ricinoleïc acid in being insoluble in petroleum ether (Fahrion, *J.S.C.I.*, 1898, 958). In separating the oxidised acids, the oil is saponified with alcoholic potash, the alcohol is evaporated, and the soap dissolved in hot water. The solution is transferred to a separating funnel and hydrochloric acid added ; it is then shaken with petroleum ether, and allowed to stand. The aqueous layer is drawn off and then the ether layer, which contains the unoxidised acids and unsaponifiable matter, leaving the oxidised acids adhering to the funnel. If their quantity is considerable, they should be redissolved in caustic soda, again separated and shaken with petroleum ether, in order to remove any residual unoxidised acids. The oxidised acids are finally dissolved in hot alcohol, the solution evaporated, the residue dried to constant weight, and weighed.

Lewkowitsch has separated the oxidised acids by Fahrion's method from a number of oils (*J.S.C.I.*, 1900, 75 ; 1902, 780), and has also thoroughly examined the oxidised acids, and the fatty acids freed from the oxidised acids, of a number of blown oils and solid linseed oil. From the results, an extract from which is given in the table on p. 148, a number of conclusions may be drawn. The saponification values of the oxidised acids are considerably higher

than the acid values, hence the acids contain lactones. Either during the oxidation of the oils, or in the saponification by potash, soluble acids are formed; the oxidised acids were also found to be somewhat soluble in water and to decompose with the formation of volatile acids when exposed to the action of steam. The separation of the oxidised acids cannot be complete, since the fatty acids from which they had been extracted had still considerable acetyl values and also contained lactones. It is also to be noted that the percentage of oxidised acids, calculated from the acetyl value on the assumption that the oxidised acids have the same molecular weight as hydroxystearic acid, is generally much higher than the percentage actually found. The agreement between calculation and experiment is much better in the case of blown cotton-seed, rape-seed, and maize oils, which contain considerable percentages of oxidised acids. The iodine values of the oxidised acids show that they are still unsaturated compounds. The oxidised acids, obtained free from lactones by neutralising with potash and extracting the lactones with ether, again formed lactones; this formation of lactones was repeated when the acids were again subjected to the same treatment. The lactones separated from the oxidised acids in the manner just described had very low saponification values, and the fatty acids recovered from the saponified lactones had acid values much higher than the saponification values of the lactones—both very noteworthy results. It is evident that the oxidised acids must have a constitution essentially different from that of any simple hydroxy-acid, such as the hydroxystearic acid which has been employed for purposes of comparison, and that

—	Specific Gravity at 15·5° C.	Acid Value, Mgrms. KOH	Saponification Value, Mgrms. KOH	Iodine Value	Total Soluble Acids, Mgrms. KOH	Oxidised Acids, per cent.	Hehner Value	True Acetyl Value, Mgrms. KOH	True Acetyl Value × 0·55
Linseed oil	—	—	—	—	0·8 C.[1]	—	—	11·7	6·4
,, ,, blown 2 hours at 120° C.	0·9334	—	189·8	—	0·68[1]	1·2	—	17·22	9·47
,, ,, blown 10 hours at 120° C.	0·9460	—	192·7	—	0·9[1]	7·1	—	31·7	17·4
Cotton-seed oil	—	—	—	—	0·1[1]	—	—	7·6	4·18
Oleic acid	—	—	—	—	0·95[1]	—	—	2·05	1·13
,, ,, blown 4 hours at 120° C.	0·9121	—	206·0	—	3·6[1]	2·6	—	14·6	8·03
Blown rapeseed oil	0·9714	—	205·6	—	8·9[1]	24·95	—	53·14	29·22
,, cotton-seed oil	0·9722	—	213·7	—	16·06[1]	26·45	—	48·54	26·7
Boiled oil, I.	—	—	188·7	—	1·6[1]	6·5	—	24·0	13·2
,, ,, II.	—	—	186·1	—	1·1[1]	4·63	—	16·9	9·29
Blown Ravison rapeseed oil	0·9685	10·47	198·31	72·66	35·89	21·22	83·52	52·93	29·11
,, East India rapeseed oil	0·9623	13·25	215·57	61·92	56·26	20·74	82·18	46·61	25·63
,, Cotton-seed oil	0·9785	9·41	224·59	65·74	46·49	29·39	82·59	64·29	35·33
Solid linseed oil	—	—	287·47	52·2	136·9	53·02	53·92	—	—
Blown maize oil	0·9806	7·33	208·63	90·7	49·13	31·93	82·34	63·37	34·85
TOTAL FATTY ACIDS:									
Blown Ravison rapeseed oil	—	175·14	191·7	73·31	7·26	—	—	42·75	—
,, East India rapeseed oil	—	171·93	190·0	60·80	10·71	—	—	55·5	—
,, Cotton-seed oil	—	194·79	210·46	72·43	12·94	—	93·76	55·67	—
Solid linseed oil	—	209·63	248·74	60·27	59·57	—	81·32	55·04	—
Blown maize oil	—	192·8	209·93	88·08	29·45	—	86·4	59·52	—
OXIDISED ACIDS:									
Blown Ravison rapeseed oil	—	171·5	208·0	49·14	22·56	—	—	80·0	44·0
,, East India rapeseed oil	—	173·3	211·3	39·79	22·35	—	—	105·65	58·1
,, Cotton-seed oil	—	174·7	220·7	48·6	36·12	—	—	118·28	65·02
Solid linseed oil	—	—	—	46·49	59·68	—	—	104·99	57·74
Blown maize oil	—	171·9	215·74	70·87	48·0	—	93·53	125·68	69·67
FATTY ACIDS FREED FROM OXIDISED ACIDS:									
Blown Ravison rapeseed oil	—	176·8	188·6	61·88	6·97	—	—	35·53	19·54
,, East India rapeseed oil	—	166·6	176·8	55·93	10·09	—	—	37·54	20·64
,, Cotton-seed oil	—	188·0	196·15	56·02	11·0	—	—	22·69	12·49
Solid linseed oil	—	179·97	187·58	61·31	18·89	—	—	31·36	17·24
Blown maize oil	—	172·37	177·68	85·52	6·14	—	85·54	36·7	20·18

[1] These values refer to volatile acids.

probably polymerisation occurs, which would account for the high specific gravity of the blown oils being produced by the absorption of a relatively small amount of oxygen.

The following figures have been obtained by the author in the examination of blown cotton-seed oil :

Specific Gravity	Acid Value	Saponification Value	The Insoluble Fatty Acids		
			Acid Value	Saponification Value	Iodine Value
0·9620	5·6	210·8	189·8	210·6	76·0
0·9730	9·0	217·4	—	—	—
0·9801	9·6	220·7	161·1	207·7	62·6

The main results, from the analytical point of view, of these investigations of oxidised oils, apart from the records of constants, may be summarised as follows : (1) The oxidised oils have increased acid and saponification values, and it may be added that the saponification is more difficult to complete ; (2) the fatty acids have higher saponification than acid values ; (3) soluble acids are formed, hence the Hehner value is very low ; (4) the iodine values are decreased ; (5) the oils acquire a considerable acetyl value and also contain oxidised acids insoluble in petroleum ether, but there is no definite numerical connection between the acetyl value and the percentage of oxidised acids ; (6) the separation of the oxidised acids by means of petroleum ether is incomplete.

Separation of the Individual Saturated Acids.—The separation of the homologous saturated acids is not easy, and is rarely required. It may, however, be advisable to indicate the possible methods. The volatile fatty acids may be more or less completely separated by adding approximately sufficient alkali to neutralise the less volatile

acid or acids and distilling off the lower and more volatile acids in steam. The non-volatile acids are separated by a process of partial precipitation with magnesium or lead acetate, when the higher acids are first precipitated ; the acids are liberated from the precipitates and separately and repeatedly recrystallised, fractions of the same melting-point being united (see p. 161).

Stearic Acid.—By utilising the difference in solubility of stearic and palmitic acids in 90 per cent. alcohol, Hehner and Mitchell (*Analyst*, 1896, 316) have devised a process for separating stearic acid quantitatively from palmitic acid and the unsaturated acids. Saturated acids higher in the series than stearic acid must of course be absent. Hehner and Mitchell used alcohol of the density 0·8183 at 0° C., 100 c.c. of which dissolve at 0° C. 1·13 grm. of palmitic acid or 0·15 grm. of stearic acid. A saturated solution of pure stearic acid in this alcohol was prepared by dissolving 3 grms. in 1 litre and leaving in ice overnight. The solution, still in the ice, was then filtered by means of a thistle-funnel, the mouth of which was covered with calico. The stem of the funnel was connected with a flask, from which the air was exhausted. To determine the stearic acid in a mixture of fatty acids, weigh 0·5–1 grm. of solid acids or 5 grms. of liquid acids into a flask, dissolve in 100 c.c. of the alcohol saturated with stearic acid, leave in ice overnight, shake, filter off the solution as described above, while the flask still remains in the ice, wash in the same manner with the same alcoholic solution cooled to 0° C., finally dry the residue in the flask, and weigh. Deduct 0·005 grm. from the result as a correction for the stearic acid in the alcohol adhering to the sides of the

flask. The separated stearic acid should not melt below 68·5° C. The exact strength of the alcohol used in this method is naturally not important, but the same must be employed throughout.

Arachidic and Lignoceric Acids.—There is one case in which the separation and estimation of saturated acids are of considerable importance. Earthnut oil contains in addition to palmitic acid the higher acids, arachidic acid, $C_{20}H_{40}O_2$, and lignoceric acid, $C_{24}H_{48}O_2$, which are very slightly soluble in alcohol and can be separated with comparative ease. The original process devised by Renard consisted in extracting the lead soaps, obtained from 10 grms. of oil, with ether in order to remove lead oleate, and recrystallising the acids, separated from the insoluble lead salts, from 90 per cent. alcohol. Archbutt (*J.S.C.I.*, 1898, 1009, 1124) has considerably shortened the process by obviating the lengthy extraction of the lead soaps with ether. He has shown that the addition of a small quantity of lead acetate is sufficient to precipitate all the arachidic and lignoceric acids ; the precipitate then contains but little lead oleate, which can be more easily extracted. The process is as follows: Saponify 10 grms. of the oil, evaporate most of the alcohol, transfer to a separating funnel by means of hot water, acidify, and extract the fatty acids by shaking with two portions of ether. Wash the ether solution and then evaporate in a flask ; the residual acids must finally be dried by aspirating or blowing air through the flask. They are next dissolved in 50 c.c. of 90 per cent. alcohol, and to the hot solution 5 c.c. of a 20 per cent. aqueous solution of lead acetate are added. The flask is cooled, shaken, allowed to stand for thirty minutes,

the crystalline precipitate filtered off and once washed with ether; it is then immediately washed back into the flask by means of a jet of ether from a wash-bottle, digested with ether for some time, again filtered, and rinsed back into the flask. Four washings performed in this manner are sufficient to remove the lead oleate. After the final washing, the small filter paper, while still wet with ether, is opened and placed in a large filter-funnel supported in the neck of a separating funnel. The lead soaps are washed off the paper with a jet of ether; the particles remaining on the paper and in the flask are decomposed and transferred to the funnel by means of hot dilute hydrochloric acid and ether. Dilute hydrochloric acid is then added, the funnel is well shaken in order to decompose the lead soaps, and is repeatedly shaken with water to remove the lead chloride. The ethereal solution is evaporated and the residue dried as before, 50 c.c. of alcohol of exactly 90 per cent. strength (sp. gr. 0·834) are added, the flask is closed by a cork carrying a thermometer, heated carefully to dissolve the acids, and cooled to 15° C., at which temperature the arachidic and lignoceric acids rapidly separate.

If the operation is quantitative, the flask is maintained in a water-bath at a constant temperature between 15° and 20° C. for one hour, and the crystallised acids then collected on a small filter, or in a Gooch crucible, using only the filtrate to rinse them on to the filter, where they are washed with three quantities of 10 c.c. each of 90 per cent. alcohol, and finally with 70 per cent. alcohol. The acids are then extracted from the filter by boiling ether, the solution evaporated in a tared flask, and the residue dried and weighed. The filtrate and washings obtained from the

90 per cent. (*not* the 70 per cent.) alcohol are collected and measured; the weight of arachidic and lignoceric acids dissolved by this volume is then found from a table (given below) and added to the weight of the acids obtained.

Weight of Acids obtained	Correction (to be added) for the Acids dissolved by 100 c.c. of 90 per cent. Alcohol		
	15° C.	17·5° C.	20° C.
grm.	grm.	grm.	grm.
0·1 or less	0·033	0·039	0·046
0·2	0·048	0·056	0·064
0·3	0·055	0·064	0·074
0·4	0·061	0·070	0·080
0·5	0·064	0·074	0·085
0·6	0·067	0·077	0·088
0·7	0·069	0·079	0·090
0·8	0·070	0·080	0·091
0·9 or more	0·071	0·081	0·091

The mixture of arachidic and lignoceric acids obtained from earthnut oil in this manner amounts almost exactly to 5 per cent. of the oil, it melts at 71–72° C.; the saponification value of the mixture shows it to contain about one part of arachidic acid to three parts of lignoceric acid. Tortelli and Ruggeri (*J.S.C.I.*, 1898, 876) obtained a higher melting-point, 74–75·5° C. for the mixture of acids. The method is accurate to within about 1 per cent., and is capable of detecting 5 per cent. of earthnut oil in olive oil. Rapeseed oil frequently (apparently not always) contains a similar mixture of arachidic and lignoceric acids, the proportion of which may amount to 1·5 per cent. Mustard oil contains a somewhat smaller quantity of the mixture, and maize oil traces of arachidic acid.

Glycerin.—The glycerin produced in the saponification of a fat may be isolated by decomposing the soaps with acid, filtering off the fatty acids, neutralising the

solution, evaporating to dryness, and extracting the resulting mixture of glycerin and inorganic salts by a mixture of two parts of alcohol and one part of ether. On evaporation of the alcohol the glycerin is left, and can be subjected to qualitative tests. The volatility of glycerin prevents this method from being used for exact quantitative purposes. If the absence of di- and monoglycerides may be assumed, the ester value gives a measure of the glycerin produced in saponification (p. 130).

There are several processes in use for the determination of glycerin, of which Hehner's is the most reliable and most generally employed. It depends on the oxidation of glycerin to carbon dioxide and water by the action of excess of potassium bichromate and sulphuric acid, and determination of the excess of bichromate. The reactions are represented by the following equations :

$$C_3H_8O_3 + 7O = 3CO_2 + 4H_2O$$

$$K_2Cr_2O_7 + 4H_2SO_4 = K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3O.$$

The oxidation of ferrous sulphate is represented by

$$2FeSO_4 + H_2SO_4 + O = Fe_2(SO_4)_3 + H_2O.$$

Therefore $6FeSO_4$ is equivalent to 3O, which is equivalent to $\frac{3}{7}C_3H_8O_3$. By means of this relation, if the bichromate solution be standardised against ferrous ammonium sulphate, the amount of glycerin oxidised by 1 c.c. of bichromate solution can be calculated.

The solutions required are : (1) potassium bichromate, containing in 1 litre about 74·86 grms., together with 150 c.c. of strong sulphuric acid ; (2) ferrous ammonium sulphate, containing in 1 litre 240 grms. of the pure recrystallised salt ; (3) dilute potassium bichromate, made by diluting

100 c.c. of solution (1) to 1 litre. The exact strength is found by titrating 10 c.c. of the ferrous ammonium sulphate solution in the ordinary manner, using potassium ferricyanide as external indicator. The quantity of bichromate in 1 c.c., divided by 7·486, gives the equivalent in glycerin. The strength of solution (1) is then ten times as great.

In the case of crude glycerin it is first necessary to remove the chlorine and organic impurities; 1·5 grm. of the sample is weighed off into a 100 c.c. flask, a little moist precipitated silver oxide is added, and 5–10 c.c. of water. After standing ten minutes, basic lead acetate (see p. 156) is added in slight excess, the flask is filled up to the mark, and the liquid filtered through a dry paper. Twenty-five c.c. of the filtrate are measured into a beaker (previously freed from grease by means of a mixture of strong sulphuric acid with a little bichromate solution), then 40–50 c.c. of the strong bichromate solution[1] are added, and finally 15 c.c. of strong sulphuric acid. The beaker is covered and heated for two hours on the boiling water-bath, the liquid is then diluted, solution (2) added in slight excess, and the excess titrated by the dilute bichromate solution.

While glycerin cannot receive a final concentration on the water-bath without loss, solutions of as much as 50 per cent. strength may be boiled for a lengthened period without any loss, if the evaporated water be replaced. In accordance with this behaviour, Hehner gives the follow-

[1] This is a strong solution, and must be very carefully measured. In making solution (3), from the strength of which the strength of solution (1) is calculated, the temperature of the solution (1) must be noted. Subsequent measurements of solution (1) must always be made at the same temperature, or a correction be made. Hehner states that solution (1) expands 0·05 per cent. for 1° C.

ing method for determining the glycerin produced in the saponification of a fat: saponify 3 grms. of the fat with alcoholic potash; do not drive off the alcohol, but dilute to 200 c.c., decompose the soaps by sulphuric acid, filter off the acids, and wash them on the filter in the ordinary manner, rapidly boil the filtrate and washings, which amount to about 500 c.c., down to about 250 c.c., add sulphuric acid and bichromate, and proceed as above.

