

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

Death.

We regret to record the death, on August 5th, of Mr. Lester Reed, Public Analyst for Croydon.

Problems in Connection with Ancient Egyptian Materials.

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(Read at the Meeting, June 2, 1926.)

It is only in comparatively recent years that the chemist has been consulted by the archaeologist and has been given an opportunity of examining antique objects, and up to the present time little more than preliminary work has been possible and a considerable amount of detailed systematic chemical analysis still remains to be done.

Antique objects are examined by the chemist for several reasons, chiefly in order to furnish information concerning the materials of which they are composed, so that they may be correctly described and sometimes so that they may be approximately dated, but also to ascertain the nature of any change or deterioration that has taken place, so that suitable treatment may be applied, and occasionally to detect forgeries.

Since antique objects are composed of the same kinds of materials as the substances that an analyst in general practice deals with regularly, and since the principles underlying the examination of these materials are well known, it follows that whatever interest such objects may have in themselves or by reason of their antiquity, there can be little that is new about the methods used in their examination. It is not proposed, therefore, to describe methods, except incidentally,

but rather to give an account of some of the unsolved chemical problems in connection with the materials.

At the outset it should be remembered, first, that many antique objects are of such a kind that no sample whatever can be taken for analysis, since this would injure or destroy the objects; second, that even when sampling is permissible, the amount of material available for analysis is generally very small; third, that an object is not always in its original condition, but may have suffered deterioration, resulting in considerable chemical change; and fourth, that frequently some examination, if only a preliminary one, must be made on the spot with very few reagents and the simplest appliances. All these factors therefore condition and limit the nature and extent of the examination it is possible to make.

As it would be impossible to discuss all the different kinds of material, that from time to time present chemical problems for solution, a few of the most important only will be briefly considered. These are flint implements, faience, glass, metals, mortar and plaster, oils and fats, pigments and varnish, pottery, resinous materials, stone, and textile fabrics; these will now be separately described.

FLINT IMPLEMENTS.—These go back to Paleolithic and Neolithic times, periods so remote as to be quite undatable. The chemical interest in these implements lies in the brown or black patina with which many of them are covered. This patina consists essentially of a mixture of oxides of iron and manganese in varying proportions, and there is considerable difference of opinion regarding the source of these oxides, the probability, in my opinion, being that they have been formed from compounds of iron and manganese contained in the flint, and that when this was exposed on the surface of the ground and was regularly moistened by dew and occasionally by rain, traces of iron and manganese existing in the ferrous and manganous conditions respectively were gradually dissolved, and being drawn by capillary attraction to the surface, became converted into a thin film of insoluble oxides by exposure and heat. If the time taken for these changes to occur could be estimated even approximately this would prove of immense value to the archaeologist, but any such estimate seems hopeless.

FAIENCE.—Faience is a product peculiar to Egypt, and may be defined as glazed siliceous ware; it is composed of a body material covered with a coloured glaze. There is, of course, no difficulty in determining, by the usual methods of chemical analysis, the composition of either the body material or of the glaze, and the former consists of practically pure silica and the latter is a soda-lime-silicate. The problems in connection with faience are: first, what is the origin of the silica of the body material; and, second, in what manner was it held together while being shaped and glazed?

A microscopical examination of the material shows that it consists essentially of angular particles of quartz that are undoubtedly the result of artificial crushing, and it is manifestly some form of powdered quartz, but what particular form it is impossible to say. Crushed white siliceous sand seems one of the most likely substances to have been employed and would have been generally available; crushed quartz rock, such as that occurring at Aswan, would also be suitable,

but would take more labour to prepare and would only be available in one locality. It was quartz, however, that at a very early date was fashioned into small objects, which were then glazed, a practice from which the making of faience probably originated.

Such a material as that described possesses no coherence whatever naturally, and the body of a faience object is generally so friable that it can readily be rubbed away with the fingers, except in those instances in which the glaze has penetrated below the surface. How then was the material held together while being shaped and glazed? It is frequently stated that a small proportion of clay, sufficient to make the material plastic, was added, but both chemical analysis and microscopical examination prove clay to be absent. Neither was gypsum the binding material, since this also is shown by analysis to be absent. Fat, gum and glue have all been suggested, and in several specimens out of a large number examined there were a few small particles of black organic matter distributed throughout the mass, which conceivably might have been the remains of some adhesive, but the evidence was too slight to be in any way conclusive. In the case of one particular lot of small faience figures, however, all from the same place, there was a grey inner core surrounded by a zone of white. This grey core contained a number of black particles, and there was little doubt that its dark colour was due to the presence of organic matter that had been partly burned. This was confirmed by strongly heating the specimens, when the core became definitely lighter in colour, though not entirely white. In those instances in which the glaze has penetrated the body material this now acts as a binder, but manifestly it could not operate while the object was being shaped before the glaze was applied. That some, at least, of the smaller faience objects were moulded is proved by the finding of a large number of red pottery moulds, in some of which were the remains of siliceous material.

GLASS.—Glaze, not only on faience, which has just been described, but also on quartz and on steatite, was used in Egypt in archaic times, that is before or about 3000 B.C., and it would seem a very simple step from glaze to glass, since they are both of the same composition and must have been made in the same way from the same materials, the only difference between them being in the manner of use. Glaze being always applied to the surface of an object, whereas glass was used independently. The development from glaze to glass, however, took a very long time, and it was not until about the beginning of the Eighteenth Dynasty, some 1800 years after glaze was employed, that glass came into general use. This date of the introduction or invention of glass is one of the most disputed points in Egyptology, and objects of much earlier date are frequently claimed to be glass, and it is not only possible, but probable, that glass was made exceptionally and in small quantity for simple objects some time before its use became general. It might seem a very easy matter to ascertain whether an object was made of glass or not, but it is not so easy as it appears at first sight. The objects in dispute are not vessels, about which there could be little doubt, but generally small pieces of inlay, often in jewellery, which naturally cannot be subjected to any analysis,

and can, at most, only be examined physically. The questioned material, if not glass, may be a glazed material, such as glazed quartz or faience, or it may be a natural stone, probably not of normal quality or colour or possibly deteriorated. The only way of settling such questions is by careful inspection by means of a powerful lens or, better, a microscope.

Ancient Egyptian glass is a soda-lime-silicate containing an excess of alkali and was apparently made from sand and natural soda (natron), the lime probably being an impurity in the sand. There was practically no colourless transparent glass, since glass was not required to transmit light. The white opaque glass owes its opacity to oxide of tin. The red glass was coloured by cuprous oxide. The blue glass was generally coloured by a copper compound, though occasionally with cobalt. The green was generally also coloured by copper, though sometimes by iron. The yellow glass was sometimes coloured by lead and antimony and sometimes by iron and manganese. The amethyst colour was produced by manganese, and the black by copper and manganese or sometimes by a large proportion of iron.

The colour of ancient Egyptian glaze and glass frequently fades or changes; thus blue becomes white, green turns brown, and red becomes covered with a coating of green. The fading of blue to white is probably due to the reduction of the copper from the cupric to the cuprous condition, but why this should take place is not clear; green manifestly turns brown owing to the oxidation of the iron present, and the change from red to green is caused by the formation of a cupric compound from the cuprous oxide present. A considerable amount of chemical work yet remains to be done on ancient Egyptian glass; thus, although cobalt has been reported by several analysts, others still deny that it was ever used. Again, a knowledge of the dates at which certain colouring materials were first employed would be valuable, for it would not only help to date the glass, but would also throw light upon Egypt's intercourse with other nations, as some of these colouring materials, antimony and cobalt, for instance, must have been imported.

METALS.—A large number of the earlier analyses of ancient Egyptian metal objects which have been made are practically useless. This is partly owing to the fact that many of the impurities occurring in small proportion were not determined, and partly because the objects have generally been corroded and often badly corroded, and, as corrosive influences never attack a metal and its constituents proportionately and uniformly, it follows that a chemical analysis of such material cannot represent the original composition.

With regard to the analysis of corroded metal or altered material of any sort the analyst is confronted with a dilemma having three horns in place of the usual two. If he presents a complete and detailed analysis of the specimen as it reaches him, he will almost certainly mislead the archaeologist; if he makes only a partial analysis, he may omit to search for traces of various ingredients the presence or absence of which might supply valuable information; whilst if he attempts to explain his results, he will almost certainly go astray unless he knows the conditions under which such materials occur, both naturally and artificially, and unless he has some knowledge of archaeology.

All analyses are made with some ulterior motive and not merely in order that the chemist may exercise his ingenuity, his manipulative skill or his chemical knowledge, nor even in order that he may produce a series of figures for contemplation in his spare moments or in his old age. In the case of antique metal objects the motives are first, to identify the metal and second, to know the nature and extent of the impurities present, as these may throw light on the locality where the ore or metal originated and the mode in which these have been treated. These points may usefully be considered with respect to specific cases.

COPPER.—Copper was used in Egypt in Predynastic times, that is, before 3400 B.C., at first for ornaments and small objects and later for weapons and tools, and it would be of value to know definitely where this copper came from. It is well known that ores of copper occur in the eastern desert of Egypt, between the Nile and the Red Sea, and also in Sinai, and it is also known that both these sources were exploited at a very early period, the Sinai mines certainly dating from the First Dynasty, that is, before 3000 B.C., and being used until the Twentieth Dynasty (1200 to 1090 B.C.); but about the mines in the eastern desert little is known beyond the fact that they show signs of ancient working. It is possible that the ores from the two localities might have notable differences of composition in respect of the nature of the impurities present, but, unfortunately, detailed analyses of these ores have never been made. It is known, too, that from the Eighteenth Dynasty onwards copper or copper ore was imported into Egypt, especially from Syria and Cyprus, and it might help to date an object if such copper could be differentiated from Egyptian copper.

A few ancient Egyptian copper objects have been found on analysis to contain a high proportion of arsenic, namely, 2·3, 3·9 and 5·6 per cent., respectively, and in a few instances bismuth has been found, varying up to about one per cent. Those who maintain that the Egyptians must have had some mysterious process for hardening copper, the secret of which has been lost, or otherwise they would not have been able to work the hard stones they certainly did work, see in the arsenic and bismuth referred to a confirmation of their argument, but analysis of the ores used would probably show these to be highly arsenical and in some cases to contain bismuth, and thus the arsenic and bismuth would be accounted for naturally. Moreover, although arsenic in the form of sulphide was used as a pigment, it was only at a comparatively late date, and there is no evidence that compounds of bismuth were ever known.

Owing to the lack of a large series of chemical analyses of metal objects from the earliest periods, there is no certainty about the date when bronze was first employed in Egypt; it was, however, known and used in the Twelfth Dynasty (about 2000 to 1788 B.C.), but whether earlier or not is doubtful.

BRONZE.—Bronze certainly did not originate in Egypt, as tin ores do not occur in the country, and, therefore, at first it must have been imported, but it is always assumed that it was subsequently made in the country from native copper and imported tin, and, although very probable, there is no proof of this. Chemical analysis, however, might furnish such proof. The earliest references to tin in the

ancient records and the earliest objects made of tin both date from the Eighteenth Dynasty, which would suggest that tin was not known in Egypt before that time. We are ignorant, too, of where and how bronze was first made, though this must have been in some locality where copper ores and tin ores occurred in close proximity to one another or where a combined copper and tin ore occurred, an accidental mixing and smelting of the former or an accidental smelting of the latter, producing bronze, and it is reasonable to suppose, if such an accident happened several times, that the superior qualities of the resulting metal compared with copper, namely, the lower melting point and the greater hardness, would not escape notice, and that the operation would then be deliberately repeated. But which of the two possibilities actually happened? It seems improbable that the combined ore of copper and tin, namely, stannite or tin pyrites, was used; first, because it occurs only in small quantity and in a few localities; second, because the principal and most important ore of tin is the oxide, cassiterite or tinstone, which does not contain copper; third, because if a combined ore had ever been employed, its use would have continued for at least some time, and in the then very elementary and purely empirical condition of metallurgy it could never have led to the use of the artificial mixture of the two ores, of the employment of which at a later period there seems no doubt, as there would have been no knowledge of the connecting link between the two; and fourth, because from the combined ore it is not likely that tin would have been produced, as it certainly was produced, probably not so early as bronze, but certainly later. The small proportion of sulphur present in the bronze is also an argument against the use of a sulphide ore. A two-fold discovery of bronze, once from the naturally-combined ore of copper and tin and once from an artificially-made mixture of the separate ores is most improbable.

GOLD.—Gold was used in Egypt as far back as Predynastic times, that is, before 3400 B.C., and, as the metal occurs abundantly in the country, both in the alluvial form and in quartz rock, there can be little doubt that the early gold was obtained locally. At a later date, however, gold was brought into Egypt from abroad, both as tribute and as one of the spoils of war, and it might help to date an object if this imported gold could be differentiated from native Egyptian gold, and manifestly the only way of doing this would be by chemical analysis. It might be found, however, that in some cases the gold was Egyptian in origin, and had merely returned from abroad in the shape of worked objects. One well-known Egyptologist has already attempted to distinguish foreign gold from Egyptian gold in this manner, but, as his data were incomplete, his conclusions were wrong. Thus he has stated that certain gold from Egyptian tombs must be of Asiatic origin, because it contains a considerable proportion of silver, being evidently unaware that practically all gold when found contains silver, and that Egyptian gold often contains more than 20 per cent. of silver.

Another question to be settled by chemical analysis is whether the ancient Egyptians refined gold, and, if so, at what date, and also at what period did they begin to debase gold. From the few analyses available it would appear as if gold were not refined, and yet the ancient records mention fine gold, "gold of two times"

and "gold of three times," which suggest refining. There is no doubt, however, that gold was debased, as the proportion of copper it sometimes contains is too high to be natural.

SILVER.—With regard to silver the important question is: whence came the early silver used in Egypt? This metal was employed in small quantity from Pre-dynastic times, but it was not until the Eighteenth Dynasty that it was much used, and it was not until Ptolemaic and Roman times that it became plentiful. At these later periods, however, it was certainly imported.

So far as is known, silver ores do not occur in Egypt, though silver is found in all Egyptian gold, and the Egyptian lead and nickel ores contain a very small proportion of silver. Only a few analyses of ancient Egyptian silver objects are recorded, and these show gold to be present in proportions ranging from 3·2 to 14·9 per cent.; and, since it is highly improbable that the metallurgical knowledge of the ancient Egyptians was sufficiently advanced at the early date at which they first used silver to enable them to separate this metal from gold, even imperfectly, and still more improbable that they even knew of the presence of silver in the nickel and lead ores, much less that they could separate it, a possible explanation of the early silver is that it was gold so rich in silver as to have a white colour; and, although no such gold is known in the country at the present time, it is suggested that it may have occurred, and may have been worked out, or may still occur and may have escaped notice in modern times. Such a metal would indeed be "white gold," which is the meaning of the ancient Egyptian word for silver. In this connection may be mentioned the light yellow-coloured natural alloy of gold and silver that occurs in Egypt, and was used anciently; it was called *electron* by the Greeks and *electrum* by the Romans on account of its resemblance in colour to amber, the Greek name for which was *electron*; *electrum* being the term now used. According to analyses of electrum that have been made, the proportion of gold varied from 72·1 to 85·9 per cent., and that of silver from 11·3 to 22·3 per cent. Electrum may be regarded as a half-way stage between gold and silver, and its occurrence in Egypt increases the probability of the occurrence of an alloy still richer in silver, which could be called "white gold."

MORTAR AND PLASTER.—Both the mortar and plaster of the ancient Egyptians were one of two materials, namely, either gypsum or clay, and, so far as can be ascertained, lime in any form or for any purpose was unknown in Egypt until the time of the Roman occupation. This was probably due, first to the scarcity of fuel in the country, since limestone is even more abundant than gypsum and more easily accessible; and, second, to the dry climate which permitted the use of gypsum for outdoor use. The scarcity of fuel naturally led to its economical use, and the much lower temperature required to burn gypsum, compared with that required for lime, namely, from 100° C. to 200° C. (generally about 130° C.) in the first case, and about 900° C. in the second case, makes a considerable difference in the amount of fuel used.

There is, of course, no difficulty in the chemical analysis of mortar or plaster, but serious errors have been made both by chemists and archaeologists in their

interpretation of the analyses, with the result that the literature of archaeology is full of mistakes about the composition of these materials. This arises from the presence of calcium carbonate and of siliceous sand, which are usually attributed to artificial admixture with lime that has become carbonated and with sand respectively. Calcium carbonate and sand, however, are natural impurities, both in Egyptian gypsum and in clay, and are not artificial additions, and they therefore occur in the mortar and plaster made from these materials.

One interesting point about ancient gypsum mortar and plaster is the pink colour which they sometimes assume. This is purely a surface coloration, and appears to be due to an iron compound, though it is not the red of ferric oxide, but a decided pink. A similar colour is also sometimes found on limestone.

OILS AND FATS.—Although fatty matter has frequently been found in Egyptian tombs, it has seldom been analysed, and, of the few analyses made, none is conclusive. This is almost inevitable, as oils and fats, unless kept under special airtight and sterile conditions, which is not the case when placed in jars in tombs, sooner or later decompose, and as some of the bodies formed escape, either by evaporation or by soaking into the material of the containing vessel, all that the analyst has for examination is merely a portion of the products of decomposition. What is left consists of a mixture of fatty acids, and it is only by the separation, purification and identification of these and by a determination of the proportion in which each occurs in the mixture that the nature of the original oil or fat can be known, and, since what remains is generally a portion of that formed and not necessarily a representative portion, the problem may often be insoluble.

The fatty matter often smells like rancid coconut oil or like rancid butter and, occasionally, of valeric acid, and the fatty acids identifiable are generally palmitic and stearic acids and, less frequently, oleic acid, and in one instance calcium butyrate was found. When the fatty acid consists essentially of stearic acid alone it seems justifiable to assume that the original material has been castor oil, which was well known to the ancient Egyptians, and which is one of the few oils or fats that contain little or no palmitic acid. When the fatty acids consist of stearic and palmitic acids in almost equal proportions it is possible that the original material was an animal fat, such as ox fat. There is, however, as will be realised, considerable scope for further chemical work on the subject.

PIGMENTS AND VARNISH.—The ancient Egyptian pigments have often been analysed, and their nature is well known; most of them are naturally-occurring mineral substances.

