# FORENSIC CHEMISTRY

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# PREFACE

Early in 1920 the author published a small book entitled *Legal Chemistry and Scientific Criminal Investigation.* This consisted of a brief introduction to the subject together with a collection of notes of cases and included a number of chapters on various aspects of legal chemistry not connected with criminal investigation.

This book was very favourably reviewed, but from the criticisms made it was evident that something more was required and that there was room for a general text-book on the subject of Forensic Chemistry. The present book therefore has been written. Such portions of the previous book as dealt with criminal investigation have been revised and enlarged and incorporated in the present book and all matters not connected with criminal investigation have been deleted. Much of the present work, however, is new and, with the exception of its predecessor *Legal Chemistry*, it is believed to be the first of the kind in English. It is true that a book with the same title was published in 1909, but this, as was indicated by the sub-title, had particular reference to the presentation of general chemical evidence in Court, and not to methods of obtaining the facts on which chemical evidence is based, much less to chemical methods of criminal investigation.

Methods of examination and details of manipulation which are known to every analyst and which are to be found in the ordinary text-books have been omitted, but special methods required by the expert are given in full.

Some of the subjects dealt with will be found described in books on Forensic Medicine. This, however, does not mean that these subjects are medical, for such is not the case. Thus the analysis of materials for poisons and the testing of stains caused by acid or other chemical substances are wholly chemical. Other subjects, including blood testing, the action of preservatives upon the dead body and the formation of adipocere are largely chemical, as is also the examination of firearms and projectiles though these have been used for wounding or killing. The subjects of microscopy and photography, which are also included, are, it is true, not specially chemical and precise measurements are the province of the physicist, but the microscopy, photography and physical determinations described are well within the competence of the chemist. The author has been careful not to encroach upon the medical side of any subject, but has limited himself strictly to the chemical and general aspects.

The chapters on Blood Stains and Poisons as originally written were submitted for criticism to Mr. W. M. Colles, B.Sc., A.I.C., of the Ministry of Justice, Cairo, who has had charge of all the testing for blood and poisons required by the Egyptian Government for many years and whose experience of these subjects therefore is very considerable. Mr. Colles generously supplied practical details and suggestions which have been freely used and the author wishes to express his indebtedness for this assistance. The chapter on Blood Stains as it now stands is largely based upon Mr. Colles' notes, which have also been drawn upon for that section of the chapter on Poisons which deals with the extraction and testing of alkaloids.

The author's thanks are also due to Mr. G. H. Hughes, Chief Inspector of the Native Parquet in Egypt, for his keen appreciation of the value of scientific evidence and for much encouragement and help.

A. L.

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# FORENSIC CHEMISTRY

## CHAPTER I

## INTRODUCTION

**Definition of Forensic Chemistry.**—Forensic or legal chemistry may be defined as chemistry applied to the solution of certain problems which arise in connection with the administration of justice. It is chemistry exercised in the service of the law.

**Scope of Forensic Chemistry.**—The scope of forensic chemistry is very wide and the boundaries of the subject are very ill-defined. Not only does it include the chemical side of criminal investigation, with which it is generally associated in the public mind, and to which it will be limited in the following pages, but it is also concerned with the analysis of any material the quality of which may give rise to legal proceedings. Forensic chemistry, too, deals not only with purely chemical questions, such as the nature, composition and quality of materials as determined by analysis, and the examination of articles for the presence or absence of particular substances, such as poisons, but it is concerned also with questions which are only partly chemical, as for example the examination of blood stains, questioned documents, counterfeit coins, fibres and textile fabrics, and the expert in forensic chemistry should be not only a capable analyst, but also an experienced microscopist and photographer.

**Practice of Forensic Chemistry.**—The practice of forensic chemistry naturally resolves itself into three stages, namely (1) the reception of the article to be examined, (2) the examination, and (3) the communication of the results. These three stages may usefully be considered in detail.

I. **Reception of the Article.**—It is most important that whenever possible the expert should himself personally take such articles as are required for examination.[1] Unless this is done, something essential to the elucidation of the problem may be omitted, or the articles may be taken, handled or packed in such an unsatisfactory manner as to render them almost useless for purposes of investigation. Sometimes too it is necessary that the expert who examines an article should know the precise condition, and even the position, in which it was found, and this can only be done when he is called in at the very commencement of the enquiry and before anything is disturbed or removed. It is impossible to lay too much stress upon this point, for just as finger-prints or foot-prints may be obliterated and thus valuable evidence lost, so other clues may be destroyed or even misleading indications may be produced. Whenever possible therefore in all cases of criminal investigation in which a chemical expert is consulted, he should visit the scene of enquiry, make a detailed examination and take notes and measurements and also any samples that may be required. A sketch as neatly and accurately drawn as possible with all the important measurements shown will be found a great help in writing the report. This sketch, however, should not be reproduced in the report, as unless drawn to scale it will be too inaccurate to be of practical value in Court and may be sufficiently inaccurate to be misleading. If a plan is necessary only one made by a trained surveyor should be used. In the case of a fire, explosion or similar occurrence, as much information as possible should be obtained regarding the exact previous condition of things, also an account of what was seen or heard at the time and the explanation that is given of the occurrence by those concerned. It should be remembered that generally it is difficult or impossible to repeat a visit, and that, unless full details are obtained in the first instance, the report will be incomplete and, in the absence of some small fact, which has been omitted, it may be found impossible to

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[1] This does not apply to viscera to be tested for poisons, since these are necessarily taken by the medical man who conducts the post-mortem examination.

express a definite opinion or to explain what otherwise might be a very simple matter. Special care should be taken to make the notes not only full but neat and clear, since it may be necessary to produce them in Court. A careless superficial examination and any inaccuracy in recording the facts are inexcusable.

When summoned to the scene of enquiry the expert should go fully prepared for all eventualities and should take with him everything that is likely to be required : a lens, or preferably a reading glass with a handle, a penknife, a few small round cardboard boxes for samples and a measuring tape will generally be found useful, and a note-book and pencil are indispensable. The note-book should be of good quality and not too small a size and the pencil should be an indelible one. A camera is also frequently of service.

In the majority of cases, however, the expert will not have the opportunity of taking his own samples, but these will already have been taken before he is called in and they will be handed or forwarded to him.

As no conclusions of value can be formed without a knowledge of all the facts, no opinion whatever should be given until the articles have been fully examined and the matter carefully considered.

As soon as practicable after an article has been received it should be registered, and the register should show the date and hour of receipt, from whom received, the nature of the article and details of all numbers or other distinguishing marks.

At this stage the package should not as a rule be opened but, whenever it is opened, this should be done by the expert himself.

It is convenient to keep one general register in which all articles received are entered in order of date, each being given a special number, the numbers being consecutive in any one year. The serial number of the article and the year should be marked on the outside of the package. When the expert himself takes the samples, particulars should be written on each at the time and full and exact notes made as to when and how and under what circumstances they were taken and, immediately on arrival at the laboratory, they should be registered and numbered in the usual manner. If a large number of articles of any one kind are being constantly received it will be found convenient to have more than one general register, though it will rarely, if ever, be necessary to have more than two, one for the class of articles that predominates and a second for miscellaneous cases.

In addition to the general register or registers, however, a series of abstracts, one for each kind of article, in which the results of the analyses are summarized, will be found useful. These are for quick reference to the details of the cases. In the Poisons abstract, for example, there would be a separate page for each kind of poison, and the cases would be entered in numerical order with details showing the nature and weight of the article analysed and the amount and percentage of poison present. In the Counterfeit Coin abstract, such particulars as the denomination of the coin, the year, weight, specific gravity, composition and whether struck or cast, would be given in tabular form. Thus at a glance the number of cases of any particular kind and the details of each case can be seen. With regard to the reports, an exact copy of each must be kept and, as these are frequently too lengthy to be conveniently transcribed into a register, it is generally best to keep typed copies in separate files according to the nature of the case, though a brief summary in the general register will be found useful. The general register should also show the number and page of the laboratory work-book in which the original facts and figures were entered at the time the article was examined.

Articles are frequently very badly packed, very inadequately marked, and insufficiently secured and sealed or sometimes even not sealed at all. This is not the fault of the expert but, by drawing attention to it each time it occurs, improvements in this respect may be brought about. The commonest faults are the use of wet or dirty bottles, dirty corks, insufficient protection of glass vessels during transit, the use of thin cardboard boxes which cannot be closed securely and which easily break or become forced out of shape, and so allow the contents to escape, the use of ordinary correspondence envelopes from which finely powdered materials always leak, the packing of several articles in one parcel in such a manner that contamination of one with another is almost inevitable, and the use of sealing wax on the corks of bottles containing alcoholic liquors or other liquids in which sealing wax is soluble. Many articles too are received without sufficient marks of identity or else bear such marks only on the outside wrapper which has to be removed. It does not seem to be sufficiently recognized that ordinary gummed labels do not adhere well to tins or to painted or glazed surfaces, with the result that such labels frequently come off and the articles are left without any means of identification. For small samples of dry material the round cardboard boxes, which are sold in nests of different sizes, will be found very useful. Oblong envelopes with the opening at the end are also satisfactory, and such envelopes are made in all sizes specially for holding samples, and many have ingenious methods of fastening. As already mentioned, ordinary correspondence envelopes are most unsuitable and should on no account be used. Acids, explosives and other dangerous materials are particularly liable to be packed in a very unsafe manner. Instructions regarding packing will be found in many of the chapters dealing with special subjects.

II. **The Examination.**—The first step in the examination of an article is to scrutinize it carefully and to write down in the laboratory note-book [1] a full description of its appearance. The outside of the package, the manner in which it is secured and particulars of the seals, if any, should first be described. The package should then be opened and any inner wrappings or packages should be examined and described, and finally a detailed description of the appearance of the article itself should be given. This will be found very helpful, since before an article can be described it must be carefully and minutely examined and definite conclusions must be arrived at on such points as

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[1] The note-book should be of good quality, with ruled paper and a stiff back, which should not be of the glazed black material so often used, but of something on which it is possible to write. All entries in this book should be in ink.

colour, shape, size and other physical characteristics which might otherwise be left vague and inconclusive.

The serial number, which was originally marked on the outside wrapper, should now be put on the article itself, if that be possible, or, if not possible, then on the innermost package. When, however, the article consists of a written or printed document, this should not as a rule be numbered or marked in any way, as by inadvertently covering up something of importance the value of the document as evidence may be seriously impaired, and what is, or is not, of importance frequently cannot be recognized until the document is critically examined. Occasionally when there are a number of different documents, which must be referred to separately in the report, it will be found convenient to mark them with a distinguishing letter or number, but this should be done carefully and generally in one corner. Frequently, however, documents will bear a distinguishing mark or number when received. The practice adopted by some experts of placing their name or initials on all documents as a means of identification is unnecessary. If a document has been carefully examined and described it can always be identified again. In the case of some articles, particularly those made of metal, it is not always easy to mark them, but this should be done as a general practice whenever possible. Coins, which may readily be confused, should always have a distinguishing mark or number scratched on them. Frequently considerable trouble is caused during a trial, in which there are a large number of different exhibits, by the confusion of similar articles or by mistakes in identification due to insufficient or unsatisfactory marking.

The original package should be opened in such a manner that if possible the seals remain intact, since sometimes questions arise in Court during a trial concerning the identity of the seals and further examination of them may be necessary. For the same reason all wrappers should be kept. Before opening any package it should be seen that the table on which this is done is clean and unencumbered with other articles.

From the time the package is opened until the examination is finished and until the article is finally sealed up again it should never leave the personal custody of the expert and should not be left about the laboratory or be unnecessarily exposed in any way; when not actually required it should be kept in a locked room or cupboard to which the expert alone has access.

The second step in the examination, if an article requires chemical analysis, is as a rule to weigh or measure it, after which, if possible, it is divided into several portions, one being kept in its original condition for further reference or for production in Court. All weights and measures should be entered directly in ink in the laboratory note-book at the time they are determined.

The third step is the chemical analysis, microscopical or other examination or physical testing, whichever may be indicated. It is not proposed to describe methods which are known to every analyst and which may be found in the ordinary text-books, since before a chemist specializes in legal cases he will naturally have had experience in general analysis, and he will be familiar therefore with the usual methods and processes and will also know where to find the description of additional methods should he require them. For matters which are outside the range of an analyst's ordinary work special books must be consulted and the names of a few such books will be given in connection with the various subjects dealt with. Certain types of cases, however, such for example as those concerning clothing, questioned documents, fires, explosives, explosions, robbery, stains and marks, will present different aspects and new features every time, and the methods of dealing with them can only be learned by practical experience. In the following chapters the methods adopted in special cases will be described.

Whenever a chemical analysis is made special care should be taken that the apparatus and vessels used are clean, that the reagents are pure and that the analysis is conducted in such surroundings and in such a manner that contamination or loss is impossible. It is not merely sufficient for the expert himself to be convinced that all these things are as they should be but he must be in a position to convince others. For example, he may feel certain that the reagents employed are pure but he cannot definitely refute a suggestion or statement to the contrary unless he has the positive evidence that can only be obtained by having tested them. During the Maybrick trial one of the witnesses, a medical man, who had made a preliminary test for arsenic, when asked about the purity of the copper and hydrochloric acid employed, could only say with regard to the copper "I do not know whether the copper was absolutely pure but I have a very strong impression that it was pure," and with regard to the acid, that he had bought it from "the chemist."[1] Blank or control experiments should be made whenever possible. At every stage full notes should be made in ink in the laboratory note-book. Pencil notes and scraps of paper should never be used.

Any important ingredient separated in the course of an analysis should be preserved for production in Court.

After the examination or analysis is finished, the article or residue should be securely sealed, and either kept for production in Court or handed over to the proper authorities. It is preferable that whenever possible the expert should retain the articles he has examined and should himself produce them in Court, as few people realize the importance of the proper care of exhibits.

The expert will generally have some help in his work but he must personally conduct all the important operations and should see and verify everything else that is done and form his own conclusions independently of the opinion of others, since it is he who is responsible and it is he who will make and sign the report and give evidence in Court, and he must therefore be in a position to describe both the article itself and the method of its examination and to justify the conclusions.

Some of the precautions mentioned may possibly appear very trivial and unnecessary, but experience proves that none of them can safely be dispensed with, and the neglect of any one may seriously discount the value of the most careful analysis or even invalidate it altogether. Thus, for instance, the authenticity of an article may be challenged and it then becomes necessary for the expert to prove, not only that it had never been out of his custody since he first

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[1] Notable English Trials: Mrs. Maybrick, pp. 105, 106.

received it, but also that it is impossible it could have been tampered with in any way or that anything could have been added or abstracted. It should not be forgotten too that the chemico-legal expert occupies a very different position from that of the ordinary analyst in that everything he does is liable to be subjected to the strictest scrutiny and adverse criticism, and his work is either done on oath or the results must be sworn to. Moreover where the liberty or the life of another is concerned it is impossible to take too much care.

In the practice of forensic chemistry there are six golden rules which should never be forgotten. These are :—

1. Go slowly : good work cannot be hurried, therefore take all the time necessary to make the case complete, no matter how urgent it may appear or how pressing others may be for the result ; it is generally possible to adjourn a case if the work cannot be finished in time.

2. Be thorough : make a careful and minute examination of everything and do not be satisfied with a qualitative analysis if a quantitative one be possible ; it always pays to do too much rather than too little and it is difficult to foresee what will or will not be required in Court.

3. Take notes : keep a full, neat and clear record of everything seen and done.

4. Consult others : many cases will lead the expert into paths with which he is not familiar, and when this happens he should consult those who are most likely to know. Unless an expert knows his subject thoroughly, or unless he consults others, he may go seriously astray ; thus a doctor, who found salt in an Egyptian mummy, reported it to be that of a person who had been drowned in salt water, apparently quite in ignorance of the very readily ascertained fact that crude natron, which contains a large percentage of salt, and at a later date, salt itself were commonly used in ancient Egypt in connection with the preservation of the body. The chemist too who thought that the ancient Egyptians used quicklime as a body preservative, because he found a small amount of carbonate of lime in a mummy, was evidently unaware of the extent to which limestone and limestone dust occur in Egypt and that burial in tombs cut

in the limestone rock was very common. Another expert, who had not made himself familiar with the methods of using liquid fuel under a boiler, but who had evidently heard of the use of compressed air in that connection, thought that compressed air was forced into the oil in the feed tank.

5. Use imagination : this is somewhat hazardous advice, since an expert with a vivid and uncontrolled imagination is a most dangerous person ; a disciplined imagination, however, which enables inferences and deductions (to be verified or discarded at a later stage) to be made from slender and incomplete premises is often very useful. In writing the report the imagination must be kept well in hand and the expert should remember that what to him may be merely a helpful working hypothesis is liable to be taken by others for more than its true worth and to be adopted by them as a final explanation.

6. Avoid complicated theories : the simplest explanation is the most likely to be the right one.

III. **Communication of the Results.**—The results of the work carried out will generally be communicated in the form of a written report, which may be supplemented at a later date by oral evidence. Both the written report and the oral evidence therefore will be considered.

The written report should commence with an enumeration of the articles received for examination, the date of receipt and from whom received and should state how they were packed and whether they were securely fastened or not and whether sealed, and if so a description of the seals should be added. A brief but sufficient record of all the significant facts noted during the examination of the article should then be given, followed by a summary of the conclusions drawn from the observed facts and from these alone, the conclusions, however, being kept quite distinct from the facts and the whole being expressed in plain and non-technical language. The conclusions arrived at should be given very definitely and without ambiguity, vague and inconclusive opinions being useless. This, however, does not mean that the expert should dogmatize on insufficient data and it is sometimes necessary to state that the facts are so few or so obscure that no definite conclusions are possible.

All opinions should be supported by the evidence on which they are formed, that is to say every statement made should be proved and unless a point can be proved to the satisfaction of others it will generally be found to be worthless.

In ordinary gravimetric or volumetric analysis it is neither necessary nor wise to report the results to more than one decimal place, as although the weights, measurements and calculations will probably be carried out to two or even to four decimal places, yet the errors inherent in the method, apparatus and manipulation are such that anything beyond the second decimal cannot possibly be correct and even the second decimal is very liable to be wrong. In special cases, however, such as the analysis of poisons, the results must frequently be given to tenths or even hundredths of a milligram. Thus in the Seddon trial quantities of metallic arsenic varying from $\frac{1}{30}$ to $\frac{1}{300}$ part of a milligram were obtained by the analyst from the various samples.[1]

It is not, as a rule, necessary to mention how an analysis or examination has been conducted and therefore, whenever possible, all references to methods should be omitted, as their inclusion only lengthens and complicates the report. Sometimes, however, it is useful to state that the analysis has been conducted by well-known methods, which are mentioned by name, or by the methods adopted by, say the Government Laboratory, London, or the Society of Public Analysts, as the case may be. The one endeavour in writing the report should be to make it as precise and clear as possible. Utility, however, should not be sacrificed to brevity, and the report should be sufficiently full and explanatory to meet any adverse criticism it may encounter, containing within itself the replies to any arguments likely to be directed against the conclusions arrived at, since the expert will seldom be able to supplement his report by further details or explanations.

If micro-photographs or photographic enlargements of objects have been made, a copy of these should be attached to the report, and it should be clearly stated on the photographs what they represent, and any special points illustrated should be indicated.

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[1] Notable English Trials: The Seddons, pp. 120, 123.

After drafting a report and before it is finally copied and forwarded it will be found a useful check on the correctness of the conclusions for the expert to put himself mentally in the place of the counsel for the opposite side and to scrutinize the report carefully from that point of view, more particularly as to whether the data are sufficient to justify the conclusions arrived at, and whether or not explanations other than those given may not agree equally well with the facts.

So far as oral evidence is concerned, the principal requirement is that the expert should have a thorough knowledge of his subject and of the collateral matters relating to it. Since the evidence may be given weeks or even months after the work has been done and the report written, it is not only permissible but necessary that the expert should refresh his memory by reference to his laboratory note-book before presenting himself in Court and he should also have the book with him in Court for further reference if required. It is not sufficient that the note-book should be consulted for a few minutes immediately before going to the Court or while waiting to be called, but it should be well studied beforehand. A witness with a bad memory, who has not taken the trouble to refresh his mind with the facts of the case, presents a very poor spectacle in the witness box. It should be remembered, however, that no notes, except those made at the time the work was done or as soon after as practicable, are allowed in Court and nothing written subsequently is permitted to be used. The expert must also anticipate and prepare himself to answer likely questions having reference to the case and should have at his fingers' ends all the details of the methods of analysis or examination employed and should be prepared to state the degree of accuracy attained. He should also know the weaknesses, if any, inherent in the methods used and be ready to meet adverse criticism directed against them. All answers to questions and all opinions should be definite and free from ambiguity and should be given in as simple language as possible. The expert should also be prepared to say what practice he has had in the particular kind of examination under consideration, how many years his experience covers and approximately how many cases of a similar nature he has investigated.

Should it be necessary to employ any term susceptible of more than one meaning, such for example as " arsenic " when either " oxide of arsenic " or " sulphide of arsenic " might be meant, the sense in which the term is used should be made quite clear. Some confusion seems to have arisen during the trial of Mrs. Maybrick owing to the neglect of this precaution, and the amount of arsenic found on analysis was sometimes referred to by the witnesses as oxide, sometimes as sulphide and sometimes merely as arsenic without any indication of what compound was meant. In the Palmer case the term " antimony " was used in the same loose way.

As an indication of the kind of questions that may be asked in cross-examination and that therefore should be prepared for, the author's experience in a few cases may be mentioned : when giving evidence in cases of counterfeit coining he has been called upon to state how genuine coins are made, to explain the difference between cast iron, wrought iron and steel and to justify his statement that certain instruments were made of steel, to give the approximate pressure required to impress the designs of some steel dies upon a given alloy and to say whether a certain press, which the prosecution contended was part of a counterfeit coining plant and which the defence explained had been employed for making small tiles, could be used for making tiles or not, and whether the accused person's description of the materials and method employed for making tiles was correct. In connection with various bomb outrages he has been asked to give the relative strength of different explosives, the radius of a particular explosion, and the probable distance that a bomb had exploded from an object which had been hit and to define explosion and detonation and to explain the difference between them.

Sometimes an object, such as a detail of the design of a counterfeit coin, a defect in a counterfeit coin, a characteristic in a handwriting or an alteration or erasure in a document, about which evidence is being given in Court, is so small that it cannot be seen clearly, or even cannot be seen at all, unless it is magnified. In such a case no attempt should be made to show the object to the judge or jury through a microscope, or even through any sort of small lens, since such instruments are not only useless but confusing to persons not accustomed to them. In such circumstances the expert should be prepared with an enlarged photograph of the object in question, though sometimes a large reading glass with a handle will be found useful, as this can be understood and managed by most people. In the Maybrick trial the judge, after vainly endeavouring to see a deposit of oxide of arsenic on the sides of a tube by means of a lens, remarked of the latter that "it was very difficult to manage."[1]

A final word of caution may be given, namely that the expert should scrupulously avoid allowing his interest in a case or his belief in the correctness of his own conclusions to make him an advocate or partisan ; he appears in order to present any facts he may have discovered, to interpret these and other facts and to help the Court with opinions, and his evidence should be given without bias to either side.

**Conclusion.**—In conclusion it is desired to emphasize the fact of the great assistance that the chemist can frequently render in the administration of justice. This is generally conceded with regard to all matters pertaining to the composition of articles or to their value as determined by analysis, but it is not sufficiently recognized in connection with criminal investigation, except in cases of poisoning, when the chemist is acknowledged to be indispensable.

The chemist is specially trained in observation and deduction ; he necessarily learns to enquire closely into the nature and meaning of things ; as a result of experience he becomes very sceptical of outward appearances ; if he practises as an analyst he is brought into intimate contact with a great variety of different materials, of all of which he must know the composition and properties ; and he habitually deals with traces of substances and weighs and measures them, all of which qualifications peculiarly fit him to act as a scientific expert in criminal investigation : observation, deduction, an enquiring turn of mind, a scepticism of appear-

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[1] Notable English Trials: Mrs. Maybrick, p. 136.

ances, a knowledge of the composition, properties and uses of articles and the ability to see and examine traces are all essential for this work, and it may confidently be stated that if criminal investigation is to keep abreast of the times it must be made more scientific and chemical analysis and microscopy must be more frequently used; in fact the criminal is becoming so scientific, not only in his work, but also in the means he adopts to escape detection, that a scientist is needed to cope with him, that is to say, a scientist must be set to catch a scientist.

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## CHAPTER II

# BLOOD STAINS[1]

As an aid in the detection of crime, the value of testing stains which look like blood, to ascertain whether they are blood or not, is well known.

This class of evidence has been enormously augmented in value by Uhlenhuth's application to the determination of the origin of a blood stain, of Bordet and Tchistovitsch's discovery of precipitin anti-sera. If a rabbit is injected intravenously several times at intervals of a week with small quantities of sterile human blood serum, the serum of the rabbit acquires the property of producing a precipitate when added in small amount to human blood serum diluted with normal physiological saline. With the possible exception of anthropoid ape serum this rabbit blood serum is specific for human blood. When compared with the previous method of examining the corpuscles in a blood stain microscopically, which was often impossible with old stains, the value of this test is obvious.

The testing of blood, although generally regarded as medical, is in fact largely chemical, more particularly since the anti-sera required for the biochemical test need no longer be prepared by the expert but may be purchased ready for use. There is no doubt too that the chemist, by reason of his training and experience in manipulation and his skill in testing and in general laboratory practice, is well qualified to undertake blood testing. It may be mentioned that the " detection of blood stains " is among the subjects required to be known by all candidates presenting themselves for the examination for the Associateship of the Institute of Chemistry. Blood testing in legal cases, however, should only be undertaken by those having consider-

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[1] See note in Preface.

able experience of the work, as most serious issues may depend upon the results.

Articles sent for examination for blood stains generally consist of clothing, weapons, tools, furniture, stones, soil and parts of buildings, such as pieces cut from a door or plaster removed from a wall.

The appearance of blood stains, although familiar, cannot be regarded as an infallible guide, as the colour may vary from chocolate-brown to orange, pink, green or even black. Stains of a similar appearance to blood may be caused by vegetable and fruit juices, rust, iron-mould, paint, coffee, sealing wax or dye.

### Search for and Preservation of Blood-Stained Articles.

This is a matter for the police, or other investigating authority, but the search for blood-stained objects at the scene of the crime or in the house of a suspected person cannot be undertaken too carefully. Unimportant as a small blood stain may appear among a number of larger stains, the determination of its origin may have great significance if, as sometimes happens, blood stains of more than one origin occur together, as may be the case, for example, if a dog is killed in defence of its master, when mixed human and dog blood might be found.

When searching for blood stains attention should be directed not only to clothing and weapons, but the fingernails, boots and linings of the pockets of the suspected person should also be examined. If too much time has not elapsed since the occurrence of the crime, the possible movements of the suspected person should be considered and search made for anything that may have been used for wiping or washing stains from his clothing or person. Recently washed clothing or rags found hidden away, partially burnt material and fresh ashes should always be submitted for examination. It cannot be too strongly emphasized that as liberal a supply of blood-stained material or suspicious objects as possible should be collected regardless of the subsequent work entailed in examining them.

When searching the scene of a crime for blood stains particular note should be taken (and if possible a photograph made) of the position, size and shape of any stains found, as this may afford a guide to the direction in which the blood was falling or spurting or in which the blood-stained object was moving or was being carried. Spraying from a cut artery causes extremely minute stains which, except for the fact that there are a great number very close together, possess an appearance closely resembling flea marks. On damp fabrics blood stains sometimes fade quickly and assume a faint green or bleached appearance.

In cases of stabbing the lips of the wound wipe the blade of the knife when it is withdrawn and only a thin film of a faint brown colour remains on drying. On the other hand blood is often found at the junction of the blade and the handle of a weapon and sometimes runs down between the prolongation of the blade and the inside of the handle, a point which is frequently overlooked by criminals when cleaning weapons after a crime. On such articles the blood when examined with a lens often looks like fresh red varnish. Wooden handles of tools too which have split or on which there are surface cracks, absorb blood into the cracks in a manner which even sandpaper fails to remove and weapons and tools which have been cleaned may therefore still show traces of blood and should always be sent for examination.

Blood Stains on Immovable Objects.—As it is much easier to examine blood stains in the laboratory, the blood-stained portion of any large immovable object should be bodily cut out or dissected away whenever possible. For example, stains on wooden flooring, doors, doorposts or furniture, should be sawn out if the stain is sufficiently adherent to withstand the vibration ; bricks or stones should be carefully dislodged from floors and walls, and plaster should be cut away in large pieces. If, however, the blood has to be scraped off, the various groups of stains should be collected in numbered packets of *soft* paper, a note being taken of their position and shape. It should always be remembered that mixed blood stains may be present and consequently only those stains close together or parts of a single splash or smear should be mixed. As the blood serum on which the biochemical test depends soaks into porous matter it is advisable to scrape off at least 0·5 mm. depth of substance below the stain, as this may be richer in serum than the superficial clot, and, as this material may contain substances which might nullify the serum test, it is advisable not to mix the deeper material with the purer superficial layer and each therefore should be packed separately. Scrapings are easily collected by means of a small scoop made by bending a piece of clean, bright tin and sweeping the blood into it with a small paint brush, which, however, should never be used again for blood work.

Blood Stains on the Person or on Animals.—The method of removing blood stains from the person or from animals that would probably occur to most people, not having experience in the matter, as being the best, would be to rub the place with a small swab of wet cotton wool, but if this be done the subsequent tests are very troublesome, as the blood is so much disseminated in the mass of the swab that it may be impossible to find any portion with a sufficient concentration to respond to the tests. Also a large volume of saline must be used for extraction of the serum, with the consequent danger of a negative test through too much dilution. A dilute solution may, however, be concentrated if necessary in a vacuum desiccator. The best method of removing blood from the person or from an animal for testing purposes is to moisten a clean piece of blotting paper or white fabric, one or two centimetres square, with physiological saline solution (0·9 per cent. sodium chloride) and allow this to lie on the stain as long as practicable and in any case until the paper or cloth is itself stained. The paper or cloth should then be allowed to dry in a gentle draught, never by heat, before packing up and despatching to the laboratory. In this way a concentrated stain on a small amount of material is obtained to which the tests are easily applied.

Blood Stains on Damp Objects.—Damp objects such as boots, recently washed clothing, and mud must be dried before being packed up, or a growth of mould may impede the subsequent tests. When the article is wet, bacterial action will also tend to denaturalize the albumins and even putrefaction of the blood matter may set in and hinder and possibly even nullify the serum test. The drying must be carried out in a gentle draught or in a warm room and never by artificial heat.

BLOOD STAINS ON FRIABLE OBJECTS.—Stains on friable objects such as plaster, dried mud or dry leaves require special packing. Unless this is done, the shaking in transit causes such material to crumble, and blood which was originally easily visible on the surface is almost hopelessly lost in a mass of powder on arrival at the laboratory. Examination with a lens may result in the finding of small stains where large stains originally existed, but considerable trouble is avoided if special means of packing are adopted. Embedding in a mass of plasticine, stained side uppermost, packing in a small cardboard box with dry cotton wool and then in a larger box, or scraping off the stain are recommended.

**Preservatives.**—In no case should any preservative whatever be added or packed with blood stains, and only clean paper or dry cotton wool should be allowed to come into contact with them. Care should be exercised that gum, candle-grease or sealing wax do not contaminate the stains, while the package is being sealed. Many hours are sometimes wasted by the expert in a fruitless attempt to obtain a test for blood from what eventually proves to be red sealing wax.

## EXAMINATION OF BLOOD STAINS.

The examination of blood stains is divided into three distinct stages, namely :—

(1) A preliminary test. This is a quick, easy method of deciding whether a stain is likely to be blood or not. At present there is no simple chemical test known which is absolute proof of the presence of blood, but there are several tests by which a stain may be excluded as not being blood.

(2) Confirmatory tests. These, which are directed to the detection of hæmoglobin, vary in sensitiveness and ease of application and may be divided into :—

   (*a*) Micro-chemical test : production of hæmin crystals by various methods.

   (*b*) Observation of a characteristic absorption spectrum of some form of hæmoglobin, usually that of hæmochromogen (reduced hæmatin).

(3) Determination of the origin of the blood by means of the biochemical or precipitin anti-serum test.

These tests will now be considered in detail.

**Preliminary Test for Blood.**—This consists in observing a colour change in a chemical reagent when applied to a part of the suspected stain or scraping. The well-known guaiacum test, although much improved by careful technique, has been superseded by other substances such as benzidine (paradiamino-diphenyl) and leuco-malachite green. More recently other substances have been proposed by various observers, such as alizarin "S" blue, phenolphthalin,[1] pyramidon, rhodamine "B" and ortho-tolidine. With the possible exception of ortho-tolidine, benzidine has been found the most convenient for use in legal practice. These tests all depend on the peculiar property of hæmoglobin in acting like a "peroxydase" in the presence of hydrogen peroxide, the transfer of the oxygen being indicated by the formation of a deep blue colour. Judging from the instability of peroxydases and from recent work, this action is not that of a peroxydase but is probably due to the catalytic property of the iron combined with the hæmoglobin.

An excellent and convenient way of obtaining hydrogen peroxide for this test is furnished by sodium perborate, which gives off hydrogen peroxide when dissolved in acetic acid.

A sensitive solution of benzidine is made as follows:—0·2 gram of dry sodium perborate is dissolved in 1 c.c. of water and 10 c.c. of glacial acetic acid. To this 1 c.c. of a 10 per cent. solution of bezidine in glacial acetic acid is added. If sodium perborate is not available, 2 c.c. of 6 per cent. hydrogen peroxide (free from acid) is substituted. The mixture has a pale straw or brown tint which darkens on standing. If the reagents are impure, it will rapidly turn blue. It should be prepared immediately before use, as it will only keep for a few hours, and recrystallized benzidine should be used. The test is carried out as follows:—

A double layer of good filter paper is spread on a porcelain tile and a minute fragment of dried blood or blood-stained fabric laid upon it. A drop of the benzidine solution is added by means of a pipette. A dense blue coloration,

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[1] The phenolphthal*in* is oxidized in the test to phenolphthal*ein* giving a red colour.

radiating out in streaks on the filter paper is produced instantaneously if blood is present. Moistening the material with pyridine before the addition of the benzidine solution promotes rapidity of action. Excluding peroxydases, peroxides and oxidizing agents, many substances, including soiled earth, manure and excrement, will give a similar blue coloration and even filter paper will do this after some time, but when blood is present the action is practically instantaneous. Stains which do not react within five seconds after being well moistened with pyridine can be disregarded unless the blood has been deliberately obliterated or rendered insoluble. The sensitiveness of the reagent towards old stains is increased by the addition of more perborate or more hydrogen peroxide, while the depth of colour is increased by adding more benzidine. Prepared as described this reagent is extremely sensitive. An instantaneous reaction is given by blood stains which have been kept in a semi-tropical laboratory for eighteen years. Blood boiled for an hour and kept for thirteen years acts readily. Blood stains on rusty iron kept for four years react within five seconds. It is almost impossible to wash out blood from cloth so that the fabric will fail to give a blue colour with the reagent.

As the value of the test lies in proving that a stain is not due to blood, it is important that the solution used should be sensitive and to make sure of this, it is advisable to test the solution against blood stains that have been artificially aged, which may be done by heating blood dried on cloth, iron or leather in an air oven to 130° C. for some hours. The solution, however, should not be made too sensitive, or much unnecessary work will be entailed in trying to confirm stains which are not due to blood.

*Negative Results.*—A brown stain of apparently recent origin that does not react within five seconds can be asserted not to be due to blood. Before definitely concluding, however, that a stain is not blood, the circumstances of the case must be considered. In the course of many thousands of tests carried out by Mr. Colles in Egypt, only one group of stains and these were old, faint, iron-grey coloured stains on the inner lining of a pocket, which were afterwards proved to be blood stains, failed to give the reaction within the specified time. Greasy, washed, boiled, grey, old-looking stains should be critically examined, especially if the garment or article appears to have been recently cleaned or scraped. A supersensitive solution should be prepared and the test repeated, moistening previously with pyridine, and the fabric or particles should be watched with a lens for any colour change. It is extremely doubtful if a stain which reacts sluggishly with benzidine will ever give the confirmatory tests for hæmoglobin, as the former test far exceeds the latter in delicacy.

In Egypt garments seized from *fellahin* (peasants) suspected of murder frequently exhibit stains which resemble blood to an extraordinary degree, but which are stated to be derived from such sources as cotton stalks, the date-palm, sugar-cane or Indian corn. A great many cases in which the benzidine test was doubted have been exhaustively examined for hæmoglobin by the spectroscope and by Beam's modification of the hæmin test and in no case has the presence of blood ever been proved after a negative benzidine reaction.

### Examination of Articles for Blood Stains.

(*a*) Clothing.—Clothes are conveniently supported on an easel or clothes horse by clips or tacks in such a way that they can readily be examined both inside and outside. Failing any prominent stains, the examination should be commenced with the cuffs and sleeves and the whole garment systematically inspected on both sides. Minute portions should be cut out of each stain with curved scissors, the pieces being held by forceps, and these pieces should be tested in the manner described. Stains positive to benzidine should be marked with small flag labels pinned on the garment and their position recorded on a rough outline sketch of the article. Observations should be made as to shape, size, surface on which present (whether inside or outside), appearance (whether fresh, superimposed dirt, rubbed or washed) and presence of hairs or insect fragments so far as can be seen with a hand lens.

There is at present no reliable test for the age of blood stains, the appearance and ease in dissolving being the only guides, though neither is conclusive. The rate of solution in 1 per cent. arsenic trioxide solution or in dilute glycerine solution, as compared with that of stains of known age, has been suggested, but is not trustworthy. In one case which occurred in Egypt a razor copiously stained with blood was kept in the drawer of a desk in a police station in the provinces for six months and on receipt at the laboratory the stains appeared fresh and clean and looked as though they might have been made the day before.

(*b*) Washed Clothing.—Sometimes even unstained portions of a garment which has been washed give a strong reaction with benzidine which may be due to incomplete rinsing from the blood-stained water. In such cases resort must be had to the more tedious method of Michel (hæmochromogen). By making a less sensitive benzidine solution and observing which parts or stains give the quickest and most intense reaction the work may be shortened.

(*c*) Insect Marks on Clothing.—These will give the serum test for human blood years after the insects have fed upon man. (Hindle.) Spraying from an artery, as already stated, results in many small stains close together which resemble insect marks, while these latter are usually scattered. Microscopical search for parts of insects should be made in doubtful cases. The general condition of the garment and the social status of the accused should also be taken into account.

(*d*) Small Objects, Weapons and Tools.—These should be placed on a large sheet of glazed paper and scrutinized carefully with a lens. Special attention should be paid to all roughened surfaces or edges, such as turned edges of knives or burred heads of hammers, on which blood may be entangled and which are difficult to clean. The space between the handle and the head or blade of a tool should be exposed, a metal-cutting bow saw being found useful for this purpose. Scrapings or detached clot fragments should be tested with benzidine as described, as little as possible of the blood matter being used for the test. Rusted iron often shows a red, shining, cracked appearance very similar to blood, and the testing of such articles is often troublesome owing to apparent positive benzidine reactions from all parts. Instances have been met with in which rust has undoubtedly given the test and also hæmatite. Dirty cooking knives soiled with remains of meat, dirt on the soles of boots, manure, articles found in lavatories all sometimes show a positive benzidine reaction. The remedy in such cases, short of trying each stain separately by the hæmin test or spectroscope, is to use a benzidine solution which is not very sensitive, so as to pick out those stains in which there is most blood matter to which a confirmatory test is then applied. The position of the stains, when important, is conveniently recorded on an outline sketch of the article.

**First Confirmatory Test for Blood.**—If blood is boiled for a short time with glacial acetic acid and a trace of sodium chloride, characteristic crystals of "hæmin" (hæmatin hydrochloride) are deposited on cooling. A minute trace of blood only is necessary but the test requires practice and fails to respond if the blood has become difficultly soluble through exposure, putrefaction or contact with rust. The two methods of carrying out this test which are recommended from among the many modifications employed are :—(*a*) by direct heating on a microscope slide, and (*b*) Beam and Freak's modification.

METHOD OF DIRECT HEATING.—The first method being quick, is recommended for cases in which fresh blood-stained fabrics or small clots are available. A few square millimetres of the stained fabric or scrapings or a minute clot are teased or crushed on a dry microscope slide and covered with a clean dry cover slip. If this makes very close contact with the slide, a small slip of filter paper or a hair should be introduced underneath it to provide sufficient space for the acetic acid. Glacial acetic acid containing 0.1 per cent. of sodium chloride or preferably of sodium iodide is then run in under the slip by means of a pipette until the space is full. The slide is now cautiously and evenly heated over a small flame (the bye-pass of a Bunsen burner) until the acid boils, and heating is continued until about half the acid is boiled away. Gentle boiling is required, as it is not a question of concentrating the acid solution of the blood but rather of subjecting the blood to the action of boiling acetic acid. Tapping the slide with forceps during the heating prevents superheating which may otherwise occur and which results in blowing the cover slip and acid into the observer's face. More acid is now run in under the slip and the heating is repeated until about a quarter of the total acid has boiled away. The slide is allowed to cool and then examined under a magnification of 300 diameters. There are two points in the microscopical search for hæmin crystals to which attention may be specially directed, namely that the fabric or clot should be examined, when crystals may be seen entangled in the undissolved blood matter, and secondly that if boiling has been too prolonged, all the blood may have become dissolved out, in which case search should then be made in the area just beyond the edges of the cover slip in contact with the remaining acid, when clusters of crystals may be seen mixed with those of sodium chloride.

The normal appearance of hæmin crystals is that of dark brown, long, narrow, thin, rhombic plates over-crossed as in an X or in clusters like a star-fish. The X formation is very characteristic. With difficultly soluble blood (old or heated blood) the rhombic plates are much shorter and broader and owing to ill-defined edges at the angles possess the shape of a bean, but, however, are quite characteristic to the experienced observer. Unsatisfactory hæmin crystals can be recrystallized by pushing the cover slip over them and heating gently for a short time and then cooling. In this way obstinate blood matter that does not give good crystals can be moved across the slide and many trials may be made.

This method is often troublesome in practice with old, heated, or washed blood or blood mixed with rust or muscle fragments. Matter obviously blood sometimes refuses to yield crystals in spite of repeated heating due possibly to the presence of albumin. Rust is also troublesome, especially when actual combination with the hæmoglobin has taken place.

*Possible Fallacies.*—Distinct brown crystals must be observed. Colourless needles of sodium chloride if small simulate hæmin crystals. Fragments of indigo-dyed fabric yield large crops of long narrow rhombic plates of a fine blue colour. These, however, are much larger than hæmin crystals and are easily distinguished by the colour. In cases in which dyed material is being examined a part of the unstained fabric should be treated with the boiling acid and examined as a control test.

Method of Beam and Freak.[1]—This method is very reliable and most valuable when minute quantities of material have to be dealt with and in cases of old, insoluble, or rust-combined blood. The only drawback to the test is that it requires considerable time, and it is recommended that the tubes be prepared over-night and examined the next morning. The details of the test are as follows:—

"A small quantity of the suspected material is placed at the bottom of a flat arsenic sublimation tube about 6 by 3 mm. and 35 mm. long. A thin round ignition tube will answer if no flat tube is procurable, but for examination under the microscope the flat form is obviously better. A few drops of acetic acid containing from 0·01 to 0·1 per cent. sodium chloride are added and a very fine cotton thread adjusted so that its upper end is near the top of the tube and the lower end reaches to the bottom of the liquid. The thread should be everywhere in contact with the tube, to which it adheres readily by being moistened with the liquid. The adjustment is readily made by means of a glass rod one end of which is drawn out for the purpose. The tube is now placed in a rack, or supported by forcing its base into a small blob of plasticine, and allowed to remain until crystallization has taken place.

"Before placing the thread into position, the solution of the blood may be aided, if desired, by cautiously heating the tube over a small flame, loss of the liquid by explosive boiling being prevented by holding the finger tightly over the mouth of the tube.

"While such heating appears rarely to be essential, the long contact with the acid in the cold sufficing for solution in practically all cases, it is nevertheless advisable in that it gives at once an indication of the strength of blood solution obtained. For the best results this should be quite weak.

"The clear liquid, filtered by its passage through and along the thread, is slowly drawn up, by capillary action,

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[1] *Biochem. J.*, Vol. IX, No. 1, March 1915.

to the mouth of the tube. Under these conditions evaporation takes place so slowly that, even when the temperature of the atmosphere is above 38° C., it is complete (if the tube is held in an upright position) only after 12 to 24 hours or more. The rate of evaporation is readily controlled, within wide limits, by altering the dimensions of the tube and the angle at which it is supported.

"The crystals usually begin to appear, on or near the upper half of the thread, and of sufficient size to be distinguished with a power of 75 diameters, in about an hour; and they ultimately become so large that, in place of requiring a magnification of 250 to 300 diameters, as usually recommended, they may readily be seen with one of 25 diameters or even, in many cases, with a good hand lens. The crystals are best viewed against a dull white background. A piece of white paper is suitable.

"The extraordinarily large size of the crystals is due, in great part, to the slow evaporation of the liquid; but it is also a result of the removal, by the thread, of the interfering action of the blood albumin on crystallization. Examination of the thread at the end of the experiment shows that the soluble, non-crystallizable, constituents of the blood are carried to its upper end, and that the maximum growth of hæmin crystals is found lower down.

"If the solution is sufficiently weak, the first crystals form at the point of greatest concentration—the upper end of the thread; but as the albuminous matter accumulates these cease to grow and the largest and most numerous crystals are found, later, about midway down the tube.

"For the best results the following precautions should be observed:—

"The tube should be thoroughly clean and dry. It is best cleaned by swabbing out successively with acetic acid, water, and alcoholic soda, followed by thorough rinsing with distilled water and drying on a hot plate.

"The thread should be clean and as fine as can be obtained. A good plan is to take No. 80, 'six-cord sewing machine cotton' and to untwist it, using one of the 'cords' for the test.

"The solution of the blood in acetic acid should not be concentrated, but very weak. The liquid should only be faintly pink. This ensures large crystals and clean fields.

"If the method is to be applied experimentally to fresh blood, the latter must be completely dried before the addition of the acid, and especial care should be taken to work with very dilute solutions."

**Second Confirmatory Test for Blood.**—When examined spectroscopically hæmoglobin and its derivatives show characteristic absorption bands in the visible spectrum. In legal practice the derivatives of hæmoglobin that are most useful are (*a*) Hæmochromogen (reduced hæmatin), (*b*) Hæmotoporphyrin, and, if the stain is fresh, (*c*) Oxyhæmoglobin or Methæmoglobin. These will now be separately considered.

HÆMOCHROMOGEN.—When blood stains, consisting as they do of a mixture of oxyhæmoglobin, methæmoglobin and hæmatin, are treated with a powerful reducing agent in the presence of a strong alkali, hæmochromogen or reduced hæmatin is produced. This possesses two marked absorption bands. ③

*Reducing Agents.*—The most efficient reducing agent is sodium hydrosulphite ($Na_2S_2O_4$), first suggested by Linossier, which is now easily obtainable in the solid form. Egyptian practice proves this to be a most useful reagent. Michel's recipe [1] does not appear to contain sufficient concentration of alkali, and the following gives a reliable solution : 2 grams of sodium hydrosulphite are shaken up in a test tube closed with a rubber cork with 5 c.c. of 10 per cent. potassium hydroxide solution and 1 c.c. of alcohol. The solution must be kept corked and should always be prepared just before use. Other reducing agents are ammonium sulphide with potassium cyanide (Sutherland) and hydrazine, the former yielding cyanhæmochromogen, which possesses almost identical absorption bands to hæmochromogen. Sutherland's method is as follows [2] :—Stains on a hard surface are moistened with potassium cyanide solution (10 per cent.) scraped off and smeared on a microscope slide. Stains on a soft

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[1] F. Michel, *Chem. Zeit.*, 1911, 35,996 through *Analyst*, 1911, p. 550.

[2] Lyon's Medical Jurisprudence for India, 1914, p. 167.

frabric are immersed in boiling water for three seconds to fix the colouring matter, placed on the microscope slide and treated with potassium cyanide solution. To the stains thus prepared add a few drops of yellow ammonium sulphide solution, cover with a cover-glass, absorb the superfluous fluid by blotting paper, and examine. If blood is present, the spectrum of cyanhæmochromogen will be seen at 540-525, the latter, however, being sometimes so faint as to be almost or quite invisible.

The hydrazine solution recommended by Riegler is made as follows :—10 grams of sodium hydroxide and 5 grams of hydrazine sulphate are dissolved in 100 c.c. of water and 100 c.c. of 96 per cent. alcohol are then added. After standing a few hours the mixture is filtered. The suspected stain is treated with a few drops of the mixture.

**Testing the Stain.**—It is not necessary to bring the blood into solution for observing absorption spectra; in fact, observation is easiest when a minute amount of blood matter, such as a clot or stained fibres of the fabric, can be brought into the field of the microscope in a sufficiently translucent condition to enable enough light to pass to give visibility of the spectrum. No objective smaller than ¼ inch should be used, or not enough light may be transmitted. If solutions of blood are prepared these are usually so dilute that a miniature observing cell must be made and filled. This is always necessary when the stain is on dark or opaque material. The micro-spectroscope having been fitted into the microscope, a powerful light, that of a demi-watt filament lamp being recommended, is concentrated by the substage condenser and a brilliant spectrum produced with as small an opening of the slit as possible. The scale is now adjusted so that the sodium line, or the " D " line if sunlight is being used, lies at the correct position, namely 589.

For the comparison spectrum the mirror is adjusted so that a brilliant spectrum is formed and a small tube containing diluted blood or a fragment of blood-stained cotton is treated with a few drops of the hydrosulphite reagent and placed in the clip at the side so that the bands of hæmochromogen are clearly seen. The concentration of blood should be such that the bands are distinct but not too marked. These consist of an intense narrow band at 556 (mean) which is the most marked and easily produced band of any of the hæmoglobin derivatives, and a wider, fainter band at 530 (mean). It will be noticed that blood shows a salmon-pink tint on addition of the hydrosulphite reagent. This is characteristic. The width of the bands depends on the concentration of the blood.

A small portion of the stained fabric, or scrapings from the blood-stained article, are placed on a microscope slide and moistened with a drop of pyridine. A few drops of the hydrosulphite reagent are added and the slide adjusted under the objective. The reduction takes place in the cold and the slide should not on any account be heated, or the hæmochromogen spectrum will disappear. In most cases the spectrum is readily seen at once and can be identified with that of authentic hæmochromogen by the comparison spectrum. It is necessary to use a small slit and move the slide about until the spectrum is observed.

If the amount of blood is very small or the material rather dark, it is best to focus a salmon-pink tinted blood fragment in the centre of the field before observing the spectrum. When as large a piece as possible is found, this is brought into the centre of the field and the spectrum of the light traversing it observed.

If the material is too opaque, even when teased or squeezed out, to show a pink zone giving the spectrum, an extraction with pyridine for about half an hour, the length of time depending on the solubility of the blood, should be made and the concentrated pyridine extract treated with the hydrosulphite reagent. If necessary a simple cell for examining the solution can be made by cutting a length of one inch off the bottom of a 3 × ¼ inch sublimation tube and supporting this in a hole in a piece of cork. The blood extract is run into this cell with a pipette and the cell so arranged that the light passes through it longitudinally, when, unless the solution is very dilute, the intense band 556 should be seen. If this fails, prolonged extraction with 10 per cent. alkali, or pyridine should be tried, but probably the blood will be so insoluble that either Beam's hæmatin test or the hæmatoporphyrin test must be applied.

This test has been found to give almost instantaneous results with recent blood stains on white or slightly coloured fabrics. With blue (indigo-dyed) garments the indigo can be completely bleached in a few minutes by performing the test in a covered watch-glass to exclude air, leaving the red hæmochromogen stain clear and visible on the fabric. With fresh stains on knives or earth the test succeeds but requires more careful observation and focussing. The test is sensitive, even old insoluble stains on white cotton fabrics giving a distinct spectrum. The presence of the intense band is considered sufficient proof of the presence of blood, assuming that the control tests with unstained material in cases of dyed substances are satisfactory.