Richardson and Jaffé (*J.S.C.I.*, 1898, 330) have somewhat shortened and simplified Hehner's method by omitting the treatment with silver oxide, which they regard as unnecessary, oxidising the glycerin in a stronger solution, and then titrating a weighed quantity of ferrous ammonium sulphate with the partially reduced bichromate solution. The process, as applied to crude glycerin, is as follows: 25 grms. of the sample are weighed off into a 50 c.c. flask and diluted to the mark, 25 c.c. of this solution are precipitated with 7 c.c. of basic lead acetate solution (made by boiling a solution of 25 grms. of lead acetate in about 70 c.c. of water with 17·5 grms. of litharge until the latter is dissolved, then filtering, and diluting the cold filtrate to 100 c.c.). The liquid is filtered into a 250 c.c. flask, and the precipitate washed until the flask is nearly full. Excess of dilute sulphuric acid is added to precipitate the remaining lead, the flask is filled up to the mark, shaken, allowed to stand a few minutes, and a portion of the liquid filtered through a dry paper. Twenty c.c. of the filtrate are then transferred to a beaker (tall shape), the mouth of which is closed by a funnel with the stem much shortened, 25 c.c. of the strong bichromate solution are added, and then (cautiously) 25 c.c. of strong sulphuric acid. The mixture

is heated on the boiling water-bath for twenty minutes, when the oxidation is finished. The liquid is cooled, transferred to a 250 c.c. flask, diluted to the mark, and transferred to a burette. Twenty c.c. of a solution of ferrous ammonium sulphate (2·982 grms. in 100 c.c.) are then titrated by the bichromate solution in the ordinary manner.

The 20 c.c. of ferrous ammonium sulphate solution are equivalent to 0·01 grm. of glycerin (see p. 154), the 25 c.c. of bichromate solution are able to oxidise 0·25 grm. of glycerin. Therefore, if b c.c. of the partially reduced bichromate solution are required in the titration, the required percentage of glycerin is $(0{\cdot}25 - \frac{250}{b}\,0{\cdot}01)\,500$.

It is convenient to give here a brief account of the character and methods of examining the various commercial qualities of glycerin ; for a full account the student is referred to Lewkowitsch (*Chemical Analysis of Oils, Fats, and Waxes*, 2nd edit.) and Allen (*Commercial Organic Analysis*). The commercial qualities of glycerin may be divided into—

Chemically Pure Glycerin.—The Pharmacopœia of 1898 requires that glycerin shall be a clear colourless syrupy liquid of the specific gravity 1·260, odourless, neutral, of sweet taste, and free from ash. It must not exhibit the reactions characteristic of lead, copper, arsenic, iron, calcium, sodium, potassium, ammonium, hydrochloric acid, or sulphuric acid. When boiled with a little dilute acid and then made alkaline, it should give no red precipitate on subsequent boiling with excess of Fehling's solution. When mixed at the ordinary temperature with

an equal volume of dilute ammonia (1 : 2) and a few drops of silver nitrate, no darkening should be observed. At the most only a slight straw colouration should be produced by mixing with an equal volume of strong sulphuric acid (cooling during the mixing). When heated with equal volumes of alcohol (90 per cent.) and dilute sulphuric acid, no fruity odour should be produced, thus indicating the absence of butyric acid. The absence of arsenic is proved by mixing 2 c.c. in a long test-tube with 5 c.c. of a mixture of 1 vol. of pure hydrochloric acid and 7 vols. of water, adding 1 grm. of pure zinc, and covering the mouth of the tube by filter paper, previously moistened with a solution of mercuric chloride and dried ; no yellow stain should be produced within fifteen minutes.

A selection of the tests here given is amply sufficient to prove the purity of a sample. The strength is obtained by means of the specific gravity, which is best determined by the specific gravity bottle or Sprengel tube, taking great care to avoid the introduction of bubbles. The specific gravities of pure glycerin solutions are given in the table on p. 32. Hehner's method (see p. 155) may of course also be used, in which case the purification by lead acetate and silver oxide is omitted.

2. *Once-distilled glycerin* is mainly used in the manufacture of nitroglycerin for dynamite and other explosives. Distilled glycerin should be almost pure ; it differs from chemically pure glycerin in containing a slight amount of ash, which should not exceed 0·1 per cent., and in being yellow in colour ; from crude glycerin it is distinguished by giving no precipitate with basic lead acetate, owing to the practical absence of organic impurities and inorganic

salts. The characters of *dynamite glycerin* should be as follows (Barton, *J.S.C.I.*, 1895, 516; Lewkowitsch, *ibid.* 1895, 1073): The specific gravity, which is best determined by means of the bottle, should not be less than 1·261 at 15·5° C., corresponding to about 98·5 per cent. of glycerin. The residue, determined by slowly evaporating 2–3 grms. in a small platinum dish at 160° C. with the occasional addition of a few drops of water to facilitate vaporisation, should not exceed 0·15 per cent. The ash, which may be found by igniting the same residue, should be much lower. The sample should be neutral to litmus (Lewkowitsch); 50 grms., when diluted with water, should not require more than 0·3 c.c. of normal acid or alkali to produce neutrality towards phenolphthaleïn (Burton). The glycerin should give no turbidity, due to fatty acids, when diluted with 2 vols. of water, or when nitrous fumes are subsequently passed through and the liquid is heated on the water-bath for two hours. Chlorine (sodium chloride) may be present only in small quantity; a good sample contains only 0·002 per cent. of NaCl (Burton); 1 c.c. diluted with water gives only a slight turbidity with silver nitrate (Lewkowitsch). No discolouration, due to organic impurity, should be produced on diluting and warming with silver nitrate, and no yellow turbidity on adding a very slight excess of ammonia and then silver nitrate (absence of more than traces of arsenic). A crucial test of the fitness of glycerin for the manufacture of nitroglycerin is obtained by simulating the manufacturing operation; the experiment, for obvious reasons, is not to be performed by the inexperienced in such matters; details are given by Lewkowitsch (*loc. cit.*).

3. *Crude glycerin* is obtained by working up the aqueous liquids produced in the lime or sulphuric acid processes of saponifying fats used in the candle industry, and by evaporating spent soap lyes and removing the salt which separates. The liquid resulting from all three processes contains a considerable quantity of impurities and only some 80–90 per cent. of glycerin. The percentage of glycerin is estimated by Hehner's method. The estimation of the ash of crude glycerin (and also of the purer qualities) offers difficulties on account of its fusible nature and the presence of salts volatile at a red heat. The ash is determined by igniting 2–3 grms. at a very low temperature (the bottom of the platinum dish must not become red-hot) over a small flame ; the ignition necessarily lasts a considerable time. The operation may be hastened by evaporating to dryness over a small flame, carbonising the residue by heating to redness for a few seconds, extracting with water, filtering, burning the black residue, and then evaporating and igniting the aqueous solution in the same vessel (Richardson and Jaffé, *loc. cit.*). The difference between the sum of glycerin *plus* ash and the weight taken is due to water and organic impurities. The latter may contain fatty acids, volatile and non-volatile, methods for determining which, if required, may be based on the ordinary processes of oil analysis. According to Davis (*J.S.C.I.*, 1900, 112), an estimate of the organic impurities in glycerin is obtained by applying Hehner's process before and after the purification by lead acetate and silver oxide. The fatty acids are not oxidised by bichromate under the conditions of Hehner's method, and would consequently not be determined in this manner.

Example of a Complete Examination of the Composition of a Fat.—It will be instructive to see how the methods indicated in this and the preceding chapters can be applied to the determination of the exact composition of a fat. As an example we give a brief account of Browne's investigation of butter-fat (*Journ. Amer. Chem. Soc.* 1899, 612, 807; *J.S.C.I.*, 1899, 780, 1132). The fatty acids obtained from 100 grms. of butter-fat were first separated into soluble and insoluble acids; the latter were divided into two main portions by crystallising from a large volume of alcohol at 0° C. The crystalline acids, called Portion I., were recrystallised from a small volume of alcohol and the filtrate added to Portion II. Then Portion I. was recrystallised from 500 c.c. of alcohol, the crystalline acids forming Part 1; the filtrate was then twice concentrated, the deposits being termed Part 2 and Part 3, the final filtrate was added to Portion II. All the recrystallisations were made at 0° C.; thus the final filtrate could not carry much stearic acid to Portion II. The acids in Portion II. were divided into Part *a*, Part *b*, and Part *c* by fractional precipitation with magnesium acetate. Thus the total insoluble acids were divided into six fractions. The iodine values and saponification values of the acids in each fraction were determined; the iodine values gave the percentages of oleïc acid, and then the residual acids were calculated to stearic and palmitic acids in Parts 1, 2, and 3, and to palmitic, myristic, and lauric acids in Parts *a*, *b*, and *c* (see p. 131). Certain corrections were then necessary. The soluble acids isolated from the same 100 grms. of fat were washed with small quantities of water at 0°, 30°, 60°, and 95° C.

in succession, and thus divided into four fractions of different solubility. The mean molecular weight of the acids in each fraction then gave the amounts of each acid present, certain assumptions being made as to the acids in each fraction. In this manner the approximate proportions of all the fatty acids were found. The percentage of glycerin was determined by Hehner's method. The hydroxy-acids, calculated as hydroxy-stearic acid, were obtained from the acetyl value. The final composition, in percentages of the different glycerides, could then be calculated. The method of procedure in making a complete examination must vary greatly according to the nature of the fat.

CHAPTER VI

DESCRIPTION AND PROPERTIES OF THE MORE IMPORTANT OILS, FATS, AND WAXES, WITH THE METHODS FOR THEIR INVESTIGATION.

THE present chapter deals in the main with the application to individual fats and waxes of the methods already described. As regards the course of the examination, general directions cannot well be given. Much depends on the purpose for which the substance is to be used, and, if adulteration be suspected, on the materials available for adulteration, having regard to market prices. An observance of prevailing prices will generally show that the oils and fats which might be used for adulteration are strictly limited in number; thus the labour of the analyst in finding the precise adulterant may be much diminished.

In the case of a sample of which the smell, taste, colour, &c., do not sufficiently indicate the nature of the oil or fat, the various properties must be examined and compared with the known values of the various oils. The oil will thus be identified, and the identification may then be made secure by the application of some colour or other test characteristic of the oil indicated.

The purity of a sample of material of stated, or evident, nature is more often in question. The specific

gravity of a liquid oil is generally first determined; it may afford indications both of purity and quality. In most cases it is advisable to make a qualitative test for unsaponifiable oils; the course to be pursued if these are present has already been indicated (pp. 69, 108). Then some other property, the numerical value of which is characteristic of the oil in question, is determined—*e.g.* the iodine value of linseed oil. If some one property is really distinctive of the oil in question, and the determination gives satisfactory results, the matter is at an end. Otherwise several properties must be determined, the indications of all summed up, the number of possible adulterants reduced, and the oil finally pronounced as genuine or adulterated; if the latter, the presence of the adulterant must be proved, if possible, by the determination of some constituent or observation of some characteristic property. In regard to the analytical constants of an oil, it is to be observed that each 'constant' has a certain range—*i.e.* the constants found for different pure samples vary within limits. It must also be remembered that the term 'pure' has more than one meaning: the seeds and other materials from which oils and fats are obtained are themselves frequently not to be obtained in commerce in a state of purity. Consequently, the oils expressed from them are also not pure in the strict sense, though they have not been intentionally adulterated. A distinction is thus to be drawn between actual and commercial 'purity.' It may be necessary to regard as 'pure,' or of 'good merchantable' quality, an oil which has not altogether the properties of the really pure oil obtained from strictly pure materials on the small scale.

Vegetable oils may differ in properties according to the age of the seed from which they are extracted, the richness of the ground on which the seed was grown, and for other reasons. The properties of animal oils vary somewhat with the food of the animal, and considerably if they are taken from different parts of the animal. In all cases different methods of refining and treatment may produce oils of slightly different character. In view of all these causes which produce differences in the characters of the product, it is desirable that chemists, who have to deal extensively with any one oil, should examine a large number of 'commercially pure' samples, and thus be in a position to state the range of each analytical property, and further to give the characters of oils which best satisfy their requirements. As an illustration, it may be stated that rapeseed oils, which in all probability are 'commercially pure,' range in specific gravity up to 0·920; it is, however, not unusual for consumers of a rape oil for burning purposes to specify that the oil delivered to them shall not exceed a specific gravity of, say, 0·916.

We now proceed to give a brief description of the individual oils, fats, and waxes, and to indicate the methods by which their purity can be ascertained either by the application of principles already laid down, or by means of special reactions. The classification and order of arrangement have been previously given (p. 101). For the numerical constants the reader is referred to the tables on pp. 62–67, 102–103.

DRYING OILS

The oils of this class are few in number, and of the seven here described linseed oil is the only one extensively used in this country. Chinese wood oil is now perhaps second in importance, and will no doubt attract increasing attention. The remaining oils are rarely used.

Linseed oil is obtained by pressing the seeds of the flax plant, *Linum usitatissimum.* The cold-pressed oil is pale yellow in colour; the hot-pressed, which is the ordinary oil of commerce, ranges from pale to dark yellowish-brown. Linseed oil is also occasionally obtained by extraction processes. Lewkowitsch states (*Analysis of Oils, Fats, and Waxes*, 2nd edit.) that extracted linseed oil is unsuitable for paint making, which is not in accordance with the author's experience. Linseed oil has a characteristic odour and taste; the odour changes somewhat when the oil is kept or heated. It dries to a transparent elastic skin in three to four days.

Linseed oil is refined by the acid process; the refined oil varies from a pale greenish-yellow to yellow in colour, and may further be bleached almost white by exposure to light. The quality of the oil varies roughly with the source of the seed: 'Baltic' oil is the best, and commands a higher price. East Indian oils frequently approach Baltic oil in quality.

The specific gravity of linseed oil ranges from 0·930 at 15·5° C. for the ordinary qualities, through 0·932–0·933 for good East Indian oils to 0·933–0·935 for Baltic oil. It should never be below 0·930, and very rarely exceeds 0·935. The specific gravity is thus in some degree a measure of

quality. The iodine value follows the variation of the specific gravity, the oils of high specific gravity having high iodine values. The iodine value of linseed oil is differently stated by different observers; the published figures of Wijs and Ingle, with which the writer's results agree, would assign limits to the iodine value of Baltic oil of about 190–200, and of ordinary linseed oil of about 170–185. Lewkowitsch has, however, recorded iodine values of 170–177 for samples of Baltic oil (*J.S.C.I.*, 1899, 51), which had been kept for some time. Values as low as 160 have been recently observed (Gill, *J.S.C.I.*, 1899, 282) by Hübl's method. If fresh Baltic linseed oil is not entirely protected from the action of air (and light?), the iodine value rapidly and continuously decreases; the writer has observed such a decrease in a sample kept in an apparently well-corked bottle. The lower iodine values of the older observers were due to the use of an insufficient excess of iodine and a short period of reaction.

The acid value of fresh linseed oil is generally very low, but samples made from damaged seed contain large quantities of free acid, amounting to 25 per cent. or even more.

Linseed oil contains a relatively large proportion of unsaponifiable matter, which should not be found to exceed 2 per cent. The oil contains about 15 per cent. of the glycerides of palmitic, stearic, and myristic acids, 4 per cent. of triolein, 13 per cent. of trilinolin, and 68 per cent. of trilinolenin (Bauer and Hazara); the relative proportions of the unsaturated acids would, however, appear, according to Hehner and Mitchell's work (p. 139), to be very different.

The usual adulterants of raw linseed oil are mineral and rosin oils, which are easily detected and estimated

from the amount of unsaponifiable matter. In view of the high price of linseed oil, which has now ruled for some time, various semi-drying oils might be used as adulterants. Maize oil is used for this purpose and is difficult to detect. Cottonseed and rapeseed oils might also be added in small quantity; the former would be detected by Halphen's reaction (p. 183), the latter by low saponification and iodine values. Hehner and Mitchell's method (p. 139) would probably detect the presence of 10 per cent. of a semi-drying or non-drying oil.

In view of the considerable range in quality of pure linseed oils, the corresponding range of the more important 'constants,' and the difficulty of detecting small quantities of certain semi-drying oils, the quality of linseed oil (if unsaponifiable oils are absent) is best estimated by means of the specific gravity, the iodine value, and a drying test. The Maumené test also gives useful indications.