The white is generally calcium carbonate, but occasionally calcium sulphate. The black is carbon, being sometimes soot and sometimes a coarser material, possibly powdered charcoal. The black pigment is very prone to disappear, more so than any other pigment, but the reason for this has not been investigated; it cannot, however, be due to fading. The grey is a mixture of black and white. The red is red ochre, sometimes natural and sometimes produced by calcining yellow ochre. In Roman times, however, both red lead and madder on a base of gypsum were also used. The browns are all natural iron oxides. There are two

yellows, the common one (that employed at the earliest date) being yellow ochre, and the other being sulphide of arsenic (orpiment). The earliest blue was azurite, a naturally-occurring basic carbonate of copper, but the principal blue was a most remarkable artificial compound consisting of a crystalline copper-lime-silicate in the condition of a frit. The composition of this has been investigated by many chemists, beginning with Sir Humphrey Davy in 1815, and both Dr. Russell and Messrs. Laurie, McLintock and Miles have not only analysed the frit, but have also reproduced it. Another blue recorded as having been used is cobalt, but this I have never found myself. There is, however, need for further systematic work to ascertain at what periods the various blues were introduced. The earliest green pigment was powdered malachite, a basic carbonate of copper that occurs abundantly in the country, but at a later date a green frit analogous to the blue frit just described was used instead.

One interesting fact about which there seems no doubt, although I have not been able to verify it personally, is that in some few instances a blue pigment has corroded the gypsum plaster on which it was painted, and in one case a pigment, that was probably blue, has corroded wood in such a manner that what were painted inscriptions are now merely a series of holes in the wood, looking almost as though they had been burned. So far, I have not been able to find any satisfactory explanation for these phenomena.

Although the ancient pigments are well known, the nature of the medium or vehicle with which they were employed has never been satisfactorily established. There is no evidence whatever of the use of oil, even as late as the Graeco-Roman period, at which date painting was done by the encaustic process, that is, by mixing the pigments with melted beeswax, and it seems most probable, and is generally accepted, that the medium was water mixed with some binding material, such as gum or glue, both of which were well known at the time, and the best chance of determining which was employed would be by the analysis of paint mixed ready for use, if this could be found. There should be no difficulty in detecting either gum or glue, if present. The use of white of egg seems improbable, since the domestic fowl, and hence the ordinary egg, is not indigenous to Egypt, but was introduced into the country at a late date. Tests for albumin, too, would probably be useless, as this is likely to have become denaturalised. Glue has been reported as having been found on painted surfaces on several occasions, but this is no proof that it was mixed with the paint, and it may well have been used to prepare the surface for painting in the manner of modern size. Two kinds of varnish were largely employed in ancient Egypt, one now yellow or reddish, but originally colourless or practically colourless, which was used to cover painting on wood, and, very occasionally, painting on plaster, and the other black, used exclusively for coating certain wooden funerary objects without any painting below. Both these varnishes consist of resin, and the problem concerning them is the manner in which they were applied. In present-day varnishes the resin is dissolved in boiled linseed oil, turpentine, alcohol or other solvent, but it is believed that all such solvents were unknown in ancient Egypt. There is no evidence, either, of

the presence of oil, and the varnish is insoluble in turpentine, though freely soluble in alcohol. Strong wine has been suggested as a possible solvent, but even in the strongest wine resin is not sufficiently soluble to produce a varnish. The alternative, therefore, seems to be a resin that did not require an extraneous solvent, which means a naturally-occurring resin already in the liquid form, such, for example, as an oleo-resin. Oleo-resins, however, although nominally liquid, are, at the best, of a thick syrupy consistence too viscous to admit of application as a thin coating, though this viscosity may be reduced by warming. A natural oleo-resin applied warm, therefore, seems a possible explanation. Another suggestion that has been made is that the resin was applied in a finely-powdered condition, and was then liquified by means of heat, but this does not appear very feasible.

POTTERY.—By pottery is meant ware made from clay, moulded into shape while wet, and then hardened by being baked.

The chemical analysis of pottery is practically never required by the archaeologist, but there is one interesting problem in connection with ancient Egyptian pottery that has not yet been satisfactorily solved, namely, the nature of the black colour of the Predynastic black and black-topped pots. This black colour, which penetrates throughout the thickness of the ware, has been accounted for in three different ways; first, by the action of smoke from the fire used in baking the pots or of some volatile organic matter that would carbonise, such as tarry matter from the fuel; second, by the production of black oxide of iron by limiting the air supply during baking by covering with ashes that part of the pot intended to be black; and, third, by the application of a black colour to the pot. The first is the usual explanation, and is supported by a certain amount of evidence, both chemical and experimental. The second solution is very attractive from a chemical point of view, and a production of black oxide by using a limited air supply is theoretically possible, though at present it lacks proof. The third method has actually been proved to have been used for certain types of pottery, the black colour being produced, though only that on the outside, by the use of graphite. The whole subject needs further work, both chemical and experimental, before it can be definitely settled.

RESINOUS MATERIALS.—Although Egypt is not now a resin-producing country, and probably never was, yet resin and resinous materials have always been largely employed. An early use of resin was in connection with burial, and lumps of this material have often been found in graves of the Archaic period. At a later date both resins and gum-resins were employed in the process of mummification, and the latter also largely as incense. Resin too was used as an adhesive, both by itself and also mixed with whiting, and its use for varnish has already been mentioned. Occasionally, too, resin was fashioned into small objects, such as beads and scarabs, and even the pupils of the eyes in statues were sometimes made of resin.

In the literature of Egyptology there are many precise statements regarding the nature of the resins used in ancient Egypt, very few of which rest on the certain knowledge that can only come from systematic chemical examination, and

most of which are merely guesswork, since the nature of these resins has been very little investigated and very few of them having been scientifically identified. This is largely due to the fact that many of them are probably of kinds that are not only not used, but are not even known, at the present day.

One analyst, who has examined these resinous materials, identifies a large number of different resins in each specimen; thus, in one sample, storax, mastic and Aleppo resin are all positively identified, and two other unknown resins are stated to be present, whilst Chios turpentine and cedar resin are also thought to be probably present; in another specimen of material, storax, three different unknown resins, a gum or gum-resin, and a balsam, were all found. This is quite contrary to my experience, and, excluding incense, which is probably a mixture, the resins are usually homogeneous bodies of well defined character, and where there has been any admixture it has been with natron or fatty matter. This analyst by well-known chemical means, such as solution and precipitation, separated certain bodies, of which he made an ultimate analysis, determining the carbon and hydrogen directly and the oxygen by difference. When, however, the smallness of the sample taken for analysis (from 0.02 to 0.22 grm.), the multiplication and division of the original figures necessitated by the calculations, and the fact, to take one example, that 77.4 per cent. of carbon and 10.4 per cent. of hydrogen represent one substance, and 77.3 per cent. of carbon and 10.2 per cent. of hydrogen a totally different substance, one may be pardoned for thinking there is room for a mistake in identification. In several instances, too, this analyst bases a probable identification upon the smell of the substance or upon a process of exclusion, assuming that, because negative results were obtained when certain specific resins were tested for and hence were probably absent, that therefore another resin, that it was thought might have been used, was probably present.

The resins used may be divided into true resins and gum-resins. Among the former is a resin from some species of pine that has a slight fragrant smell and gives the Liebermann-Storch colour reaction, and among the latter is myrrh. This is about all that can be definitely stated, and there is, therefore, need for much work on the subject.

USE OF BITUMEN.—Certain classical and early Arab writers state that bitumen of Judaea was used by the Egyptians in embalming, and most Egyptologists and modern writers on mummification have followed this old tradition, and, without any evidence, save that many mummies are black, definitely state that bitumen or mineral pitch was used. Although the use of bitumen about the Ptolemaic period is probable, no definite evidence of it has yet been found. The analyst previously mentioned states that he found bitumen in all the specimens of mummy material he examined, but his identification is based on the fact that a tiny residue of black material obtained in the course of the analysis smelled somewhat like bitumen and contained sulphur. This same analyst, however, finds bitumen in ancient Egyptian perfumes. The use of a very small proportion of bitumen with a complex mixture of different resins or its use in perfumes seems most improbable. Although mineral pitch has not been found, wood pitch has been identified on several

occasions. It may be mentioned that these ancient resins frequently become dark brown or black, and that mummy flesh becomes dark brown and very resinous-like and behaves very like resin when treated with solvents.

COLOUR OF MUMMY HAIR.—In connection with mummies the colour of mummy hair may be mentioned. Hair is very resistant to decomposition, but it is generally stated that the colour is not so resistant and even that all hair eventually becomes red, owing to the disappearance of the colouring matter originally present, and the oxidation of the iron compounds in the hair. As an example of this colour change, Petrarch is cited. Petrarch died at the age of seventy, but from the age of twenty-five his hair was white, and it is recorded that many years after his death his coffin was broken open by robbers in the hope of finding treasure, and that the hair was then red. In the case of Egyptian mummies I have been unable to find any evidence of any change to red, though in a few instances the hair is flaxen and suggests *post mortem* bleaching due to the natron bath and in one case the hair had been dyed red, probably by the use of henna.

STONE.—The chief purpose to be served by the examination of stone is to identify it in order that it may be correctly described. This is essentially a matter for the petrologist or for the petrologist and chemist working together, but in the absence of a petrologist or geologist the chemist is often called upon to identify stone. It is very seldom that a chemical analysis of stone is necessary, the only examination usually required being a critical inspection, both with the naked eye and with a lens, an approximative determination of the hardness by ascertaining whether it can be scratched with a knife and whether it will scratch glass, sometimes the determination of the specific gravity, and in cases of special difficulty the microscopic examination of the powdered material or better, of a thin section, and, above all, the comparison of the specimen with one of which the identity is well established. Occasionally a drop of acid may be required to confirm, say, the distinction between calcite and gypsum.

As an example of the confusion existing in the matter of the nomenclature of stones, it may be mentioned that there is still discussion whether the material always called "alabaster" in Egyptology, and which chemically is calcium carbonate, is calcite or aragonite. Here the most useful test is the determination of the specific gravity, that of calcite being 2.7, and that of aragonite 2.9.

Other mistakes frequently made are in the recognition of the various green stones used in ancient Egypt, among which are malachite, beryl, green felspar, green jasper, green quartz, and green chalcedony, and the confusion in the literature is considerable. The archaeologist, too, is inclined to call all dark-grey or black stone, if of coarse texture, granite, and, if of fine texture, basalt; and, although basalt was used, much of what is called basalt is a fine-grained crystalline schist.

TEXTILE FABRICS.—There are many points of interest in connection with ancient Egyptian textile fabrics, which for burial purposes were always linen, namely, the method of retting and preparing the flax, as also the methods of weaving and dyeing, but I propose to confine myself to one aspect, *i.e.* the nature

and cause of the disintegration these fabrics have often undergone during their prolonged sojourn in the tombs.

In some cases old Egyptian linen is in as good condition when found as when it was made, whilst in other instances it is discoloured and rotted, and may even be reduced to the condition of a black powder. The date of the burial apparently is not a factor in the problem, and light, which is known to rot textile fabrics, may also be eliminated from the question, as none of the fabrics were exposed to light while in the tomb. The determining factors are damp and a certain amount of air, and it is suggested that the damage is caused largely or wholly by fungi, which induce a sort of slow spontaneous combustion.

DISCUSSION.

The PRESIDENT, in thanking the author for his most interesting paper, said that he did not know whether he ought to feel sad or glad at hearing that chemists were now being consulted, as he felt that they should have been consulted long ago. It was, however, a happy chance that Mr. Lucas should have been the man on the spot when a chemist was eventually consulted. He enquired whether there was any outstanding example of a disclosure of a method of doing something we did not know before; one was accustomed to hear much of the Egyptians' inimitable ingenuity and of their lost arts, yet Mr. Lucas seemed to indicate that there was nothing really inimitable after all.

He was interested in the remarks about the condition of the fats found in the tombs, and he suggested that the analytical figures might be compared with those on record in *THE ANALYST* (1907, 32, 89) for Irish bog butter. It was the custom in Ireland to preserve butter by burying it in the bog (peat), and specimens, which had been found after many years, had been analysed, and had afforded interesting information as to the decomposition of the fat.

Miss M. A. MURRAY explained that she was speaking as an Egyptologist and not as a chemist. She had been trained by Prof. Flinders Petrie, who was the first to realise the importance of chemical analysis in connection with Egyptology. She, herself, had had many analyses made, and regarded them as valuable.

Following Professor Petrie's system of dating, she contended that flint implements had been discovered which were in existence before 5000 B.C. Experiments had been made with a view to assessing the length of time required for patination, but so far without success. It would first be necessary, she thought, to arrive at the approximate date of the implements. As there was now some possibility of determining the date of the later palaeolithic implements, she hoped that in due course the earlier palaeolithic specimens would also be dated, and that, ultimately, useful information would be obtained with regard to patination.

Reviewing the various classes of substances mentioned by the author, Miss Murray pointed out that the Ashmolean Museum was in possession of a glass bead of the first dynasty, which Prof. Sir Henry Miers had identified by a specific gravity test; also an inlay of glass on a piece of ebony.

Coloured glazes were not in general use until the end of the eighteenth dynasty, after the Syrian conquest; though before that time all shades of blue and green had been used, together with a little black (manganese).

Speaking of metals, Miss Murray said that the early Egyptians used malachite for medical purposes—covering the under eyelid with green paint which protected the eye from the white glare off the ground; malachite was imported, as it did not

occur in the Nile valley. There was a "pilgrim bottle" from Egypt in the Ashmolean Museum made of tin. An analysis of the gold leaf covering of a coffin of the twelfth dynasty revealed that it contained 97.5 per cent. of gold.

Castor oil was common in Egypt, and was probably used for lamps, as it has been so used in India till recent times. Giving a white light, without smoke, it was used for lighting up the tombs.

With regard to perfumes, she suggested that the scent was extracted with dilute alcohol, by putting the scent-bearing leaves in fat and adding wine.

The possibility of the use of egg albumin for the fixation of pigments had been suggested, but there were no fowls in ancient Egypt, nor did they arrive in the West until the days of Alexander the Great, who introduced them from India.

What strength of alcohol was required to dissolve resin? Evidence that the ancient Egyptians distilled alcohol was supplied by the representations of stills. A resin had been found which was described as amber; how did it compare with a genuine sample?

With regard to minerals, it was known that salt and quicklime, with a little alum, were in use for mummification in the twelfth dynasty.

Pottery of the prehistoric periods was often covered with a wash of ground haematite, and then fired. Often the vases were placed mouth downwards in the ashes on the floor of the kiln, thus excluding the oxygen during the firing. On the parts so covered the haematite became black, *i.e.* formation of oxide (?).

All the Egyptian amulets in the Ashmolean Museum at Oxford were identified as to material by Sir Henry Miers by their specific gravity and microscopical appearance.

Dr. PLENDERLEITH considered that the brown colour of flint implements was due to oxide of iron. He referred to samples of pre-Stonehenge implements which, although purple in the centre, had turned white on the outside. Their age was unknown, nor was it exactly known why the colour had changed; yet the effect of caustic soda on the blue was to turn it white, ammonia having no effect. Had the author come across white patination in the Egyptian world?

It had been suggested that faience was sometimes made from glazed sandstone rather than from free sand when sand was employed. Another suggestion was that it was held together by some such substance as sodium silicate, but, personally, he attached more importance to the disc of carbon that had been found in the core.

With regard to fats, he mentioned that Mr. Chaston Chapman and he had been examining a cosmetic found in the tomb of Tutankhamen, which had a coconut aroma and consisted largely of saturated fatty acids. They had been unable to identify the sweet smell, which reminded them of "open broom blossom in the sunlight." Vegetable fibre had been found in one fat recently analysed.

Mr. A. CHASTON CHAPMAN said that the Tutankhamen cosmetic appeared to resemble very closely in appearance the specimen which the author had exhibited. It contained chiefly stearic and palmitic acids, together with a smaller quantity of some lower saturated fatty acids (chiefly myristic) and about 30 per cent. of the so-called "oxidised" acids. An interesting point was that after so long a period of time 6 per cent. of the glycerin should have remained undecomposed. The reason, of course, was that the fat was sterile, and there was not any evidence of bacterial decomposition. Sterols were absent, but, so far as could be stated, the cosmetic appeared to consist of about 90 per cent. of some animal fat with about 10 per cent. of a fragrant resin or balsam. The coconut smell was almost certainly due to the oxidation products formed during the process of time, and did not appear to have any connection with coconut oil.

Dr. H. P. STEVENS asked whether the fibre used was entirely linen. Also, did the author suggest that modern glass free from manganese would change colour in the sun, or did he imply that the darkening was due to the presence of traces of this metal? The carbon core in faience, he suggested, might be due to the fact that the original wooden centre had been covered with an alkaline copper salt, which, by solvent action, had brought about decomposition of the wood.

Mr. C. A. MITCHELL referred to some sequins of red gold which the author had sent to him for examination. The bright red surface deposit on these contained traces of iron, and he asked whether there was any evidence to show that the colour had been intentionally produced with the aid of an iron compound, or whether it was accidental.

He also mentioned a piece of graphite given to him by Sir Flinders Petrie. This dated back to about 1500 B.C. (*cf.* ANALYST, 1922, 47, 380), and he asked the author whether any other discovery of ancient graphite had been made in Egypt.

Dr. J. J. FOX stated that the colour of a specimen of black pottery examined by him was due, not to carbon, but to the reduction of oxide of iron to a lower oxide. Speaking on the disintegration of fats, he referred to a specimen of beeswax, 600 years old, the constants of which, even after long exposure in a museum, had remained normal.

Mr. HARDING asked whether there was any indication of the use of lead in glazes prior to Roman times; and whether antimony was responsible for any of the yellow tints.

Mr. LUCAS, replying, said that he was acquainted with the inlay mentioned by Miss Murray, but that, although manifestly a vitreous material, it was not necessarily glass, and might conceivably have been faience, glazed on one side only, from which the siliceous body material had been removed or had fallen away. Malachite occurred in Egypt, both in Sinai and in the Eastern desert. Referring to castor oil, he mentioned that the oil described by Herodotus as being used for temple lamps was apparently castor oil, to which salt was added to prevent smoking.

Both beer and wine were well known in ancient Egypt. The beer was possibly very similar to the *booza* used at the present day, both in the Sudan and in Egypt, among the Nubians. It resembled thin gruel in appearance, and generally contained about 8 per cent. of absolute alcohol. It had not been proved that the Egyptians of the eighteenth dynasty separated alcohol by distillation from either beer or wine.