HÆMATOPORPHYRIN.—Hæmoglobin when digested with concentrated sulphuric acid decomposes into an iron-free purple pigment, hæmatoporphyrin, which shows three absorption bands, one at 608–594, and two others lying so close together as to appear often as one broad band 584–572 and 572–548. The bands are not so intense as that of hæmochromogen. The dry substance or fabric should be cautiously and gently warmed with concentrated sulphuric acid, avoiding charring, and the purple-coloured zone focussed and observed. A control test should be made by grinding a small quantity of dried blood up in a glass mortar with 1 c.c. of concentrated sulphuric acid. Only strong acid should be used, as moisture tends to precipitate the hæmatoporphyrin.

In alkaline solution hæmatoporphyrin exhibits four bands—620–612, 594–568, 553–536, 527–488. An alkaline solution can be made from the acid preparation by draining off the acid and adding concentrated potassium hydroxide solution. The alteration in the position of the bands affords a perfect proof of the presence of blood.

This test is especially applicable to old or insoluble blood and, if negative, there is no other test that is likely to succeed. It should be regarded as the last resort for difficult stains. It is not so easy of application as the hæmochromogen method.

OXYHÆMOGLOBIN.—In recent stains the spectrum of this substance showing two bands, a narrow sharp band at 579 (mean) and a broader less marked band at 542 (mean), may be seen, with the bands of methæmoglobin possibly mixed with them. These spectra cannot be considered conclusive alone and the hæmochromogen spectrum must be produced by addition of a reducing agent.

*Possible Fallacies of the Spectrum Test for Hœmoglobin.*—The presence of dye may entirely mask the spectrum. Certain substances such as alkanet root, madder and cochineal exhibit absorption spectra simulating that of oxyhæmoglobin, but none of these substances show a spectrum similar to that of hæmochromogen. In every case in which dyed or coloured fabrics are examined, a control test should be applied, and stains or matter which do not give the benzidine test but which show absorption spectra must be confirmed by the hæmin test. Such a case, however, has never yet been met with in Egyptian practice. If possible two spectra should be observed or the change in spectrum on the addition of reagents.

**Origin of the Blood.**—This is determined by means of a biochemical examination. The details of the method vary in the hands of different workers. Dr. W. H. Willcox's description is as follows[1]:—

"In performing the test on blood stains the suspected stains are treated with a solution of salt of the same strength as that of blood serum (normal saline).

"The solution of the stains is clarified by centrifugalization and to the clear solutions in different small test tubes a few drops of different anti-sera are respectively added.

"If the stain is one of human blood, a precipitate will be given with a human anti-serum, but not with sheep or ox or other anti-serum than man.

"The test is a reliable one provided that a series of sensitive anti-sera are available and provided also that the blood stain is sufficiently large to give a solution of blood serum of sufficient strength to give a *marked obvious precipitate* when the test is applied."

Lieut.-Colonel Sutherland, Director of the Imperial Seriological Institute, Calcutta, uses the following method[2]:—

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[1] Circumstantial Evidence. *Wills,* 1912, p. 165.

[2] Lyon's Medical Jurisprudence for India, 1914, pp. 174–180.

"When the observer desires to determine the origin of a blood stain he makes an extract of it by soaking the stained fabric or scrapings of the stain in physiological salt solution. Some stains are hard to extract and for these the addition of a few drops of solution of potassium cyanide to the salt solution in which they are immersed is a good plan. The stain extract is then tested as to its alkalinity or acidity. If it be acid it must be rendered neutral or slightly alkaline by the addition of a drop or two of a weak solution of caustic soda or potassium cyanide. If it be strongly alkaline, as it will be if the cyanide solution has been used to hasten extraction, it must be rendered only slightly alkaline by the addition of a drop or two of a solution of tartaric acid.

"The extract having been thus treated is diluted with salt solution until it corresponds to a 1 : 1000 dilution of serum.

"The observer having made water-clear extracts of all the blood stains that have to be examined, sets these out in taper tubes, and to the contents of each adds two drops of an anti-human serum, which he knows is on the day in question specific and highly potent. Those extracts which within twenty minutes give a distinct reaction are noted and the stains from which they have been obtained are proved to have been caused by human blood."

*Possible Fallacies.*—The extract of the stain must not contain any free acid or alkali or any acid or alkaline salts. If the extract is acid a precipitate will be produced with any anti-serum, while if alkaline the precipitate which tends to form may remain in solution or its formation may be retarded beyond the specified interval. Acid stains should be extracted with saline containing a little magnesium oxide in suspension; alkaline stains should be carefully neutralized with dilute tartaric acid. Extracts should be made only with physiological saline solution, never with tap water or even distilled water as these latter sometimes give a turbidity with anti-sera. The anti-sera from different rabbits, even if of the same kind, should never be mixed.

**Mammalian Reaction.**—Precipitin anti-sera contain anti-sera for many proteins and with powerful sera and strong blood solution a faint turbidity due to a precipitin common to all mammals is observed. This varies according to the anti-sera, the rabbit, and the particular blood tested, but it is often observed with all anti-sera for mammalian bloods. The reaction is very weak, but in order not to mistake it for a weak positive precipitin reaction it is necessary to use dilute blood solutions about 1 : 500 to 1 : 1000 with powerful anti-sera. The strength of blood extracts can be judged either by the "foam" test, that is to say by comparison of the duration of foam on blowing into it with that produced by blowing into a known 1 : 1000 dilution of blood serum in normal saline (Bubbles should persist for about half a minute) or by observing the turbidity produced by the addition of a few drops of nitric acid (sp. gr. 1·2) to a small quantity of the extract as compared with that produced from a solution of blood serum of known strength.

The strength of the blood extract to be used necessarily depends on the "strength" of the anti-serum at disposal. By this is meant the maximum dilution of blood serum in normal saline with which the anti-serum will produce a definite ring or turbidity within the specified time period. Sutherland observes after twenty minutes, the German observers after two minutes. In Egypt the two minutes' limit is adopted. The dilution of the blood extract should be 10 per cent. within this maximum dilution or the reaction may be missed owing to slight turbidity or to traces of alkali.

**Micro-precipitin Test.**—To overcome the difficulty of preparing clear extracts of small stains without loss or over-dilution of the extract, the following method was devised by Mr. W. M. Colles and has given good results after several years' use in Egypt :—

The stains or scrapings are placed in small square white glass blocks having a circular hollow basin-shaped cavity, such as are commonly used by pathologists for staining sections, moistened with a drop or two of normal saline and thoroughly teased until the whole fabric is wetted. Clots or dust are ground with a glass rod. At least two square centimetres of stained fabric should be taken although the test will succeed with very small stains if the albumin is still soluble. Enough saline is now added drop by drop to ensure excess of liquid beyond that soaked up by the material, so that, if the pot is tilted, supernatant liquid will flow easily to the side. Usually, four to six drops are sufficient, but as little as possible should be taken. The pot is now covered with a square of glass and placed in a damp chamber (a desiccator with water) and, in hot weather, in order to avoid putrefaction, the desiccator is put in the ice-chest and left until next morning.

In the tropics, extracts usually require at least twelve hours' extraction owing to the climatic conditions tending to cause greater insolubility of the blood than in temperate climates. If necessary, however, tests can be made after a few hours' extraction, and, if not positive or conclusive, the extraction can be allowed to proceed overnight and the tests repeated. If very spongy material such as thick wool, which soaks up much liquid, is under extraction, it is a good plan to squeeze it out with a glass rod after six hours' extraction and then leave it overnight. This ensures thorough mixing of the extract. Next morning, the pot is taken out of the ice-chest and allowed to warm up to room temperature.

A number of capillary tubes about 12 to 15 cms. long and 1 mm. diameter are made by drawing out test tubes in the blowpipe and cutting up the capillary so produced into lengths. These should be made just before use or kept stored in corked tubes, as, if dusty or dirty, the reaction is obscured.

The tubes of anti-sera having been allowed to warm up slowly to room temperature are opened and drops of the anti-sera placed on white tiles with hollows such as are used for volumetric " outside " indicators, using sterile pipettes. The tiles are labelled anti-human, anti-bovine, anti-avian and so on respectively, and 1 : 500 solutions of human, bovine or avian serum are prepared in normal saline, especially that of any blood which may be alleged by the accused to be the origin of the stains, and also a blank extract of the material or fabric upon which the blood is found. The anti-sera should now be tested as described under Control Tests.[1] The control tests having been made the suspected stains are examined as follows :—

*The Test.*—The white glass pot containing the case under

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[1] P. 40.

test is cautiously tilted on one side by pushing some microscope slides under it and a little of the supernatant liquid, which should be clear and will be coloured according to the amount of hæmoglobin present, is drawn off into a capillary tube held slightly obliquely. A column about 2 to 3 cms. long should be drawn off, but less will suffice if only small quantities of extract are available. If this is done cautiously, avoiding any matter floating on the surface, a clear column of liquid is obtained. This method has been found to give clear extracts with scrapings from such objects as rusty knives, mud, bricks, earth and wood. The clear liquid is now blown out on to a watch-glass and tested for serum concentration by blowing bubbles in it or by drawing off a minute quantity in another capillary tube and then drawing up a drop of 25 per cent. nitric acid and comparing the turbidity with that produced by one of the 1 : 500 serum dilutions treated in the same way. If the extract is too concentrated, drops of saline are added and the mixture stirred. It must also be tested for neutrality and, if not neutral, it is made so by treatment either with magnesium oxide or with dilute tartaric acid. Columns of the diluted extract of 2 to 3 cms. length are now drawn up into two capillary tubes, the tubes being held one in each hand. That in the right hand is now immersed in a drop of anti-human serum or that anti-serum corresponding to the blood which the accused is alleged to have shed. A column of anti-serum about 2 cms. in length can be readily induced to run up into the capillary by holding it obliquely. Care should be taken that no air bubbles collect between the column of blood extract and the column of anti-serum. The capillary held by the left hand is introduced into a drop of anti-serum for the blood claimed by the accused to be present or the blood supposed to be present, and a column drawn up in the manner described.

The two tubes held obliquely, one in each hand, are now viewed against the shadow cast under the working table, it having been previously arranged that no light comes from the side of or from behind the observer. In this way, the appearance of a faint colourless ring at the point of contact of the extract and anti-serum is easily detected. Within one minute or, at the most, two minutes (according to the strength of the anti-serum) a turbid ring should be observed if blood corresponding or related to the anti-serum is present. If, as is usually the case, the right hand tube is positive, human blood or the suspected blood is present. The tubes are now supported in an oblique position in plasticine on a horizontal wooden rod fixed in a retort stand and their position numbered and recorded.

If a positive result with abundant turbidity has been obtained, a control test using another capillary should be made immediately with a greater dilution and the result confirmed. At the end of five to ten minutes, the turbidity should have collected into flocks of colourless matter resembling aluminium hydrate. The second capillary tube should remain perfectly clear ; if a turbidity is produced, this may be due to mixed bloods or to acidity. If no result is observed in five minutes with the suspected blood anti-serum, the test should be repeated with undiluted extract. If negative results are obtained, tests should be made with other anti-sera using undiluted extract until a result is obtained. This, however, may not always be possible owing to lack of the necessary anti-serum. In this way, a number of tests may be made with a few drops of extract.

*Positive Results.*—If the extract from the case repeatedly gives a definite result with one anti-serum (or, at the most, with two, if related) and remains clear with other anti-sera, showing the absence of any precipitant for albumin in the anti-sera and the control test for the unstained material is clear, a positive result can be considered to have been obtained. In recording positive results, the family relationship of the blood detected should be taken into account ; for example, pigeon blood can only be reported as "avian," goat or sheep as "ovine," fox or dog as "canine."

Sutherland states that anthropoid ape blood gives practically no reaction with human anti-serum, but the defence that the blood is of anthropoid ape and not human origin is not one likely to be advanced.

Concentrated extract of human hair (after washing with light petroleum spirit) was found only to give an almost imperceptible ring after five minutes with an extremely powerful human anti-serum and a concentrated extract from finger-nail scrapings gave a negligible ring with the same anti-serum. Blood on such articles therefore can be tested for human origin with certainty if sufficient is present to give a marked reaction.

**Family Relationship.**—If the accused alleges blood suspected to be pheasant is that of a fowl, practically nothing can be done to disprove it if the blood proves avian. Similarly, a defence that cow blood is buffalo or ox blood or that sheep blood is goat blood cannot be disproved. Relationships, however, which are more distant, can be distinguished if quantitative tests are made. For example, alleged goat blood suspected to be cow blood can be distinguished by comparing the time required for a turbidity to form with anti-ovine and anti-bovine sera respectively, employing anti-sera of the same strength or differing strengths of extract to compensate for differences in the strength of the anti-sera. The amount of precipitate formed is also a useful guide.

*Negative Results.*—If a strong extract has been tested with powerful anti-serum without result after fifteen minutes and the extract is free from alkali or obvious impurity, it can be stated that the blood tested for is not present. It is manifestly more satisfactory to report the origin of the blood whenever possible but when this is not proved, it is necessary to state simply that the blood is not human, or as the case may be.

*Inconclusive Results.*—If turbidity is produced with different anti-sera, there may be a mixture of bloods present, but this is usually due to the presence of acid substances acting as a preciptant for the albumins in the anti-sera and any turbidity from this cause is generally intense and rapidly formed. The remedy is to digest the extract with excess of magnesium oxide.

TURBID EXTRACTS.—Obstinate turbid extracts which cannot be clarified by centrifuging prevent any accurate observation unless used in a concentrated form, when there is a danger of the mammalian reaction. Results can sometimes be obtained if control tests for the mammalian reaction are carried out.

**Control Tests.**—These are extremely important. The anti-serum giving a positive result must be proved specific, powerful, free from marked mammalian reaction for fifteen minutes with 1 : 100 concentration of the blood alleged present by the accused, and must remain clear when mixed with normal saline or extract of the unstained material upon which the blood was detected.

These tests are easily and quickly carried out in capillary tubes; drops of human anti-serum, for example, being tested with (*a*) normal saline, (*b*) blank extract of material, (*c*) 1 : 100 dilution of bovine, avian, equine serum, etc., in normal saline, (*d*) human serum, strength 1 : 500.

Occasionally, a batch of serum having a marked mammalian reaction is met with and should be discarded, but usually the controls remain perfectly clear for half an hour or longer. In hot weather with extracts beginning to putrefy, a general cloudiness commences in all tubes after half an hour.

The advantages of Mr. Colles' method are (*a*) that small quantities of clear extract are easily obtained even from powdered material, (*b*) that a considerable number of tests can be applied to a drop or two of extract, (*c*) that the tests are easily repeated. The anti-serum rising inside a tube wetted with the extract mixes at once with it in sufficient quantity to ensure precipitin reaction. The drawbacks of the method are twofold, namely first the difficulty experienced with porous material and, secondly, that as the extract must not be shaken or stirred the concentration of the supernatant layer may be weaker than that at the bottom of the pot. This cannot be avoided entirely but may be diminished to some extent by stirring or testing the mixture the last thing before leaving the laboratory in the evening.

**Illustrative Cases.**—In England, the result of the precipitin test was accepted in the Slough murder case tried in 1910 and in the Gorse Hall case tried the same year. It has been recognized in France and Germany for a number of years and is also regularly accepted in Australia, Canada, India, Egypt and other countries.

## BOOKS OF REFERENCE.

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## Chapter III

# BULLETS AND OTHER PROJECTILES FOR FIREARMS

The various projectiles for firearms, such as bullets, slugs and small shot, differ not only in size and shape, but also in composition, particularly if made by different manufacturers or in different countries, hence in a case of murder, attempted murder or wounding by shooting the examination and chemical analysis of the projectile may provide very valuable evidence. The different kinds of projectiles will now be described, as also wads and cartridges.

### Bullets.

Bullets are made generally either of lead or of lead coated with some harder metal. The lead employed, which is ordinary trade lead, usually contains small proportions of various impurities, among which arsenic, antimony and tin may be specially mentioned. In addition to any antimony present as an accidental impurity this metal is frequently added for hardening purposes in order to give greater penetration. One manufacturer states that for shrapnel bullets and case shot he uses seven parts of lead to one part of antimony (12½ per cent. of antimony). The lead core of the British Army bullet Mark VI contains 2 per cent. of antimony, as also does the rear portion of the core of bullet Mark VII. As already stated, bullets usually consist of a lead core (either soft or hardened) coated with some harder metal such as copper, cupro-nickel or steel or copper or steel plated with nickel, but sometimes they are of lead tipped with another metal such as aluminium or an alloy of aluminium and zinc and enclosed in a cupro-nickel envelope. The British Army bullet Mark VII is of this type. Bullets used for sporting purposes are frequently made entirely of lead and are not coated at all, or sometimes they have the end left uncovered (soft-nose). Revolver bullets are also generally uncoated. Automatic pistol bullets are always coated. Pure aluminium has also been employed for bullets as well as alloys of this metal with tungsten and magnesium, and solid copper bullets have also been used. During the war of 1914-18 rifle bullets of special material and construction were employed for armour piercing and incendiary purposes.

The exact diameter of a bullet does not always correspond with the nominal calibre; thus a nominal 0·380 bullet for an automatic pistol has sometimes an actual diameter of only 0·358, a nominal 0·32 bullet may be only 0·301 and a nominal 0·35 S. and W. only 0·320.[1] In the John C. Best trial two experts stated that a nominal 0·38 calibre rifle bullet was actually of 0·40 calibre.[2] In one case an automatic pistol bullet measured by the author had an actual diameter of 0·448 inch and was probably a nominal 0·450.

A bullet takes the imprint of the rifling of the barrel from which it is fired and may also be marked by rust or other defects in the barrel. Such marks, therefore, should always be looked for. Enlarged photographs are useful for demonstrating in Court any marks that may be found. When rifling marks are present the number and width should be determined, as also whether the twist is right-handed or left-handed. In the case of the automatic pistol bullet mentioned above, which was probably a Webley and Scott bullet, the width of the grooves was 0·04 inch and the depth 0·004 inch. Many of these factors vary with different firearms and hence may serve to indicate the particular kind of weapon used. Thus on a bullet fired from a Webley and Scott "Service" revolver or from a Webley and Scott automatic pistol there are seven main grooves and these are not parallel to the principal axis of the bullet but are slightly inclined to the right, while on a bullet fired from a Colt automatic pistol there are only six main grooves and these are inclined to the left. In most cases there are also other characteristic markings between the grooves. It should not be forgotten, however, that cartridges are not always employed

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[1] Automatic Pistols. *H. B. C. Pollard.* 1920, p. 99.

[2] Trial of John C. Best. Boston, pp. 570, 605.

with the particular kind of weapon for which they are designed. In the Dickman case for example four bullets were found, two being uncoated lead bullets, 0·320 calibre, and two being nickel-coated automatic pistol bullets, 0·250 calibre. The editor of the account of the trial states that all four bullets had been fired from the same weapon, the smaller cartridges having been wrapped in paper to make them fit the barrel. One such piece of paper was found, but its significance was not realized at the time. These facts did not come out in evidence and the presence of two different kinds and sizes of bullets puzzled both judge and counsel.[1]

On a lead bullet which has passed through clothing it is sometimes possible to see an imprint of the texture of the cloth first penetrated.[2] In order to identify this an enlarged photograph is taken of the imprint and of similar imprints made by firing bullets of the same kind and size into the suspected clothing.

**Slugs.**—Slugs are composed of soft lead, which, being of commercial quality, generally contains small proportions of antimony and tin and occasionally traces of arsenic. Two makes of cartridges have been examined. One (Remington) contained seven slugs which varied in weight from 1·65 grams to 1·82 grams each, and the other (Sneider) contained eleven slugs with weights varying from 1·78 grams to 1·83 grams each. Eight of the former and two of the latter were analysed. In the first case four of the slugs contained no tin and four contained tin, which varied in amount from 0·17 to 0·55 per cent. and all contained antimony, the amount varying from 0·30 to 0·90 per cent. In the second case the amount of tin was 0·23 per cent. in both slugs and the antimony 0·54 and 0·60 per cent. respectively. Arsenic was present in several instances, but only as a trace. Slugs from different cartridges may vary considerably in shape, some being almost cubical and others much flattened.

**Small Shot.**—Small shot practically always consists of lead to which a small proportion of arsenic (variously stated to be from one-tenth per cent. to five per cent.) is added for the purpose of enabling the lead to assume the spherical

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[1] Notable English Trials. *J. A. Dickman*, pp. 11–89.

[2] Précis de Médicine Légale. *V. Balthazard.* Paris, 1911, p. 239.

shape when the molten metal is cooled by being dropped into water. On one occasion some small shot examined was found to be iron, it was coated with graphite, was irregular in shape and varied in size in the same cartridge.

The question of the size of the shot is sometimes of considerable importance, as for example in the Monson trial.[1] In this case there were two guns involved, one a 12-bore gun with which No. 5 shot was used and the other a 20-bore gun with which No. 6 shot was used, and a great deal depended upon from which of the two guns the fatal shot had been fired. Four pellets were found in the brain of the victim, and the prosecution contended that the weight of these pellets established the fact that death had been caused by a discharge from the 12-bore gun loaded with No. 5 shot. In view of the importance of the size and weight of shot, the particulars given in the table on p. 46 may be quoted. These figures should be taken as only approximately and not rigidly correct.

The following few determinations made by the author may also be recorded :—

| | | A. | B. | C. | D. |
|---|---|---|---|---|---|
| Number of Shot . . . . . . . | | 10 | 10 | 6½ | 7 |
| Bore of Gun . . . . . . . . | | 12 | 12 | 12 | 12 |
| Number of Cartridges examined . | | 5 | 6 | 12 | 12 |
| Weight of Shot in Cartridge (grams) | Min. | 31·7 | 31·5 | 24·5 | 32·1 |
| | Max. | 32·8 | 32·2 | 31·0 | 33·0 |
| Number of Pellets in Cartridge . . | Min. | 1,377 | 983 | 184 | 383 |
| | Max. | 1,949 | 1,097 | 290 | 397 |
| Diameter of Pellets (mms.) . . . | Min. | 0·90 | 1·72 | 2·26 | 1·93 |
| | Max. | 3·50 | 1·75 | 3·07 | 2·63 |

From the above facts it is evident that not only does the number of pellets sometimes differ considerably in cartridges of the same make, but that the size of the pellets may vary in the same cartridge, and therefore great care is required in making any general deductions from the size and weight of isolated pellets.

**Analysis of Projectile.**—Whatever the nature of the projectile is, it should be not only weighed and measured but analysed.

As a rule in shooting cases the amount of material available

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[1] Notable Scottish Trials. *A. J. Monson*, pp. 58, 91, 359, 365.

for analysis is very small, since not only is the total amount small, but of this as large a quantity as possible must be reserved for further analysis or for production in Court. In such cases where a comparison between several different samples of lead only is required the following method will be found useful:—Take half a gram of each sample in a finely divided condition, put into separate small conical flasks

Details of Shot Sizes.[1]

| Designation. | Pellets per oz. | Weight per Pellet. | Diameter. |
|---|---|---|---|
| | | Grains. | Inch. |
| LG | 5 | 87·5 | 0·388 |
| MG | 7 | 62·5 | 0·347 |
| SG | 8 | 54·7 | 0·332 |
| *Special* SG | 11 | 39·8 | 0·298 |
| SSG | 15 | 29·17 | 0·269 |
| SSSG | 20 | 21·89 | 0·245 |
| SSSSG | 25 | 17·50 | 0·227 |
| SSSSSG | 30 | 14·58 | 0·214 |
| AAA | 35 | 12·50 | 0·203 |
| AA | 40 | 10·94 | 0·194 |
| A | 50 | 8·75 | 0·180 |
| BBB | 60 | 7·29 | 0·170 |
| BB | 70 | 6·25 | 0·161 |
| B | 80 | 5·47 | 0·154 |
| 1 | 100 | 4·38 | 0·143 |
| 2 | 120 | 3·65 | 0·135 |
| 3 | 140 | 3·12 | 0·128 |
| 4 | 170 | 2·57 | 0·120 |
| 4½ | 200 | 2·19 | 0·113 |
| 5 | 220 | 1·99 | 0·110 |
| 5½ | 240 | 1·82 | 0·107 |
| 6 | 272 | 1·61 | 0·102 |
| 6½ | 300 | 1·46 | 0·099 |
| 7 | 340 | 1·29 | 0·095 |
| 8 | 450 | 0·97 | 0·087 |
| 9 | 580 | 0·75 | 0·080 |
| 10 | 850 | 0·51 | 0·070 |

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[1] From The Shooter's Year Book, 1920–21, by permission of Explosives Trades Ltd., London.

and add to each 5 grams of ammonium sulphate[1] and 10 c.c. of strong sulphuric acid and heat strongly until the metal is all dissolved and the precipitated lead sulphate is white or practically white. Although the bulk of liquid is small, there is no fear of the flask breaking. The lead is fully precipitated as sulphate and any arsenic, antimony and tin present remain in solution.[2] Cool and add to each flask 25 c.c. of a 10 per cent. solution of tartaric acid. This prevents the antimony and tin from being precipitated by the water, which, if it were not added with the tartaric acid, would have to be added to dilute the solution before filtration. Shake well and boil for five minutes in order to granulate the sulphate of lead. Filter into Nessler tubes, using ordinary filter paper and making no attempt to transfer all the lead sulphate to the paper, and wash with dilute sulphuric acid (10 per cent. solution) using a minimum quantity and approximately the same amount in each case. To the filtrates add a saturated solution of sulphuretted hydrogen. Make up to the same volume (50 c.c.) with water in each case, stir well with a glass rod and compare. The contents of the tubes may be transferred to glass stoppered bottles and preserved for presentation in Court.[3] If tall narrow bottles of clear glass are used, the different amounts, colours and general appearance of the various precipitates are very marked and demonstrate the results in a manner that no mere recital of percentages can possibly do. If required, this method may be made quantitative, in which case, however, the filtration from the lead sulphate should be through asbestos, using a Gooch crucible. The antimony and tin are best determined volumetrically, after separating any arsenic present, the former by means of potassium permanganate and the latter with iodine.

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[1] If more than about 5 per cent. of tin is present, the ammonium sulphate should be omitted.

[2] Owing to the fact that stannic sulphate tends to form and that this compound is insoluble in strong sulphuric acid, there is danger of some tin being left with the lead, and the lead sulphate therefore should always be tested for tin.

[3] If the bottles are completely filled and are provided with well-fitting stoppers coated with wax, the contents remain practically unchanged for a considerable time.

The precipitate formed on the addition of sulphuretted hydrogen will be composed of one or more of the following named compounds :—(*a*) Arsenic sulphide ($As_2S_3$) : lemon yellow. (*b*) Antimony sulphide ($Sb_2S_3$) : orange. (*c*) Tin sulphide ($SnS_2$) : yellow. Copper sulphide, which is sometimes present in small amount, will darken the precipitate.

The analysis is continued as follows :—Filter, using a small paper, and wash with sulphuretted hydrogen water. Transfer the paper with the precipitate to a small beaker and warm with 10 c.c. strong hydrochloric acid until the sulphides of antimony and tin are dissolved and the sulphuretted hydrogen is all expelled, which may be tested by removing a small portion of the solution with a pipette, and diluting it largely with water. If a coloured precipitate is produced, the operation is not yet complete and the portion tested must be returned to the main bulk of solution and the heating continued. The liquid must not boil, or chloride of antimony may be lost. When all the sulphuretted hydrogen is expelled, dilute the liquid with water. If antimony is present, a slight milkiness due to basic chloride is produced. Add dilute hydrochloric acid drop by drop until the liquid clears. Filter through asbestos, using a Gooch crucible, and wash well with water acidified with hydrochloric acid.

*Residue.*—This will consist of arsenic sulphide. If more than a trace, the amount may be determined quantitatively as follows :—Wash the residue on the filter paper with water, dry and weigh. Treat repeatedly with hot ammonium carbonate solution, which dissolves the arsenic sulphide wash, dry and again weigh. The difference between the two weights represents arsenic trisulphide. Arsenic may also be separated from antimony and tin by distillation, the method depending upon the volatility of arsenic trichloride. The arsenic is determined volumetrically by titration with iodine.

*Solution.*—Dilute to 200 to 250 c.c. Titrate with $\frac{N}{20}$ potassium permanganate solution.

$$5SbCl_3+16HCl+2KMnO_4=5SbCl_5+2KCl+2MnCl_2+8H_2O$$

The potassium permanganate should be standardized with tartar emetic, which contains 36·16 per cent. of antimony. Copper in small amount does not interfere.[1] After titration of the antimony boil the solution down to about 150 c.c. Add 50 c.c. strong hydrochloric acid and about 0·05 gram metallic antimony dissolved in 5 c.c. strong sulphuric acid. Add a small rod of pure soft iron. Both the antimony and iron are for the purpose of reducing the tin to the stannous condition, and either may be used alone. Nickel is sometimes employed instead. Boil gently for about an hour. Transfer the solution quantitatively, without the iron, by means of boiling water to a small conical flask containing about two inches of pure thin iron wire and a small quantity of hydrochloric acid which have been generating hydrogen for about two minutes. Boil gently until all the wire is dissolved (about five minutes). Cool in ice in an atmosphere of carbon dioxide. This is essential to avoid oxidation of the stannous chloride. When cold, titrate with $\frac{N}{10}$ iodine solution, using starch as indicator.

$$SnCl_2 + I_2 + 2HCl = SnCl_4 + 2HI$$

Iron, lead and antimony do not interfere. Copper in quantities below 10 per cent. does not interfere.[2]

**Wads.**—Not only may the examination and analysis of the projectile prove of value, but the nature of the wads, if any, may also provide valuable evidence. In shot-gun cartridges there are two varieties of wads, generally one felt wad and three card wads, and these differ very much in different makes of cartridges. Thus the felt differs in colour and in thickness, and the card wads also differ in thickness, and may be faced with waxed paper, white glazed paper, black glazed paper or coloured paper, and the wad situated at the end of the cartridge immediately over the shot is generally covered with a paper label (often coloured) giving the description of the powder and the size of the shot. Even in the same make of cartridge the wads are not always uniform. Sometimes a composition, one kind of which is called " Feltine," is used in place of felt, or a com-

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[1] *J. Amer. Chem. Soc.*, 1907, p. 72.

[2] Standard Methods of Chemical Analysis. *W. W. Scott.* New York, 1917, p. 427.

bined cloth and card wad may be substituted for one of the card wads.

The following table shows the relation between the diameter of the wads and the bore of the gun [1]:—

| Bore of Gun. | Diameter of Card Wadding. Inches. |
|---|---|
| 4 bore | 0·948 |
| 8 bore | 0·845 |
| 10 bore | 0·784 |
| 12 bore | 0·738 |
| 14 bore | 0·702 |
| 16 bore | 0·671 |
| 20 bore | 0·623 |
| 24 bore | 0·587 |
| 28 bore | 0·557 |
| 32 bore | 0·509 |
| 0·410 bore | 0·414 |
| No. 3 L.S. | 0·305 |

The $2\frac{1}{2}$ in. case is actually $2\frac{9}{16}$ in. long in 12, 16 and 20 bore sizes. In other calibres the nominal length is also the real.

Card Wads.—A plain card wad, one-twelfth of an inch thick, should be used over the powder, and a similar one over the felt. The overshot card wad should be one-sixteenth of an inch thick.

Felt Wad.—The thickness of the felt wad, whether three-eights, seven-sixteenths, half-inch, or any other size, must be adjusted according to the amount of powder and shot, so as to ensure a compact cartridge and a properly made turnover.

Frequently the card wad which comes into immediate contact with the shot bears indentations and marks made by the pellets, from which the number corresponding to the area of the wad, and hence their size, can be determined. Occasionally the paper label from the end of the cartridge, which bears the description of the powder and the size of the shot, is found intact. In the Monson case, already referred to, a blood-stained wad found near the body corresponded in size with those from the cartridges for the 12-bore gun.[2] In rifle, revolver and automatic pistol cartridges there is never more than one wad and sometimes not even that. When a wad is present it is a glazed or waxed cardboard disc placed between the powder and the bullet.

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[1] From The Shooter's Year Book, 1920-21, by permission of Explosives Trades Ltd., London.

[2] Notable Scottish Trials. *A. J. Monson*, p. 374.

**Cartridges.**—When dealing with cartridges these should be measured and all marks indicating the manufacturer, place of manufacture, date or size should be recorded. The cartridge should then be opened, and the bullet or pellets, the charge, the wads and the case all separately weighed, measured and examined and the charge analysed. The number of the pellets should be specially noted. The easiest way of extracting a bullet from a cartridge is to put the bullet point downwards, and surrounded by paper to protect it from injury, in a small vice or clamp and to work the case gently from side to side until it is felt that the bullet is quite loose, when it is removed from the vice and extracted with the fingers. To examine a shot-gun cartridge this should be laid on its side and cut through about the centre with a sharp knife, when the charge will be found in one half and the pellets in the other, each kept in place by a wad.

It may be mentioned that cartridges belonging to revolvers and automatic pistols respectively may be distinguished in several ways ; thus revolver cartridges invariably have a rim at the base and the bullet is not coated, while automatic pistol cartridges either have no rim at all or an almost imperceptible one, but there is a recess round the base for the extractor to engage in and the lead of the bullet is always coated with some harder metal such as cupro-nickel or copper plated with nickel. The reason for omitting rims in automatic pistol cartridges is to prevent the bullets from jambing in the magazine.

Sometimes the expert may be asked to express an opinion as to the nature of a projectile which has been distorted by impact against something hard, whether for instance it is a rifle bullet, a revolver bullet or slug.[1] No opinion, however, should be given unless the evidence is very definite and unless the expert has had previous experience. As examples of the difficulties in such cases it may be mentioned that the envelope of a coated bullet may sometimes be entirely stripped off and detached from the lead core and that a square slug may become so distorted as to make an almost round hole and when found may be oval or round with definite

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[1] The weight of the bullet and the weight and thickness of any casing of harder metal are useful indications.

"mushrooming" and may show little or no evidence of ever having been square.[1] Other problems such as those concerning the spread of shot, the distance and direction from which a firearm has been fired, should be left to sportsmen or gunsmiths or others having considerable experience in these matters. For an example of how experts may differ in such cases, the trial of A. J. Monson, which has already been alluded to, should be consulted. In connection with the spread of shot the following details may be quoted[2] :—

DIAMETER OF SPREAD.

Being the Diameter in Inches covered by the whole charge of a Gun at various Ranges for all Calibres.

| Boring of Gun. | Range in Yards. | | | | | | |
|---|---|---|---|---|---|---|---|
| | 10 | 15 | 20 | 25 | 30 | 35 | 40 |
| True Cylinder | 19 | 26 | 32 | 38 | 44 | 51 | 57 |
| Improved Cylinder | 15 | 20 | 26 | 32 | 38 | 44 | 51 |
| Half Choke | 12 | 16 | 20 | 26 | 32 | 38 | 46 |
| Full Choke | 9 | 12 | 16 | 21 | 26 | 32 | 40 |

**Illustrative Cases.**—1. Some white metal adhering to a piece of lead with which a man had been shot, but fortunately not wounded, was found not to be nickel, as was at first supposed, and which would have indicated a nickel-coated bullet, but silver from a cigarette case which had been penetrated. A night watchman suspected of the crime escaped conviction because the projectile did not agree in composition with the slugs in the cartridges with which he was supplied.

2. A man, who was suspected of wounding another by shooting, escaped conviction because the shot from a cartridge seized in his house was found on chemical analysis to differ in composition from the shot extracted from the

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[1] The presence of straight lines or slight ridges, which are sometimes all that remain of what have been the edges or corners of the slug, should be specially looked for.

[2] From The Shooter's Year Book, 1920-21, by permission of Explosives Trades Ltd., London.

wounded man, the former containing a comparatively large amount of tin and the latter containing no tin but a trace of antimony.

3. In a somewhat similar case to the one just mentioned a night watchman shot at some unknown persons suspected to be thieves who, however, all escaped. In the morning, blood was found on the ground and subsequently a man was arrested with a shot wound in his leg for which he failed to give any satisfactory explanation. Analysis, however, proved that the lead extracted from the wound did not agree in composition with the slugs in the watchman's cartridges and consequently the man was released. The impurities present in the lead were the same in each case and consisted chiefly of antimony, but the shot from the wound contained much less antimony than the slugs.

4. Two slugs found in the house of a person accused of murder by shooting agreed in composition with the lead extracted from the brain of the victim.

5. A distorted slug and six small pieces of very thin doubled-up sheet lead were extracted from the body of the victim. The slug contained 0·66 per cent. of antimony and no tin, which was exactly the composition of the slugs in some suspected cartridges. These cartridges contained seven slugs enclosed in a thin lead sheath. This sheath was of pure soft lead free from both antimony and tin. The six pieces of the sheet lead from the body of the victim were also pure soft lead free from antimony and tin and the lead was exactly the same thickness as the sheath from the cartridges. There was no doubt therefore that the cartridge with which the man had been shot was identical with those examined, and that the small pieces of lead were from the same cartridge as the slug and not from a second shot as at first seemed possible.

6. In the John C. Best trial in America two lead bullets with which a man had been murdered were identified by experts as having been fired from a particular rifle, the identification being based on the kind and size of bullet, the number and width of the marks made on the bullet by the rifling of the barrel, and also by other distinctive and characteristic lines and marks on the bullet caused by rust in the

barrel. One of the experts forced a bullet similar to the two taken from the body of the murdered man through the rifle in question by means of a steel rod, and produced markings, including those caused by the rust in the barrel, similar to the marks found on the two bullets with which the murder was committed.

## BOOKS OF REFERENCE.

Criminal Investigation. *Hans Gross.* Trans. Madras, 1906.
Notable Scottish Trials. A. J. Monson. Edited by *J. W. More.*
The Official Report of the Trial of John C. Best for Murder. Superior Court of Massachusetts. Boston, 1903.
The Book of the Pistol and Revolver. *H. B. C. Pollard.* London, 1917.
Automatic Pistols. *H. B. C. Pollard.* London, 1920.
Text Book of Small Arms. War Office, London, 1904.

## Chapter IV

# CLOTHING

There are few articles which afford so many clues to the personality, habits and history of the owner as clothes and few articles better repay careful examination. This examination is in the main chemical, and therefore work for the chemico-legal expert. Clothes may be considered under various aspects, as for example in relation to the nature of the material of which they are made, their age and state of repair, and the stains, marks, dust and dirt which may be found upon them. There are also other features, too numerous to specify, which should be taken into account and each case must be considered as a separate and special study.

Before being sent to the expert, clothes, if wet, should always be dried, but this must be done with care and with as little handling as possible. Each garment should be wrapped in a separate sheet of clean paper, tied, sealed and marked with a distinguishing number or letter. The parcel or parcels should then be further packed in stout paper or in a box.

For the purposes of examination, the clothes should be either hung up or spread upon a large table. A good light is essential, and daylight is preferable to artificial light. The examination may be very disagreeable, as the clothes are frequently blood-stained, sometimes wet and generally dirty.

The kind of garment, the material, colour and condition are all recorded. In some cases clothes are submitted for a specified purpose, such as to ascertain whether there are stains of a particular kind or for the determination of the nature of certain manifest stains or for the examination of holes with a view to ascertaining how they have been caused, whether for instance by acid, burning, shot or bomb fragments. In such cases, beyond a general inspection of the clothes to enable them to be described in the report and identified again in Court, the examination is limited to the specific purpose required. In other cases the clothes must be examined in detail, special attention being directed to any tailor's name or mark, to any inscription on the buttons, to the nature and condition of the lining, to any holes or repairs, and particularly to the pockets, and all dust and dirt in the pockets should be carefully examined. Much of this work is not chemical, but the material of the clothes, the stains, the dust and the dirt on the clothes and in the pockets may all require analysis.

In the case of stains and holes, the position, size and general appearance should be recorded, and it will be found very helpful in writing the report and for future reference if a sketch giving these particulars is made in the laboratory note-book at the time of the examination, or better if a photograph is taken of them. This latter, however, is not always possible.

Nature of Material.—The method of examination will be described under Fibres and Textile Fabrics respectively.

Blood Stains.—These have been considered in a previous chapter.

Seminal Stains.—These will not be dealt with, as the method of examination is not chemical.

### Stains other than Blood and Seminal Stains.

All stains before being tested should be examined with a lens. As a rule before applying any tests the substance producing the stains should be dissolved out from the cloth. In order to do this, however, the solvent should not be applied directly to the garment, as this causes the stains to spread and results in considerable loss, and also because a large amount of solvent is required, which is soaked up by the cloth and must be squeezed out again, a process which it is impossible to carry out without contamination and further loss. The best procedure in most cases is to cut out portions of the stains and soak them in the appropriate solvent. The whole stained area should not be taken, sufficient always being left for subsequent reference and further examination if necessary. The solution obtained is then tested, and reliance should never be placed upon the result of any one test, but as many confirmatory tests as possible should be applied. A control experiment should always be made with a portion of the cloth free from stains.

**Acid Stains.**—The presence of acid stains on clothes, and holes caused by acid, is frequently very useful as evidence in connection with such crimes as acid-throwing, the making of explosives or bombs and the forgery of banknotes, in which latter process acid may be used for etching purposes.

In order to ascertain whether a given stain is acid or not, a piece of blue litmus paper moistened with distilled water is pressed against the spot. This should be done by means of a glass rod, and never with the fingers, as in warm weather or in a hot climate the fingers may be sufficiently acid with perspiration to redden the litmus.

Sulphuric Acid.—Strong sulphuric acid, which is the acid generally used for throwing at the person, and which is also used for making explosives and bombs, acts energetically upon cotton and linen fabrics, destroying the material and almost instantly producing holes: on woollen fabrics the action is less pronounced. The colour produced upon clothes by sulphuric acid depends to some extent upon the colour of the cloth and the nature of any dye present, and varies generally from red to reddish brown, though sometimes it may be black.

The best solvent for sulphuric acid is water. Taylor recommends[1] that the stained fabric should be digested in rectified spirit at a gentle heat. This is intended to avoid the solution of sulphates. Sulphates, however, although insoluble in absolute alcohol, are slightly soluble in rectified spirit, and are dissolved by weak alcohol, and therefore if the clothing is wet or the alcohol not very strong misleading indications might be produced. Sodium hydrogen sulphate too is readily acted upon by alcohol with the liberation of free sulphuric acid.[2] Alcohol also is always slightly acid,

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[1] Principles and Practice of Medical Jurisprudence, 1920, Vol. II, p. 391.

[2] The Action of Alcohol on the Sulphates of Sodium. *G. S. Butler and H. B. Dunnicliff.* Chem. Soc. Trans., 1920, pp. 649–667.

and this might introduce an error, since acid is what is being looked for. Water is the best solvent for sulphuric acid, and should always be used. If, however, there is a large area of staining, it is useful to cut out an additional portion and extract it with alcohol and make a confirmatory test. The best tests for sulphuric acid are the acidity test with litmus and the white precipitate with barium chloride insoluble in hydrochloric acid.

NITRIC ACID.—Nitric acid may be used criminally in the making of explosives or for etching purposes in connection with the forgery of banknotes, and is occasionally used for throwing at the person, and has been so employed on several occasions in Egypt. This acid is not so immediately destructive of clothing as strong sulphuric acid, but the stained areas, which are generally of a yellow or yellowish-red colour, eventually become very friable and break into holes.

Nitric acid should be extracted with water, and is best tested for as follows :—Add a few drops of sodium carbonate solution to prevent loss of free acid by volatilization and evaporate the solution to dryness over a water-bath in a small platinum or porcelain basin ; moisten the residue with phenol-sulphonic acid, allow to stand for a few minutes, and transfer by means of a jet of water from a wash-bottle to a small beaker, using the minimum amount of water possible : add strong ammonia in excess. A bright yellow colour indicates nitric acid. Any brown coloration should be disregarded.

In one case of acid-throwing fragments of a bottle were submitted for examination. This contained traces of acid, and the neck of the bottle was stoppered with a screw of paper which contained crystals of oxalic acid. The acid in the bottle, however, proved to be nitric acid and not oxalic acid.

HYDROCHLORIC ACID.—This is less used for criminal purposes than either sulphuric or nitric acid. It is also less energetic in its action than either of the acids mentioned. Stains on dark clothes caused by hydrochloric acid are usually of a red or reddish-brown colour. This acid is best extracted with water, and is tested for in the usual way by means of silver nitrate, with which it forms a white precipitate insoluble in nitric acid but readily soluble in ammonia.

In all cases of acid stains on clothes, portions of the unstained material must always be tested as a control, since sulphates and chlorides may be present originally in the cloth.

Picric Acid.—In several instances in connection with bomb outrages in which picric acid was employed as the explosive material, the presence of this substance on clothing has provided valuable evidence. The best solvent for picric acid is alcohol. The alcohol solution is evaporated to dryness on a water-bath, and the acid re-dissolved in water and tested. The most satisfactory tests are the colour, the acidity to litmus, the extremely bitter taste, the dyeing properties with wool and silk, the red coloration on warming with a solution of potassium cyanide and the green precipitate with ammonia-copper sulphate, which latter, however, may not be formed for about twenty-four hours if the solution is very dilute. The dyeing test should always be done, since the result, which is very conclusive, is one that can be exhibited in Court.

**Oil and Grease Stains.**—Oil and grease stains on clothing are sometimes of importance, particularly stains of paraffin oil (kerosene) in cases of suspected incendiarism. Generally such stains are very manifest, but if the amount present is small and the clothes dark coloured or dirty they may not be visible. Oil and grease, however, can always be detected by placing the garment between several layers of tissue paper or blotting paper and ironing with a hot iron. The best solvent for oil and grease is ether or petroleum ether, but if paraffin oil (kerosene) is suspected petroleum ether should on no account be used, as it may, and frequently does, contain a little paraffin oil, and in such cases ether or chloroform should be employed.

**Shot Marks.**—The chemical examination of shot marks on clothing may occasionally prove of value, and Lochte states [1] that black powder fired at short range leaves an alkaline residue in the cloth which gives the reaction for

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[1] E. H. Th. Lochte, *Chem. Ztg.* 35, 1107, through *Chem. Abst.*, 1912, p. 3184.

potassium. Potassium nitrate, however, is a component of some smokeless powders, and therefore the presence of potassium is not conclusive evidence of the use of black powder. Lochte also states that when a greased bullet is used, which is wrongly said to be only the case with black powder, the grease is rubbed off on passing through the clothing and may be recognized by transferring it to tissue paper by means of a hot iron. The presence of grease round a shot hole, however, is an indication that should be interpreted with the greatest caution, since grease is sometimes used with steel-coated bullets employed with smokeless powder, as is the case with the Austrian military bullets.[1] The external lubrication of bullets is not permitted in the British Army, but that part of the bullet which enters the case is coated with beeswax. This is very usual in both rifle and revolver ammunition, the wax being held in recessed rings or cannelures round the base of the bullet inside the cartridge case. Sometimes greased or waxed paper is wrapped round the bullet. Wax, if present on clothes, leaves a stain on paper like grease when ironed with a hot iron. A waxed or greased wad might also cause a similar stain.

**Dust and Dirt.**—To remove dust from a garment for the purpose of examination the use of a miniature vacuum cleaner has been suggested, but unless all parts of this could be taken to pieces and thoroughly cleaned such an apparatus would be most dangerous and might give rise to very misleading indications, as contamination with dust previously collected could not be avoided.

In a case mentioned by Hans Gross,[2] a coat, from which it was desired to examine the dust, was placed in a strong paper bag and then beaten with a stick and the dust collected. This method is very satisfactory.

The pockets of clothes should always be carefully examined, and any dirt or dust present should be collected and its nature determined.

**State of Preservation of Clothing.**—The state of preservation of clothes and other buried articles depends not only on the nature of the clothes or articles themselves,

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[1] Text Book of Small Arms. War Office, London, 1904.

[2] Criminal Investigation, p. 212.

but also upon the nature of the soil in which they are buried and the depth at which burial has taken place, and no general rule can be laid down. Thus, linen wrapping cloth from Egyptian mummies several thousands of years old is still frequently in an excellent state of preservation, while in other instances it is in a very friable condition or has entirely perished. During the Archæological Survey of Nubia the author had an opportunity of seeing most of the excavations near Aswan and was particularly struck by the variation in the state of preservation, not only of the bodies, but also of the perishable objects found with them. These burials are fully described in the published reports.[1]

In the Predynastic graves, which were at least 6,000 years old, the body was frequently wrapped in goatskin, and often was lying upon and covered with matting of coarse fibre or twigs, and the clothes, if any, generally consisted of a short leather kilt or loincloth. In a few cases linen was found. The wrappings were frequently in a very poor condition and sometimes only a few small pieces were left. In the early Dynastic and Old Middle Empire graves loose linen wrappings, goatskin and matting were usual.

In an early Christian cemetery on the island of Biga near Aswan, which probably dated from between the fourth and seventh centuries A.D., more than five hundred bodies were exhumed. All were wrapped in coarse white cloth, but many were also wearing clothes, which generally consisted of a white shirt, and in some instances of two or even three such shirts, but which included in one case a pair of drawers, in another case a coat (kuftan), in another case a pair of socks, and in several other cases shoes or boots. Unfortunately no critical examination of the material of the clothes was made ; the archæologist, however, states in one instance that a wrapping consisted of wool.

**Illustrative Cases.**—1. A waistcoat was submitted with

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[1] The Arch. Survey of Nubia.
Report for 1907–8, Vol. I. *G. A. Reisner.* Cairo, 1910.
Report for 1907–8, Vol. II. *G. Elliot Smith, M.D., F.R.S., and F. Wood Jones, M.B., B.Sc.* Cairo, 1910.
Report for 1908–9. *C. M. Firth.* Cairo, 1912.
Report for 1909–10. *C. M. Firth.* Cairo, 1915.

a request that it might be examined for clues concerning the immediate history of the owner, who was discovered under suspicious circumstances near the Suez Canal during the war of 1914–18. The following facts, which appeared significant, were found :—

The waistcoat was clean, in good condition, and looked almost new.

The bone button of an inner pocket bore a name which was probably that of the tailor and the address Batavia.

One of the pieces of cloth of which the lining was formed bore a portion only of a stain (probably due to perspiration) which ceased abruptly at a seam where two separate pieces of the lining joined and was not present on the other side of the seam, but was continued on the turned-down edge where the first piece of material was folded in order to sew it to the second.

The waistcoat was impregnated, both at the top and bottom, with chlorides, sulphates and compounds of calcium, magnesium and potassium.[1] These salts are all characteristic of sea water. The magnesia too was present in greater proportion than the lime, which is also a characteristic of sea water. It should not be forgotten, however, that these same salts occur in the sand of the sea-shore, in the dust on any roads near the sea, and even in the sea air. They are found also in the dust of the desert, and hence on clothes that have been worn in the desert. Chlorides might be derived from perspiration. If, in addition to salts derived directly or indirectly from sea water, dust from limestone rocks, from a limestone road or from calcareous sand was present, this would change the original proportion of lime and magnesia, and so might obscure the origin of the salts.

Each pocket contained a small amount of very fine quartz sand with rounded grains of practically uniform size, which closely resembled dune sand.

The deductions from the above facts were as follows :—

The owner of the waistcoat had bought a ready-made suit of clothes in Batavia. The stain proved that the lining

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[1] These naturally were not visible, but portions of the waistcoat were soaked in distilled water, and the solutions thus obtained examined chemically.

had been used before, since evidently the stain had not been acquired while the lining was in its existing position, and the fact of the waistcoat being almost new pointed to ready-made clothes in which some old lining had been used, rather than to a second-hand suit.

The man had travelled to Egypt in a Dutch steamer, since Dutch steamers were then passing through the Suez Canal and were the only ones that called at Batavia.

The man had left the steamer surreptitiously while it was passing through the Canal and had swum to the shore. Entire submersion, which pointed to swimming, was indicated by the fact that even the top of the waistcoat showed evidence of having been wetted with salt water, and it followed therefore that the mode of leaving the steamer was unusual and probably surreptitious.

The man had landed from the Canal at a place where there were sand dunes. This was indicated by the nature of the sand found in the pockets.