The drying power of linseed oil may be tested as follows: heat 100 c.c. of the sample to 130° C., stir in slowly 0·5 grm. of litharge in fine powder, continue to heat at 130° C. for five minutes, stirring vigorously, then add 0·5 grm. of precipated manganese resinate. After cooling, put two drops on a plate of glass, spread out with the finger over an area of about six square inches, and put in a horizontal position to dry. A standard sample of oil, similarly treated at the same time, must be put to dry alongside the first. The two patches of oil must be exposed to exactly the same conditions of air and light. The time of drying depends on the temperature, illumination, &c.; thus results, expressed as the time required to

dry, obtained on one day are not comparable with results obtained on another day. The layers of oil are examined by touching with the finger every 15 minutes after drying commences; when the skin of oil just ceases to adhere to the finger the oil is to be regarded as dry. It must then continue to harden further, and after another twenty-four hours should be hard. A good oil dries in a short time and subsequently becomes quite hard; an oil which dries but remains sticky must be regarded as of very low quality, and is most probably adulterated.

The drying of linseed oil may also be accelerated by heating in order that it may take place within a convenient time: 2 drops of the oil are spread out to cover an area about 3 × 3 inches on a sheet of ground glass (perfectly clean and dry), a standard sample is similarly spread out on the same sheet of glass, which is then placed in the water-oven at 100° C. Good Baltic linseed oil dries under these conditions in seventy-five minutes, ordinary linseed oil in ninety minutes. Archbutt (*J.S.C.I.*, 1899, 347) performs the test by brushing the oil over two pieces of polished plate glass 2¾ inches square, while the glass is hot, then adding more oil to make the total weight exactly 0·1 grm., and drying in an air-oven at 50° C. Pieces of glass similarly coated with a standard oil are placed alongside. After nine hours one glass coated with each oil is withdrawn, and the second glass after twelve hours. Good linseed oil is still tacky after nine hours, but quite dry in twelve hours.

Linseed oil intended for varnish making is required not to separate any considerable quantity of 'mucilage' when it is heated to 300° C. The experiment is best performed in a metal vessel, but a flask may be used; the

oil is *rapidly* heated to 300° C., transferred to a test-tube, and allowed to cool. Good 'varnish oil' shows little or no deposit; in the case of a bad oil the volume occupied by the separated matter may be equal, or almost equal, to the volume of the oil.

Linseed oil is used in the manufacture of paints, boiled oil, linoleum, soft soap, &c. Boiled oil is now generally made by heating raw linseed oil in steam-jacketed pans, adding 'driers' (generally lead and manganese compounds), and blowing in a current of air; it may dry in as little as five hours. Lithographic varnish or 'stand oil' is made by heating linseed oil to high temperatures, at which it gives off inflammable vapours and thickens. In the manufacture of linoleum, linseed oil, which has been dried in thin layers or has been otherwise oxidised until solid, is incorporated with ground cork, pigments, &c.

Wood oil, also known as tung oil and Chinese or Japanese wood oil, is not to be confounded with the essential oil which is also known as gurjun balsam. It is obtained in China and Japan from the seeds of the tree *Elæococca vernicia* or *Aleurites cordata*. It is a thick oil, which becomes semi-solid at low temperatures; it has a peculiar clinging and somewhat nauseous odour, the colour ranges from pale yellow to pale brown. On exposure to light it is slowly converted into a solid fat, owing to the conversion of the glyceride of elaomargaric acid into that of the isomeric elaostearic acid. Wood oil dries more rapidly than linseed oil; the raw oil requires about two days, but when 'boiled' with 'driers' it dries much more rapidly than boiled linseed oil. The dried films are very hard, but are crinkled and adhere but little to the surface

beneath. When heated to 200° C., wood oil is converted into a jelly, which does not melt at higher temperatures and is insoluble in solvents.

From the very diverse results obtained in the examination of different samples of the oil (see pp. 62–63, 102–103), it is probable that it is often much changed in the crude method of extraction employed in China. The oil dries better than linseed oil, but has a lower iodine value; this may be due to previous oxidation of the oil, but there are reasons for supposing that wood oil has a different constitution from the other drying oils (Ingle, *J.S.C.I.*, 1902, 594). The greater drying action may be due to other causes than the absorption of oxygen.

Until a number of authentic samples of wood oil have been examined it will be difficult to detect small adulterations of wood oil by other fatty oils. The high specific gravity and iodine value are characteristic, as also the high melting-point of the fatty acids. Wood oil gives no precipitate with bromine (Hehner and Mitchell); thus the presence of linseed oil could be detected. The readily recognised odour of wood oil prevents its fraudulent addition to other oils, which is not probable for other reasons.

Wood oil is used in China for varnishing furniture and in very large quantities for oiling junks. In this country it will probably be applied to special purposes, for which the peculiar hardness of its dried films renders it particularly suitable. Broadly speaking, wood oil cannot be regarded as a substitute for linseed oil. Its property of gelatinising at a temperature of 200° C. renders difficult its employment in the manufacture of oil varnishes.

Candle-nut oil is extracted from the kernel of the nut of *Aleurites molucanna* or *triloba.* It is a somewhat thick oil, light yellow in colour, it readily turns rancid, and is stated to be intermediate between linseed oil and wood oil in regard to drying properties. The iodine values recorded for candle-nut oil are divergent: de Negri found 136·3–139·3 and Lewkowitsch 163·7, both working on extracted oils. The latter value is more in accordance with the drying properties, the former corresponds better with the specific gravity (0·920–0·926). Candle-nut oil contains the glycerides of palmitic, myristic, oleïc, and linolic acids.

Candle-nut oil is not yet an article of commerce in this country. It is employed as a drying oil, also for burning and soap-making.

Walnut oil is expressed from the kernels of the walnut, the fruit of the common walnut tree, *Juglans regia.* The cold-pressed oil is colourless to pale yellow, and has an agreeable flavour; the hot-pressed oil is greenish-yellow.

Walnut oil contains the glycerides of myristic, lauric, oleïc, linolic, and linolenic acids. It gives 1·4–1·9 per cent. of the insoluble bromine compound when treated by Hehner and Mitchell's process (p. 140). The iodine value serves to characterise the oil; an addition of linseed oil would give an increased iodine value and an increased quantity of the insoluble bromine compound. An addition of cotton-seed oil would be recognised by the lower iodine value and by Halphen's reaction.

Walnut oil is used as a drying oil by artists, the hot-pressed oil is employed on the Continent in place of (or

together with) linseed oil in making boiled oil. The cold-pressed oil is also used for edible purposes.

The cold-pressed oil of the black walnut, *Juglans nigra* L., which is an American species, is stated by Kebler (*J.S.C.I.*, 1901, 727) to be limpid, straw-yellow in colour, and to possess an agreeable odour and taste. It becomes turbid at 12° C., the specific gravity is 0·9215 at 15° C.; it has the acid value 8·6–9, saponification value 190·1–191·5, Hehner value 92·77, Reichert-Meissl value 15, iodine value 141·4–142·7. The fatty acids melt at 0° C.

Poppy-seed oil is obtained by expressing the seeds of the poppy, *Papaver somniferum*. The cold-pressed oil is almost colourless, with a yellow tinge; the taste is pleasant and odour is almost absent. The hot-pressed oil is reddish, has an acrid taste, and characteristic odour.

Poppy oil contains the glycerides of palmitic, stearic, oleïc, and linolic acids; it gives no precipitate with bromine (Hehner and Mitchell, p. 140), and linolenic acid is probably absent.

Poppy oil is recognised in non-drying oils (olive oil) by the increased iodine value and specific gravity. The presence of earthnut, sesamé, or cotton-seed oils in poppy-seed oil would be recognised by the reactions characteristic of those oils and by a decreased iodine value.

The cold-pressed oil is largely used as a salad oil and generally for culinary purposes; it is also employed by artists as a drying oil. The hot-pressed oil is used in boiled oil, as an illuminating oil, and for soap-making.

Niger-seed oil. This oil is obtained from the seeds of *Guizotia oleifera*, a plant cultivated in India and Germany. The oil is pale brownish-yellow in colour, fluid, and has a

pleasant nutty flavour. Niger oil has only feeble drying properties and almost belongs to the class of semi-drying oils. It is used in India for culinary and illuminating purposes; in this country its application, which is not extensive, is confined to soap-making.

SEMI-DRYING OILS

These oils are of a considerable importance, which is not due to the feeble drying properties they possess. They absorb oxygen from the air, especially when heated, and become more viscid and specifically heavier, but they are not converted into a dry film. The oxygen absorption is commercially utilised in the manufacture of 'blown oils' from cotton-seed and rapeseed oils; these thickened oils are valuable lubricants. The properties and composition of the oils of this class are intermediate between those of the drying and non-drying oils. The semi-drying oils contain linolin, but contain little, if any, linolenin.

Sunflower-seed oil is expressed from the seeds (achenes) of the sunflower, *Helianthus annuus*. The oil of the first pressure is pale yellow in colour, has little odour, and a bland taste. The oil obtained by a second pressing is darker.

Sunflower oil is used very largely in Russia as an edible oil, and for burning and soap-making. It is also stated to be used as a paint oil.

Maize oil. The germ of maize (Indian corn) contains a considerable quantity of oil, which would be injurious to the flour and whisky prepared from the grain. In distilleries, after malting, the germ is separated

by sifting and then subjected to pressure, when maize oil is obtained (Archbutt, *J.S.C.I.*, 1899, 346). The oil is yellow or yellowish-brown in colour, when refined it is much paler; it has a slight, pleasant odour. When examined by Archbutt's drying test (p. 169) maize oil dried in eighteen hours, while cotton-seed oil required twenty-one hours. It is therefore unsuitable for lubricating purposes.

In accordance with its drying properties, maize oil has an iodine value approaching those of the drying oils. The Reichert-Meissl value may be as high as 10, though some samples appear to be free from volatile acids.

Maize oil contains a high proportion of unsaponifiable matter: 1·4–1·7 per cent. extracted in the ordinary manner, which is mainly phytosterol, and in addition 1·1–1·5 per cent. of lecithin. When maize oil is dissolved in carbon bisulphide and a drop of sulphuric acid is added, a violet colouration, due to the phytosterol, is developed after twenty-four hours. The glycerides of the oil contain palmitic, hypogæic, oleïc, and linolic acids, and traces of arachidic acid; the presence of stearic acid is doubtful. Maize oil has a considerable acetyl value, 11·1–11·5 (Winfield, *J.S.C.I.*, 1899, 1031), which may indicate the presence of ricinoleïc acid. Among the volatile acids, formic, acetic, caproïc, caprylic, and capric are probably present. The iodine value of the liquid fatty acids is 136–140. In the elaïdin test it gives a dark red liquid with an orange-coloured deposit.

The general reactions must be relied upon for the detection of maize oil in other oils. Maize oil itself is probably only liable to adulteration with cotton-seed oil, which can be detected by its special reactions.

Maize oil is somewhat extensively imported into this country ; it is used for soap-making and probably for the adulteration of other oils, of which linseed oil is certainly one. It is also stated to be used as a burning oil.

Pumpkin-seed oil is obtained from the seeds of the pumpkin, *Cucurbita pepo.* According to Poda (*J.S.C.I.*, 1898, 1054), the oil is obtained by heating and hot-pressing, while Graham (*J.S.C.I.*, 1901, 1003) states that it is usually obtained by extraction with solvents. The cold-pressed oil is greenish in colour, with a faint red fluorescence ; the hot-pressed oil appears brownish-green by transmitted and red by reflected light (Poda). The oil extracted by acetone is red and has a pleasant odour and taste (Graham). It readily becomes rancid.

The variations in the iodine value are due, according to Poda, to oxidation occurring in the drying and roasting of the seeds, which are preliminary to hot-pressing.

Pumpkin-seed oil is largely used in Austria and Hungary for culinary purposes ; it approaches olive oil in price. The oils used as adulterants are linseed, sesamé, cotton-seed, and rape oils, an addition of any of which can be detected by means of the refractometer, the modified iodine value, specific gravity and melting-point of the fatty acids. Pumpkin-seed oil gives negative results in Becchi's reaction for cotton-seed oil and Baudouin's reaction for sesamé oil.

Sesamé oil is obtained from the seeds of *Sesamum orientale*, which is extensively cultivated in semi-tropical countries. It is a pale-yellow oil, odourless, and of bland flavour.

Sesamé oil is composed of the glycerides of palmitic,

stearic, oleïc, and linolic acids; according to Farnsteiner (p. 142), it contains 15–16 per cent. of the last-named. The unsaponifiable matter extracted by ether in the ordinary manner contains phytosterol, sesamin, $C_{11}H_{12}O_3$ (?), and a red oil; on treatment with a little ether, sesamin is left. It is colourless, and crystallises either in round irregular crystals or in aggregates of needles thicker than those of phytosterol, from which it is easily distinguished by treating the dried substance with a mixture of equal parts of acetic anhydride and strong sulphuric acid, when the crystals become in turn brownish-green, bluish-green, cherry-red, and finally reddish-blue. Phytosterol remains unchanged. Pure sesamin melts at 120–122° C., and is dextro-rotatory; its presence accounts for the optical rotation of the oil. If the solution of phytosterol and the red oil in ether be filtered through animal charcoal, and the charcoal extracted by ether, a brown resin is obtained which gives Baudouin's reaction (see below) at great dilutions (Bömer, *J.S.C.I.*, 1899, 1054).

Sesamé oil is used for adulterating olive oil and other edible fats and oils. In Germany 10 per cent. of sesamé oil must be present in margarine in order that the latter may readily be detected in butter. It may itself be adulterated with earthnut, cotton-seed, and rapeseed oils. Earthnut oil is detected by isolating the less soluble saturated acids (see p. 151), and by the decreased specific gravity and iodine value. An addition of cotton-seed oil, which may be detected by the colour reactions (p. 181), increases the iodine value. Rapeseed oil decreases the saponification value.

Sesamé oil gives a number of characteristic colour

reactions, which afford the best means for its detection in other oils.

Baudouin's reaction was originally performed by shaking the oil in the cold with half its volume of strong hydrochloric acid and a little cane sugar. In the presence of small quantities of sesamé oil, a red colouration is produced which, when the liquids separate, is seen to be in the acid layer. The reaction was found to be due to the production of furfurol by the action of the hydrochloric acid on the sugar, and the subsequent action of the furfurol on a constituent of the oil. Furfurol is now generally used at once, and in the form of a 1 per cent. solution in alcohol, 0·1 c.c. of which is shaken with 5 c.c. of the oil and 10 c.c. of strong hydrochloric acid for half a minute. Furfurol itself produces a colouration with hydrochloric acid, but no reddish colouration is obtained if the proper quantity of furfurol solution is not exceeded. The method is capable of detecting 0·2 per cent. of sesamé oil. It may be applied with some loss of delicacy to the fatty acids; the fatty acids of certain olive oils, which themselves give a colouration with Baudouin's reaction, give no reaction (see p. 194).

Butter and other fats may be coloured by dyestuffs which give a red colouration with hydrochloric acid, and must therefore be removed before applying Baudouin's reaction. This is accomplished by washing repeatedly with hydrochloric acid until the acid is no longer coloured. The separation of the oil and acid is accelerated by heating to 60° C. Filtering through animal charcoal, which removes turmeric and other colouring matters, also removes the compound which gives the colouration with

furfurol and hydrochloric acid. Butter should also be freed from albuminous matter, which might give a violet colouration with hydrochloric acid, by melting and filtering, or by dissolving in ether, filtering, and evaporating the solvent (Amthor, *J.S.C.I.*, 1900, 473). Rancid fats containing sesamé oil do not give Baudouin's reaction (Soltsien, *J.S.C.I.*, 1899, 301), neither does sesamé oil which has been heated for some time at 100° C. This must be remembered in extracting the oil from chocolate (Utz, *J.S.C.I.*, 1902, 642.

Bishop's Reaction.—Rancid sesamé oil, sesamé oil after exposure to air and light for several days, and rancid fats to which fresh sesamé oil is added, give a green colouration when shaken with strong hydrochloric acid (*J.S.C.I.*, 1899, 1158). This method is capable of detecting 5–10 per cent. of sesamé oil.

Tambon's Reaction.—Certain olive oils give a reddish colouration in Baudouin's test. These oils give, however, no reaction when treated in the following manner: 3–4 grms. of pure dextrose are dissolved in 100 c.c. of hydrochloric acid, 5 c.c. of this solution are well shaken with 10 c.c. of oil, the mixture is heated just to boiling, and then allowed to stand. In the presence of 2 per cent. of sesamé oil a rose-red colouration, soon changing to cherry-red, is slowly produced (*J.S.C.I.*, 1901, 285, 1121). The reaction is also given by the fatty acids isolated from the oil.

Tocher's reaction may also be mentioned. The pure olive oils which give a colouration with Baudouin's reagent are unaffected. Fifteen grms. of the oil are shaken with a solution of 2 grms. of pyrogallic acid in 30 grms. of hydro-

chloric acid. After standing, the oil is drawn off and the acid layer boiled for five minutes ; in the presence of sesamé oil a reddish-purple colouration is produced (*J.S.C.I.*, 1898, 275 ; 1899, 1054).