There was no evidence that amber was known in ancient Egypt until a late period, the material so frequently termed amber being an entirely different resin. Although aluminium compounds and calcium carbonate had been found associated with mummies, this in no way proved that alum and quick lime were used in mummification. The aluminium compound was not in the form of sulphate, and other compounds, *e.g.* the silicate, were widely distributed. Calcium carbonate, too, occurred extensively in Egypt, and the mummy referred to came from a limestone district, and both during mummification and burial and particularly during exhumation, it would have been difficult to avoid contamination with calcium carbonate. With regard to myrrh, this material was certainly employed in ancient Egypt, possibly, as suggested, for making incense; it was undoubtedly used in mummification.

The fibres used by the ancient Egyptians included wool as well as linen, but wool was not used for burial purposes before Christian times; ramie fibre had also been reported on one occasion. Silk and cotton were not used until a very late period.

So far as was known, lead glaze was not used in Egypt before Arab times. The formation of the amethyst colour only occurred in glasses containing manganese.

Referring to Mr. Mitchell's observations, Mr. Lucas said that gold from Egyptian tombs was not always yellow in colour, but often various shades of red, due sometimes to chemical changes in the traces of impurities in the gold and sometimes to organic matter on the surface. The very remarkable pink colour referred to, however, was very different; it was intentional and not accidental, and, so far as had been ascertained, seemed to be due to a trace of iron, and might possibly have been produced by the use of an iron compound with subsequent heating.

The lump of graphite analysed by Mr. Mitchell was the only piece that had yet been found in ancient Egyptian graves, though graphite had been found in graves in the Sudan. Small amounts of graphite occurred in the gold-bearing quartz of Nubia, but it was otherwise unknown in Egypt.

The Determination of Potassium in the Presence and Absence of Sulphates.

BY M. A. HAMID, Ph.D.

THE perchloric acid method for the determination of potassium having failed in the presence of sulphates, a need arises for a general and satisfactory method for the quantitative determination of potassium.

Streker and Jungck (*Z. anal. Chem.*, 1923, **63**, 161-180) have compared a number of methods for the determination of potassium both gravimetrically and volumetrically. They state that the precipitation of potassium by means of sodium cobaltinitrite does not lead to concordant results, but that the whole of the potassium is precipitated in this way, and can then be subsequently determined by the perchlorate method.

In the following work a number of determinations of potassium have been made both in the presence and absence of sulphates, and the results have been compared with those obtained by other methods. That the precipitate is not potassium cobaltinitrite, $K_3Co(NO_2)_6$, as Streker and Jungck suppose it to be, but the di-potassium mono-sodium cobaltinitrite mono-hydrate, $K_2NaCo(NO_2)_6 \cdot H_2O$, has been confirmed by a number of experiments.

The failure of Streker and Jungck to get concordant results is therefore undoubtedly due to this error. The precipitate is neither potassium cobaltinitrite nor the di-potassium mono-sodium salt, as these authors describe it, but the mono-hydrate of the latter salt. By assigning the formula $K_2NaCo(NO_2)_6 \cdot H_2O$ to the dried precipitate very satisfactory results are obtained, and the method seems to be very useful.

DETERMINATION OF POTASSIUM BY THE COBALTINITRITE METHOD.—The amount of potassium in a given sample can be determined by this method either gravimetrically or volumetrically. The potassium ion in the solution is first precipitated, under certain conditions to be described presently, by means of sodium cobaltinitrite. The precipitate is then either dried and weighed as such, or its amount determined volumetrically by titrating the nitrite groups with standard permanganate solution. In the present determinations only the first of these methods has been used.

PREPARATION OF SODIUM COBALTINITRITE REAGENT.—Sodium nitrite (220 grms.) was dissolved in 400 c.c. of water, and 113 grms. of cobalt acetate were dissolved in 300 c.c. of water and 100 c.c. of glacial acetic acid. The two solutions were carefully mixed and gently warmed. The nitrogen peroxide which was evolved was removed by evacuation with a pump, and the solution left for a day. A slight yellow precipitate, due to the presence of potassium as an impurity in the sodium nitrite employed, was removed by filtration, and the solution made up to a litre and kept in a dark place.

DETERMINATION OF POTASSIUM IN THE ABSENCE OF SULPHATES.—Pure recrystallised potassium nitrate (0.9439 gm.) was dissolved in water, and the solution made up to 100 c.c. Ten c.c. of this solution were acidified with a drop or two of concentrated acetic acid, then treated with an excess of sodium cobaltinitrite reagent, and the whole evaporated to dryness on a water bath. The residue was washed by decantation with 5 per cent. acetic acid, and then with cold water, and the precipitate transferred to a weighed Gooch crucible (previously dried at 100° C.), washed with cold water till free from the acid, and dried at 100° C. to a constant weight.

DETERMINATION OF POTASSIUM BY THE PERCHLORIC ACID METHOD.—Ten c.c. of the above solution were evaporated down with 1 c.c. of 20 per cent. perchloric acid. About 10 c.c. of hot water and a little more perchloric acid were added to it, and the evaporation repeated till heavy fumes of perchloric acid were given off. After cooling, the residue was treated with about 20 c.c. of 0.2 per cent. perchloric acid in 97 per cent. alcohol. A special wash bottle, with a very fine jet, was made out of a test tube, and the precipitate washed once by decantation with the washing solution and transferred to a Gooch crucible, previously dried at 130° C. The precipitate was washed thoroughly with the washing solution, the Gooch crucible and its contents dried at 130° C. and weighed till constant.

The results obtained by each of these methods were as follows:

		Weight of Potassium nitrate calculated by	
		Cobaltinitrite method.	Perchloric Acid method.
		Grm.	Grm.
1.		0.9423	0.9445
2.		0.9458	0.9437
3.		0.9428	—
4.		0.9441	—
	Mean =	0.9438	0.9441

In another sample the potassium was determined both by the cobaltinitrite and the perchloric acid methods, and also by conversion into the sulphate. The results are given below in terms of potassium oxide:

	Cobaltinitrite method K ₂ O per cent.	Perchlorate method K ₂ O per cent.	Weighed as K ₂ SO ₄ K ₂ O per cent.
1.	38.67	38.71	38.69
2.	38.68	38.69	38.65
3.	38.70	—	—
Mean =	38.68	38.70	38.67

DETERMINATION OF POTASSIUM IN THE PRESENCE OF SULPHATES.—It is a well-known fact that the perchloric acid method breaks down when there are sulphates present in the solution. In order to see whether the cobaltinitrite method could be used in the presence of sulphates, determinations of potassium were made with different quantities of "pure reagent" potassium sulphate. Analyses of the sample of potassium sulphate used were made by determining the sulphate as well as the potassium ion. The potassium ion was determined by the cobaltinitrite method, and also by conversion into the chloride. The sulphate was precipitated as barium sulphate. The precipitate was filtered off, and the filtrate evaporated to a small bulk. The excess of barium in the filtrate was precipitated as barium carbonate by means of ammonium carbonate and removed by filtration. The second filtrate was evaporated to dryness with a small quantity of hydrochloric acid, the ammonium salts expelled by ignition, and the residue weighed as chloride.

	Weight of potassium sulphate		
	Taken Grm.	Determined by	
		Cobaltinitrite method. Grm.	Conversion into the chloride. Grm.
1.	0.2187	0.2182	0.2179
2.	0.1062	0.1068	0.1059
3.	0.1104	0.1106	0.1100
4.	0.0471	0.0463	0.0466
5.	0.0856	0.0859	0.0860
6.	0.1236	0.1232	0.1228

With another sample the following results were obtained:

Theoretical K ₂ O Per Cent.		Calculated from the weight of K ₂ NaCo(NO ₂) ₆ ·H ₂ O Per Cent.
44.87	1.	44.82
	2.	44.85
	3.	44.79
	Mean =	44.82

The above figures serve to demonstrate that the cobaltinitrite method works very satisfactorily both in the presence and absence of sulphates. Several determinations of potassium were made by the method in mixtures of sodium sulphate,

potassium nitrate, potassium sulphate, and sodium nitrate, and the results agreed satisfactorily with the theoretical amounts. This is clearly an advantage over the perchlorate method which fails when there are sulphates present in the solution. There seems to be no reason, therefore, why it should not be adopted as a general method for the quantitative determination of potassium.

SUMMARY.—The cobaltinitrite method for the quantitative determination of potassium has been described and the results compared with those obtained by other methods. It has been shown that this method works very satisfactorily, both in the presence and absence of sulphates, and it is recommended for general use.

The author is indebted to Professor F. G. Donnan for the suggestion and details of this method.

NOTE.—The method described by Dr. Hamid has been in constant use in my laboratory since 1920. It was communicated to me privately in 1919 by Mr. Mellott, who was at that time Chief Chemist at the Pena Grande Oficina of the Du Pont Nitrate Company (Chile). As I do not think the value of the method is sufficiently appreciated in many laboratories, I hope the check analyses given by Dr. Hamid will prove of some service.

F. G. DONNAN.

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The Miscometer: An Apparatus for Obtaining Composite Samples.

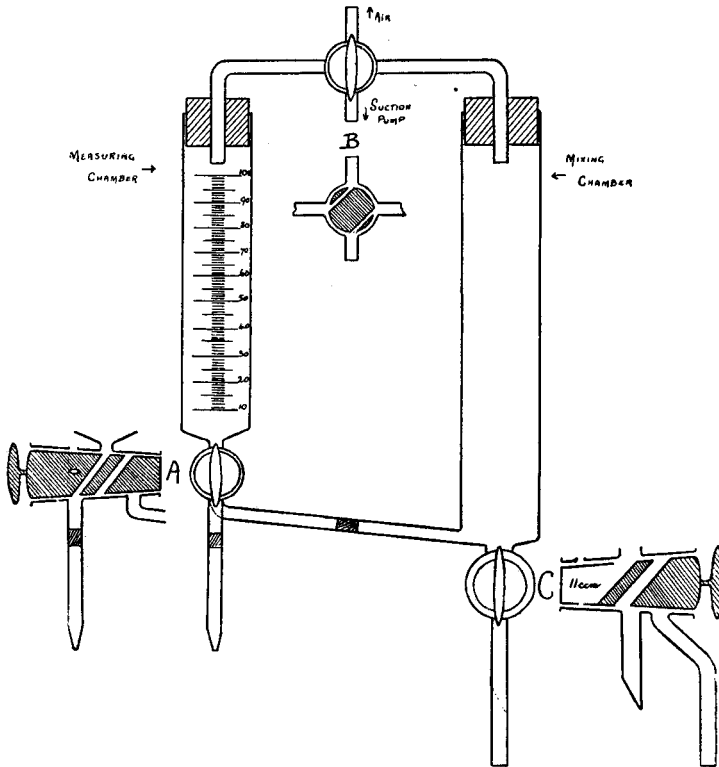
By JOHN HOUSTON, M.Sc.

ALTHOUGH primarily designed for milk samples, this apparatus may be used to obtain a composite sample of any two (or more) miscible liquids. In addition, it measures off a given quantity of the composite sample and delivers the remainder to a suitable receptacle.

CONSTRUCTION.—The apparatus consists essentially of two cylindrical glass chambers (one of which is graduated), fixed to a stand (see Figure). The cylinders are connected at their upper ends, through a four-way stopcock, with any device which will create a partial vacuum. For obtaining composite milk samples, an ordinary laboratory tap pump answers this purpose. The cylinders are connected at their lower ends by a length of glass tubing. At the lower end of the

measuring chamber there is a three-way stopcock, and at the lower end of the mixing chamber there is a specially devised stopcock.

In order that the apparatus may not be rendered wholly useless by the fracture of any small part, the rubber connections shown in the sketch are included. The support for the apparatus consists of a crossbar about 15 in. long, to which are attached two adjustable clamps.



STOPCOCKS.—A. The inlet stopcock, is a three-way one. In one position it connects the measuring chamber and the inlet tube, and in the reverse position it connects the measuring chamber and the mixing chamber. In a position half-way between these two positions the top of the inlet tube is connected with the air, so that the excess left in the inlet tube may flow out.

B. The suction stopcock is a four-way one, which in one position connects the measuring chamber and the suction pump, and at the same time allows air to enter the mixing chamber. When the stopcock is turned through 90° the connections are reversed.

C. This is a specially devised stopcock, so made that, when closed, a measured quantity (11 c.c.) flows into the narrower end. When the stopper is opened the

measured quantity flows into a butyrometer, and at the same time the residue in the mixing chamber flows out by the side tube. This device may be best understood from the sketch.

MODE OF WORKING.—With the suction pump operating and connected with the measuring chamber, and C in a neutral position, the requisite quantities of the samples to be made composite are drawn in turn into the mixing chamber, by opening A, which is so left that the inlet tube drains. B is then turned through 90° and A turned so that it connects the two chambers. The mixture is thus drawn into the mixing chamber and thoroughly mixed by allowing air to bubble through it. A is then closed and C allowed to fill up after which B is turned to its first position and C turned so that the 11 c.c. and the residue of the composite sample flow out at the same time. The process is then repeated as often as required.

ADVANTAGES.—1. The time taken to test a number of samples is reduced by at least 30 per cent. Furthermore, speed and accuracy can be combined, which is impossible in the method of mixing by hand.

2. The pipette is eliminated from milk testing, an important consideration when dealing with preserved or impure samples of milk.

3. Mechanical devices to deliver samples and convey butyrometers to and from the apparatus may be installed.

4. The operator may remain seated while working.

5. The routine testing of composite samples of milk is divided definitely into two parts—a skilled man carries out all the measuring parts, and an unskilled assistant unpacks and shakes the samples, and fills and washes butyrometers.

In the laboratory of this University upwards of 100,000 samples per annum are tested for butter-fat by Gerber's acid method. A miscometer has been installed and is working satisfactorily.

CHEMICAL RESEARCH DIVISION, MINISTRY OF AGRICULTURE,
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THE SEPARATION OF IRIDIUM FROM IRON.

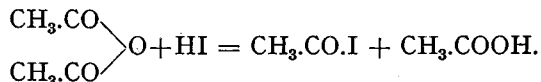
ERRATUM,—On page 395, eleven lines from the bottom, *for* "chloroiridate" *read* "chloroiridite."

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE USE OF ACETIC ANHYDRIDE IN ZEISEL'S METHOD FOR THE DETERMINATION OF METHOXY GROUPS.

It is customary to add acetic anhydride and to re-heat the Zeisel apparatus at intervals in cases where values are obtained which fall below the theoretical. (Compare, for example, Herzig, *Monatsh.*, 1898, **9**, 544; Goldschmiedt, *Ber.*, 1902, **35**, 1728; Perkin, *J. Chem. Soc.*, 1903, **83**, 1370.) In the course of the work on hemlock-tannin (Manning and Nierenstein, *J. Chem. Soc.*, 1919, **115**, 662) use was made of these modifications of Zeisel's method with apparently good results, but at the same time it was noticed that acetic anhydride and hydriodic acid *by themselves* yield weighable quantities of silver iodide when heated in a Zeisel apparatus, which was also independently confirmed in this laboratory by Dr. A. Geake. A note to that effect (Manning and Nierenstein, *Ber.*, 1913, **46**, 3983) was not favourably received by Goldschmiedt (*Ber.*, 1914, **47**, 389) or by Perkin (*J. Chem. Soc.*, 1914, **105**, 2352), who insisted on the reliability of the above-mentioned modifications of Zeisel's method. It must, therefore, be noted that I find that acetic anhydride and hydriodic acid are nearly quantitatively converted into acetyl iodide on standing for 10 to 11 years:



Since acetyl iodide, which yields silver iodide by interaction with silver nitrate, is also produced on heating acetic anhydride with hydriodic acid (Gauthier, *Annalen*, 1857, **103**, 335) and, further, since acetyl iodide boils at 108° C., which is more than 30 degrees below the temperature used in Zeisel's method for the production of the methyl iodide by the interaction of the hydriodic acid with the methoxy derivative, it becomes evident that some acetyl iodide is formed and distilled into the silver nitrate solution when acetic anhydride is used. It is, therefore, not advisable to use acetic anhydride in connection with Zeisel's method, as already pointed out by us in 1913.

M. NIERENSTEIN.

THE UNIVERSITY, BRISTOL.

THE ABSENCE OF MIRROR FORMATION IN VORTMANN'S METHOD OF SEPARATING SILVER AND LEAD.

IN reply to the question which has been put to me, why no silver mirror is formed in the method devised by Hecht and myself for the separation of silver and lead (*ANALYST*, 1926, 158), I may say that such mirror formation has never been observed when the directions given have been accurately followed (and this also applies to the analogous method of separating lead and antimony). It is essential that the liquid should be heated only to 70 to 80° C., and that, after the addition of tartaric acid or ammonium tartrate, only a slight excess of ammonia should be

added. A silver solution can be boiled for a considerable time with even large quantities of tartaric acid and ammonia without yielding a mirror. Too large an excess of ammonia must be avoided, however, since otherwise a small amount of basic lead phosphate will be formed and will interfere with the determination of the lead.

Mirror formation usually occurs when an ammoniacal silver solution containing but little tartaric acid is treated with potassium or sodium hydroxide solution. All the published methods for the production of silver mirrors direct that a large excess of a fixed alkali hydroxide shall be used. Even when Rochelle salt is used instead of tartaric acid in the ammoniacal silver solution there is more or less mirror formation on heating.

G. VORTMANN.

SALMGASSE 25, VIENNA.

EFFECT OF CELLULOSE ON ARSENIC DETERMINATIONS.

THE rapidity with which deterioration of the contents of the gas purifying tube occurs in both the Gutzeit and Marsh apparatus, with consequent reduction in size of the stains or mirrors for the same amount of arsenic, is well known. Experiments have shown that when cellulose in any form is used in the tube it is degraded by the small amounts of acid present in the evolved gas and then retains the arsenic, probably as the hydride. In order to overcome this difficulty the substitution of glass wool for cotton wool or filter paper in the purifying tube has been tried, and has been found to prolong the life of the tube very considerably. For example, a series of Gutzeit apparatus, the tubes of which required refilling every few days when cotton wool and lead acetate filter paper were employed, has, since the introduction of glass wool (either plain or treated with 10 per cent. lead acetate solution) been in almost continuous use for over three months with no diminution in the intensity of the stain for a given amount of arsenic. The glass wool should be well washed in hot water and dried before use.

T. J. WARD.

THE LABORATORY, STAG BREWERY, S.W.1.

Notes from the Reports of Public Analysts.

The Editor will be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

CITY OF PORTSMOUTH.

REPORT OF THE PUBLIC ANALYST FOR THE YEAR 1925.

DURING the year 1380 samples were examined, of which 1200 were taken under the Food and Drugs Acts, and 99 under the Milk and Dairies Amendment Act. The "sample rate" of food and drugs was 5.1 per 1000 persons.

MILK.—Of the 595 samples examined, 31 were adulterated and 12 were inferior.