The above conclusions were subsequently proved to be correct.

2. The remains of a man in European clothing were found buried on some unoccupied land, and it was hoped that some clue to his identity might be discovered from an examination of the clothes. The following facts were noted:—

The coat and waistcoat were alike and were of thick striped, brown tweed. Parts of some bone buttons still remained, but these did not bear any tailor's name or other inscription.

The trousers did not match the coat and waistcoat, but were of thinner material, though almost of the same colour and pattern. There were six metal buttons left on the trousers, all of which were very corroded but, when cleaned, the name and address of a tailor in Duke Street, Portland Place, London, were legible.

Most of the stitching of the clothes had perished, leaving the garments in separate pieces: the lining was much decayed and the pockets were missing.

The undervest, of which only a part remained, was of cotton.

The shirt, of which only a part remained, was of coloured cotton, the predominant colour being green, but there were brown stripes which possibly had been red originally. The front was pleated and was ornamented with cross stitches in red.

The one sock was of thick red wool and in fairly good condition.

There was a part of a good quality handkerchief, which, however, bore no name.

Collar, boots, and hat were missing, but no very careful search had been made for these or other articles.

The conclusions drawn from the facts were :—

The man was most probably a European but not an Englishman, and not the man for whom the clothes had been originally made, since Englishmen do not as a rule wear green shirts with coloured stripes and pleated fronts ornamented with red stitches, neither is such a shirt worn by one who obtains his clothes from a West-end London tailor.[1]

The man was probably not destitute although the clothes were second-hand, since they were in good condition and included a good handkerchief. The one sock may be taken as presumptive evidence that at the time of his death the man was wearing two socks and also boots, as to wear only one sock, or socks without boots, would be most unusual.

Naturally only the articles forwarded could be examined and, so far as was ascertained, no systematic search had been made for others.

This case, although not chemical, is recorded on account of one very interesting feature, namely the fact that the articles made of cotton, such as the shirt and vest, the lining of the clothes and the thread used for stitching, had perished to a much greater extent than the articles composed of wool, like the clothes and the sock.

3. An Egyptian dressed in European clothes, who had been killed by having his throat cut, was found in a garden. Round his neck there was tied a gallabia,[2] probably to soak

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[1] An Englishman who had lived with people of other nationalities for many years, or who had married a non-English wife, might conceivably wear such a shirt.

[2] Loose outer garment worn by both men and women of the poorer classes in Egypt.

up the blood, and lying near the body was a small piece which had been torn from the gallabia, on which, in addition to blood stains, were black smears, marks of yellow material, much blackened, and patches of white metal in very thin layers. Analysis proved the black material to be carbon, the yellow to be resin blackened by carbon, and the metal to be plumber's solder. The small piece of material therefore had been used by a plumber for "wiping" a joint, and thus a valuable clue was afforded for the police to follow up.

There were also other clues; thus the nature of the pattern on the gallabia, which consisted of circles, indicated that the garment had belonged to a woman or girl, since men's gallabias are plain or striped and never have patterns of circles, spots or flowers,[1] while women's gallabias, unless plain, possess these patterns and are seldom, if ever, striped. The gallabia had also a pleat down each side of the upper part of the front, which again indicated a woman's or child's dress, and was very much torn at the front and had a patch of different material inserted from inside in order to mend one of the torn places.

4. A policeman who witnessed the explosion of a bomb and gave chase to the thrower was shot at twice by this individual, and was found to be slightly wounded in several places, and it was desired to know whether the wounds were caused by the explosion or by the shots. His clothes were examined, and in the tunic, which was of white drill, there were several holes, the edges of some of which were slightly stained with a yellow substance which on testing was found to be picric acid, thus proving that the wounds had been caused by fragments of the bomb. In this same case the man who threw the bomb was also slightly wounded by the explosion and there was a stain of picric acid on the torn edge of a small hole in the cuff of his shirt.

## BOOK OF REFERENCE.

Criminal Investigation. *Hans Gross.* Trans. Madras, 1906.

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[1] The author has only seen one exception, which was that of a gallabia worn by a boy, which had a pattern consisting of ovals.

## CHAPTER V

# COUNTERFEIT COINS

THE examination of counterfeit coins, although not wholly chemical, has a very important chemical aspect, as it is generally necessary to determine the weight, specific gravity and composition of the suspected coins.

Counterfeit coins are essentially of two kinds, cast and struck, the former being those made in moulds and the latter those made by means of dies. There is, however, a third way in which counterfeit coins might be made, namely by means of the electrotype process, a method frequently employed for reproducing rare coins for museum purposes, but this method is seldom used by false coiners.

Occasionally counterfeit coins are cast and then silvered or gilt, but examples of this are rarely met with and the author has seen only a few such cases, one of which was of counterfeit English sovereigns which were cast in lead and then gilt, the imitation being very crude.

There have been several instances in Egypt of the passing as genuine coins of the imitation sovereigns made in England and sold at one time in the streets of London at a penny each. These, which are of the same size as the sovereign, though much lighter in weight, are of copper gilt and, since they are struck and not cast, the design is good and sharp; they are also milled. As made originally these imitation coins are provided with a small ring at the top which is easily removed, but evidence of this is left.

For obvious reasons, any detailed description of the manner in which counterfeit coins are made will be omitted, though a few general particulars may usefully be given.

Cast coins are made in one operation by casting the metal in moulds prepared by taking impressions of genuine coins in suitable material such as plaster of Paris, clay, moulding sand or sometimes copper. The "runner"[1] is then removed and the edge of the coin touched up with a file. Cast coins are generally much poorer imitations than struck coins, and hence are more easily detected. A really first-class casting, however, with good milling, which fortunately is rarely met with, is very difficult to detect, more especially if it is worn, since even the smallest details of the design are correct. The surface of a cast coin is frequently pitted and uneven, the edges of the lettering and design are rounded instead of being sharp, there are little projections of metal where the lines should be straight and, in that section of the circumference from which the "runner" has been removed, the milling is often irregular or defective and shows evidence of having been made or adjusted with a file. Sometimes, however, the runner is so skilfully taken off and the milling so cleverly adjusted that, if the casting has been well done, it is difficult to say whether the coin has been cast or struck.

A large proportion of the counterfeit coins made in Egypt are struck and many are excellent imitations.

To make struck coins at least two separate operations are required, first the punching of circular pieces of metal of the correct size and weight from a sheet or strip, and secondly the impressing on these "blanks" the design of the coin. This latter is done from dies, the pressure being applied by means of a press. Generally, in order to produce the milling, a steel collar, which is milled inside, fits round the dies, though sometimes other devices are employed, such as pressing the blanks against milled wheels.

The dies, which are of steel, are usually engraved, but may be cast and then touched up by an engraver. In the case of engraved dies, which necessarily take considerable time and skill to prepare, the counterfeiter sometimes follows the practice of the mints and only employs the original die as a means of obtaining secondary dies, and it is these latter, which can readily be duplicated when broken or worn, which

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[1] The thin rod remaining attached to the coin where the metal has entered the mould. Any vents made for the escape of air would produce similar, though thinner rods, but although the author has examined a very large number of coinage moulds, he has never yet seen any provided with special air vents.

are actually employed for striking the coins. These secondary dies naturally all exhibit the same features and any slight peculiarity or defect existing in the original is reproduced in the copies.

Struck counterfeit coins are often very well executed, and their detection is by no means always an easy matter, but one which requires considerable experience, since the weight, size, specific gravity, milling and composition may all be good, and only a careful comparison of the smaller details of the design with those of a genuine coin enables the deception to be discovered. A counterfeit coin is less easily detected when it is worn or when it simulates a worn coin.

When examining coins, the most important points to which attention should be directed are first the details of the design and the manner in which the design has been executed, that is to say the quality of the workmanship, and secondly the milling, and a careful inspection with a lens and a careful comparison with a genuine coin are essential. In a genuine coin the milling is regular and at right angles to the face of the coin, while in counterfeit coins the milling is often irregular and slanting. In cast coins there is sometimes a fault down the centre of the milling, showing that the two halves of the mould have not fitted well together.

The examination of the milling of a coin should include the determination of the number of ridges. These can best be counted by colouring the edge of the coin by means of a pad moistened with aniline ink, carefully removing the colour from one of the ridges, and then taking the impression of the circumference of the coin on paper, and counting the number of lines made by the ridges, using the blank spaces caused by the removal of the colour from one of the ridges as the starting and finishing points, the number of ridges on the coin being of course the number of lines counted plus one. It should not be forgotten, however, that the diameter of genuine coins, and hence the number of ridges, sometimes varies with different issues of coins of the same denomination from the same mint, and that the number of ridges may also vary with coins of the same denomination from different mints although the diameter of the coins may be the same. Counterfeit coins made with the same dies may have different milling owing to the use of different milling collars or wheels.

Both the weight and specific gravity of suspected coins are useful indications, especially the latter, and should always be determined, the weight of course being necessarily ascertained when taking the specific gravity.[1] Any marked excess of weight above the legal limits is a certain proof that the coin is counterfeit; a deficiency in weight may be due to the coin being worn.

The density of coins composed of the same alloy and from the same mint is only constant for coins of the same size, and it varies inversely as the size of the coin, the smaller the coin, the greater being the density. The explanation of this is that the blow required for impressing the design on the coin acts on a smaller area and on a thinner piece of metal in the case of the smaller coins, and hence produces a greater effect.

The determination of the hardness of a coin, although possibly of some use to the person who tests a coin by biting it, is not of much assistance in any scientific examination.

The "ring" of a coin also is of very little value, since many counterfeit coins ring well, while genuine coins, if cracked, ring badly. A yellowish or a leaden colour in a silver coin is a certain proof that the coin is counterfeit.

Sometimes genuine coins which have been subjected to the action of chemicals or fire or which have been run over by a heavy vehicle or have been bored and the hole filled up, or fitted with a ring which has been removed and the milling adjusted with a file, are submitted to the expert because of their suspicious appearance, but such cases rarely present any real difficulty.

Secret marks are sometimes placed on coins by the mint, and unless a coin is worn their absence is a certain proof that the coin is counterfeit. Too much reliance, however, should not be placed upon the presence of secret marks,

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[1] For the determination of the specific gravity a little basket made from fine platinum wire in which to suspend the coin when weighing it in water will be found useful.

since they may be reproduced by the counterfeiter. Such marks never act as deterrents against forgery.

Counterfeit coins should always be carefully examined for slight flaws and marks, since these if present on a number of different coins prove that all have been made in the same mould or with the same dies, or with dies made from the same punch or matrix, and so may enable the police to trace coins passed in different places to one central factory. This has been helpful on many occasions. Thus in several cases it has been possible to trace cast coins seized with different persons to the same source, because all the coins had the same defects and had manifestly been made in the same mould. In one important coining case, which had ramifications in different parts of the country, and in connection with which about forty persons were condemned, struck coins from places far apart all bore evidence in the details of the design, that they had been made either from the same dies or from dies made from the same punch or matrix, and in many instances they had certainly been made from the same dies, since a flaw due to a crack in one of the dies occurred in the same position on each of the coins.

Coins made with the same dies may exhibit slight differences due to a different manner of striking or to the dies or blanks not having been rigid during striking.

Photographic enlargements are very helpful for attaching to the report, or for presentation in Court, as they enable similarities or differences between various coins to be readily demonstrated. Special methods for use when photographing coins will be described in the chapter on Photography.

In connection with counterfeit coins, it is sometimes useful to mark on a map the places where particular kinds of coins have been passed, and the number of coins from each place. This may show the centre of distribution and so indicate a particular district in which special search can profitably be made.

The services of a chemist are required not only for the examination of counterfeit coins, but also for the analysis of the materials seized on the premises of suspected persons, which may include metals, moulding composition and miscellaneous chemicals. Sometimes an ingot or other piece of metal found on the premises of a suspected person or a little fused metal left in a crucible can be proved to be identical in composition with certain counterfeit coins seized.

Since the chemist is consulted about so many aspects of the question, his opinion is also frequently asked about the use of various apparatus and appliances used in making counterfeit coins, such as crucibles, casting frames, moulds, flattening mills, punches, punching machines, dies and presses. Frequently such apparatus is very primitive, but occasionally it is very complete and ingenious. Naturally no details or descriptions can be given.

The expert to whom questions in connection with counterfeit coining are referred should familiarize himself with the methods used in the minting of genuine coins.

Most cases of counterfeit coining are devoid of any special interest, apart from the ingenuity displayed in adapting primitive apparatus and appliances intended for other purposes to the needs of the coiner. In the important coining case already referred to, in which a large number of persons were condemned, considerable use was made of small portable punching machines such as are used for punching holes in iron plates. These machines were not only employed for preparing the blanks, but they were also fitted with dies and used as presses, although unsuitable for this purpose.

**Analysis of Counterfeit Coins.**—A qualitative analysis of counterfeit coins or alleged counterfeit coins is practically always required, as it is generally necessary to state in the report of what metal or metals the coins consist. A quantitative analysis, however, is frequently not absolutely necessary, though it is always useful, and should be made whenever possible, but it becomes essential when the composition of the coins has to be compared with that of metal found on the premises of suspected or accused persons.

No general scheme of analysis applicable to all cases can be laid down, as the methods adopted depend upon the nature of the alloy and vary considerably in the presence of certain metals such as silver and antimony. The methods, however, are all standard methods which will be found described in the text-books. The usual constituents of counterfeit coins are one or more of the following, namely antimony, copper, lead, silver, tin and zinc.

Sometimes counterfeit coins are of the same composition as genuine coins, and several instances of this occurred in Egypt before the war, when the price of silver was low.

**Illustrative Cases.**—1. As possible connecting links between certain counterfeit coins and a suspected person, several pieces of white metal, and some similar-looking metal adhering to the end of an iron rod, which had manifestly been used as a stirrer, were examined. The defence set up was that these articles had no connection whatever with counterfeit coining but had been used for tinning copper saucepans, a common practice in Egypt. Analysis, however, proved that the pieces of metal and the metal on the rod were both identical in composition with the counterfeit coins, and differed considerably from the material ordinarily employed for tinning saucepans, and the man was convicted.

2. This was a case of English sovereigns which had been sweated, in some instances being reduced as much as half a gram each in weight. Examination proved that the faces of the coins had been protected, probably by grease or wax, and the edges then sweated by means of aqua regia, and traces of both nitrate and chloride were found on the coins.

3. Among a number of articles seized in one case were a small cylindrical piece of wood and a hammer. One end of the wood had the appearance of having been repeatedly struck, and on the other end there were impressions of parts of the design of a coin, and also some tiny particles of white metal, which on analysis proved to be largely silver, and wood fibres were found in a depression on the head of the hammer. Manifestly, therefore, both the wood and hammer had been employed together in some connection with silver coins, and it was confessed that they had been used to force counterfeit coins out of the milling collar. A similar piece of wood, also bearing the impressions of parts of the design of a coin, and adhering to which were tiny particles of white metal too small to allow of analysis, were subsequently found in another case. Other instances in which impressions of coins have been found on various articles will be mentioned in connection with the subject of Stains and Marks.

4. A minute fragment of metal, from which the test for copper was obtained, but which was too small for the reactions for other metals to be observed, though judging from its colour the metal was not wholly copper, was found on a file seized in the house of a person suspected of making counterfeit coins, the coins in question being composed of copper and tin.

## BOOK OF REFERENCE.

Manuel de Police Scientifique. *R. A. Reiss.* Vol. I. Lausanne, 1911.

## CHAPTER VI

# DOCUMENTS

AT first sight it might appear that the examination of documents was hardly within the province of the chemist, but when it is considered that a chemical and microscopical examination of the paper, a chemical analysis of the ink and a chemical examination of any discoloration or stains present, are frequently necessary, it will be recognized that there is a large amount of purely chemical work in this connection. There are, of course, other aspects of the question which are in no way chemical, such as that of the identity or non-identity of different specimens of handwriting, but since the chemical side of the question is so important, it is not unreasonable that the whole matter should be entrusted to a chemist who has specialized in such matters. In any case, however, the expert undertaking such work should possess some chemical knowledge, should have considerable experience in the use of the microscope, and should be an expert photographer.

**Care of Documents.**—It cannot be too strongly insisted upon that suspected documents are precious things and should be treated accordingly. As an example of the lack of care sometimes bestowed upon documents the following case may be cited :—On one occasion when dealing with an anonymous letter it was found that the mode of formation of the capital letter I, and whether it was made with one stroke of the pen or in two strokes, was of considerable importance, and this letter only occurred twice in the whole document and one of the two occurrences had been mutilated by the document having been perforated for filing.

Documents which are to be examined by the expert should be handled, folded and marked as little as possible.

On receipt, all documents should be placed between plain white paper in a cardboard cover. The examination should be made on a clean table on which there are not any bottles containing ink, liquids or chemicals or anything that could soil or damage the document if accidentally upset and even the reagents used for testing purposes should be placed elsewhere. Documents should not be touched with pen, pencil or anything that could possibly mark them, and should be handled as little as possible. In most cases it is advisable to photograph a document before examination, in order to have a record of its exact condition on receipt in case there should be any subsequent question or dispute, but this is not always practicable, especially if a large number of documents are received together. When being photographed the document should not be pinned or nailed, but should be placed on a piece of cardboard and covered with a sheet of glass and held in position in an adjustable wooden frame.

**Examination of Documents.**—A careful scrutiny is first made of the document as a whole in the usual way by reflected light and afterwards by transmitted light, both sides of the paper being inspected. The document is then examined in detail with a lens and any doubtful portions further examined with a microscope, special search being made for evidence of alterations and erasures if these are suspected. In some cases the material of the paper must be tested, and for this purpose a very small piece is cut from one corner. The ink too should generally be analysed and the handwriting critically examined. Frequently an enlarged photograph will be found helpful, especially in cases of alteration or erasure or when a comparison of the handwriting with that of another document is required.

## Paper.

One of the earliest substances used for writing upon was Egyptian papyrus, the fine layers of fibrous material from the stem of which were made into sheets, and it is from the name "papyrus" that the word "paper" is derived. After papyrus came parchment, which is still used for many legal documents. Parchment is made from the skins of certain animals, chiefly sheep and goats. The finer qualities, made from the more delicate skins of calves and kids, are termed "vellum." Parchment was succeeded by rags, at first linen rags, and afterwards a mixture of linen and cotton, or cotton only. A letter dated A.D. 874, and documents which from internal evidence are believed to belong to the year A.D. 792, made of linen, were found some years ago in the Fayum province in Egypt.[1] The date of the oldest manuscript in England on cotton paper is stated to be 1049.[2] Although experiments in paper-making were made with a great variety of fibrous materials, rags were generally employed until the middle of the nineteenth century. Straw was first used in 1800, a book made from it being published in England at that date,[1] though it was not employed on a large scale until more than fifty years afterwards. The manufacture of paper from mechanical wood pulp dates from Keller's process in 1840,[2] that of soda wood pulp from 1854,[2] and that of sulphite wood pulp from Tilghmann's patent in 1866.[2] One authority states that a patent was granted to some paper makers in Italy in 1826 for the use of wood pulp.[3] The general use of mechanical wood pulp, however, dates from 1870–80 and that of wood cellulose from 1880–90.[4] Esparto was introduced into England about 1860.[2] Before 1799 all paper was hand-made, but in that year the first paper machine was invented in France, and in 1807 a paper machine was erected in England.[2] Blotting paper is mentioned as early as 1465 and fragments have been found in fifteenth-century account books, and in the sixteenth century it is referred to as though it were well known ; [5] it was not in general use, however, until the beginning of the nineteenth century, a fine powder, such as sand, being employed instead, and even to-day sand is still sometimes used for this purpose in Egypt. Brown paper is referred to in 1570–1.[5]

Two cases in which the composition of paper has been of importance may be mentioned. In one instance a docu-

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[1] The Manufacture of Paper. *R. W. Sindall.* London, 1908, pp. 6, 89.

[2] Paper Technology. *R. W. Sindall.* London, 1906, pp. 208, 209.

[3] Chambers' Encyclopædia, Vol. VII, 1908.

[4] Deterioration of Paper. Society of Arts, London, 1898.

[5] Encyclopædia Britannica, 11th Edition, Cambridge, 1911. Vol. XX. Article "Paper."

ment dated 1213 A.H. (A.D. 1798) was composed entirely of chemically prepared wood cellulose although this material was not employed for paper-making until more than fifty years afterwards, and in another case it proved a useful point in favour of the accused that paper found in his house, which superficially resembled that of some anonymous correspondence, and was stated by one expert, who, however, had not examined it microscopically, to be the same, was of entirely different composition. In this connection it may be mentioned that a number of genuine old Arabic documents, mostly title-deeds of land dating between 1136 A.H. (A.D. 1723) and 1287 A.H. (A.D. 1870), which have been examined were all found to be composed entirely of linen.

**Testing of Paper.**—The determination of the nature of the fibre is made by means of the microscope after the paper has been suitably prepared. For this purpose the fragment to be examined, which need only be very minute, is placed in a small beaker and boiled with dilute sodium hydroxide solution (2 to 5 per cent.) to get rid of sizing and loading materials. The liquid is poured off and the paper washed repeatedly with water by decantation. If the sample is large enough, it is then transferred to a small porcelain mortar and well broken up, dried between filter paper, and small portions picked off with microscope needles, teased out on a slide, stained with iodine, or better with zinc-chlor-iodine solution, and examined, using a ⅙th inch or ⅛th inch objective. If the sample is very small, it is transferred, after washing, direct from the beaker to a slide, dried with filter paper teased out, stained and examined. At least two or three different slides should be made from each sample of paper. The various fibres prepared as described give the results shown in the table on p. 78.

It is important always to employ solutions of the same strength, since variations in the strength of the reagent affect the intensity of the colour produced. If wet, the paper must be dried before staining. The staining is not only of use in distinguishing the various kinds of fibre from one another, but it also serves to intensify differences of structure and to bring out details.

**Water-mark.**—Sometimes a limit may be placed to the age of a document by means of a water-mark, the earliest known dating from 1301.[1] In an important case in which the title to a large area of land was in dispute certain receipts were produced dated 1294, 1295, 1296 and 1297 A.H. respectively (A.D. 1876, 1877, 1878 and 1879), all of which bore the Egyptian Government water-mark although this was not introduced until 1885.[2] In the same case two other receipts dated three months apart had originally formed part of the same sheet of paper and a portion of the same water-mark appeared on each, and it seemed improbable that part of a sheet of paper should be used on one occasion and the other part not until three months later. In another case a number of receipts, the dates of which extended over a period of one year and ten months, were examined and it was noticed that all were parts of similar sheets of foolscap,

| Fibre. | Iodine (*a*). | Zinc-chlor-iodine (*b*). | Phloro-glucine (*c*). | Aniline (*d*) Sulphate. |
|---|---|---|---|---|
| Linen .. .. | light to dark brown | weak to strong wine-red | nil | nil |
| Cotton .. .. | | | | |
| Straw .. .. | almost colourless to light brown (*e*) | blue | nil | nil |
| Esparto .. .. | | | | |
| Wood (chemical) | | | | |
| Wood (mechanical) | yellow to brown | yellow | red | yellow |

(*a*) Potassium iodide 2 grams : iodine 1·5 grams : glycerine 2 c.c. : water 20 c.c.

(*b*) (1) Zinc chloride 20 grams : water 10 c.c.: (2) potassium iodide 2·1 grams : iodine 0·1 gram : water 5 c.c. Mix (1) and (2) and allow the mixture to stand, and decant off the clear supernatant liquid for use : add a few small crystals of iodine.

(*c*) Phloroglucine 2 grams : alcohol 25 c.c. : hydrochloric acid (conc.) 5 c.c.

(*d*) 10 per cent.

(*e*) As seen in the brown iodine solution.

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[1] The Manufacture of Paper. *R. W. Sindall.* London, 1908, p. 68.

[2] The ordinary correspondence paper of the Egyptian Government had a date added to the water-mark for the first time in 1915, although certain stamped paper had borne a dated water-mark for many years previously, and certainly as early as 1887.

and after considerable trouble it was found possible to fit the various pieces together. It was seen that the order in which the pieces occurred did not correspond with the dates on the receipts, thus for example a receipt dated January of one year was part of the same sheet as a receipt dated January of the following year, while parts of other sheets, in no regular order, had been used for intermediate dates. This at once raised a presumption of forgery, since it seemed improbable that one part of a sheet of paper should be used one year and a second part not until twelve months afterwards, although meanwhile other sheets had been torn into similar pieces and used for a similar purpose. The water-mark on the paper was very helpful in fitting the various pieces together. In connection with the forgery case already mentioned the fact that blank sheets of paper bearing an unusual water-mark, which occurred on some of the forged documents, were found in the houses of two of the accused persons was a small link in the chain of circumstantial evidence which convicted them.

A series of three crescent moons of different sizes is an old water-mark on paper used in Egypt and certainly occurs as early as 1188 A.H. (A.D. 1774) and it occurs in at least two different forms. In Brown's Travels to Darfur[1] "papier des trois lunes" is mentioned as an article of commerce from Egypt to Darfur in 1793. A water-mark of three stars has been noticed on documents dated 1190 A.H. (A.D. 1776).

**Discoloration of Documents.**—Occasionally documents are discoloured intentionally in order to give them a fictitious appearance of age, or they are scorched and partly burned and sometimes creased and torn for a similar reason. These same devices are also resorted to for the purpose of hiding evidence of fraud.

Discoloration due to age is largely a process of oxidation brought about by natural means and it takes place in proportion to the extent to which the paper has been exposed to the air and light, and hence the outsides and edges of old documents, which are the most exposed, become the most discoloured, the discoloration progressively diminishing towards the less exposed parts. In addition to the general

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[1] Second Edition, p. 348.

discoloration, however, there are frequently on old documents brown spots due to mould which are very characteristic both in appearance and distribution. It may be mentioned that these spots become translucent when wetted with water, regaining their original appearance when dry, the smaller spots as a rule becoming entirely translucent while the larger ones remain opaque at the centre. This is a very simple test and one that may be applied to almost any document without fear of injury. Sometimes isolated brown spots not caused by mould but by iron or other impurities also occur.

Other natural causes for the discoloration of documents are exposure to dust and dirt and occasionally staining by fruit juice, grease and the excreta of rats, mice and insects. In the latter case the outsides and edges of the documents generally suffer the most. Where a document has been intentionally discoloured with dust, dirt or mud, this is evident as a rule by the discoloration showing definite streaks or lines when carefully examined, the dirt generally having been rubbed on either with a cloth or with the hand.

Discoloration may also be due to heat, in which case it is an actual partial burning of the surface of the paper, which becomes very brittle if the burning is severe, and when one side becomes scorched, unless the paper is thick, the opposite side also shows signs of burning. When a number of documents are exposed to fire while together those on the outside naturally suffer most, and those on the inside may be only slightly burned or not burned at all, and any holes produced correspond in relative position in the various documents. The edges of the documents too, being loose, always burn first. These facts are frequently forgotten by forgers whose handiwork shows a definite selective action which is unnatural, certain documents or parts of documents being burned or spared in a manner that could not possibly be accidental. In one case a piece of blank paper which had been scorched in the same suspicious manner as some forged documents was found in the house of one of the accused persons.

Artificial discoloration made to simulate age is produced by means of a coloured solution. The author has never been able definitely to establish the nature of any solution employed, but in the East coffee is very probable, while in the West tea might be used. A water extract of tobacco or a dilute solution of potassium permanganate would also serve the same purpose. The use of a coloured solution is generally indicated by the characteristic shape of the edges of the discoloured areas, or the way in which the liquid has run may be plainly visible, and a thin dark line sometimes occurs where there were any very marked creases on the paper at the time it was treated. Occasionally too portions of the paper, often very small, may be found which have altogether escaped the action of the solution.

**Documents Mounted on other Paper.**—In cases where documents have been intentionally creased or torn to give them a fictitious appearance of age and constant use they are sometimes mounted on another sheet of paper. This may also be done to hide signs of erasure or to prevent too minute an examination. When a document mounted on other paper is in several pieces the edges at the joins should always be carefully examined, since sometimes these do not correspond, the separate pieces being parts of different documents put together to make an entirely new and fictitious document, or the bottom piece bearing the signature may be part of a genuine document to which writing specially prepared for the purpose has been joined. In other cases the two pieces of paper at a join overlap, and although a line of writing is partly on one piece and partly on another, it does not always extend to the edges of both, but only to the edge of the piece which is uppermost, leaving a margin without writing on the under piece, which is conclusive proof that the writing has been done after the pieces have been joined together : it is also sometimes found that small portions of some of the letters of the writing at a join exist on the paper used for mounting, which again is proof that the mounting paper was in place when the document was written.

In one case in which a document consisted of two pieces joined together it was manifest that the paper had been cut intentionally and not torn or worn, since not only were marks of short-bladed scissors apparent, but the cut was not horizontal across the paper but occurred between two lines of writing following the very marked slant of the writing. A piece of the document also had been cut away and was missing, since the paper was a laid paper with the usual "water-lines" crossing it horizontally, and these lines where they occurred above and below the join did not coincide.

It is quite a common occurrence in a country like Egypt for old title-deeds of land to be written on more than one sheet of paper, the separate sheets being joined end to end; thus one such document examined was written on nineteen separate pieces of paper and measured altogether about seven and a half metres in length: sometimes such deeds are twenty or even thirty metres long. In the particular case above mentioned the safeguard against the possible fraud of tampering with one or more of the pieces consisted in the fact that the writing had been so arranged that a line was across each join of the paper.

**Ink.**—The principal kinds of ink employed are carbon ink, iron-tannate ink, logwood ink and aniline ink.

The ancient Egyptians used a carbon ink, as also did the Greeks and Romans, the latter of whom too made an ink from sepia. The earliest reference to an iron-tannate ink is in the eleventh century.[1] Colouring matters, such as indigo, were used to a slight extent to strengthen the colour of the ink during the eighteenth century, but it was not until the nineteenth century that the modern blue-black ink containing a provisional colouring matter was employed.[1]

The use of a decoction of logwood as an ingredient in ink was known as early as 1763, and at first the logwood was employed as an addition to iron-tannate ink, but in 1847 logwood was used with potassium chromate and in 1859 with alum, in both cases without any iron compound.[1]

The first aniline dye was mauve, discovered by Perkin in 1856;[2] in 1859 fuchsine was made and aniline blue in 1861;[2] in 1863 Hofmann discovered the violet that bears his name;[2] eosin was made in 1874,[3] and malachite green

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[1] Inks. *Mitchell and Hepworth.* 1916, pp. 6, 11, 92, 94, 95.

[2] J. Soc. Arts. *W. H. Perkin.* 1868.

[3] The Chemistry of the Coal Tar Colours. *Benedekt and Knecht.* 1889.

in 1878.[1] The first British patent for the use of aniline dyes in ink was dated 1861, followed by others in 1862.[2] Nigrosine was introduced for stylographic pens in 1867.[2] Coloured inks, however, were known before the introduction of aniline dyes ; thus green ink, probably a mineral product, was used both by the ancient Egyptians[3] and also by the Romans.[2] Red ink was also employed.

*Carbon Ink.*—Carbon ink is still largely employed for writing purposes in Egypt and the East generally at the present day. The best quality consists essentially of carbon obtained as a deposit during the partial combustion of organic materials such as oil and resin. A recipe for making ink of this kind from incense, the ink being used for writing religious books, has been kindly supplied by a priest of the Coptic Church. The recipe is as follows :—Put a quantity of incense on the ground and round it place three stones or bricks, and resting on these, an earthenware dish bottom upwards covered with a damp cloth ; ignite the incense. The carbon formed will be deposited inside the dish, from which it is removed and made into ink by mixing with gum arabic and water. An old Arabic book in the Sultania library, Cairo (unfortunately anonymous and undated), contains a recipe for making what is called Persian ink. The method is to take date stones, put them in an earthenware vessel stoppered with clay and put the vessel over a fire until the next day, then remove, allow to cool, grind and sift the contents and make into ink with gum arabic and water. Such an ink is of a very different composition from that previously referred to and may contain little or no free carbon.

When wood or similar organic matter is charred by heat it is generally assumed that the resultant blackening is due to the formation of free carbon, and under certain conditions of temperature this is undoubtedly the case, but various black organic compounds, such as tarry matter, are also

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[1] J. Soc. Chem. Ind. *W. H. Perkin.* 1885, p. 426.

[2] Inks. *Mitchell and Hepworth.* 1916, pp. 95, 99, 100, 240.

[3] A Guide to the Egyptian Collections in the British Museum. London, 1909, p. 31.

formed, and it seems possible that at relatively low temperatures free carbon may be almost or entirely absent. As the result of experiments made on the solubility of charred wood in a solution of bleaching powder and in nitric acid, Boodle[1] comes to a similar conclusion.

Having occasion to consult, in the Government archives, Cairo, some old Arabic documents dating between 1026 A.H. (A.D. 1617) and 1288 A.H. (A.D. 1871), it was noticed that although generally speaking the ink was black, yet in some cases it was brown or partly brown. It was not possible to examine the ink chemically in these particular instances, but in the case of other documents which have been examined, in one instance a series of eight title-deeds for land varying in date from 1246 A.H. (A.D. 1830) to 1287 A.H. (A.D. 1870) the same phenomenon occurred, and the ink was undoubtedly nominally a carbon ink, and the fact of such ink becoming brown with age must be accepted. This at first sight seems extraordinary and even impossible, since one of the characteristics of carbon is its durability, carbon ink being always regarded as the type of a permanent ink. It is of course not contended that ink in which such change has taken place was ever a good quality carbon ink, for this is manifestly not the case, but it is stated that the ink in question is not an iron ink, but has been originally a black or practically black ink, which was accepted and used as an ink of the carbon type, and to-day carbon ink is still generally employed for documents of the class of those in question. An ink made according to the second of the methods mentioned might well be of this sort. In any case it must be assumed that when the document was finished, the writing was uniformly of a dark and practically black colour, and hence the disappearance of this colour must be accounted for, and it is suggested that the ink contained little or no free carbon in the first instance, and that a definite colour change has taken place, any small amount of carbon present being masked by the brown colour formed. In this connection a number of old manuscripts in the Sultania library, Cairo, were examined, and it was found that here too in many

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[1] The Nature of Charred Wood. *L. A. Boodle.* Bull of Misc. Inf. Royal Botanic Gardens, Kew, London, 1917.

cases the ink was brown. Thus the ink on manuscripts written on papyrus dating from between A.D. 622 and 719 though often black is sometimes brown, and in some of the old manuscript Korans dating between A.D. 719 and 912 the ink is also brown and sometimes faded. In several instances where the writing is black there is a brown-coloured zone round the letters, and where writing shows through the paper from one side to the other it is often brown. It has also been noticed that where writing has become accidentally copied on to an opposite page, probably owing to the paper having been damp at some time, the copy may be brown although the original writing is black. In the case of two manuscripts on leather, dated 233 and 239 A.H. respectively (A.D. 848 and 853), the ink evidently has originally been black, and is still black in places, but in other places it is white and manifestly some change has taken place, but of what nature it is impossible to say without a chemical examination; the white, however, does not appear to be a fungus growth on the surface of the ink as was at first thought possible.

When carbon ink is used on a good quality well-sized paper, such as the parchment-like paper frequently employed in Egypt for title-deeds of land, the ink does not penetrate and can be washed off. A common illustration of this is that when testing carbon ink it is frequently noticed, after applying the usual tiny drop of reagent and using blotting paper to absorb the excess, that the ink on the small portion of the writing tested has partly or wholly disappeared from the paper, and if the test solution employed happens to be one that bleaches, the explanation at once suggests itself that the colour has been bleached and therefore is not carbon, though what has actually happened is that the ink has become loosened from the paper by the solution and has been removed mechanically by the blotting paper, to which it can be found adhering. Care therefore is necessary to distinguish this washing off from bleaching. That carbon ink could be removed from documents was known to Pliny, who states that it could be done with a wet sponge.[1]

*Iron Ink.*—As originally made iron ink consisted essentially

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[1] Inks. *Mitchell and Hepworth.* 1916, p. 6.

of a mixture of ferrous sulphate and an extract of nut galls. Such an ink when fresh gives only a very faint colour on paper but gradually oxidizes and darkens and finally becomes black. In order therefore that there should be an immediate coloration when the ink was used it was customary to oxidize it during manufacture. Later it was found preferable that oxidation should take place after the ink had penetrated into the fibres of the paper, and so oxidation during manufacture was dispensed with and, in order to obtain an immediate coloration, a provisional blue colouring matter, sometimes indigo but more generally an aniline colour, was added, and such a mixture constitutes the modern blue-black ink. At first only the blue colour shows, but after a time this becomes masked by the formation of the black compound resulting from oxidation. Such an ink also oxidizes and gradually becomes black if left exposed in an ink pot. This colour change provides a valuable means of determining the age of an ink within certain limits. Thus ink which is very blue to the naked eye is very recent, if it is violet it is less recent, and if black still less recent. But an ink which is black to the naked eye may still be violet or even blue when seen through a lens or microscope, and it is only after a considerable time that the ink becomes completely black. Mitchell states that a thin wash of an iron-tannate ink when exposed to strong light becomes almost completely oxidized in about a week, but that if the ink is applied in the ordinary way with a pen and afterwards kept in a closed book the final intensity of colour may not be reached for upwards of a year.[1]

Osborn [2] states that for a blue-black iron-tannate ink to appear black to the naked eye a period of from one to two weeks is required in the summer, and from six to eighteen weeks in the winter, while for the ink to be of a real neutral black, even when examined microscopically, a period of from fourteen to twenty-four months is required, and that the colour is then fixed for a period of from six to ten years, after which it begins to turn yellow at the edges, finally becoming a yellowish brown.

In some tests made by the author it was found that in

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[1] *Analyst,* 1920, p. 250.

[2] Questioned Documents, p. 353.

the month of January in Cairo, Stephens' blue-black writing ink lost its blue colour to the naked eye in two days, one locally-made blue-black writing ink lost its blue colour in one day, while in the case of a second locally-made blue-black ink for fountain pens, after the blue colour disappeared a violet tint formed which persisted up to and including the twelfth day from the time the writing was made. With the microscope all three inks showed a slight dark violet colour two years after the writing was made.

The following observations may also be quoted :—

The writing in a number of registers extending over a period of fourteen years was examined. It was found that some of the writing fourteen and a half years old (the oldest of the registers dated fourteen and a half years from the time the examination was made) when seen with the naked eye was grey, some black and a small amount brown. Of the writing in the subsequent years part was grey and part black, but none brown ; the writing from six months to eighteen months old was chiefly black. In no instance was there any blue or violet tint. No writing less than six months old was examined. The ink used was a blue-black iron-tannate ink, but possibly not of the same make in every case. A number of dated documents were also examined. These varied in age from one month to seven years. To the naked eye none of the ink showed any blue or violet tint, but with one exception all were grey or black, the exception being brownish black and the document four and a half years old. Under the microscope most of the documents, up to and including those three years old, showed a definite blue or violet tint, the colour being more marked the more recent the writing. There were, however, two exceptions, one being four months old and the other two and a half years old, and in both instances the colour of the ink varied from grey to black. Beyond three years no blue or violet tint was distinguishable, the ink being brown in one case (four and a half years old) and grey or black in all other instances.

In the above-mentioned cases therefore no blue or violet was visible to the naked eye after one month (no observations of ink less than one month old were made), but blue or violet was visible with the microscope up to three years, with only two exceptions, one being four months and the other two and a half years old respectively.

Another indication of the age of iron-tannate ink when in the form of writing is that when recent it reacts rapidly with certain reagents, but after a time this reaction proceeds more slowly and finally the ink remains comparatively unaffected. For this test C. A. Mitchell[1] recommends a 5 per cent. solution of oxalic acid, a drop being applied to the writing by means of a capillary pipette and the result observed with a lens. In order for a correct comparative estimation of the age of two inks to be made it is necessary that the dates of the inks should not be too close together, that the inks should be of the same type and that the differences found should be very pronounced. The fact that ink has been blotted may considerably modify any conclusions to be drawn from either the colour or the time of reaction to various reagents.

No general rule exists for determining the age of an ink from the colour, since much depends upon the nature of the ink and whether or not it was fresh when the writing was made, or whether it had already been exposed for some time in the ink pot, also upon the degree of exposure to which the writing has been subjected, the temperature and humidity to which it has been exposed and the nature of the paper, and whether or not the writing has been blotted.

In this connection it may be mentioned that in a prominent advertisement of a well-known ink a curious mistake has been made. This advertisement is one showing a large blot of ink with the name of the manufacturer written across it. This blot is coloured black in the centre and blue at the edges, which is the very reverse of what actually happens, a blot being entirely blue at first and gradually darkening at the edges where the film of ink is thinnest and becoming black in the centre last of all.

Neither carbon ink nor aniline ink can be dated even approximately from chemical evidence, since neither undergoes that progressive chemical change with lapse of time that is so characteristic of a blue-black iron-tannate ink.

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[1] *Analyst*, 1920, p. 253.

A special ingredient is sometimes put into ink in order that forgery may readily be detected and demonstrated.

*Aniline Ink.*—Coloured aniline inks, chiefly violet, are used largely in the East, even for important documents such as wills, promissory notes, deeds of sale and receipts. Ink of this kind fades on exposure and the nature and extent of the exposure condition the appearance of the ink much more than mere age. It may be mentioned, however, that aniline colours in the form of ink of the kind used for duplicating purposes with such an apparatus as the hectograph for example, are frequently very resistant even to strong sunlight, apparently on account of the thickening ingredients present, whereas copies of documents made with such ink fade readily.

Sometimes a signature written with violet ink is suspected because it is lighter coloured than the ink on the body of the document or than that of other signatures, but little can be done in such cases and the lighter colour may simply be due to the ink having been blotted. In one case the authenticity of a document written in violet ink was questioned because the ink was darker in colour than that on another document bearing the same date; the evidence, however, was in favour of it being genuine.

In a case in which the validity of a document relating to the sale of land was contested, the whole of the document, including the signatures, was written with violet ink and therefore no chemical examination of this could give any indication of its age. The prosecution contended that the signature of the most important witness had been added at a later date than the other signatures, while the defence produced photographs (both negative and prints) showing this signature and the absence of a certain dated registration stamp which seemed to prove that the signature had been on the document at a date prior to that assigned to it by the prosecution. The fact that the grain of the paper of the document and considerable gradations of light and shade existed on the photographs, as also two numbers which showed through the paper from the opposite side, was definite proof that the registration stamp had not been blocked out or otherwise removed, and that the photograph had actually been taken before the document was registered. The registration stamp too crossed a fold where a corner of the paper had been turned down to allow an endorsement on the other side to show on the photograph and the sequence of the fold and some writing on the registration stamp corroborated the other evidence that the photograph was taken before the document had been registered.

When dealing with photographs it should not be forgotten that both negatives and prints are very easily "faked." Thus a negative may not be the original negative, but a secondary one on which something not on the original has been added or from which something has been omitted, and in the same way the print may have been made from a "faked" original or from a secondary negative. It is a commonplace of photography to block out undesirable features in a negative and to make additions, such as a suitable background to a portrait or clouds in a landscape.

In another case examined a document bore various genuine consular registration stamps and was endorsed on the back with violet ink which showed through the paper. As seen from the face, however, the colour of the endorsement was not violet but green, and since it is a well-known chemical fact that certain acids turn aniline violet a green colour, acid was tested for and was found to be present to an extent impossible in a paper that had not been specially subjected to acid treatment, and on further examination it was discovered that part of the original writing had been effaced and a forged name and other details substituted. The fact that gum had been used in this and in several other instances to reglaze the paper after the original writing had been removed was additional proof that the documents had been tampered with, since gum is never used for such a purpose by the paper manufacturer.

**Testing of Ink.**—The nature of the ink on a document should as a rule be determined, since this may serve as an indication of age or as a proof of alterations or additions. The testing is carried out by applying minute drops of various reagents to the writing by means of a capillary pipette and examining the result with a lens, and this can be done so carefully that any marks left are so small that it is difficult afterwards to find the spot tested. When writing has been chemically tested a note should always be made of the exact spots to which the reagents have been applied and these should be indicated in the report.

It cannot be insisted upon too strongly that suspected documents should be treated with the greatest care, and no one who has not had considerable experience in delicate manipulation should be allowed to test them. Sometimes an alteration to a document consists of a few letters or figures only, or even of part of a single letter or figure, and unless the greatest precautions are taken, the disputed portion of the writing will be so defaced as to become quite useless as evidence. In cases of this nature it is inadvisable to test the ink at all.

In view of the comparatively large number of reagents for testing ink on documents recommended in various books, perhaps it will be as well to state that very few are actually required, the most useful being dilute hydrochloric acid (strength not more than 5 per cent.), dilute sodium hydroxide (strength about 2 per cent.) and a solution of bleaching powder, or better, of sodium hypochlorite. Oxalic acid, tartaric acid and acetic acid are occasionally useful substitutes for hydrochloric acid and may be employed in greater strength than the mineral acid without fear of injuring the paper. Sulphuric acid, although frequently recommended, should never be used, since it will inevitably seriously damage the document. This damage may not take place at once, but only gradually, and hence may not be apparent until some time afterwards. When testing fresh or comparatively fresh ink, potassium ferrocyanide, which is frequently employed as a test for iron in ink, is less valuable than sodium hydroxide, since it labours under several disadvantages, one being that it readily decomposes, giving the very reaction (a blue colour) it is used to test for, and another that it reacts with any iron compounds in the paper as well as with those in the ink. Also, since potassium ferrocyanide solution is acidified with hydrochloric acid before use, this latter renders visible the blue colour due to any provisional colouring matter in the ink, which masks any other blue due to iron compounds. When testing old inks both potassium ferrocyanide and potassium thiocyanate are necessary and are then to be preferred to sodium hydroxide. When using these reagents a minute drop of hydrochloric acid is first placed on the ink in order to dissolve the iron, and this is followed by a drop of ferrocyanide or thiocyanate. Sodium hydroxide has also a disadvantage in the way it spreads on the paper, but the effect of this may be minimized by using the smallest possible quantity and by removing the excess with blotting paper as soon as the result of the test has been noted. No matter what reagent is used, it is a wise precaution to remove the excess remaining after the reaction has terminated, by means of blotting paper, or better to wash it off by applying successive drops of water which are afterwards absorbed by blotting paper.

Carbon ink is distinguished from most other writing inks by the fact that it remains unaltered when treated with a solution of bleaching powder or of sodium hypochlorite. Other reagents are also without effect.

The fact already mentioned that carbon ink can readily be removed from documents provides a good test for this ink. A drop of water is placed on a small piece of filter paper, which is then applied to the writing and pressed down with the thumb-nail. If the ink is a carbon ink, a little will generally come off and will be found as a black stain on the filter paper and can be tested. Occasionally an iron ink, especially an old iron ink, will transfer in the same way, but the presence or absence of iron can readily be ascertained by applying a drop of hydrochloric acid, followed by a drop of potassium ferrocyanide or of potassium thiocyanate.

Both iron ink and logwood ink, unlike carbon ink, give very marked positive reactions with various reagents. The principal results are shown in the table opposite.

In several cases investigated alterations to documents written with carbon ink have been made with an iron-tannate ink containing a provisional blue colouring matter, and in one instance a document written with carbon ink had been altered twice, in one place with an iron-tannate ink and in a second place with logwood ink. In another case a document written with iron-tannate ink had been altered with carbon ink.

Many of the results of the chemical examination of ink on documents, which are to be found in the literature of the subject, have reference to an iron tannate ink without any provisional blue colouring matter, and therefore are very misleading, since practically all ordinary writing inks now contain a blue colour.

It is sometimes suggested that in order to test the nature of aniline ink on a document, or even of any kind of ink, a press copy of the writing should be taken so that this may be tested chemically instead of the original. This should never be done. In the case of aniline ink it is sometimes allowable to take a copy of a few unimportant letters or words, if such exist, but in no circumstances whatever should a press copy be made of a suspected alteration or suspected signature or date. As already mentioned, if the testing is carefully done there need not be any disfigurement of the document. With respect to violet ink only one substance is generally employed, namely methyl violet, and therefore as a rule chemical tests yield no useful results.

| Reagent. | Iron-gall Ink without Blue Colouring Matter. | Iron-gall Ink with Blue Colouring Matter. | Logwood Ink. |
|---|---|---|---|
| Hydrochloric acid .. | slight yellow stain | blue | red |
| Oxalic acid .. .. | bleaches | blue | red |
| Tartaric acid .. .. | bleaches | blue | light brown |
| Acetic acid .. .. | bleaches | blue | violet |
| Sodium hydroxide .. | reddish brown | reddish brown | brown |
| Potassium ferrocyanide (*a*) | blue | blue | — |
| Potassium thiocyanate (*a*) | red | red | — |
| Sodium hypochlorite .. | bleaches | bleaches | bleaches |

(*a*) After the iron has been dissolved by a drop of hydrochloric acid.

For blue inks different compounds are used, and some of these may be distinguished from one another even when in the form of writing. When dealing with blue ink the following caution should be noted:—An ordinary iron-tannate ink containing a blue colouring matter if very recent is blue, and when tested with hydrochloric acid, oxalic acid, tartaric acid or acetic acid it remains blue or may become still more blue, owing to the bleaching of any colour due to the iron-tannate, and the consequent rendering visible of all the blue already present. Such an ink too gives a reddish-brown colour when tested with sodium hydroxide. When blue aniline inks, which are sometimes used for writing, are tested with these same reagents, they also remain blue with the acids, and in some cases give a reddish purple colour with the sodium hydroxide, and therefore, unless care is taken, such an ink might be mistaken for a very recent blue-black iron ink.

For red inks there are satisfactory tests. The dye principally used for making red ink is eosine, but cotton scarlet, ponceau scarlet or fuchsine might also be employed, and the following method of distinguishing between them when on a document may be mentioned:—With bromine water eosine becomes yellow, but the red colour returns on the addition of sodium hydroxide; cotton scarlet also becomes yellow with bromine water, but on the addition of sodium hydroxide to the yellow spot a brown colour is formed; sodium hyroxide applied directly to the cotton scarlet produces a puce colour which disappears after a time; with bromine water ponceau scarlet at first becomes yellow and then bleaches; bromine water turns fuchsine a violet colour, which becomes yellow or orange on the addition of sodium hydroxide.

Occasionally it is found that a few words or figures in red ink are suspected because of their dark colour, but this may simply be due to a black-ink pen having been used by mistake at the beginning of the writing or to the pen having been subsequently and inadvertently dipped into black ink.

In one book dealing with questioned documents, the chemical testing of ink is described as "quite simple," and it is further stated that "the cost of the necessary material and the time required to make oneself proficient as a capable tester are so slight that even the small fee that would be charged by a chemist is scarcely worth paying." It is certainly true that the cost of the reagents required is slight, chiefly because the quantity used is so minute, and the method of testing like everything else is simple when one knows how to do it, but the way proposed of determining the presence of acid in an ink on a document by placing "the tip of the tongue to a thick stroke" cannot be recommended, nor is it advisable to put so much water on a document that some of it can subsequently be poured into a test tube, as is advocated in the book in question. A "point of a pin" as suggested as a means of applying acid is also unsatisfactory. The only right way of testing ink on a document is that already mentioned, namely to apply an exceedingly small drop of the requisite reagent by means of a fine capillary pipette, and to examine the result with a lens.

**Pens.**—The first steel pen on record was made in England in 1780 and others were made and sold in 1803. The first patent for the manufacture of metallic pens is dated 1808. Machine-made pens were introduced in 1822.[1] In England and many other countries the only pen now used is the ordinary steel pen, the quill pen formerly employed having become practically obsolete, but in the East pens analogous to the quill are still employed; thus in Egypt a reed pen is very common, though it is being gradually superseded by a special form of steel pen. The writing made with these different kinds of pen can often be distinguished by the general appearance, the shading and the width of the ink lines. Even writing made with different pens of the same kind may be distinguished if the points are of different fineness. It is a useful precaution in connection with the examination of forged or anonymous documents to ascertain whenever possible the kind of pen used by the suspected writer.

**Pencil Writing.**—Pencils used for writing may be either black-lead or they may be coloured, the colour generally being violet, and such pencils are termed "indelible" or "copying" or "copying ink" pencils, but blue or red pencils are also used.