Sesamé oil is an excellent salad oil, and is largely used for edible purposes. It also finds employment in soap-making. Its compulsory presence in margarine made in Germany has already been mentioned.

Cotton-seed oil is obtained by expressing the seeds of the cotton plant. The crude oil is dark brownish-red ; it is refined by treatment with caustic soda, which leaves a pale yellow oil of slight characteristic odour and pleasant flavour. When cooled, the refined oil separates cotton-seed stearin. There are in commerce several qualities of cotton-seed oil superior to the ordinary refined oil and intended especially for culinary use, for burning, and apparently for adulterating other oils ; these superior qualities may differ slightly in specific gravity and iodine value from the ordinary refined oil, than which they are generally paler and have less odour.

Cotton-seed oil contains the glycerides of palmitic, stearic, oleïc, and linolic acids. The oil gives no precipitate with bromine, and linolenic acid appears to be absent. Farnsteiner (p. 142) finds that cotton-seed oil contains about 18 per cent. of linolic acid, which, however, hardly agrees with the following calculation : the iodine value of the total acids is about 112, the iodine value of the liquid fatty acids is about 148 (Wallenstein and Finck, p. 138), hence the total acids contain $\frac{100 \times 112}{148} = 75$ per cent. of liquid acids. Now the iodine value of the liquid acids, 148, shows

that they contain about 2 parts of linolic acid to 1 part of oleïc acid, hence 50 per cent. of the total acids should be linolic acid. The oil also contains two volatile compounds, one of which gives the red colouration with Halphen's reagent, the other the brown colouration in Becchi's reaction.

Cotton oil contains a small quantity of hydroxy- or oxidised acids, as indicated by the acetyl value (see the table on p. 148).

Cotton-seed oil is the cheapest saponifiable oil, and is therefore generally not adulterated. On the other hand, it is very widely used for adulterating other oils. Apart from the colour reactions given below, the constants of cotton-seed oil which serve to detect it in other oils are the iodine value, specific gravity, and high melting-point of the fatty acids; the iodine value of the liquid fatty acids is also of great service (see pp. 137, 138, 198). Cotton oil is refined by means of alkalis, hence the acid value of the fresh oil is very low.

A large number of colour reactions have been proposed for the detection of cotton-seed oil, and the literature on the subject is voluminous. Becchi's and Halphen's reactions are the most reliable, and the latter, except in certain special cases, is apparently perfectly trustworthy.

Becchi's reaction is based on the production of a brown colouration, due to the formation of silver sulphide or reduced metallic silver, when cotton-seed oil is heated with silver nitrate. The silver nitrate solution is obtained by dissolving 1 grm. of silver nitrate in 200 c.c. of strong alcohol and adding 0·1 c.c. of nitric acid (the exact quantity is important). 10 c.c. of the oil are well shaken with

2 c.c. of the reagent; 1 c.c. of ether may be added to obtain intimate admixture. The tube is then heated in the boiling water-bath. In the presence of more than 10 per cent. of cotton-seed oil, a brown or black colouration is produced.

Tortelli and Ruggeri (*J.S.C.I.*, 1898, 607) have rendered Becchi's test more delicate by applying it to the liquid fatty acids: 5 grms. of the oil are saponified by alcoholic potash, the liquid exactly neutralised by acetic acid, and poured into a hot solution of lead acetate. The lead soaps are well washed, dried between paper, and boiled with anhydrous ether under a reflux condenser for twenty minutes. The ethereal solution is cooled, filtered, the lead removed by shaking with hydrochloric acid, the ethereal solution washed, and the solvent evaporated. The residue is heated at 70–80° C. with 10 c.c. of 90 per cent. alcohol and 1 c.c. of a 5 per cent. solution of silver nitrate in water. In the presence of as little as 1 per cent. of cotton-seed oil, a reduction is seen within two minutes, while with pure olive oil the liquid remains clear for thirty minutes.

Cotton-seed oil which has been heated to 250° C. does not give Becchi's or Halphen's reaction; 10 per cent. of such heated oil may, however, be detected by this modification of Becchi's test (*J.S.C.I.*, 1901, 753).

According to Tolman (*J.S.C.I.*, 1902, 643), the substances, which cause rancid olive oils frequently to give colourations with silver nitrate, can be removed by extracting in the cold with 95 per cent. alcohol, washing with 2 per cent. nitric acid, and finally with water.

Halphen's reaction depends on the production of a red

colouration when cotton-seed oil is heated with a solution of sulphur in carbon bisulphide. The test is performed as follows (*J.S.C.I.*, 1897, 1045): in a test-tube put 2 c.c. each of the oil, amyl alcohol, and a 1 per cent. solution of sulphur in carbon bisulphide. Heat by plunging the tube in a boiling brine bath, removing the tube for a minute or two at first to permit the carbon bisulphide to escape. If no colouration appears in thirty minutes, add 1 c.c. more of the sulphur solution and continue to heat. In the presence of 5 per cent. of cotton-seed oil a strong red colouration is produced, while 1 per cent. gives a perfectly recognisable colouration if the original oil is pale. The reaction is given by cotton-seed oil refined in various ways, but not by oil which has been heated at 250° C. for some time or by oil which has been thickened beyond a certain point by 'blowing.' Van Kettel records one sample of crude yellow Egyptian oil which did not give the reaction (*J.S.C.I.*, 1900, 471). No other oil gives a similar colouration; Soltsien believes that a slight colouration, about equal to that produced by 1 per cent. of cotton-seed oil, given by certain American lards, was due to feeding the pigs on cotton-seed meal (*J.S.C.I.*, 1901, 393). The butter from cows fed on cotton cake gives a similar reaction (*J.S.C.I.*, 1900, 172).

Soltsien is of opinion that cotton-seed oil, which has been heated to 250° C., cannot be used in edible fats, to which, therefore, Halphen's reaction may be applied in all cases. The fatty acids also give the reaction, though not after drying at 100° C. (Wauters, *J.S.C.I.*, 1900, 172).

Cotton-seed oil is used for culinary purposes for

adulterating lard and olive oil, as a burning oil, and in soap-making. Blown cotton oil is very largely used in lubricating oils for heavy machinery.

Cotton-oil Stearin.—This term is applied to two very different substances: the solid fat which separates from cotton-seed oil at a low temperature, and a semi-solid mixture of fatty acids obtained by working up the 'foots' produced in refining cotton-seed oil. The composition of the former, to which the term should be restricted, varies according to the temperature at which it was made. Thus the iodine value ranges from 90 to 103. Cotton-oil stearin gives the same reactions as cotton-seed oil, and may thus be detected in lard and butter.

Rapeseed oil is expressed or extracted by solvents from the seeds of varieties of *Brassica campestris*. There are two chief qualities of rape oil: colza oil and ordinary rape oil, to which may be added Ravison rape oil as a variety of the second class.

Crude rape oil is brown in colour and has a characteristic odour; the colour of the refined oil ranges from a very pale greenish-yellow to pale yellow. The specific gravity of colza oil is 0·912–0·914, while that of ordinary rape oil rises to 0·917 or higher. Archbutt states the specific gravity of pure rape oil should not exceed 0·916. Samples of specific gravities up to 0·919, which there is no reason to consider are intentionally impure, are, however, frequently found. In contracts for burning oil it is usual to specify that the oil shall not exceed the specific gravity of 0·916 or 0·917. The acid value is frequently high, 6–8 per cent. of oleïc acid is not uncommon, but the acid value of rape oil for burning or for

lubrication should be much lower. The saponification value of rape oil is lower than that of other fatty oils, with the exception of the allied mustard-seed oils; it should not exceed 179, and therefore serves to indicate adulterated oils and the presence of rape oil in other oils. Rapeseed oil has little drying power, and on this account is preferred to cotton-seed oil for lubrication.

Rapeseed oil is liable to adulteration with mineral oils, cotton-seed oil, and linseed oil. The presence of cotton-seed oil raises the iodine and saponification values, the specific gravity, and the melting-point of the fatty acids; its presence is detected with certainty by means of Halphen's reaction (p. 182). Linseed oil largely increases the iodine value and the specific gravity, and lowers the melting-point of the fatty acids; its presence would be detected by means of Hehner and Mitchell's bromine reaction (pp. 139, 140).

Rapeseed oil contains the glycerides of stearic, erucic, rapic, and arachidic acids (p. 153), and apparently a small quantity of linolenic acid (Hehner and Mitchell, p. 140; Halphen, p. 142).

Rapeseed oil is very largely used as a burning oil, for which purpose colza oil is superior to ordinary rape oil. It is also used for lubricating, generally in admixture with mineral oils, after thickening by blowing, and for soap-making.

Black and white mustard-seed oils much resemble rapeseed oil; they have similar low specific gravities and saponification values. They are golden-yellow oils, with little odour and an agreeable taste; in other respects they are sufficiently described by the properties given

in the tables (pp. 62, 102). One sample of mustard-seed oil examined by Archbutt contained arachidic acid (p. 153).

NON-DRYING OILS AND SOLID FATS

The oils in this class include the remaining vegetable oils, all the animal oils and fats except those from marine animals, and also the vegetable fats which are solid at the ordinary temperature. There does not appear to be any essential difference in the composition of the animal and vegetable fats, if the unsaponifiable matter be disregarded, neither can any strict line be drawn between liquid oils and solid fats.

Earthnut or **arachis oil** is obtained from the earthnut, ground nut, or monkey-nut, which is the seed of *Arachis hypogæa.*

The cold-pressed oil is pale in colour and has an agreeable flavour; the hot-pressed oil is yellow. Certain samples of Indian earthnut oil, undoubtedly genuine, had specific gravities higher than those generally recorded—viz. 0·920–0·9256—whilst the iodine value and other properties were normal. These oils gave deposits on standing. The iodine value of the liquid fatty acids is 128·5 (Wallenstein and Finck).

Earthnut oil contains palmitic acid, arachidic acid (1·3 per cent.), lignoceric acid (3·8 per cent.), oleïc acid, and about 6 per cent. of linolic acid (p. 142). Advantage is taken of the slight solubility of arachidic and lignoceric acids in alcohol to detect earthnut oil in olive and sesamé oils, which it is used (the former especially) to adulterate.

The absence of rapeseed oil, which contains smaller quantities of arachidic and lignoceric acids, must be proved by the saponification value before the presence of earthnut oil is definitely shown. The process for the quantitative estimation of arachidic acid has already been given (p. 151). A simpler method, adapted to qualitative work, is given by Bellier (*J.S.C.I.*, 1899, 303): 1 c.c. of the oil is saponified by boiling for two minutes with 5 c.c. of alcoholic potash of 8·5 per cent. strength, the solution is neutralised by dilute acetic acid, and rapidly cooled in water at 17–19° C. When the precipitate ceases to form, 50 c.c. of 70 per cent. alcohol, containing exactly 1 per cent. by volume of hydrochloric acid, are added, the flask is well shaken, and again cooled. If the oil contains more than 10 per cent. of earthnut oil, a distinct precipitate of arachidic acid is formed; in the presence of less than 10 per cent. the bottom of the flask is obscured by a cloud after standing for thirty minutes. Occasional samples of Tunis olive oil, particularly rich in solid acids, leave a turbidity after the addition of the alcohol, but it disappears on warming and does not reappear when the liquid is again cooled at 17–19° C. for one or two hours.

Earthnut oil may be adulterated by cotton-seed oil, which is recognised by Halphen's reaction and possibly by the higher melting-point of the fatty acids and increased iodine value. Drying oils are recognised by means of the iodine value. Soltsien (*J.S.C.I.*, 1901, 1121) states that sesamé oil is added to the best (edible) qualities of earthnut oil; the addition is detected by means of Baudouin's reaction (see p. 178).

In addition to the illegitimate use of earthnut oil for

adulterating olive and sesamé oils, it is extensively used under its own name for culinary purposes. The hot-pressed oil is used for soap-making in very large quantities.

Almond oil is expressed from either sweet or bitter almonds, which are the seeds of varieties of *Amygdalus communis*. It is a thin, very pale yellow oil, practically without odour, and has a nutty flavour. It is mainly composed of the glyceride of oleïc acid ; it also contains palmitic acid, about 6 per cent. of linolic acid (Farnsteiner), but no linolenic acid. The British Pharmacopœia requires that almond oil shall remain liquid at —20° C., while the German Pharmacopœia specifies — 10° C. In the elaïdin test, which is performed by shaking 2 c.c. of the oil with 1 c.c. of *fuming* nitric acid and 1 c.c. of water, a whitish mixture (not brownish-red) should be formed, which after standing six hours at 10° C. should separate into a white solid mass and an almost colourless liquid ; indications to the contrary denote the presence of peach-kernel or other oils (Pharmacopœia).

The oils mainly used for adulterating almond oil are those of peach, cherry, and apricot kernels, which are very similar and therefore difficult to detect. The low melting-point of the fatty acids serves to indicate the presence of other oils.

Almond oil enters into the composition of certain ointments, and is also used for culinary purposes.

Croton oil is expressed from the seeds of *Croton tiglium*, Linn. It is brownish-yellow to reddish-brown in colour, is viscid, has an unpleasant odour and burning taste. It has violent purgative and vesicating properties, for the sake of which it is employed in medicine.

Croton oil contains the glycerides of both higher and lower saturated acids, oleïc acid, and crotonoleïc acid. The latter acid, which is allied to oleïc acid, is stated to be made on the large scale by extracting croton oil with alcohol, saponifying the extracted portion with baryta, extracting the barium salts with ether, which dissolves the oleate and crotonoleate, and finally extracting the latter by ether (? alcohol). The vesicating constituent, which is a resin $(C_{13}H_{18}O_4)_x$, accompanies the crotonoleïc acid. (Dunstan and Boole, *J.S.C.I.*, 1895, 768, 985.)

Croton oil is soluble in absolute alcohol and in petroleum ether. It does not give an elaïdin ; according to the Pharmacopœia, 2 c.c. when shaken with 1 c.c. of fuming nitric acid and 1 c c. of water should not solidify, but only thicken slightly after standing two days. The constants upon which reliance is to be placed in detecting adulteration are the high specific gravity (0·940–0·950), high saponification value (210–216), Reichert-Meissl value, and acetyl value. The acetyl value is given by Benedikt as 8·5 (Benedikt and Ulzer's method), by Dulière as 38·6 (Lewkowitsch's method), and the apparent acetyl value (see p. 85) by Lewkowitsch as 41·1–53·5. Dulière also gives (*J.S.C.I.*, 1899, 1133) the following constants: specific gravity at 15° C., 0·9437. ; at 100° C., 0·8874 ; solubility in 92 per cent. alcohol, 1 in 63 ; butyro-refractometer reading, 77·5° at 27° C. ; saponification value, 215·6 ; solidifying point of the fatty acids, 16·4–16·7° C. ; Reichert-Meissl value, 12·1 ; iodine value (Hübl, two hours), 100·4–101·9 ; (twelve hours), 103·6–104·4 ; iodine value of fatty acids (two hours), 111·2–111·8. Dulière states that the percentage of castor oil, which may be present in an adulte-

rated sample, can be calculated from the acetyl value. The presence of castor oil may be detected by the formation of sebacic acid on fusion with caustic potash.

Grapeseed oil is obtained by pressing or extracting the seeds of the grape. According to Benedikt (*Analyse der Fette und Wachsarten*), the extracted oil is green, readily soluble in glacial acetic acid at 70° C., and only partially soluble in alcohol. The expressed oil is brownish-yellow.

Grapeseed oil gives a high acetyl value (144·5) by the older method of Benedikt and Ulzer ; no determination by Lewkowitsch's process has yet been published. The acetyl value indicates a close relationship with castor oil. Grapeseed oil is used in wine-growing countries for illuminating and table purposes.

Castor oil is expressed or extracted by volatile solvents from the seeds of *Ricinus communis*. It is a very thick viscous oil, the best qualities of which are colourless and odourless ; the lower qualities vary from a pale yellow to dark brown, and also possess an odour.

Castor oil is mainly composed of triricinolein (p. 143) ; it contains a little tristearin. This exceptional composition accounts for the characteristic properties of castor oil, which make its examination easy. The specific gravity, 0·960–0·970, is higher than of any other fatty oil, except the blown oils. The Pharmacopœia limit of specific gravity is 0·950–0·970, which, according to Dowzard (*J.S.C.I.*, 1901, 370), should be reduced to 0·960–0·967.

Castor oil mixes with absolute alcohol in any proportions, it dissolves in 3–4 vols. of 90 per cent. alcohol at 15° C. (Dowzard), which is confirmed by Allen (*Com-*

mercial Organic Analysis), while the Pharmacopœia test gives 5 vols. Castor oil dissolves in an equal volume of glacial acetic acid ; it is insoluble in petroleum ether and mineral oils, but dissolves about its own volume of these solvents. In the presence of other oils castor oil dissolves in petroleum ether. Castor oil has a high viscosity : 1,160-1,190 secs. in Redwood's viscosimeter at 100° F. Its optical rotation is characteristic, and would serve to detect any large addition of another oil.

