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A GENERAL ACCOUNT OF MICRO-CHEMICAL METHODS IN CRIMINAL INVESTIGATIONS

J. B. Firth

J. B. Firth, D. Sc., F. R. I. C., M. I. Chem. E., is Director of the North-Western Forensic Science Laboratory, Preston, England. During 1948 Dr. Firth was asked to organize a meeting dealing with the subject of Micro-Chemical Methods in Forensic Investigations. To open this meeting he presented an illustrated lecture which gave a general account of the special chemical methods that have been developed to further scientific criminal investigation. His present article is based on the material included in this lecture.—Editor.

Until comparatively recently the application of science to criminal investigations was directed mainly to major offences such as murder, or more generally, crimes against the person. With the development of civilization there have been a very considerable change and augmentation of the resources of the criminal, not only in weapons—from bludgeon to firearms,—but also in poisons—from snake venom and poison pot to toxic chemicals. Poisoning seems to exert to some degree a fascination for the human race. History records many cases of death by criminal poisoning as well as the production of alleged antidotes. Thus, Sir William Willcox records that in the first century B.C., Mithridates conducted toxicological experiments on condemned criminals and others, and invented an antidote alleged to have contained 36 ingredients. It is further claimed that he became so immune to poisons that, wishing to commit suicide, he was compelled to ask a mercenary to kill him by sword.

Although poisoning in England was not as prevalent before 1500 as it was on the Continent, it did exist. It is recorded that Henry VIII ordered poisoners to be boiled to death. After this era, criminal poisoning developed fairly rapidly and became quite common. Up to the 19th century the chief poisons were probably preparations containing arsenic and corrosive sublimates.

The methods of diagnosis of poisoning were at first limited, depending for the most part on observations before and during illness together with post-mortem appearances. This was the beginning of scientific investigations relating to poisoning cases (toxicology) and were undertaken by qualified scientific men working in a field which has become known as Forensic Medicine or Medical Jurisprudence. The development of toxicology was in some measure restricted and controlled by the advances in modern chemistry and reliable analytical methods and ran parallel with developments in forensic medicine.

The foundations of toxicology were laid towards the end of the eighteenth and beginning of the nineteenth centuries. Thus,
we have the discovery by Scheele of arsenic (1775), hydrogen cyanide (1783), the Marsh test for arsenic (1836), the detection of arsenic in alimentary tract and organs by Orfila (1839), which indicated the importance of the determination of the degree of absorption of this poison. The first systematic descriptions of chemical test for poisons in Britain appear to have been given by Christian in his Treatise on Poisons (1829). In 1844 Fresenius and Von Babo devised a scheme for the search and detection of mineral poisons including methods for the destruction of organic matter (viscera, etc.,) prior to analysis. In 1850 Stas described a process by which alkaloid poisons could be extracted from viscera, a modified form which (Stas-Otto) is frequently used to-day.

The developments of toxicology falls into three phases: (a) The detection and recognition of poisons by their physiological effects and observations made at post mortems, (b) the detection and isolation of the poison, and (c) the quantitative estimation of and the distribution of the poison within the body with the view of ascertaining the total amount taken and the period involved.

Modern medicine has become an applied science, and its progress is closely interlocked with progress in chemistry. Many products of the chemist have been of considerable value in medicine but many substances used in modern medicine are scheduled poisons and when taken in excess produce fatal results. Thus, in the majority of poisoning cases, the subsequent detection and estimation falls naturally into the province of the chemist.

Again, with the discovery and use of highly powerful and toxic substances, the amount required to produce fatal results becomes correspondingly small, and the subsequent detection and determination of such substances in the body become correspondingly difficult. In these cases highly specialized techniques have had to be developed involving microchemical, colorimetric, electrometric, and nephelometric methods. It would naturally follow, therefore, that toxicological investigation has passed from the province of the medical man to the chemist, and in many cases to a chemist highly skilled in microchemical techniques.