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[1] Encyclopædia Britannica, 11th Edition. Cambridge, 1911, Vol. XXI, Article "Pen."

*Black-lead Pencils.*—Black-lead pencils were not known before the discovery of graphite in Cumberland about 1560, earlier pencils being made either of metallic lead or of a lead alloy.

It is sometimes stated that it is impossible to distinguish between one black-lead pencil and another when in the form of writing, but C. A. Mitchell [1] has recently shown that under certain conditions writing done with different pencils may be differentiated both by microscopical examination and by chemical tests.

For the microscopical examination a 1-inch objective and a strong side light are employed. The variations in the marks made by different pencils consist of (*a*) differences in the colour and distribution of the pigment, (*b*) the presence and nature of certain striations and (*c*) a brilliant lighting effect sometimes observed on the paper. Mitchell states that too much importance should not be placed upon differences in the depth of colour observed, since under variations of pressure marks from hard pencils may resemble and may be mistaken for those made by soft pencils.

The chemical tests applied to black-lead pencil writing consist principally of tests for iron and chlorides, but may include a test for sulphates and also a test with dilute nitric acid. For iron a drop of 80 per cent. acetic acid or of strong nitric acid is placed on the writing, followed by a drop of potassium ferrocyanide. Different pencil marks show pronounced differences in the colour produced. The presence or absence of chlorides also serves to distinguish between the writing done with different pencils. For this reaction a drop of dilute nitric acid is applied to the writing and silver nitrate solution is then added to the drop, which should retain its globular form throughout the test. A test for sulphates with nitric acid and barium chloride or nitrate may also show differences, or sometimes there is a difference in the behaviour with dilute nitric acid alone.

*Copying Ink Pencils.*—The only reference to this subject

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[1] Black-lead Pencils and their Pigments in Writing. *C. A. Mitchell.* J. Soc. Chem. Ind., 1919, p. 383T.

that can be traced is a recent paper by C. A. Mitchell.[1] The tests applied are both microscopical and chemical. When examined with the microscope definite differences in colour may sometimes be seen with writing made with different pencils. Different pencil marks also show characteristic differences when tested with different solvents. Water, alcohol, ether and acetic acid should all be tried, a drop of the solvent being placed on the writing and the result observed with a lens. It should be noted whether there is any solution of colour and whether this solution is immediate or not, and finally the drop should be soaked up with a small piece of filter paper. The residue left on the paper, particularly that left after acetic acid, should be examined microscopically when graphite if present will be seen and a difference in the amount may be found in the case of different pencils.

To test the colour chemically a little is extracted by placing a drop of alcohol or acetic acid on the writing and then absorbing it by means of filter paper. To the spot of colour thus obtained various reagents are applied, such as nitric acid, hydrochloric acid, stannous chloride, sodium nitrite with acetic acid and sodium hypochlorite with acetic acid and the colour effects produced observed with a lens. The residue left on the paper after the removal of the colour with acetic acid should be tested for iron with a 5 per cent. solution of potassium ferrocyanide. Another useful test is that for chlorides, and for this a drop of silver nitrate is applied directly to the writing.

In a murder case Mitchell gave evidence that writing in copying ink pencil on some fragments of partially burnt paper from the grate of the room where the body was found gave identical results when tested with that of a copying ink pencil belonging to the accused person, who finally admitted that the writing had been done by him. The accused, however, was acquitted on other grounds.[1]

Mitchell has shown that writing done with copying ink pencils is very little affected when immersed in sea water for long periods.[1]

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[1] Copying Ink Pencils and the Examination of their Pigments in Writing. *C. A. Mitchell.* Analyst, 1917, pp. 3–11.

**Handwriting.**—In the examination of suspected forgeries and anonymous documents a study of the handwriting is of great importance. It seems at first sight to be a very simple matter to determine whether one specimen of writing is in the same hand as another or not, and it is quite a common experience for the ordinary man to be appealed to on such a question, and most people are quite ready to give a decision. Even the few who disparage handwriting experts because of the mistakes that have sometimes been made employ the method of comparison of handwriting in their own private affairs, and notice, and even act upon, resemblances and differences found. The untrained person, however, bases his comparison upon the broad features of the writing only and does not as a rule pay any attention to details. Since, however, the aim of the forger is to deceive, a clever forgery, in order to pass muster at all, must necessarily resemble in its general aspect the writing forged, and therefore something more than a superficial likeness or difference is required before any useful opinion can be pronounced regarding the genuineness or otherwise of a specimen of writing. The study of handwriting to be of any value must be conducted according to strictly scientific methods, which can only be done by those possessing special experience, and hence in all cases of doubt or dispute regarding the authenticity of documents the matter should be referred to an expert, for not only may he discover characteristics in the handwriting that make its origin certain, but not infrequently small points in connection with the writing, the paper or the ink, which would be passed unnoticed by an untrained observer, will be found and may prove of considerable value as evidence. Thus in a certain suspicious letter that was being examined it was stated that it had been commenced in a train and finished afterwards in the house, but the internal evidence did not bear this out, the writing being very regular and uniform and no part of it showed any signs of having been done in a moving train and a similar pen and ink had been employed throughout. A mere opinion, however, that different documents are or are not in the same handwriting is of very little value unless the expert is able to demonstrate this in his report or in Court, and when the similarities or differences are substantial this can usually be done by means of enlarged photographs.

All writing may be considered as consisting of a series of strokes, and it is not necessary to know what the strokes mean or how to pronounce them in order to compare them, all that is required being a sufficient knowledge of the language to recognize and follow the same letter or word whenever it occurs.

In theory, the examination of one form of writing should not present any greater difficulty than the examination of any other form of writing, since whatever language a man uses, he unconsciously imparts to the writing certain characteristics which are personal and peculiar, and by means of which his individuality is expressed and may be recognized. In practice, however, the author has found Arabic-writing much more difficult to examine than either English or French, probably because of his imperfect acquaintance with Arabic.

When dealing with suspected forged or anonymous documents the known and genuine writing is first carefully examined and all the important characteristic features noted. The forged or anonymous writing is then similarly treated. Small agreements or differences such as those of size, slant or angle should generally be disregarded, since few people write the same letter or word in an absolutely identical manner on two different occasions, complete identity being generally a proof of forgery by means of tracing or transfer. It is only a mechanical process like printing or photography which produces results that never vary. Small agreements or differences in non-essentials between two specimens of writing and which are of no value as proving the identity or non-identity of the writers, and which should not be taken into account, are a frequent cause of the unfortunate conflict of opinion between handwriting experts, one basing his conclusions on facts which undoubtedly exist but which the other rightly refuses to regard as essential. The expert should always mention in his report that he only claims identity or non-identity between the writings compared in respect to certain fundamental characteristics and not with regard to small superficial details.

A frequent criticism made against handwriting experts is that too few documents are examined and that a certain letter or word stated to be a characteristic of a particular writing may occur in a large number of other cases. This criticism is based on a misconception of the expert's position. In order to prove the identity of the writing on two different documents, the expert establishes the presence in each of the documents of a number of characteristics or peculiarities, as many as possible being unusual or uncommon. Reliance, however, is not placed upon the presence of any one characteristic, since one or even more may be accidental or may occur in other writings, but reliance is placed upon the presence of the same combination of the same characteristics in each of the two documents, care being taken that the characteristics are sufficient in number to exclude the possibility of chance. The absence of fundamental differences is also established. These are the same principles that are employed in personal identification and in the finger-print system. Thus measurements of various parts of the body, the colour of the hair and eyes, deformities and scars are all personal characteristics, but no single one is sufficient by itself to establish identity, which can only be done by a combination of characteristics. In the Bertillon system of identification a maximum of eleven different measurements are taken, and although many hundreds of thousands of people have been examined only a very few cases are known in which two different persons have been found to possess the same combination of measurements, and in such cases other features, and notably the finger prints, are different. As a rule fewer characteristics than eleven are quite sufficient to prove identity, and even one characteristic if it be very marked and uncommon is almost enough. With regard to the finger-print system, Locard states [1] that identity is proved by the agreement of twelve or more characteristics.

The expert should be particularly careful to avoid being misled by a general resemblance of style due to nationality or other cause and should never forget that writing done by different members of the same family is frequently super-

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[1] L'Enquête criminelle et les Méthodes scientifiques. *E. Locard.* Paris, 1920, pp. 128-9.

ficially much alike. It has been noticed too that many Egyptian clerks writing English often have a general similarity of style. As an example of a national characteristic the French custom of making a bar across the down stroke of the figure 7 may be mentioned. In England such a feature would be very unusual and therefore characteristic, but in a country such as Egypt where there is a considerable French influence, it is very common and as a rule need not be specially taken into account.

In the case of anonymous documents not only should the writing be examined and compared with that of the suspected person, but the paper and ink should also be carefully examined, since these may provide important clues. In many instances the writer of anonymous letters is never traced, but in a few cases very definite proof of identity may be obtained, and even when this cannot be done it is sometimes possible to clear a person who has been wrongly suspected. In a country like Egypt evidence of nationality, which can sometimes be proved from the writing, spelling or composition, may be very useful.

In one instance investigated, in which it was important to trace the origin of an anonymous document, this was done from the evidence afforded by the ink, that on the document and some found in a bottle in the house of the suspected person, when examined and compared, being characteristic and identical; it was a blue-black writing ink of poor quality and contained a large amount of finely divided insoluble matter, and had probably been made from ink powder. In another case, well-marked finger prints were found. In another case an anonymous letter was written on a half sheet of note paper, on which a faint reversed impression of a name and address could be deciphered and which had manifestly been present as a stamped heading on the other half of the sheet.

The examination of signatures resolves itself largely into a comparison of the suspected signature with as many others known to be genuine as possible, but includes also as a rule an examination of the nature and age of the ink. Sometimes, however, it may be inadvisable to test the ink, particularly in the case of an Arabic signature, which may consist of a few strokes only, since in some cases it can be foreseen without testing that any evidence obtained would not be conclusive, whereas it may be most important that there should not be even the slightest mark made on the writing, since this might be construed into an attempt to tamper with the signature.

When dealing with suspected signatures it should not be forgotten that although as a rule when once a particular form of signature has been adopted it varies very little and then not fundamentally but only in superficial details, yet there are a small number of people, often not well educated, who habitually modify definite characteristics of their signature, thus for example letters like *a* or *u* may sometimes be written with one stroke of the pen or with two strokes, and the manner in which letters are joined or not joined may constantly be changed, and it is only by examining a comparatively large number of genuine signatures that it can be determined whether or not such changes occur. If possible not less than five or six genuine signatures should always be examined and preferably double that number. In one case in which three cheques each for £E3000 had been forged 83 different genuine signatures were examined and these differed so much among themselves that it was impossible to prove that the suspected signatures were forged merely because certain tricks of writing were present or absent. The proof of forgery relied upon in this particular instance was the practical identity of the three forged signatures among themselves, which extended not only to the manner of writing, the size of the letters and the space occupied by the signature, but even to the precise position of the signature upon the cheque, whereas no two genuine signatures exhibited such identity. It is noteworthy too that the writing of the forged signatures was bolder and freer from tremors and hesitancy than the genuine signatures. The forgeries were probably done by means of freehand drawing over traced signatures, the forger being a better caligraphist than the original writer.

Arabic names as written are frequently very short and offer a very small field for comparison, and superficial differences between two Arabic signatures are sometimes due to one having been written with an Arabic reed pen and the other with an Arabic steel pen or with a European pen, or arise from the fact that one has been written with the paper placed flat on the table or other hard surface in the Western manner, while the other has been written with the paper held in the hand [1] or resting on the knee in the Eastern way. Sometimes, too, some of the letters are written over a second time or are touched up without any fraudulent intention. The greatest caution is necessary before giving a definite opinion regarding the genuineness or otherwise of a signature, and more particularly of an Arabic signature.

In handwriting cases the expert is frequently expected to examine and report upon a document in a few minutes or at most in a few hours when days may be necessary. Whenever such a request is made it should be explained that a superficial examination is not only worthless but may be misleading, and that the results at the best could only be very non-committal, and that no definite statement can be made unless adequate time for a thorough examination is allowed.

*Alterations and Erasures.*—Generally these can be seen with the naked eye, but if skilfully done a microscopic examination is required to detect them. Enlarged photographs are very helpful in this connection. Special hints on the taking of such photographs will be given in the chapter on Photography.

Occasionally documents are submitted to the expert in which it is stated that alterations have been made, but which on examination are found to be perfectly genuine. Alterations may be alleged because the ink has "run" in places, and fraudulent additions may be suspected because some of the writing is unnecessarily close together. It may, however, be possible to prove not only that no alteration or erasure has been made, but that any "running" of the ink is due entirely to the poor quality of the sizing of the paper, and if any part of the writing touches or crosses another

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[1] This can sometimes be proved from the presence of creases in the paper showing where it has been folded in order to hold it. These creases are very characteristic in appearance, and are never horizontal but always inclined across the surface of the paper.

part, the determination of the sequence of the strokes will settle the question of whether additions have been made or not.

Sometimes a suspected erasure turns out to be a smear, or a suspicious appearance may be due to the use of thick ink or a bad pen. Sometimes too, although an alteration has been made it is in an unimportant detail and is manifestly due to the correction at the time of writing of a slight inadvertent error and is of no consequence. Instances occur also in which an unconscious mistake is made at the time of writing in a figure such as a date, an amount representing a payment or a price, and it is claimed afterwards that the figure has been tampered with, but in such cases it can generally be shown that no alteration has been made.

The testing of documents for erasure by means of iodine vapour or heating, both of which are sometimes recommended, is not as a rule either advisable or necessary. If iodine vapour is used, the colour partly disappears on standing and may be completely discharged by means of sodium thiosulphate. Heating is never satisfactory and will probably seriously injure the document.

*Sequence of Strokes.*—A very common kind of fraud in documents is the addition of further writing after the document is completed. In such cases there is frequently very little spare space and the added writing often touches or crosses some part of the original, and when this occurs the sequence of the strokes may generally be determined by microscopical examination and the facts observed with the microscope may be demonstrated in Court by means of enlarged photographs. Great care, however, is necessary before a definite opinion is given as to the sequence of crossed strokes, particularly if either of the strokes has been blotted, since if one stroke is thick and the other thin, the thick stroke will always appear uppermost whether this is actually the case or not. Under certain conditions, however, the second of two strokes crossing one another widens out where it touches the first stroke and the ink from the second spreads upon the first in a manner that is very definite and characteristic, and when this occurs it furnishes absolute proof of the true sequence of the strokes.

**Illegible Writing.**—This refers to writing which has faded or which has been partly effaced, and not to that illegible on account of the manner in which it is written. The possibility of restoring faded writing depends upon the composition of the ink and the conditions which produced the fading. Inks containing iron offer the best prospects of restoration, while aniline inks as a rule cannot be restored. Iron ink may be restored by brushing over with a dilute solution of tannic acid or of ammonium sulphide ; the use of an infusion of galls for a similar purpose was described as early as 1660.

Pencil marks which have been partly effaced may sometimes be deciphered if they are photographed, and for this purpose the light should fall upon the writing from one side and at an angle or they may be developed at the back of the paper by brushing it over with a coloured solution or a coloured powder.

In one case of a partly effaced document written with black-lead pencil on poor quality paper and found on the body of a drowned person it was impossible to develop or intensify the writing, and although ten different photographs were taken under varying conditions of illumination and exposure, none of them was helpful, the writing being best deciphered with the naked eye when the paper was held obliquely.

Sometimes it becomes necessary to decipher writing or printing which has been deliberately obliterated. As a rule this can easily be done, and indelible pencil, blue pencil, blue ink, blue-black ink and printer's ink can all be removed sufficiently by treatment with suitable solvents to enable the matter underneath to be read.

On one occasion in connection with a crime, a letter was found so soaked in blood as to be quite illegible, and it was desired to remove the blood so that the writing could be read. This, however, proved impossible, since the ink used was aniline blue soluble in water and in all the other solvents in which the blood was soluble.

When deciphering writing which has been partly effaced the task is much more difficult when the language is a foreign one, since with a language which is familiar a certain amount of reasoned guesswork can be indulged in by means of which a working hypothesis is obtained, which can be verified or discarded later.

**Secret Writing.**—The most important kind of secret writing is that by means of ciphers and codes, and after this in importance comes the use of invisible or sympathetic ink.[1] The former will not be dealt with at all, as it is entirely outside the author's experience, and also it is not a chemical question. The latter will be treated very briefly, since there are many things in connection with the subject which it is inadvisable to publish.

Any colourless or almost colourless solution that can be used with pen and paper may be employed as an invisible ink, even plain water.

Descriptions of a few invisible inks may be found in various books, as also the methods of developing them, but the possibilities are almost endless, and a large number of such inks could be devised by any chemist for which it would be practically impossible, without being informed of the nature of the ink, to find the correct developer. This does not mean, however, that development would be impossible, but merely that chemical development might be impossible. To realize the distinction it will be necessary to consider what an invisible ink is and in what development consists.

Invisible inks, with a few exceptions, which will be referred to later, generally consist of water containing some chemical ingredient in solution, and such inks are invisible merely because they are colourless or practically colourless.

Chemical development consists in so changing the nature of the ink that it becomes coloured and therefore visible. This change may be brought about by light, by heat or by appropriate chemical reagents.

But the act of writing, whether the ink be invisible or not, does more than merely place the writing fluid on the

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[1] During the war of 1914–18 a proclamation by the General Officer Commanding in Chief His Britannic Majesty's Forces in Egypt prohibited the use of secret writing in the following terms:—"No person shall make use of invisible ink or of any other medium for writing which is not visible unless subjected to heat or to some other treatment."

paper. In the process the surface of the paper, the special physical condition of which is produced by pressure and heat, generally becomes scratched by the writing instrument, and is also partly destroyed, or at least affected to some extent, by the solution used, and even writing made with distilled water and a soft brush affects the paper and can therefore be developed. In some cases too the solution may possibly act upon the sizing or the loading material of the paper. The result of the alteration of the surface is to make the paper more absorbent, and hence if a coloured solution be applied, the colour is retained mechanically to a greater extent in those parts where the writing occurs than on the rest of the paper, and the writing therefore becomes visible. Coloured powders also adhere more firmly to those places where the surface of the paper has been affected by the writing. The application of coloured solutions or coloured powders therefore constitutes a valuable mechanical method for developing invisible inks.

When secret writing is suspected both chemical and mechanical development should be tried, beginning with the latter.

The value of secret writing is not that it cannot be developed unless the nature of the ink is known and the correct chemical developer applied, for most invisible inks can be developed by mechanical methods, but the value consists in the fact that secret writing will probably escape detection unless suspicion is aroused and special search made.

Before applying any method of development, however, the suspected document should be carefully examined in a good light, both in the ordinary way by reflected light and also by transmitted light. When making the examination by reflected light, the light should be allowed to fall both directly on the paper, and for this purpose a strong light is required, and also obliquely, and for this the light should not be too strong.

When glazed paper is used the presence of secret writing can practically always be detected by careful inspection, no matter what the nature of the ink is, unless after the writing is finished the paper is treated in a special manner, which is very simple and not generally known, but which cannot be divulged. This treatment does not affect subsequent chemical development of the ink but reduces the efficacy of some forms of mechanical development or prevents it altogether.

An examination for invisible ink by means of a lens or microscope is not of much value and is seldom necessary.

The methods of development already referred to will now be described.

LIGHT.—Exposure to light develops silver salts, and also dyes, such as fuchsine, which have been decolorized, but in the latter case it is the air and not the light which brings about the development, the process being one of oxidation.

HEAT.—This develops writing made with such fluids as dilute sulphuric acid, urine, milk, lemon juice and other fruit and vegetable juices, solutions of sugar, copper salts and cobalt salts. The most satisfactory manner to apply this test is by means of a carefully regulated air oven, but a hot iron also gives good results, an electrically heated one being the best. Documents on which there are wax seal impressions should of course never be heated in an oven, but always ironed, the wax being avoided : documents made of celluloid, which is sometimes used for Christmas, New Year and birthday cards, should not be heated, since this material unless specially prepared is very inflammable.

CHEMICAL REAGENTS.—The chemical compounds which might be required as developers are innumerable and only a few general reagents of different classes can be tried. The following named are the best :—

*Ammonia.*—For substances, such as phenolphthalein, which form coloured compounds with an alkali and for salts, such as those of copper, which form coloured hydrates.

*Ammonium Sulphide.*—For the salts of metals which form coloured sulphides, such as tin, copper, iron, cobalt and nickel.

*Ferric Chloride.*—For tannic acid, potassium ferrocyanide[1] and some organic compounds.

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[1] Potassium ferrocyanide is most unsatisfactory for secret writing, since it decomposes slowly on the paper and becomes visible.

*Ferrous Sulphate.*—For tannic acid and potassium ferricyanide.

*Potassium Ferrocyanide and Potassium Ferricyanide.*—For ferric and ferrous salts respectively.

*Tannic Acid.*—For iron salts generally.

*Iodine.*—This may be employed either in solution or as vapour and develops a large number of invisible inks. Iodine sometimes acts as a chemical developer, as for instance with starch, but more often merely mechanically. Iodine after use may be decolorized by means of sodium thiosulphate.

The above reagents, with the exception of iodine, are best applied by means of a soft brush, a stroke being made diagonally across the paper as a preliminary trial. Iodine solution is best applied with a moderately hard flat brush. When using iodine vapour a few crystals of iodine are placed at the bottom of a glass vessel, such as a large beaker or dish, in which the suspected document is also placed, and the vessel is then covered and gently heated.

Although it is possible to employ all the various compounds above mentioned as secret inks, it is doubtful whether more than a very few are actually used. The author's experience includes the use of lime juice, onion juice, a lead salt and of several different organic compounds, for one of which there was a simple chemical developer and for another a very complex developer consisting of two solutions. All these inks, however, could be developed by coloured powders or better by coloured solutions.

Coloured Powders.—The powder should either be sprinkled or sprayed on the document or applied by means of a soft brush, the latter method being the best, any excess being blown or brushed off. Almost any coloured powder, even dust or ashes, will do if it is in a sufficiently fine state of division, but graphite, red lead and iron oxide (such as jeweller's rouge) are the best. Coloured powders develop most invisible inks, including water, saliva, urine, milk, sugar, salt, lemon juice, and most chemical reagents.

Coloured Solutions.—The use of such a solution appears to have been first suggested by Forgeot in 1891 for the purpose of developing invisible finger prints on paper, and he employed ordinary writing ink.[1] The modification of this test suggested by Nicefero,[2] also for developing finger prints on paper, is very satisfactory, not only for that purpose, but also for developing secret writing. It consists in diluting ordinary blue-black writing ink with water, applying this to the paper by means of a brush and then washing off the excess. Almost any coloured water solution, however, will do, thus red ink may be used or a solution of any aniline colour. Although the finger print or the secret writing is developed without the subsequent washing of the paper, this latter is essential if the best results are to be obtained. A strong solution of some water soluble blue, such as methylene blue, will be found very satisfactory. This is painted on the paper by means of a moderately hard flat brush, and the paper is then immediately put under a tap and rinsed well with running water. When dealing with a large sheet of paper, or with one that requires treatment on both sides, it will generally be found advantageous to put the paper bodily in the solution instead of using a brush. The procedure followed is identical with that employed for developing photographs, the dish containing the developer being rocked from side to side. This method develops a very large proportion of invisible inks and is one of the most satisfactory for general use. A solution of an aniline dye in alcohol will answer the same purpose.[3] In this case the paper is allowed to remain several minutes in a dilute alcoholic solution of the dye and is then rinsed with alcohol. This alternative method might prove useful in case there was any writing on the paper, other than the invisible ink, which was soluble in water but not in alcohol, and which it was desired to retain.

Most methods of developing invisible ink disfigure the paper, but in some cases, from his knowledge of the compo-

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[1] Des empreintes digitales, étudiée au point de vue médico-judiciaire. *R. Forgeot.* Thèse de Lyon, 1891.

[2] La Police et l'Enquête Judiciaire. *A. Nicefero:* Paris, 1907, p. 174.

[3] An alcoholic solution of an aniline colour is sometimes used to develop finger prints on glass. Manuel de Police Scientific, *R. A. Reiss*, p. 422.

sition of the developer, a chemist can decolorize it after it has been applied or can decolorize the compound formed, and so remove all visible traces of the development.[1]

Dilute solutions of oil in alcohol or other solvent are sometimes employed as invisible inks, and hence form an exception to the rule that invisible inks are water solutions. The oil used is generally castor oil, which is one of the very few fixed oils soluble in alcohol, but essential oils could also be employed. These latter, however, are most unsatisfactory, since they entirely disappear in time by evaporation. Such inks may be developed by soaking the paper in water, when the writing becomes translucent and readily visible. Occasionally, however, the secret writing is of the nature of an artificial water-mark which is made while the paper is wet by placing over it a dry sheet and writing on this latter with a hard and blunt pointed pencil or similar instrument, writing made in this way being invisible when the paper is dry, but can be seen plainly if examined by transmitted light when the paper is wet, and hence can be developed by soaking the paper in water. A solution of bismuth nitrate in dilute nitric acid, which is sometimes recommended for use as invisible ink, can also be developed by water. Both dilute nitric acid and dilute hydrochloric acid can also be used for secret writing and can be developed in the same way, the writing when wet becoming translucent and visible.

It should not be forgotten that all seemingly blank pieces of paper, even though sent as letters through the post, do not necessarily bear invisible writing, a blank sheet being sometimes placed round a letter to prevent the writing showing through the envelope. Sometimes, too, a sheet of blank paper is sent as a specimen or to supply writing paper

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[1] It has been stated in the public press that during the war the Censor's Department employed a developer for the detection of secret writing of such a nature that after use it could be chemically removed without leaving any visible signs that the document had been tested. It would be impossible, however, to eliminate entirely the evidence of the use of this or of any other developer, and although altered in character and no longer visible, the developer could always be detected by suitable chemical means.

to some one likely to be without,[1] or as a pre-arranged signal, or even as a practical joke. Sometimes what is suspected to be secret writing is merely an accidental transfer of other writing, laterally inverted, with which the paper has been in contact.

Writing which has been developed and is still not very distinct often gives excellent results when photographed.

In some instances the manner in which a sheet of paper is folded, creased or marked by indented lines constitutes a code or signal : the water-mark too may be specially prepared and may convey a message or have a secret meaning.

In the case of letters sent through the post, the inside of the envelope and underneath the postage stamp should be specially examined. Even the position of the stamps on the envelope may be a signal. In one case in the author's experience a portion of the lettering on the postage stamp had been obliterated, leaving the figure indicating the value of the stamp to serve as part of a message.

Some of the substances used during the war of 1914–18 as invisible ink were articles of everyday use which could be bought openly and carried without suspicion, others were uncommon organic compounds which were intended to be developed with special reagents, but it was found that they could also be developed by mechanical methods. Some of the methods of carrying and sending supplies of ink were very ingenious. Particulars respecting many of these inks have recently been published. In some cases the ink was made up to resemble medicine, throat gargle, pomade or perfume. One method of carrying supplies was to saturate socks, handkerchiefs, or soft collars with the writing solution and afterwards dry them, and when the ink was required it was recovered by soaking the article in water. The ink was generally colourless or almost colourless and so did not show, and when socks were used they were often black.[2]

**Typewritten Documents.**—At first thought it might appear to be quite impossible to distinguish between one typewritten document and another, if both are done with ink of the same colour, but frequently such discrimination

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[1] Especially in the case of business letter paper with printed heading.

[2] German Spies at Bay. *S. T. Felstead.* London, 1920.

is a fairly simple matter. Typewriting machines have all a distinct individuality and the documents made, not only with different makes of machines but with different machines of the same make, can generally be recognized and even the approximate age of a document can sometimes be determined. It is well known for instance that different makes of machines have type of a different size, and hence the make of machine can be ascertained from a careful measurement of the type used. All typewriting machines, too, deteriorate with age and constant use, and characters made with any machine show defects which did not exist when the machine was new and which do not exist in any other machine even of the same make, and hence a typewriter records its own individuality and its own history in a way that cannot be disputed. A frequent defect is that some of the letters soon get out of alignment, and this provides one of the ways in which the writing of any particular machine may be identified.

With respect to the age of a document, it is well known for instance that typewriters of certain kinds were not made before certain dates and also that alterations are constantly being made in practically all makes of machines in order to improve them and, since many of these changes affect the size or style of the letters, all such alterations therefore constitute definite evidence of age. Thus the capital letters of the Smith Premier machine were narrowed in 1896, and when a few years later a document dated 1893 was produced in connection with an important case which was being tried in Court and which bore the narrow capitals only introduced in 1896 it was manifest that the document was a forgery, and it was finally traced to a particular machine and its full history discovered.[1]

The examination of typewritten documents consists principally in a careful scrutiny and measurement of the type, more particularly with respect to the general form, small peculiarities of form and small defects. The measurement of the size of the letters is best carried out by means of a microscope fitted with a micrometer eyepiece.

**Printed Documents.**—The principal printed documents the expert will be called upon to examine will be banknotes

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[1] Questioned Documents. *A. S. Osborn.* 1910, p. 441.

and postage stamps and possibly share certificates and lottery tickets, but occasionally the identification of the type used for a circular or pamphlet or the comparison of one printed type with another may be required. The examination of printed documents consists principally in a careful scrutiny of the type, together with measurements of the size of the letters, but the composition of the paper and the nature of the sizing should also be determined.

**Banknotes.**—The notes of all banks are occasionally forged, and those of the National Bank of Egypt are no exception. Most of the forgeries are poor, both the design and the colouring being very crude, and it is a wonder they deceive any one, but some have been very well executed. The forgeries have included lithographed notes, notes printed from relief half-tone blocks in zinc, direct photographs on paper coloured by hand and tracings drawn over by hand and coloured. The Egyptian Treasury notes which are of low value (approximately one shilling and two shillings respectively) are also occasionally forged. One case submitted as forgery was merely a clever confidence trick in which a rough negative copy of a banknote played a part. Finished notes were not made, nor was there any attempt to make them, the victim, however, being persuaded that forgeries, which would be quite undetectable, could be executed in a simple and inexpensive manner if he would supply the necessary notes for reproduction, the intention of those who worked the scheme being to secure the genuine notes and then to disappear. A similar case has recently occurred in England.

Some forged notes, as already mentioned, were entirely hand-drawn, and from internal evidence it was manifest that they were the work of at least two different persons. In most instances the outlines of the main features had been traced from a genuine note and the design then worked over by hand and the details filled in. At a distance the general appearance of the face of these notes was fairly good, since the colour approximated closely to that of the original, but the colouring on the back was crude. Some of these notes were of a value of fifty piastres each (approximately ten shillings) but others were for five pounds.

The evidence indicated that the forger of most of the notes was unacquainted with English, but was familiar with Arabic and was able to write it well, though not perfectly, and therefore either that he was not an Egyptian or that he was not well educated. This man was finally arrested and proved to be an uneducated Egyptian whose usual occupation was that of tinsmith. Other forged notes were probably the work of an Englishman or British Colonial, the English being correct and the Arabic nonsense, and were signed " D. Spoof." Forgeries of banknotes executed by hand, however, are not unknown elsewhere, and in 1913 pen and ink copies of Scotch notes for £1 and £5 respectively were made in a convict prison, and during the war of 1914–18 two similar cases were reported in the daily press, one being the forgery of English ten-shilling Treasury notes and the other the forgery of ten-shilling and one-pound Treasury notes, the forgeries being clever pieces of penmanship in both instances.

On one occasion a few specimen notes of a new issue of Egyptian notes were found in circulation, the word " specimen " having been effaced and a forged signature added in indelible pencil.

In several cases a note having very little design or colour has been presented at the bank for payment, with the explanation that it had been sent to the laundry by accident in the pocket of some clothes. This explanation has been corroborated by chemical examination and traces of soap and alkali were found to be present.

The expert to whom forged banknotes are submitted should make himself acquainted with the details of the methods employed for the production of genuine notes.

It is a very general practice for secret marks to be placed on banknotes. These usually consist of a slight intentional defect in the engraving, or of a slight alteration in the form of a particular letter or figure or other part of the design, or in the addition of some small special mark. Such indications are helpful in enabling a genuine note to be easily and quickly recognized, and their absence is a certain proof that a note is counterfeit, no matter how good an imitation it may be. Secret marks, however, are necessarily reproduced by all photographic processes, and hence their value is limited and their presence is not a proof that a note is genuine. Such marks probably never act as a deterrent against forgery.

Banknotes, except those of the Bank of England, are protected to some extent against the photographic reproduction of the design by the use of specially selected colouring. When an ordinary photographic plate is used, red, orange and yellow appear practically black, green is dark, while blue and violet are light, blue photographing almost like white. These difficulties, however, may be overcome by the use of orthochromatic plates and light filters. The colouring too is difficult to match exactly, and hence in this respect also it serves as a protection.

In this connection postal orders and cheques may be mentioned. The former are protected in various ways, one of which is by the use of water-marked paper. Cheques may be protected against alterations by the use of special security paper, the paper being treated with certain chemical ingredients which become discoloured by any reagent employed to remove the writing. In some cases cheques are printed on paper having a coloured surface on the side used, the colour being sensitive and readily showing any attempts at the removal of the original writing. A more usual safeguard against the alteration of cheques, however, is the use of coloured safety ink for printing the groundwork of that portion on which the amount and signature are written, and as this ink is sensitive to the action of chemical reagents, and generally also to the action of water, any attempt to efface the writing is at once betrayed.

**Postage Stamps.**—Forged postage stamps are generally reproductions of rare specimens made to sell to collectors and not forgeries of stamps in current use. During 1873, however, many thousands of forged English one-shilling stamps were sold at a particular London post office for use on telegrams and the fact was only discovered some twenty-six years later.[1] In cases of suspected forgery a photographic enlargement both of the suspected stamps and of genuine ones of the same kind will be found helpful for comparison purposes.

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[1] Stamp Collecting. *A. B. Creeke, Jun.* London, p. 245.

The method of examination of postage stamps includes a careful scrutiny with a lens and a determination of the colour and composition of the paper, the water-mark, the kind and size of the perforations, the colour of the printing ink, the kind and size of type used for lettering, including that of overprints and surcharges, and the nature of the printing process employed, and above all a careful search for minute differences in the design and a careful comparison with genuine stamps of the same kind. It should not be forgotten, however, that even in the case of genuine stamps, slight differences in design and mistakes in printing sometimes occur.

In this connection the re-use of postage stamps from which the cancellation marks have been removed may be considered. The usual method of cancelling postage stamps is by means of a metal stamp and carbon-oil ink, and if the stamp is well impressed with a good blow, the oil penetrates the paper carrying some of the carbon with it, and although the oil may be dissolved out by suitable solvents, the carbon remains and cannot be removed by any chemical means, but only by erasure, which is easily detected. In several cases investigated the removal of the cancellation marks had been clumsily done and with a lens both the signs of the erasure and the remains of the marks could be plainly seen.

The colours with which postage stamps are printed are frequently intentionally sensitive and fugitive and readily show any alteration or attempt at removal of the cancellation marks, the ink being acted upon and partly dissolved by any solvent employed. Stamps for fiscal purposes or combined postage and fiscal use are sometimes printed with doubly sensitive ink. Sometimes, too, safety paper has been used, as for example in the English fourpenny stamps of 1855–6. Another form of protection which has been applied to paper for postage stamps and postage envelopes consisted of the insertion in the pulp of threads of cotton, linen or silk. The chalk surfaced paper now so extensively used for postage stamps also constitutes a protection against the fraudulent removal of cancellation marks.

**Impressions of Seals.**—In the East it is a very common practice for names to be impressed on documents by means of an inked metal seal instead of the documents being signed in the Western manner. This practice is not confined to those who are illiterate, but is very general, and even in Government offices the less important documents are frequently sealed. The seal is inked as a rule from a pad, a coloured aniline ink being generally used. Such seal impressions when made with aniline ink can readily be transferred from one document to another, and such transfers cannot easily be detected. The methods of transfer mentioned in several books yield very poor results, and much better ones are available, though it is not advisable to describe them. The practice of using an aniline ink for the seal impression on an important document is therefore most dangerous. Aniline inks, too, are unsuitable for such a purpose on account of the ease with which they fade on exposure. Oil-carbon inks are much safer, and with a suitable ink transfers cannot be obtained when the impression is more than a few days old. The metal seals themselves are sometimes counterfeited, but impressions made with forged seals are generally easily detected when sharp, though less easily recognized when the impression is a poor one, which unfortunately is very frequently the case. It should not be forgotten, however, that in cases where the same seal is used it is sometimes cleaner or better or more uniformly inked than at other times, and so the impression may appear superficially different. Sometimes, too, the ink spreads on one side of a seal in such a way as to cause the impression to appear larger and of a somewhat different shape than usual. It is also by no means an uncommon occurrence in cases where a seal impression is not very distinct for the outlines to be traced over with pen and ink, and this is not done with any fraudulent intention. Sometimes, however, a seal impression is cleverly forged by means of a tracing inked over. Such a forgery is very evident when an enlarged photograph is made.

Before it can be stated whether a given impression is a forgery or not it is necessary to compare it carefully with an impression of the genuine seal and to measure all the important lines and angles. For measuring purposes a thin glass or celluloid plate ruled in small squares which can be placed over the impression will be found very useful. Although much may be done by direct comparison, indirect methods, in which the impressions to be compared are photographically enlarged, are much more satisfactory. This subject will be dealt with further in the chapter on Photography.

By means of a chemical and microscopical examination of the ink it is sometimes possible to state whether two or more seal impressions having the same superficial appearance and colour have been made with the same ink or not, but as a rule neither the age nor the relative age of the ink can be determined from chemical evidence, since it is generally either carbon or an aniline colour.

The date when seal impressions in ink were first used on documents in Egypt is not known, but a seal impression in black ink has been noticed on a document at the Government archives dated 1184 A.H. (A.D. 1770) and seal impressions in green ink were found on a number of documents dated respectively 1284, 1286, 1287 and 1288 A.H. (A.D. 1867, 1869, 1870 and 1871).

In connection with seal impressions, attention may be called to the results of the examination of a number of mediæval English wax seals by Dobbie and Fox,[1] ranging in date from the thirteenth to the beginning of the sixteenth century. The seals were found to consist either of beeswax alone or of beeswax mixed with resin in various proportions. In two cases the resin was colophony, but in the other cases it could not be identified. The red seals were coloured with vermilion, the green with verdigris, the brown and black with verdigris and organic matter. The beeswax which formed the sole constituent of an impression of the Great Seal of 1350 was practically unaltered in chemical and physical properties. A thirteenth-century seal, which consisted of beeswax alone, had been attacked by mould and had become flaky and disintegrated.

**Illustrative Cases.**—1. In an important land case in connection with which one hundred and sixty-eight documents were examined, one hundred and sixty-three being

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[1] Chem. Soc. Trans., 1914, p. 795.

forged, the methods of forgery adopted embraced practically all those known, some of the documents being entirely counterfeit, others being genuine documents tampered with in almost every possible manner, such as by the alteration of names and dates, the forging of signatures and seal impressions, the piecing together of parts of separate documents to make an entirely new document, the addition of forged endorsements, the burning and staining of certain parts, the mounting on other paper to hide signs of erasure, and the removal of the whole or part of the original writing to make room for other writing. In the case of the title-deed of the land in dispute, which was a large document of between seventy and eighty lines and containing some seventeen hundred words, the whole of the original writing, except the signatures, had been removed and replaced by other writing, this being rendered possible by the document having been written with carbon ink on a thick, glazed, parchment-like paper. Several interesting points in connection with this document may be mentioned. In most deeds of this kind which have been examined it has been found that if the last line of writing would not normally end at the left-hand side of the paper or at the left-hand margin (Arabic writing is from right to left) the last words are stretched out, or the last letter is elongated, so as to reach the edge of the paper or the margin, and immediately below the last line of writing and immediately above the signatures of the witnesses (the signature to a deed of this kind is at the top, but the witnesses sign at the bottom) a line is begun at the right-hand edge of the paper or at the right-hand margin and stretches right across the paper, ending with the word "*shehoud*" (witnesses); that is to say, the first letter of the word "*shehoud*" commences as a long stroke at the other edge or margin of the paper. In one such document examined the line stretched continuously across the paper from one margin to the other, the word "*shehoud*" being absent, while in another document the writing was continued down almost to the bottom of the paper and no line was present, the signatures of the witnesses being in the margin. In only one other case among those examined (about a hundred altogether) was a line absent.

In the case of the document in question only part of the characteristic line alluded to was present, the missing portion being that on the right-hand side where the line normally begins, and the writing of the document, where the line was missing, extended down to the signatures. Parts of the missing portion of the line, however, were still faintly visible in places, and it was evident that the line at one time had extended across the paper in the usual way. The last few lines of writing too were unduly crowded together, although plenty of room existed below the signatures, which could only mean that this space was not available and that the writer therefore was limited to the space above, and hence that the signatures already existed at the time the writing was made.

The ink with which this document had been rewritten was of a brown colour and was possibly sepia, though this could not definitely be proved. Sepia, however, was found in the house of one of the accused persons.

The original glazing of the paper had in large part disappeared and the paper had been scorched by fire in order to hide this and other suspicious indications.

2. A register was submitted for its age to be determined, it being suspected that an original register had been made away with and another specially written one substituted. The book was new-looking and only slightly soiled, and had the appearance of having been very little used. The paper label on the back was also remarkably clean, and was partly detached. In the case of other similar registers the labels had come off entirely at some time and had been fastened on again with extra gum, abundant traces of the operation being left. Nothing of the sort, however, had happened with the register in question. At the end of the register a sheet of foolscap paper had been inserted for use as an index. Other registers had been treated in a similar manner, but in this case the foolscap bore a dated water-mark which showed it to be of a later issue than would probably have been the case had the register been the original one. A chemical examination of the ink proved it to be an ordinary iron ink, and provided abundant evidence that it was all approximately of the same age, and that part of it at any rate was

not as old as certain of the entries purported to be, some of which were dated more than twelve months previously: all the ink was fresh-looking and had probably been blotted, and in no case was it black, even to the naked eye, and it was all very blue when seen under the microscope.

3. A register which was presented in Court in support of a case was found on examination to have been specially prepared. The dates of the entries extended over rather more than a year, and it was stated that the register had been kept and entered up in the office of a mill. The book, however, was clean and new-looking and quite free from dirt and dust, the ink was a bluish-green aniline ink, and so could not be dated from chemical evidence, but it was exactly the same colour throughout, and the writing was in the same hand and was all done with the same or a similar pen, but showed signs of haste or fatigue towards the end. In two instances an entry which belonged to several lines below had been made and afterwards erased, and the dots under a word to indicate repetition, which occurred plentifully on every page, had in some cases been written vertically on a number of different lines at the same time, the ink showing progressively diminishing intensity of colour from the top to the bottom of a page.

4. In several instances letters enclosed in envelopes, which have been submitted in support of cases, have been found on examination to be fraudulent, a genuine envelope with a postmark of the required date having been obtained, and a letter forged to accompany it. Forgeries of this kind may generally be exposed by a difference in the nature or age of the ink or by a difference in handwriting. In one case a grease stain, going through the envelope from front to back, did not occur on the letter; in other instances the envelopes have been mutilated.

5. A suspicious recent appearance of some writing, which was being produced as evidence in Court, was stated to be due to the fact that the ink used was a mixture of writing ink with copying ink. Fortunately writing made with each of the inks in question was available for comparison and the explanation given was shown to be untenable.

6. In one case the author gave evidence that a certain

anonymous document in Arabic written in copying pencil was in the same handwriting as a document written in ink and signed with the name of one of the accused persons, who, however, denied all knowledge of either document and stated that even the signed document had not been written or signed by him. At the request of the prosecution the accused wrote the contents of the signed document from dictation in Court, once in pencil and once in ink. The writing on these dictated documents was so different from both that of the signed document and that of the anonymous document that the author was unable to certify that it was in the same hand. The accused, however, who was a student, had made a written application while under arrest to be allowed to sit for an examination and this had been allowed, and the application and the examination papers were obtained and compared, with the result that the writing of all these was found to be identical with that of both the signed document and the anonymous document and quite different from that done in Court. The explanation was that the accused, who had naturally been present when the characteristic features of his writing had been pointed out in evidence, had spent the intervening time (about a month) in practising a disguised hand, which differed considerably from his usual writing, and from which the normal characteristics had been eliminated.

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## Chapter VII

# DUST AND DIRT

Dirt has been described as "matter in the wrong place," and it is the business of the chemico-legal expert to discover the place to which this matter, when found in connection with a crime, rightly belongs, and in so doing he may provide a valuable clue to the identity of the criminal.

The subject of dust and dirt on clothing has already been mentioned in the chapter on Clothing, and the methods of collecting the dust and dirt for examination have been given and need not be further discussed.

In a country such as Egypt one kind of dust frequently met with consists of sand, and the examination of sand if found in connection with a crime may prove very useful, since the sand occurring in different localities may be of totally different appearance and composition, the river sand, for example, being siliceous, the desert sand frequently calcareous, the sand on the Mediterranean coast calcareous with numerous shell fragments, the sand at Aswan largely composed of granite detritus, while dune sand is very characteristic.

It cannot be hoped that evidence of first-class importance will be obtained every time a specimen of dust is examined, and in many instances the results will be negative, but even these may be of value. As examples of negative results, the following may be mentioned :—In one case the ashes of a fire were examined for the presence of calcined bones or other human remains, but nothing of the sort was present; in another case a suspicious black deposit in thin flakes, which proved to be carbon, found adhering to the arm of a chair in a room where a murder was committed, could not be connected with the crime, and in two instances in which the finger-nails of persons suspected of being concerned in the making of bombs were examined nothing incriminating was found.

METHOD OF EXAMINATION.—The dust and dirt should first be examined with a lens and afterwards with a microscope. If there is a sufficient quantity, a small portion should be heated to redness in a small platinum dish or crucible, to destroy any organic matter present. Any smell during burning should be noted, and the inorganic residue left should be examined microscopically. As a rule the amount of dust and dirt available will not be sufficient for a quantitative chemical analysis, and this is rarely necessary, but qualitative tests should be made, and micro-chemical tests will frequently be found of service.

**Illustrative Cases.**—1. Some sand found in the pockets of the waistcoat mentioned in connection with the subject of Clothing proved to be extremely fine quartz sand with well-rounded grains of uniform size similar to dune sand, and from this it was concluded that the wearer of the waistcoat had been in a locality where there were sand dunes.

2. Sand taken from the barrel of a gun was proved to be identical with the sand from the place where a crime had been committed, and of which the owner of the gun was suspected. The value of this fact, however, was discounted by the further fact that similar sand could be found in many other localities, but in connection with the particular circumstances of the case the fact was of value.

3. Sand taken from the stomach and duodenum of a drowned man was examined. As the quantity of material available was very small indeed, only a microscopical examination was possible. The sand was found to consist essentially of colourless grains, all very angular and many having definite crystalline form which proved to be selenite: there was an absence of quartz, but on one slide there was part of a diatom. The presence of selenite in large proportion and the absence of quartz seemed to preclude the Nile valley and suggested the northern shores of Egypt. It was probable therefore that the man was drowned off the coast where his body was found and that he had not been transported there after having been killed elsewhere, as was at first thought might possibly have been the case.

4. In several cases of counterfeit coining the fact that the working parts of machines seized on the premises of the accused persons were well oiled and were free from accumulated dust and dirt has made it very probable that these machines had been recently used. In another coining case a tiny particle of metal, similar in composition to that used for making the counterfeit coins, was found on a file in the house of one of the accused persons.

5. A safe had been robbed, the door having been bored through, and some of the finely powdered material used as a fireproof packing between the two walls of the safe was scattered about the room. This was found to be entirely mineral, to be free from fibres and organic matter, and to contain a number of small rounded particles of various colours, which manifestly had been fused and which were very characteristic. Some dust found on the clothes and between the fingers of two persons suspected of the robbery was identified as consisting of this same material and the men were convicted.

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## Chapter VIII

# EXPLOSIVES AND EXPLOSIONS

Questions having reference to explosives and explosions submitted to the chemico-legal expert generally concern high explosives used or intended to be used for criminal purposes, or are connected with explosions in cases where crime is suspected. The examination is largely chemical.

In the case of explosives, and particularly of bombs, it is usually better that the expert should see the articles where they are found rather than that they should be sent to him.

### Explosives.

The principal manner in which high explosives are employed criminally is in the form of bombs, which have been a favourite weapon of assassination for many years. During the war of 1914–18 immense numbers of bombs were employed by the various armies and the knowledge of the manufacture and use of bombs became very widespread, and it will not be surprising therefore if bomb outrages are more common in the future than has been the case in the past.

Bombs vary considerably in size, shape, nature of envelope, nature of explosive and mode of initiating the explosion, but it would be unwise to give any detailed description of them.

The examination of live bombs is always attended with danger, and all possible precautions should be taken to avoid accidents. The best preliminary examination of a suspected bomb is to have a radiograph taken, when in many cases the nature and disposition of the contents become plainly visible.[1] To radiograph anything, however, necessitates a considerable amount of handling, and this is generally inadvisable when dealing with bombs of unknown composition. In the case of a series of bomb outrages the later bombs will probably be of the same general type as the earlier ones, and hence by studying these a good idea of the nature and disposition of the explosive material may be obtained which will be of great assistance in opening any live bombs found.

It is impossible to lay down any but very general rules to be applied to the opening of bombs, since these are of so many different kinds, each of which needs special precautions, and it would be impossible to describe the various types of bombs without supplying information that might be of service to criminals. The following guiding principles, however, may be helpful:—

(1) On no account to shake or invert a bomb.

(2) If there is a lighted fuse, this must be extinguished, which can be done with water. A general impression exists that the best way of treating a bomb in order to render it harmless is to place it in water, but such a method should not be adopted indiscriminately, since bombs in which metallic sodium is used, or even those containing sulphuric acid, might be exploded in this manner; water, however, is very useful in putting out a lighted fuse attached to a bomb.

(3) To place the bomb on a perfectly flat solidly constructed table or other flat surface and, unless it is of a shape that will not stand upright, to keep it absolutely vertical.

(4) If the bomb is of an army type, an ordnance officer with experience of bombs should be consulted before attempting to open it, unless the expert has had personal experience of such bombs.

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[1] Several instructive reproductions of such radiographs are given by Brouardel. Les Explosifs et les Explosions. *P. Brouardel.* Paris, 1897.

(5) To examine or open the bomb in such a place and under such conditions that an explosion would cause the minimum damage to life or property.

(6) To study carefully the probable effect of each step before it is taken.

(7) To work slowly and gently, using as little force as possible.

(8) If a detonator or percussion cap is visible, this should be removed the first thing whenever possible.

(9) If any screws are visible near the top, these should be taken out and, if the cover is screwed on, it should be carefully unscrewed.

(10) If when the bomb is opened a glass bottle or tube is seen, this will almost certainly contain sulphuric acid. If the bottle is not already securely corked, which is unlikely, this must be done (taking care not to break it by corking too tightly) before either the bottle or any of the other contents of the bomb are removed. If the acid is contained in a closed glass tube this will probably be very fragile, and may contain a lead weight at the bottom, hence great care is necessary in handling it. Sometimes it is preferable and possible to remove the rest of the contents, leaving the acid to the last.

(11) If the package suspected to be a bomb is tied with string or fastened with wire, and if this is not all visible, but if a part enters the package, this particular piece must not be untied or cut, since it might hold up a hammer or weight which when released would explode the bomb. The string or wire must also not be pulled, since it might be attached to a glass tube containing acid, which would thus be broken.

(12) If when the bomb is opened a spring is seen, this will probably be attached to a hammer, the fall of which would explode the bomb, and the spring therefore must not be released until something is effectively interposed between the hammer and

the object below, or until this object, which may be a percussion cap or a tube of acid, is removed.

(13) Clockwork mechanism for firing a bomb can generally be detected by the sound. Such a bomb must be so handled as to prevent the hammer from striking any explosive composition or any glass vessel containing acid that may be present, and anything of this sort should be carefully removed.

(14) As a last resort the bomb should be taken on to a large vacant plot of ground and exploded and the fragments examined.