The high acetyl value, low saponification value, and absence of soluble acids serve to differentiate castor oil from blown cotton-seed and rapeseed oils, which have equal or greater specific gravities but which would probably be detected by their smell. The acetyl value (146–150) also distinguishes castor oil from all other oils, except grapeseed oil, which might be difficult to detect.

Castor oil is used for medicinal purposes, in soap-making, for the preparation of Turkey-red oil (see p. 231), and as a lubricant.

Olive-kernel Oil.—In the manufacture of olive oil the olives are crushed, means being taken to keep the kernels whole, and the pulp is then expressed. The residual mass, known as *bagasses*, after keeping some time, is again pressed or extracted with solvents. According to Klein (*J.S.C.I.*, 1898, 1055), the oil so obtained is generally known as 'kernel' oil, and the properties hitherto recorded for olive-kernel oil have been obtained by examination of the *bagasses* oil. It has a dark green or brown colour, a sharp and burning taste, and contains large quantities of free fatty acids, which explains its solubility in alcohol and acetic acid. Klein obtained the following

results: specific gravity, 0·9277; free fatty acids, 71 per cent.; iodine value, 71·6; saponification value, 190·5.

Klein also examined a true kernel oil, which was free from oil obtained from the pulp, and obtained the figures: specific gravity, 0·9186–0·9191; iodine value, 87–87·8; saponification value, 182·3–183·8; free acids, 1·0–1·8 per cent.; refractive index, 1·4682–1·4688. It is thus very similar to olive oil, but has a somewhat higher specific gravity and iodine value. The true kernel oil was found to be no more liable to turn rancid than olive oil itself. It contains 9·7 per cent. of solid fatty acids (palmitic and stearic), and also oleïc and linolic acids.

Olive oil is obtained from the fleshy pulp of the olive, the fruit of *Olea europæa*, many varieties of which are known. Although there is little difference between the chemical properties of the oil of the pulp and of the kernel of the olive (see above), and consequently no appreciable difference between the oil expressed from the pulp alone and that obtained from the pulp after crushing the kernels, yet the former commands a considerably higher price on account of its superior flavour (*J.S.C.I.*, 1900, 912).

Olive oil ranges in colour from almost colourless to yellow or green. The better qualities have a bland, pleasant flavour; the lower qualities, in the preparation of which the pulp has been allowed to ferment, may contain considerable quantities of free acid and are not suitable for culinary purposes. Olive oil may become turbid, and deposit solid fat at relatively high temperatures (10° C.); the deposit and the residual oil have practically the same composition.

The fatty acids of olive oil contain 13–30 per cent. of solid acids; Holde and Stange (*J.S.C.I.*, 1901, 1003) isolated from the oil about 1 per cent. of oleodimargarin (pp. 6, 12). The liquid acids consist mainly of oleïc acid, but contain a small quantity of linolic acid and a trace of linolenic acid (p. 142). Olive oil contains much smaller quantities of phytosterol than most other vegetable oils.

Olive oil is adulterated by sesamé, earthnut, cotton-seed, and lard oils, and possibly also with rapeseed and other oils. All these adulterants, with the exception of lard oil, increase the specific gravity and the iodine value; the latter constant is the one of most value in detecting fraud. The iodine value of olive oil does not normally exceed 84, but certain (Tunis) oils have iodine values of 85–89, and a sample of Indian oil examined by Crossley and Le Sueur had a value of 93·67 and a specific gravity of 0·9203 (*J.S.C.I.*, 1898, 994). The melting-point of the fatty acids is raised by considerable additions of cotton-seed oil, and lowered by rapeseed and linseed oils. The saponification value is also decreased by rapeseed oil. The elaïdin yielded by olive oil is characteristic (see p. 97).

Cotton-seed, earthnut, and sesamé oils, which are the more usual adulterants, are detected by the special reactions already given for each of those oils. The substances which give Becchi's reaction for cotton-seed oil and Baudouin's reaction for sesamé oil are present in the liquid fatty acids obtained from the lead salts. Tortelli and Ruggeri state that the whole of the substance which gives Baudouin's reaction for sesamé oil passes into the

liquid fatty acids, but that the compound, which is present in certain pure olive oils and gives a rose or violet colouration in the reaction, is entirely absent from the liquid acids. In accordance with these facts, Tortelli and Ruggeri (*J.S.C.I.*, 1898, 607) have devised a process for detecting cotton-seed, sesamé, and earthnut oils by working on the same sample: 20 grms. of the oil are saponified by boiling with 120 c.c. of alcoholic potash (60 grms. in 1 litre of 90 per cent. alcohol), the liquid fatty acids are then isolated as described on p. 182. One half of the liquid acids is tested for cotton-seed oil (p. 182), and the other half is examined for sesamé oil by the furfurol reaction. The insoluble lead salts may be treated by one of the methods for isolating arachidic acid described on pp. 151, 187, in order to detect the presence of earthnut oil.

The quantity of unsaponifiable matter (phytosterol) in olive oil is very small; it has been proposed to detect the addition of other vegetable oils by means of the increased percentage of unsaponifiable matter. The presence of lard oil would be detected by the presence of cholesterol in the unsaponifiable matter (see pp. 115–118). Lard oil might also be detected by its odour.

Olive oil is the table oil *par excellence*; the poorer qualities, obtained in the second and subsequent pressings and by extraction of the residues, are used as lubricants, wool oils, and for soap-making.

Ben or **behen oil** is obtained from the seeds of certain species of *Moringa*. It is a pale odourless oil of pleasant flavour. It contains the glycerides of myristic, palmitic, stearic, oleïc, and behenic acids.

Ben oil is used as an edible oil and for lubricating

purposes. It does not appear to be imported into England.

Neat's-foot oil is properly obtained by boiling the feet of oxen and calves with water and removing the fat which rises to the surface. The oil from the feet of other animals—sheep, pigs, horses—is no doubt also often present in commercial neat's-foot oil. The oil is pale yellow in colour, and has a distinct and characteristic odour; at low temperatures it deposits 'stearin' or turns semi-solid. It contains very little free acid.

Neat's-foot oil is very frequently adulterated; the following oils are said to be added: mineral oils, marine animal oils, lard oil, rapeseed oil, cotton-seed oil. Mineral oils and cotton-seed oil are easily detected. As regards other adulterants, the iodine and saponification values give the most useful indications. The figures given in the following table indicate the range of the properties; the corresponding values obtained for samples of horse oil are added.

—	Specific Gravity	Saponification Value	Iodine Value	Iodine Value of Fatty Acids	Titer Test °C	Observers
Neat's-foot oil, 5 samples.	15° C., 0·914–0·919	—	67·1–72·9	63·6–69·5	16–26·5	Gill and Rowe
Horse oil, 3 samples.	15° C., 0·916–0·922	—	75·1–86·3	72·9–78·7	25·0–33·5	Gill and Rowe
Horse oil, 2 samples.	100° C., 0·798–0·799	—	78·8–79·9	80·4–82·1	30–35	Gill and Rowe
Neat's-foot oil, 11 samples.	—	194–199	66–77·6	—	—	Holde and Stange
Neat's-foot oil, 2 samples.	$\frac{15·5}{15·5}$, 0·9169–0·9174	195·5–197·4	71·1–72·4	74 75·8	28·5–29·2 (melting-point of acids)	Coste and Parry

The fatty acids isolated by Coste and Parry (*J.S.C.I.*, 1898, 5) were white, crystalline, and almost odourless.

In order to detect the presence of vegetable oils in neat's-foot oil, Holde and Stange (*J.S.C.I.*, 1901, 484) examined the unsaponifiable matter (see p. 118). A somewhat different method from those already described was used in examining the extracted matter: the soap from 50 grms. of the fat was extracted by shaking with three successive volumes of 600–700 c.c. of ether. The extracts were evaporated, the residue dissolved in 10 c.c. of absolute alcohol on the water-bath, and water added until a turbidity began to appear. After cooling, the precipitate was collected and repeatedly recrystallised in the same manner until, after drying on a porous plate, the crystals had a constant melting-point. The cholesterol thus obtained from neat's-foot oil melted at 145–147° C. (one sample at 147–148° C.). Adulterated oils gave mixtures of phytosterol and cholesterol melting at 143–144° C. to 130–131° C. In view of the difficulty of detecting additions of certain vegetable oils (rape oil), the desirability of examining the unsaponifiable matter is evident.

Neat's-foot oil is a valuable lubricant, and is largely used in preparing leather.

Lard is obtained from the fat of the hog by subjecting it to the action of heat (generally steam-heat) and straining off the impurities. The best quality is obtained from the 'leaf,' the sheet of fat covering the intestines; probably fat from other parts of the animal enters into the composition of most commercial lards. The consistency, and consequently the iodine value, of lard obtained from different parts of the animal vary to some extent. Similar variations are found in the fat of hogs fed on different foods and in different climates.

Lard is an almost white semi-solid fat, with a slight characteristic odour and taste. The melting-point and consistency are variable. Lard is mainly composed of the glycerides of stearic, palmitic, and oleïc acid ; it contains a little linolin and linolenin (Farnsteiner). An examination of the refractive index by means of the refractometer (p. 58) may be of service in the detection of adulteration.

Lard is extensively adulterated ; the substances used for this purpose are beef and mutton tallow, cotton-seed oil, and cotton-seed stearin ; sesamé oil, cocoanut oil, and earthnut oil may also be used. By selecting the more solid and the liquid constituents in proper proportions, the consistency of genuine lard is exactly reproduced. Lard occasionally contains a considerable quantity of water, which is incorporated by the aid of alkaline solutions ; if a sample does not readily yield a clear oil on warming, it may be necessary to determine the water (p. 104). Other adulterations of a gross character would be found as matter insoluble in ether.

Cotton-seed, sesamé, and earthnut oils are readily detected by means of the special reactions already given (pure lard may give a reaction in Becchi's test). Their presence may also be proved by examining the unsaponifiable matter for phytosterol (p. 118). Cocoanut oil would be indicated by a high saponification value and the presence of volatile acids (Reichert-Meissl value). It should be mentioned that Soltsien (*J.S.C.I.*, 1901, 393), in examining American lards, has obtained with Halphen's reagent a colouration equal to that given by 1 per cent. of cotton-seed oil, which he believes to be due to the hogs having been fed on cotton-seed meal. Maize oil is also

probably used for adulterating lard; it would increase the iodine value, and phytosterol would readily be detected in the unsaponifiable matter.

The iodine value is the constant upon which the main reliance is to be placed for the detection (apart from special reactions) and estimation of vegetable fats. The iodine value of lard does not exceed 65 and is generally about 60, though it may be considerably lower; pure American lard reaches the upper limit. Since the iodine value of cotton-seed oil is 106–108 and that of cotton-seed stearin about 90, the amount of either present in lard could readily be estimated, if tallow, which has a lower iodine value (41–46), were not used in conjunction with cotton oil and if the iodine value of lard were not variable. It is not possible to prove the presence of small quantities of vegetable oils in this manner.

Wallenstein and Finck emphasise the importance of estimating the iodine value of the liquid fatty acids, which may be seen by examining the figures in the table given on p. 138. Assuming average values for the 'absolute' iodine number of cotton-seed oil and of the animal fats, the percentage of the former present in a mixture can be calculated with some accuracy.

The detection of tallow in lard is somewhat more difficult; a considerable proportion may be present without reducing the iodine value below the normal limits. A small proportion is best detected by examining microscopically the stearin, which crystallises from a solvent in which the lard has been dissolved. In the presence of tallow characteristic crystals are obtained, and the amount of the deposit is also more considerable. Whatever

method be adopted, more certainty is obtained by examining samples containing 5, 10, and 20 per cent. of tallow at the same time. The method given by Goske (*J.S.C.I.*, 1896, 53) is as follows: 1 grm. of a hard lard or 2 grms. of a soft lard are dissolved in 10 c.c. of ether in a test-tube, which is then closed by a plug of cotton-wool, and set aside to cool at a temperature of 12–13° C. (not below). When a sufficient quantity of stearin has crystallised out, the liquid is poured off and replaced by a little colourless oil to form a medium for the microscopic examination. A sample of the best crystals is transferred to an object-glass, covered by a cover-slip, which is gently pressed down and the slide examined. The stearin which separates from pure steam lard forms sharp-edged plates of considerable width ; occasional bundles of needle-shaped crystals are also seen. In the presence of tallow, needles disposed in curved tufts predominate. The crystals obtained from butcher's lard contain many needle-shaped ones and are with difficulty distinguished from those of tallow. About 5 per cent. of beef-tallow, or 15 per cent. of mutton-tallow, may be recognised in this manner.

Gladding gives a different process, in which the crystals first obtained are recrystallised (*J.S.C.I.*, 1896, 560, 831) : 5 c.c. of melted lard are dissolved in 20 c.c. of absolute alcohol and 10 c.c. of ether in a small conical flask, which is plugged with cotton-wool and the contents allowed to cool. The stearin is filtered off, washed once with the same mixture of alcohol and ether, dried, and again dissolved in 25 c.c. of ether. The flask is put in a slanting position overnight in water to cool. The crystals are then examined as above.

According to Cochran (*J.S.C.I.*, 1898, 74, 697), 2 c.c. of the melted fat are warmed in a graduated cylinder with 22 c.c. of fusel oil until dissolved; the solution is then cooled slowly to 16–17° C., at which it is kept for from two to three hours. The deposit obtained from beef-fat occupies 16 c.c., from oleostearin the whole 24 c.c., while the maximum from pure lard is 4 c.c. The deposit is thoroughly drained on paper, recrystallised from ether, and examined in oil under the microscope. In the presence of 10 per cent. of beef-fat the typical crystals are plentiful, with 20 per cent. no lard crystals are seen, with 5 per cent. of oleostearin the form of the crystals is somewhat modified, but they can still be identified as due to beef-fat.

Hehner states that these various methods for detecting beef-fat must be applied with great caution. The distinctive shape of the crystals obtained from lard containing beef-fat is due simply to the presence of a larger proportion of tristearin than can be obtained from pure lard by a single crystallisation. It is therefore not proper to recrystallise the first deposit. A pure lard which contained a high proportion (24·9 per cent.) of stearic acid gave a crystallisation which could not be distinguished from that yielded by an ordinary lard mixed with much beef-fat.

'Compound lard' is a mixture of oleostearin from beef-fat with cotton-seed oil; genuine lard is sometimes also added. A simple mechanical process is used in the United States to separate the solid and liquid constituents: 150 grms. are melted, allowed to cool to 24–27° C., and then left for twelve hours more in a moderately warm place.

The solid fat then crystallises out; the contents of the beaker are well stirred, 50 grms. weighed off, wrapped in flannel, and subjected to a slowly increased pressure in a screw-press. The highest attainable pressure is finally applied; the process requires about an hour. The cake of solid fat is then weighed. According to Wainwright (*J.S.C.I.*, 1896, 620), this process is reliable to within 1·5 per cent., but Tennille (*ibid.*, 1897, 363) has obtained results 7–8 per cent. too high or too low.

In addition to the well-known culinary use of lard it is also employed in pharmacy as a base for ointments.

Lard Oil.—When lard is subjected to pressure, it is separated into a fluid portion, lard oil, and a solid portion, lard stearin. According to the temperature at which the lard oil was expressed, it will separate stearin more or less readily on cooling. Lard oil is a pale, almost colourless oil, with the lard odour somewhat accentuated. According to Schweitzer and Lungwitz (*J.S.C.I.*, 1895, 129), the lower grades are deep brown in colour, rancid, and offensive in odour. In consequence of the dark colour the application of the colour reactions for vegetable oils is difficult. The specific gravity should be between 0·913 and 0·919 at 15·5° C. (water at 4° C. =1). Lard oil contains very little free acid.

The iodine value of lard oil varies in accordance with the variable iodine value of lard. The 'congealing point' of the oil is low in proportion as the iodine value is high. Schweitzer and Lungwitz (*loc. cit.*) determine the 'congealing point' by noting the temperature at which the oil becomes cloudy when placed in a 4 oz. bottle immersed in

a freezing mixture of ice and salt and vigorously stirred with a thermometer. The following figures indicate the connection between congealing point and iodine value :

Specific Gravity at $\frac{15{\cdot}5^{\circ}}{4^{\circ}}$	Iodine Value	Congealing Point
0·9136	78·8	25° F.
0·9146	76·4	28° F.
0·9174	76·0	28° F.
0·9151	71·5	35° F.
0·9159	67·8	40° F.
0·9160	63·9	42° F.
0·9186	62·8	Solid at 40° F.

The presence of vegetable oils could be detected by examining the unsaponifiable matter for phytosterol. The ordinary reactions for cotton-seed could be obtained in the usual fine qualities of the oil. Maize, cotton-seed, and marine animal oils would increase the iodine value and specific gravity, and the last-named would be evident to the smell. Rapeseed oil decreases the saponification value and increases the iodine value.