"*Certified Milk.*"—Thirty-four samples were examined, 30 passed, and 4 were rejected. The 34 samples gave an average of 1672 bacteria per c.c.; 4 failed

to pass the *B. Coli* test. The average amount of fat was 3·51, and of solids-not-fat 8·76 per cent. On five occasions the milk fat was below 3 per cent. In one case there was a deficiency of 25 per cent. in milk fat, but the producer satisfied the Magistrates that the sample was "as drawn from the cow," and the summons was dismissed. The average price of this milk throughout the year was 1s. 2d. per quart.

Grade A (Tuberculin Tested) Milk.—Of the 12 samples examined, 2 were rejected. The 12 samples gave an average of 3072 bacteria per c.c.; two failed to pass the *B. Coli* test. The average amount of fat was 3·85, and of solids-not-fat 8·58 per cent. Two samples were deficient in solids-not-fat, and, in consequence, a sample was taken under the Food and Drugs Act, and the producer was summoned and fined £5 and costs. The average retail price for the year was 8d. per quart.

Grade A Milk.—The 53 samples examined contained, on the average, 4550 bacteria per c.c. Five failed to pass the *B. Coli* test and were rejected. The average amount of fat was 3·55, and of solids-not-fat, 8·82 per cent. On three occasions the fat fell below 3 per cent. The average price was 7d. per quart, as compared with 6d. per quart for milk of ordinary quality.

BUTTER.—Sixty-two of the 139 samples examined contained boron preservatives.

MARGARINE.—Fifty-one (85 per cent.) of the 60 samples contained boron preservatives. This indicates that the practice of adding these preservatives is decreasing.

DRUGS.—Ninety-three samples were examined, and all found to be genuine, with the exception of 2 samples of camphorated oil, which were slightly deficient in camphor.

REGINALD P. PAGE.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

STALE MILK AND THE REDUCTASE TEST.

ON May 28 three vendors of milk were prosecuted by the New Zealand Health Department in the Magistrates' Court at Auckland, N.Z., for the sale of milk which did not withstand the reductase test for the regulation time.

Mr. Hubble, for the prosecution, said that since prominence had been given to the reductase test legislation had been introduced requiring a sample to be tested within four hours of its sale. Good milk would not cause decolorisation for at least three hours, whilst milk which caused decolorisation in shorter periods was unfit for sale, owing to the presence of an excessive number of bacteria. If the test took less than two hours the milk was definitely bad.

In the first case, in which a dairy company were the defendants, the sample of milk had withstood the test for only 20 minutes.

The defendants pleaded guilty, and explained that the inspector had been served by a little girl who had, unfortunately, drawn the milk from the wrong can, but the inspector stated that the "little girl" appeared to be at least 24 years of age.

The Magistrate imposed a fine of £10, and said that next time fines of £25 and £50 would be imposed.

In the second case a milkman of Takapuna pleaded not guilty. The inspector stated that he had just purchased the milk when the defendant arrived with a fresh supply. He told witness that he had been delayed with the morning's supply.

The sample of the milk had withstood the test for $1\frac{1}{4}$ hours.

The Magistrate remarked that it was a mistake, but that he could not have mistakes with milk. A fine of £5 was imposed.

The defendant in the third case also pleaded not guilty. Evidence was given that the sample had withstood the test for only 2 hours, but it was suggested by counsel that the milk might have become contaminated during transit to the laboratory.

The defendant was convicted and fined £1.

NOTE.—The reductase test was included in the New Zealand Sale of Food and Drugs Act in June, 1924, the clause being as follows:—"When subjected to the reductase test it shall not completely decolorise the methylene blue in less than three hours."

Prior to the three cases mentioned above prosecutions either failed or, if successful, secured convictions without a fine. Legislation was introduced last year requiring that a sample should be tested by the Government Analyst within 4 hours of its sale.

The official reductase test is as follows:—A stock solution is prepared by dissolving 1 part of powdered methylene blue in 2000 parts of water. Immediately prior to use 1 part of this solution is diluted with 9 parts of water.

One c.c. of the diluted solution is mixed with 10 c.c. of the milk in a test-tube and then placed in a water bath or oven kept at a temperature of 38° C. The proportion of methylene blue to milk is 1 to 200,000, as compared with 1 to 270,000 proposed by Orla-Jensen.

W. R. MUMMERY.

CASE UNDER THE CONDENSED MILK REGULATIONS.

A FIRM of grocers was summoned on July 15th, at Rotherham, for selling condensed milk which was deficient in quantity. On the tin it was stated that the contents were equivalent to two pints of ordinary milk, whereas, when tested, the tin was found to contain the equivalent of only 1.68 pints.

The solicitor for the defence said that the deficiency was due to the fact that the tin only contained 14 ozs., which, again, was due to an error of the machinery or of some employee of the manufacturers.

The managing director of the manufacturers said that the deficiency might have been due to a defect in the plunger, which automatically measured the quantity of milk to go into the tins. This plunger was examined three times every day.

A fine of £20 was imposed.

MINERAL OIL IN MARGARINE: AN INVOICE WARRANTY.

ON June 21st, a margarine dealer was summoned at Thames Police Court for the sale of adulterated margarine. Owing to complaints six samples were taken (five informally) from cases of various brands, and one sample formally from margarine which was exposed for sale.

This sample was certified by Mr. Douglas Henville, Public Analyst for Stepney, to contain "a foreign ingredient, namely, mineral oil, which is present to the extent of 11.9 per cent. of the fat or 9.5 per cent. of the sample," with the observation that "Mineral oil is devoid of any food value."

The solicitor for the defence produced two invoices stamped with the warranty and asked the defendant why he had not been instructed sufficiently early to give notice of a warranty defence. The defendant replied that he had been in communication with the wholesale firm.

The Magistrate (Mr. Sharpe) observed that the production of the invoices could only be considered as "mitigating circumstances"; he noted that the sums shown on the invoices were considerable, and that the invoices were stamped in all directions with the warranty and bore June dates.

The Council's solicitor (Mr. Young) pointed out that the sample had been taken on May 18th.

The Public Analyst explained the nature of the adulterant, and compared its value with that of vegetable oils.

The defendant was fined £1 with £2 2s. 0d. costs.

SULPHURIC ACID AS "GOITRE CURE."

A GROCER was charged on May 24th, at the Auckland Police Court, New Zealand, with having sold a drug under the label of "Goitre Cure," which was false and misleading.

Evidence was given by the Analyst of the Health Department that the article consisted of 7.3 per cent. of sulphuric acid in water. The Medical Officer of Health said that it was misleading to describe such an article as a cure for goitre. None of the recognised text-books included sulphuric acid among the possible cures for goitre.

The defendant said that he did not know what the article contained. He had continued to sell it because he had found that certain people had derived benefit from its use. He would undertake not to sell any more of it.

Counsel for the Health Department having stated that the maximum penalty under the Act was £50, the Magistrate fined the defendant £5.

MEAT WINE DEFICIENT IN MEAT EXTRACT.

ON July 15th, at Richmond, Surrey, at the instance of the Surrey County Council, a firm of druggists was summoned under Section 6 of the Sale of Food and Drugs Act, 1875, for selling a certain article of food, a meat wine, which was not of the nature, substance and quality demanded; and, further, under Section 27 of the same Act, for unlawfully giving a label with the bottle of meat wine which falsely described the same. The manufacturers of the wine were summoned for aiding and abetting both offences.

Mr. T. R. Ubsdell, Chief Officer, Public Control Department, Surrey County Council, appeared for the prosecution, and Mr. Walter Frampton, barrister, represented the manufacturers.

Mr. Frampton said that he was prepared to accept full responsibility, and suggested that the summonses against the retailers should be withdrawn, in which case he would plead guilty if the charge were amended to giving a false warranty. Mr. Ubsdell said that if his friend was prepared to plead guilty on behalf of the manufacturers he would agree to the summonses against the first defendants being withdrawn; he was not prepared to agree to any amendment of the summonses. The Bench decided to proceed with the whole of the summonses as they stood, and to take the cases against the manufacturers first. Mr. Frampton then pleaded guilty, and said that, although the analysis made by the defendants' analyst differed in some particulars from that of the County Analyst, he was not going to contest the analysis put forward by the prosecution.

Mr. Ubsdell said that this was a serious case, in which the composition of the article sold was guaranteed to be in accordance with the analysis of Sir Charles Cameron given on the label on the bottle. The Public Analyst would say that in respect of the important ingredients of meat extract the sample he analysed contained only from one-third to one-half of the proportions stated to be present.

Mr. E. Hinks, B.Sc., F.I.C., Public Analyst, gave evidence in support of his certificate. He said that the sample contained not more than one-half of one per cent. of a meat extract containing 8 per cent. of nitrogen; the proportion might be, and probably was, less than this, but it could not be more. He found that the proportion of nitrogen compounds present was only slightly more than one-third, and the compounds of phosphorus less than one-half, the proportions stated to be present in Sir Charles Cameron's analysis. He did not agree that there was much nutritious substance present; he considered that there was very little. A question put to him as to what he considered would be a proper proportion of meat extract in a meat wine was objected to by Mr. Frampton. He said that he laid particular stress upon the proportion of nitrogen compounds present because the value of meat extract depended on them and emphasis was laid upon them in a pamphlet accompanying the sale.

Dr. Joseph Cates, M.D., D.P.H., County Medical Officer, said that, in his opinion, the proportion of meat extract stated to be present by the Public Analyst would be of practically no value.

Mr. Frampton, addressing the Bench, said that during the time the article had been upon the market hundreds of samples had been taken by local authorities, and no complaint had previously been made with regard to it. The Company had a high reputation, and how it came about that this particular sample was not up to the usual standard they could not understand. Whether or not it was due to deposition of a portion of the meat extract caused by the particular wine used in this case he could not say. He asked the Bench to treat the matter as a mistake made in the manufacture. His clients took full responsibility, and the retail vendors were in no way to blame. With regard to what had been said as to a false label, he contended that that was a case for the Merchandise Marks Act, not for the Sale of Food and Drugs Act.

The Chairman of the Bench said that there must be convictions, and a fine of £10 in respect of each offence was imposed, with £10 10s. costs.

The summonses against the firm of druggists were withdrawn.

Trinidad and Tobago.

REPORT OF THE GOVERNMENT ANALYST FOR THE YEAR 1925.

THE Government Analyst (Mr. H. Shrewsbury, F.I.C.), reports that 3265 samples were examined during the year, of which 1788 were official samples of foods and drugs (150 adulterated), 278 were submitted by private persons, and the remainder were for different Government Departments. A lengthy investigation, in which 455 specimens were examined, was made for the auditor, with a view to determining whether signatures were super-imposed on the stamps of documents or *vice-versa*.

TOXICOLOGICAL.—Ground glass was found in three samples of food, and creosote and Cooper's Cattle Dip in other samples. In an unusual case of poisoning $1\frac{3}{4}$ ozs. of caustic soda was found in a cupful of chocolate.

AERATED WATERS.—Of the 678 samples examined, 8 were found to be adulterated; the impurity being lead, the amount of which ranged from 7.7 to 63 hundredths of a grain per gallon. In the most grossly contaminated case the magistrates inflicted a penalty of only 2s. 6d.

COOKING BUTTER.—A sample was found to consist wholly of margarine and to contain only 73.5 per cent. of fat, whereas the Food and Drugs Ordinance requires margarine to contain 80 per cent. of fat. The Magistrate, however, held that the purchaser was not prejudiced since he obtained what he expected when he purchased cooking butter; under this decision a vendor may sell adulterated margarine as cooking butter.

Vendors have frequently sold watered butters under such names as "cooking butter," "cooking grease," etc., and it was to meet this that Standard 2(c) was framed. According to this standard "any substance consisting of butter, but sold as cooking butter, cooking grease or under any other name than butter, shall contain not less than 75 per cent. of butter fat." The sample in question could not be dealt with under this standard, as it did not consist of butter, but of margarine, but it was plainly not of the nature, substance and quality of cooking butter, and was therefore dealt with under 9(e) of the Food and Drugs Ordinance. Even if cooking butter can be considered to be a correct synonym for margarine, the sample was still deficient by 6.5 per cent. of fat under No. 4 of the Standards for Food and Drugs.

"LARD."—Mixtures of beef fat and cottonseed oil devoid of hogs' fat are sold all over the Colony as "lard," and pure lard can only be obtained in 5 lb. tins. Prosecutions have failed because it has been held that people mean lard substitute when they ask for lard. Whether they mean lard or not, they certainly get the substitute; but a vendor should not be excused for selling such a mixture as lard, even though the name of the genuine product has been degraded until it has come to mean a substitute which contains no lard at all.

ARROWROOT.—A sample was found to contain 33 per cent. of cassava starch; the vendor was prosecuted, and fined £4 11s. 3d.

DRUGS.—Of the 18 samples examined, 55 per cent. were adulterated or of inferior quality. Six samples of turpentine bought from 6 different vendors were analysed, and only one was genuine.

Siam.

THE GOVERNMENT LABORATORY, BANGKOK.

SECOND REPORT, FROM APRIL 1, 1922, TO MARCH 31, 1924.

THE Director of the Government Laboratory of Siam, Mr. A. Marcan, F.I.C., has given a report (dated March, 1926) of the work done in the two years ending March 31, 1924 (*cf.* ANALYST, 1924, 49, 88).

PREPARATION OF ESTERS OF HYDNOCARPUS OIL.—The oil is cold-pressed from the seeds of *Hydnocarpus anthelmintica*, and esterified by heating it for 20 hours with alcohol and sulphuric acid. The esters are washed with water, caustic soda, and again with water and are finally distilled *in vacuo*, mixed with animal charcoal, blown with air, filtered and sterilised. In all, 72·8 litres were prepared and distributed for the treatment of leprosy.

CHEMICO-LEGAL WORK.—Twelve of 51 articles tested for poison were found to contain it. Arsenic was found in 3, atropine in 4, atropine and arsenic in 1, calcium oxalate in 3; and boric and salicylic acid in 1 article. Human blood was detected in 5 of 23 cases investigated.

Of the 124 drugs examined under the Harmful Habit-Forming Drugs Law, B.E. 2465, twenty were found to contain opium alkaloids and one cocaine.

INDUSTRIAL MATERIALS.—The bark from a species of oak (Kaw Nun), which is reported to be much used for chewing purposes, was found to contain 28·8 per cent. of tannin, and to be a suitable tanning agent.

Various samples of bamboos, examined as to their suitability for paper-making, contained from 50·4 to 56·1 per cent. of cellulose (calculated on the dry material).

Various samples of hay (for fodder) and of paddy husk (for use as fuel) were examined.

LOCAL DRUGS.—“Kratom” leaves from *Mitrogyne speciosa* are widely used for chewing in Peninsular Siam and, to a certain extent, in Bangkok, where it is now planted in the market gardens. Two new alkaloids, mitrogynine and mitro-versine, were discovered in *M. speciosa* and *M. diversifolia*, respectively. The chemical and physiological properties of these are under examination.

Power Alcohol Production from Sugar Beet.

REPORT TO THE MINISTER OF AGRICULTURE *

THE cost of raw material, allowing for the value of residual products, is placed at 5 per cent. of the price per ton of beet, and the total cost of conversion at 9d. per gallon, so that with beets at £1 and £2, respectively, the cost per gallon of 95 per cent. alcohol, at the works, would be 1s. 9d. and 2s. 9d. respectively. Costs of denaturation, packages, transport and selling charges are additional to this. One gallon of 95 per cent. alcohol is taken as equivalent to three quarters of a gallon of petrol for internal combustion engines of present design, since full realisation of the higher compression ratios and thermal efficiencies possible with alcohol demands engines of special design. A moderate amount of fuel alcohol might be

* Obtainable at Adastral House, Kingsway, W.C.2. Price 6d. net.

marketed at approximately the same price as petrol. The theoretical yield of alcohol from sucrose is 53·8 per cent. by weight or 8 gallons of 95 per cent. alcohol from 1 cwt. of sucrose, and the practical yield about 6·8 gallons per cwt. The prospects for the production of alcohol by synthetic processes are regarded as rather obscure, but the effect of development in this direction would be a restraining influence on the price of petrol, etc.

D. G. H.

Poisons and Pharmacy Acts.

THE Lord President of the Council has appointed a Committee, consisting of Messrs. E. A. Mitchell-Innes, C.B.E., K.C. (Chairman), L. G. Brock, C.B., Sir Malcolm Delevingne, K.C.B., F. W. Gamble, Sir William M. Graham-Harrison, K.C.B., Sir Donald MacAlister, Bt., K.C.B., M.D., E. T. Neathercoat, C.B.E., Colin Smith, G. Stubbs, C.B.E., F.I.C., Sir William H. Willcox, K.C.I.E., M.D., F.I.C., and A. E. Young; with M. D. Perrins (Home Office), and Dr. E. W. Adams, O.B.E., M.D. (Ministry of Health), as Joint Secretaries.

This Committee is to consider and report whether any modifications are necessary or desirable in the Poisons and Pharmacy Acts (1) In regard to the conditions relating to the sale of poisons; (2) in regard to the procedure for the modification or extension of the Schedule of Poisons to which the Acts apply; (3) in the system of making and enforcing Regulations in regard to the keeping, selling and dispensing of poisons; (4) in regard to the Central Authority for the purposes of the Acts; and (5) in regard to any other matters to which the attention of the Committee may be drawn.

The Committee have decided to take evidence on matters within their terms of reference. Any person, association or firm wishing to give evidence or place their views before the Committee should communicate as soon as possible with Mr. M. D. Perrins (Joint Secretary), Home Office, Whitehall, S.W.1. The Committee will inquire generally into the existing machinery regulating the sale (retail and wholesale), keeping, distribution and supply of poisons; any consideration of proposals for the inclusion in or exclusion from the Schedule of Poisons of any specific substance is held *not* to fall within the Committee's terms of reference.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Unsaturated Fatty Acids Associated with Corn (Maize) Starch. T. C. Taylor and L. Lehrman. (*J. Amer. Chem. Soc.*, 1926, 48, 1739-1743.)—Corn starch (α -amylose), completely freed from extraneous fat by means of ordinary solvents, yields, on hydrolysis with acids, 0·5 to 0·6 per cent. of fatty acids, which consist of approximately 24 per cent. of palmitic, 40 per cent. of oleic, and 36 per cent. of linolic acid.