Another phase in application of science to forensic medicine followed the compulsory appointment by public authorities of a public analyst of proved ability, as an analyst and microscopist. Although the primarily duties of a public analyst related to the purity of foods, his work on occasions involved giving of evi-
dence in courts of law. Not infrequently, he came to be regarded as the local scientist and was called upon to give assistance in many problems requiring scientific investigation including investigations relating to crime. Thus, the public analyst on occasion functioned as a forensic chemist. Some public analysts showed considerable ability in this field; whilst others showed marked lack of interest in this type of work, treating problems submitted to them in a very routine manner. On occasions, private consultants developed limited fields in forensic science with marked success. For example, C. Ainsworth Mitchell focused his attention of the scientific examination of documents, and made a comprehensive study of the chemistry of inks with the result that he became an outstanding English authority on these subjects.

The field of forensic science investigations is considerable, and it would be unreasonable, if not impossible, for an individual to become an expert on all subjects likely to occur in scientific criminal investigations, nor would it be economic to attempt to staff and equip each forensic science laboratory so as to be able to undertake every conceivable problem. This is provided for in Britain by the utilization of the services, not only the other government laboratories, including research laboratories, but also the facilities afforded by the research and technical laboratories of many industries. The willingness with which industrial organizations have placed at the disposal of the director of a regional laboratory any special resources they may have, has materially contributed to the efficiency of the laboratory. Even the individual specialist can often be pressed into services when required. Thus, it becomes possible for a director to bring to bear on a criminological problem the scientific resources of the country.

Coming now to the main theme of this paper with few exceptions it cannot be said that there are micro-chemical methods only applicable to forensic investigations. It is rather a case of adapting known methods to deal with small quantities of material involved in criminal investigations. It is true, however, that in some cases modification of apparatus and technique may be necessary, particularly with regard to the isolation of material in a form suitable for examination. Thus in many toxicological examinations, the amount of parent material may be relatively great, whilst the final material to be examined may be extremely small, and in obtaining this final material micro-physical methods have frequently to be used, e.g., fractional extractions, crystallizations, distillations, sublimations, and selective absorptions, followed in some cases by elusion by selective solvents.
As with all branches of analysis, there are qualitative and quantitative methods, i.e., methods for detection and methods for estimation. In some forensic investigations, the presence of or absence of a material may be all that is required, whilst in other cases, exact quantitative data may be necessary.

In the past the major attention has been focussed on toxicological investigations, and with the ever increasing flood of narcotic drugs on the market, the problems confronting the forensic chemist have increased. In view of the fact, that many of these drugs are used in the medical treatment of disease, it is of paramount importance to determine whether the drug is in excess of the medicinal dose, and if so, the degree of excess.

Some drugs modify or breakdown in the body and in endeavouring to obtain a value for the total amount taken, both unchanged material and decomposition products may have to be determined. In many cases, however, it is not possible to assess accurately the total amount taken from analytical data alone; in other cases the material examined is by no means comprehensive. To cite an example, in a simple case of aspirin poisoning, the limited material submitted provided evidence of aspirin equivalent to 2.3337 gms. or 7.2, 5-grain tablets. This amount included some unchanged aspirin from digestive tract, yet it was known that deceased had taken the contents of at least two—50 tablet bottles.

Many present day problems of toxicology require very refined micro-chemical techniques. While in general the victims of narcotic drugs are known to have consumed large quantities, the amount of these drugs recovered is relatively small in comparison. With the ever increasing number of barbituric acid derivatives, specific indentification of the barbiturate becomes increasing difficult. In an effort to overcome these problems the use of absorption spectra has been tried at the Preston laboratory, but without much success. G. E. Turfitt has proposed a method involving the use of micro mixed melting points with which he has had good success.¹

Criminal investigations alone allows only a limited experience in the field of toxicology so that many of the laboratories carry out investigations on behalf of the Coroners as well. In this way the personnel is able to maintain as wide experience as possible.