Lard oil is used as a high-class burning and lubricating oil.

Mahwah Butter, Mowrah Seed Oil, and Illipé Nut Oil.—There appears to be considerable uncertainty in regard to the oil to which the last-mentioned name should be applied. According to Crossley and Le Sueur (*J.S.C.I.*, 1898, 991), who examined certain Indian oils of undoubted source, mahwah (or mahua) butter is also known as Illipé oil and is obtained from the seeds of *Bassia latifolia*, while Mowrah seed oil is the produce of *B. longifolia*. Allen, however, states that Illipé oil is obtained from *B. longifolia*.

In the table, constants obtained by Crossley and Le Sueur from nine samples are compared with figures given by Becker (*J.S.C.I.*, 1898, 161) for Illipé oil from the Dutch East Indies, and with the older determinations of De Negri and Fabris, as given by Benedikt. The sample examined by Becker is evidently a different substance from the Indian oils examined by Crossley and Le Sueur.

Illipé oil is employed in candle-making; its use is stated to be steadily increasing.

—	Illipé Oil (Becker)	Mahua Butter = Illipé Oil, *B. latifolia* (Crossley and Le Sueur)	Fat of *Bassia latifolia*	Fat of *Bassia longifolia*
Specific gravity at 100° C.	0·8854	0·8943–0·8981	—	—
Saponification value	194·04	187·4–194·0	199·9	188·4
,, ,, of fatty acids	206·0	—	—	—
Iodine value	29·93	53·4–67·8	60·4	50·1
,, ,, of fatty acids	31·64	—	—	—
Melting point of fat	35·5–36·5° C.	23–29° C.	28–31° C.	42° C.
,, ,, of fatty acids	54·5–55·5° C.	—	—	45° C.
Solidifying point of fat	24·6° C.	—	19–22° C.	36° C.
,, ,, of fatty acids	52·5° C.	—	—	40° C.
Reichart-Meissl value	1·23	0·4– 0·8	—	—
Acid value	34·5	4·83–70·8	—	—

Palm oil is obtained from the fruits of a palm, *Elais guineensis*, which grows on the tropical coasts of West Africa. The nuts are crushed, and then allowed to lie in heaps or pits to ferment, after which they are heated with water, when the oil collects on the surface and is removed. The kernels are separated from the pulp and exported to Europe, where the fat they contain is extracted.

Palm oil is of a lardy consistency, yellow to red in colour, has an odour recalling violets, and, in consequence of the crude method of preparation, contains water, dirt, &c. It may be bleached by heating to a high temperature, or by the action of oxidising agents.

The fermentation process to which the fruits are subjected causes the hydrolysis of a considerable proportion of the glycerides; thus palm oil frequently contains 50 per cent. of free acids, and Lewkowitsch records samples consisting entirely of free acids. Palm oil is used in the manufacture of soap and candles, for which purposes the presence of free acids is not detrimental. For candle-making the value of palm oil varies according to the height of the 'titer' of the fatty acids (p. 47). The percentage of water and non-fatty impurities should also be determined (see pp. 104); these may be considerable in quantity. An estimation of the iodine value of the purified fat may to some extent replace the 'titer' test. Palm oil does not appear to be adulterated by other fats; in any case, the 'titer' of the fatty acids and the iodine absorption determine the value.

Bone fat is obtained by boiling crushed bones with water and removing the oil which rises to the surface, or by extraction with a volatile solvent. When obtained from fresh bones it is yellow in colour, but the fat from old bones is dark and of disagreeable odour. The consistency is about that of butter. The acids of bone fat contain approximately 60 per cent. of oleïc acid, 20 per cent. of palmitic acid, and 20 per cent. of stearic acid.

In addition to glycerides, bone fat may contain calcium phosphate and the lime salts of fatty acids; also water and organic impurities. It yields a relatively high percentage of cholesterol.

The determination of water by drying at 100–110° C. requires a considerable time if lime soaps are present: according to Shukoff and Schestakoff (*J.S.C.I.*, 1898, 383),

more than twenty-four hours' drying may be necessary to attain constant weight. In estimating the dirt and organic impurities, it is to be remembered that the lime soaps are more or less soluble in the ordinary solvents; if the dried fat be extracted by petroleum ether boiling at 85° C., nearly the whole of the lime soap passes into the solution and is therefore calculated as fat by difference. If the bone fat is to be used for candle-making, there is little error, because the fatty acids combined with the lime are available. For soap-making the presence of a large quantity of lime soaps is objectionable. The amount of calcium carbonate in the ash is a measure of the lime soaps; the combining weight of the acids is to be taken as 260 in calculating the result.

In determining the total fat it is necessary to remove the lime; Shukoff and Schestakoff (*loc. cit.*) warm 10 grms. of the fat with 3–5 drops of strong hydrochloric acid, and shake at frequent intervals during one hour; the fat is then extracted by petroleum ether, the solvent evaporated, and the residue weighed. The dirt may be determined in the same portion, and the water then found by difference, unless lime soaps are present.

The 'titer' of the fatty acids of bone fat intended for candle-making is determined in the usual manner (p. 47).

Bone fat is used in the candle industry and in the manufacture of inferior soaps.

Tallow.—The fat of oxen and sheep is known as beef or mutton tallow, but no doubt the fat of other animals is often included in the commercial products. Beef tallow is somewhat yellower than mutton tallow, melts at a lower temperature, and has a slightly higher iodine value.

The oleomargarine, which is used in making butter substitutes, is obtained by melting beef tallow and straining off impurities, when the product is known as *premier jus*; it is then cooled to about 35° C., and pressed. The liquid portion constitutes oleomargarine, whilst the solid is known as pressed tallow or tallow stearin. Tallow oil is another product obtained by expressing the more fluid portions of tallow at a lower temperature.

The quality of tallow is determined by means of the 'titer' test (p. 48). According to Lewkowitsch (*Chemical Analysis of Oils, Fats, and Waxes*), the 'titer' of tallow for use in the candle industry should not be below 44° C. Smetham recommends for general use, in evaluating tallow for the soap and candle manufactures, the determination of the iodine value; low iodine values correspond to the high 'titers.' The iodine value, which does not necessitate the isolation of a large quantity of fatty acids, is much more easily determined. Smetham (*J.S.C.I.*, 1899, 330) gives the iodine values of about 1,000 samples of commercial tallows examined by him; 5 samples had values below 36; 47 between 36 and 40; 750 between 40 and 45; 180 between 45 and 50; and 21 between 50 and 57. Most of these samples were accepted as 'commercially pure.' The average iodine value of 592 samples of English tallow was 42·81.

In order to eliminate the personal equation in performing the 'titer' test, Shukoff (*J.S.C.I.*, 1899, 406) employs a beaker-shaped vessel 3 cm. wide, narrowed towards the top and fused into a larger glass vessel 5 cm. wide and 10 cm. high; the space between is exhausted of air to a very high vacuum. The principle of Dewar's tubes is

thus employed to prevent the escape of heat. Thirty to forty grms. of the melted fatty acids are poured into the apparatus, a cork carrying a delicate thermometer is inserted, and when the temperature has sunk to within 3° of the expected solidifying point the apparatus is shaken steadily until the contents become decidedly turbid. The temperature is then recorded.

Tallow might be adulterated by other solid fats, such as cotton-seed stearin, cocoanut-oil, and palm-kernel oil. The first-named can be detected by Halphen's reaction and the increased iodine value; the two latter by the increased saponification value and the occurrence of a Reichert-Meissl value.

Tallow is directly made into candles; a much larger quantity is saponified (p. 9), the solid fatty acids separated, and then used in the candle manufacture. It is largely used in soap-making, and also as a lubricant for slow-running heavy machinery.

Tallow oil is the more fluid portion of tallow (see p. 206). It is generally a pale yellow oil, with a decided odour of tallow; it more or less rapidly deposits 'stearin,' or turns semi-solid, on standing at the ordinary temperature. Tallow oil has a much higher iodine value than tallow (see table, p. 102); it is liable to adulteration by vegetable oils, of which rape oil would be detected by the low saponification value and cotton-seed oil by the colour reactions (p. 181) and the high iodine value. The presence of a vegetable oil would be proved with certainty if phytosterol were found in the unsaponifiable matter. Tallow oil is employed as a lubricant in admixture with mineral oils.

Cocoa or cacao butter is not to be confounded with cocoanut oil; it is produced in the manufacture of cocoa from the seeds of *Theobroma cacao* by grinding the nibs (husked seeds) and pressing the resultant paste in a heated press.

Cacao butter is a hard yellow fat, which is bleached by exposure to light and does not readily turn rancid; it has an odour of cocoa and an agreeable taste. It contains chiefly stearic, palmitic, and oleïc acids. Klimont (*J.S.C.I.*, 1901, 1121; 1902, 486) points out that the melting-point (26–33° C.) is much lower than corresponds to a mixture of the triglycerides of stearic, palmitic, and oleïc acids in the proper proportions. By crystallising cacao butter from acetone, Klimont obtained three fractions; the first contained tristearin and tripalmitin; it was free from arachidic acid, which has been stated to occur. The second fraction was a mixed glyceride of stearic, palmitic, and oleïc acids, which is to be regarded as the main constituent of the fat. The third fraction was probably a mixed glyceride of myristic, palmitic, and oleïc acids.

Cacao butter is practically free from volatile acids: Lewkowitsch obtained Reichert-Meissl values of 0·2–0·9; the Reichert value of 1·6 obtained by Allen must be erroneous (*J.S.C.I.*, 1899, 556). The acid value should be low.

According to the Pharmacopœia, cacao butter softens at 26·6° C. and melts at 31·1–33·9° C., and a solution obtained by dissolving 1 grm. in 3 c.c. of ether at 17° C. should not become turbid, or give a deposit in less than three minutes, when placed in ice at 0° C. Lewkowitsch (*loc. cit.*) recommends a somewhat different form of this test; the fat is shaken with twice its weight of ether in a

closed tube at 18° C., when a clear solution should be formed. In the presence of 10 per cent. of tallow, complete solution is obtained, but more slowly. On cooling at 0° C., genuine cacao butter deposits tufts of crystals, while if 5 per cent. of tallow be present flocks separate in the liquid.

Cacao butter may be adulterated by tallow, stearic acid, cocoanut oil, palm-kernel oil, beeswax, Japan wax, and paraffin wax. Beeswax and paraffin wax would be recognised by means of the unsaponifiable matter. Palm-kernel and cocoanut oils give rise to high saponification and Reichert-Meissl values. Stearic acid would be readily detected by a high acid value and high 'titer' test. Tallow may be found by means of the test given above; considerable quantities would raise the melting-point, give a lower 'titer' of the fatty acids, and increase the iodine value. The presence of small quantities of tallow is difficult to prove; Lewkowitsch (*loc. cit.*) for this purpose examined the unsaponifiable matter, which should consist entirely of phytosterol (see p. 118). The presence of Japan wax (which see) would also not be easy to detect; it would give a low iodine value and a high proportion of soluble acids.

A 'vegetable butter,' proposed as a substitute for cacao butter, examined by Possetto (*J.S.C.I.*, 1902, 55), appeared to be a mixture of 70–75 per cent. of cocoanut oil and 25–30 per cent of Japan wax. It was partially soluble in hot alcohol; the solution on cooling deposited a semi-crystalline precipitate which had almost the same constants as Japan wax.

Cacao butter is employed in pharmacy.

Vegetable or Chinese tallow is obtained in China from the seeds of *Croton sebiferum* Linn., which are

enveloped by a coating of the fat. It is a hard white or greenish-white substance without smell or taste ; it contains palmitin and olein. De Negri and Sburlati state that the commercial tallow often contains linseed oil (*J.S.C.I.*, 1897, 339).

Vegetable tallow is used in the soap and candle industries, for which purposes it is valued by means of the 'titer' test.

Butter-Fat.—In addition to the actual fat, butter contains water, salt, and other preservatives, casein, milk sugar, colouring matters, lecithin, cholesterol, and phytosterol. According to Browne (p. 161), butter-fat (i.e. butter freed from water, salt, &c.) contains dihydroxystearin, 1·04 ; olein, 33·95 ; stearin, 1·91 ; palmitin, 40·51 ; myristin, 10·44 ; laurin, 2·73 ; caprin, 0·34 ; caprylin, 0·53 ; caproïn, 2·32 ; and butyrin, 6·23. Apart from the fact that acetic, arachidic, and linolenic acids have also been found in butter, Browne's results can only be taken as showing the proportions in which the different acids are present, for a mixture of artificial triglycerides in the above proportions solidifies at a considerably higher temperature than butter itself (Partheil and von Velsen, *J.S.C.I.*, 1900, 919). This fact points to the occurrence of mixed glycerides, of which a butyro-palmito-oleate has actually been isolated.

The analysis of butter may be divided into three sections : the examination of the non-fatty constituents, a preliminary test, and finally the determination of analytical constants.

Non-fatty Constituents.—The water is determined by drying at 100° C. (p. 104), and the ether-insoluble matters by extracting the dried butter with ether. The difference

then gives the percentage of butter-fat. The limit for the permissible amount of water in butter has been fixed in this country at 16 per cent.

In order to detect artificial colouring matters, which are generally harmless, the butter is gently warmed with alcohol (90 per cent.) and the mixture shaken. The natural colouring matter is insoluble in alcohol, but artificial colouring matters dissolve and give a yellow solution.

If required, the amount of common salt in the residue insoluble in ether may be determined in the ordinary manner by means of silver nitrate.

Other preservatives present (boracic acid, glucose) may also be sought in the aqueous extract of the ether-insoluble residue, or of the original butter.

Preliminary Examination.—For the purpose of a preliminary sorting of samples into 'genuine' and 'doubtful,' either the specific gravity or the refractive index may be utilised. The specific gravity of butter-fat at 100° C., compared with water at the same temperature, is not lower than 0·910 ; oleomargarine (see p. 206) has a specific gravity under the same conditions of 0·901–0·906. Since, however, the specific gravity of butter-fat may be as high as 0·913 , it is easy to make mixtures containing oleomargarine, the specific gravities of which fall well above the lower limit for pure butter. According to Allen, $D\frac{99^\circ}{15^\circ}$ is for butter-fat 0·867–0·870 and for oleomargarine 0·8585–0·8625.

The evidence furnished by the refractive index is much more reliable, and the butyro-refractometer (p. 58) is widely employed for testing butter-fat. According to

Wollny, the refractive index of butter-fat at 25° C. is 1·4590–1·4620, and of oleomargarine 1·4650–1·4700. The corresponding scale readings on the butyro-refractometer are 49·5–54° and 58·6–66·4°. Thus samples which give a reading above 54° will always be found adulterated when subjected to analysis. Wollny places the limit for undoubtedly pure butter-fat still lower—viz. 52·5°—regarding those butters which give readings between 52·5° and 54° as suspicious. The readings obtained at other temperatures than 25° C. may be reduced to that temperature by means of the correction given on p. 59. Attention is also directed to the fact (previously indicated) that the dividing line in the case of pure butter fat is colourless, while that obtained from oleomargarine has a blue fringe; these appearances may not, however, be taken as absolute criteria of purity or adulteration.

Chemical Examination.—The Reichert-Meissl and Hehner values give the most important results; the saponification and iodine values may also be determined.

The Reichert-Meissl value of pure butter-fat varies somewhat according to the season (lower in summer than winter) and the food of the cows, but more especially according to the time which has elapsed since calving. Differences due to the latter cause should almost disappear when the butter of a whole dairy and not of a single cow is under consideration. Browne gives the following limits for the properties of pure butter-fat (*J.S.C.I.*, 1899, 780):

—	General Limits	Extreme Limits
Reichert-Meissl value . .	20–33	11·2–41
Saponification value . .	220–236	216–245
Iodine value	26–38	19·5–49·6

Butter-fat giving a Reichert-Meissl value below 24 is, however, exceptional, and is to be suspected of adulteration. Values below 26 are indeed not frequently obtained.

The Hehner and saponification values naturally vary in accordance with the Reichert-Meissl value. The normal Hehner value is about 87–88. In the determination, the insoluble acids must be very thoroughly washed.

While the usual adulterant for butter is oleomargarine (p. 206), cotton-seed, sesamé, and cocoanut oils may also be found. The two former may be detected by the colour reactions; artificial colouring matters are removed as described on p. 178. Butter from cows fed on cotton-seed cake may give slight Becchi and Halphen reactions. Cocoanut oil might be mixed with oleomargarine in proportions to give the proper Hehner value (see p. 102), but the adulteration would be detected by the Reichert-Meissl value.

The detection of oleomargarine in butter, by means of the microscopic examination of crystals deposited from solutions, is more difficult than in the case of lard. In Cochran's method (p. 200) only 8 c.c. of fusel oil are used to 2 c.c. of the butter fat.

Palm-kernel Oil.—Palm-kernels (see p. 203) contain 46–52 per cent. of oil (Nördlinger); they are imported to Europe and crushed.