T. H. P.

Influence of Fatty Acids on Bömer's Melting-Point Method. F. J. F. Muschter and R. Smid. (*Chem. Weekblad*, 1926, 23, 284-285.)—Bömer's differential melting-point method (*ANALYST*, 1913, 38, 204, 214; 1914, 39, 84, 171) gives fallacious results if applied to lards containing free fatty acids (in excess of 0·3 per cent of free oleic acid). For instance, a neutral fat showing a Bömer

value of 70, gave a value of 71.5 when mixed with 2.5 per cent. of stearic acid, and of 72.0 when mixed with 5 per cent. of palmitic acid. Again, a grease with a Bömer value of over 71 gave, after removal of free fatty acids, a value below 71, and was therefore adulterated.

Kapok Oil. (*Bull. Imp. Inst.*, 1926, 24, 18-36.)—Kapok seed from Travancore, Federated Malay States, Gold Coast and Zanzibar varied in its oil content from 27.4 per cent. on the dry seed for Travancore to 21.6 per cent. for F.M.S. The oils had the following characteristics:—Sp gr at 15° C., (T) 0.9217 (G.C.) 0.9226, (Z.) 0.914; acid value, (T.) 30.9, (F.M.S.) 54.1, and 61.0, (G.C.) 6.1, (Z.) 26.0; saponification value, (T.) 192.2, (G.C.) 193.5, (Z.) 194.1; iodine value, Hübl. 17 hours, (T.) 90.4, (G.C.) 89.6, (Z.) 101.5; and for Travancore oil unsaponifiable matter, 1.2 per cent.; n_D^{40} , 1.463; and "titer," 29.8° C. The meal from Travancore seed closely resembled commercial kapok cake but contained proportionately more (about 4 per cent.) protein. D. G. H.

Oxidation of Chaulmoogric Acid by Permanganate. G. A. Perkins. (*J. Amer. Chem. Soc.*, 1926, 48, 1714-1725.)—The primary products of the oxidation of chaulmoogric acid by permanganate in alkaline solution are described. α -Dihydroxy-dihydrochaulmoogric acid ($[\alpha]_D^{30} + 4.9^\circ$, m.pt. 106° C.) has been obtained purer than heretofore, and the so-called β -isomeride has been shown to be a mixture of the α -acid with another acid, with $[\alpha]_D^{30} - 38.2^\circ$, and m.pt. 85°, to which the name β -acid is transferred. These acids are stereoisomeric and presumably ω -2:3-dihydroxycyclopentanetricyclic acids; both yield the same tricarboxylic acid on oxidation, and in each the hydroxyl groups are in the *cis*-positions. T. H. P.

Yocco, a new Caffeine-containing Drug. E. Perrot and A. Rouhier. (*Comptes rend.*, 1926, 182, 1494-1496.)—The Yocco is found in the forests of Columbia and is cultivated by the natives, and the drug is prepared by scraping off the bark to obtain the thin warty layer of cork, small portions of which are macerated in water. From this drug a crystalline body can be readily prepared which shows the reactions of caffeine, dissolving in water and chloroform, and with a m.pt. (after drying at 100° C.) of 232° C., volatile at 177-180° C., and polarimetrically inactive. It gives the tetramethyl-alloxantin reaction, yields a blue precipitate with potassium ferricyanide in the presence of nitric acid, and is precipitated from aqueous solutions by silicotungstic acid and by tannin. Two samples contained, respectively, 26.1 and 26.3 per cent. of nitrogen. The amounts of caffeine extracted varied with the solvent, the maximum yield (2.73 per cent.) being obtained by extraction with 2 per cent. hydrochloric acid. D. G. H.

Adulteration of Liquorice with Masticogna. P. Casparis. (*J. Suisse de Pharm.*, 1925, 63, 121-126; *J. Pharm. Chim.*, 1926, 118, 28-29.)—Masticogna, a thickened extract of the root of *Atractylis gummifera*, a plant abundant in Sicily, was mixed during the war with liquorice. It was found in the present examination in 4 samples of liquorice (all made before 1923) out of 37 samples. The root

of *Atractylis* contains the poisonous substances atractylin (a sulphonated glyceride) and atractyligenin. Since atractylin is soluble in absolute alcohol, masticogna may be detected in liquorice by Bertolo's method. The finely powdered dried juice is extracted with absolute alcohol, the extract filtered, and the filtrate evaporated to dryness. The brown residue is fused with pure sodium hydroxide and potassium nitrate, and heated until white, cooled, dissolved in water and nitric acid, and barium chloride added. In the presence of masticogna a precipitate of barium sulphate is formed.

D. G. H.

Reaction for Xanthin. G. Schweizer. (*Chem. Zeit.*, 1926, 50, 430).—When a trace of xanthin is heated with a few crystals of potassium chlorate and one or two c.c. of hydrochloric acid, it is converted into carbamide and alloxan, and, if the liquid is evaporated to dryness on a clock glass and the residue heated for some time on a water bath, the alloxan is transformed into its carmine red modification. On addition of 1 or 2 drops of potassium hydroxide solution, this residue is immediately changed into potassium iso-alloxanate, which has a deep ultramarine blue colour.

T. H. P.

Polarimetric Examination of Oil of Cade. R. Massy. (*J. Pharm. Chim.*, 1926, 118, 61-65).—Although true cade oil obtained from the wood of the trunk has a laevorotatory power of the order described by Huerre (*ANALYST*, 1926, 417), the oil from the branches is only slightly active under polarised light, and that of the root is dextrorotatory. Oils from *Juniperus phoenicea* and *J. thurifera* cannot be polarimetrically differentiated from true cade oil, but that of *Cedrus atlantica*, alone of those examined (which, in addition to the above, included *Pinus halepensis* and *Callitris quadrivalvis*), showed a pronounced dextrorotatory power.

D. G. H.

Biochemical, Bacteriological, etc.

Cyanmethaemoglobin and the Determination of Methaemoglobin. Balthazard and M. Philippe. (*Compt. Rend. Soc. Biol.*, 1926, 94, 522; *J. Pharm. Chim.*, 1926, 118, 69-70).—Five c.c. of blood are placed in a 1 cm. cell with parallel sides, 10 c.c. of water and 1 drop of pure acetic acid added, and, on direct spectroscopic examination, the very dark band of methaemoglobin is seen. A 0.1 per cent. solution of potassium cyanide is added from a burette reading to 0.01 c.c. until the dark band disappears. Then for every c.c. of potassium cyanide added a quantity of methaemoglobin of respiratory capacity in oxygen of 0.1715 c.c. is present in the blood. Potassium cyanide has no action upon oxyhaemoglobin under these conditions. Free methaemoglobin may be titrated with potassium cyanide solution in the same way, 2 molecules of cyanogen converting 1 molecule of haemoglobin into cyanmethaemoglobin.

D. G. H.

Comparison of the Folin-Wu and the new Benedict Method for Sugar in Blood and Cerebrospinal Fluid. J. D. Lyttle and J. E. Hearn. (*J. Biol. Chem.*, 1926, 68, 751-757).—In a recent study of nephritis with nitrogen retention

simultaneous blood and cerebrospinal fluid (drawn by lumbar puncture) sugar determinations were made on 26 cases by the method of Folin and Wu (*J. Biol. Chem.*, 1919, **38**, 81), and by the new method of Benedict (*J. Biol. Chem.*, 1925, **64**, 207). For cerebrospinal fluid a modification is given of the Folin-Wu method in which proteins are precipitated with tungstic acid; this causes a 1:5 dilution. In the majority of cases the Folin-Wu method gave higher values than did the Benedict method. In the blood the average difference by the two methods was 12.4 mgrms., with 13.8 per cent. of the cases showing practically no difference. The non-protein nitrogen content of the blood had no relation to differences in sugar content by the two methods. In the cerebrospinal fluid the average difference was 3.1 mgrms., with 52.6 per cent. of the cases showing practically no difference. The fact that in 21 per cent. of the cases there was a difference of 5 mgrms. or more shows that the unknown substance can, and does, get into the fluid, probably through the choroid plexus. It is not known whether this happens in a normal individual or not. The protein and non-protein nitrogen content of the cerebrospinal fluid has no relation to differences in sugar content by the two methods.

P. H. P.

Determination of Sugar in Blood and Normal Urine. S. R. Benedict. (*J. Biol. Chem.*, 1926, **68**, 759-767.)—The technique described by Benedict (*J. Biol. Chem.*, 1925, **64**, 207) for the determination of sugar in blood, which yields lower figures than those obtained by the use of older methods, was subjected to a severe criticism by Folin (*J. Biol. Chem.*, 1926, **67**, 357). Folin also proposed new reagents for the determination of sugar in blood and urine. The author now discusses Folin's method and replies to his criticism. A new technique is described, essentially the same as the previous one, but with modifications which make for greater speed and convenience in the analysis, and with provision for always having present an adequate amount of sulphite in order to gain a normal yield of cuprous oxide from a given amount of glucose. The use of the tungstic acid colour reagent has been retained, although Folin champions a not easily obtained complex molybdic acid reagent for colour development. *Alkaline Copper Solution.*—In about 800 c.c. of water 200 grms. of sodium citrate and 60 grms. of anhydrous sodium carbonate are dissolved. With stirring or shaking 6.5 grms. of pure crystallised copper sulphate, dissolved in 100 c.c. of water, are added; then 9 grms. of ammonium chloride are added, the whole is diluted to 1 litre and mixed. Not more than 1 month before use to each 100 c.c. of the reagent 2.5 to 3 grms. of pure anhydrous sodium sulphite should be added. *Complex Tungstic Acid Colour Reagent.*—In a litre flask, 100 grms. of pure sodium tungstate are dissolved in about 600 c.c. of water, 50 grms. of pure arsenic pentoxide are added, followed by 25 c.c. of 85 per cent. phosphoric acid and 20 c.c. of concentrated hydrochloric acid. The mixture is boiled for 20 minutes, cooled, 60 c.c. of commercial formalin, 45 c.c. of concentrated hydrochloric acid and 40 grms. of sodium chloride added, diluted to 1 litre and mixed. The technique is as follows:—Two c.c. of the 1:10 tungstic acid filtrate are measured into a Folin-Wu sugar tube, then 2 c.c. of the copper reagent.

The contents are mixed, and the tubes placed in boiling water for 5 minutes, then cooled by immersion in cold water and 2 c.c. of the complex tungstic acid colour reagent are added. After 1 to 2 minutes the contents of the tube are diluted to 25 c.c. with water, thoroughly mixed, and compared with a standard (1:10 dilution of a 0.1 per cent. solution of pure glucose in water, preserved with 1 or 2 c.c. of toluene) in a colorimeter. The standard will keep so long as any toluene remains on the surface. The solution analysed should not be more than twice as strong or less than half as weak as the standard. Results for blood sugar are in close agreement with those previously reported. For sugar in normal urines the best method is that of Folin and Berglund (*J. Biol. Chem.*, 1922, 51, 209) with the use of Lloyd's reagent, and the new reagents in place of the Folin-Wu copper and molybdic acid reagents. P. H. P.

Determination of Glucosan. H. J. Deuel, Jr., S. S. Waddell and J. A. Mandel. (*J. Biol. Chem.*, 1926, 68, 801-820.)—In a paper on "The Physiological Behaviour of Glucosan" experiments are described which were carried out on dogs made diabetic with phlorhidzin, to find to what extent glucosan, administered orally, is absorbed and oxidised, as judged by the effect on the respiratory quotient, what effect it exerts on the glycosuria, and its fate after subcutaneous and intraperitoneal injection. For the experiments a tetraglucosan preparation called "Salabrose" was used. A method was devised for the quantitative determination of glucosan in the urine. Urinary glucose was determined by the Bertrand method owing to the high colour of the glucosan solutions. It was found that, on boiling with dilute acid, glucosan was quantitatively converted into glucose, which, after neutralisation, could be determined by the usual analytical methods. A table shows the results obtained when salabrose solutions were boiled with water and with different concentrations of hydrochloric acid. Boiling with water for 15 or 30 minutes was without effect on the glucosan, but it was destroyed to a considerable extent when boiled with 20 per cent. hydrochloric acid. Maximum conversion to glucose occurred on boiling with 5 per cent. of acid for 30 minutes. Glucosan was calculated by subtraction of the free glucose from the total glucose obtained after hydrolysis. This figure, which represents the glucosan as glucose, is changed to that of glucosan as such by the subtraction of 10 per cent. of this amount. The procedure is satisfactory for the determination of glucosan in diabetic urine, since urine containing glucose, when boiled with hydrochloric acid, remains unchanged in glucose content, whilst after the addition of glucosan to such a urine subsequent hydrolysis yielded quantitative results for glucosan. P. H. P.

Simpler Nitrogenous Constituents of Yeast. I. Choline and Nicotinic Acid. H. B. Vickery. (*J. Biol. Chem.*, 1926, 68, 585-592.)—Since the problems presented by the metabolic changes which take place in the living cell are fundamentally chemical in nature, it was thought that a study of the constituents of the cell might lead to a fuller knowledge of the chemical environment in which the metabolic processes take place, and to a better understanding of what these processes may be. A study of the nature of the simpler nitrogenous substances extracted

from fresh brewers' yeast by hot water, with the use of the methods previously employed by the author in the study of alfalfa juice (*J. Biol. Chem.*, 1925, **65**, 81; 1924, **60**, 647) is in progress. A brief description is given of the method used for the fractionation of the yeast extract, and the basic substances not precipitated by Neuberg's reagent are dealt with. These contained nitrogen equivalent to 3.35 per cent. of the nitrogen of the yeast extract. When fractionated with mercuric chloride in acid solution, 2.41 per cent. of the nitrogen of the yeast extract was found in the precipitate. Of this nitrogen, about 86 per cent. belonged to choline and 6 per cent. to nicotinic acid. Further quantities of nicotinic acid were found in the filtrate from the mercuric chloride precipitate. Choline accounted for 2.07 per cent. of the nitrogen of the yeast extract, and 0.29 per cent. of the nitrogen of the dry yeast; nicotinic acid accounted for 0.28 per cent. of the nitrogen of the extract and 0.039 per cent. of that of the dry yeast. Bases of the betaine type, if present at all in yeast, can occur only in very small proportions. Since the yeast extract was not subjected to hydrolysis, these substances occur free in the cell solution to at least the extent given.

P. H. P.

Differences in Water-soluble Vitamins of Yeast Extracts of Varied Origin. L. Randoin and R. Lecoq. (*Comptes rend.*, 1926, **182**, 1408-1409.)—

Brewers' yeast was found to contain two factors, one capable of maintaining a pigeon's life and the other of curing polyneuritis, whilst distillery yeast contained only the former.

D. G. H.

Anti-Rachitic Substances. IV. The Polymerisation of Cholesterol. C. E. Bills and F. G. McDonald. (*J. Biol. Chem.*, 1926, **68**, 821-831.)—

Cholesterol on treatment with activated floridin behaves in general as an olefinic terpene. It is first polymerised and the polymer is then degraded by further catalytic action into a resinous mass. The polymer, which appears to be tricholesterol, $C_{81}H_{138}O_3$, was isolated as an early product of the catalysis by abbreviating the period of "refluxing" and not allowing the entire series of colour changes to occur. It is obtained in fine, colourless, microscopic needles from equal volumes of carbon tetrachloride and alcohol; molecular weight, theoretical, 1159, found 978; $[\alpha]_D^{20} = -40.8^\circ$ (2 per cent. in carbon tetrachloride). A characteristic of the pure substance is that when it is precipitated from carbon tetrachloride in the amorphous condition by the rapid addition of an equal volume of alcohol, this precipitate suddenly becomes crystalline when the solution is warmed. Its solubilities are recorded in a table. Like several other cholesterol derivatives, tricholesterol exhibits a multiple melting-point; principal m.pt., 203° ; secondary m.pt.s., 209° and 196° . Tricholesterol gives the Burchard-Liebermann, Salkowski and Moleschott cholesterol colour reactions with slight differences; it absorbs iodine erratically from Hübl's solution, and is not acetylated on treatment with acetic anhydride. Tricholesterol is not anti-rachitic, and does not become so by irradiation. However, on further treatment with floridin it gives an anti-rachitic degradation product which apparently is identical with the product obtained directly from cholesterol. The anti-rachitic

substance is different from the anti-rachitic vitamin and irradiated cholesterol, and its mode of action is not understood. Tables and plates show the results of experiments on rats.

P. H. P.

The Solubility of Vitamin B in Benzene. R. R. Williams and R. E. Waterman. (*J. Biol. Chem.*, 1926, 68, 499-501.)—It has been shown by McCollum and Simmonds (*J. Biol. Chem.*, 1918, 33, 55) that, under certain special conditions, vitamin B can be extracted by benzene. Benzene does not extract the vitamin from white beans or wheat embryo, but if the raw material is extracted with 95 per cent alcohol and the alcoholic extract is evaporated on dextrin, extraction with benzene in a continuous type of extractor will remove the vitamin from the dextrin. The authors have carried out similar experiments on rice polish and find that benzene does not extract the protective substance directly from it, but does dissolve the vitamin if the residue of the alcoholic extract is repeatedly treated with the hot hydrocarbon in a continuous type of extractor. The explanation appears to be a simple one; *viz.* that pure benzene never dissolves the vitamin, but does remove it from alcoholic extracts which still retain small amounts of water and alcohol, as alcoholic extracts of foodstuffs are very prone to do. The effective solvent thus seems to be the low-boiling ternary mixture of alcohol 18.5 per cent., water 7.4 per cent., and benzene 74.1 per cent., which distils at 64.85° C. from a mixture of any proportions. In a continuous type of extractor any small amount of alcohol and water present would be utilised again and again with the much larger amount of benzene present. The low-boiling mixture of alcohol, water and benzene is by no means highly selective, and there is little promise in the use of immiscible solvents for the separation of vitamin B.

P. H. P.

Toxicological and Forensic.