Several ways of opening bombs are mentioned in various books, but the author has had no personal experience of any of them. One of the methods is very ingenious, but is only applicable to metal receptacles which are closed by means of solder. This method consists in fixing the bomb in a deep stone basin into which mercury is poured until the basin is full, when the sides, bottom and cover of the receptacle fall apart, the solder being dissolved by the mercury. Another method recommended when dealing with thin metal vessels having a cover simply fixed on, but not soldered or rivetted, is to place the vessel between two vertical supports, one of which is fixed and the other movable, and the latter, being operated from a distance, is then gradually pressed against the vessel in order to deform it, when the cover becomes loosened and may easily be removed.

Neither of these last two methods, however, would be suitable for bombs of the kind used in the attempts on the lives of the Egyptian Ministers in 1919 and 1920, which were particularly dangerous to examine, as they contained a small open bottle of strong sulphuric acid packed in a mixture of potassium chlorate and picric acid, and exploded immediately a drop of the acid reached the chlorate. Two of these bombs, while in possession of a youth who intended to use them, were exploded accidentally by being incautiously placed on a seat the surface of which was not level, one apparently falling over on to its side and exploding, thus causing the other to explode also, with the result that two persons were seriously injured and the room wrecked.

In the examination of a bomb, its appearance, nature and construction should be carefully noted and described, and the bomb, after having been opened, should be weighed and measured and the explosive material weighed and analysed. Fragments of bombs which have exploded should also be weighed and measured, since details concerning the size and capacity of the bomb and the thickness of the metal are likely to be required subsequently, either for comparison of one bomb with another or in order to answer questions raised in Court or to meet arguments advanced by the defence if the case is tried. All fragments should be carefully examined for traces of the explosive.

The expert should also visit and examine the scene of the outrage at the earliest possible moment, since if there is any subsequent trial he is almost certain to be questioned as to the manner in which the explosion occurred. It is also better whenever possible for the expert to assist the police in collecting the débris and in searching for indications likely to be useful in proving the nature of the bomb or explosive used.

When picric acid is exploded it is not as a rule all consumed, a little of the unburned acid being scattered about, and yellow or greenish-yellow stains can generally be found on the fragments of the bomb and sometimes on articles which have been hit. The tests for picric acid will be found in the chapter on Clothing.

In the case of bombs such as those described, traces of free sulphuric acid will be found on the pieces of the metal holder of the acid bottle, and sulphate on most of the bomb fragments, as also potassium and chloride, the presence of which will indicate the use of potassium chlorate. In one instance a particle of unaltered potassium chlorate was found in the depression on the head of a brass screw on the outside of a piece of the casing. Sulphuric acid, sulphate, chloride and potassium are tested for in a water solution in the usual way, the best test for potassium being the flame test. The best test for potassium chlorate is also the flame test, together with the nature of the action of strong sulphuric acid upon the solid material.

Cartridges of dynamite, gelignite or similar material should be weighed and measured and the explosive material analysed, and the wrappers should be examined for indications, such as the manufacturer's name or other marks of origin.

Analysis of Explosives.—As a rule all that is necessary in the way of chemical analysis of explosives is a qualitative testing to ascertain the nature of the material. Occasionally, however, a quantitative analysis may be required, as for example in order to identify an explosive used or intended for criminal purposes with some other explosive which is possibly the source from which the quantity used has been obtained, or when it is thought that the results of a complete analysis might indicate the place or country of origin, or when the explosive is of amateur make and the details of its composition might be useful. Explosives are of so many possible varieties that no attempt will be made to give methods of analysis. The nature of the material can generally be recognized from its appearance, and a qualitative analysis is then made to confirm this. The methods of analysis will be found described in the standard text-books on the subject. The tests for picric acid have already been given in the chapter on Clothing.

Gelignite and similar explosive material should always be cut with a horn or bone knife and never with steel, and care should be taken not to handle an explosive containing nitro-glycerine more than is absolutely necessary, otherwise the very unpleasant symptoms of nitro-glycerine poisoning (violent headache, profuse perspiration, vomiting and diarrhœa) will probably be experienced.

One difficulty the expert will have in dealing with explosives is that of getting rid of the residues of the samples. In the case of materials such as blasting gelatine, gelignite, dynamite and cordite, the materials should be placed in an open space, saturated with petroleum spirit and burned, the fire being started by means of a fuse, care being taken so to arrange and light the train that the flame will travel against the wind. Black powder and potassium chlorate explosives may be rendered harmless by dissolving out the soluble ingredients with water. Nitro-glycerine may be destroyed by means of an alcoholic solution of sodium hydroxide, but is better burned in small quantities at a time. Detonators cannot be destroyed with safety, and therefore should be taken out to sea and thrown into deep water.

### Explosions.

None of the cases concerning explosions submitted have been of sufficient interest to merit detailed description, though several may just be mentioned to show the nature of such cases. One involved the testing of the stability of the explosive (ballistite) in a shell which had escaped destruction during an explosion at an ammunition store, as a consequence of which one man had been killed and certain damage to property had occurred, for which claims for compensation were made. Another case necessitated an opinion on the facts elicited at an enquiry concerning an explosion at a store containing high explosives (blasting gelatine and gelignite) used for mining and quarrying. The cause of the accident in both instances was undoubtedly spontaneous decomposition due to the fact that the explosives in question had been stored in a hot climate for several years, in one case partly in the Sudan and partly in Cairo, and in the other case chiefly in Upper Egypt. In a third case the gelignite charge of some bombs made locally in Egypt during the war of 1914–18 was found to have deteriorated so much after about two years' storage as to have become unsafe. The gelignite was packed in an inner zinc cylinder enclosed in a brass case, and the former had become very corroded, and several small explosions took place on handling the outer brass cases after the inner cylinders had been removed. As the bombs were manifestly unsafe, they were destroyed by throwing them into the sea.

**Illustrative Cases.**—1. In the case of the bombs thrown at various Egyptian Ministers during 1919 and 1920 it was possible from an examination of the fragments to say exactly what the bombs had been and to follow the progress of evolution of the different types used. Thus in the first few bombs made, which were found by the police before they were used, the casing consisted of a long cylinder of thin galvanized iron. The cylinder was then made smaller and three bombs of this smaller kind were employed, the casing, however, in one instance being tinned iron in place of galvanized iron. After this the type was changed, and a small bomb with a thick iron casing was used. Portions of six such bombs, differing slightly in size but identical in construction, were examined. All the bombs contained iron nuts or pieces of iron rod as projectiles, but the fragments of the casing itself in the last type used made much more dangerous projectiles and frequently weighed between 100 and 200 grams each, and in one instance a single piece weighed 362 grams. The explosive material was picric acid in every case and there was no detonator or fuse, the explosion being initiated by the action of strong sulphuric acid upon potassium chlorate. The acid was contained in a small open or loosely closed bottle fixed upright in the case, and the explosion took place when the bomb was inverted, and not on impact, most of the bombs exploding in the air. Although fortunately none of these bombs hit or hurt the Minister at whom they were aimed, the motor-car in which the Minister was travelling was seriously damaged on several occasions. One man was killed, a number of others were wounded, and some slight injury was done to property in the vicinity of the explosions. Other and larger bombs of the same type were subsequently found by the police and were examined.

2. The bomb thrown at the late Sultan Hussein of Egypt in 1915, and which fortunately did not explode, consisted of a mass of gelignite weighing 338 grams, into which were inserted a large number of the iron studs used for fastening the bands of cotton bales, as also a detonator and fuse, the whole being wrapped in a newspaper and then in thin pliable leather to make it resemble a harmless ball. The failure of this bomb to explode was due to the fact that a small amount of sawdust had been left in the detonator,[1] and although the fuse burnt through, the amount of sawdust was sufficient to prevent the flame from reaching the fulminate. A second and similar bomb was found in the room from the window of which the first was thrown, but in this case there was no sawdust in the detonator, and in all prob-

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[1] Detonators are usually packed in sawdust.

ability therefore the second bomb would have exploded had it been used instead of the first.

In the case of these bombs the composition and weight of the gelignite made it probable that each had been made by massing together eight of the large size of Nobel's cartridges, and pieces of parchment paper, such as is used for wrapping gelignite cartridges, remained embedded in the mass: these were all cut out and carefully examined, but unfortunately there was no inscription on them. The exact size of the bomb proved of importance, since one of the questions raised by the defence during the trial was whether it was possible to pass the bomb through a certain opening in a shutter, it having been proved that the shutters of the window from which the bomb was stated to have been thrown were partially closed.

3. In several cases connected with explosives which have been investigated, the size, weight and composition of blasting gelatine and dynamite proved that these materials were not of Nobel's manufacture, which alone can legally be imported into Egypt, and therefore that they were contraband. In one case a large dynamite cartridge, which manifestly had been cylindrical at one time and had become somewhat flattened, was examined, and it was found that there was no nitro-glycerine at all present, and that the material contained a little chloride and a trace of sulphate, and it was concluded that the cartridge had been immersed for a considerable period in water, during which time the nitro-glycerine had escaped, and that while wet the cartridge had been subjected to pressure.

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## Chapter IX

# FIBRES

These may be either animal or vegetable. If in the form of a few small loose fibres the recognition is necessarily microscopical, but if in the shape of a woven material the recognition may be partly microscopical and partly chemical. Loose fibres only will be considered here, woven cloth being dealt with under Textile Fabrics.

There are few subjects that will repay the chemico-legal expert better than a study of the microscopical appearance of fibres, and certainty of recognition can only be attained by long practice. The published illustrations of the structure of fibres, although sometimes helpful, are on the whole disappointing, and anyone relying upon a book illustration and not on his own experience is apt to be misled. The only certain method of recognition is by means of careful comparison with other fibres known to be genuine. In the case of animal fibres it should not be forgotten that hair from different parts of the body of the same animal may vary very considerably, most animals producing two kinds of hair, a long, coarse, stiff hair termed "beard hair" and a shorter, finer, softer and more curly hair called "wool-hair," and these differ not only in the properties mentioned but also with respect to the presence or absence of scales and medulla.

The expert should make himself acquainted not only with the microscopical appearance of the hair of the ordinary common animals and of fibres used for woven fabrics, but he should also be able to recognize most of the principal textile fibres of commerce, such for example as those employed for paper, cordage, brushes, mats, hats, and in the fur industry.

It is a comparatively simple matter to distinguish hair from other fibres and human hair from animal hair, and with experience it is not difficult to differentiate between the hair of different kinds of animals, and occasionally it is even possible to determine the region of the human body from which a particular specimen of hair has been derived.

Sometimes it is useful to know whether certain hair has fallen out naturally or has been forcibly torn out, and this may be determined by a careful examination of the roots. If the root is small and club-shaped with a rounded extremity and smooth surface, the hair has probably fallen out, whereas if the root possesses an irregular surface and adherent root-sheaths, the hair has been plucked out.

**Method of Examination.**—As a rule only a few small fibres will be available. A preliminary examination is made with a lens, the appearance, colour, length, nature of ends and whether pointed, blunt or split, and any special features are all noted and recorded. In the case of human hair, it should be remembered that the colour may not be natural, but that the hair may be either artificially coloured or bleached. If the former is suspected, a little of the hair should be digested in dilute nitric acid and the solution tested for metals such as silver, bismuth and lead, which are among the most usual constituents of hair dyes. Fibres other than hair may of course also be dyed.

The fibre is next examined microscopically, but before doing this it is frequently necessary to clean it. Immersion in warm water is as a rule sufficient, after which the fibre is carefully dried. If the fibre is hair, it is then treated with ether to remove fatty matter. Hairs are best examined in water or in Canada balsam, and vegetable fibres either in water or glycerine. It is frequently helpful to stain vegetable fibres before examination, since different fibres give different colours with various reagents. Animal fibres unfortunately cannot be differentiated in this manner.

Many of the colour tests for vegetable fibres given in books are very unsatisfactory, and are not recommended. Some of the more useful of these tests are described when dealing with the subjects of Paper, and String and Rope respectively. Fibres may also be distinguished by their solubilities in various reagents. This test is described in connection with Textile Fabrics. In all colour tests it is important always to employ solutions of the same strength, since variations in the strength of the reagent affect the intensity of the colour produced. If wet, the fibre must be dried before staining. The colour obtained will vary somewhat with the treatment the fibres have received in any manufacturing process to which they have been subjected, and raw fibres frequently give different results from prepared fibres.

For the microscopical examination of fibres any good microscope may be used, but it should be furnished with an iris diaphragm, a polarizing apparatus, a screw-micrometer eyepiece, objectives up to and including $\frac{1}{6}$ inch and $\frac{1}{8}$ inch, and, if possible, a mechanical stage, though this last is not essential. Hair should always be examined not only with full illumination, but with the light restricted by means of the iris diaphragm, and also by polarized light, as in this way the different structures may be seen to the best advantage. Any measurements of the thickness of fibres can best be made with a screw-micrometer eyepiece having two cross wires, one movable and the other fixed. It is impossible to convey any reliable impression of the appearance of fibres by means of a written description, and this can only be learnt by direct observation from fibres of known origin, aided to some extent by illustrations such as those given in the books referred to at the end of the chapter. Micro-photographs of fibres are often useful for attaching to a report or for demonstration purposes in Court.

**Illustrative Cases.**—1. In connection with a question which concerned the identity of several pieces of cotton material, one found wrapped round the body of a murdered woman and others taken from the house of one of the accused persons, it was found that each of the pieces of cloth had attached to it a few threads of the material with which it had been sewn to something else. All these threads were linen, a somewhat unusual material with which to sew thin cotton cloth, and all were of the same diameter and general appearance.

2. On the wax seal impression on an envelope which

figured in a case of robbery a few tiny black-looking fibres were found which proved to be dark blue wool and to correspond exactly with the material of the frayed edge of the turned-up sleeve of the coat of one of the persons employed by the firm who had been robbed. This man, however, had possibly helped to seal up the envelope in the first instance.

3. A small quantity of material tightly packed into the nipple of a muzzle-loading pistol with which a woman was shot was found to be wood fibre impregnated with paraffin wax, and therefore was almost certainly part of a match the wood of which had been dipped in the customary way in paraffin wax during manufacture. In all probability the head of a match had been used to fire the pistol in place of a percussion cap.

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Fibres used in Textile and Allied Industries. *Mitchell and Prideaux.* London, 1910.

The Textile Fibres. *J. Merritt Matthews.* New York, 1913.

Studio Microscopico E Chemico Pel Reconoscimento delle Fibre vegetali, Lane, Peli, Pelliccie. *A. Solaro.* Milan, 1914.

Atlas der Menschlichen und Tierischen Haare. *J. Grimm.* Lahr, 1884.

Le Poil de l'Homme et des Animaux. *Marcelle Lambart and V. Balthazard.* Paris, 1910.

## Chapter X

# FINGER PRINTS

A full consideration of finger prints and finger-print methods of identification is outside the scope of forensic chemistry, and therefore will only be briefly alluded to. The chemico-legal expert, however, should know sufficient about the subject to be able to decide whether or not an impression that he may find in the course of his work possesses sufficient clearly defined characteristic detail to make it worth while submitting to the proper authorities. A chemist, too, can frequently advise as to the best way of developing finger prints. Almost any article touched is liable to retain some impression of the fingers, but many impressions, including almost all those on rough surfaces, are not of much value for identification purposes.

During the examination of questioned documents finger prints are frequently noticed. These are sometimes very manifest and have been made with inky or greasy or otherwise dirty hands. Care should be taken that such marks are not defaced or obliterated, and it is a useful precaution to make a photographic enlargement of them, and should the document be an anonymous communication, such as a threatening or libellous letter, this procedure becomes essential. These photographs should be attached to the report, so that they may be dealt with by the finger-print expert. It may happen, too, that papers are sent to the chemist for investigation, which manifestly should also be examined for finger prints, and in such cases special care should be taken in "handling" them, and they should not be touched except with gloved fingers or by means of forceps. Sometimes, however, finger prints which are almost or entirely invisible exist on the paper. If required, these may be intensified and rendered visible in several different ways; for example (1) by brushing the paper over quickly and uniformly with a dilute solution of silver nitrate (strength about 1 per cent.)[1] and then exposing it to direct sunlight, (2) by exposing the paper to the action of iodine vapour, (3) by brushing the paper over with a coloured water solution, such as ordinary blue-black writing ink as originally suggested by Forgeot, or similar ink diluted with water, and then immediately washing the paper in running water as recommended by Nicefero, or by using a water solution of methylene blue and subsequently rinsing the paper under a tap, and (4) by applying a very finely divided coloured powder such as graphite, an aniline colour, red lead or jeweller's rouge. The powder may be dusted on from a receptacle with a perforated cover, sprayed on or applied by means of a soft brush, the excess being removed by blowing or brushing it off.

To develop finger prints on a dark surface a light-coloured powder such as "grey" powder (mercury and chalk), magnesia or dry white lead is used. All four methods of treatment disfigure the paper, more particularly the first named, and they should not be employed unless and until it is quite certain that the document will not be required again for other purposes. In every case, however, before any attempt is made to develop finger prints, the document should first be photographed. In one case in the author's experience a very faint but just visible finger print was noticed on a document. This was photographed and was then treated with silver nitrate solution in order to intensify it so that a second and better photograph could be obtained, but under the treatment the mark disappeared altogether, probably being washed off mechanically by the solution.

Any finger prints which develop should be photographed and forwarded to the proper authorities for examination: photography is particularly necessary when silver nitrate is employed as the developer, since this gradually darkens on the paper and finally obscures the finger marks. If the

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[1] *Nicefero* (La Police et l'Enquête Judiciaire, p. 171) recommends an 8 per cent. solution, but the author has found a 1 per cent. solution sufficiently strong.

finger prints are to be photographed, neither blue-black ink nor a blue solution nor blue powder should be used for development, since blue is not an easy colour to photograph, but a red dye such as eosine, fuchsine or cotton scarlet either in solution or in the form of powder should be employed instead.

C. A. Mitchell[1] states that the results yielded by the various methods of development of finger prints on paper depend very largely upon the absorptive capacity of the paper, and that in some cases the prints are permanent and can be developed after several years, whereas in other cases the prints show no details after about a week.

To prevent finger prints from being smudged after development Stockis[2] recommends coating them with a solution made from gum arabic, alum and formaldehyde.

When comparing finger prints developed by powders with those developed by solutions, it should not be forgotten that the former are produced by the powder adhering to the moisture, grease and dirt on those parts of the paper with which the ridges have come in contact, and it is the prints of the ridges therefore that are coloured, the result being a positive. In the second case the part of the paper touched by the ridges, being greasy, repels the solution and therefore takes less colour than the rest of the paper, and the result is a negative.

Sometimes it is desired to obtain the finger prints of those who touch certain objects. This is readily done by coating the object in question with a thin film of something that will take and retain the impression of the fingers. For glass a thin layer of gelatine and glycerine, the latter being added to prevent the gelatine drying, will be found satisfactory, while for many opaque objects a thin coating of oil or grease is effective.

If the suspected person can be detained and examined, it is sometimes helpful to coat the article likely to be touched with something that will stain the hands, such as aniline

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[1] *Analyst*, 1920, p. 124.

[2] Annales Soc. Méd. Lég. Belge, 1906, p. 7, quoted by *C. A. Mitchell*, Analyst, 1920, p. 124.

colour or a solution of silver nitrate. This latter may be prevented from drying by adding a deliquescent material like calcium nitrate.

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Classification and Uses of Finger Prints. *Sir E. R. Henry.* London 1913.
Instructions in the Method of Taking Finger Prints. New Scotland Yard. London, 1909.
Dactylography. *H. Foulds.* Halifax.
Circumstantial Evidence. *Wills.* London, 1912.
La Police et l'Enquête Judiciaire. *A. Nicefero.* Paris, 1907.
Criminal Investigation. *Hans Gross.* Madras, 1906.
Manuel de Police Scientifique. *R. A. Reiss.* Vol. I. Lausanne, 1911.
The Detection of Finger Prints on Documents. *C. A. Mitchell.* Analyst, 1920.
Personal Identification. *H. H. Wilder and B. Wentworth.* Boston, 1918.

## CHAPTER XI

# FIRES

FIRES ought to provide a fruitful field of work for the chemico-legal expert, but it is one in which his services are too little utilized, with the result that the origin of many fires is never ascertained even when they are the work of an incendiary, and sometimes they are attributed to spontaneous combustion when this is chemically impossible.

Spontaneous combustion is a comparatively rare occurrence, and should never be accepted as the cause of a fire unless the circumstances have been investigated by a chemist.

There are in Egypt some very interesting cases of what may be regarded as slow spontaneous combustion, but which are never likely to become so accelerated as to give rise to fire. One such example may be seen at the Museum of Antiquities, Cairo. This consists of a mummy in which the wrappings and body are so carbonized that they look exactly as though they had been burned with fire. Other examples of the same phenomenon have been noticed. It seems probable that this carbonization is due to the slow oxidation of the resinous and gummy materials used in embalming, and it is probably this occurrence which has given rise to the statements sometimes met with that the ancient Egyptians burned their dead.

In the Government Analytical Laboratory, Cairo, it has been noticed on several occasions that a cardboard or felt pad on which a wash-bottle containing water was standing on the bench has been charred by the sun's rays focussed through the bottle. Naturally such an occurrence might easily start a fire.

No general rules can be formulated concerning the procedure to be adopted in investigating the origin of a fire, but each case must be studied and dealt with as a separate and special problem. The principal points requiring attention are, first, that a general examination of the whole of the damaged premises should be made; secondly, that a more detailed examination of the spot where the fire originated, or is supposed to have originated, is necessary; and, thirdly, that an explanation of the possible origin of the fire and a description of its progress should be obtained from the officer in charge of the fire brigade, policemen on duty, owner, watchman, employees and those who first saw the outbreak. In all such investigations it should never be forgotten that a competent fireman is an experienced expert, and his opinion should be specially asked for and carefully considered. In many cases it is necessary to take for analysis samples of materials stored on the premises if there is anything that is likely to have caused the fire. Thus inflammable liquids or explosive materials should always be sampled if any portion is left. Oils, particularly oil fuel, should also be sampled. Such obvious things as paraffin oil (kerosene) or oily rags, if present, will probably have been discovered by the firemen, but they should always be looked for if the fire has been extinguished at an early stage. Even in cases where the premises have been completely gutted, and where it is thought that little that is helpful will be found, the expert should not neglect to make an inspection.

Among the points that call for special attention are the following:—

(*a*) The gas meter, to ascertain whether the gas is turned on or off.

(*b*) Gas fittings, to see whether any of the taps have been left open.

(*c*) The presence and position of gas jets, lamps, fires, stoves and furnaces, since vapours from volatile liquids may reach a light or fire from a considerable distance.

(*d*) The presence of inflammable liquids, explosives, strong mineral acids and anything liable to spontaneous combustion.

The fact that petrol vapour is heavier than air and will travel comparatively long distances along the ground is generally unknown, as is also the fact that a very small proportion (from $2\frac{1}{2}$ to 5 per cent.) of certain inflammable vapours in the atmosphere will produce explosive mixtures.

Many fires are of electrical origin, and result from a short-circuit due to some defect in the electrical installation, or occasionally from an electrically heated iron being left in position after use. An electric iron left "on" will in course of time burn anything inflammable with which it is in contact, and a very large fire in Cairo recently originated in this manner. An iron filled with live charcoal of the kind used in Egypt by native tailors is also sufficient to produce a fire. These causes, however, are not primarily chemical, and only concern the chemical expert indirectly, and he should remember that he is not called in to replace the head of the fire brigade but to aid him in chemical matters. When an electric origin for a fire is suspected, an electrical expert should be consulted.

Cotton fires are of frequent occurrence in a cotton-growing country like Egypt, and the loss sustained is sometimes considerable. A brief reference to these fires may be of interest.

For the purpose of fire risks cotton may be divided into two kinds, namely seed cotton, that is to say cotton in the state in which it is gathered and still containing the seeds, and lint or ginned cotton, which is the cotton after the seeds have been separated by the process of ginning.

Seed cotton is comparatively difficult to burn, and although it can readily be ignited, it merely flashes over and the fire soon goes out. This is generally recognized, and unginned cotton is never regarded as a hazardous risk and is not usually insured and very few fires take place, although it is exposed to much the same risks during transport and storage as ginned cotton. It is stated, however, that seed cotton from Upper Egypt, being drier than that from Lower Egypt, burns more readily. During the American Civil War when orders were issued to burn cotton, this proved to be a difficult matter, and the only way that was found at all satisfactory was to set fire to the building in which the cotton was stored and even then some of the cotton frequently escaped destruction.

During ginning a very large number of fires occur, as many as one hundred a day having been recorded in a single ginnery. These fires, however, are provided against and are promptly extinguished, and as a rule cause very little direct damage : they are due to two principal causes, namely matches carelessly dropped into the cotton by workmen at some previous stage, and small stones, sand and grit in the cotton which, coming into contact with the steel knives of the gins, either cause sparks, or probably more generally give rise to sufficient heat, without sparks, to ignite the cotton. The presence of plant stalks and other vegetable matter and also string, all of which are sometimes found in the cotton, will increase the friction and cause heat.

After ginning, the cotton is pressed by means of hydraulic presses into bales which are sent to Alexandria, this being the principal Egyptian port from which cotton leaves the country. At Alexandria, however, the bales are opened and the cotton is sorted and afterwards re-pressed by steam presses into more compact bales for export. Very few fires originate in these export bales, since they are as hard as wood, having a density of about 0·6, and do not readily ignite.

All the serious fires take place in the hydraulic pressed bales from the ginneries, either during transport to or storage at Alexandria. These bales are not very tightly packed, their density being only between 0·2 and 0·3, and after much handling they become very loose, particularly at the corners. Such bales are readily ignited, and ginned cotton when once alight is very difficult to extinguish and may smoulder until the whole is consumed.

When a fire originates on the outside of a bale it is manifestly due to extraneous causes, which may be a lighted end of a cigarette carelessly thrown away or other negligence, to sparks or red-hot cinders from a locomotive, or to incendiarism. When, however, the fire originates inside a bale, as undoubtedly is sometimes the case, it is most probably due to particles of smouldering cotton from an incompletely extinguished fire having been included in the bale at the ginnery.

It is sometimes suggested that since cotton naturally contains a little oil and since additional oil may gain access from crushed seeds, spontaneous combustion is therefore possible. Although there is no doubt that under certain conditions cotton impregnated with cotton seed oil will take fire spontaneously, there is no evidence that cotton fires in Egypt are ever due to this cause. The amount of oil present in the cotton naturally is only a trace (about 0·5 per cent. of wax, however, also occurs, but this is not likely to inflame spontaneously), the amount that gets into the cotton in any one area from crushed seeds is very small and is probably present in every bale, and there are so many other causes that can be proved to operate that it is unnecessary to invoke the aid of spontaneous combustion in order to account for the fires that occur.

In connection with some experiments on the spontaneous inflammability of cotton, the temperature of ignition of various kinds of matches was determined and it was found that ordinary matches ignited at temperatures varying from 40° C. to 85° C. (104° F. to 185° F.) and safety matches from 120° C. to 175° C. (248° F. to 347° F.).

Sodium nitrate is employed in considerable quantity as a fertilizer, and fires sometimes occur during the unloading of the steamers and during the transit of this material by rail or river. This compound is not inflammable, but fabrics and other organic material impregnated with it readily take fire, and when this happens the nitrate aids the combustion by furnishing oxygen. When bags containing sodium nitrate are exposed to wet, some of the nitrate dissolves and saturates the bags, and these when dry again are in the condition of touch paper or tinder and take fire readily and can be ignited even by a spark. In a country like Egypt where cigarette smoking is very prevalent and where men smoke during their work, the immediate cause of such fires is almost invariably a lighted cigarette end carelessly thrown away, except in those cases where the fire occurs on the railway, when it may be due to sparks or red-hot cinders from a locomotive.

**Illustrative Cases.**—1. A portion of a sack which was very much burned and which smelled strongly of paraffin oil (kerosene) was discovered, and when this was carefully examined some wheat husks and a few particles of wheat starch were found inside the bottom seam and bottom corners, and a small amount of whitish material caked in patches on the outside was found to consist of wheat flour. These facts enabled the police to fix the crime upon a certain baker.

2. A sack saturated with paraffin oil was found. This, however, was not a matter of incendiarism of premises but of attempted murder by burning. A sleeping man who had wrapped his head up in the usual Arab fashion woke feeling very hot, and discovered that while asleep he had been covered with a sack saturated with paraffin oil which had been lighted. As it is not an easy matter to light paraffin oil with a match, the greater part of a box of matches had been used up in the attempt and the burned remains of these were found scattered about. The sack had on it an inscription which was illegible, and which it was thought might provide a clue, but although the sack was cleaned and the inscription deciphered, the author of the crime was never traced.

3. One serious fire was caused by the leakage of oil fuel from a small tank placed above a boiler (which was oil fired) into the furnace. The tank was an old one made of thin sheet iron and had a hole in the side which had been repaired. The patch, however, was not rivetted, but soldered, and came off, or partly off, allowing the oil to run down the front of the boiler into the furnace, where it took fire, which spread to the oil in the tank and then to some wooden rafters in the roof. The small tank was filled by a hand pump from a large covered masonry tank below the ground and adjoining the boiler. The flash point of the oil was satisfactory, being 238° F., and the oil in the large tank did not take fire. The oil was very thick and was heated in the small tank by means of a steam coil in order to render it sufficiently liquid to flow readily. There was manifestly either ignorance or negligence on the part of the owner in using an old tank which had been badly repaired and in placing this tank in such a position that in case of a leak the oil would run into the furnace.

4. Three cases proved to be examples of spontaneous combustion, one being caused by the ignition of freshly made

charcoal in small pieces, another by locally made rat poison which contained too large a proportion of phosphorus, which took fire spontaneously and ignited some material with which it was in contact, and the third by the action of nitric acid. A quantity of this acid, which was in a corked bottle, was packed in wood shavings, and adjacent to it in the same box was another bottle containing a mixture of bitumen and turpentine. Probably the box was placed upside down, and if so the acid would gradually find its way through the cork and in contact with the shavings would develop sufficient heat to break the bottle containing the turpentine mixture, or this might have been broken in transit. In any case it is certain either that the action of the acid on the shavings gave rise to sufficient heat to start the fire or that this was caused by the acid coming in contact with the turpentine, on which it would act vigorously.

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Fire and Explosion Risks. *Von Schwartz.* Trans. London, 1904.

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The Chemistry of Fire and Fire Prevention. *H. and H. Ingle.* London, 1900.

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Manuel de Police Scientifique. *R. A. Reiss.* Vol. I. Lausanne, 1911.

Cotton Fires in Egypt: Their Cause, Extent and Prevention. *A. Lucas.* Cairo Sci. J., 1917.

## CHAPTER XII

# FIREARMS[1]

In cases of wounds or death from shooting it is frequently of importance in tracing the weapon used, and so possibly finding a clue that may point to a definite person, to know what kind of powder was last employed with a particular firearm, and also whether this firearm has been recently used or not. Hence a chemical examination of the nature of any fouling present in the barrel may be of considerable value.

The powder used for firearms is of two kinds, namely black powder (ordinary gunpowder) and smokeless powder (nitro-powder).

**Black Powder.**—This consists of an intimate mixture of charcoal, potassium or sodium nitrate and sulphur. There is, however, also always a little moisture present, which in English black powder generally amounts to rather more than 1 per cent. Owing to a temporary shortage of potassium nitrate during the war the use of sodium nitrate has been allowed in England for ordinary gunpowder and H.M. Inspectors of Explosives have now permanently adopted the view that " gunpowder ordinarily so called " may be made with either potassium or sodium nitrate.[2] Although every effort is made by the manufacturer to obtain the required ingredients of as uniform and pure a quality as possible, slight differences of composition cannot be avoided, and although all black powder for firearms, whatever its origin, approximates closely to the same composition, the proportion of the various ingredients is not always absolutely identical. It follows, therefore, that the products of combustion must show variations corresponding to the differences

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[1] This term includes shot guns, rifles, pistols and revolvers.

[2] Annual Report of H.M. Inspectors of Explosives, 1914.

in composition of the original powder, but these variations are so slight as probably to be negligible from a chemico-legal point of view. Although smokeless powder has displaced black powder for many purposes, the latter is still used for some varieties of sporting cartridges. The cartridges loaded with slugs which are supplied to some of the night watchmen (ghaffirs) in Egypt contain black powder. Modern pistol and revolver ammunition is generally smokeless powder.

When gunpowder is burned both gaseous and solid products are formed. This was observed at a very early date, and at the beginning of the eighteenth century the volume of the gas produced was measured. Owing, however, to the primitive state in which chemistry was at that time it could not be determined whether the gaseous products of combustion consisted of one or of several gases. Gay Lussac and Chevreul were the first to give an approximately correct idea of the composition both of the gases and of the solid residue. Other and later investigators were Bunsen, Schischkoff, Linck, Karolyi, Vignotti, Craig, Fedorow, and especially Noble and Abel, the latter two carrying out a large amount of detailed experimental work on the subject.[1] The results obtained will now be briefly summarized.

The principal final products formed when black powder is exploded in a firearm are as follows[2] :—

| | |
|---|---|
| *Gases* | Carbon dioxide. |
| | Carbon monoxide. |
| | Hydrogen. |
| | Methane. |
| | Nitrogen. |
| | Sulphuretted hydrogen. |
| *Solids* | Potassium carbonate. |
| | Potassium sulphate. |
| | Potassium sulphide. |
| | Potassium thiocyanate. |

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[1] Phil. Trans., 1875, p. 49, and 1880, p. 203.

[2] The cap composition (generally mercury fulminate and potassium chlorate with sometimes antimony sulphide) may affect the results very slightly both with black powder and also with smokeless powder.

A little of the original charcoal is left unconsumed but the greater part of this escapes as smoke. A little sulphur is also left, but this probably combines with the potassium monosulphide produced to form polysulphides. It is always stated too that a little potassium nitrate is left, but although the author has frequently tested for nitrate, the results have invariably been negative. A trace of nitrite, however, is generally found after the sulphide has disappeared. Potassium thiosulphate is also stated to be formed, and was always found when tested for, but it is probably not an original product but a secondary product resulting from the oxidation of potassium sulphide. The work undertaken by the author was not a research into the exact composition of the compounds produced by the explosion of gunpowder nor into their methods of formation, but merely an enquiry into what compounds, for which there are good positive tests, that may be applied away from the laboratory if necessary, are present in the barrel of a firearm at various intervals after the firearm has been discharged.

From the nature of the residue left in the barrel attempts have been made to fix the time that has elapsed since discharge took place. Thus Hans Gross,[1] on the authority of Sonnenschein and Classen, makes some very precise statements concerning this matter with reference to black powder. These statements, however, are both incomplete and wrong, and hence may be seriously misleading, especially in a country like Egypt where persons having little knowledge of firearms, and less of chemistry, are sometimes apt to accept definite statements found in a well-known book, like that of Hans Gross, as though they had been conclusively proved and generally admitted, and were true for all time and under all conditions. The statements are as follows:—

| Results of Examination. | Period since Fired. |
|---|---|
| No rust: no crystals of ferrous sulphate: solution yellow, smells of sulphuretted hydrogen and contains sulphide. | Within 2 hours. |
| No rust: no crystals of ferrous sulphate: solution clearer and contains traces of sulphate. | Between 2 hours and 24 hours. |

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[1] Criminal Investigation, 1906, p. 229.

| Results of Examination. | Period since Fired. |
|---|---|
| Rust present: solution contains iron. | Between 24 hours and 5 days. |
| Rust in large amount: solution does not contain iron. | Between 10 days and 50 days. |

The editor of Taylor's Medical Jurisprudence[1] is much more guarded. Briefly summarized, his conclusions regarding black powder are as follows:—

| Results of Examination. | Period since Fired. |
|---|---|
| Sulphide present: charcoal present: strong alkaline solution smelling of sulphuretted hydrogen. | Recently discharged. |
| No sulphide: sulphate present. Solution neutral. | Hours or days depending upon exposure. |
| Oxide of iron present: trace sulphate present. | Considerable time. |

Aitchison Robertson writes[2]: "Gunpowder in a recently fired gun leaves a coating of carbon and the barrel smells strongly of sulphuretted hydrogen. Any further information is inconclusive."

Reiss states that sulphate of iron is found after a lapse of time varying from 48 hours to 72 hours from the discharge of the firearm.[3]

A considerable number of experiments have been made by the author in connection with this matter, and under the conditions of the experiments the results were as follows[4]:—

A characteristic smell which decreases in intensity with lapse of time is present immediately after firing, but even

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[1] Taylor's Principles and Practice of Medical Jurisprudence, 1920. Vol. I, p. 558.

[2] A Manual of Medical Jurisprudence, Toxicology and Public Health. *W. G. Aitchison Robertson.* London, 1913, p. 107.

[3] Manuel de Police Scientifique. *R. A. Reiss.* Vol. I. Lausanne, 1911, p. 492.

[4] The experiments with very few exceptions were made with a 12-bore double-barrelled shot gun, using either Eley cartridges and No. 10 shot or Belgian cartridges and No 6½ shot. About 100 experiments were made altogether. The gun was cleaned from oil before use.

after several weeks some slight smell remains. The greatest caution is required in respect to the identification by odour alone of the components of this smell, and although it can be proved that sulphuretted hydrogen is present at first, the author has never been able positively to identify this compound except by chemical tests.

Sulphuretted hydrogen is present in the gaseous state and may be detected by means of lead test paper, but it quickly disappears, the time taken for the complete disappearance largely depending upon whether the breech has been opened and whether the empty cartridge case has been removed.

Solid matter is deposited in the barrel. As seen at the muzzle end, where alone it can be critically examined, this deposit is generally black and frequently a very intense black but sometimes dark grey, and a clean white fabric introduced into the barrel becomes considerably soiled. The colour of this deposit gradually changes with lapse of time; the black disappears and light-coloured particles are formed, the whole deposit becoming light grey or sometimes even practically white. After a further period, however, brown streaks and patches appear and the deposit becomes partly brown. The black is not entirely, nor even principally, carbon, but consists of sulphide of iron with a little carbon and sometimes a little sulphide of lead. The light-coloured particles contain sulphate, carbonate, potassium, iron and sometimes lead. The brown coloration is due to oxide of iron (rust). These changes are manifestly largely due to the oxidation of ferrous sulphide to ferrous sulphate with the subsequent formation of ferric oxide.

The solution obtained by washing out the barrel with water immediately after firing contains black particles in suspension, is of a greenish or yellowish colour, smells of sulphuretted hydrogen, is alkaline and contains sulphide, sulphate, no nitrite or nitrate, but sometimes a trace of ferrous iron. If various intervals of time, gradually increasing in length, be allowed to elapse before the barrel is washed out, the suspended matter, which is black at first, finally becomes grey or brown, while the solution gradually loses its green or yellow colour, and becomes colourless or brown. The sulphide disappears, but the alkalinity, sulphate and iron remain, and a little nitrite develops. The suspended matter at first contains iron in the ferrous condition, but later part of the iron is in the ferric state.

The time limits found during which the compounds originally present persisted, and after which others were formed, were as follows :—

COLOUR.—As already explained, the colour of the deposit in the barrel, even immediately after a firearm has been discharged, is not always the same, being much blacker on some occasions than on others, and even the two barrels of the same gun fired at the same time with cartridges from the same box sometimes differ. The light-coloured particles generally begin to form very soon and are sometimes noticeable even after one day, but rusting as a rule does not commence for several days. The results, however, are not always the same, and no definite rule can be laid down.

SULPHURETTED HYDROGEN IN THE GASEOUS STATE.—When the breech is kept closed this compound persists for periods varying between two and three hours, but when the breech is opened and the empty cartridge case removed it sometimes entirely disappears in less than one minute and never lasts for more than five minutes.

SULPHIDE.—The greater part of this disappears in about four to five hours, but frequently a trace remains for some time longer, the longest period during which sulphide persisted in the experiments made being ten hours.

SULPHATE.—This is always present from the first, and it does not appear only at a later stage as Hans Gross, Taylor and Robertson would seem to imply : the amount, however, increases, additional sulphate being formed by the oxidation of the ferrous sulphide present.

THIOSULPHATE.—After the sulphide has disappeared thiosulphate may be recognized by the precipitate formed with silver nitrate, which is white at first, but which quickly turns yellow, then brown and eventually black. This compound was occasionally tested for and found to be present. Thiosulphate, as already stated, is a secondary product formed by the oxidation of potassium sulphide.

THIOCYANATE.—This compound was occasionally tested for and found to be present. The intense red coloration produced by ferric chloride in hydrochloric acid solution and destroyed by mercury perchloride was relied upon for its identification.

Iron.—Iron in the ferrous condition is always present in the solid residue from the first, and is also generally present as the merest trace in the solution. After a time some of this ferrous iron becomes oxidized, and ferric iron makes its appearance, first in the solid matter and at a later stage in the solution. The earliest period at which ferric iron has been definitely found in the solid matter has been after 28 hours and in the solution after 33 days. On one occasion a trace of brown, most probably rust, was seen in the barrel after 24 hours, but this was not tested, as it was desired to continue the observations. Since the production of rust is very dependent upon the moisture that can gain access to the gun barrel, the humidity of the atmosphere must be an important factor in conditioning the length of time that elapses before this compound is formed.

Nitrite.—This is never found earlier than after a lapse of four hours and generally does not appear for five hours. Nitrite, however, is decomposed in the presence of alkaline sulphide and hence it never occurs until the sulphide has disappeared or has practically disappeared, when a small amount is always found.

Nitrate.—This was never present on any occasion, but nitrate like nitrite is decomposed by alkaline sulphide; unlike nitrite, however, nitrate was never obtained after the sulphide had disappeared.

Alkalinity.—Alkalinity to phenolphthalein was always present up to periods of sixty days, beyond which the experiments were not carried. Taylor states that the solution becomes neutral, but this was never the case in the experiments made although after two months the alkalinity appeared to be less than after shorter periods. This may be accounted for by assuming that part of the potassium carbonate becomes converted into bicarbonate which is not alkaline to phenolphthalein, though alkaline to methyl orange.

It is not claimed that the above results necessarily represent those found under all conditions, since variations in the powder, the cartridges and the atmospheric conditions might conceivably influence the nature or duration of the compounds formed. One very important fact in actual practice which is frequently not taken into account is that all theoretical conclusions are liable to be invalidated if the firearm was foul before it was fired.

**Smokeless Powder.**—Smokeless powder as used in firearms varies very much in composition. The principal ingredient and sometimes practically the only one, as in the case of Walsrode powder, is nitro-cellulose (gun cotton).[1] Another frequent constituent is nitro-glycerine; thus Ballistite consists of nitro-cellulose and nitro-glycerine, Cordite contains nitro-cellulose, nitro-glycerine and a little vaseline, and Axite consists of nitro-cellulose, nitro-glycerine, vaseline and a small proportion of potassium nitrate. This latter compound is not uncommon as a constituent of smokeless powders, the proportion present in different powders varying from about 1 to about 6 per cent. Barium nitrate is another ingredient sometimes found, and when present it occurs in proportions varying from about 6 to about 29 per cent.; thus ordinary Schultze powder[2] consists of nitro-lignin, barium nitrate, potassium nitrate, vaseline and starch. Amberite[2] consists of nitro-cellulose, barium nitrate, vaseline, potassium nitrate and wood meal. One variety of French smokeless powder contains both ammonium and potassium bichromates. It follows therefore that with such varying composition the nature and amount of the products formed on explosion must also show considerable differences. The principal gaseous products formed are carbon dioxide, carbon monoxide, hydrogen, methane and nitrogen.

The nature of the solid products formed differ with the composition of the powder, and nitrite, nitrate and sulphate are frequently found.

From what has been stated it will readily be recognized that the difference in the nature of the products of combustion of black powder and smokeless powder respectively is

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[1] Except in the case of Schultze powder in which the principal ingredient is nitro-lignin.

[2] This figured in the Monson shooting case.

considerable, and hence there is no difficulty whatever in ascertaining which of the two kinds of powder has been used, though it may not always be possible to determine the exact variety of the smokeless powder.

Very little appears to have been written from a chemico-legal point of view on the subject of the nature of the fouling left in a barrel from which smokeless powder has been fired. The editor of Taylor's Medical Jurisprudence[1] states, however, that so far as he is aware smokeless powders " do not leave behind them any products liable to undergo further changes by lapse of time," but he " has noticed in shooting that white particles undoubtedly collect in the barrel mixed with darker ones, but he is unable to find that any experiments have been made to determine the character of these particles." Aitchison Robertson[2] writes: " Nitro-powders leave only a slight yellow deposit in the barrel."

Since the variation in composition of smokeless powders is so considerable, it is manifest that there must also be great variations in the products of combustion, and this being so it is misleading to group all such powders together and to discuss them as though the differences in composition were trifling and not more than those existing between different makes of black powder.

A considerable number of experiments have been made by the author with various kinds of smokeless powder. The cartridges used were Ballistite, Bonax, Cordite, Eley " Grand Prix," Nobel's " Empire," Primax, Remington, Schultze and Walsrode respectively.[3] The greater number of the experiments, however, were carried out either with Schultze powder or Remington powder, the latter of which consists essentially of nitro-cellulose.

The differences between the results given by the smoke-

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[1] Taylor's Principles and Practice of Medical Jurisprudence, 1920, Vol. I, p. 559.

[2] A Manual of Medical Jurisprudence, Toxicology and Public Health. *W. G. Aitchison Robertson.* London, 1913, p. 107.

[3] The experiments with cordite were made with a rifle: those with the other powders with a 12-bore shot gun and various sized shot, chiefly however No. 10. About 50 experiments were made altogether.

less powders enumerated and those given by black powder are considerable, the two most important being, first that smokeless powder does not form that intense black coloration in the interior of the barrel which is so characteristic of black powder, and secondly that with smokeless powder both sulphuretted hydrogen and sulphide are entirely absent. The principal results obtained may be briefly summarized as follows :—

A characteristic smell is present after firing which decreases in intensity with lapse of time : this smell differs from that given by black powder.

Sulphuretted hydrogen in the gaseous state is absent.

A slight deposit, which is generally black or dark grey in colour, is noticeable at the muzzle end of the barrel immediately after firing : this deposit is never of the intense black colour produced by ordinary gunpowder, though sufficiently dark to soil a clean white fabric introduced into the barrel. The colour does not become lighter with lapse of time as in the case of black powder. Only with Schultze powder and Eley " Grand Prix " cartridges were white particles, such as are mentioned by Taylor, noticed in the barrel. Traces of both ferrous and ferric iron have been found in the solid matter immediately after firing.

The solution obtained by washing out the barrel with water is colourless or slightly brown, and contains a small amount of matter in suspension, generally of a light brown or light grey colour, but sometimes dark grey, and sometimes there is a slight black film (carbon) floating on the surface. This solution does not contain sulphide, but sometimes sulphate (only a trace, and never so much as with black powder), nitrite (always more than with black powder), and generally nitrate (varying from a trace to a considerable amount). The solution is also neutral, which in view of the very general opinion that the fouling in firearms from which smokeless powder has been fired is acid, is noteworthy, and shows that the alkali frequently found in special oils recommended for cleaning purposes, if added to neutralize acidity, is unnecessary and its presence based on a wrong assumption. This is confirmed by the work of Huff published since the statement was first made in Legal Chemistry.[1] Huff states [2] that "The present high-pressure smokeless cartridge leaves no nitro-cellulose residue and no corrosive acid residue. After-corrosion following the use of such cartridges is caused by the explosive deposition of a water-soluble salt or salts in whose aqueous solution steel corrodes, together with subsequent exposure to a high humidity and the presence of oxygen. In the present service ammunition this salt is potassium chloride from the decomposition of the chlorate in the primer."

The different smokeless powders vary considerably in their effects on the barrel; thus a rifle from which Axite has been fired may be left twenty-four hours without cleaning and no marked rusting occurs, whereas after using Cordite the rifle must be cleaned at once, otherwise rust may be found the following day, even in a dry place like Cairo, while in a damp atmosphere, such as that of the Sudd region of the Sudan, the rusting would be considerable.

No marked difference was found either in the composition or colour of the residue left in the barrel when examined immediately after the firearm had been discharged and when examined after various intervals of time had elapsed. It was noticed, however, that the amount of nitrate formed differed considerably with different powders; thus for instance Ballistite gives less nitrate than Bonax, Nobel's "Empire," Schultze or Walsrode powders. This might provide useful negative evidence and it might be possible to prove that a certain kind of powder which was suspected had not been used. The presence of such substances as barium, chromium or potassium would provide useful indications of the nature of the powder.

**Method of Examining Firearms.**—On the receipt of a firearm the first thing to do is to note whether there is any smell at the muzzle end, and if so, its nature and intensity, and also the colour of the interior of the barrel at this end.

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[1] Legal Chemistry and Scientific Criminal Investigation. *A. Lucas.* London, 1920, p. 130.

[2] The Cause and Prevention of After-Corrosion on the Bores of Firearms. *Wilbert J. Huff.* J. Ind. and Eng. Chem., 1920, pp. 862-870.

Sulphuretted hydrogen is then tested for at the muzzle end by means of lead test paper. The breech is then opened and the empty cartridge case removed, if it is still in place, and lead test paper at once applied.[1] If there is no indication of sulphuretted hydrogen at either end, the barrel should be gently blown through from the muzzle end while the test paper is inserted at the breech. The whole length of the barrel is then carefully inspected and its condition noted.

After these preliminary tests the firearm should be held in a slanting position and a small quantity of distilled water[2] poured down the barrel, a little at a time, an effort being made by turning the firearm to reach the whole area of the inside of the barrel. A receptacle to receive the water as it comes out is of course placed at the other end. An alternative method is to swab out the barrel with wet cotton wool, which is then squeezed and the solution obtained examined, but the direct washing out with water is preferable. In the laboratory a wash-bottle or pipette from which to deliver the water, and a small beaker in which to receive it, will be found convenient, but away from the laboratory two small glass bottles with funnels will answer the purpose. As a rule about twenty cubic centimetres of water altogether are quite sufficient. The water that has been through the barrel a first time should be passed through once or twice again, the firearm being turned and held in a different position each time, so that all sides of the interior of the barrel are well washed. It will be found better not to cork the barrel and not to try and shake the solution in the barrel, as this invariably leads to loss. The colour, appearance and smell of the solution should all be noted, and the solution should be filtered. A small portion should be tested for reaction to phenolphthalein and another small portion for sulphide (with acetate of lead). A further portion is acidified

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[1] Since sulphuretted hydrogen, if present, disappears very quickly when the breech is opened and the empty cartridge case removed, this should not be done until the test paper is quite ready to be applied.

[2] Hot water, being a better solvent than cold water, is to be preferred whenever possible, though cold water is quite satisfactory.

with hydrochloric acid[1] and tested for sulphate (with barium chloride), ferrous iron (with potassium ferricyanide) and ferric iron (with potassium thiocyanate). Another portion should be tested for nitrite (with sulphanilic acid and naphthylamine acetate or chloride) and a further portion for nitrate (with phenolsulphonic acid). The colour of the residue on the filter paper should be noted and this residue should be treated with strong hydrochloric acid and the solution obtained tested for ferrous iron (with potassium ferricyanide) and ferric iron (with potassium thiocyanate).

All the tests mentioned except that for nitrate, which requires evaporation, can be carried out in a few minutes almost anywhere. If it is necessary to test for nitrate away from the laboratory, the "brown ring" test can be applied, although strong sulphuric acid, which is required for the test, is not a satisfactory reagent to carry about. The test solutions can be carried in small corked bottles (about 15 c.c. capacity), and the whole outfit can be packed into a very small compass.

Hans Gross states that the examination is "so simple that it may be employed if necessary in the country by any doctor or hospital assistant." Although the testing of firearms like everything else is easy enough when one knows how to do it, nevertheless it should not be undertaken except by an experienced analyst.

In all enquiries connected with wounding or killing by shooting the chemical expert should be called in at the very commencement of the enquiry, and the firearm should be examined by him at once and before it has been handled by others. In this way evidence that otherwise would certainly be lost may be preserved and utilized. If the firearm has to be sent to the expert it should be carefully wrapped up in clean paper, tied and sealed and sent by special messenger.