Palm-kernel oil is a soft white or yellowish fat with a pleasant odour. It contains lower fatty acids, and thus resembles cocoanut oil in composition and properties. It is used in soap-making.

Japan wax is obtained in Japan from the berries of several species of *Rhus*, chiefly from *R. succedanea*, by hot-pressing. The crude wax is a greenish tallow; it is

bleached by converting into flakes, exposing to light, and frequently sprinkling with water (*J.S.C.I.*, 1900, 832). Japan wax is not a true wax; it is composed of glycerides, just as are the fats already considered. The bleached wax as it is imported into this country is in smooth hard cakes, which break with a shining fracture.

According to Geitel and van der Want (*J.S.C.I.*, 1900, 356), Japan wax contains palmitic, oleïc, and japanic acids. Japanic acid is a saturated dibasic acid, $C_{20}H_{40}(CO_2H)_2$, which melts at 118° C.; it is probably present in the wax in the form of a mixed glyceride with palmitic acid. Two samples contained 90·62 and 90·66 per cent. of insoluble acids, and 5·96 and 4·66 per cent. of soluble acids. The latter were of the consistency of lard, and had a mean molecular weight of 162; they are regarded by Geitel and van der Want as products of the oxidising agents employed to bleach the wax.

The bleached wax is stated to melt at 42–55° C. (*J.S.C.I.*, 1900, 832); four samples examined by Bernheimer and Schiff (*J.S.C.I.*, 1902, 56) melted at 52·6–53·2° C. The iodine value of Japan wax is given by Hübl as 4·2; Geitel and van der Want found 8·3–8·5; Bernheimer and Schiff, 10·56–11·3. A sample prepared by Ahrens and Hett (*J.S.C.I.*, 1901, 909) had the iodine value 11·9–12·8, which after bleaching in the sun fell to 7·6. The saponification value has also been found to vary; the following are recent figures: 220–222·1 (Bernheimer and Schiff), 206·6–212·0 (Ahrens and Hett, unbleached), 208·1 (Ahrens and Hett, bleached), 217·5–237·5 (Geitel and van der Want). The differences may be due to bleaching by oxidising agents rather than by exposure to light.

Japan wax is apparently not adulterated ; only paraffin wax, palm-kernel oil, and tallow would be available for the purpose. The detection of paraffin wax and tallow (by the iodine value and 'titer' test) would be simple.

Myrtle wax is obtained from the berries of certain species of *Myrtica*, mainly in America and at the Cape. It is mainly composed of the glycerides of palmitic, stearic, and myristic acids, and thus is a wax only in name, not in composition. The principal interest of myrtle wax in this country appears to be that of a possible adulterant of beeswax.

Cocoanut oil is obtained from the dried kernels (known as coprah) of the cocoanut, which is the fruit of a palm, *Cocos nucifera*. It is a soft white fat of pleasant odour and taste, but readily turns rancid.

Cocoanut oil differs from other vegetable fats, except palm-kernel, croton, and maize oils, by the large proportion of volatile acids it contains. The insoluble acids of the oil are also of comparatively low molecular weight ; thus the saponification value is higher than that of any other fat. According to Ulzer (*J.S.C.I.*, 1899, 1133), cocoanut oil contains 2·32 per cent. of the triglycerides of volatile acids (mainly caproïc and caprylic), about 10·45 per cent. of olein, and the remainder is laurin, myristin, and caprin. Tripalmitin is probably not contained in the fat.

The high saponification and Reichert-Meissl values and the low iodine and Hehner values distinguish cocoanut oil from other fats except palm-kernel oil, the detection of which would be difficult. Cocoanut oil is soluble in 2 vols. of absolute alcohol at 30–31° C. (Benedikt).

Cocoanut oil is used in soap-making ; the soaps are

soluble in dilute salt solutions, and hence lather with sea-water. It is also used in the candle-manufacture and in the preparation of butter substitutes.

MARINE ANIMAL OILS

The oils included in this class are obtained from a variety of sources: from fish, the livers of fish, and the fat of marine mammals. They all possess the disagreeable fishy odour, have high iodine values but do not dry like the drying oils, and give high Maumené tests. The constitution of the unsaturated acids is not understood; it must differ from that of the unsaturated acids in the fats of land animals and vegetables. The marine animal oils give considerable precipitates with bromine (see p. 140).

The results obtained by Bull (*J.S.C.I.*, 1900, 73, 176) show that the marine animal oils contain, in addition to unsaturated acids of the oleïc, linolic, and linolenic series, also still less saturated acids to the series $C_nH_{2n-8}O_2$. These acids are separated by means of the solubility of their sodium salts in ether; 7 grms. of the oil are boiled with 25 c.c. of a solution of sodium ethylate (23 grms. of sodium in 1 litre of absolute alcohol). The dry soap is broken up with a spatula, treated with 144 c.c. of ether (anhydrous and free from alcohol), the flask corked, shaken at intervals during thirty minutes, and the contents then filtered. An aliquot part of the filtrate is measured off and shaken with three successive quantities of 25 c.c. of water made just alkaline to phenolphthaleïn. The free acids are then liberated from the aqueous solution, extracted by ether, and the solution evaporated, avoiding

oxidation by concluding the evaporation in a tared round-bottomed flask connected to the filter-pump. The high iodine values of the acids thus obtained point to the presence of acids less saturated than linolenic acid. The figures in the following table refer to the more important oils examined by this method:

—	Acid Value	Iodine Value	Highly Unsaturated Acids		
			Percentage of	Acid Value	Iodine Value
Cod-liver oil, crude Japanese	5·68	140·8	11·61	182·8	341·8
,, ,, white, Lofoden	13·0	144·6	17·2-17·7	202·2	314·0
,, ,, brown, Hamburg	31·9	147·9	13·04	193·7	319·5
Sardine oil, clear, Japanese	2·2	134·1	12·12	188·2	285·8
Arctic whale oil, refined	1·9	117·4	7·19	206·3	300·3
,, ,, crude white	2·5	127·4	8·83	201·4	275·5
Porpoise body oil	0·75	126·9	14·3	313·2	285·4
Newfoundland seal oil	0·77	143·4	11·96	182·8	330·3

In all probability these highly unsaturated acids are the cause of the large precipitates the oils give with bromine; a method for the analysis of marine animal oils might be based on these properties.

The great variations found for the analytical 'constants' of many of these oils are no doubt due to several causes: alterations in the material before the oil is extracted; crude methods of extraction; the oils obtained from different parts of the body differ greatly in composition; the oils are produced by different species.

Menhaden oil is extracted in large quantities from the menhaden, a fish peculiar to the western coast of North America. It is a brown oil, which is stated to dry rapidly. It is employed in currying, and for adulterating linseed oil.

Sardine oil is extracted from sardines by boiling with

water. A large quantity is made in Japan. Very varying and conflicting results have been obtained in the examination of this oil. It is used in the leather industries.

Cod-liver Oil.—The medicinal oil is obtained from the perfectly fresh livers of the common cod, *Morrhua vulgaris*, by the application of a moderate heat and pressure; the oil is then cooled below 0° C. and the 'stearin' filtered off. The residual oil, obtained by means of higher temperatures and pressures, is used industrially under the name of 'cod oil,' under which term is also included the oil extracted from the mixture of livers of the cod and other fish caught by the larger fishing vessels.

Cod-liver oil contains the glycerides of stearic, palmitic, and certain unsaturated acids, the nature of which is not definitely known, but, according to Bull, they probably contain members of all the series down to $C_nH_{2n-8}O_2$. It contains cholesterol and small, variable quantities of iodine.

Cod-liver oil is a pale yellow oil, with a slight fishy taste; there should be no rancid taste, and free acids should be almost absent. According to the Pharmacopœia, the specific gravity is 0·920–0·930, strong sulphuric acid gives a violet colouration with a drop of the oil on white porcelain, and when strong nitric acid is carefully poured into the oil a precipitate of coagulated albumin is seen at the surface of contact. No 'stearin' should separate when the oil is cooled at 0° C. for two hours.

The iodine value of cod-liver oil should be high: Harvey (*J.S.C.I.*, 1902, 694) gives 160–177 as the values obtained for thirteen samples by Wijs's method. Additions of vegetable oils, except linseed oil, would give materially

lower values; vegetable oils could also be detected by examining the unsaponifiable matter for phytosterol.

According to Dowzard (*J.S.C.I.*, 1898, 696), the presence of seal oil can be detected by means of the refractometer. In Amagat and Jean's oleorefractometer pure cod-liver oil gives readings of 43·5–45° at 22° C., while pale seal oil gives 32–32·5°. The oils must first be shaken with alcohol of 0·800 specific gravity, the mixture heated to 30° C., allowed to separate, and the oil finally dried at 110° C.

By the action of bromine on cod-liver oil, Hehner and Mitchell (see p. 140) obtained 42·9 per cent. of precipitate, whilst the other marine animal oils examined yielded much smaller quantities. Walker and Warburton (*J.S.C.I.*, 1902, 1144), however, only obtained 33·76–35·33 per cent. of insoluble compound; the fatty acids yielded 29·86–30·36 of insoluble hexabromide. These results are worthy of consideration, but it does not yet appear possible to base upon them a method for detecting the presence of individual marine animal oils in mixtures of two.

Cod oil is a dark brown oil with a strong fishy odour; it is used in tanning and currying.

Seal oil is obtained from the blubber of a large number of species of seal. The best quality is almost white and has comparatively little odour; lower qualities are brown in colour and unpleasant of smell.

A sample of Caspian seal oil was found by Ljubarsky (*J.S.C.I.*, 1898, 358) to contain palmitic (17 per cent.), oleïc, and physetoleïc acids. Walker and Warburton (*vide supra*) obtained 27·5–27·9 per cent. of the insoluble compound from the oil and 19·8–19·9 per cent. of the

hexabromide from the fatty acids. The presence of a less saturated acid than physetoleïc and oleïc acids is therefore proved (see also p. 217). The detection of seal oil in cod-liver oil has just been mentioned.

White seal oil is employed for burning, the lower qualities in tanning and currying.

Whale oil is extracted from the blubber of many species of whale. It is a yellow or brown oil of fishy odour.

Whale oil contains palmitic, stearic, oleïc, and physetoleïc acids, also volatile acids in varying amounts, and less saturated acids. Hehner and Mitchell obtained 25 per cent. of the insoluble bromine compound from the oil; Walker and Warburton (*vide supra*) 15·5–16·1 per cent. from the oil and 12·4 per cent. of the insoluble hexabromide from the acids (also see p. 217).

According to Schweitzer and Lungwitz (*J.S.C.I.*, 1895, 130), the characteristics of pure refined whale oil are as follows: the oil is clear and pale yellow in colour, the specific gravity is 0·921–0·923 at 15·5° C., the iodine value is 120–130, the saponification value 190–200. The fatty acids melt at 20° C. or below, in a capillary tube; they remain perfectly clear at the ordinary temperature, and are unaltered in colour by silver nitrate (Becchi's reaction). Whale oil is used in the leather industries; the refined oil is also employed for burning and lubrication.

Porpoise oil and **dolphin oil** are of little importance. They are fluid oils, containing a considerable proportion of volatile acids (mainly valeric acid), and hence giving high Reichert-Meissl values.

LIQUID WAXES

The oils obtained from the sperm and bottlenose whales are entirely different in character from the oils hitherto considered; they consist mainly of the esters of monovalent unsaturated alcohols with unsaturated acids, and thus resemble the solid waxes in composition.

Sperm Oil.—The oil contained in a large cavity in the head of the sperm whale, *Physeter macrocephalus*, which lives in tropical seas, separates on cooling the solid crystalline wax known as spermaceti. The residual liquid oil is sperm oil.

Sperm oil is a pale limpid oil of slight fishy odour, its specific gravity is much lower than that of any other fatty oil, except bottlenose oil. Sperm oil is composed, as stated above, of the esters of saturated and unsaturated acids, which latter are probably oleïc and physetoleïc, with monatomic alcohols, which, according to Lewkowitsch (*Chemical Analysis of Oils, Fats, and Waxes*), are unsaturated. It also contains small quantities of volatile acids and probably of less saturated acids, since Walker and Warburton (*J.S.C.I.*, 1902, 1144) have obtained slight precipitates with bromine: 2·4–3·7 per cent. from the oil and 2 per cent. of hexabromide from the fatty acids. The alcohols of sperm oil appear in analysis as unsaponifiable matter, of which 39–41 per cent. is present; it is readily soluble in cold alcohol (Allen).

The low specific gravity, low saponification value (126–130), and high proportion of unsaponifiable matter at once distinguish sperm oil (and bottlenose oil) from all other oils. A mixture of mineral oil and vegetable or

animal oil might, of course, give the correct saponification value of sperm oil, but its specific gravity could not well be lower than 0·892 unless such a mineral oil were used, that it could be readily detected by its low flash point. If such a mixture were added to sperm oil, the amount of the unsaponifiable matter and its nature (see p. 114) would at once indicate the extent and character of the adulteration.

A determination of the glycerin (p. 156) in adulterated sperm oil gives an estimate of the amount of the saponifiable oil added, since sperm oil only contains traces of glycerin.

Sperm oil is used as a burning oil, and is a valuable lubricant for light, quick-running machinery.

Bottlenose oil, Arctic sperm oil, is obtained in the same manner as sperm oil from the bottlenose whale, which inhabits the North Atlantic Ocean. It resembles sperm oil in all respects, and is used for the same purposes.

SOLID WAXES

The solid waxes are of animal or vegetable origin. They are composed mainly of the esters of the higher saturated alcohols with saturated acids, but contain also both free acids and free alcohols, and unsaturated compounds, which give rise to small iodine values.

Spermaceti, as stated above, is the solid which separates from the oil of the sperm or bottlenose whale. It is a white shining, translucent mass of crystalline structure, which dissolves in hot alcohol and crystallises from the solution on cooling.

Spermaceti is mainly composed of cetyl palmitate; it

contains in addition small quantities of octadekyl alcohol and of other saturated acids, also apparently of free alcohols and soluble acids.

Spermaceti may be adulterated by stearic acid, tallow, and possibly palm-kernel or cocoanut oils. The presence of stearic acid would be at once indicated by the acid value, since spermaceti is practically free from uncombined acids. According to Hirschsohn (*J.S.C.I.*, 1897, 639), stearic acid is readily detected by dissolving 1 grm. of the spermaceti in petroleum ether and shaking the solution, which should be clear, with a 0·1 per cent. aqueous solution of copper acetate. The ether layer becomes green in the presence of only 2 per cent. of stearic acid. Stearic acid is detected, according to the Pharmacopœia, by dissolving in hot 90 per cent. alcohol, cooling, filtering, and adding water, when no flocculent precipitate should be formed. An addition of tallow, palm-kernel, or cocoanut oils would raise the saponification and iodine values. Spermaceti is employed in pharmacy and perfumery. An examination of twelve samples by Kleber (*J.S.C.I.*, 1898, 383) gave the following range of properties: melting-point, 43–45° C.; solidifying point, 42–44·5° C.; acid value, 0·09–0·47; saponification value, 124·8–136·3.

Beeswax.—Bees secrete a wax from which they build up the combs. After the honey has been removed, the combs are melted in water, and then yield the wax in mass. Beeswax is usually yellow or brownish-yellow in colour, with an odour of honey, and somewhat brittle at the ordinary temperature, so that it breaks with a coarse granular fracture. It softens under the heat of the hand. When exposed to light it is bleached (see below).

Beeswax is generally stated to consist mainly of free cerotic acid and myricyl palmitate ; the free acids, however, contain only about 60 per cent. of cerotic acid, the remainder being melissic acid and other homologues (Marie, *J.S.C.I.*, 1895, 591). There are also present ceryl alcohol, and 12–17 per cent. of hydrocarbons (Ahrens and Hett, *J.S.C.I.*, 1899, 591). The acetyl value recorded by Lewkowitsch points to the presence of about 10 per cent. of free alcohols.

Beeswax dissolves entirely in cold chloroform ; hot alcohol takes up the free acids and part of the esters, but on cooling separates them almost entirely. In other solvents—ether, benzene, turpentine, &c.—beeswax dissolves entirely when hot, but separates more or less completely on cooling. It melts to a clear liquid at about 64° C.

The examination of beeswax for purity conveniently commences by quantitative determinations, of which the acid and saponification values are the most important. The two values are determined on the same sample by Hübl's method : 3–5 grms. of the dried wax are heated with neutralised absolute alcohol in a flask on the water-bath ; phenolphthaleïn is added, and the free acid titrated by semi-normal alcoholic potash ; 25 c.c. of the same potash solution are then added, and the mixture boiled vigorously under an inverted condenser for one hour, when the excess of potash is titrated. Beeswax does not saponify readily, and erroneous results may easily be obtained. Büchner states that the chief source of error is the use of weak alcohol ; the potash solution should be made from absolute alcohol. According to Benedikt (*Analyse der Fette*), it is necessary to boil down almost to dryness on

the water-bath, and even then correct results cannot be obtained if paraffin or ceresin be present. The saponification may be done under pressure in a closed bottle (see p. 76).