Determination of Strychnine in Poisoned Grains. J. W. Elmore. (*J. Assoc. Off. Agric. Chem.*, 1926, 9, 224-230.)—Determination of strychnine in poisoned grain used for rat bait (1) by extracting the alkaloid with kerosene and chloroform, and titrating with sulphuric acid, or (2) by extracting with chloroform and weighing as strychnine picrate, gives unreliable results, but the following procedure has proved satisfactory. The barley, dried overnight at 50° to 60° C if necessary, is finely ground, and 25 grms. weighed into a dry 300 c.c. Erlenmeyer flask, and shaken occasionally during 30 minutes with 120 c.c. of an ether-chloroform mixture (2:1) in the stoppered flask. The mixture is next shaken for an hour with 25 (for wheat, 15) c.c. of 10 per cent. ammonium hydroxide solution, allowed to stand overnight, shaken for 15 minutes, and for a further period of 15 minutes with 5 c.c. of a clarifying syrup, such as "Karo." One hundred c.c. of the ether and chloroform mixture are mixed in a separating funnel with sufficient ether to cause the layer of solvents to float in the subsequent extractions. The liquid is extracted with 50 c.c., and then with six portions of 25 c.c. of 0.5 per cent. hydrochloric acid solution, the combined extract being evaporated in a 400 c.c.

beaker to 50 c.c., cooled, made just alkaline with ammonia, then slightly acid with acetic acid, and warmed gently to flocculate suspended matter. When cool, the liquid is mixed with 2 c.c. of 10 per cent. neutral lead acetate solution, made up to volume in a 100 c.c. flask, shaken and filtered into a dry 100 c.c. cylinder without washing, the volume (say, 97 c.c.) being noted. This filtrate is shaken with 3 c.c. of 3 per cent. sodium oxalate solution and, after 15 minutes rest, filtered into a dry 100 c.c. cylinder, the volume (say 96.9 c.c.) being again noted. The liquid is transferred to a 250 c.c. beaker, evaporated to 70 c.c., cooled, mixed with 25 c.c. of recently filtered, saturated picric acid solution, and allowed to stand for three hours, with occasional stirring during the first half-hour. The strychnine picrate is collected on a tared Gooch crucible, washed with 50 to 80 c.c. of cold water, dried at 105°C., and weighed; strychnine picrate $\times 0.5932 =$ strychnine. The weight of the dried material represented by this precipitate is $\frac{25 \times 97 \times 96.9}{99.9 \times 120} = 19.6$ grms. In calculating

the proportion of strychnine in the moist grain, a correction is made for the loss of weight at 50° to 60° C. The determination is required for enforcing the California Economic Poison Law.

T. H. P.

Water Analysis.

Determination of Iodine in Natural Waters. H. W. Brubaker, H. S. Van Blarcom and N. H. Walker. (*J. Amer. Chem. Soc.*, 1926, 48, 1502-1504.)—

For determining the small amounts of iodine usually present in natural waters, the method used by Hunter in the case of thyroid tissue is recommended (*J. Biol. Chem.*, 1909, 7, 336). A quantity of 1 to 2 litres of the water is evaporated to about 200 c.c. and the iodine then oxidised to iodate by boiling with a sodium hypochlorite solution (the chlorine content being known approximately) acidified with phosphoric acid solution prepared by diluting the 85 per cent. acid with an equal volume of water; the boiling is continued until the free chlorine is eliminated. A small quantity of 1 per cent. potassium iodide is added, and the iodine thus liberated titrated with 0.00474 *N* sodium thiosulphate solution, 1 c.c. of which corresponds with 0.1 mgrm. of iodine. If much bromine is present in the water, prolonged boiling may be necessary to expel it. The presence of iron in large proportion vitiates the results slightly.

T. H. P.

Organic Analysis.

Determination of Hydrocyanic Acid in Gaseous Mixtures. G. E. Seil. (*Ind. Eng. Chem.*, 1926, 18, 142-143.)—A special cylindrical funnel (Tutwiler burette), having a tap at the bottom, and a three-way ray at the top, connecting with a small stoppered cylinder, is filled to the 540 c.c. mark with dilute starch solution saturated with sodium hydrogen carbonate. This solution is then withdrawn and titrated, to distinct acidity, with dilute (1 : 8) hydrochloric acid. The tapped

funnel is next filled with water, this is displaced by the gas sample, the flow of gas being continued for a few minutes, and the volume of the gas is adjusted at 500 c.c. by means of a levelling reservoir. Approximately 0.1 *N* iodine solution is then added slowly, with constant shaking, until the starch solution just turns blue, the blue solution is then washed into another vessel and standard thiosulphate solution is added until the blue colour disappears. The amount of hydrochloric acid used previously is added and, after one minute, the liberated iodine is titrated with the standard thiosulphate solution— $\text{CNI} + \text{HI} + \text{HCl} = \text{HCl} + \text{HCN} + \text{I}_2$. The thiosulphate solution is prepared by diluting 84.75 c.c. of 0.1 *N* solution to 1 litre; each c.c. is equivalent to 0.2288 mgrm. of HCN per litre when 500 c.c. of gas are taken for the analysis. Hydrogen sulphide does not interfere with the determination.

W. P. S.

***α*-Naphthyl Isocyanate as a Reagent for Phenols and Aliphatic Amines.**

H. E. French and A. F. Wirtel. (*J. Amer. Chem. Soc.*, 1926, **48**, 1736–1739.)—As well as for alcohols (*ANALYST*, 1926, 263), *α*-naphthyl isocyanate serves as a satisfactory reagent for phenols and for primary and secondary aliphatic amines, forming urethanes which crystallise well from petroleum spirit and have sharp melting-points. The isocyanate must be heated for a few minutes with a phenol, but with amines the reaction takes place at the ordinary temperature. The reaction, which is catalysed by dry ethereal solutions of tertiary aliphatic amines, is not given by polyhydroxy-phenols under the conditions employed. The same reagent acts well with oximes and with acetamide and acetanilide.

T. H. P.

Determination of Small Quantities of Water in Methyl Alcohol.

M. M. Rising and J. S. Hicks. (*J. Amer. Chem. Soc.*, 1926, **48**, 1929–1933.)—The temperatures at which mixtures of methyl alcohol (containing traces of water as impurity) and twice its volume of a suitable solvent just begin to appear cloudy when cooled, are plotted against the percentage of water. The curves obtained are used for the determination of the water. Fractionated ligroin (b.pt., 50° to 55° C.), or pure normal hexane are suitable solvents and enable 4 and 3 per cent. of water to be determined for temperature ranges extending over 31° and 15° C., respectively. Hexane is to be preferred, as it is a definite substance.

J. G.

Determination of Acetone by means of Hydroxylamine Hydrochloride.

M. Marasco. (*Ind. Eng. Chem.*, 1926, **18**, 701–702.)—Acetone reacts with hydroxylamine hydrochloride according to the equation: $(\text{CH}_3)_2\text{CO} + \text{NH}_2\text{OH} \cdot \text{HCl} = (\text{CH}_3)_2\text{CNOH} + \text{HCl} + \text{H}_2\text{O}$. The reaction is slow (*cf.* *ANALYST*, 1922, **47**, 146), and is not complete when the hydrochloric acid is allowed to accumulate. If, however, the acid is neutralised as it is formed the reaction proceeds and comes to an end within about five minutes, at which stage 94.4 per cent. of the acetone has reacted. A solution containing not more than 0.2 gm. of acetone is added to about 400 c.c. of 0.2 per cent. hydroxylamine hydrochloride solution which has been neutralised just previously, methyl orange being used as indicator. The

mixture is titrated with 0.1 *N* sodium hydroxide solution, stirred for twenty seconds and again titrated, and so on. After the fourth titration the end-point will be approached very nearly, and when the mixture liberates no appreciable amount of acid after one minute's standing, the reaction is complete. Each c.c. of 0.1 *N* sodium hydroxide solution is equivalent to 0.00614 gm. of acetone, approximately, but the hydroxylamine hydrochloride solution should be standardised against known amounts of pure acetone. The stock of hydroxylamine hydrochloride solution keeps for a few days, but it gradually becomes acid and requires neutralising each time it is used. The method may also be used for the determination of aldehydes; formaldehyde reacts like acetone in liberating only about 94.4 per cent. of the theoretical amount of hydrochloric acid. W. P. S.

Head Oils of the Dolphin (*Delphinus Delphis* Lin.). H. Marcelet, (*Compt. rend.*, 1926, **182**, 1416–1417.)—Detailed analyses of the oils obtained from the maxillary glands (M), from between the upper jaw and nose (N), and surrounding the skull (H), revealed considerable differences. The figures given include:—Sp.gr. at 15° C., (M) 0.9206, (N) 0.9308, (H) 0.9330; n_D^{17} , (M) 1.4548, (N) 1.4640, (H) 1.4790; acidity (per cent. oleic), (M) 0.05, (N) 0.08, (H) 0.07; saponification value, (M) 267, (N) 259, (H) 212; iodine value, Wijs, (M) 17, (N) 56, (H) 133; per cent. of solid fatty acids, (M) 30.82, (N) 19.28, (H) 10.08; of liquid fatty acids, (M) 18.7, (N) 43.17, (H) 74.04. D. G. H.

Cuttlefish Oil (*Todarus Sagittatus*, Lk.). E. André and H. Canal. (*Compt. rend.*, 1926, **183**, 152–154.)—Eight cuttlefish, weighing about 2 kilos., were dissected, and the head and tentacles (*a*), visceral sac and swim bladder (*b*), and viscera and genital glands (*c*), separately analysed. The percentages of oil found were as follows:—(*a*) 1, (*b*) 1.25, (*c*) 10, having the following characteristics:—Sp. gr. (*a*) 0.983, (*b*) 1.008, (*c*) 0.929; iodine value (Hanus), (*a*) 144.5, (*b*) 153.9, (*c*) 173.6; saponification value, (*a*) 135.7, (*b*) 141.9, (*c*) 163.4; fatty acids, per cent. (*a*) 76.6, (*b*) 75.0, (*c*) 88.0; unsaponifiable matter, (*a*) 21.6, (*b*) 25.0, (*c*) 8.0; acetyl value, (*b*) 94.2, (*c*) 28.7; glycerol, (*a*) positive reaction. *Fatty acids*: Neutralisation value, (*a*) 190.3, (*b*) 187.3, (*c*) 188.3; iodine value (Hanus), (*a*) 205.2, (*b*) 180.7, (*c*) 175.4; mean molecular weight, (*a*) 294.0, (*b*) 298.0, (*c*) 297.0. It is concluded that fat is not stored as such by the cuttlefish, but transformed into muscle. No aliphatic alcohols were found in the fat. D. G. H.

Determination of the Composition of Tung (Chinese Wood) Oil by means of Thiocyanogen Absorption. H. P. Kaufmann. (*Ber.*, 1926, **59**, 1390–1397.)—The glyceride of pure β -elaeostearic acid was prepared from tung oil by Grün's method (*Analyse der Fette u. Wachse*, 1925, I., 294), and its thiocyanogen absorption determined as previously described (ANALYST, 1926, 157, 264). The result, calculated into the equivalent iodine value, gave a mean value of 90, whilst for the saturation of one double bond the theoretical iodine value is 91. Hence the addition of thiocyanogen only takes place at one of the three double

bonds of the glyceride. Analogous results were obtained with β -elaeostearic acid, the mean thiocyanogen value (in terms of iodine) being 86, as against the theoretical iodine value of 87.

In determining the bromine value 20 c.c. of a carbon tetrachloride solution of β -elaeostearic acid (0.2328 gm.) were treated with 75 c.c. of an approximately 0.1 N bromine solution in methyl alcohol saturated with sodium bromide, and left in the dark, and the amount of unabsorbed bromine was determined in successive portions of 15 c.c. of the solution. The following results, calculated as iodine values, were obtained:—

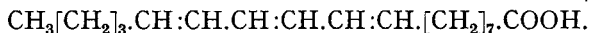
10 minutes.	20 minutes.	1 hour.	2 hours.	6 hours.	15 hours.	24 hours.
171	174	180	182	183	191	196

Thus, after one hour a value corresponding to an addition at two double bonds (theory, 182) was reached. Not until after several hours did slow addition at the third double bond begin. Analogous results were obtained with the glyceride.

Addition of bromine at three double bonds.—Forty c.c. of the carbon tetrachloride solution of the β -elaeostearic acid (=0.4656 gm.), were made up to 250 c.c. with the solution of bromine in methyl alcohol saturated with sodium bromide, and exposed at a distance of 50 cm. to the rays of a mercury vapour (uvio) lamp. The following results were obtained on determining the unabsorbed bromine in successive portions of 20 c.c. :—

Time, minutes	5	10	20	30	40	50	60	70	80
Iodine value ...	180	215	229	256	273	272	273	276	293
Acidity (in c.c. of 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution) ...	—	—	—	—	—	—	—	0.1	0.15 0.3

The calculated iodine value for addition at three double bonds is 273. After this value is reached substitution of bromine does not begin for some time. These results confirm the conclusions drawn from refractometric determinations by Boeseken and Ravensway (*Rec. trav. chim.*, 1925, **44**, 241) that elaeostearic acid has three double bonds and belongs to the linolenic acid series—



On using the results obtained in the thiocyanogen and bromine absorption in the method of calculation previously described (*ANALYST*, 1926, 265), and assuming tung (Chinese wood) oil to contain no unsaturated fatty acids other than oleic and elaeostearic acids, three specimens of the oil were calculated to have the following composition:

- I. Elaeostearin, 78.5; olein, 22.8 per cent.
- II. Elaeostearin, 83.9; olein, 7.6; glycerides of saturated acids and unsaponifiable matter, 8.5 per cent.
- III. Elaeostearin, 87.1; olein, 13 per cent.

Examination of Kemp Fibres. H. Bliss, J. Duerden, J. F. Roberts, J. Blyth, and H. Hirst and A. T. King. (*J. Text. Inst.*, 1926, 17, T264–304.)—This series of five papers deals at some length with the occurrence, characteristics, effects, and determination of kemp in wool from various breeds of sheep and in mohair. Numerous photomicrographs are given. In certain East Indian wools kemp is quite flat and broad, resembling, under the microscope, an empty hose-pipe. In merino it is more like a wool fibre, except that the root is frayed out into a number of delicate fibrils. Above this, viewed by transmitted light, is a clear transparent part, and beyond this a black central core, surrounded by a clear margin and extending nearly the full length of the fibre. The tip of the fibre is clear and solid. Viewed by reflected light on a dark background the central core appears as a silvery streak. Like true wool, the clear parts toward the root and tip are uniform in structure throughout their thickness, and consist only of solid fusiform cortical cells and an outer serrated cuticle. The remainder of the fibre has an additional thick central core or medulla occupied by minute air inclusions, and it is this medulla which gives kemp its peculiar characteristics, as distinct from wool. Kemp fibres are made up of three layers—cuticle, cortex and medulla. The cuticle possesses some special features, as compared with those of ordinary hair-fibres of the Welsh fleece, in that the scales are more compressed in a vertical direction; this gives the fibres a more serrated edge in section. The medulla of kemp is rather less regular than that of hair fibres, being less evenly rounded and appearing more compressed. Also kemp shows extreme irregularity of shape.

Quantitative Determination.—Methods involving counting the fibres are laborious and unsatisfactory; also, the fibres are liable to break. A much better procedure is to weigh the kemp. From 0.2 to 0.3 grm. of the wool is separated, so as to get a complete staple, and is washed with ether. The sample is then dissected, bit by bit, on a board covered with black velvet, the kemp- and non-kemp fibres severally weighed, and the percentage of kemp calculated. The method of sampling has yet to be standardised, but a very fair indication of the kempiness of a fleece can be obtained by sampling from the point of the shoulder and from the point of the rump. The latter is about the worst place in the fleece, and if the kemp is low there, the fleece may be passed as relatively free from kemp. A figure of 2 to 3 per cent. of kemp in the shoulder is certain indication of a fleece that is thoroughly kempy all over.

R. F. I.

Determination of Aromatic Hydrocarbons in Light Petroleum. A. M. Erskine. (*Ind. Eng. Chem.*, 1926, 18, 694–698.)—The lowering of the critical temperature of solution of light petroleum and nitrobenzene by the presence of aromatic hydrocarbons is directly proportional to the percentage by weight of the latter. In the fractions distilling at 50° to 95° C., 95° to 124° C., and the residue above 124° C., the lowering in degrees by each 1 per cent. of aromatic hydrocarbon is 1.18 for benzene, 1.12 for toluene, and 1.07 for xylene; these factors apply exactly up to 20 per cent. by weight of the aromatic hydrocarbons. As applied to a sample

of light petroleum, the method is as follows:—500 c.c. of the sample are fractionally distilled, the most effective fractionating column available being used, and the fractions 50° to 95° C., 95° to 124° C., and 124° to 150° C., (or the residue above 124° C.), at 760 mm., collected separately. After removal of any olefines by treatment with mercuric acetate, a 50 c.c. portion of each fraction is sulphonated with 2 volumes of 98 per cent. sulphuric acid, the mixtures being shaken for ten minutes except in the case of the 50° to 95° C. fraction, for which thirty minutes are required if the benzene content is high. The acid layer is removed, and the petroleum layer washed first with water, then with 10 per cent. sodium hydroxide solution, finally with water until neutral, and dried over calcium chloride. Two grms. of each fraction, before and after sulphonation, are placed in a series of tubes, 2 grms. of pure dry nitrobenzene are added to each, and the tubes are sealed. The critical solution temperatures of these mixtures are then determined in an apparatus that will give thorough shaking and a controlled slow rise and fall in temperature. The rise in critical solution temperature after deducting 0.60 and 0.52 degree in the toluene and xylene fractions, respectively, multiplied by the factor 0.845 for benzene, 0.890 for toluene, and 0.938 for xylene, gives the percentage by weight of the aromatic hydrocarbon in the corresponding fraction. The amount of each in the original sample may then be calculated. The 2 gm. portions of the fractions taken for the determinations may be obtained with sufficient accuracy by measurement from a pipette graduated to 0.05 c.c., with suitable temperature control.

W. P. S.

The Aromatic Hydrocarbon Content of Natural Gas Petroleum. A. M. Erskine. (*Ind. Eng. Chem.*, 1926, 18, 722-723.)—The presence of small amounts of benzene, toluene and *m*-xylene in a sample of Pennsylvania natural gas petroleum was proved by fractionation, concentration of each aromatic hydrocarbon by extraction with liquid sulphur dioxide, and identification as the corresponding dinitro or trinitro derivative. Quantitative determinations on the fractions by means of the critical solution-temperature method with nitrobenzene (*cf.* preceding abstract), showed that the original sample contained 0.6 per cent. of benzene, 0.6 per cent. of toluene, and 1.2 per cent. of *m*-xylene, by weight. W. P. S.

“Boiling off” Value of Bleached Cotton. Angele. (*Leipziger Monats. Text. Ind.*, 1925, 40, 480-481; *J. Text. Inst.*, 1926, 17, A202.)—A method of detecting damage due to over-bleaching consists in boiling a sample of the fabric with sodium hydroxide solution, which dissolves the damaged cellulose portion. The solution is filtered, acidified and titrated with 0.1 *N* potassium permanganate solution. The number of c.c. required to effect complete oxidation of the dissolved organic matter is termed the “boiling-off value.” A standard value of 10 to 12 has been adopted in many laboratories.

Inorganic Analysis.