A fairly common micro-chemical investigation is that of alcohol in the blood and/or urine of a motorist, particularly in case

¹ For a brief description of this method see Dr. Turfitt’s abstract of his paper “Micro Methods in Forensic Toxicology with Particular Reference to the Barbiturate Drugs,” Jr. of Crim. Law & Criminal. (Police Science Technical Abstracts) 59 (6): 894 (Mar.-Apr. 1949).
of accident, and in some instances it is essential to test blood of
the accident "victim" as well. The method usually adopted is
that of oxidation by standardized acid-dichromates; the excess
dichromate being estimated by the addition of potassium iodine
followed by titration with sodium thiosulphate. When the avail-
able blood is very small the liberated iodine is estimated colori-
metrically after extraction with carbon bisulphide. Alcohol in
the urine may be determined by similar methods, but it should
be noted that allowance for oxidizable acetone bodies must be
made in the case of urine. While opinions differ with regard to
the stability of alcohol in urine it is generally held that alcohol is
more stable in urine than in blood. Since it is known that the
alcohol content of the blood falls off with time it is essential
that these tests are made at the earliest opportunity. These tests
have definite importance in investigations in which there is
suspicion that the person under investigation has been drinking
for there are instances when overdose of certain drugs as well
as other causes may produce similar effects to drunkenness.

Many of the so-called spot tests, well known to the micro-
analyst, are of considerable value in preliminary qualitative exa-
minations and can be followed by colorimetric estimation with
or without the use of the absorptiometer. These methods can be
applied to the many forensic investigations in which only very
small amounts of material are available. These may be in the
form of stains, metallic smears, paint smears on objects or
instruments, of tiny fragments of metal, glass, and the like from
clothing or found at the scene of an incident. In other cases
involving comparatively large amounts of material very positive
identity may be established by the detection and estimation of
impurities. Actually in these cases it may be the impurities
which are the most valuable in the ultimate identifications of
the material.

There is the occasional forensic problem in which micro-
precipitations, followed by crystal identification and micro-
weighing can be of assistance.

In dealing with minute quantities of inorganic materials, or
in general material containing metals, the spectrograph is one
of the most valuable instruments from both the qualitative and
quantitative aspects. In the latter problem care must be exer-
cised in the preparation of electrodes, controlling the arc, and
using the same amount of material in both the control and test
fragment. As has already been pointed out, in identifying
material from the scene of a crime with specimens from a sus-
pect vehicle, instrument, or clothing it is often the impurities,
sometimes present as only traces, which are of significance and not the basic substances, e.g. glass, paint, alloys, etc.

Micro-physical methods can be used to augment micro-chemical techniques in certain classes of problems. This is particularly true in the case of glass fragments. Specific gravity established by the flotation method, refractive index determined with mixed liquids, and appearance of the fragments under ultra-violet light may be of significance. In comparing two specimens of a substance like glass, say one specimen from the crime scene with one recovered from a suspect vehicle or person, it is essential that agreement in both chemical and physical properties be established. The chemical analysis alone may be insufficient and inconclusive. Differences between glasses have been detected even when there was agreement in the chemical composition.\(^2\)

Paint fragments present a special problem to the forensic chemist. The layer which has been built up is often of importance, because, as in the case of an impact, only surface layer may be of significance and a general analysis of a flake may be misleading. To cite a motor car accident investigation, the bumper bar had been painted twice giving two layers of different composition paints. A general analysis of the flake taken from the vehicle did not agree with the paint on the victim’s clothing, but the outer layer of paint did agree.

From this brief survey it can be seen that chemical problems of the forensic laboratories are widely diversified. In many instances chemical analysis may be of fundamental importance, in others it constitutes one or two pieces in the jigsaw which ultimately is to solve the crime. Again in others the negative results are of extreme value from the standpoint of elimination. While the resources of the criminal has seen a marked change with the growth of civilization forensic chemical analysis has kept abreast with his progress so that today it plays an important part in criminal investigations.

\(^2\) N. A. Morris, “Identification of Glass Splinters,” the Analyst, 1934, page 686, describes an investigation in which 65 specimens of glass were examined from various sources and only one agreed in all three respects with the broken window, viz., ultra-violet examination, refractive index determination, and specific gravity of the specimens.