The chemist, too, should be given the projectile for analysis. The value of this has already been discussed when

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[1] The freedom of this acid from iron should be confirmed by a blank experiment.

dealing with the subject of Bullets. Search should also be made at the scene where the shooting took place for wads, and these should be handed over to the chemist for examination.

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Text Book of Small Arms. War Office, London, 1904.
Treatise on Service Explosives. War Office, London, 1907.

## CHAPTER XIII

# HASHISH (INDIAN HEMP)

THE subject is included here on account of its connection with crime, many criminals in the East being confirmed hashish takers and crime committed under the influence of hashish being not uncommon.

Hashish is the Arabic term for grass, weed or herbage generally, but in addition to this meaning the word has acquired a special and narrower signification, and is applied to a particular plant, the Indian hemp (*Cannabis indica*), and to preparations made from it.

In temperate climates hemp is grown for its fibre and seed, and possesses no pharmacological interest whatever, but in warm countries it develops a peculiar resinous secretion upon its leaves and flowering heads, and this resinous matter when smoked or swallowed induces marked derangement of the nervous sytem. *Cannabis indica*, however, which is the hemp grown in Northern India, Turkestan, Persia, Arabia, in many parts of Africa, in Greece and in Brazil, is identical botanically with the ordinary hemp *Cannabis sativa* of temperate climates. The intoxicating and narcotic properties of Indian hemp have been known in the East from a very early period, and are referred to both by Herodotus and Galen.

As ordinarily used in Egypt, the word hashish means the resinous material from the plant, containing a certain amount of leaf tissue and mineral matter,[1] the whole compressed

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[1] The mineral matter in various samples examined ranged from 24 to 58 per cent. It is not known to what extent this indicates adulteration but in addition to the mineral matter present in the plant tissue, a certain amount will accumulate on the plant during growth owing to the sticky nature of the resinous secretion and additional dust and dirt will gain access during packing.

into a hard cake. Hashish as thus defined corresponds to the Indian "Charas." The Indian "Bhang" and "Ganja" are also derived from hemp. Bhang is generally stated to consist of the dried and broken leaves and small stalks of the plant, together with a few fruits, the whole in the form of a coarse powder, while Ganja is stated to be the flowering and fruiting tops of the female plant compressed into bundles.

Hashish is used in three different ways, and may be smoked, eaten or drunk, smoking and eating, however, being the usual modes of consumption. When smoked, the drug is mixed with tobacco and used either in a water pipe or in cigarettes. In some samples of cigarettes examined the amount of hashish varied from 7 to 16 per cent. of the net weight of the tobacco. When present in cigarettes hashish is readily detected, since it is introduced either in the form of small pellets or more generally in thin strips. If no visible hashish is found, the tobacco itself should be tested by Beam's test, since it is quite conceivable that it might have been soaked in an alcoholic solution of hashish. The author has never actually known a case where this particular method of introducing hashish into tobacco has been employed, but he once examined a strong alcoholic solution of hashish seized by the Customs, which could have been used for such a purpose.

For eating, hashish is mixed with spices and made into a paste with sugar, honey, molasses or fat, or it is introduced into sweetmeats. The paste is taken in the form of pills. When required for the use of excessive consumers to whom hashish alone is no longer sufficient, it is stated that opium or datura seeds are sometimes added.

**Tests for Hashish.**—The tests for hashish are microscopical and chemical. Before the introduction of Beam's test [1] the only way of detecting hashish apart from the microscopical recognition of any leaf tissue present, was by the very characteristic smell, the usual procedure being to extract the suspected substance with some solvent such as alcohol, ether or petroleum ether, to evaporate off the solvent, and to note the smell of the dried extract while warm. Such

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[1] Wellcome Tropical Research Laboratories. Fourth Report, Vol. B, pp. 25-26. Khartoum, 1911.

a test, however, is not very satisfactory even at its best, and it fails entirely when the hashish is present in small amount, or if it is mixed with spices or other odoriferous ingredients, a not unusual condition. Beam's test as described by Dr. Beam himself is as follows: "The suspected material is extracted with petroleum ether of low boiling point and which leaves no perceptible residue when evaporated in the cold. The petroleum ether extract is separated, passed through a filter and evaporated to dryness in a small porcelain capsule. Both extraction and evaporation should be carried out in the cold. To the residue a few drops of weak alcoholic solution of potash or soda (about deci-normal strength) are added, and the liquid allowed to evaporate at room temperature. In the presence of hashish a rich purple or reddish purple colour gradually develops which on dilution with water takes a more bluish cast. It is very permanent."[1]

It has been found, however, that sometimes undoubtedly genuine preparations of *Cannabis indica* such as Ext. Cannabis Ind. B.P. and Merck's purified alcoholic extract, both of which are made by extraction with alcohol accompanied by prolonged heating, fail to respond to this test, and Beam therefore devised a modified test to which he states all hashish plants and preparations respond. This is as follows: "The petroleum ether extract is made as usual, and the evaporation of the solvent is carried out in a short test tube. To the residue is added a few cubic centimetres of a reagent prepared by passing dry hydrogen chloride gas through absolute alcohol to saturation. In the presence of *Cannabis* extract the liquid strikes a bright cherry-red colour, which disappears on dilution with alcohol or water."[1]

The author has frequently employed the original test, and has always found it most satisfactory, but has had very little experience of the modified test, and so cannot criticize it. Occasionally a genuine hashish plant is found which does not respond to either test. In such cases it is possible that the plant is young and that the resinous material has not developed.

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[1] Wellcome Tropical Research Laboratories. Chemical Section. Bulletin No. 3, April 1915. Khartoum.

Hashish is principally used on account of its reputed virtues as an aphrodisiac.

From time to time a number of attempts have been made to isolate the active principle of Indian hemp, and it has been variously stated to be a resin, a glucoside and an alkaloid, but there is still uncertainty about the matter.

## BOOK OF REFERENCE.

Some Notes on Hashish. *A. Lucas.* Cairo Sci. J. 1911.

## CHAPTER XIV

# MICROSCOPY AND PHOTOGRAPHY APPLIED TO FORENSIC CHEMISTRY

### MICROSCOPY.

THE chemico-legal expert would be very helpless indeed without a microscope, since there are few examinations in which this instrument, either in its simple form of a lens or in its more complex form of a compound microscope, is not used. One great advantage the microscope has over chemical analysis is that it does not destroy the material tested. The microscope is especially useful for the examination of documents, fibres, dust and dirt, and is essential in blood-testing for the examination of hæmin crystals and, in conjunction with a spectroscope, for the examination of the various derivatives of hæmoglobin. A microscope too is necessary for the recognition of the crystals formed in the testing of alkaloids with such reagents as picric acid and gold chloride. For general purposes any good microscope may be used, and no particular make is specially recommended, each kind having its own mertis. As a rule, four objectives will be found sufficient, namely 1 inch, $\frac{1}{4}$ inch, $\frac{1}{6}$ inch and $\frac{1}{8}$ inch.

For use with documents a particular form of microscope or stage is necessary in order to avoid folding the documents. Osborn[1] mentions a special document microscope, but the author has found the " Process " microscope[2] a very useful instrument, and it has the advantage of not being expensive. This instrument is made for the examination of lithographic stones and zinc and aluminium plates, and has no stage at all, the tube carrying the objective and eyepiece being at

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[1] Questioned Documents. 1910, p. 80.

[2] Made by Messrs. R. & J. Beck, Ltd., London.

the end of a long arm which reaches over the document, which therefore can be examined without folding. With a No. 2 eyepiece and a 1-inch objective, this instrument gives a magnification of 48 diameters, and with a $\frac{2}{3}$-inch objective a magnification of 72 diameters, and as a rule nothing more than this is required. For the measurement of the width of ink lines in a written document or the size of the letters in a typed document, as also for the measurement of fibres, the microscope should be provided with a good micrometer, either a simple micrometer to be inserted into the eyepiece or, better, a screw-micrometer eyepiece having two parallel wires, one being movable and the other fixed.

For the examination of crystals, mineral fragments, insoluble residues from analytical processes, dust and dirt of mineral origin and also hair, a polarizing apparatus will be found useful, and for general use a mechanical stage is a great convenience though by no means essential.

Three different kinds of lens are recommended, a good high-power folding lens for the pocket, a good reading glass with a handle for examining objects to which a pocket lens cannot be applied, and a "counting glass" with a field a square inch in size such as is used in the cotton trade. This last is a very useful type of lens, since it has a large field, and being in a stand, it can be placed on the article examined, thus leaving both hands free.

Microphotographs are often very useful, and it has even been suggested that for legal cases a permanent record taken by means of microphotography should be made of everything examined under the microscope. While there is no doubt that microphotographs should be utilized more often than is done at present in order to demonstrate in Court facts which can only be seen under the microscope, in actual practice it will be found impossible to take a photograph every time the microscope is used.

As already mentioned,[1] the author is not in favour of taking a microscope into Court, since only those who have been specially trained can either use a microscope or appreciate what they see: a microscopical demonstration, too, occupies considerable time and requires a much better light

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[1] P. 14.

than is available in most Courts, and all the advantages arising from it may be better and more easily attained by the use of microphotographs.

## PHOTOGRAPHY.

Although the value of photography in criminal investigation is well known, and although photography is gradually being employed more and more, it is not yet made use of to anything like the full extent that is possible. In chemico-legal work photography is most important, and every expert should himself be a good photographer, not necessarily in order to take all the photographs required, but more particularly that he may realize the use and also the limitations of photography and so be in a position to know exactly what is required and how the best results may be obtained, and also that he may be able to do the work if at any time a professional photographer is not available. It is, however, generally uneconomical for the expert to undertake personally the photography needed for his work, and he should have access to a fully equipped photographic laboratory with all the technical assistance and apparatus necessary for taking any photographs required, including copies of documents and enlargements of all sorts.

There are few branches of chemico-legal work in which photography is not at some time helpful, but it is more particularly needed in connection with counterfeit coins, questioned documents, fibres, finger prints, robbery from letters and parcels, and stains and marks. These particular uses of photography will now be separately considered.

COUNTERFEIT COINS.—Photography is not as a rule necessary for any examination of counterfeit coins, but photographic enlargements are very helpful for attaching to the report or for exhibition in Court, as they allow similarities in various counterfeit coins or differences from genuine coins to be readily demonstrated. Considerable difficulty will be experienced in successfully photographing coins. This is due to the low relief of the design and the consequent absence of contrast in light and shade. There may also be difficulty arising from reflection from the surface of the coin, especially if the coin is new. The first difficulty may be overcome by arranging the coins in such a manner that they receive the light from one side and at an angle to the surface, so that the shadows formed increase the relief. In order to keep the coins in position while being photographed they should be fastened to a piece of cardboard by means of beeswax. Slow plates should be used, "process" plates or lantern slides being particularly good. For printing, "Bromide" or "Gas-light" paper is recommended.

A simple and effective method of photographing coins is mentioned by Guebhard.[1] This consists in very slightly fogging, by the briefest possible exposure, an ordinary photographic plate, placing this in a dish already containing sufficient developer to cover it, then putting the coins directly on to the sensitized surface of the plate and allowing development to proceed. After the lapse of a few minutes the plate is taken out and fixed in the usual way. All the operations, except of course the preliminary fogging, are done in a dark room.

The other difficulty mentioned may be surmounted by dulling the surface of the coins, which is done by heating them in a Bunsen flame and immediately plunging them into a 5 per cent. solution of sulphuric acid, if such a treatment is permissible on other grounds, or by covering them with a thin layer of magnesium oxide. This latter is accomplished by fastening the coins with gum or beeswax to a piece of cardboard, which is then held upside down, and underneath it a little magnesium ribbon is burned, when the oxide produced forms a thin uniform coating on the coins. If a number of similar coins are available, one or more may be treated in the manner first suggested, but if there is only one coin of any particular kind this should on no account be heated or treated with sulphuric acid, and in this case the second method described should be used.

Documents.—Whenever possible all questioned documents should be photographed before they are examined in order that a record may be kept of their exact condition when received, and as a rule such photographs should be the same size as the document. Photographs of documents,

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[1] La Nature, 1898, p. 128.

however, are useful or necessary for other purposes. Thus by means of a microscopical examination the expert may find definite evidence of alteration or erasure, but this can only be satisfactorily shown to others by means of enlarged photographs. Sometimes, however, the fact of alteration[1] or erasure or the presence of traces of obliterated writing are only visible, or are more clearly visible, in a photograph.

In case of an erasure where traces of original writing, which it is desired to decipher, still remain, it will be found that a photograph taken by transmitted light will sometimes show greater detail than one taken by reflected light, and a very simple and satisfactory method of taking the transmitted light photograph is by contact, employing a plate such as is used for lantern slides. If the document is small or one that can be folded without injury, the exposure is best made in an ordinary printing frame, the document taking the place of the usual negative and the photographic plate that of the printing paper. If the document is one that cannot be put in a frame, such for example as a register, the photographic plate is placed, with the sensitized face upwards, immediately beneath the writing it is desired to photograph, which is covered with a plate of clear glass to keep it flat. The exposure is made in artificial light and frequently several trials are necessary before the correct time of exposure is found. The negative gives a laterally reversed print if this is made by contact in the usual way, but by projection a correct print may be obtained. For purposes of examination, however, the negative is generally more satisfactory than the print, and if viewed from the film side, it shows the writing uninverted. Prints, however, are necessary for attaching to the report or for presentation in Court.

Frequently enlarged photographs are helpful in the comparison of handwriting, and similarities and differences can best be seen and studied in this way. In all such cases, however, it should never be forgotten that the writing under examination is an enlargement, otherwise too much stress may be laid upon what in reality are very small points.

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[1] For the examination of erasures on a document a stereoscopic binocular microscope is recommended.

In the identification or comparison of handwritings photographic enlargements of the characteristic letters or words upon which reliance is being placed to prove identity or non-identity are absolutely essential for demonstration purposes.

Sometimes the expert is given only a photograph of a document for examination. Photographs, however, may be misleading; for instance, they do not distinguish between ink and pencil writing. In one case in the author's experience only a photograph of a certain document was available when he was first consulted, and neither the fact that the body of the document was written with copying pencil nor that certain corrections had been made with black-lead pencil was apparent in the photograph, and this latter was an important point.

As already pointed out in the chapter on Documents, photographs are very easily "faked," and therefore care is necessary before accepting them as of any evidential value.

In the examination of forged banknotes and forged postage stamps, particularly the latter, photographic enlargements will be found very helpful, as also for the comparison of inked seal impressions on documents.

A very good yet simple method of carrying out a comparison of two or more seal impressions is to put the documents side by side in a photographic frame and, by means of an enlarging camera, to obtain enlarged images on a ground glass plate. Tracing cloth is then placed touching the glass plate and is held in position by a wooden framework or other device, and the enlarged images of the impressions are traced by hand with Indian ink directly on to the cloth. These tracings are then superimposed one on the other and examined by transmitted light. If the case is one likely to be brought into Court, the best method is to make photographic enlargements, and from the negatives to prepare positives on glass which can be produced in Court and directly compared by superimposing one on the other. Since seal impressions are frequently made in coloured ink, it is necessary in such cases to use special orthochromatic plates and light filters in order to obtain satisfactory photographs, and sometimes the best results are very poor or even useless.

Fibres.—The only useful photographs of fibres are microphotographs, and these are often helpful for attaching to a report or for demonstration purposes in Court.

Finger Prints.—Enlarged photographs are essential for the proper examination of finger prints.

Robbery from Letters and Parcels.—It is a useful precaution to photograph all envelopes and parcels immediately on receipt and before they are dealt with, since the subsequent examination frequently necessitates making some change in their appearance, such for example as opening them in a new place or to a greater extent than was the case originally.

Stains and Marks.—Photographs of stains and marks are most useful for attaching to a report or for exhibition in Court, as a much more accurate description can be conveyed by means of a photograph than is possible by means of a written account. Whenever possible therefore stains and marks should be photographed before they are dealt with. In the case of small marks enlarged photographs should be made.

**Radiography.**—Photography by means of X-rays is frequently very useful and, as already mentioned, it may be employed for determining the nature of the contents of closed packages, such as bombs, which it is not desirable to open, and there are also many other ways in which it can be of service in criminal investigation.

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## CHAPTER XV

# POISONS

THE whole science of poisons is termed Toxicology, but this includes the origin, properties, physiological action of and treatment for poisons as well as their detection. The chemist, however, deals only with the detection of poisons, and the chemico-legal expert limits himself to the detection of poison in cases of crime or suspected crime.

A knowledge of the methods employed for the detection and estimation of poisons forms part of the training of every analytical chemist, and the syllabus of the examination for the Associateship of the Institute of Chemistry includes "toxicological analysis" as also "the toxicological effects of chemicals and drugs and a knowledge of the dangers from poisoning by chemicals used in trade processes," and "a general knowledge of the Acts relating to . . . the Sale of Poisons."

Toxicological analysis, however, in legal cases is work for an expert, and should never be undertaken except by those having special experience, since the life or liberty of others depends upon the results. The rough qualitative testing sometimes attempted in cases of suspected poisoning by medical men, having little or no experience of chemical analysis, is most unwise, as at the best it uses up valuable material, while at the worst it may lead to a miscarriage of justice. Any qualitative testing for poisons should always be carried out with the same care and accuracy as in the case of quantitative work, and only by an experienced analyst. In this connection the trial of Mrs. Maybrick[1] should be read, as also Dr. Taylor's evidence in the Smethhurst case,[2] in both of which cases mistakes were made in

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[1] Notable English Trials: Mrs. Maybrick, pp. 104, 105, 106.

[2] Forensic Chemistry and Chemical Evidence. *W. Jago,* p. 150.

testing for arsenic. In the former, a medical man with no experience of analysis attempted Reinsch's test, and naturally the results were neither satisfactory nor conclusive. In the latter, Dr. Taylor, when giving evidence in the preliminary examination before the magistrate, stated that arsenic was present in the material tested, whereas in the final trial he stated that his assistant had made a mistake, and that the arsenic found had come from the copper used in the test.

A chemist undertaking toxicological analysis, in justice to his work and to himself, must insist upon being supplied with the fullest information possible about all the cases in which he is consulted, and especially with information that may serve as a guide to the particular poison or class of poison to look for, otherwise he may waste both time and valuable material in searching for something that from the symptoms and circumstances of the case is not likely to be present, and when he does obtain evidence of the nature of the poison, there may not be sufficient of the sample left for a quantitative determination or even for a conclusive test.

In India the Government regulations state that the chemist shall be supplied with the following information in all cases of suspected poisoning[1] :—

(1) The interval between taking the last food or drink and (*a*) the first appearance of symptoms of poisoning and (*b*) death (if this has occurred).
(2) The nature of the first symptoms.
(3) Whether any of the following symptoms were present, and if so, which :—
    (*a*) Vomiting and purging.
    (*b*) Deep sleep.
    (*c*) Tingling of the skin and throat.
    (*d*) Convulsions or twitchings of the muscles.
    (*e*) Delirium and clutching at imaginary objects.
(4) The nature of any other symptoms noticed.
(5) Whether any other person partook of the suspected food or drink, and whether they also suffered from symptoms of poisoning.

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[1] Lyon's Medical Jurisprudence for India, 1914, p. 780.

(6) Any other information available likely to prove serviceable as a guide to the class of poison administered.

If a medical man has been in attendance in any case of poisoning or suspected poisoning, the symptoms and history of the case will probably indicate, if not the particular poison used, at all events the class to which the poison belongs. If a medical man has not been called in until immediately before or immediately after death, a search should be made in the room for the remains of food and medicine, and for bottles, drinking glasses and hypodermic syringes, any of which may provide evidence of the nature of the poison.

In the Crippen case counsel for the defence tried to make capital out of the fact that the analyst knew that Crippen had bought hyoscine before "he tested for hyoscin and before he formed the opinion that he had found hyoscin," and referred to it as "unfortunate." This reflection upon the analyst was strongly condemned by counsel for the prosecution, and the judge when dealing with the point in his summing up said, "I do not think it is right to suggest that it is 'unfortunate' that they knew that the man had bought hyoscin," and "I do not think if you are satisfied with Dr. Willcox's evidence that you ought to come to the conclusion that he was influenced one single bit by what he knew."[1]

**Samples for Analysis.**—Samples of viscera will necessarily be taken by the medical man who conducts the post-mortem examination. Other samples, such as food or medicine, will generally be taken by the police or other investigating authority. A generous supply of viscera and dejecta should be sent for analysis, and in fatal cases should comprise (1) the entire stomach and its contents in one receptacle, (2) not less than one pound (half a kilo) of small intestines with their contents in another receptacle, (3) not less than one pound (half a kilo) of liver and a kidney in a third receptacle, (4) the bladder and any urine in a fourth receptacle, and (5) vomit, if any, in a fifth receptacle. In non-fatal cases, the vomit, stomach washings, the urine

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[1] Trial of H. H. Crippen, pp. 149, 160, 178.

passed during twenty-four hours and some fæces, each in a separate receptacle, should be sent for analysis. In every case strong spirit, either plain or methylated, should be added as a preservative, and a large sample of this should be sent to the analyst.

Formalin should never be used as a preservative, as in the case of alkaloid poisoning it is liable to cause decomposition even of such a relatively stable substance as strychnine, and in all cases it hardens the viscera and makes their extraction troublesome. The receptacles for the samples should be clean, wide-mouthed, white glass jars with glass stoppers, of about one litre capacity. These jars should not be filled more than two-thirds full, or they will probably burst owing to the pressure generated by the evolution of gases due to putrefaction. The stoppers of the jars should be greased with mineral oil or vaseline to prevent them sticking, and should be tied down with clean white cloth or strong paper, and sealed in such a manner that the jars cannot be opened without breaking the seals. Care should be taken that the seals are legible. The bottles should be carefully packed in a strong wooden box, and preferably each in a separate box.

Excellent rules for the guidance of those forwarding articles for analysis in suspected cases of poisoning are in use in India and may be found described in books dealing with Medical Jurisprudence in India.[1]

When a part only of an organ or a portion of the vomit, stomach contents or urine is sent for analysis, the chemist must be informed of the total weight of the organ or the total amount of fluid in order that in the event of poison being found the quantity present in the whole organ or fluid may be calculated. It is essential therefore for the medical man to weigh the organs and to measure all fluids.

**Methods of Examination.**—The actual testing for poisons presents no great difficulty, but considerable skill and experience are required in separating poisons from other substances with which they may be mixed and in isolating

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[1] Lyon's Medical Jurisprudence for India, 1914, pp. 776–786.
Outlines of Medical Jurisprudence for India. *P. Hehir and J. D. B. Gribble.* Madras, 1908, pp. 645–656.

them in the condition of purity required before the specific tests are applied.

The articles it is usually necessary to test for poisons in connection with crime or suspected crime are foods, liquors, medicines, vomit, urine and viscera. The methods employed for the actual testing are the same whatever the material examined may be and only vary with the kind of poison present, but the preparation previous to the testing differs with the nature of the material. It would be impracticable, however, to give all the possible variations in the methods of separating poisons from different materials, since they differ every time, and also quite unnecessary, as the fundamental principles are familiar to most analysts and may be found described in the ordinary text-books. It is equally unnecessary to give the tests for every known poison, since of the large number of poisonous substances known only a comparatively few are used for criminal purposes, and of those so used a large proportion are employed so rarely that they need not be considered in a general text-book. The methods and tests given, therefore, will be limited to those for the most usual poisons, and these will be described with particular reference to the examination of vomit, urine and viscera.

## Preliminary Examination.

(*a*) VOMIT, STOMACH CONTENTS AND STOMACH WASHINGS.—These should be examined for reaction to litmus paper, colour and odour, and the sediment should be carefully scrutinized with a lens in order to detect any white or yellow arsenic, match heads, vegetable fragments such as seeds, leaves and other portions of plants, opium or any poisonous material visibly present. Anything suspicious that is found should be separated, washed with water, then dipped into alcohol, allowed to dry and carefully examined.

(*b*) STOMACH AND INTESTINES.—These should be slit open, if not already opened, and spread out separately in white porcelain photographic dishes and carefully examined with a lens for general appearance, for remains of the last meal and for particles of poison.

## Detailed Examination.

The first necessity is to make sure that all the apparatus used is chemically clean and that the reagents are absolutely pure, and this can only be done by the chemist personally superintending the cleaning of the former and personally testing the purity of the latter.

Glass apparatus may be thoroughly cleaned in the following manner :—(*a*) First wash in a 10 per cent. solution of sodium hydroxide, (*b*) rinse with water, (*c*) treat with a warm mixture of potassium bichromate and sulphuric acid (made by dissolving 500 grams of commercial bichromate in 6 litres of water and slowly pouring this into a kilogram (850 c.c.) of commercial oil of vitriol) and (*d*) finally wash well with water.

The methods used for the examination depend upon the nature of the poison present. The various kinds of poison therefore will be considered.

Poisons may be classified in many ways; thus the medical man considers them with reference to their physiological action, while the analyst regards them according to the methods used for their extraction and isolation. Following the latter system, poisons may be divided into four main groups, namely :—

(1) Volatile substances separated by distillation. Examples : hydrocyanic acid, carbolic acid and phosphorus.

(2) Non-volatile substances separated by extraction with solvents. Examples : most alkaloids and many poisonous synthetic drugs.

(3) Metallic substances which remain when admixed organic matter has been destroyed by oxidation. Examples : arsenic, antimony, mercury, copper and lead.

(4) Substances requiring special methods of extraction. Examples : oxalic acid and oxalates.

Owing to the necessarily limited amount of material available, and to the fact that any poison present is usually in very small amount, the greatest economy must always be practised in dealing with the samples, and, unless the symptoms clearly indicate the particular poison or class of poison used, it is recommended that each sample should be divided into at least three portions, one for distillation, a second for extraction with solvents, and the third to be reserved for control or reference purposes or for use in the possible event of an accident happening to one of the others. In this division of the samples metallic poisons are not specially provided for, but they may be tested for either in the residue from the distillation or in the residue from the extraction by solvents. A special portion of the samples must always be reserved for the search for alkaloids, since these can only be tested for in the original material before it has been subjected to any treatment owing to the fact that most of the alkaloids, except strychnine, are very easily decomposed both by acids and alkalies, especially if heated.

The various methods of separating poisons will now be described in detail.

### Volatile Poisons.

The separation of volatile substances by distillation is performed in the following way :—A wide-mouthed, round-bottomed flask of about a litre capacity is fitted with a two-holed cork and inlet and outlet tubes, the former being connected with a steam generator and the latter with a long glass condenser. The end of the condenser is fitted with an adapter. The distillate is received in test tubes which are surrounded by crushed ice, and if hydrocyanic acid is being searched for the adapter dips into a small quantity of a 2 per cent. solution of potassium hydroxide.

About 100 grams of stomach contents, or of minced viscera mixed with water to the consistency of thin gruel, are placed in the flask and are acidified with a few cubic centimetres of a 10 per cent. solution of tartaric acid. The flask is now warmed and the steam supply connected up and steam slowly passed in. The distillate is collected in fractions of 8 to 10 c.c. at a time in numbered test tubes. Hydrocyanic acid, if present, distils over at once, and will be found almost entirely in the first fraction. The distillate is now tested for hydrocyanic acid and carbolic acid. If either of these substances is present, it may frequently be recognized by the characteristic smell both in the stomach contents and also in the distillate. Phosphorus is tested for separately, as described later.

**Hydrocyanic Acid (Prussic Acid).**—1. Add to a portion of the distillate which has been collected in potassium hydroxide two drops of a 2 per cent. freshly made solution of ferrous sulphate and allow to stand for ten minutes, then acidify with dilute hydrochloric acid and warm gently. If a cyanide is present, Prussian blue is formed. If only traces of cyanide are present, the blue coloration will only be faint. Blank tests with the reagents used must always be made.

2. A second portion of the distillate is placed on a watch-glass and a few drops of dilute sulphuric acid added. Invert over the watch-glass another similar glass moistened with yellow ammonium sulphide. Place in the water-oven for a few minutes. If a cyanide is present, gaseous hydrocyanic acid is liberated and forms ammonium thiocyanate with the ammonium sulphide on the upper watch-glass. Remove the upper glass, dry gently in the oven, add a few drops of water and a drop of ferric chloride solution. If a cyanide is present, a blood-red colour is produced.

**Carbolic Acid. (Phenol).**—Sometimes traces of carbolic acid are produced in putrefying viscera, and the presence of mere traces therefore is of no importance. Separate portions of the distillate are tested as follows :—

1. Add Millon's reagent (made by dissolving a little mercury in twice its weight of strong nitric acid (sp. gr. 1·4) and diluting with twice its volume of water) and warm. If carbolic acid is present, a red coloration is produced. This, however, is not specific, as other substances besides carbolic acid give the same coloration.

2. Add bromine water until a permanent brown colour is obtained. A white to yellowish-white precipitate or turbidity (tri-bromophenol) indicates carbolic acid.

3. Add a little dilute ammonia, then a few drops of a

solution of bleaching powder or of sodium hypochlorite and warm. If carbolic acid is present, a blue colour will be produced, which is turned red on the addition of acids.

4. Ferric chloride produces a purple colour with carbolic acid.

**Phosphorus.**—When employed for criminal purposes, phosphorus is generally obtained either from lucifer matches or from rat poison, in both of which it is present in the yellow or poisonous form. The best preliminary test for phosphorus is Scherer's test, which depends upon the fact that paper saturated with silver nitrate is blackened by the fumes from a solution containing phosphorus. The test is carried out as follows :—Place the suspected substance in a finely divided condition in a small flask, cover with water and add a few cubic centimetres of lead acetate solution in order to fix any sulphuretted hydrogen formed ; make very slightly acid with sulphuric acid. Close with a cork having a groove cut in one side and from the cork suspend two test papers, one being moistened with 5 per cent. silver nitrate solution and the other with a solution made by adding sodium hydroxide solution to 5 per cent. lead acetate solution until the precipitate that first forms is redissolved. The flask is gently warmed. If the silver nitrate paper does not darken, phosphorus is absent. If both papers darken, the test is inconclusive. If only the silver nitrate paper darkens, phosphorus may be present but its presence needs confirmation. The best confirmatory test is that proposed by Mitscherlich, which consists in the distillation of the suspected substance in the dark after acidifying with sulphuric acid, the vapour in the condenser being luminous in the presence of phosphorus.

## POISONS EXTRACTED WITH SOLVENTS.

This class of poisons includes (*a*) alkaloids, the principal of which are aconitine, cocaine, hyoscyamine, morphine and strychnine, and (*b*) poisonous synthetic drugs, the most important of which are acetanilide (antifebrin), antipyrine, phenacetin, sulphonal and veronal.

The solubilities of these substances in various liquids are as follows :—

| Poison. | Alcohol. | Benzol. | Chloroform. | Ether. | Petroleum Ether. | Water. |
|---|---|---|---|---|---|---|
| Aconitine .. | soluble | readily soluble | readily soluble | soluble | almost insoluble | almost insoluble |
| Cocaine .. | readily soluble | readily soluble | readily soluble | readily soluble | readily soluble | almost insoluble |
| Hyoscyamine | readily soluble | soluble | readily soluble | slightly soluble | slightly soluble | almost insoluble |
| Morphine .. | soluble | almost insoluble | almost insoluble | almost insoluble | almost insoluble | almost insoluble |
| Strychnine.. | soluble | soluble | readily soluble | almost insoluble | almost insoluble | almost insoluble |
| Acetanilide . | readily soluble | soluble | soluble | soluble | almost insoluble | slightly soluble |
| Antipyrine.. | readily soluble | almost insoluble | readily soluble | slightly soluble | almost insoluble | readily soluble |
| Phenacetin.. | soluble | slightly soluble | soluble | soluble | almost insoluble | almost insoluble |
| Sulphonal .. | soluble | soluble | soluble | slightly soluble | almost insoluble | almost insoluble |
| Veronal .. | readily soluble | slightly soluble | slightly soluble | soluble | slightly soluble | slightly soluble |

The principal solvents employed in chemico-legal practice are alcohol, chloroform, ether-ethyl acetate mixture and ether-chloroform mixture. The method adopted for the systematic separation of alkaloids and poisonous synthetic drugs, which is known as the Stas-Otto process, is briefly as follows :—

(*a*) Extraction of the viscera with alcohol, which dissolves the alkaloids and synthetic drugs, but which also extracts albumin, fat, colouring matter and other substances which would interfere with any subsequent tests, and from which therefore the solution must be purified.

(*b*) Separation of the alcohol, evaporating to dryness and extracting the residue with water faintly acidulated with sulphuric acid. The acid converts the alkaloids into acid salts, which are soluble in water. The water, however, only partly dissolves some of the synthetic drugs mentioned, several of them being only slightly soluble in cold water, and these therefore must be specially extracted from the residue.

(*c*) Purification of the acid water solution with various solvents. The solvents used are petroleum ether, ether, chloroform, and mixed ether and ethyl-acetate (or amyl alcohol). The ether and chloroform, in addition to taking out impurities, will also extract any poisonous synthetic drugs of acidic or weak basic nature present. These solvents are the very ones finally employed to extract the alkaloids, but used here they do not dissolve the acid salts of the alkaloids but merely separate various impurities, which if left in the solution would eventually be extracted with the alkaloids and would contaminate them.

(*d*) Making the water solution alkaline in order to liberate the alkaloids from their salts and extracting the alkaloids with chloroform and ether-ethyl acetate (or amyl alcohol).

(*e*) Purification and testing of the extracts.

The details of the process as carried out by Mr. Colles in Egypt, which vary somewhat with different workers, are as follows[1] :—

**Preparation of Material.**—Solids, such as viscera, are minced finely and a weighed portion taken, which should be as large as possible having regard to other tests and to the keeping of a reserve portion of the material for contingencies. Fluids like vomit are measured and almost neutralized with sodium bicarbonate solution, care being taken that they are never made alkaline but are always left faintly acid, after which they are evaporated to a syrupy consistency. The evaporation is carried out at a temperature not exceeding 45° C. and is expedited by

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[1] A modification is given by Hankin in Lyon's Medical Jurisprudence for India, 1914, p. 520.

blowing hot air on to the surface of the liquid either from a pressure pump or from an electric blower. This also acts as a corrective of any tendency to over-heating, and prevents the formation of a scum which would retard evaporation.

**First Extraction with Alcohol.**—Put the material into a large flask and add twice the weight of 95 per cent. alcohol made faintly acid with acetic acid, warm to about 45° C., cork the flask, shake well from time to time with occasional warming and finally allow to digest for twenty-four hours. Decant off the liquid through several layers of muslin placed in a large funnel, transfer the solid matter to the filter by means of a little alcohol, and free the residue from as much liquid as possible by squeezing it in the muslin. Digest both the solid matter and the muslin for a further twenty-four hours with fresh alcohol made faintly acid as before with acetic acid. Again decant off the liquid through fresh muslin and again squeeze the solid residue, and finally wash it well with alcohol. If the second extract is highly coloured, a third and similar extraction is made. The solid residue is reserved for the search for metallic poisons.

The alcoholic extracts, which are never clear, are mixed together, acidified faintly with acetic acid and warmed quickly on a water bath to 70° C. to coagulate as much albumin as possible. Cool under the tap and filter through muslin. The filtration is often very troublesome, and it may be necessary to allow the liquid to settle overnight in a tall cylinder and to decant off the following morning. The solid matter, after well washing with alcohol, which is separated either by filtration or decantation, is added to the previous residue and the washings are added to the main liquid.

**Evaporation of the First Alcoholic Extract.**—The mixed alcoholic extracts and washings are transferred to a large shallow porcelain basin, treated with sodium bicarbonate solution until only faintly acid, and are then evaporated at a temperature not exceeding 45° C., using either a stream of hot air blowing on to the surface of the liquid to hasten evaporation or a vacuum distillation apparatus. As the liquid concentrates it is necessary to watch the acidity carefully and to add a little sodium bicarbonate solution from time to time in order to keep the solution only just acid. Oil and fat will now separate out and are filtered off, washed with alcohol and rejected, the washings being added to the solution. The evaporation is continued until the liquid is syrupy.

**Second Extraction with Alcohol.**—The syrupy extract is covered with absolute alcohol and allowed to stand three minutes. The alcohol is poured off and the process repeated several times. In about fifteen minutes the surface changes to a lighter colour and becomes granular. The mass, which is still brown and sticky internally, is cut across in all directions with a spatula, stirring, however, being avoided, and repeatedly drenched with absolute alcohol, which is renewed every five minutes. All lumps must be broken up with the spatula so that the inner sticky portions are brought into contact with the alcohol. In about an hour the whole mass disintegrates into a granular powder. This is transferred to a glass or porcelain mortar and ground up with absolute alcohol until no more colour is extracted. The process must be carried out continuously, as if the granular powder is left exposed it absorbs water and reverts to the sticky condition. Throughout the whole operation the mixture must be faintly acid, a drop of dilute acetic acid being added to the alcohol if necessary. The residue is rejected.

**Evaporation of the Mixed Alcohol Extracts.**—The united absolute alcohol extracts are made nearly neutral and filtered. The residue is washed with a little absolute alcohol and rejected, and the washings are added to the filtrate, which is evaporated until all the alcohol is expelled and the liquid becomes syrupy.

**Extraction with Water.**—The syrupy mass is treated with a small quantity of water and well stirred. The mixture should be faintly acid. If an emulsion forms, more water is added and the mixture warmed to 40° C. After thorough digestion with the water, the mixture is filtered and the residue well washed with water, the washings being added to the main solution. The residue, which consists chiefly of fat, will also contain any substances originally present which are not very soluble in cold acidulated water, such for example as acetanilide, phenacetin, sulphonal and veronal. This residue therefore is reserved for further examination. The filtrate contains all alkaloids and other substances soluble in cold acidulated water.

### PRELIMINARY TESTS WITH WATER EXTRACT.

Before making any tests a general caution may be given, namely that as the amount of poison present is always very small, the tests made must be chosen with discrimination and carried out with the greatest possible care, as only a few tests can be applied and as a rule it is impossible to repeat a test.

At this stage a few preliminary tests are sometimes useful.

*Taste.*—A tingling and numbing sensation may indicate aconitine: a bitter taste with numbing may indicate cocaine: an intensely bitter taste may indicate strychnine.

*Physiological Tests.*—(*a*) A few drops made neutral and put into the eye of a cat may produce mydriasis, which indicates atropine, hyoscine or hyoscyamine.

(*b*) A quarter of a cubic centimetre of the extract is injected into the dorsal lymph sac of a frog to ascertain whether it is toxic and whether it produces convulsions, which latter may indicate strychnine.

(*c*) To a few drops of the solution in a small white dish or on a white porcelain tile add one drop of ferric chloride solution. A red coloration indicates meconic acid, which denotes opium.

**Opium.**—If opium is present in quantity, the ferric meconate reaction described above will be distinct. The coloration is distinguished from a somewhat similar coloration produced by acetates and thiocyanates by the following facts:—(*a*) That the colour of ferric acetate is destroyed at once by dilute hydrochloric acid, while ferric meconate is not decolorized until strongly acidified, and (*b*) that ferric thiocyanate is decolorized by mercuric chloride, while ferric meconate is not affected.

If the test is not distinct and opium is suspected, meconic acid, if present, is separated as follows:—To the whole of the remaining acid aqueous extract a 10 per cent. solution of basic lead acetate solution is added drop by drop until no further precipitate is produced. The mixture is warmed to 50° C. and allowed to settle. The precipitate is filtered off and washed once with water, the washings being added to the filtrate, which contains any alkaloids and synthetic drugs present, and which is reserved for further treatment. A hole is made in the apex of the filter paper and the residue washed into a test tube with as little water as possible. Sulphuretted hydrogen is then passed through the liquid to saturation and decomposes the lead meconate forming lead sulphide and liberating meconic acid. The lead sulphide is filtered off, washed and rejected and the filtrate evaporated on the water bath at a low temperature to a volume of about 1 or 2 c.c. A drop of dilute hydrochloric acid is added, followed by a drop of ferric chloride solution. Any red coloration formed is distinguished in the manner already described from the similar colour produced by acetates and thiocyanates. A positive result is conclusive of opium. A special test for Indian opium is described below; the tests for morphine will be given later.

### SPECIAL TEST FOR INDIAN OPIUM.[1]

The acid water extract, made by treating the residue from the absolute alcohol extraction with acidulated water, is made alkaline with ammonia, immediately shaken with ether, the ether separated and allowed to evaporate to dryness spontaneously. A portion of the residue is warmed with dilute hydrochloric acid, when a fine rose-red coloration indicates porphyroxin, the red colouring matter of Indian opium, which according to Dey is absent from Turkey and Smyrna opium.

**Purification of the Water Extract.**—If meconic acid has been tested for, this solution will contain lead acetate, to separate which it is treated with sulphuretted hydrogen, the lead sulphide filtered off and the filtrate freed from sulphuretted hydrogen by evaporating it to small bulk in a shallow dish at a temperature of 50° C., a faint acid reaction

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[1] Allen's Commercial Organic Analysis, Vol. VI. London, 1912, pp. 403, 410.

being maintained throughout. The final residue is diluted with water. This solution, or the original water solution if meconic acid has not been tested for, is acidified with a drop of dilute sulphuric acid, which converts any alkaloids present into sulphates, transferred to a small separating funnel and agitated by gentle rotation successively with the following solvents, which are afterwards separated in the usual manner:—(*a*) 20 c.c. of petroleum ether, (*b*) 20 c.c. of ether, (*c*) 15 c.c. of chloroform, (*d*) 15 c.c. of chloroform. The ether and chloroform extracts are mixed and washed by agitation with 5 c.c. of water made faintly acid with sulphuric acid, the washings being returned to the main water solution. The mixed extracts are then transferred to a small basin and allowed to evaporate to dryness by simple exposure without heat. The petroleum ether extract consists chiefly of fat, and need not be further considered. The residue from the mixed ether and chloroform extract will not contain any alkaloids, the acid salts of the alkaloids, unlike the alkaloids themselves, not being soluble in these solvents, but may contain acetanilide, antipyrine, phenacetin, sulphonal and veronal. The purification and testing of this extract will be described later.

The acid aqueous liquid after extraction with the solvents mentioned, and which still contains all the alkaloids, is warmed gently to drive off dissolved ether.

SEPARATION OF ALKALOIDS.—The method used for separating the alkaloids depends upon (*a*) whether morphine only is being looked for, (*b*) whether morphine together with other alkaloids may be present or (*c*) whether morphine may be excluded.

*Morphine only Present.*—If morphine is the only alkaloid that need be searched for, the acid liquid is transferred to a separating funnel and extracted with a mixture of 20 c.c. of equal volumes of ether and ethyl acetate. The solvent is separated and washed with 5 c.c. of water slightly acidified with sulphuric acid, the washings being added to the main solution. As ethyl acetate is frequently acid, it should be shaken before use with dilute sodium carbonate solution, which is separated and rejected. If ethyl acetate is not available, warm amyl alcohol may be used instead of the mixture of ether and ethyl acetate. This treatment does not dissolve morphine, the acid salt of which is insoluble in the solvents used, but removes various impurities which would interfere with the subsequent tests.

The acid liquid is now made alkaline with sodium bicarbonate, which liberates morphine from its acid salt, and is extracted with mixed ether and ethyl acetate or warm amyl alcohol, employed exactly as before, but the solvents now dissolve the alkaloid. At least three separate extractions should be made. The extract is purified and tested as described later.

*Morphine and other Alkaloids Present.*—The acid solution is treated with mixed ether and ethyl acetate, or amyl alcohol, as previously described in order to remove impurities. It is then warmed to 40° C. to drive off any ether present, filtered if necessary, placed in a separating funnel, 20 c.c. of chloroform added and a piece of litmus paper. Very dilute sodium hydroxide solution is now added drop by drop with gentle agitation until the litmus turns blue, when the funnel is corked and shaken. The alkali liberates the alkaloids from their acid salts and dissolves the morphine, which behaves differently to other alkaloids. The chloroform is separated and the extraction repeated twice with 10 c.c. more chloroform each time. The chloroform extracts, which contain all the alkaloids except morphine, are mixed and reserved for subsequent purification and testing as described later. The morphine remains in the solution and is extracted with mixed ether and ethyl acetate as already described and the extract purified and tested as given later. Some chemists use a mixture of equal parts of chloroform and ether in place of chloroform, but aconitine, hyoscyamine and strychnine are all more soluble in chloroform. Marden and Elliott show that three extractions each with 10 c.c. of chloroform will extract more than 99·9 per cent. of the aconitine from 50 c.c. of an ammoniacal aqueous solution, while six similar washings with ether only extract 99 per cent.[1]

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[1] A Study of the Methods for Extractions by Means of Immiscible Solvents from the Point of View of the Distribution Coefficients. *J. W. Marden and V. Elliott.* J. Ind. and Eng. Chem., 1914, pp. 315–320 and 928–934.

*Morphine Absent.*—The acid solution is made alkaline with sodium carbonate solution and extracted with chloroform as described above. The extract contains all the alkaloids, and is purified and tested as described later.

NOTE ON EXTRACTION WITH SOLVENTS.—It is a well-known fact that may be proved mathematically and demonstrated chemically, that if a precipitate on a filter paper is washed twice with a given quantity of water any soluble salts present are reduced in amount more than if the precipitate be washed once with double the quantity of water. In the same way when a liquid is being extracted with a solvent, the best results are obtained by using a larger number of extractions with a smaller volume of solvent each time rather than a small number of extractions with a large volume of solvent.[1]

**Purification of the Extracts.**—These extracts, which are too impure for the specific tests for the poisons to be applied, consist of (*a*) the ether-chloroform extract from the acidulated water, which may contain acetanilide, antipyrine, phenacetin, sulphonal and veronal; (*b*) the chloroform extract from the alkaline water solution containing any alkaloids except morphine, and (*c*) the ether-ethyl acetate or amyl alcohol extract containing morphine. These extracts are purified as follows:—

ETHER-CHLOROFORM EXTRACT FROM ACIDULATED WATER.—The various synthetic drugs mentioned, being only slightly soluble in cold acidulated water, were partly left behind in the residue from the absolute alcohol when this was treated with water. This residue should be extracted with hot water, filtered hot and extracted when cold (without further filtration if a turbidity appears) with chloroform. This chloroform extract is added to the ether-chloroform extract from the acidulated water and both are purified together. This purification is difficult, and may best be carried out by allowing the solution to evaporate to dryness spontaneously, separating the fat present by means of petro-

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[1] A Study of the Methods for Extractions by Means of Immiscible Solvents from the Point of View of the Distribution Coefficients. *J. W. Marden and V. Elliott.* J. Ind. and Eng. Chem., 1914, pp. 315–320 and 928–934.

leum ether and extracting the residue with hot alcohol which is evaporated in a small weighed basin. The residue after weighing is tested as described later.

Chloroform Extract.—This is placed in a small separating funnel and 10 c.c. of water acidified with a drop of sulphuric acid are added. The mixture is shaken, when the alkaloids combine with the acid forming salts which leave the chloroform and dissolve in the acid water. The impurities remain in the chloroform which is separated from the water, shaken with a further 10 c.c. of acidified water and again separated and finally rejected. The two lots of acid water are mixed and contain the alkaloids in a fairly pure state. If the solution is coloured brown, it may be further purified by shaking with petroleum ether, ether or ethyl acetate. The acid water solution is now transferred to a separating funnel with 10 c.c. of chloroform and a solution of sodium carbonate is added drop by drop until the liquid is distinctly alkaline. The sodium carbonate liberates the alkaloids from their salts, and on shaking the solution the alkaloids dissolve in the chloroform. The chloroform is run off and the alkaline liquid left in the separating funnel is shaken at least three times with 5 c.c. of chloroform each time, the various lots of chloroform being mixed. The chloroform is now filtered through a small filter paper moistened with chloroform in order to separate any small amount of water present, and the filter paper is washed with a few cubic centimetres of chloroform. The solution is now transferred to a small weighed glass basin and allowed to evaporate to dryness spontaneously when it is weighed. The residue in the basin appears as a brown ring of varnish-like matter on the side of the dish, while at the bottom is a brownish deposit or amorphous powder which is purer than the ring round the sides. Recrystallization from warm alcohol should be tried and the final residue weighed.

Ether-Ethyl Acetate Extract.—This is purified exactly in the same way as the chloroform extract except that ether-ethyl acetate mixture (or amyl alcohol) is used in place of chloroform and sodium bicarbonate instead of sodium carbonate.

## TESTS FOR ALKALOIDS.

**General Tests.**—The various alkaloidal precipitants are not specific, and a precipitate may be obtained even if no alkaloid is present, as many substances besides alkaloids give precipitates with the reagents. A positive result therefore needs confirmation. If no precipitate is obtained, alkaloids are absent. A very slight precipitate usually means a negative case unless some very sensitive and specific confirmatory test, such as the taste test for aconitine, is applicable.

As the quantities both of solution available and of reagents to be used for each test are very minute, consisting of a drop or two at the most, capillary pipettes or very fine glass rods must be employed for taking up the liquids, and the tests must be carried out in small watch-glasses. For the preliminary tests the material from the brown ring on the sides of the basin is used, the purer residue at the bottom of the basin being reserved for confirmatory tests. A little of the brown ring is removed, dissolved in a few drops of chloroform and drops of the solution placed on a number of small watch-glasses and allowed to dry, and the residue on each is then dissolved and tested as follows:—

(1) The best general test for alkaloids is with sodium phosphomolybdate (10 per cent. solution in 10 per cent. nitric acid). This produces a yellow amorphous precipitate which usually turns blue on standing. To apply the test the residue to be tested is dissolved in a drop of dilute nitric acid. Ammonium salts give a similar precipitate.

(2) The smallest possible drop of dilute sulphuric acid is added, and then iodine solution (1 gram iodine : 2 grams potassium iodide and 50 c.c. water). If alkaloids are present, a chocolate-coloured precipitate is produced.

(3) The smallest possible drop of dilute sulphuric acid is added and then a drop of Mayer's reagent (made by adding a solution of potassium iodide to one of mercuric chloride until the red precipitate first formed is just dissolved). If alkaloids are present, a white or yellowish precipitate is produced.

## Special Tests.

ACONITINE.—There is no good chemical test. The most satisfactory test is the physiological one, which consists in the characteristic tingling and numbing sensation produced when the smallest drop of a solution containing aconitine is applied to the tongue or lips. Veratrine also produces tingling, but in a lesser degree, and cocaine produces numbness, but the difference in the sensation produced by the three alkaloids can with practice readily be distinguished. In the Lamson case, Dr. T. Stevenson was asked about the difference between aconitine, veratrine and delphinine when tested on the tongue.[1] The toxicity test on a frog or small animal should also be made.

COCAINE.[2]—The best test is that suggested by E. H. Hankin,[3] which is as follows :—

Spread a drop of strong potassium permanganate solution on a microscope slide and allow it to dry. On the same slide near the permanganate film place a drop of saturated alum solution. Put into the alum solution a drop of the solution or a portion of the residue to be tested. With a cover slip draw the drop of alum solution up to the edge of the permanganate film and then allow the cover slip to fall so that the alum runs over the permanganate. If cocaine is present, characteristic crystals of permanganate of cocaine form. Antipyrine interferes with the test, and if present must be removed, which is done as follows:—Dissolve in water, add ammonia which precipitates the cocaine, filter and test the residue on the filter paper. Tropacocaine, alypin and scopolamine give somewhat similar crystals, which, however, with experience can be distinguished from one another and from the permanganate of cocaine. Cocaine also gives characteristic crystalline precipitates with gold chloride and picric acid, the tests being carried out under the microscope. Cocaine has a bitter taste, and produces numbness

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[1] Notable Trials Series : Dr. Lamson, pp. 95, 96.

[2] See the Detection and Estimation of Cocaine, Heroine and Veronal in Viscera. *P. A. Ellis Richards.* Analyst, 1919, p. 192.

[3] *Analyst*, 1911, pp. 2–6. Also Lyon's Medical Jurisprudence for India, 1914, p. 612.

when applied to the tongue. The toxicity test on a frog or small animal should also be made.