Henriques introduced a *cold saponification* method, which is stated to give more reliable results (*J.S.C.I.*, 1896, 476; 1898, 805), particularly in the presence of ceresin and paraffin. Three grms. of the wax are dissolved in 25 c.c. of hot petroleum ether, the free acid titrated by semi-normal alcoholic soda, 25 c.c. more of the alcoholic soda are added, the liquid is shaken and allowed to stand for twenty-four hours, when the excess of alkali is titrated.

As regards the results of these determinations: the acid value of beeswax is about 20, the ester value is 73–76. Hübl found that a high acid value is generally accompanied by a high ester value, a low acid value by a low ester value; consequently, the ratio of the two values is more nearly constant than either. The ratio of the ester value to the acid value for pure beeswax lies between 3·6 and 4, but is more usually 3·65–3·8. A ratio greater than 4 renders probable the presence of tallow, lard, carnaüba wax, or Japan wax. A ratio lower than 3·6 indicates the addition of rosin or stearic acid. A correct ratio together with a low saponification value indicates the addition of unsaponifiable substances—paraffin or ceresin. As regards the extreme limits of the values given by pure beeswax, Büchner considers that a wax may be regarded as pure if the qualitative tests give negative results and the values fall within the following limits: acid value, 17·5–21; ester value, 70–78; saponification value, 87·5–99 (*J.S.C.I.*, 1901,

286). Waxes from India, China, and other countries have given values far outside these limits.

It will easily be seen that it is possible to combine acid substances (rosin, stearic acid) with paraffin and Japan wax or tallow in proportions such that the mixture has the correct acid and ester values, and therefore the correct ratio. To meet such cases Büchner (*J.S.C.I.* 1895, 1070) has devised a method based on the small solubility of cerotic acid in alcohol. Five grms. of the sample are placed in a flask with 100 c.c. of alcohol of 80 per cent. strength. The flask is weighed, boiled for five minutes with frequent shaking, then cooled, and alcohol added to bring the flask up to the original weight. The solution is filtered through a folded filter, and 50 c.c. of the filtrate are titrated with deci-normal alcoholic potash, using phenolphthaleïn as indicator. The figures given in the following table show results obtained for possible adulterants of beeswax, and certain mixtures of these substances made to give normal acid and saponification values:—

Substance	Acid Number
Beeswax, yellow	3·6–3·9
,, white	3·7–4·1
Palm wax	1·7–1·8
Carnaüba wax	0·76 0·87
Japan wax	14·93–15·3
Tallow stearin	1·1
Rosin	150·3
Stearic acid	65·8
I Mixture of stearic acid, stearin, and paraffin	21·4
II. ,, ,, Japan wax, and paraffin	17·8
III. ,, rosin, stearin, and paraffin	22·0
Pure beeswax *plus* 25 per cent. of mixture I.	8·42
,, ,, 50 ,, ,,	11·3

In this manner it may be possible to estimate roughly the proportion of such a mixture present.

The price of beeswax is much higher than that of most of the adulterants, so that a small addition would be profitable. Methods for detecting small quantities should therefore be applied. Resin may be detected by the Liebermann-Storch reaction (p. 124), and woolwax by the reactions for cholesterol (p. 34) and its partial insolubility in chloroform. To test for stearic acid, boil 3 grms. of the wax with 10 c.c. of 80 per cent. alcohol, immerse the tube in cold water, shake until a thick paste is formed, and let stand for one hour. Then filter and add to the filtrate an alcoholic solution of lead acetate (the addition of water in place of this solution gives a less delicate test). Pure waxes give only a faint opalescence ; a wax is to be regarded as pure if it gives no deposit of stearic acid after one to two hours (Büchner, *J.S.C.I.*, 1901, 286).

Glycerides (Japan wax, tallow) may be estimated by determining the glycerin (p. 156) ; their presence may be detected by evaporating the alcohol from the liquid left after determining the saponification value, adding water, filtering, evaporating, and heating with acid potassium sulphate, when the odour of acrolein is perceived (Büchner).

Small quantities of paraffin wax or ceresin may be detected by the following method, due to Weinwurm, based on the solubility of the normal unsaponifiable matter in glycerin (*J.S.C.I.*, 1897, 939) : saponify 5 grms. of the wax by boiling with 25 c.c. of semi-normal alcoholic potash, evaporate off the alcohol, add 20 c.c. of glycerin, and continue to heat on the water-bath for some time after a clear liquid is obtained, then add 100 c.c. of boiling water. The liquid obtained from pure beeswax remains more or less clear, so that print placed beneath the flask

can be read through it, which is no longer possible when 5 per cent. of ceresin or rosin is present. A second determination is now made, adding to the wax 5 per cent. of ceresin ; if a slightly turbid solution is obtained no ceresin was present originally, but if there is a marked precipitate about 3 per cent. was present. If in the first determination a turbid solution was obtained, a second experiment is made adding 3 per cent. of ceresin ; if the substance causing the first turbidity was ceresin there is now a precipitate; but if not, still only a turbidity. Ceresin is used in nearly all wax substitutes.

The presence of ceresin gives a lower specific gravity, stearic acid and rosin give higher acid values, and the latter can be extracted by cold 70 per cent. alcohol. Tallow gives a higher iodine value and lowers the melting-point. Carnaüba wax raises the melting-point and specific gravity.

Bleached beeswax differs somewhat in properties from the original wax. Beeswax is bleached either by exposing it in a thin ribbon to the action of light, generally after incorporating with it about 5 per cent. of tallow or turpentine, or by means of chemical agents.

Bleached wax (white wax) is white or very pale yellow, is more brittle than yellow wax, and gives a finer fracture. The iodine value is almost invariably reduced. According to Büchner (*J.S.C.I.*, 1901, 286) and Berg (*ibid.*, 1902, 1032), the effect of bleaching by chemical means is to diminish the ratio of the ester value to the acid value to such an extent as to make the wax appear adulterated. This is due to a considerable increase in the acid value ; the saponification value is also generally raised.

Beeswax is used for candles, in pharmacy, and in the manufacture of polishes.

Carnaüba wax is an excretion of the leaves of a palm, *Corypta cerifera.* It comes into commerce as hard greenish-yellow lumps, which are very brittle and can be rubbed to powder between the fingers. It has a high melting-point, and is even less soluble than beeswax in cold solvents.

Carnaüba wax is mainly composed of myricyl cerotate; it also contains free ceryl and myricyl alcohols, which must be present in considerable quantity judging by the large acetyl value (p. 144) found by Lewkowitsch.

Carnaüba wax is employed in the manufacture of candles and polishes.

Woolwax.—The fat, which is extracted from natural wool by solvents, consists essentially of esters of higher alcohols; other constituents are free acids, free alcohols, and potassium salts of fatty acids. The wool-fat, which is removed from wool by washing, contains also the fatty acids of the soaps used for scouring the wool. Lewkowitsch therefore proposes to term the neutral portion of wool-fat—consisting of free alcohols and esters—'woolwax,' in order to show that it resembles the other waxes in constitution (*J.S.C.I.*, 1896, 14). Pure woolwax is now made on a large scale. Its composition is still a matter of research; it contains cholesterol, lanolinic alcohol, and other unsaturated alcohols; the fatty acids appear to contain hydroxy-acids.

Pure woolwax (*lanolinum anhydricum, adeps lanæ*) is an almost white butter, has a slight pleasant odour, and can be mixed with considerable quantities of water. It should

be free from water and practically free from free fatty acids and ash. According to Lifschütz (*J.S.C.I.*, 1898, 587), it possesses the following characters: it does not darken on heating; when 0·5 grm. is boiled with 5 c.c. of glacial acetic acid, the cooled and filtered solution must not have more than a brownish-yellow tinge and must give no green colouration when 4–5 drops of strong sulphuric acid are added; 2 grms. must give an alkaline reaction to phenolphthaleïn with 1–2 drops of deci-normal caustic potash; it must leave no ash and must be free from chlorine, which is tested by boiling with absolute alcohol and a drop of nitric acid, filtering, and adding alcoholic silver nitrate. Other tests given in the Pharmacopœia state that 1 grm. should dissolve almost completely in 75 c.c. of boiling 90 per cent. alcohol, and the greater part should separate in flocks on cooling; also that the chloroform solution poured over strong sulphuric acid turns purple-red.

Woolwax is employed in pharmacy as a base for ointments.

CHAPTER VII

THE EXAMINATION OF CERTAIN COMMERCIAL PRODUCTS

Turkey-red oil is obtained by treating castor oil with strong sulphuric acid, and neutralising with ammonia or soda, with or without previous washing with brine. Turkey-red oil consists essentially of ricinoleïc acid and sulpho-ricinoleïc acid, $C_{17}H_{32}(CO.OH)O.SO_2.OH$, which is soluble in water but insoluble in salt solutions; it is decomposed by heating with acids into sulphuric and ricinoleïc acids.

Qualitative Examination.—The oil must be entirely soluble in ammonia and contain no iron. To detect iron, mix 10 c.c. with a mixture of equal parts of strong sulphuric acid and water, add a little potassium ferro-cyanide, shake, add 50 c.c. of ether, and shake again. A blue ring at the junction of the liquids denotes the presence of iron.

Total fat and total sulphuric acid. Boil 4 grms. of the oil with 30 c.c. of dilute hydrochloric acid for forty minutes under an inverted condenser (Herbig, *J.S.C.I.*, 1902, 365). Transfer to a separating funnel, and wash the flask out with water and ether, adding the washings to the contents of the funnel. Wash the ethereal solution three times with water and add the washings to the aqueous liquid.

On evaporation of the ether, the total fat is obtained. The aqueous solution, or an aliquot portion, gives the total sulphuric acid by precipitation with barium chloride.

Sulphoricinoleïc acid. (1) Weigh 5 grms. of the oil into a separating funnel, dissolve in ether, and shake repeatedly with a 20 per cent. solution of pure sodium chloride. The washings are united, acidified, and finally precipitated by barium chloride. The result gives the sulphuric acid present as sodium or ammonium sulphate; the difference between this quantity and the total sulphuric acid gives the sulphuric acid present as sulphoricinoleïc acid, from which the quantity of the latter may be calculated.

(2) Weigh 10 grms. of the oil into a separating funnel, add 20 c.c. of water, and a little ammonia until the solution is clear, then add dilute sulphuric acid in slight excess, shake well, add 20 c.c. of petroleum ether, shake again, and let stand to separate. Then draw off the lower layer, and wash the ether solution repeatedly with water, until a portion of the washings is not rendered at all turbid by the addition of an equal volume of strong hydrochloric acid. Unite the washings, extract once with petroleum ether, take an aliquot portion, boil with strong hydrochloric acid for one hour, extract the oil which separates by means of ether, evaporate, and weigh. The result is the ricinoleïc acid present in soluble form as sulphoricinoleïc acid. This method is preferable to (1) in that it gives a direct estimate, but it may be tedious owing to the formation of emulsions.

Unsaponifiable matter and neutral fat. Weigh 20 grms. into a separating funnel, add 30 c.c. of water and ammonia until the solution is clear, then extract with ether, wash

the ether solution with water, evaporate, and weigh. The residue should be very small.

Lubricating Oils.—Apart from the chemical analysis of the oils, which is conducted by ordinary methods, it may be necessary to determine (*a*) mineral acids, which should be absent; (*b*) fatty acids, which should be low; (*c*) the cold test; (*d*) the flash point, which is best taken by the 'open' test (oils of low flash point are dangerous in use, 150° C. may be given as an approximate lower limit); (*e*) the viscosities at temperatures which depend on the purpose for which the oil is to be used, cylinder and gas-engine oils being tested at high temperatures. It is far from true that lubricating oils of the same viscosity are equal in value; a proper estimate of the lubricating power of an oil must be based on a knowledge of its composition and of the practical qualities mentioned above.

Lubricating Greases.—Solidified oils and greases are examined in an entirely different manner. The only practical test to which they need be subjected is a determination of the melting-point, which may be taken by observing the behaviour of a portion of the grease on the sides of a test-tube immersed in a beaker of water heated by a small flame. The melting-point desirable depends on the purpose for which the grease is to be used.

Solidified oils are generally mineral oils gelatinised by means of soap, they may or may not contain water. The viscosity cannot be determined, since on melting the oil separates from the soap solution. The examination is simple: about 5 grms. are dried at 100° C. to constant weight, and then extracted with petroleum ether in a Soxhlet apparatus until colourless; the extraction is

much accelerated by occasionally stirring the residue. The ether solution, after washing with water, leaves on evaporation the total oil contained in the sample, which can then be examined by ordinary methods; it will generally be found to consist of mineral oil with a small proportion of saponifiable oil. The residual soap is decomposed by boiling with dilute sulphuric acid, the washings of the ether solution are added, and the fatty acids are extracted by ether and the melting-point taken. Talc and other mineral matters may also be found along with the soap. If such substances are present, the ash of the original grease should be estimated and examined; while talc is not entirely to be regarded as an adulterant, gypsum and barytes are certainly valueless.

Rosin greases are essentially composed of rosin oil and calcium resinate (see *J.S.C.I.*, 1901, 1193). They may also contain water, mineral oils, coal-tar oils (anthracene oil), talc, gypsum, barytes, &c. The examination may be conducted by practically the same method as the above.

Boiled Linseed Oil, Lithographic Varnish, Linoleum.—Boiled linseed oil varies in colour from a pale yellow to a dark reddish-brown, and in specific gravity from 0·935 to about 0·980. The drying power in commercial oils is roughly in proportion to the colour and specific gravity, the dark oils drying more rapidly and having a greater density. Good dark boiled oil should dry in about five to six hours, pale oil in twelve to sixteen hours at the outside. The dried films should become hard, and not remain or become at all 'tacky.' Boiled oil may be at once detected by the dark colouration it gives with ammonium sulphide, due to the presence of the 'driers.'

Boiled oil which is qualitatively (p. 108) shown to be free from unsaponifiable matter, dries well, has the correct specific gravity, and a normal acid value, may generally be accepted as pure. The following analytical constants (*J.S.C.I.*, 1898, 305) are given by Williams (also see pp. 146–149, 170) :

Consistency	Specific Gravity at 15·5° C.	Unsaponifiable Matter, per cent.	Saponification Value	Iodine Value (calculated from Br value)	Acid Value
Very thin .	0·947	2·34	182·2	158·7	8·2
Thin . .	0·948	1·27	180·9	154·8	7·1
Thin . .	0·961	2·11	179·5	150·6	12·7
Stout . .	0·972	2·01	189·3	98·9	19·8
Stout . .	0.982	2·04	185·6	106·6	21·1
Very stout .	0·983	2·14	183·0	98·2	25·2
Solid . .	—	2·08	193·9	113·3	14·1

The usual adulterants of boiled linseed oil are mineral oils, rosin oils, rosin, and metallic resinates ; the latter may legitimately be present in small quantity as 'driers.' A normal specific gravity and consistency for adulterated boiled oil may be obtained by using rosin oil and mineral oil in conjunction, or rosin and mineral oil, or by adding mineral oil to boiled oil blown very stout. If any considerable proportion of a heavy mineral oil be added, the mixture would dry very badly ; the presence of light mineral oils is indicated by a low flash-point. For the detection of mineral and rosin oils see pp. 107–114 ; rosin is detected qualitatively by the Liebermann-Storch reaction and estimated quantitatively by Twitchell's process or by means of the acid value (see p. 125). The normal acid value of boiled oil is low, in fact generally lower than the values given in the table above for boiled oil of an ordinary consistency (sp. gr. 0·947–0·948). The

quantity of resinates and metallic salts of other acids in boiled oil is determined by taking the acid value of the sample and then dissolving 5 grms. in 40 c.c. of benzene, thoroughly shaking with 40 c.c. of 10 per cent. nitric acid, washing with water until the mineral acid is removed, and then running the benzene solution into 60 c.c. of alcohol and determining the acid value. The difference between the two acid values is equivalent to the resinates or salts of fatty acids which are present as driers or otherwise. Assuming the acid value of rosin to be 160, the percentage may be calculated; it should not be higher than about 2 per cent.

Lithographic varnish is produced by heating linseed oil to high temperatures without any addition of driers. Decompositions of an unknown character take place, the oil becomes thicker and specifically heavier until, on cooling, it is gelatinous. Lithographic varnish (or stand oil) contains a normal amount of unsaponifiable matter, and therefore gives a clear solution in the qualitative test. The consistency is strictly in proportion to the specific gravity.

Linoleum is essentially a mixture of oxidised (solid) linseed oil with ground cork, rosin, and kauri copal. For mechanical methods of testing linoleum see *J.S.C.I.*, 1895, 587; 1900, 255.

Paint oils. The term is applied to raw linseed oil adulterated in a similar manner to boiled oil: by rosin and mineral oil, rosin oil and mineral oil, &c.

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