Colorimetric Method for the Determination of Free Chlorine in Air. L. E. Porter. (*Ind. Eng. Chem.*, 1926, 18, 730-731.)—A definite volume (100 c.c. to 500 c.c.) of the air is aspirated through 10 c.c. of a solution containing 1 gm.

of *o*-tolidine and 100 c.c. of concentrated hydrochloric acid per litre; the coloration obtained is then compared with standards. The standards recommended by the author are prepared by diluting mixtures of copper sulphate and potassium dichromate solutions, and a table is given showing the quantities of these solutions corresponding with amounts of chlorine ranging from 0.01 to 1.0 part per million.

W. P. S.

Determination of Sulphur Dioxide in the Presence of Excess of Air.

E. D. Ries and L. E. Clark. (*Ind. Eng. Chem.*, 1926, 18, 747.)—The most satisfactory solution for the absorption of the sulphur dioxide was found to be sodium hydroxide solution containing a small quantity of stannous chloride. The gas is passed through a 4-bulb Mitscherlich apparatus in which is placed 10 c.c. of 10 per cent. sodium hydroxide solution containing 0.002 mol. of stannous chloride per litre. The solution is then rinsed into a flask, diluted to 50 c.c., cooled, acidified with 50 c.c. of 12 *N* hydrochloric acid, cooled, and 2 c.c. of carbon tetrachloride are added as indicator. The mixture is titrated with 0.003 *M* potassium iodate solution, the flask being shaken vigorously during the titration until the pink colour of the carbon tetrachloride disappears. The reagent must be titrated separately to determine the small but constant amount of iodine absorbed by the stannous chloride.

W. P. S.

Iodimetric Titration of Acids. I. M. Kolthoff.

(*Chem. Weekblad*, 1926, 23, 260–261.)—Iodimetric titration of weak acids, with a dissociation constant greater than 10^{-6} , is possible, provided that 1 grm. of potassium iodide, 5 c.c. of 3 per cent. potassium iodate solution and an excess of thiosulphate are added to 10–25 c.c. of the acid solution, and the excess titrated back after 15 to 30 minutes. The P_H value of the solution is then about 7. This method of titration may be of especial use with coloured solutions in which the colour change of an indicator cannot be seen.

Determination of Traces of Mercury in the Presence of Organic Matter.

H. S. Booth, N. E. Schreiber and K. C. Zwick. (*J. Amer. Chem. Soc.*, 1926, 48, 1815–1823.)—One litre of the solution containing the mercury salt is boiled under a reflux condenser with 100 c.c. of sulphuric acid, and oxidised with potassium permanganate, any manganese dioxide being removed by hydrogen peroxide, excess of which is destroyed by boiling. Hydrogen sulphide is then passed through the solution, and the coagulation of the mercury sulphide assisted by a few flocks of manganese hydroxide, which are produced by the addition of sodium hydroxide solution. The filtered, alkali-free, dry precipitate is then well ground in an agate mortar with 0.5 grm. of dry precipitated lead chromate, and the mixture placed in a tube in the sealed end of which is a decomposition-mixture consisting of magnesite and lead chromate. A layer of sodium carbonate (0.1 grm.) is added, and the tube loosely plugged and dried inside a larger tube at 200° C. It is then placed in a decomposition tube, having a constricted end, and the magnesite heated in a furnace. The mercury collects in the constriction, and, after it has

been dried, is transferred to a capillary burette and its length measured in a comparator in which Canada balsam is used to counteract the lens effect of the capillary. The length is corrected for temperature, and the weight obtained from the calibration-curve of the comparator. The method prevents loss of mercury due to volatilisation and provides for the simultaneous determination of mercury in its ionic and colloidal forms. For weights of mercury taken varying from 0.27 to 4.21 mgrms. an almost constant error of -0.02 mgrm. was found. J. G.

Mercuric Bromide Paper for Use in the Gutzeit Test for Arsenic.

G. Kemmerer and H. H. Schrenk. (*Ind. Eng. Chem.*, 1926, 18, 707.)—The paper to be sensitised should be dried at 105° C. for one hour, then cut into strips and saturated with a 1.5 per cent. mercuric bromide solution in 95 per cent. alcohol; the strips are allowed to drain, dried for ten minutes in a large desiccator, and used. The treated strips should not be kept for more than two hours before they are used. Uneven and spotted colour bands are obtained in the test if the paper is not dried before it is impregnated with the mercury bromide solution.

W. P. S.

Improvement of the Cobalt Aluminate Test for Aluminium. C. Otto.

(*J. Amer. Chem. Soc.*, 1926, 48, 1604–1605.)—Aluminium hydroxide precipitated in presence of cobalt, decrepitates and tends to fall when heated on a platinum wire. This trouble may be avoided if the lump of precipitate is moistened with a small drop of concentrated sulphuric acid, applied by means of a stirring rod, before the ignition. The aluminium and cobalt sulphates remain on the wire after the water and excess of acid are expelled and form a dull, dark blue mass when heated. Precipitated silicic acid, which is sometimes mistaken for aluminium hydroxide, does not dissolve readily in the sulphuric acid and ultimately yields a fusible, shining blue mass easy to distinguish. When a mixture of silica and aluminium hydroxide is treated, the silicic acid remains segregated and yields a bead of fusible cobalt silicate, whilst the rest of the wire is coated with the dull blue aluminate.

T. H. P.

Action of Concentrated Hydrobromic and Hydriodic Acids on the Cobalt Ion. New Reaction for Nickel. G. Deniges.

(*Compt. rend.*, 1926, 183, 55–57; cf. *ANALYST*, 1925, 50, 419.)—Substances giving characteristic absorption spectra are formed by the addition of about 10 *N* hydrobromic and hydriodic acids to salts of cobalt. With 5 c.c. of hydrobromic acid for 0.1 to 0.2 c.c. of a 5 per cent. salt solution 3 lines are seen in the orange region, λ :700,662 and $640\mu\mu$, and sensitiveness is about twice that obtained with hydrochloric acid. If to 2 c.c. of 10 *N* hydriodic acid a 5 per cent. solution of cobalt salt is gradually added a green colour is obtained, of maximum intensity when 0.7 to 0.8 c.c. of solution have been added, after which dissociation occurs. The colour corresponds to a line in the red λ : 698 $\mu\mu$, a clear interval, 705–715, absorption of the red to infra-red, two approximately equidistant bands in the green (λ , 555 and 535), and a more intense one at the beginning of the blue (λ , 517), a clear

interval to 490 and total absorption of the indigo and violet. Nickel shows no absorption bands or variations of colour with hydrochloric or hydrobromic acids, but with hydriodic acid a very sensitive colour reaction occurs. One drop of a solution of nickel sulphate solution (4 per cent. of nickel ion) added to 1 or 2 c.c. of conc. hydriodic acid results, on mixing, in the formation of an intense blood-red coloration, a reddish tint being produced by 0.1 mgrm. of nickel ion per c.c. of hydriodic acid, but dissociation starts if excess of the solution of hydriodic acid is used.

D. G. H.

Determination of Sodium. L. Barthe and E. Dufilho. (*Compt. rend.*, 1926, 182, 1470-1473.)—In the absence of organic matter and of phosphates Blanchetière's method (*ANALYST*, 1923, 48, 456) should be closely followed. Uranium is recommended for the precipitation of phosphoric acid (hot), and direct determination of sodium in mineral waters indicates that calculation of sodium from the chlorine figure may introduce considerable error. To determine sodium in milk the casein is precipitated by hydrochloric acid, the excess of acid in the filtrate neutralised with ammonium hydroxide, and a few drops of acetic acid added, followed by slight excess of Blanchetière's reagent A. After boiling and cooling, the volume is measured, and to a quantity of filtrate corresponding to a known volume of the original milk is added 10 times its volume of uranium magnesium reagent. After being mixed and left for half an hour the liquid is filtered, the filter washed with B reagent followed by 95 per cent. alcohol, heated at 110° C. for half-an-hour and weighed. If sodium bicarbonate has been added to the milk the quantity of sodium found exceeding 0.5 gm. per litre is to be regarded as added, and if dichromate has also been added, chromium will be precipitated by prolonged boiling in the presence of a large excess of ammonium chloride, introduced by adding hydrochloric acid in excess of that required for the first precipitation of casein.

D. G. H.

Analysis of Prussian Blue. F. G. A. Enna. (*J. Soc. Leather Trades Chem.*, 1926, 10, 172-174.)—Previous methods are reviewed and criticised. The author's method is as follows:—*Moisture.*—One gm. of the sample is heated in a porcelain capsule for 2 hours at 105-110° C., cooled, weighed, again heated, and weighed at hourly intervals till constant in weight. The loss should not exceed 2.5 to 3 per cent. *Total Iron.*—The sample (0.25 gm.) is treated with cold strong sulphuric acid in a porcelain capsule for 15 minutes, and then heated gently for 30 minutes at a temperature at which fumes appear, after which the mass should be white. When cool, it is washed with water into a 400 c.c. conical flask, the total volume being under 200 c.c. Any insoluble matter is collected on a tared filter-paper, well washed and weighed. The filtrate is rendered strongly acid with sulphuric acid, granulated zinc added, and the solution heated till reduction is complete. It is then diluted to 250 c.c., the usual precautions being taken to guard against oxidation. One hundred c.c. are treated with 5 c.c. of strong hydrochloric acid, and 150 to 200 c.c. of water and titrated with 0.1 *N* potassium

permanganate solution (1 c.c.=0.012128 grm. $\text{Fe}_4[\text{Fe}(\text{CN})_6]_3$). *Actual Blue*.—One grm. of the sample is treated with hot pure 5 per cent. hydrochloric acid, and well washed. The soluble iron in the filtrate is reduced with zinc and determined as above. The difference between the total iron and this soluble iron gives the amount of actual Prussian blue. It will be seen that this method will readily reveal the presence of any ultramarine, a frequent adulterant. R. F. I.

Determination of Uncombined Lime in Portland Cement. W. Larch and R. H. Bogue. (*Ind. Eng. Chem.*, 1926, 18, 739-743.)—The method described consists in titrating the lime, in alcohol-glycerol solution, with ammonium acetate solution— $\text{CaO} + 2\text{CH}_3\text{COONH}_4 = (\text{CH}_3\text{COO})_2\text{Ca} + 2\text{NH}_3 + \text{H}_2\text{O}$. The mixture is boiled during the titration, and the liberated ammonia is volatilised. The details of the method are as follows:—One grm. of the freshly-ground cement is placed in a flask containing 30 c.c. of absolute alcohol (anhydrous methylated spirit may be used), 6 c.c. of pure glycerol and 8 drops of phenolphthalein solution, the mixture is boiled, and titrated with ammonium acetate solution. The operations of boiling and titrating are repeated until the pink colour does not reappear after boiling for several minutes. The ammonium acetate solution used should contain about 16 grms. of dry ammonium acetate per litre of absolute alcohol and is standardised by titration against pure calcium oxide in alcohol and glycerol solution. W. P. S.

Determination of Soluble Silica in Cement, Mortar and Concrete. D. Florentin. (*Compt. rend.*, 1926, 183, 53-55.)—Under the following experimental conditions sand and clay furnish no appreciable quantities of soluble silica: One grm. of cement (4 grms. of mortar) is added in small portions to 50-60 c.c. of cold hydrochloric acid (sp. gr. 1.12), the temperature being kept down by a stream of cold water. The small portion of insoluble impurities is filtered off, and the silica rendered insoluble by the usual methods. If a small proportion of silica is precipitated, the residue is treated with a hot 5 to 6 per cent. solution of sodium carbonate, and the solution added to the filtrate. The proportion of soluble silica in the original cement being known, that soluble in the mortar can be deduced. Artificial Portland cements contain 20 to 21 per cent. of soluble silica. D. G. H.

Physical Methods, Apparatus, etc.

Chemical Reactions and Volumetric Titrations in Wood's Light. R. Mellet and M. A. Bischoff. (*Compt. rend.*, 1926, 182, 1616-1619.)—The fluorescence of salts of quinine in filtered ultra-violet light, and the non-fluorescence of the base itself, enable this substance to be used as an indicator in acid or base titrations. The fluorescence varies sharply in its nature for various ranges of concentrations of acid, but depends on the specific action of the acid used, and not wholly on the hydrogen ion concentration. Titrations of hydrochloric, sulphuric and oxalic acids with sodium hydroxide have been carried out, but the

use of ammonia is not successful. The successive neutralisations of the two first hydrogen atoms of ortho-phosphoric acid may be followed by these means, and the method is also suggested for use with coloured and turbid liquids. J. G.

Fluorescence of Acetone Extracts of Tanning Materials. L. Meunier and A. Jamet. (*J. Soc. Leather Trades Chem.*, 1926, 10, 166-168.)—Continuing their work on this subject (*ANALYST*, 1925, 50, 641; 1926, 54) the authors show that the yellow fluorescence is characteristic of all catechol tans if a more sensitive mode of operation is employed. The ground tannin material, or the dry powdered residue from an aqueous extract, is shaken in a stoppered test-tube from time to time with 5 c.c. of acetone under the rays from a Wood lamp. Quebracho and tizerah immediately produce the characteristic brilliant yellow fluorescence. Mimosa, cachou and gambier, give at first a violet fluorescence, becoming paler and eventually yellow, though not so brilliant. Pine and hemlock give similar results, the fluorescence being fainter, but distinctly visible if in certain dilutions. Mangrove, although classed as a catechol tan, seems to be an exception to the rule, giving a fluorescence in which the yellow tints are only very slightly visible. Pyrogallol tans, such as chestnut and oak woods, myrobalans, valonia, divi-divi and various galls, give a characteristic violet fluorescence. Plants containing a mixture of the two classes of tans (*e.g.* oak-bark, gonakie) give a bluish-white fluorescence. Lastly, leaves which contain tannin (sumac, arbutus, lentiscus) give up chlorophyll to the acetone, and this produces a dark red fluorescence. Dried extracts of the leaves do not give this effect. The best results are obtained by making the acetone extract as concentrated as possible and pouring it, drop by drop, into pure acetone. R. F. I.

Determination of Absorptive Powers of Charcoals. Surun. (*Comptes rend.*, 1926, 183, 44-46.)—By shaking 50 c.c. of a 0.01 *N* solution of a suitable substance, such as resorcinol, phenol, choral, etc., for half-an-hour in a series of flasks with increasing quantities of the charcoal until the filtrate contained less of the substance than could be detected by a convenient test, it was found that the quantity of charcoal required to bring this about varied with its source and with the substance used for absorption. The method can be conveniently used to compare absorptive powers of different charcoals by taking the quantity of a given substance absorbed by one charcoal as a standard. D. G. H.

Adsorbing Properties of Cellulose Compounds. M. J. Duclaux. (*Rev. Gen. Colloid.*, 1926, 4, 137-142.)—The use of nitrocellulose as an adsorbing agent, similar to charcoal, is described. It is best used in layers forming the bed of an ultra-filter, as this enables larger volumes of liquid to be used, ensures speedy equilibrium between the adsorbent and the solution, and enables the course of the filtration to be followed when the filter is subsequently unpacked. The method is used for the separation of mixtures of dyes, when the lower layers of the filter are coloured with the dye present in excess. The dye may then be

removed in a suitable solvent, or the whole layer mounted and examined spectroscopically by transmitted light. In spite of the negative nature of the nitrocellulose micelle, its adsorbing action is not wholly confined to basic radicals. The fixation of substances dissolved in very dilute solutions may also be effected; the filter will decolorise a solution containing 1/25,000 part of methylene blue, adsorbing 5 per cent. of its weight of dye. J. G.

Analytical Balance without Rider. A. Wirth. (*Chem. Zeit.*, 1926, 50, 438.)—In place of a rider, use may be made of a rod arranged vertically on the beam of the balance. This rod may be rotated from the outside of the closed case, the rod and beam being automatically freed after each displacement of the rod. Since the mass centre of the rod lies outside its axis of rotation, the leverage of the weight of the rod is altered by its rotation, which is read on a scale. The sensitiveness and accuracy are similar to those of an ordinary balance, but manipulation is simplified and weighing accelerated, since the position of the rod may be adjusted while the beam is swinging. T. H. P.

References to Scientific Articles not Abstracted.

THE DISCOVERY OF EKA- AND DVI-MANGANESE. By J. G. F. Druce. *Chem. Weekblad*, 1926, 23, 318.

Description in English of author's method of isolating Mendeljéef's eka- and dvi-manganese from manganese ores—These elements now known as mazurium and rhenium—Results of X-ray spectroscopic analysis—Attempts to prepare pure compounds.

OBSERVATIONS ON THE PHARMACOLOGY OF SPRINGS AND PEATS. By Prof. Zörkendörfer *Arch. Med. Hydrology*, 1926, 4, 165 (August).

Pharmacological action of the mineral waters—The Glauber salt springs—The carbonated alkaline earth springs—Chalybeate waters—Carbonic acid baths—Peat baths—Combined effect of the various forms of spa treatment.

TOBACCO FROM PALESTINE, NIGERIA AND MAURITIUS. *Bull. Imp. Inst.*, 1926, 24, p. 187 (August).

Description—Analyses—Smoking trials.

Reviews.

INTERNATIONAL CRITICAL TABLES OF NUMERICAL DATA OF PHYSICS, CHEMISTRY AND TECHNOLOGY. (Produced under the direction of the National Academy of Sciences and the National Research Council.) Vol. I, pp. 413. New York and London: McGraw-Hill Book Co., Inc. 1926. Price for the set of 5 vols, \$60.

There are few workers in any part of the field of natural philosophy who do not at times urgently need exact numerical data relating to chemistry or physics.

Few books are devoted exclusively to such data. "Landolt and Börnstein" is full, good and expensive. The data are well set out, but frequently many values are given for one constant, and it is difficult to choose the best value without referring to the original papers, which may not be available. Kaye and Laby have issued a small and excellent book, the price of which has been more than trebled in ten years. The great work, the first volume of which is under review, differs from the two works mentioned and, in a sense, resembles Castell-Evans' work in that it is critical. It is not, however, the unaided work of one man who had a passion for computation. It "is the result of the co-operative labours of a large number of specialists, each of whom has been charged with the responsibility for the critical compilation of the quantitative information available on his topic. The word critical in this connection means that the co-operating expert was requested to give in each instance the 'best' value which he could derive from all the information available, together, where possible, with an indication of its probable reliability."

It is interesting to note, as illustrating the difference between physical and chemical data, on the one hand, and purely numerical data, on the other, that, whereas tables such as these, even although they are "critical," are likely to begin to be out of date as soon as the final proof is passed, Vega's 7-figure logarithmic tables were used in computing the conversion tables. Vega's last edition was published in 1794, and was a revision of Vlacq's tables published in the previous century.