HYOSCYAMINE.—The use of hyoscyamine as a poison is of frequent occurrence in Egypt, and this arises from the fact that both *Hyoscyamus* and *Datura*, each of which contains hyoscyamine, grow wild. Of the latter there are several species, but only one variety of *Hyoscyamus*, namely *H. muticus*, is common. The seeds of *H. niger*, however, are also obtainable in Egypt, but these are imported from India. Many of the cases of poisoning by hyoscyamine originate in drugging for the purposes of robbery, which is carried out in Egypt much in the same manner as in India, the seeds of either *Hyoscyamus* or *Datura*, generally whole though sometimes crushed, being mixed with something which the victim is persuaded to eat and in which they are not very noticeable, such as rissoles or figs or the paste made from crushed dates, all of which form effective disguises for the poison.

Hyoscyamine is closely allied chemically to atropine and hyoscine (scopolamine), and therefore all three alkaloids will be dealt with together. F. H. Carr states[1] that the best procedure is as follows :—

1. To show that the substance is a mydriatic alkaloid. This may be done by applying one or two drops of a 1 : 10,000 solution to the eye of a cat and observing the dilation of the pupil.

2. To prove that the substance is a vegetable mydriatic alkaloid. This may be shown by (*a*) the strong alkaline reaction with phenolphthalein and (*b*) Vitali's test, which consists in adding a few drops of strong nitric acid, evaporating to dryness on the water bath, and adding an alcoholic solution of potassium hydroxide when a magnificent reddish-violet colour is produced.

3. To identify the alkaloid. If sufficient material is available, this may be done by preparing the gold chloride or the picrate and observing the crystalline form and melting point of these salts. These are shown in the following table :—

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[1] *The Chemical World*, 1912, pp. 3–5. Also *Carr and Reynolds*, Chem. Soc. Trans., 1912, pp. 101, 949. *King* (Chem. Soc. Trans., 1919, pp. 115, 478–9) gives different figures.

| | | Hyoscyamine. | Hyoscine. | Atropine. |
|---|---|---|---|---|
| Gold Chloride | Appearance : | Brilliant golden-yellow scales | Yellow leaflets | Dull pulverulent crystals |
| | Melting Point | 165° C. | 198° C.–200° C. | 137° C.–139° C. |
| Picrate | Appearance : | Rectangular plates | Slender matted needles | Rectangular plates |
| | Melting Point | 165° C.–166° C. | 180° C.–181° C. | 175° C.–176° C. |

MORPHINE.[1]—Until recently opium was easily obtained in Egypt, the poppy being cultivated in Upper Egypt, where its growth was allowed although the sale of Egyptian opium in the country was forbidden.[2] The growth of the opium poppy, however, was prohibited in October 1918.

Hankin and Chatterji[3] state that the only tests of any use for opium in the viscera are (*a*) the porphyroxin test, (*b*) the Husemann reaction, and (*c*) the urotropin or formaldehyde reaction. The porphyroxin test has already been described and is only applicable to Indian opium. The other two tests are as follows :—

*Husemann's Test.*—Dissolve a little of the suspected morphine residue in a drop of strong sulphuric acid, heat to 150° C. until white fumes are given off. Cool and add a drop of strong nitric acid. If morphine is present, a red-violet or blue coloration is produced which rapidly changes to dark blood-red. A better method is to allow the suspected residue, moistened with a drop of sulphuric acid, to stand in the desiccator for twenty-four hours before applying the nitric acid. A particle of potassium nitrate or potassium chlorate may be used in place of nitric acid.

*Formaldehyde Reaction* (*Marquis Test*).—To a minute portion of the suspected residue on a small watch-glass a drop of a mixture consisting of 3 c.c. of strong sulphuric acid and two drops of formalin (40 per cent. formaldehyde

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[1] See Opium Poisoning: Detection of Morphine in Acute and Chronic Cases. *J. Webster.* Analyst, 1917, pp. 226–228.

[2] 1,379 feddans (1 feddan = 1·038 acres) of poppy were grown in 1917.

[3] Note on Opium Poisoning. *E. H. Hankin and D. Chatterji.* Analyst, 1920, pp. 171–172.

solution) is added. A purple-red coloration changing to violet and finally to blue indicates morphine. The same colours are produced by codeine, dionine and heroine.

Other tests for morphine are as follows:—

(1) A drop of Fröhde's reagent (0·1 gram ammonium molybdate dissolved in 10 c.c. of cold strong sulphuric acid), which must be freshly prepared, gives with morphine a violet colour changing to blue and finally to dirty green.

(2) Strong nitric acid gives an orange-red colour changing to yellow.

(3) Pellagri's Test. Dissolve the solid morphine residue in two drops of strong hydrochloric acid to which a small drop of strong sulphuric acid has been added. Heat on the water bath for half an hour, a pink to brown colour being produced. Cool, dilute with water, and add a few drops of sodium bicarbonate solution until alkaline. The colour changes to red. Add a small drop of an alcoholic solution of iodine, when an emerald-green colour slowly develops. Excess of iodine spoils the test. If a little ether is poured on the surface of the liquid, the ether takes a reddish tinge, the water solution remaining green.

(4) Dissolve a little of the residue in dilute sulphuric acid (1 in 50). Add a drop of 6 per cent. iodic acid in water or 6 per cent. potassium iodate solution (free from iodide) in 2 per cent. sulphuric acid. A faint yellow colour due to liberated iodine is produced. A drop of the mixture placed on a little powdered starch gives a blue colour, and the colour of the remaining liquid when treated with a drop of very dilute ammonia deepens to brown.

STRYCHNINE.—The best test for strychnine is that with strong sulphuric acid and an oxidizing agent such as potassium bichromate or manganese dioxide. To carry out the test a portion of the suspected material is moistened with about two drops of strong sulphuric acid and a minute particle of potassium bichromate or better a little manganese dioxide is stirred in. A purple-blue colour changing to purple, crimson and finally to bright red is produced. The presence of strychnine should be confirmed by the physiological tests, namely the taste and the result of injection into the dorsal lymph sac of a frog. Strychnine is extremely bitter, and when injected into a frog produces convulsions.

## Poisonous Synthetic Drugs.

The most important of these compounds are acetanilide, antipyrine, phenacetin, sulphonal and veronal. The tests will now be described.

### Acetanilide (Antifebrin).

(1) Melting point 113° C. to 114° C.

(2) The distinguishing test for acetanilide is the formation of the characteristic and disagreeable odour of phenyl-isocyanide when heated with a few drops of alcoholic potassium hydroxide solution and a drop of chloroform. Heated with potassium hydroxide alone, the odour of aniline is given off.

(3) The following confirmatory tests should also be applied :—Boil a portion of the residue suspected to contain acetanilide with a few drops of strong hydrochloric acid for one minute, cool, dilute with ten to twenty drops of water and divide into three parts and test as follows :—

(*a*) Indophenol reaction : To one portion add carefully one to three drops of a solution of chlorinated lime (1 : 200) in such a manner that the solutions do not mix. A beautiful blue colour formed at the junction of the two liquids indicates acetanilide.[1]

(*b*) Mix another portion with a small drop of 3 per cent. chromic acid solution. Acetanilide gives a yellow-green solution, changing to dark green on standing five minutes, and with the formation of a dark-blue precipitate on addition of a drop of potassium hydroxide solution.[1]

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[1] J. Amer. Chem. Soc., 1905, p. 721.

(*c*) To the third portion add a few drops of 5 per cent. potassium nitrite solution, cool and make alkaline with sodium hydroxide and add a drop of an alcoholic solution of β naphthol. If acetanilide is present, a fine red azo dye is produced.

ANTIPYRINE.[1]

(1) Melting point 111° C. to 113° C.

(2) Steensma's reagent affords an excellent test. This reagent is prepared by dissolving one gram of paradimethylaminobenzaldehyde in 100 c.c. of a solution of 5 c.c. of 25 per cent. hydrochloric acid in 100 c.c. of absolute alcohol. When a liquid containing a trace of antipyrine is evaporated to dryness with 5 c.c. of this reagent, a beautiful fairly permanent rose-red colour is developed.

(3) Ferric chloride gives a blood-red coloration with antipyrine which is destroyed by mineral acids.

(4) A few drops of a solution of potassium or sodium nitrite followed by a drop of dilute sulphuric acid produces a green colour if antipyrine is present.

PHENACETIN.

(1) Melting point 135° C.

(2) Add a few drops of hydrochloric acid and boil for half a minute, dilute with ten times its volume of distilled water, cool, filter and add several drops of chromic acid solution; if phenacetin is present, a deep red colour is produced.

(3) Heat the suspected material to boiling with a few drops of 10 per cent. nitric acid. If phenacetin is present, a yellow to orange coloured solution is formed. Acetanilide gives a colourless solution when treated in the same way.

(4) Phenacetin gives the indophenol reaction as described under acetanilide.

(5) When phenacetin is heated with alcoholic potash and chloroform it does not give the smell of

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[1] Antipyrine in Toxicological Analysis. *G. D. Lander.* Analyst, 1913, pp. 97, 98.

phenyl-isocyanide which serves to distinguish it from acetanilide.

SULPHONAL.[1]

(1) Melting point 125° C.

(2) When sulphonal is heated with a little potassium cyanide in a dry tube, mercaptan is formed which is recognized by its disagreeable smell. If the residue is dissolved in water and acidified with hydrochloric acid, the solution yields a reddish colour with ferric chloride.

(3) That the material is a sulphone may be proved as follows :—Fused with sodium peroxide an orange-red mass results. A solution of a portion of this mass in water gives a purple colour with sodium nitroprusside : sulphur separates when the mass is treated with hydrochloric acid and the addition of barium chlorides gives a precipitate of barium sulphate. That the particular sulphone present is sulphonal and not trional or tetronal may be proved by a determination of the melting point.

VERONAL.[1]

(1) Melting point 191° C.

(2) If to a little fused potassium hydroxide veronal be added while the hydroxide is molten, ammonia gas is evolved and the residue, when dissolved in water, gives Prussian blue with a solution of ferrous sulphate, a purplish-violet colour with a solution of copper sulphate and carbon dioxide and a fatty odour with dilute sulphuric acid.

(3) Add two drops of nitric acid and test with Millon's reagent. A white gelatinous precipitate soluble in excess of the reagent is formed if veronal is present.

(4) Boil with a drop of 25 per cent. hydrochloric acid, make alkaline with sodium hydroxide and test with Nessler's solution. If veronal is present, a red precipitate is formed from the ammonium salt produced.

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[1] The Detection and Estimation of Cocaine, Heroine and Veronal in Viscera. *P. A. Ellis Richards.* Analyst, 1913, p. 192.

## METALLIC POISONS.

**Arsenic.**—This is one of the commonest forms of poison used. In England oxide of arsenic (white arsenic) is generally employed, but in the East (India and Egypt for example) not only the oxide but the yellow sulphide (orpiment) and occasionally the red sulphide (realgar) are employed.

The most important factor conditioning the nature of the particular substance used for poisoning purposes is probably the ease with which the substance in question may be obtained. Thus the extensive use of sulphide of arsenic as poison in Egypt is due to the fact that it can easily be obtained and can be bought clandestinely almost anywhere in the country despite the restrictions imposed on its sale by the Pharmacy Law, which results from its extensive employment for making the depilatory powder used for removing hair from certain parts of the body, a custom which Mohammedans consider essential to personal cleanliness. The powder consists of a mixture of sulphide of arsenic and slaked lime, which is made into a paste with water immediately before use and is applied in that condition. It is a frequent mistake in connection with these powders to regard the arsenic as the important and essential ingredient, whereas it is the calcium sulphide, formed by the interaction of the two constituents which is valuable, and other sulphides are equally effective, and depilatory powders containing sulphide of barium in place of sulphide of arsenic are made.

During the trial of Mrs. Maybrick, one of the witnesses (a hairdresser), who was evidently under the common and erroneous impression that it is the arsenic which is the active agent, stated that in the course of his business he made depilatory powders, using arsenic and lime, and that he generally employed yellow arsenic, though sometimes white arsenic. White arsenic, however, is quite useless for this purpose, and any depilatory effect produced by a mixture of white arsenic and lime is due entirely to the lime.

It may be mentioned that the very poisonous nature of artificial arsenic sulphides is due to the fact that, as a result of their method of manufacture from sulphur and arsenious oxide (white arsenic), they generally contain a large proportion of oxide. The naturally occurring sulphides are insoluble and therefore non-poisonous.

The best methods of testing for arsenic in cases of poisoning or suspected poisoning, where the amount of poison present is small, are Reinsch's test and Marsh's test, the former being used as a preliminary qualitative test and the latter either as a qualitative test or for a quantitative determination.

*Reinsch's Test.*—This test depends upon the fact that metallic copper in boiling dilute hydrochloric acid reduces soluble compounds of arsenic, antimony, mercury, bismuth and a few other substances to the metallic state, in which form they are deposited as a thin film on the copper. The test is very sensitive, and will readily detect as little as half a milligram of arsenic in 50 c.c. of liquid. Another good feature of the test is that it can be applied without the destruction of organic matter. Urine should be evaporated on the water bath to about one-fourth of its volume, vomit is mixed with water, and solid matter is cut or minced finely and sufficient water added to make the mixture the consistency of thin gruel. If the liquid is acid, it should be made slightly alkaline by the addition of sodium bicarbonate.

The hydrochloric acid employed must be absolutely free from arsenic. If arsenic-free acid is not available, it can be made by treating the purest acid obtainable with potassium bichromate and distilling.[1] The copper should also be free from arsenic, and such copper is readily obtainable. In one case on record, the presence of arsenic in the copper and chlorate in the solution led to a serious mistake being made, owing to part of the copper dissolving and the arsenic it contained being deposited on the rest of the foil.[2]

Reinsch's test should always be begun by a control experiment to test the purity of the materials used. For this purpose 50 c.c. of about 10 per cent. pure hydrochloric

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[1] Royal Commission on Arsenical Poisoning, Vol. II, 1903, p. 220.

[2] Reg. *v.* Smethurst, quoted in Circumstantial Evidence, *Wills* p. 368.

acid are placed in a conical flask and a thin strip of clean, bright copper foil about half an inch (12 mm.) by a quarter of an inch (6 mm.) in size is inserted and the solution kept gently simmering for about fifteen minutes. If at the end of this time the copper is still bright, the materials are pure and the test can be proceeded with, but if any signs of a grey deposit appear on the copper, the materials are not sufficiently pure and must not be used.

The copper foil may be suspended in the liquid by means of fine platinum wire or held in a clip made by bending the end of a glass rod twice round parallel to itself.[1]

It is essential that the copper used should be bright and clean, and in order to clean it fuming nitric acid is used, or the following mixture recommended by Hankin, namely 100 parts water, 100 parts strong sulphuric acid, 50 parts strong nitric acid and 2 parts strong hydrochloric acid.[2] In either case the foil is dipped momentarily into the acid, well washed in running water and used immediately.

When the reagents have been proved to be free from arsenic, about 100 c.c. of the material to be tested is added to the flask, which is kept simmering, the evaporated liquid being replaced from time to time by adding a little hot distilled water. If much arsenic is present, a film is formed on the copper within a few minutes, but if at the end of half an hour the copper remains bright, the material is free from arsenic. If a deposit is obtained on the copper, although this may be due to arsenic it is not necessarily a proof of its presence, since other substances, as already stated, notably antimony, mercury and bismuth, may also be deposited on the copper under the conditions of the test or the copper may become stained by organic matter or by sulphur compounds. Some indication of the possible nature of the film may be obtained from its colour, arsenic films varying from steel-grey to black, antimony films being generally purple, mercury films silvery to zinc grey and bismuth and sulphur films black. The presence of arsenic must therefore be confirmed. This is done as follows :—

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[1] Indian Toxicology. *F. N. Windsor.* Calcutta, 1906, p. 15.

[2] Lyon's Medical Jurisprudence for India, 1914, p. 485.

Remove the foil, rinse it with water, then with alcohol and ether, or simply with petroleum ether, dry it thoroughly either between filter paper or at a temperature not exceeding 100° C., cut it into strips and introduce them into a small (5 mm. diameter) dry tube, preferably a flat sublimation tube, and gently heat in a small flame, the upper portion of the tube being warmed before beginning the sublimation in order to obtain better crystals if arsenic is present, since arsenious oxide is liable to condense on a cold surface in the amorphous condition. Any sublimate obtained is examined under the microscope. If the material tested contained arsenic, the sublimate will consist of arsenious oxide and is in the form of well-marked octahedral crystals which are characteristic and absolute proof of the presence of arsenic. Unless satisfactory crystals are obtained, the presence of arsenic is not proved.

Sometimes on heating the copper in order to obtain sublimation, charring occurs and liquid is given off which obscures the sublimate. If this happens, another piece of copper having the deposit should be taken and placed in ether for twenty-four hours, after which it is dried with blotting paper and heated in the usual way (Hankin).[1]

With reference to Reinsch's test the Royal Commission on Arsenical Poisoning say[2]: "But we are now satisfied that the use of the Reinsch test entails considerable risk of missing quantities of arsenic which, though small, are not to be regarded as negligible."

*Marsh's Test.*—This consists in introducing the material to be tested into an apparatus evolving nascent hydrogen which reduces any arsenic or antimony compounds present, combining with the metal to form arseniuretted hydrogen (arsine) or antimoniuretted hydrogen (stibine) as the case may be. The apparatus consists essentially of a small flask fitted with a stoppered funnel, a tube filled with calcium chloride to dry the evolved gases and an exit tube drawn out to a point at the end and having one or more constrictions in the middle. This tube is made of hard glass or

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[1] Lyon's Medical Jurisprudence for India, 1914, p. 485.

[2] Part I, Final Report, 1903, p. 20.

transparent silica, and is strongly heated at the constricted sections, the arsine or stibine being decomposed and the metal deposited on the inner walls of the tube in the form of a dark-coloured metallic-looking film termed a "mirror."

In the Report of the Royal Commission on Arsenical Poisoning, three different forms of Marsh apparatus are described,[1] one, the ordinary form recommended by a Joint Committee of the Societies of Chemical Industry and Public Analysts,[2] the second, an electrolytic modification recommended by the Inland Revenue Departmental Committee,[3] and the third, a modification of the usual method by Dr. G. McGowan and Mr. R. S. Finlow in which the greater part of the hydrogen is evolved in a separate apparatus. The detailed descriptions of these different methods will not be transcribed, since the originals are so readily available to all chemists. With reference to the electrolytic method, Treadwell states[4] that when the platinum becomes dull, as it does with use, low results are obtained owing to the retention of arsenic. Hefti uses lead in place of platinum[4] for the cathode while Kunkel and Witthaus prefer silver.[5]

In a modification of the Marsh test recently described by B. S. Evans,[6] the arseniuretted hydrogen is passed over ignited copper, the gain in weight of which is a measure of the amount of arsenic present.

When using the Marsh test for arsenic determination, the following precautions must be observed, namely (*a*) that the reagents are free from arsenic, (*b*) that zinc when used must be "sensitive" and permit all the arsenic present to be evolved, and (*c*) that substances liable to retard or prevent the evolution of arsenic are absent. These possible sources of error will now be considered.

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[1] Vol. II, 1903, pp. 206, 208 and 221.

[2] See also *Analyst*, 1902, pp. 48–53, and *J. S. C. Ind.*, 1902, pp. 94–96.

[3] See also *J. Chem. Soc.*, 1903, pp. 974–986, and *Analyst*, 1903, pp. 349–359.

[4] Analytical Chemistry. *Treadwell-Hall.* Vol. II. New York, 1911, pp. 213–214.

[5] Manual of Toxicology. *R. A. Witthaus.* 1911, p. 607.

[6] *Analyst*, 1920, p. 8.

**Purity of the Reagents.**—This may be proved by making a blank experiment of an hour's duration after ensuring that conditions (*b*) and (*c*) are satisfactory.

*Hydrochloric Acid.*—This may be freed from arsenic as under :—

1. Take the "pure" acid as purchased for analysis. Dilute with distilled water to a specific gravity of 1·1 and add sufficient bromine to colour it strongly yellow (about 5 c.c. per litre) ; sulphurous acid, either gaseous or in aqueous solution, is then added *in excess*, and the mixture is allowed to stand for at least twelve hours. Or hydrobromic acid and sulphurous acid may be used. The acid is then boiled until about one-fifth has evaporated. The residue can then be used direct, the arsenic having volatilized with the first portion.[1]

2. Take the "pure" acid as purchased for analysis. Dilute with distilled water to a specific gravity of 1·1 and distil with potassium bichromate.[2]

With neither of these processes is the acid obtained always satisfactory, and it should invariably be tested before use.

*Sulphuric Acid.*—To about half a litre of the acid sold as "pure for analysis" add a few grams of pure sodium chloride and distil from a non-tubulated glass retort, rejecting the first 50 c.c. of the distillate.[1]

The purified acids should not be stored for any length of time, since most bottle glass is liable to communicate traces of arsenic.

*Calcium Chloride.*—This frequently contains arsenic and may be purified by moistening with hydrochloric acid, fusing and re-granulating.[1]

Dyer states[3] that much of the calcium chloride obtainable is strongly absorptive of arseniuretted hydrogen, especially if used for more than two experiments. The

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[1] Report of the Joint Committee of Societies of Chemical Industry and Public Analysts. Royal Commission on Arsenical Poisoning, Vol. II, 1903, p. 206. Also *J. S. C. Ind.*, 1920, p. 94, and *Analyst*, 1902, pp. 210–211.

[2] Report by Dr. G. McGowan and Mr. R. S. Finlow. Royal Commission on Arsenical Poisoning, Vol. II, 1903, p. 220.

[3] *Analyst*, 1920, p. 17.

calcium chloride therefore should be renewed after each positive test and also after every two negative tests.

*Sensitiveness of the Zinc.*[1]—Pure zinc is often insensitive and does not permit the evolution of all the arsenic in the solution. The best remedy is to coat the zinc with metallic cadmium before use. This is done as follows:—Twenty-five grams of pure granulated zinc are treated quickly with dilute nitric acid and then washed thoroughly with water and finally put into a flask containing one gram of cadmium sulphate or chloride dissolved in 50 c.c. of water. This is allowed to stand on the water bath with occasional shaking until a grey spongy deposit of cadmium completely covers the zinc. The liquid is poured off and the zinc washed twice with water by decantation. The sensitiveness of the zinc is controlled by making a test and seeing that a distinct arsenic mirror is obtained with one-hundredth of a milligram of arsenious oxide. If insensitive, the cadmium coating is increased, after which, if still unsatisfactory, the zinc must be rejected.

*Absence of Deleterious Substances.*—Among the substances which retard or entirely prevent the evolution of arseniuretted hydrogen are iron, platinum, copper, nickel and cobalt. The reagents therefore must be free from these substances. The presence of oxidizing agents such as potassium chlorate, chlorine, bromine, nitrates and nitric acid must also be avoided, as these would prevent the formation of arseniuretted hydrogen by inhibiting the reducing action of the hydrogen.

Marsh's test should always be begun by a control experiment to test the purity of the reagents and the satisfactory working of the apparatus. This experiment is allowed to run for twenty minutes, after which the solution to be tested is introduced in portions of 10 c.c. at a time, 20 c.c. being added altogether. In from five to twenty minutes, a mirror should begin to form if arsenic is present. If there is no formation of a mirror, a further 15 c.c. of solution is added, together with 10 c.c. of acid. If no mirror is formed after

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[1] The Estimation of Traces of Arsenic by the Marsh-Berzelius Method and the "Insensitiveness" of Zinc. *A. C. Chapman and H. D. Law.* Analyst, 1906, pp. 3–16.

half an hour, arsenic is absent. If the test is positive, the experiment should be allowed to continue for forty-five minutes.

*Standard Mirrors.*—When a satisfactory blank experiment has been obtained, a series of standard mirrors representing known quantities of arsenic must be prepared in order that, by comparison with the standards, the amount of arsenic in the sample tested may be estimated. The standard mirrors are made as follows[1]:—A hydrochloric acid solution of arsenious acid containing in each cubic centimetre 0·001 milligram of oxide is prepared by diluting a stronger solution with distilled water. Two c.c. of this solution (equal to 0·002 mg. of arsenious oxide) are introduced into the apparatus and, after twenty minutes, the portion of the tube containing the mirror is sealed off while still filled with hydrogen ; in contact with air, the mirrors gradually fade. Mirrors are now similarly made with 0·004, 0·006, 0·008, 0·01 mg. and so on, of arsenious oxide. With a little practice, it is easy to obtain the deposits of arsenic neatly and equally distributed. The standard mirrors, properly marked, are mounted on a white card. If sulphuric acid is used for the actual testing and not hydrochloric acid, then the standard mirrors must be made with sulphuric acid, as with small quantities of arsenic there is a difference in intensity between the mirrors made with the two acids. The Marsh test is so sensitive and detects such minute traces of arsenic that in viscera, vomit or urine in legal cases no mirror indicating an amount of arsenic less than one-fiftieth of a milligram should be considered positive, since less than this amount may be derived from sources other than poison administered criminally, such for instance as food, glass bottles and chemicals and apparatus, even though these latter are specially purified. In the case of hair and fingernails, however, amounts of less than one-fiftieth of a milligram of arsenic may be significant.

With reference to Marsh's test the Royal Commission on Arsenical Poisoning make the following statements[2] :—

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[1] Joint Committee's Report. Royal Commission on Arsenical Poisoning, Vol. II, p. 206.

[2] Part I, Final Report, 1903, pp. 16–18.

"The evidence shows that it is now recognized that a satisfactory estimation of the arsenic in a given substance can be made by comparing mirrors obtained by the Marsh-Berzelius method, after the substance examined has been subjected to appropriate treatment by which any arsenic present is obtained in a solution suitable for the proper application of the test.

"This method has lately been investigated in much greater detail than formerly, and certain new and important points have been worked out. It is, for instance, now recognized as essential, not only to obtain zinc and acid which are free from traces of arsenic and to verify this freedom by control experiments, but also to see that the zinc used is "sensitive," and will permit all the arsenic in the solution to be evolved as arseniuretted hydrogen. Another matter brought out by recent investigation is that the presence in the solution of certain metals, notably iron, is liable to retard, or entirely to prevent, the evolution of arseniuretted hydrogen, and thus may seriously affect the estimation.

"We are satisfied that careful analysts who estimate arsenic by this method can obtain results sufficiently exact and comparable for practical purposes, although the details of their procedures may differ.

"General experience of this test has shown that when in the preparation of 'standard' mirrors the quantities of arsenic added to the apparatus differ by amounts such as 0·002 milligram, a series of mirrors can be obtained showing differences in intensity which are sufficiently distinct and constant to be utilized for comparison. If the test is applied to a solution obtained from 10 grams of a given substance, a difference in mirrors corresponding to 0·002 milligram represents 2 parts of arsenic per million in the substance taken, or 0·0014 ($\frac{1}{720}$th) of a grain of arsenic per lb. If applied to 50 c.c. of a given liquid, a difference of 0·002 milligram in the mirror represents 0·04 parts of arsenic per million in the liquid, or 0·0028 ($\frac{1}{360}$th) of a grain of arsenic per gallon.

"Distinctions of greater or less delicacy can of course be made (within practicable limits) by increasing or diminishing the quantity of substance taken for the purposes of the test.

"As regards *detection* of arsenic by the Marsh-Berzelius method, the evidence shows that when various substances are taken in quantities which have been found practically convenient, the presence of arsenic will be detected when in amounts well below $\frac{1}{1000}$th grain per pound or (in the case of a liquid) well below $\frac{1}{300}$th grain per gallon." These figures represent 0·14 milligram per kilogram and 0·05 milligram per litre respectively.

The Commission also state that "A difference in reading corresponding to 0·002 milligram of arsenic too much or too little appears to be regarded as an outside limit" that is possible for errors due to differences in personal judgment and other factors.

It may be mentioned here that the Royal Commission's Report on Arsenical Poisoning contains much valuable information with respect to (*a*) the possible presence of arsenic in various foods, (*b*) the examination of enamelled iron cooking vessels for arsenic, (*c*) the accumulation of arsenic in human issues and the manner in which it is eliminated, and (*d*) the elimination of arsenic by the hair.

*Destruction of Organic Matter.*—Before using Marsh's test organic matter present must usually be destroyed. A discussion on the extent to which this is necessary and various methods employed will be found in the Report of the Royal Commission.[1] The details of the method applicable to viscera are as follows:—

Mince the material finely in a mincing machine, take 150 grams if available, or as much as possible up to this amount, place in a porcelain basin and add 70 c.c. hydrochloric acid (sp. gr. 1·1) mixed with three times its volume of water, or less in proportion if less material is used. Stir well and heat on a boiling water bath. Small quantities of pure potassium chlorate (about one-third of a gram at a time) are now added at intervals of a few minutes, the volume of the liquid being kept constant by the addition of water from time to time. The solid matter gradually goes into solution and the liquid becomes yellow. The addition of chlorate is continued until the yellow colour ceases

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[1] Vol. II, 1903, pp. 223, 224.

to change to brown, after which the liquid is further heated until it just begins to darken in colour, an indication that the chlorate has been decomposed. The liquid is now allowed to become quite cold, when a considerable quantity of solid matter separates out. The clear liquid is decanted off and filtered. The residue in the basin is washed two or three times by being warmed with water and a little hydrochloric acid. The liquid is cooled each time, filtered and added to the main bulk. It is important that the liquid should be quite cold before filtration, otherwise more solid matter will be deposited later and will require filtering off. The mixed filtrate and washings are returned to the basin and evaporated on a water bath to a volume of about 200 c.c., one or two further crystals of chlorate being added from time to time should the liquid tend to become dark coloured. The liquid is allowed to cool and 10 to 15 c.c. of a saturated aqueous solution of sulphurous acid are added in order to reduce the arsenic to the arsenious condition. The liquid is allowed to stand for some time and the sulphurous acid evaporated off, care being taken that the hydrochloric acid never becomes too concentrated, otherwise arsenic will be lost by evaporation as trichloride. The liquid is cooled and if necessary filtered. From this liquid the arsenic is precipitated by sulphuretted hydrogen, re-dissolved and then tested in the Marsh apparatus, or the liquid is diluted to a given volume and an aliquot part taken for direct testing. If the former method is decided upon, a slow current of sulphuretted hydrogen is passed through the liquid for an hour and the flask (which should be nearly full) is tightly corked and allowed to stand in a warm place until the next day, when more sulphuretted hydrogen is passed in and the flask again corked. If after two days more the liquid still smells of sulphuretted hydrogen, it is filtered through asbestos in a Gooch crucible, the precipitate being washed with a little sulphuretted hydrogen water. It is important that the volume of this wash water should not be more than about 20 c.c., otherwise the sulphide of arsenic is apt to assume the collodial form and to pass through the filter.

The asbestos containing the washed precipitate is then transferred to a small porcelain basin, the crucible being rinsed out with a little water. After warming the basin on a water bath to disintegrate the asbestos, 2·5 c.c. of aqueous ammonia (1 in 5) are added and the warming continued for a short time and the liquid filtered through a small filter paper, and this extraction with small quantities of ammonia and water is continued until both the asbestos and the solution are colourless. The filtrate is evaporated on a water bath to dryness or until all the ammonia is expelled, 2 c.c. of concentrated nitric acid are added and the liquid is digested on the water bath with subsequent small additions of nitric acid if necessary (about 1 c.c. at a time) until it is pale yellow. The free nitric acid is then evaporated off and the residue extracted with a few cubic centimetres of a warm aqueous solution of ammonium carbonate (1 in 4) in order to get rid of any sulphur present. The liquid is again filtered, evaporated on the water bath until free from ammonia, and the residue treated with nitric acid and evaporated to dryness. Then 8 to 10 drops of concentrated sulphuric acid are added and the basin heated until the contents are thoroughly charred. The sulphuric acid is then driven off, a Bunsen flame being used, but this operation must be performed very cautiously and no part of the basin must ever approach a red heat. If the smell of sulphurous acid can be detected in the escaping fumes, the heating must be stopped and the contents of the basin again treated with nitric acid as before. When all the sulphuric acid has been driven off the charred residue is extracted several times with small quantities of water acidulated with sulphuric acid and filtered. To the filtrate, which must be clear, colourless and without smell, a few drops of an aqueous solution of sulphurous acid are added, and after the excess of this acid has been removed by evaporation the liquid is ready for testing in the Marsh apparatus.

Another method of destroying organic matter which is particularly suitable for small amounts of material is as follows[1] :—

Ten grams of the substance are placed in a 3½-inch porce-

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[1] Royal Commission on Arsenical Poisoning, 1903, Vol. II, p. 207.

lain crucible and covered with pure re-distilled nitric acid (about 10 to 15 c.c.). The crucible is then heated on a sand bath until the evolution of brown fumes ceases. Three cubic centimetres of concentrated arsenic-free sulphuric acid are then added and the heating continued till the mass just begins to char, when a further 5 c.c. of nitric acid are added. The heating is now continued till all the acid is expelled, leaving in the crucible a black, nearly dry, charred mass. The crucible is then filled about half full with water and a few cubic centimetres of hydrochloric acid or of dilute sulphuric acid (according to which acid will be used in the Marsh apparatus) are added and the residue allowed to extract on a water bath for about half an hour, after which it is filtered into a porcelain basin, the charred mass washed with hot water and the filtrate and washings evaporated to about 30 c.c., which is allowed to cool, and is then ready to be tested in the Marsh apparatus.

**Antimony.**—Antimony may be detected both by Reinsch's test and Marsh's test. In the Reinsch test the film deposited on the copper is generally of a purple colour. The sublimate obtained by heating the copper, which consists of antimony trioxide and is amorphous in character, requires a higher temperature for its formation than that of arsenic. If the sublimate is moistened with a minute drop of ammonium sulphide or sodium sulphide introduced into the tube by means of a capillary pipette or a very thin glass rod, an orange coloration of antimony sulphide can be seen if the edges of the drop are examined under the microscope. When Marsh's test is employed it is conducted in exactly the same way as for arsenic. The antimony mirror formed in the heated tube differs from that of arsenic by being black and of a velvet or mat appearance, whereas the arsenic mirror is brown-black and glistening. If sulphuretted hydrogen, dried by means of strong sulphuric acid, is passed slowly through the tube and the deposit heated, an orange-red sulphide confirms the presence of antimony.

Antimony may also be deposited from solution electrolytically by means of zinc and platinum. For this purpose it is not necessary to destroy the organic matter. Strong hydrochloric acid is added to the material to be tested and a strip of platinum and a piece of zinc in contact with it are placed in the solution, or the acid solution is placed in a small platinum dish in which there is also a piece of zinc. If antimony is present, a black or brown-black deposit of metallic antimony will form on the platinum. The nature of this deposit may be confirmed by heating the foil in a sublimation tube and applying a drop of ammonium sulphide or sodium sulphide to the sublimate formed, when the orange-red sulphide of antimony will be produced.

When testing for antimony, if it is necessary to use rubber corks, these should on no account be red rubber, as this contains antimony compounds.

Antimony is best determined quantitatively by weighing the metal separated by electrolytic deposition.

**Mercury.**—After arsenic, probably mercury is one of the most common poisons used.

Mercury perchloride is frequently sold coloured with a red organic dye, and therefore any such coloration in the stomach or stomach washings may indicate the presence of mercury.

Mercury may be tested for by Reinsch's test in exactly the same way as arsenic and antimony. If mercury is suspected, after introducing the strips of copper foil into the sublimation tube the open end of the tube is drawn out into a capillary, care being taken to avoid heating the foil while doing this. The tube is now heated in the usual way, and if mercury is present the sublimate consists of minute globules of metallic mercury, which if examined with a lens cannot be mistaken. To confirm the presence of mercury, a tiny particle of iodine is dropped into the tube and is gently heated, when the iodine vapour passing over the mercury globules forms the brilliant red iodide of mercury.

Mercury is also deposited from solution electrolytically in the same manner as antimony. The nature of any deposit formed should be confirmed by sublimation with subsequent examination of the sublimate produced. A cathode of gold or heavily gilded platinum is used, and care must be taken when subliming the deposit or when cleaning the electrode not to heat it too strongly, or the gold will fuse.

Mr. W. M. Colles uses an electrolytic method of testing for traces of mercury which he finds so sensitive that minute amounts of mercury vapour present in the atmosphere of a laboratory where mercury is used are easily detected.[1]

A quantitative determination of the amount of mercury present may be made by weighing the cathode with and without the deposited mercury.

**Copper and Lead.**—The residue from the extraction of alkaloids may be used to test for general metallic compounds such as those of copper and lead. The organic matter present is burnt off and the ash extracted repeatedly with strong nitric acid, washed, filtered and the solution obtained tested in the usual way.

## POISONS REQUIRING SPECIAL METHODS OF EXTRACTION.

The most important are oxalic acid and oxalates, which are fairly common poisons in England though not in India or Egypt.

**Oxalic Acid and Oxalates.**—The method generally recommended for separating oxalic acid or soluble oxalates from vomit or the stomach contents is either by dialysis or by boiling with distilled water and filtering. In either case the resulting liquid is concentrated by evaporation and precipitated by acetate of lead, which is then decomposed by sulphuretted hydrogen, the sulphide of lead being filtered off and the filtrate evaporated to small bulk and tested in the usual way. Wynter Blyth states[2] that this process is "by no means to be recommended as generally applicable," and gives the method, of which the following is a summary, as being better:—Digest with hydrochloric acid until a fluid capable of being filtered is obtained, add ammonia in very slight excess, allow to settle, decant through a filter paper and to the filtrate add a slight excess of calcium acetate to precipitate the oxalic acid as calcium oxalate, filter, wash with acetic acid, wash into a beaker, dissolve by hydrochloric acid with the aid of heat, re-precipitate by

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[1] The Chemical Investigation of Medico-Legal Cases. *W. M. Colles.* The Practice of Medicine in the Tropics. Edited by *W. Byam.* London, 1921. (In the press.)

[2] Poisons: Their Effects and Detection, 1920, p. 550.

ammonia and allow to settle completely. Decant off the liquid, wash the precipitate by decantation and dry over the water bath. Test (*a*) by ignition, when a grey-coloured calcium carbonate is left if the substance is calcium oxalate, (*b*) by the reducing action on potassium permanganate in the presence of sulphuric acid, and (*c*) by warming with strong sulphuric acid and testing for carbon dioxide.

A better method, however, of separating oxalic acid or soluble oxalates from the stomach contents is to extract with hot alcohol acidified with hydrochloric acid, filter and evaporate to dryness. The residue is then dissolved in water and tested in the usual way. Oxalic acid, if present, is soluble in the alcohol, while soluble oxalates are decomposed by the hydrochloric acid and yield oxalic acid which is then dissolved.

The best tests for oxalic acid are as follows :—

(1) Neutralize the solution with ammonia and add a solution of calcium chloride or calcium sulphate. If oxalic acid is present, a white precipitate of calcium oxalate is formed which is almost insoluble in acetic acid but readily soluble in hydrochloric acid.

(2) Solutions containing oxalic acid give with silver nitrate a white precipitate of silver oxalate soluble both in nitric acid and in ammonia.

(3) Solutions containing oxalic acid reduce (decolorize) potassium permanagnate.

**Illustrative Cases.**—The best illustrative cases of poisoning are the well-known ones, descriptions of many of which are published in the Notable Trials Series. In only three cases, however, namely that of Dr. Pritchard, that of E. M. Chantrelle and that of Madeleine Smith, are copies of the chemical reports given. It would be most helpful if all editors of such trials gave a full copy of the chemical report, since the details of the chemical analysis are often very difficult to follow from the evidence. In several cases too there are manifest misprints and mistakes in the transcription of the chemical evidence; thus in the Lamson case, Unguentum Aconitia B.P. is stated to consist of "8 grains of aconitia to 1 grain of lard,"[1] and the residue from the ethereal extract in the Stas-Otto process is stated to have been "invisible"[2] in water. In the Maybrick trial ordinary glycerine is described in one place as "nitro-glycerine."[3] In the Seddons' trial the amount of arsenic in a mirror obtained in the Marsh apparatus is given in one place as being "one-three thousandth of a milligram" and a few lines below as "one-three hundredth,"[4] while in the Crippen case cresol is printed "creosol,"[5] carbolic acid "carbonic acid,"[5] and minced up "mixed up."[6]

The table on p. 220 summarizes the results of the chemical analysis in some of the more notorious poisoning cases.[7]

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  H. H. Crippen. Edited by *Filson Young.*

Notable Scottish Trials.
  Dr. Pritchard. Edited by *W. Roughead.*
  Madeleine Smith. Edited by *A. Duncan Smith.*

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[1] P. 90. [2] P. 95. [3] P. 132.
[4] P. 123. [5] P. 68. [6] P. 70.

[7] Notable Trials Series, except the case of Chapman, for which see Taylor's Principles and Practice of Medical Jurisprudence, 1920, pp. 366–380.

| Nature of Poison. | Amount Found. | Case. | Date. |
|---|---|---|---|
| Aconitine | .......................... | Dr. Lamson | 1882 |
| Antimony | 0·5 grain antimony. Strychnine also suspected but none found. | Dr. Palmer | 1856 |
| Antimony | Victim (*a*). More than 2·55 grains as metallic antimony: also more than 3 grains of mercury as metallic mercury.<br>Victim (*b*). More than 0·68 grain as metallic antimony. | Dr. Pritchard | 1865 |
| Antimony | Victim (*a*). 20·12 grains calculated as tartar emetic.<br>Victim (*b*). 29·12 grains calculated as tartar emetic.<br>Victim (*c*). 3·83 grains calculated as tartar emetic. | G. Chapman | 1903 |
| Arsenic | More than 87·9 grains as arsenic trioxide. | Madeleine Smith | 1857 |
| Arsenic | From 0·01 to 0·05 grains as arsenic trioxide in various organs. | Mrs. Maybrick | 1889 |
| Arsenic | From $\frac{1}{300}$ mgm. to $\frac{1}{80}$ mgm. as metallic arsenic in various organs: total arsenic calculated as trioxide in whole body, 2·01 grains. | The Seddons | 1912 |
| Hyoscine | $\frac{2}{7}$ grain. | H. H. Crippen | 1910 |
| Opium | No poison in body: opium on a sheet and bed gown. | E. M. Chantrelle | 1878 |

## Chapter XVI

# PRESERVATION OF THE HUMAN BODY AFTER DEATH

On first thought this subject might appear to be wholly medical and therefore beyond the range of forensic chemistry, but as will be shown it has an important chemical aspect and hence its consideration here is not out of place.

**Mummification.**—In Egypt the question of mummification is forced upon one's attention, and this method of preserving the dead was essentially a treatment by chemical reagents such as natron[1] and salt. Other preservative materials used included resins, gums, gum-resins, beeswax and wood pitch.

Many ancient writers state that mineral pitch (bitumen of Judea) was employed by the Egyptians for embalming purposes and most modern writers on the subject of mummies make the same statement. Apparently the black material found in mummies has been called pitch or bitumen chiefly on account of its colour and general superficial resemblance to pitch, coupled in some few cases with its behaviour on burning, but no record of any systematic examination or chemical analysis can be traced. The author, however, after extensive research and numerous analyses, has not been able to find a single instance of the use of mineral pitch in mummies, the greater proportion of what is ordinarily called pitch being resin, gum-resin or gum blackened, like so many organic materials, by the changes brought about by age and exposure. In some cases the material is wood pitch. It is not denied that

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[1] A compound of sodium carbonate and sodium bicarbonate mixed with sodium chloride and sodium sulphate in varying proportions, and which occurs naturally in Egypt.

mineral pitch may have been used for mummification, but so far no instance of its use has been proved.

In order to demonstrate the value of natron and salt as preservatives the author soaked two fowls (plucked and with the internal organs removed) for seventy days (the period mentioned by Herodotus) in solutions of different qualities of natron, each 8 per cent. strength, and a third fowl in an 8 per cent. solution of common salt, and afterwards rapidly rinsed them with water and then allowed them to dry. This was done in 1908, and after thirteen years all three fowls still exist and are in much the same condition as when taken out of the solutions.

The success of the old Egyptian method of treatment is shown by the large number of mummies in an excellent state of preservation which still exist, the oldest known example being a mummy of the Fifth Dynasty (about 2700 B.C.) now in the museum of the College of Surgeons in London.

**Desiccation.**—In addition to mummies, however, there are numerous examples of well-preserved bodies, some dating from Predynastic times, that is before 3300 B.C., but these are merely desiccated by natural means and are not mummified in any way, and owe their preservation to burial without coffins in shallow graves in hot, dry and porous sand, thus allowing heat to gain access and the moisture from the body to escape, the almost complete absence of rain and subsoil moisture aiding desiccation.

**Hair.**—In this connection the following note on human hair may be of interest, since the colour of the hair of an exhumed body may possibly be an important factor in the identification of the person buried. Hair itself is very resistant to putrefaction, but it is generally stated that the colour is not so resistant. This, however, requires qualification, and it will be shown that under certain conditions the colour of human hair may remain unchanged for several thousands of years. It is also sometimes stated that all hair eventually becomes red, and the author has a letter from a well-known chemist in which this statement is made in connection with an exhumed body in which such a colour change is said to have taken place due to the oxidation of the iron compounds in the hair. Wilkinson would seem to support this view, for he writes[1]: "Many of the mummies of women have been found with the hair perfectly preserved, plaited in the manner I have mentioned; the only alteration in its appearance being the change of its black hue, which became reddened by exposure to great heat during the process of embalming."[2]

As there seemed to be some uncertainty about the matter, it was thought that it might be useful to collect as many definite facts as possible concerning the colour of the hair of mummies and other preserved bodies, and the following have been noted.

During the Archæological Survey of Nubia a very large number of bodies were carefully examined and described by Professor G. Elliot Smith and Dr. F. Wood Jones.[3] These bodies belonged to all periods varying from Predynastic to Early Christian. In the published report reference is made to the hair in 128 different cases, and the following tabular statement shows the results :—

HAIR FROM NUBIAN GRAVES.

| | |
|---|---|
| Colour not stated | 43 |
| Black | 46 |
| Brown | 8 |
| Dark | 1 |
| White | 16 |
| Black with white | 3 |
| Brown with white | 3 |
| Dark with white | 2 |
| Grey | 6 |
| Total | 128 |

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[1] The Ancient Egyptians. *Sir J. Gardner Wilkinson, F.R.S.* London, 1890. Vol. II, p. 335.

[2] There is no proof that the body was subjected to great heat during the process of embalming. Doubtless the resinous material employed as a preservative, especially that introduced into the skull, was applied in a molten or semi-molten condition, but this does not require great heat.

[3] The Archæological Survey of Nubia. Report for 1907–8, Vol. II. Report on the Human Remains by *Prof. G. Elliot Smith, F.R.S., and Dr. F. Wood Jones.* Cairo, 1910.

In eight of the cases in which the colour is not given the hair is described as being "typically negro," in two other cases as "typically negroid," in one case "woolly negroid," and in one case "woolly." None of these twelve cases therefore are likely to have been red. In seven of the cases of white hair the person is specifically called "old": in one instance, however, the person was apparently young, since a suggestion is made that possibly the white colour of the hair was due to post-mortem bleaching caused by the salt used in embalming. In two of the cases of brown hair the description given is "now brown," as though it was desired to suggest that brown had not been the original colour. In no single case is the hair described as red or even reddish.

In the catalogue of royal mummies in the Cairo Museum, Professor Elliot Smith describes fifty mummies.[1] The results so far as the hair is concerned are as follows:—

### HAIR OF ROYAL MUMMIES.

| | |
|---|---|
| Colour not stated | 26 |
| Black | 1 |
| Dark brown | 4 |
| Brown | 2 |
| Dark reddish-brown | 1 |
| Reddish-brown | 2 |
| Yellow | 1 |
| Black with grey | 1 |
| Dark brown with grey | 1 |
| Grey | 2 |
| Brown with white | 1 |
| White | 7 |
| *Stained* a brilliant reddish colour | 1[2] |
| Total | 50 |

In addition to the above there were three wigs, all of which were brown.

Two specimens of hair from mummies were examined by the author for the late Sir M. Armand Ruffer, one of

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[1] Catalogue Général des Antiquités Egyptiennes du Musée du Caire. The Royal Mummies. *G. Elliot Smith, F.R.S.* Le Caire, 1912.

[2] Interplaited with strands of hair of a black colour.

which was of a very light brown colour, and the other black. The former belonged to the Twentieth Dynasty and the second could not be dated. The light brown hair was matted together with a paste that proved on analysis to be a mixture of natron and soap,[1] but whether the natron had affected the colour of the hair in any way cannot be stated, though this seems possible.

The hair of all the human and animal mummies, as well as of all the wigs, found exposed in the Cairo Museum,[2] was also examined. The results were as follows :—

### Hair of Human Mummies.

| | |
|---|---|
| Black, curly | 1 |
| Brown | 2 |
| Light brown | 1 |
| Dark | 1 |
| Flaxen | 2 |
| Impossible to determine whether any hair at all without closer inspection : certainly no red hair | 6 |
| Total | 13 |

### Hair of Ape Mummies.

| | |
|---|---|
| Dark | 2 |
| Dark brown | 1 |
| Brown | 3 |
| Total | 6 |

### Wigs.[3]

| | |
|---|---|
| Black | 2 |
| Very dark brown | 4 |
| Brown | 4 |
| Total | 10 |

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[1] The soap was probably a product of the action of the natron on fatty material with which it had been mixed.

[2] In 1918.

[3] Inside one of the wigs there was some fibre which resembled coco-nut fibre in appearance, and the colour of this had a slight suggestion of red.

The Alexandria Museum was also visited, but there were only four mummies of which the hair could be seen and this was brown in all cases.[1]

In view of the foregoing facts, therefore, there can be no doubt whatever that under Egyptian conditions of burial the hair certainly does not always become red even after several thousands of years, and it seems very doubtful whether it ever undergoes such a change ; in the one case where the hair is red Professor Elliot Smith specially states that it is stained, and suggests henna, which is still used for this purpose in Egypt, as the dye probably employed.

**Effect of Lime on the Body.**—Not infrequently in murder cases in which the body is buried lime is employed, either in the hope of hastening decomposition or of preventing the offensive products of putrefaction from manifesting themselves. Thus in the Crippen case lime was found with the remains and two of the medical experts were asked at the trial what the effects of this lime would be.[2]

Before discussing this subject it should be stated that the word " lime " as ordinarily used may mean either quicklime or slaked lime or even chlorinated lime (bleaching powder), frequently called " chloride of lime." Quicklime is calcium oxide produced by strongly heating limestone ; as ordinarily made for building purposes it is in the form of large irregular lumps and is very impure. Slaked lime is calcium hydroxide, a dry white powder obtained from quicklime either by exposing this latter to a moist atmosphere or by the addition of water. In both cases heat is produced, but in the former case the reaction proceeds so slowly that the effect is small, the heat being dissipated almost as quickly as it is formed, whereas when water is added directly to quicklime a large amount of heat is evolved, which may be sufficient to ignite combustible bodies in contact with the lime. Thus even wood may be ignited in this way. In 1894 seventeen fires in London were reported to be due to the slaking of quicklime in contact with combustible bodies, eight of these being caused by

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[1] In 1920.

[2] Trial of H. H. Crippen, pp. 58, 59, 65.

rain.[1] Slaked lime necessarily contains the impurities present in the original quicklime from which it is made and also a certain amount of additional carbonate due to the absorption of carbon dioxide from the atmosphere. Chlorinated lime is a product of the action of chlorine gas upon slaked lime and is extensively employed as a disinfectant. It soon loses its efficiency if exposed to the air. The name "chloride of lime" for this substance, although in common use, is not only ambiguous but incorrect, dry chlorinated lime consisting chiefly of a substance intermediate between calcium chloride and calcium hypochlorite.

No record can be found of any observations or experiments showing the effect of lime upon the dead human body. From purely theoretical considerations, however, it may be stated that neither slaked lime nor chlorinated lime has any corrosive action upon flesh, but that on the contrary both these substances, especially chlorinated lime, would tend to exert a preservative action, owing to their germicidal properties, and if placed round a dead body would prevent, or partly prevent, the attacks of micro-organisms and insects from without. They would also act as deodorants, the lime by absorbing some of the offensive gases of putrefaction and the chlorinated lime by decomposing them. Neither lime nor chlorinated lime outside a body, however, could arrest the decomposition taking place from within, and it is this which is one of the main factors in the putrefaction of the body. Slaked lime, which is used in the form of milk of lime in the de-hairing of hides, might cause the hair to fall off.