The tables proper begin with a review by E. C. Guillaume and C. Volet of the weights and measures of the world, literally "from China to Peru," and including much information on the weights and measures of antiquity. Very comprehensive conversion tables with logarithms of the factors will be welcomed by many. Analysts, in particular, will find the graph on p. 33 for converting the results of various technical viscometers into terms of one another and into c.g.s. units very useful. The glossary of technical terms by N. E. Dorsey, with definitions and dimensional formulae, should save much hunting in text books. Much information is given as to the grosser properties of atoms and the structure of the isolated atom.

The group of monographs on laboratory technique includes thermometry, the production and maintenance of constant temperatures, high and low, methods for maintaining constant humidity, useful in investigating the "natural" moisture content of such materials as coal, flour, textiles, etc. The volume of liquid menisci is a matter of frequent interest to the worker with gases. Weights and weighing are usually too much taken for granted by chemists, who will find much exact information here. Nearly two hundred indicators and their properties and several groups of buffer solutions are described by Mansfield Clark.

A very valuable article on Errors of Observation considered, not from the view of the calculus of probability, but as psychological phenomena, is contributed by R. S. Woodworth. The data mostly refer to vision, but sound perception,

time of reaction to stimulus, discrimination of differences of weight and temperature and number limitation are dealt with. It appears that a fine dark line on a light ground is more easily discriminated than a light one on a dark ground. Aligning power, as in setting a vernier, exceeds the power of resolving two points. Subdivision of a scale division 1 mm. long is easy to 0.1 division, but attempts at finer division are mere guesses. This article is of special interest to readers of *THE ANALYST* in view of Dr. Tocher's recent paper.

About half of the volume is taken up with tables of the physical properties of the elements, of air and of 3359 inorganic and 6175 organic compounds. Each substance is identified by an index number, and each element in a compound is given a key number, so that by a very simple system a substance can be looked up by means of its formula or in an index by its name. The index number characterises the substance in tables in this volume and in those which are to follow. Tables in ascending order of temperature of melting and boiling points and similar tables of density facilitate the identification of pure substances.

Quantitative information as to the sweetening power of sweetening agents, from lactose, which is 0.27 to 0.28 as sweet as cane sugar, to perillaldehyde and anti-aldoxime, which is 2000 times as sweet, introduce a rather novel feature, which is followed up by a considerable amount of information as to odours, including values for many odoriferous materials given in molecules per c.c. One notes that 16-32 hundred thousand molecules of ionone per c.c. of air are detectable, whilst 17,000 billions of apiol molecules are needed for detection. These data are undoubtedly of a very low order of precision, but they represent a real beginning in the scientific investigation of taste and smell.

It is a difficult task to discuss the value of data collected in as authoritative a manner as the contents of this volume have been. Nevertheless, a few points for consideration may be indicated. The specific gravity of ice at 0°C. is given as 0.917 Leduc in 1906 came to the conclusion that 0.9176 was the most probable value. His work was very carefully done, and I know of none later. The value in the conversion tables for the 20° calorie = 4.181 joule is only 1 in 4000 different from Laby's weighted mean of what he considered (1926)* the best values, but the ratio $20^\circ \text{calorie}/\text{mean calorie}$ deduced from the data on the same page is $4.181/4.186 = 1.0012$, whereas Ezer Griffiths and Awbery have stated that, in data prepared for these tables they adopt 1.002₂. Some of the latent heats of metals (*e.g.* aluminium) differ rather from the values recently obtained by these same workers (G. and A.) in a very large calorimeter. One notes that, like most of us, the contributors occasionally refer to the once familiar *c.c.* rather than to the *ml.*

A work containing data as to the stars, the solar system, the earth and its contents, down to individual atoms, presented in the authoritative and attractive manner of this volume is useful—almost indispensable—to all chemists and physicists. The price alone is likely to repel most individual workers. It is difficult to see how it could be reduced, unless the hope of the trustees that it may

* The figure in Laby's paper differs from that in the abstract by 0.002. I quote from the paper.
J. H. C.

be endowed is fulfilled. The only other scientific object equally worthy for commendation to millionaires is the augmentation of the payment of university scientific staffs. Books of this kind are useful in a reference library, but they are many times more useful on the chemist's own bookshelves.

J. H. COSTE.

A TEXT-BOOK OF ORGANIC CHEMISTRY. By Dr. JULIUS SCHMIDT. English Edition by H. GORDON RULE, D.Sc. Pp. 798+xxiv. London: Gurney & Jackson. 1926. Price 25s. net.

A translation of this excellent text-book has long been needed. It is a valuable reference work, and yet no mere dictionary, for it is thoroughly interesting and readable in every part. The translator has revised the text of the German edition, added much new material, and inserted many references to English and American authors.

The subject matter is treated under four headings, viz., a general section, aliphatic compounds, carbocyclic compounds and heterocyclic compounds. The general part treats of analysis, formulae, molecular weight determinations, molecular structure, isomerism, etc., and gives interesting summaries of such subjects as optical activity and the stereochemistry of nitrogen. The concluding part of this section deals with the nomenclature of organic compounds and might, with advantage, be enlarged. From this it is at once evident that to appreciate this book the reader must have a sound knowledge of the elements of the subject. In fact, Schmidt's *Organic Chemistry* is no book for the novice, but is rather a work for advanced students.

The aliphatic section (Part I) begins by discussing paraffin hydrocarbons, both saturated and unsaturated, and proceeds systematically through the various groups of compounds, giving prominence to substances which, although of great importance in nature, are often left out in ordinary text-books.

In Part II. the cyclo-paraffins receive a very generous share of the space available, and additional interest is created by brief accounts of the chemistry of such substances as rubber. Throughout the book references are given which will allow the reader to satiate the curiosity that the text inspires. Benzene and its derivatives naturally take up much space, but, nevertheless, the hydro-aromatic compounds are not crowded out. Polynuclear hydrocarbons are treated fully, rare ones such as pyrene and picene receiving mention.

The final section of the book contains an excellent account of the chemistry of the heterocyclic compounds, alkaloids, proteins and plant pigments, and good subject and author indexes complete the volume.

Schmidt's *Organic Chemistry* is comprehensive and well suited to advanced students. It is not encumbered with too much detail, but gives broad outlines in an extremely interesting way. For the most part, the subject matter is correct, but here and there it is a little incomplete. For instance, in dealing with the unsaturated aliphatic acids, oleic and sorbic acids are treated fully, but the more highly unsaturated acids which occur in such quantities in animal and vegetable

oils are not even mentioned. Also, in the aromatic section a little more might be said concerning the preparation of *m*-nitrotoluene. In spite of minor defects, the book stimulates thought and, so it appears to the reviewer, has a distinct bias towards that branch of chemistry which probably will be the subject of much research in future years, namely, the chemistry of natural products. The translator has done his work so well that the awkward sentences which characterise the German language have been turned into such lucid English that it is difficult to believe that it is a translation. The publishers, too, have done their share by producing a well-bound, clearly-printed, book, which deserves a place in every chemical library and can be thoroughly recommended to all advanced students in both chemistry and biochemistry.

HAROLD TOMS.

PRACTICAL PHOTOMICROGRAPHY. By J. E. BARNARD, F.R.S. and F. V. WELCH.
Second Edition. Pp. xii.+316. London: E. Arnold & Co. 1925.
Price 18s.

The authors have undertaken the task of revising and extending the first edition of this work published fourteen years ago, and from their wide experience have compiled a manual which is perhaps of greater value to the photomicrographer than any other volume dealing with the subject at the present time. The reader is assumed to be more or less familiar with the manipulation of the microscope for visual purposes, thus allowing of greater space being given to the subject denoted by the title.

The contents of the volume are subdivided under the following headings: The Microscope, Optical Equipment, Sources of Illumination, Photo-micrographic Cameras, Use and Manipulation of the Microscope, Preliminary Preparations, Colour Screens, Plates and Exposure, Photographic Processes, and Special Processes. Under the last heading are described the methods employed in photomicrography with ultra-violet light and X-rays, photography of opaque micro objects, bacterial tube and plate cultures, stereophotomicrography and the production of lantern slides in colour. Chapter XII introduces an exceedingly valuable feature entitled "Some Progressive Examples," and comprising a series of forty photomicrographs ranging in magnification from $\times 20$ to $\times 2700$, whilst appended to each are full details of the apparatus and procedure employed and useful comments upon the method or the result obtained. A series of appendices is provided containing various data, formulæ and a bibliography relevant to the subject, and the combined name and subject index at the end of the volume is both adequate and accurate, an exhaustive test having failed to detect an incorrect reference therein.

The book is well printed in legible type, and the illustrations, both diagrammatic and otherwise, are clear, whilst the numerous plates bear a variety of photomicrographs, bacterial cultures, etc., which exhibit the outstanding qualities one has learned to associate with the authors' productions.

The volume is essentially practical in character, and throughout the text is explicit and to the point without the inelegancies of diction often noticed in a work

of this nature. The value of the book is greatly enhanced by the inclusion of many minute details of procedure derived from the authors' experience, but which are frequently omitted from similar productions, although such details contribute greatly to ease of procedure and final success.

On the whole, the text is commendably free from errors, although a few of minor importance have crept in. Thus, on p. 14, Van Heurck is given as Van Heurch, and two pages further on reference is made to "an axplanatic condenser with a numerical aperture of 1-40," whilst the first line on p. 257 appears to have the word "focussed" omitted. The *Journal of the Royal Microscopical Society* is stated, on p. 306, to be published "every two months," but up to the present time this periodical has appeared quarterly. Some experienced photographers will probably differ from the authors on a few minor points of procedure, particularly the use of a plain "hypo" fixing bath, the suitability of mercury salts for intensification, and the inability to mount glazed prints with aqueous mountant without damage.

Although intended for the use of photomicrographers, the volume contains much information of value to the ordinary microscopist who has no desire to make use of photography in his work, especially as emphasis is laid upon the simplification of apparatus, for which reason description of apparatus is subordinated to that of method throughout the text.

The authors have carried out their task in an admirable manner, and have been ably seconded by the publishers in the production of a work which should be in frequent demand and well deserves a place on the shelves of every photomicrographer.

T. J. WARD.

PHYSICO-CHEMICAL METHODS. By JOSEPH REILLY, D.Sc., F.I.C., WILLIAM NORMAN RAE, M.A., F.I.C., and THOMAS SHERLOCK WHEELER, Ph.D., F.I.C. Pp. xi.+735. London: Methuen & Co. 1926. Price 30s. net.

This book represents a new departure in the writing of text books on practical physical chemistry. It caters exclusively for the man who has had some considerable training in physico-chemical methods, and will probably reach its maximum of utility in the case of a chemist who, after one or two years of research training, finds himself left to his own resources in a work's laboratory with few or no facilities for consulting the literature. At the same time, its thorough treatment of some branches of the subject and its frequent allusions to original papers make it a valuable work of reference for the physical chemist of wider experience and resources.

The book is somewhat arbitrarily divided into ten sections, the first of which deals with the evaluation of experimental results. It treats (perhaps with unnecessary elaboration) of the calculation of probable error, the solution of simultaneous equations by the method of least squares, the use of the slide rule and graphical methods for the expression of experimental data. The next section is devoted to general laboratory design, and is followed by an account of standard laboratory operations, such as distillation, the determination of melting point,

vacuum technique, etc. In this section the chapters on distillation and on vacuum pumps seem excellent. It would appear, however, that the authors have made a serious omission in failing to treat in detail the determination of setting points. Phase rule methods are in common technical use, and the examination of setting points is of prime importance for the elucidation of the problems of condensed systems of two or three components. The fourth section, dealing with physical measurements—mass, pressure, temperature, viscosity, etc.,—is especially valuable. It is thorough, occupying about 250 pages in all, and particularly admirable are the constant emphasis of accepted standards of accuracy and the clearness with which the practical range of applicability is stated for each instrument. The calibration of volumetric apparatus and thermometers is described in detail, and the construction and use of many types of balance are very thoroughly treated.

The rest of the book seems hardly up to the same high standard of excellence. It is devoted to a description of accepted physico-chemical methods and is, as the authors themselves state, largely culled from well-known, text books on practical chemistry. In many cases sample calculations are given. These seem hardly necessary in a book of this type and might well make way for more important material. The methods given are not treated in the critical manner one might expect. For instance, in the chapter on the determination of molecular weights of dissolved substances, many modifications of Beckmann's ebullioscopic method are discussed, but no distinction is drawn between them, and no mention is made of the fact that for some of these methods the adjustment of conditions is so delicate as to make them comparatively useless. It is a pity that no mention is made of the very convenient differential vapour pressure thermometer recently developed by Menzies for this class of work.

The reviewer feels that the authors have, to some extent, spoilt the book in an attempt to make it complete. Evidence of this may be found in such chapters as those on electro-chemistry and photo-electric effect—subjects which surely merit more than the few pages allotted to them. The successful application of such methods would demand far more practical information than could be given within the confines of a work of this type, and it would seem wiser to omit such subjects entirely.

Appendices give atomic weights and numbers, corrections for exposed thread of mercury thermometers and the boiling points of liquids suitable for constant temperature jackets; surface tensions and viscosities of some common liquids are also tabulated.

W. J. SHUTT.

INTRODUCTION TO QUALITATIVE ORGANIC ANALYSIS. By HERMANN STAUDINGER Ph.D. Translated by WALTER T. K. BRAUNHOLTZ, M.A., Ph.D., F.I.C. Pp. xvi. + 112. London: Gurney and Jackson, 1925. Price 6s. 6d. net.

The difficulties of Qualitative Organic Analysis need no emphasis, and it seems doubtful if a method of procedure will ever be devised which will be both systematic and thoroughly comprehensive. Prof. Staudinger attempts the solution of the problem by making a preliminary classification of organic compounds according to their volatility and solubility. Substances are regarded as

easily or slightly volatile according as their boiling points are below or above $140^{\circ}\text{C}.$; these two classes are then sub-divided, according to the solubilities in water and ether; in this way compounds and the components of mixtures are placed in one or more of eight groups; these groups are still further sub-divided, taking into account the presence of elements other than carbon hydrogen and oxygen. Proceeding along these lines, it is possible to locate a substance so nearly that the final identification by means of physical constants and special tests should not be a lengthy business.

The first twenty pages of the book outline the principles underlying this scheme of analysis, and the remaining ninety detail the expansion and application of those principles. No lists of individual substances or tables of physical constants are given, hence the student could not identify compounds by the aid of this book alone; nevertheless, it can be recommended as likely to be of considerable assistance to anyone beginning organic analysis. No index is provided, but the book opens with a highly detailed table of contents which serves the same purpose. The printing and general style are quite satisfactory. A. F. KITCHING.

THE STATES OF AGGREGATION: THE CHANGES IN THE STATE OF MATTER IN THEIR DEPENDENCE UPON PRESSURE AND TEMPERATURE. By GUSTAV TAMMANN. Authorised translation from the Second German Edition by Robert Franklin Mehl, Ph.D. Pp. xi.+297. 128 Figs. London: Constable & Co., Ltd. 1926. Price 24s.

As the translator points out, "Professor Tammann's name is so well known in scientific circles that no apology need be made in offering an English translation to the public. The present work is a very complete outline of the equilibria in one-component systems, and includes the work for which Professor Tammann is so well known, namely, the experimental and theoretical work upon crystallisation and melting at ordinary and also at high pressures."

The book deals with a highly specialised field in physical chemistry, but the treatment is clear. A reasonable knowledge of the calculus and of thermodynamics is necessary. The value of the book lies in its appeal to those who wish to gain data from any branch of science which will assist in elucidating the nature of intermolecular forces. The student of the phase-rule and its applications cannot afford to ignore this book, with its constant use of phase diagrams. Another matter which receives full treatment is that of velocity of crystallisation, deformation of crystals, and the transition of unstable into stable states. The book concludes with a short but very interesting account of liquid crystals.

WILLIAM CLAYTON.

CHEMISTRY IN INDUSTRY. A CO-OPERATIVE WORK INTENDED TO GIVE EXAMPLES OF THE CONTRIBUTIONS MADE TO INDUSTRY BY CHEMISTRY. Edited by H. E. HOWE. Vol. I. (1924), Vol. II. (1925). Pp. 372 and 392. New York: The Chemical Foundation Inc. \$1 each.

At the present time, when specialisation is essential, there is a marked tendency for chemists of all kinds to get sadly out of touch with branches of industrial

chemistry which are not directly connected with their own particular domain. This is regrettable, though to some extent unavoidable, and one feels that scientific workers in general must welcome a work of the type of that under review, which enables them, in their leisure moments, to improve their acquaintance with fields of labour other than their own, and to bring themselves, to some extent, up to date in their general knowledge of chemistry and of its diversity of application in industry.

Those responsible, however, for "Chemistry in Industry," aim at a wider field of appreciation. Their intention is not only to tell the story of the relation of chemistry to industry, but also to tell it in such a form as to appeal to and grip the popular mind.

The two volumes contain 21 and 22 chapters respectively, each forming a monograph dealing with some particular industry, pointing out its dependence on chemistry, and sketching, more or less briefly, the principles that underlie its application to the production of articles of common life.

The authors, with the exception of a few official chemists and university professors, are mostly chemists holding important positions in big American industrial concerns. With varied success, they deal with a wide range of subjects, which include coal, cotton, fertilisers, gas, leather, paper, perfumes, petroleum, soap, and textiles. The writers differ somewhat in their conception of what the general reader wants to know, and also in their ability to give, in the space available, a succinct account of the activities of their particular sphere.

One notices occasional loose writing and undue labouring of points; the statement on p. 54 (Vol. I.), that lactic acid is sour milk, requires modification. Some of the chapters suffer from that extravagance of expression and of metaphor which is so frequently met in the pages of science for popular consumption, such as, for example, "Kekulé, dreaming of giddy molecules, gambolling in an exhibition tango."

A certain amount of overlapping occurs. An interesting chapter on "Packing-house Processes" includes nearly four pages on gelatin, which subject is extensively treated in a separate section. Similarly, nitrogen-fixation receives double attention.

Notwithstanding these and other minor defects, the result of this collaboration of experts is, in the main, an excellent one. Here the intelligent reader, with no special chemical training, will find much to interest and instruct. He will gain a clear appreciation of the basic services of chemistry in providing for modern needs and comforts, realising, in addition, something of the part played by science in the making of history, of the frequent dependence of political and international problems on the chemical factor.

To the chemical student, "Chemistry in Industry" should prove fascinating and useful, and chemists in general will find its perusal (with, perhaps, a little judicious skimming), more than worth their while.

A. F. LERRIGO.