When giving evidence in the Crippen case Dr. Pepper stated[2] that "absolute quicklime" destroys the body by abstracting moisture and that given a sufficient quantity of quicklime the whole body would be destroyed. In this I believe him to have been mistaken. If a dead body be surrounded by a large amount of quicklime and if this be suddenly slaked, as was done in the Mannings' case,[3] a

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[1] The Handling of Dangerous Goods. *H. J. Phillips.* London, 1896, p. 262.

[2] Trial of H. H. Crippen, p. 58.

[3] Taylor's Principles and Practice of Medical Jurisprudence, 1920, Vol. I, p. 294.

small amount of superficial burning might result and a certain amount of drying of the body, caused by the heat generated, would take place, the extent of both of which would depend largely upon the amount of quicklime present and upon its quality, and also upon whether there were any clothes or wrappings on the body. The body, however, would not be destroyed, or even partially destroyed, since it contains so large a proportion of water as to make destruction extremely difficult. The drying, in fact, by delaying putrefaction would conduce to preservation. If on the other hand only gradual slaking of the lime took place by the abstraction of water from the body, air or the surrounding soil, and this is the condition Dr. Pepper assumes, then there would simply be a very partial desiccation of the body with the consequent slightly delayed putrefaction. The lime in both cases would tend to preserve the body from destructive agencies from without.

In order to obtain positive evidence of the nature of the action on the body of the different kinds of lime the following-mentioned experiments were made. Young pigeons were killed and plucked but not opened, nothing being removed but the feathers, and were then treated separately as follows :—

A. Buried in dry earth.
B. Buried in freshly slaked and sifted lime.
C. Buried in fresh chlorinated lime.
D. Buried in quicklime in the form of small lumps.
E. Buried in quicklime in small lumps which was suddenly slaked by the addition of water.

In all cases the burials took place in boxes which were provided with loosely fitting covers and which were placed on the laboratory roof in Cairo in July. After twenty-four hours, specimen E was taken out and examined. The lime was in the condition of a dry white powder and the pigeon was somewhat discoloured, considerably drier and firmer than it was originally, but no blistering or rupture of the skin or other damage caused by the heat except the discoloration mentioned could be found. The pigeon was re-buried in the same slaked lime. After three days pigeon C was found to be partly uncovered owing to the considerable swelling caused by putrefaction which had taken place, there was a most objectionable smell and the chlorinated lime had become wet and pasty. The pigeon was re-buried in the same wet lime. After five days a number of large maggots and several small beetles were visible in box A and the smell was very strong. After fifteen days all the pigeons were disinterred and examined, when the following results were found :—

A. Active decomposition taking place : strong smell.
B. No visible decomposition : some smell : still soft.
C. Decomposition proceeding : smell very objectionable : chlorinated lime pasty.
D. Lime all slaked : pigeon dry and intact (not quite so dry as E) : no burning or rupture of the skin.
E. Dry and firm : slight discoloration but no rupture of the skin.

The pigeons were re-buried and left for a further period of five and a half months, making six months in all, when they were re-examined, after which the experiments were discontinued. The results were as follows :—

A. In very bad condition : considerable smell of putrefaction : large part of flesh had disappeared.
B. In good condition : very slight smell : shrunken : flesh still soft and skin pliable : skin unbroken.
C. In active state of decomposition : swollen : considerable smell of putrefaction.
D. In good condition : dry and hard : skin unbroken : shrunken.
E. In good condition except the feet, both of which had come off : dry, hard and brittle : skin unbroken : shrunken.

These results bear out the statements already made, namely first, that lime is a preservative, and secondly that the act of slaking lime in contact with a dead body, whether the slaking is brought about gradually or done all at once, does not destroy the body.

**Adipocere.**—Adipocere is a waxy-looking substance having a greasy feel, which is frequently formed when a body is buried in damp soil or is submerged in water. A few statements from well-known books on Medical Jurisprudence with respect to the formation of adipocere may be quoted.

Taylor writes[1]: "The whole of the soft parts may have become converted into this white substance"; "The white substance into which all the organs had been completely transformed . . ."; "Any part of the human body may undergo this change . . ."

Professor Dixon Mann writes[2]: "It is probable, however, that the fatty acids concerned in the formation of adipocere may be partly derived from protein as well as from the fatty tissues actually present at the time of death; the appearances found in many advanced cases in which the soft structures of the body have been almost entirely converted into adipocere are inconsistent with the view that the acids are solely derived from the pre-formed fat."

Professor Glaister writes[3]: "Dr. Guy has seen whole bodies transformed completely . . ."; " . . . we observed whole limbs, chiefly the lower, the chest and shoulders and in several cases the buttocks so changed and in more than one case the whole body."

Dr. Aitchison Robertson writes[4]: "This is a change which the fat as well as the other tissues of the body may undergo when exposed to very damp conditions"; "The formation of adipocere may be noticed in the dissecting-room. Bones . . . are often placed in a sink and covered with water for several weeks: during this time the fragments of tendon and ligament attached to the bones become soft and greasy or in other words have become changed into adipocere"; "The subcutaneous fat undergoes this

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[1] Principles and Practice of Medical Jurisprudence, 1920, Vol. I, pp. 298, 307.

[2] Forensic Medicine and Toxicology, 1908, p. 63.

[3] A Text Book of Medical Jurisprudence and Toxicology, 1915, pp. 155, 156.

[4] Manual of Medical Jurisprudence, Toxicology and Public Health, 1913, p. 70.

change first of all, then the skin follows and last of all the muscle."

These statements are very definite and leave no room for doubt that the writers believe that the whole of the soft parts of the body, under favourable conditions, may be converted into adipocere. This view, however, has been challenged by other medical men: thus, commenting on some descriptions by Dr. Coull Mackenzie of the formation of adipocere in India, Dr. G. H. F. Nuttall states[1] that "internal organs are not converted into adipocere," and suggests that Dr. Mackenzie was mistaken and quotes a previous writer to the effect "that muscular tissue which has macerated and putrefied in water presents an appearance sufficiently similar to deceive the unpractised eye."

Lieutenant-Colonel Waddell states[2] that "Well authenticated cases supported by an authoritative chemical analysis are still required to settle this question for India."

Professor Abderhalden writes[3]: "By carefully following this process, and especially by direct experiments, it has been proved that there is no such conversion of albumin into fat, but that the fat already present in the body is responsible for the production of adipocere. This is mainly due to the fat present already in a given locality and to such infiltrated fatty masses as may be deposited there by the water."

Dr. Leonard Hill writes[4]: "It was supposed to result from the actual change of muscle proteid into fat. What happens, in truth, is the putrefactive colliquation of the proteids, and the fat set free from its depôts floats in the water and percolates through the tissues."

Since differences of opinion on a subject frequently arise from an absence of exact definition of the terms used,

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[1] Taylor's Principle and Practice of Medical Jurisprudence, 1910, Vol. I, pp. 311, 312.

[2] Lyon's Medical Jurisprudence for India, 1914, p. 87.

[3] Text Book of Physiological Chemistry. Trans. New York, 1908, p. 326.

[4] Recent Advances in Physiology and Bio-Chemistry. London, 1908, p. 293.

it would be well to have a more precise definition of adipocere than the usual one of "a waxy-looking unctuous substance." It is manifest at the outset that no exact definition of adipocere and no certain identification of a substance as adipocere can be given without chemical analysis. The only analyses of adipocere that can be traced are as follows :—

(*a*) An early analysis by Chevreul.[1]
Result: An ammoniacal soap with colouring matter.

(*b*) Analysis of three specimens by Schmelck in 1902.[2]
Result:

| | % |
|---|---|
| Insoluble fatty acids .. .. .. | 83 to 84 |
| Unsaponifiable matter .. .. .. | 16·7 |
| Ash (containing 83 to 84 per cent. lime) .. | 1·6 to 1·8 |

(*c*) An analysis by L. van Itallie and A. J. Steenhauer in 1917[3] of a museum specimen of material from the same source as the sample examined by Chevreul.
Result: Essentially a mixture of fatty acids (probably palmitic and stearic) with calcium soaps.

(*d*) A recent analysis by R. F. Ruttan and M. J. Marshall of pig adipocere.[4]
Result:

| | % |
|---|---|
| Palmitic acid .. .. .. .. .. | 67·5 |
| Stearic acid .. .. .. .. .. | 3·3 |
| Oleic acid .. .. .. .. .. | 5·2 |
| Hydroxy-stearic acids .. .. .. .. | 15·8 |
| Calcium soap .. .. .. .. .. | 4·4 |
| Fat and unsaponifiable matter .. .. | 2·2 |
| Total .. .. .. .. .. | 98·4 |

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[1] Taylor's Principles and Practice of Medical Jurisprudence, 1920, Vol. I, p. 306.

[2] Chemical Technology and Analysis of Oils, Fats and Waxes. *J. Lewkowitsch.* London, 1914, pp. 686–7.

[3] *Pharm. Weekblad,* 54, 121–5 (1917), through *Chem. Abst.,* 1917, p. 1437.

[4] *J. Biol. Chem.,* 1917, 29, 319–327, through *J. C. S.,* 1917, i., 364.

(*e*) Recent analysis by R. F. Ruttan of specimens of human adipocere.[1]

Result: Essentially composed of saturated fatty acids.

From these analyses it is evident that adipocere is composed almost entirely of fatty acids, but that it contains a certain amount of calcium soap, and probably in the early stages of its formation some ammonia soap, and therefore from its chemical composition there can be little doubt that adipocere is the residue of the fat pre-existing in the body, the greater part of which has undergone slow hydrolysis by water, but some small part of which has been saponified by ammonia (derived from decomposing nitrogenous tissue), this ammonia being ultimately replaced by lime. If this be so, what then is the explanation of the numerous observations by competent medical men who have declared that large parts of the body, or sometimes even the whole body, became converted into adipocere? It is suggested that the observations were generally correct but that the explanation given was wrong, and that what was thought to be muscle and organs converted into adipocere was merely a semblance of these things, the protein, which had largely disappeared, not having been changed into adipocere but having been replaced in part by adipocere. As is well known, a layer of fat exists underneath the skin and fat is found throughout most of the organs and tissues of the body, in some cases enveloping them, and if the protein putrefied and disappeared while the changes were taking place in the fat (and at least a partial putrefaction and disappearance of the protein must occur if ammonia is to be formed to combine with the fat as ammonia soap), there would remain the semblance of the muscle or organ, much shrunken in size, composed of adipocere. If also, as several authorities state, fat may be transferred from one part of the body to another during putrefaction, then a greater amount of adipocere in any one place than the pre-existing fat would account for could be explained. In some few

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[1] *Trans. Roy. Soc. Canada*, 1917 (iii.) 10, 169–170, through *J. C. S.*, 1917, i., p. 495.

cases possibly a mistake may have been made and what has been described as adipocere may have been something which resembled adipocere in appearance or was merely covered with a layer of adipocere. In any case the subject of adipocere formation requires further investigation to free it from the existing confusion, and chemical analysis must form an essential part of any such investigation. In all investigations, too, the body or part of the body consisting of adipocere should be weighed in order to ascertain what relation this weight bears to the amount of pre-formed fat normally present. The proportion of water in all specimens should also be determined, otherwise no true comparison with the normal body would be possible.

In this connection it may be mentioned that certain white crystalline efflorescences occurring on a number of specimens of Egyptian mummy tissue, and resembling salt very much in general appearance, which have been examined by the author all proved on analysis to be fatty acid, generally palmitic acid but in one case stearic acid.

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Taylor's Principles and Practice of Medical Jurisprudence. *F. J. Smith.* London, 1920.

Lyon's Medical Jurisprudence for India. *L. A. Waddell.* Calcutta, 1914.

Preservative Materials Used by the Ancient Egyptians in Embalming. *A. Lucas.* Cairo, 1911.

## Chapter XVII

# ROBBERY FROM LETTERS AND PARCELS

It is a comparatively easy matter to open a letter or parcel fastened in the usual way or even sealed with wax and to re-fasten or re-seal it in such a manner that it will pass muster without giving rise to suspicion unless critically examined, but it is very difficult to do so without leaving some traces of the operation, and these traces may be found by any one having experience, if careful search is made. As a rule the better the quality of the sealing wax and the worse the quality of the envelope or paper the more evident are the traces left. A cloth-lined envelope and a number of seals made with wax of an unusual colour, such as green, blue or yellow, afford some measure of protection. A special mineral ingredient, which can readily be tested for, if added to the wax, simplifies the detection of false seals.

In order to prevent envelopes being opened by wetting or steaming it has been suggested that the flap should be coated with isinglass or glue in place of gum, and that the part of the envelope beneath the flap should be treated with chromic acid, and it is stated that the isinglass or glue becomes so hard as to be absolutely insoluble in water, and hence the envelope cannot be opened except by cutting or tearing. It has also been suggested that the flap should be impregnated with one chemical ingredient and the part of the envelope immediately beneath it with another, so that when water or steam is applied to open the envelope the chemicals will react with one another and discolour the paper. The same action, however, would take place if the envelope were wetted accidentally, or if it were wetted too much when being closed in the first instance.

It would manifestly be impolitic to describe the various ways in which letters and parcels may be tampered with, though the expert to whom such questions are referred naturally acquires considerable experience in these matters.

Before the expert receives a letter or parcel for investigation it will almost necessarily have already been opened by the receiver, and it will generally be found that no examination whatever was made of it at the time of receipt, that no notice was taken of its condition, that little or no care has been taken to preserve it in the exact state in which it was received, and frequently valuable evidence has been destroyed by the careless way in which it has been handled, with the result that the expert is handicapped from the very commencement of the enquiry.

Envelopes and parcels which it is suspected or alleged have been tampered with should be photographed on receipt, since the subsequent examination frequently necessitates making some change in their appearance, such for instance as opening them in a new place or to a greater extent than was the case originally.

The examination of letters and parcels from which something is alleged to have been abstracted during transit consists very largely of a careful scrutiny (both with the naked eye and also with a lens) of the envelope or wrapper, and also of the wax seals and string, if any, together with a chemical analysis of the gum on the envelope and the wax of the seals. It is therefore a matter eminently fitted for the chemico-legal expert.

If an envelope of which the paper has a glazed surface has been steamed or otherwise wetted in order to open it, evidence of this will generally be found in the different and duller appearance of the paper in those places where the water has touched it, the smoothness and brilliance of the original surface being destroyed: if the paper is not glazed, the envelope may be opened without any evidence of the operation being left unless the paper is of very poor quality, in which case even though unglazed it will show signs of having been wetted. If the edge of the flap only has been wetted, this can be shown by means of iodine solution or iodine vapour, either of which produces a difference in colour between the wetted and unwetted portions.

When gum is applied to an envelope in order to re-fasten it after it has been opened an excess is sometimes used which squeezes out from under the flap and can readily be seen. Such an appearance, however, although always suspicious, does not necessarily indicate that the envelope has been opened and re-fastened, since slight smears of gum are often found on envelopes that have not been tampered with, these being due to the fact that with a well-gummed envelope, freely moistened, a little of the original gum may be squeezed out and smeared on the envelope just beyond the edge of the flap.

If the envelope has been opened by cutting one of the edges and has afterwards been closed again, this operation may have been so skilfully performed that no evidence whatever will be found on a careful examination of the outside of the edge even with a lens. To detect such a mode of opening and re-closing it is necessary to slit open all the four sides of the envelope with a sharp knife and to examine the edges inside. In every case where tampering is suspected the envelope should be opened in such a way as to allow the inside, and particularly the inside of the flap, to be carefully examined. It should be remembered, however, that it is not always the top flap of an envelope which is tampered with, but sometimes the bottom flap or one of the flaps at the sides.

Numbers and marks placed on envelopes by the post office employees are sometimes written with indelible pencil, and such marks may show signs of having been wetted if the envelope has been tampered with, and the name and address of the sender or other indications on the envelope, if made by means of a rubber stamp and aniline ink, as is often the case, may show the same thing. Wetting, however, may be accidental, as for example by rain.

False wax seals may frequently be detected on account of the metal seal used not being an exact reproduction of the original, or because the colour or the composition of the wax is not an exact match. It is possible, however, to reproduce a wax seal in such a manner that the impression itself defies detection, but even in such a case evidence that the envelope has been tampered with is frequently left either during the removal of the original wax or during re-sealing. It will be found a great help in the detection of forged seals and the opening of envelopes and parcels if the expert acquires personal experience of how to do these things. A few references to methods may be found in several books, such as that on Criminal Investigation by Hans Gross, but these should be accepted only as preliminary suggestions, since far more satisfactory methods exist though they cannot be described.

A considerable amount of valuable information may be obtained from a careful examination of the sealing wax on an envelope or parcel which has been sealed. Thus finger prints, or marks made by a fabric, or even threads from a fabric, may be found on the wax: sometimes, too, it is possible to prove that a seal consists of two separate and distinct layers of wax, one superimposed on the other: the nature and percentage of the ash may also furnish valuable information which may allow of one wax being distinguished from another of the same appearance: even small differences in colour are important, since they may indicate not only different kinds of wax, but may also show the manner in which the wax has been melted. Thus, for instance, sealing wax may be melted in the flame of a lamp or candle, or in larger quantity, in a pot by means of a burner. When melted in a flame the wax generally takes fire unless only a very small seal is made, and when this happens, that portion which has been alight is partly burned, and particles of black carbonized material can be seen in the wax, the colour of which is generally darkened. The amount of burning and hence of carbonization, however, may be so slight that it is not noticeable, especially in small seals. Frequently in post offices and banks, where sealing wax is required in large amount, it is melted in a small pot over a burner, which may be a spirit lamp, a gas flame, or an electric heater. When heated in this manner the wax does not inflame, and hence does not carbonize, although some colour change may and frequently does take place. The colour of a wax seal depends therefore not only upon the colour and composition of the original wax, but also on the way it has been melted. When melted in a flame the wax generally becomes darker owing to carbonization, and in addition particles of carbonized material are frequently visible: when melted over a burner there is generally no darkening, though sometimes a change of colour, and there are no particles of carbonized matter. The amount of visible blackening, however, also depends upon whether or not the molten wax on the paper has been stirred with the stick of burning wax as is sometimes done, since in this case the black particles may become partly or wholly masked by fresh wax and so may not be apparent. The spots and streaks of different colour sometimes seen in a wax seal are generally caused by the existence of particles of differently coloured wax in the original stick due to imperfect mixing.

**Illustrative Cases.**—1. This was a robbery of a large sum in banknotes from a sealed envelope, which had been sent through the post. Part only of the notes had been taken. The following clues were found:—

The wax seals were all identical and had all been made with the same metal seal, which was the genuine seal of the firm forwarding the letter or one identical with it.

There were two different and distinct layers of wax on two of the seals, one layer being subsequent in point of time to the other, and superimposed upon it.

There were wavy lines, and a few very small black-looking fibres on one of the seals. The fibres were found on microscopic examination to be wool of a very dark blue colour, and the lines to be the impression of a woven fabric.

In two places adhering to the under side of the flap of the envelope there were fragments of paper torn from the surface of the envelope beneath the flap, and these were embedded between two layers of wax, and on the envelope below the flap there were places from which the surface layer of the paper were missing, and there was wax on these torn surfaces. This could not possibly have happened unless the envelope had been sealed with wax beneath the flap (the usual method adopted by the firm forwarding the letter), then opened, the surface of the paper being torn during the process, then re-sealed with more wax.

All the wax was identical in colour and chemical composition, and corresponded to that used by the firm sending the letter.

The sealing wax used by the addressee, although of the same colour as the wax on the envelope, was of very different chemical composition.

The post office sealing wax was also very different in chemical composition from that on the envelope.

The dark blue woollen fibres found on the wax of one of the seals corresponded to the material of the coat of one of the employees of the firm sending the letter, and the right-hand sleeve of the coat was turned up and frayed at the turned-up edge. There was evidence, however, that this employee might have been called in to assist in the sealing of the envelope.

It was evident from the above facts that after the envelope had been sealed in the first place it was opened at the flap and then re-sealed in the same manner as before, the additional wax being identical in both colour and composition to that originally employed, and the impressions on the wax being probably made with the firm's own seal. Everything therefore pointed to the robbery having taken place before the envelope left the premises to be taken to the post, and that it was done hurriedly, since all the notes were not taken. It seems probable that after the flap of the envelope had been fastened down and when some, but not all of the wax seals had been put on, the clerk in charge, thinking the envelope was then secure, had gone away temporarily and that some one, taking advantage of his absence, re-opened the envelope, hurriedly abstracted some of the notes and then re-sealed it. Against this, however, was the fact that the postage stamps on the envelope represented the correct postage and registration fee for a package of the weight which the envelope originally had when it contained the full complement of notes, whereas when the letter reached its destination its weight was some sixty grams less, and hence it was then considerably overstamped.[1] To meet this difficulty, however, it might be

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[1] Apparent differences in weight may be due to incorrect scales and weights, or to careless weighing: actual differences in weight occur from pieces of sealing wax being inadvertently detached or from the presence of dirt or moisture.

assumed that the messenger who took the letter to the post, either mechanically or intentionally, asked for stamps to represent the original weight. It was admitted that letters of the same kind and weight had been sent to the same address on several previous occasions.

2. This case, like the last, was one of the robbery of banknotes from a sealed envelope which was sent through the post, the sum stolen being L.E. 5,000. The clues found were very similar in some respects to those in the previous case, and it was possible to prove that in several instances the wax seals were formed of two different layers of wax, one superimposed on the other. The lower and original seals in at least some cases had been made with a rectangular seal which had been taken out of use only a few days previous to the robbery, whereas the top seals were all made with a new circular seal. If a mistake had been made in the first instance and the wrong seal used, it would be a natural way of correcting the mistake for some one to be instructed to add more wax and to re-seal the envelope with the new seal and, if the gum on the flap was not quite dry, the occasion might be taken to open the envelope and carry out the robbery before re-sealing it. Both lots of wax were identical in composition and the seal used to make the impressions was the genuine one or an exact reproduction of it. In this case all the banknotes had been taken out and replaced by a packet of the same weight consisting of pieces of Arabic newspaper. The robbery had certainly been carried out by some one having access to the firm's wax and seal, and also to a balance for adjusting the weight of the newspaper packet. This had occupied some time, or careful preparation had been made beforehand for it. The papers were pieces of nine different newspapers, all published in Cairo where the robbery took place, and the dates extended over a period of six months. The origin of these was never traced.

3. This again was a robbery of banknotes from a sealed envelope sent through the post, the amount stolen being L.E. 3,000. In this case, as in the one last mentioned, all the notes were abstracted and pieces of newspaper substituted. The following significant facts were noted :—

Of the nine wax seals on the envelope five were on the flap and four elsewhere. The wax of the latter was identical both in colour and in composition with the firm's wax, and the seal used was identical with the firm's seal, and these four seals were undoubtedly part of the original sealing. The colour of the wax of the five seals on the flap was slightly but definitely different and darker than that of the other seals though the composition was the same: small differences in detail proved that the metal seal used to make these impressions was different from that used for the other four impressions, and that it was not the firm's seal.

The post office sealing wax was different both in colour and in composition from any on the envelope.

A portion of the surface layer of the paper of the envelope beneath the flap was torn away and was adhering to the flap, and in several places small pieces of paper were found between two layers of wax, all of which proved that the envelope had been opened and re-sealed after it was originally fastened.

The newspapers, with one exception, were all published in Alexandria, the place from which the envelope was sent: the exception was a Cairo paper having an extensive circulation in Alexandria. One of the Alexandria papers was dated the same day that the envelope was posted.

The conclusions arrived at were that the robbery certainly took place in Alexandria, and not at the post office, and unless the firm had other wax different from that of the four seals and from that of a specimen submitted for examination, which point was not satisfactorily cleared up, then the robbery probably did not take place on the firm's premises, since in such a case it would have been a very easy matter to have obtained some of the firm's wax; hence by exclusion the robbery occurred while the envelope was in transit between the firm's premises and the post office.

4. Two similar envelopes were sent by post by the same person from the same place on the same day to the same address. Each was stated to contain banknotes, one for L.E. 400 and the other for L.E. 320. On arrival the L.E. 320 was intact, while the other envelope contained pieces of thick pink paper instead of banknotes. This paper

consisted of wrappers from packets of a well-known local cigarette tobacco. Both envelopes were sealed in the same way with five wax seals, all of which were identical and had been made with the same metal seal. There were no signs whatever that the envelope from which the banknotes were missing had been opened and re-sealed, nor that it had been tampered with in any way. The envelopes were of the kind sold only at the post office, and it was proved that they had been purchased several days before the robbery: the writing was that of the local postmaster, who admitted that in contravention of regulations he had addressed and sealed the letters, using the post office wax and the seal of the sender; he stated, however, that the envelopes were already gummed down when they were handed to him. As a result of the enquiry it was proved that the owner of the money had robbed himself and had arranged matters in such a manner as to throw suspicion on the postmaster.

5. A key had been abstracted from a sealed envelope, used for the purpose of robbery and then replaced in an envelope similar to the first, but this was very clumsily done and it was readily proved that the envelope and sealing were not the originals.

6. In several cases there have been no signs that envelopes from which it is alleged that money or papers have been stolen in transit have been tampered with. In one instance the envelope showed no evidence of having been wetted; there was no extra gum; six seal impressions across the flap made with blue aniline ink had not been wetted, and the two parts of these seal impressions (the one on the flap and the other on the envelope) corresponded exactly; two pieces of stamp edging placed across the flap also showed no evidence of having been removed and replaced, and there was no evidence of the envelope having been opened in any other way. It seemed probable that the money had never been placed in the envelope. In another instance an envelope sent through the post was found on arrival to contain a blank sheet of paper instead of banknotes for L.E. 13. The evidence all indicated that the envelope had not been tampered with and hence that the money

had never been forwarded. One unusual feature was that the flap of the envelope had been gummed twice, but this second gumming was not the work of an amateur, but had been done when the envelope was manufactured, and when search was made other instances of the same mistake were found. In a third case an important document was missing from an envelope which had been delivered by the post in the usual way. On examination it was found that although the flap of the envelope had been sealed it had never been gummed down, and it seemed probable either that the letter had been abstracted before the envelope had been sealed, or that it had not been put in the envelope. There was no evidence to show that the envelope had been tampered with. Other cases have been without any particular interest and need not be specially mentioned.

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## CHAPTER XVIII

# STAINS AND MARKS

CHEMISTRY is the science of small things, and the chemist habitually tests for ingredients which are only present to the extent of a few parts per million or even per hundred millions, and the tenth, the hundredth and even the thousandth part of a milligram of a substance can sometimes be determined. The chemist, therefore, is peculiarly fitted to search for and to examine stains and marks. This, however, is rarely recognized except in the case of blood stains, with the result that much valuable evidence is frequently lost.

The testing of stains and marks does not as a rule obliterate or destroy them, but if this seems likely to happen they should first be photographed, so that a permanent record may be preserved for future reference or for production in Court. In any case a photograph will always be helpful, since it is difficult to convey an exact description of the appearance of a stain or mark by means of a written account. In the case of small stains and marks any photograph taken should be enlarged.

No general rules for the examination of stains and marks can be laid down, since no two cases are alike, but each must be made the subject of special study and individual treatment.

MARKS ON BULLETS.—These have already been considered.

STAINS AND MARKS ON CLOTHING.—These have also been dealt with.

**Stains and Marks on Documents.**—Stains and marks on documents are sometimes of considerable value as evidence. Thus it frequently happens that the post office cancellation and date marks on envelopes are impressed so vigorously that the outlines of these marks are indented on the letters inside. In several cases it has been possible to prove that the contents of certain envelopes received through the post had been through the post on a previous occasion, since they bore marks made by a post office stamp which did not correspond to any on the existing envelope. In one instance the marks were on an inner sheet only and hence the paper had been folded differently on the previous occasion. Also, if a particular post office mark shows through on to the far side of the envelope to that on which it was impressed, any letter inside must also have been marked, and hence in such a case a letter, folded sufficiently large to fill or almost fill the envelope, and which does not show the mark in question, cannot possibly have been in the envelope at the time it was stamped. In one case the fact that a certain grease stain which occurred on both the back and front of an envelope did not exist on a letter which was alleged to have been in the envelope, strengthened other evidence that the letter was a forgery. Sometimes stains of ink or colour on a document enable a connection to be established between one document and another or between a document and a particular person.

**Stains and Marks on the Person.**—In addition to marks on the person such as those resulting from wounds, operations or disease, the examination and identification of which are matters solely for the medical man, or of tattoo marks or marks on the hand indicating a particular occupation which fall within the province of the detective, there may be stains produced by chemicals which should be examined and tested by the chemico-legal expert. Such stains may be caused by acid, dyes, photographic reagents, ink, grease, tar and many other chemical substances. If any stains are found, the position and appearance should be noted: they should then if possible be removed by some solvent, and analysed. The solvent, using the minimum amount possible, is best applied, as a rule, by means of small swabs of cotton wool. To remove chemical stains from round the finger nails very small swabs formed by twisting a little cotton wool round a plain undyed wooden match-stick will be found useful. From the appearance of the stain its nature may frequently be surmised, and the particular kind of solvent to apply will then generally be known. Thus stains of iodine may be removed by sodium thiosulphate, stains of silver nitrate by potassium cyanide, stains of picric acid or dye stains by alcohol or water. If the nature of the stain is not known, water should be tried first and then alcohol and afterwards other organic solvents, for example petroleum spirit, the object being of course not simply to remove the stain but to remove it in such a condition that it may be analysed. On this account many reagents such as bleaching powder cannot be employed. It is not possible, however, to dissolve off all stains; thus for example tobacco stains and those caused by nitric acid cannot be removed. If nitric acid stains on the skin are kept under observation, it will be seen that the skin eventually peels off. The solution obtained by the removal of the stain should be squeezed from the cotton wool and examined chemically in the way indicated by its probable nature.

Claims made against insurance companies and others for alleged damage to goods by water can frequently be decided on chemical evidence, more particularly if the claim is one for damage by sea water. One of the important factors in such cases is sometimes the presence and nature of stains and marks on the goods, and in other cases the entire absence of staining.

**Illustrative Cases.**—1. In connection with a murder, the question of whether some pieces of white cotton cloth found in the house of a suspected person were identical with a piece of similar-looking material, which was wrapped round the body of the victim, was of considerable importance. This case will be described when dealing with the subject of Textile Fabrics, but among the minor points investigated were some pink-coloured stains on all the pieces of cloth. The stains on the several pieces were alike, and as the pieces of cloth had manifestly been sewn together at one time and had formed the loose washable cover for a couch, in all probability there had been a pink-coloured material underneath from which the stains had been derived.

2. In connection with the subject of Fires a case is quoted in which some whitish material caked on the outside of a

sack proved to be wheat flour, and this enabled the sack to be traced to a certain baker.

3. When dealing with the subject of Counterfeit Coins two cases are mentioned in which impressions of parts of the designs of coins were found on pieces of wood. These proved to be valuable links in the chain of evidence. In other cases impressions of coins on metal (lead, copper and iron) have been found, and in some instances it was possible to state that the coins with which the impressions had been made were counterfeit. In another case a large number of circular impressions the size of one of the Egyptian silver coins were found on two blocks of wood which had been used as a support for a press, and on several of these impressions there were parts of the design of a coin. Although the presence of the press, which was without dies, was suspicious in itself, it was not sufficient to connect the man on whose premises it was found with counterfeit coining, since it was possible that such a press might have been used for legitimate purposes, but the impressions of the coins on the wooden base placed it beyond doubt that it had been employed for making counterfeit coins. In another instance, from the marks left on the wooden parts of an apparatus which had been employed for putting the milling on counterfeit coins it was possible to prove, not only that the instrument had been used considerably, but also the size of coins which had been milled.

4. In various other cases of counterfeit coining, small amounts of moulding composition (sand, clay, plaster of Paris) found in brass and iron casting frames proved that these had been used, and in a few instances where the moulding composition was intact and bore impressions of coins, it was possible to state that certain coins found on the premises had been made in those particular moulds.

In some cases from the presence or absence of rust it has been possible to say whether certain machines and dies used for making counterfeit coins had been recently employed or not, and in one case, from some splashes of colour wash on the iron base of a press it was possible to place a limit to the time since which this had last been used.

5. In one case which occurred in Egypt when a man's

hands were being examined purple stains similar to the stains produced by a gold solution were noticed, and it was subsequently proved that the man had been engaged in "sweating" sovereigns by means of aqua regia. In another case a child was poisoned by means of silver nitrate and a stain of silver nitrate was found on the hand of the accused person.

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## CHAPTER XIX

# STRING AND ROPE

THE examination of string or rope found in connection with a crime will often furnish valuable clues.

Not only should the nature of the fibre be determined, but also that of any sizing material present, and attention should be directed to the ends to ascertain whether they have been cut or frayed, and also to any knots or loops, and to the manner in which these have been tied.

The fibres used for making string and rope[1] are very numerous, though the principal are hemp, jute and coir (*Cocos nucifera*), the first, however, not being confined to hemp proper (*Cannabis sativa*),[2] but including such fibres as Sisal hemp (*Agave rigida*), Manila hemp (*Musa textilis*), Sunn hemp (*Crotalaria juncea*), New Zealand flax or hemp (*Phormium tenax*), and many others generally termed hemp. In Egypt ropes are extensively made from date-palm fibre and string from Deccan hemp (*Hibiscus cannabinus*).[3] Cotton is also used for rope-making.

The nature of the fibre may be ascertained both microscopically and chemically. For the microscopical examination a small piece of the rope is unravelled, boiled in a small beaker with dilute sodium hydroxide solution (2 to 5 per cent.), washed well by decantation, rubbed up in a small porcelain mortar and dried between filter paper. A minute portion is then teased out on a slide with microscope needles, covered with a cover glass and examined.

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[1] By rope is meant cordage only, iron and steel wire rope being excluded.

[2] Of hemp proper there are several varieties which differ in appearance and quality, the two principal met with in Europe being Italian hemp and Russian hemp.

[3] Also known as Ambari hemp and called *Teel* in Egypt.

The various fibres may be distinguished chemically by the following reactions :—

| Fibre. | Phloroglucine (*a*). | Aniline Sulphate (*b*). | Iodine and Sulphuric Acid (*c*). |
|---|---|---|---|
| Flax .. .. .. | nil | nil | blue |
| Hemp (*Cannabis sativa*) | nil | slightly yellowish to yellowish brown | yellow or greenish |
| Jute .. .. .. | deep red | bright yellow | brownish yellow |
| Coir .. .. .. | red | yellow | — |
| Date-palm .. .. | deep red | slightly yellowish | — |

(*a*) and (*b*). See p. 78.

(*c*) The fibre is treated with a 1 per cent. solution of potassium iodide saturated with iodine. The excess of this is removed by filter paper and a few drops of a solution consisting of one volume of water, two volumes of glycerine and three volumes of sulphuric acid added. This is not a very satisfactory test, but may be useful sometimes. The blue coloration with flax is frequently so poor that it can only be distinguished under the microscope.

When examining a rope it is useful to determine (*a*) the "lay" which is the system on which the strands forming the rope are twisted, and which is measured by the angle between the direction of each strand and the direction of the centre line of the rope; (*b*) the size, which is the circumference in inches; (*c*) the number of strands; (*d*) the number of yarns per strand; and (*e*) the number of fibres per yarn.

**Illustrative Cases.**—1. In the second murder case described under Textile Fabrics the rope with which the body was tied was of two kinds, one being native rope made from date-palm fibre, and the other a better quality machine-made rope not of local manufacture. One of the pieces of rope was tied in a manner similar to that used in Egypt for hobbling animals.

2. In another case a piece of thick rope which had been employed in connection with scaffolding, and which had given way, and so caused an accident, was submitted to ascertain whether it had broken naturally or whether it had been cut intentionally. On examination it was found that the rope was strong and of good quality, and although it showed signs of wear along its whole length, yet apart from the broken portion, it was still fit for use. It had not been cut with a sharp instrument, but manifestly had worn through, probably by friction against a rough surface. From the appearance of the broken ends, the rupture had been gradual, and if the rope had been inspected the weakness could hardly have escaped detection and so the accident might have been prevented.

## Chapter XX

# TEXTILE FABRICS

The examination of textile fabrics as far as the work concerns the chemico-legal expert is generally for the purpose of discovering a clue that will establish the origin of some material found in connection with a crime, with sometimes the question of its identity or non-identity with some other material of similar appearance. The material is carefully examined, measured and described. The examination, which is largely microscopical and chemical, comprises the nature of the fibre, the colour, the method of weaving, whether for example it is plain, twilled or figured (i.e. has a pattern), the number of threads per inch in each direction of the cloth, the position and nature of any stains and marks and the nature of any dust or dirt.

**Nature of the Fibre.**—A small piece of the cloth should be unravelled, and, since the warp and weft threads are frequently of different material, these should be separately examined.

Preliminary Examination.—If the fabric consists of either animal or vegetable fibre alone and not a mixture of the two, a rough way of testing it is by burning, and for this purpose a few threads should be pulled out and held in a flame. Vegetable fibres burn readily and without disagreeable smell, whereas animal fibres burn with some difficulty and give off a disagreeable smell of burning nitrogenous matter. The burnt end of the fibre is also characteristic, vegetable fibres burning off sharply at the end, while animal fibres fuse to a rounded nodule. Any further examination is either microscopical or chemical.

Microscopical Examination.—This has already been dealt with under the subject of Fibres. As a rule before examination a small portion of the fabric should be boiled with dilute hydrochloric acid (2 per cent.), well washed with water by decantation and dried between filter paper in order to free it from dyes, loading and finishing materials.

CHEMICAL EXAMINATION.—There are two main principles underlying the chemical method of examination, one being the different colours produced in different fibres by various substances and the other the different solubilities of different fibres in various reagents. Both these methods may be used, not only for qualitative testing, but also to determine the proportion in which the several kinds of fibre occur in a mixed fabric.

*Colour Reaction.*—These have already been described when dealing with the subjects of Paper, Fibres, and String and Rope respectively.

*Solubilities.*—These are shown in the following table :—

| Fibre. | Sodium Hydroxide (*a*). | Sulphuric Acid (*b*). | Basic Zinc Chloride (*c*). | Hydrochloric Acid (*d*). |
|---|---|---|---|---|
| Wool .. | soluble | insoluble | insoluble | insoluble |
| Silk .. | soluble | insoluble | soluble | soluble |
| Cotton .. | insoluble | soluble | insoluble | insoluble |
| Linen .. | insoluble | soluble | insoluble | insoluble |

(*a*) 10 per cent.; hot.

(*b*) Strong; cold.

(*c*) Zinc chloride 100 grams, water 85 c.c.; zinc oxide 4 grams: heat until dissolved as completely as possible : allow to stand : decant : use boiling.

(*d*) Strong; cold.

Differences in the concentration of the reagent and whether used cold or hot make a considerable difference in the results obtained; thus silk which is soluble in hot sodium hydroxide is insoluble if the solution be cold, and though insoluble in cold sulphuric acid, it is soluble if the acid be hot, while hydrochloric acid acts strongly upon cotton if hot but not in the cold. In many cases the action is not instantaneous and therefore the time limit is important.

If it is desired to make the results quantitative, a small piece of fabric is first boiled in dilute hydrochloric acid (2 per cent.) to free it from finishing, loading and colouring matters, washed, dried and weighed, then treated with the appropriate solution and again washed, dried and weighed, the difference being the weight of the particular fibre dissolved. The results, however, can only be regarded as approximate, since most of the fibres shown as insoluble in various reagents are in reality attacked to a slight extent.

METHOD OF WEAVING.—This can be ascertained by examining the fabric under a lens. Only the broad features are required as a rule, any elaborate dissection being unnecessary.

*Count.*—Both the warp and weft fibres are counted. In the Gill form of counting apparatus the fabric is placed on the glass top of a small box the inside of which is illuminated by a tiny electric light. This is very useful if there are many examinations to be made and for certain kinds of cloth, but for occasional purposes an ordinary counting glass with a field one inch square will be found quite satisfactory.

STAINS AND MARKS.—These should be examined as described under the subject of Clothing.

DUST AND DIRT.—If present, dust and dirt may be collected in the manner mentioned under the subject of Clothing and examined as described when dealing with the special subject of Dust and Dirt.

**Illustrative Cases.**—1. This concerned the question of whether a piece of material found wrapped round the body of a murdered woman was identical with several other pieces of similar-looking material found in the house of one of the persons suspected of the murder. On examination it was found that the various pieces of cloth were cotton, that all the pieces were of the same fineness, that is to say the number of threads to the inch was the same in each case, and that all were of the same width between the selvedge edges. These facts, together with the further facts that all the pieces bore a similar coloured stain, and all had been sewn with similar and somewhat unusual thread, small portions of which still remained, and that all fitted together, justified the report that the several pieces were of identical material, and that at one time they had been sewn together and had probably formed the loose washable cover for a couch.

2. The naked body of a man was discovered in a canal, tied up in a sack which was weighted with stones. The sack and stones were submitted to ascertain whether any clues to the murderer could be found.

The sack was of a kind and quality largely used in Egypt for cotton seeds and cereals, and similar sacks may be obtained almost anywhere in the country. The sack, however, had been patched in two places, and in both instances in a similar manner and with similar material, and on a microscopical examination this material was found to be camel hair, and the thread with which it was sewn was also of camel hair. Similar thread had also been used to mend one corner of the sack. From these facts the following conclusions were drawn :—

That the sack had at one time belonged to some one who could not readily obtain a new sack and to whom therefore a sack was a thing of value and something to be repaired.

That since similar sacks are extensively employed in Egypt, the ordinary Egyptian would not experience much difficulty in obtaining sacks in good condition, and hence would have no need to mend an old sack. Moreover, it is specially characteristic of the ordinary Egyptian that as a rule he does not mend things, but uses them until they cannot be used any longer, and then discards them.

Hence the fact of a mended sack seemed to point away from an ordinary Egyptian, and at the same time to some one poorer and less able to obtain sacks, that is by exclusion, to a bedouin.

A bedouin was indicated too by the nature of the stones used to weight the sack, which, with two exceptions, were rounded flints, the exceptions being silicified wood, all of which must have come from the desert.[1] It was advised that search should be made for a bedouin, and it was subse-

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[1] It was of course by no means certain that these stones had been taken directly from the desert, similar stones being sometimes used in Egypt for railway ballast.

quently ascertained that the crime had been committed by a bedouin.

3. In several cases in which cloth suspected to have been stolen has been forwarded for identification, it has been found impossible, in the absence of any special distinguishing mark, to prove the theft. Although the cloth has been of similar material (linen in some cases, cotton in others, and linen warp and cotton weft in others) and of similar count to the samples from stock submitted for comparison, it has been of a kind that is very common, and hence no definite proof that it was stolen was possible.

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## Chapter XXI

# TOBACCO

The chemico-legal expert may occasionally be called upon to identify a specimen of tobacco found in connection with a crime. Any tobacco so found will almost certainly be in the form of the unsmoked end of a cigar or cigarette. Thus, in the case of the attempt on the life of H.H. the late Sultan Hussein of Egypt in 1915, several cigarette ends, one being still alight, were found on the floor of the room from which the bomb was thrown, and it seemed possible that this latter had been used to light the fuse of the bomb. These cigarette ends bore the name of a certain tobacconist, and cigarettes bearing the same name and mark were found in the house of one of the accused persons. The tobacconist stated that he supplied the accused with a special mixture. In view of these facts it became important to ascertain whether the tobacco in the cigarette ends and that in the cigarettes of the accused was identical, and whether it was the particular mixture in question.

The chief differences between various tobaccos are the size, shape, colour and general appearance of the leaf, and more particularly the flavour on smoking. When a tobacco is cut, the size, shape and appearance of the leaf are lost, and in the case of a cigar or cigarette that has been partly smoked, the colour of the tobacco may be modified by the moisture and products of combustion which accumulate in the unsmoked end, and the best test, namely the flavour, cannot as a rule be applied at all, since even if the amount of tobacco available were sufficient, such a test would entirely destroy the evidence. For the same reason a chemical analysis is also precluded, since as a rule a single small specimen of the tobacco is all that is available. Hence one is forced back upon such facts as the appearance of the tobacco, both to the naked eye and also when examined under a lens or with a microscope. In the case already referred to the tobacco was particularly finely cut, and this fact together with the proportion and size of the cross sections of stem present made it very characteristic and quite different from a number of other cigarette tobaccos examined, and since the tobacco of the cigarette ends and that of the cigarettes belonging to the accused had the same characteristics as well as the same general appearance and colour, it was probable that they were the same tobacco.

Whenever there is a sufficiently large sample of tobacco a chemical analysis should always be made. The determinations likely to be most helpful are the amount and nature of the ash. The following table shows some of the constituents of the ash in which the most marked differences occur[1] :—

TOBACCO ASH.

| Kind of Tobacco (*a*). | Total Ash. | Potassium Chloride. | Lime. | Magnesia. | Potash. | Sand. |
|---|---|---|---|---|---|---|
| | % | % | % | % | % | % |
| China .. | 14·8 | 3·7 | 31·1 | 12·7 | 14·4 | 2·1 |
| German .. | 25·9 | 25·0 | 35·7 | 6·5 | — | 0·9 |
| Havana .. | 21·4 | 12·7 | 33·1 | 5·3 | 11·8 | 1·8 |
| Java .. | 18·8 | 19·6 | 22·3 | 8·5 | 14·1 | 1·7 |
| Kentucky .. | 18·0 | 2·4 | 29·5 | 6·8 | 16·8 | 5·3 |
| Latakia .. | 18·2 | 4·1 | 34·7 | 5·5 | 19·6 | 0·9 |
| Manila .. | 21·4 | 6·5 | 33·3 | 7·2 | 16·3 | 0·6 |
| Turkey .. | 12·9 | 19·3 | 22·5 | 9·2 | 14·5 | 1·8 |
| Virginia ripe | 15·0 | 2·5 | 18·9 | 6·7 | 34·2 | 1·0 |
| Virginia bright | 12·3 | 7·0 | 21·7 | 12·4 | 22·2 | 1·3 |

(*a*) The tobacco was dried at 100° C. before being analysed.

When dealing with cigarettes, if the size of the sample will allow, the tobacco and paper should be separately examined, the former for the nature of the tobacco, of which one important feature is the amount and composition of the ash, and the latter for the nature of (*a*) the fibre, (*b*) the sizing material, and (*c*) the loading material.

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[1] A Dictionary of Applied Chemistry. *Sir Ed. Thorpe.* Vol. V. London, 1913, p. 509.

It has been stated that since the ash of different tobaccos varies considerably in composition (as may be seen from the figures given) therefore from an analysis of ordinary tobacco ash, such as might be found on the scene of a crime, the nature of the tobacco smoked by the criminal may be ascertained. Such an argument, however, neglects to take into consideration one very important factor, namely that tobaccos as sold are never pure and unmixed; thus both pipe and cigarette tobaccos are invariably blends of different tobaccos and even cigars are not made from a single kind of tobacco, the outside leaf or "outer wrapper" being generally cf a different tobacco from the rest of the cigar. Also there is no certainty that the same blends are always employed for the same brands of tobacco or that they are employed in the same proportion. Cigarette ash too necessarily includes the ash of the paper.

In order to determine if possible the value of tobacco ash as a clue in criminal investigation, the author has made analyses of (*a*) the ash of several different brands of cigars, (*b*) the ash of fourteen kinds of cigarettes, each specially made from a pure unmixed tobacco, and (*c*) the ash of several different brands of cigarettes as found on the market. The analyses were only partial, the determinations made being confined to the constituents that appeared likely to be the most useful for differentiating the various tobaccos. The following results were obtained :—

CIGAR ASH.

| Kind of Cigar. | Colour of Ash. | Chlorine as Potassium Chloride. | Matter Insoluble in Hydrochloric Acid (Sand). |
|---|---|---|---|
| | | % | % |
| Havana .. .. | light grey | 12·2 | 14·5 |
| Indian A .. .. | dark grey | 25·0 | 13·8 |
| Indian B .. .. | dark grey | 24·0 | 12·1 |
| Indian C .. .. | dark grey | 22·3 | 18·4 |

Cigarette Ash.

| Kind of Tobacco. | Total Ash. | Chlorine as Potassium Chloride. |
|---|---|---|
| | % | % |
| Algerian .. .. .. | 17·3 | 27.5 |
| American .. .. .. | 15·6 | 2·0 |
| Bulgarian .. .. .. | 18·5 | 1·6 |
| Burmese .. .. .. | 19·0 | 0·7 |
| Chinese .. .. .. | 10·0 | 2·5 |
| Greek .. .. .. | 19·2 | 10·3 |
| Indian .. .. .. | 15·8 | 42·3 |
| Japanese .. .. .. | 18·0 | 3·1 |
| Porto Rico .. .. .. | 16·1 | 23·9 |
| Russian .. .. .. | 22·3 | 3·4 |
| Serbian .. .. .. | 17·3 | 1·7 |
| Turkish (Samsoun) .. .. | 14·5 | 5·0 |
| Turkish (Smyrna) .. .. | 19·7 | 5·5 |
| Turkish (Cavalla) .. .. | 15·2 | 2·8 |

Cigarette Ash.

| Kind of Cigarette. | Chlorine as Potassium Chloride. | Matter Insoluble in Hydrochloric Acid (Sand). |
|---|---|---|
| | % | % |
| English .. .. .. | 3·6 | 36·0 |
| Egyptian A .. .. | 7·5 | 20·6 |
| Egyptian B .. .. | 7·4 | 18·1 |

These results show that there are considerable differences in the proportion of chloride present in the ash of various tobaccos. Thus with respect to the particular cigars and cigarettes examined there was approximately twice as much chloride in the Indian cigars as in the Havana cigars and twice as much chloride in the Egyptian cigarettes as in the English cigarettes. Both from the colour of the ash and from the proportion of chloride present there would be no difficulty whatever in distinguishing between the ash of the Havana cigar and that of the Indian cigars.

In the same way the ash of the English cigarettes could readily be distinguished from that of the Egyptian cigarettes. The matter insoluble in hydrochloric acid did not show a sufficiently large difference in amount in the various kinds of cigars to be helpful, but in the case of the cigarettes the English ones contained approximately twice as much as the Egyptian ones. The insoluble matter, which was examined microscopically, both with and without polarized light, consisted largely of silica, the only marked difference of any sort being that in the Havana cigar ash the silica was in a finer state of division than was the case with either the Indian cigars or the cigarettes. The differences in the proportion and nature of other minerals besides the silica were very small and not enough to be characteristic, though it seems possible that some special mineral, such as mica, might be present in notable amount in the ash of particular tobaccos. The experiments, however, have been too few to enable any general rule or hard and fast limits to be laid down, but manifestly from the appearance and chemical analysis of tobacco ash the nature of the tobacco smoked may sometimes be proved. The principal value of such evidence will probably consist in the negative fact that a certain suspected person does not smoke the particular kind of tobacco indicated by the ash found, and hence may be eliminated from the case. The positive fact that a certain person does smoke the kind of tobacco shown by the ash found may mean very little, since no matter what the kind of tobacco may be it will certainly be smoked by a great number of other people besides the criminal.

## Methods of Analysis.

*Ash.*—A weighed quantity of tobacco is incinerated by means of a Bunsen burner in a small platinum or porcelain dish, the heat employed being the lowest possible to ensure complete combustion of the carbon and not sufficiently high to decompose any carbonates present. The end of the operation is indicated by the white or light grey colour of the ash. A muffle furnace should never be used. The contents of the dish should be stirred with a stiff platinum wire from time to time during the ignition, the wire being weighed with the dish.

*Chloride.*—A weighed portion of the ash is transferred to a small standard stoppered flask, water added to the mark, the flask well and repeatedly shaken, the solution filtered and an aliquot portion taken and titrated with standard silver nitrate solution in the usual way. The result is calculated to chlorine or potassium chloride.

*Sand.*—A weighed portion of the ash is boiled with hydrochloric acid, the solution filtered and the residue washed, dried, weighed and examined. A microscopical examination both with and without polarized light, should be made.

*Lime.*—This is determined in the usual way in the filtrate from the sand.

*Magnesia.*—This is determined in the usual way after the lime has been separated.

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