THE ANALYST

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

An Ordinary Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, April 7th, 1926, Mr. E. Richards Bolton, F.I.C., President, being in the chair.

Certificates were read for the first time in favour of Messrs. Arthur Barraclough, B.Sc., A.I.C., George Gordon Elkington, Kenneth Massy Griffin, M.Sc., A.I.C., Herbert Firth, A.I.C., and Thomas Pickerill, B.Sc.

Certificates were read for the second time in favour of Messrs. John Allan, Michael Thomas Casey, M.Sc., George Henry Davis, Julius Grant, M.Sc., A.I.C., and Miss Monica Mary Ruston, B.Sc., F.I.C.

The following were elected Members of the Society:—Messrs. Sydney Back, B.Sc., A.I.C., Howard Henry Bagnall, B.Sc., F.I.C., William Percival Crocker, Bernard William Alfred Crutchlow, B.Sc., A.I.C., Alec Munro Ferguson, M.A., B.Sc., A.I.C., Ralph Henry Klein, A.I.C., Oswald James Napier, M.A., A.I.C., George Stubbs, C.B.E., F.I.C., Joseph Hughes Williams, B.Sc., F.I.C., Kenneth Alan Williams, B.Sc., A.I.C.

The following papers were read and discussed:—"The Analysis of Acetic Anhydride," by H. Droop Richmond, F.I.C., and J. A. Eggleston, B.Sc.; "The Analysis of Glacial Acetic Acid," by H. Droop Richmond, F.I.C., and E. H. England; (i) "Errors of Personal Judgment in Chemical Work," (ii) "Results of Further Work on Variations in the Composition of Milk," by J. F. Tocher, D.Sc., F.I.C.; and "A Test for Tung Oil," by E. Richards Bolton, F.I.C., and K. A. Williams, B.Sc., A.I.C.

Obituary.

SIR J. B. HARRISON, C.M.G.

In the death of Sir John Burchmore Harrison there passed away one who, though probably little known in this country and only to some of the older generation,

was a great personality in the colony of British Guiana, where he spent the greater part of his active life, and where he died on February 8, 1926.

Born in Birmingham in 1856, and graduating from Christ's College, Cambridge, in 1878 (Nat. Science Tripos), he was appointed, in 1879, Professor of Chemistry and Agricultural Science in Barbados. This post he held until 1889, when he was transferred to British Guiana, which he henceforth made his home, only visiting the old country at long intervals. In 1889 he took up the position of Government Analyst and Professor of Chemistry, British Guiana, and subsequently, in 1904, was appointed Director of the Department of Science and Art.

Harrison, in addition to being a good chemist, was distinguished by his great versatility and aptitude for taking up other branches of science—so that, not chemistry only, but other subjects also, came under his purview and activity. Notably was this the case with geology, and he made several valuable contributions to the geological knowledge of the West Indian Islands and of his own Colony, in recognition of which he was, in 1899, given an award from the Wollaston Fund of the Geological Society of London.

His chief work in British Guiana was, however, in connection with the development of the staple industry—sugar cultivation—and he was instrumental in experimenting on and introducing new and better varieties of sugar-cane, and in improving the output of sugar. He also published many reports on the chemistry of the vegetable products of the colony.

Though never possessing robust health, Harrison succeeded in standing the exigencies of a tropical climate for over 40 years, in the course of which he survived an attempt made in his own laboratory to poison him (with arsenic; *cf.* ANALYST, 1906, **31**, 315); and, in later years, a severe operation—both of which necessitated a return home, though only to find him again, and somewhat unexpectedly, at his old post in the Colony.

He was created C.M.G. in 1907, appointed a member of the Executive Counci. of British Guiana in 1918, and knighted in 1921.

An Accurate Method for the Determination of Mercury in Solution.*

By B. S. EVANS, Ph.D., F.I.C., and S. G. CLARKE, B.Sc., A.I.C.

(Read at the Meeting, March 3, 1926.)

THE process described below was worked out to provide a rapid and accurate method of determining mercury in the form of mercurous nitrate in presence of relatively large amounts of copper and zinc nitrates. The analytical chemistry

* Communication from the Research Department, Woolwich.

of mercury is a wide subject, involving, as it does, a very large number of papers; considering these, it occurred to the authors to re-investigate the possibility of complete removal of mercury by deposition on copper and its subsequent determination by sublimation.

PREVIOUS METHODS OF DEPOSITION AND SUBLIMATION.—A great deal of work has already been done on this method, but the results are, in the main, unsatisfactory. In the first place the quantities dealt with, are, as a rule, very small; in the second, the fact that with larger quantities, at any rate, the mercury has a strong tendency both to detach itself from the copper and to precipitate, presumably as basic salts in the liquid, has not adequately been dealt with. The most recent paper that could be found was that of Gordon (ANALYST, 1920, 45, 41). This author used coils of copper wire which were rotated in the solution, thus stirring it; he dealt only with quantities of the order of 0·005 grms., because he found that larger quantities required so much copper that the weight of the coil stopped his motor.

The only outstanding work on larger quantities by this method appears to be that of T. W. Richards of Harvard (Richards and Singer, *J. Amer. Chem. Soc.*, 1904, p. 300). That author used copper wire coils, leaving one in the solution for five hours, and then removing and replacing it by another for twenty hours; the mercury was then determined by the loss in weight of the copper when heated in hydrogen. The time occupied by this determination is prohibitive, and, in addition to this, volatilisation of the copper appears to introduce an error if the temperature is taken much above 350° C. Richards notes the formation of a precipitate in sulphuric acid solutions; he tests his method with quantities up to 0·03 grm. of mercury.

CONDITIONS FOR QUANTITATIVE DEPOSITION.—Work done on the Reinsch test seemed to indicate that the separation on copper should give accurate results if the right conditions were discovered. Preliminary experiments were carried out, both with hot and cold solutions, various acids being used, and attempts were made to determine the mercury by sublimation. At first the mercury was deposited on foil; later this was abandoned in favour of coils of wire. The results, in the main, were completely untrustworthy, but two or three facts emerged:— (a) Hot solutions always gave lower results than cold. (b) In marked contrast to the Reinsch tests for arsenic, etc., hydrochloric acid was a distinct hindrance rather than help (this was attributed to precipitation of mercurous chloride), whilst the observations of Richards and also of Gordon (*loc. cit.*) on the non-interference of nitric acid were confirmed. (c) The results became worse as the quantity of mercury was increased.

Thinner wire was next used, and the solution was stirred. With this arrangement it proved possible to determine quantities of mercury up to 0·02 grm. with fair certainty and accuracy; above this amount, however, the method broke down, and a grey precipitate collected at the bottom. The result also appeared to be less reliable in the presence of large amounts of copper salts.

At this stage it became obvious that for the process to be available for larger amounts of mercury the following conditions must be fulfilled:—(a) The surface of copper must be as large as possible. (b) The solution must be cold. (c) Filtration of some sort is necessary, the precipitate being added to the mercury on the copper. (d) The time of contact must be reasonably long. (e) Agitation of some kind is desirable.

PERCOLATION THROUGH COPPER. Filtration through a bed of copper powder was the obvious answer to most of these demands, but continuously passing the same solution through a filter entailed a good deal of labour and the requisite time of contact could not be ensured in any other way. For this reason the apparatus for continuous percolation described in the following paper (Evans, *infra*, p. 229) was devised, and with the aid of this a cold solution can be kept circulating over a bed of copper as long as may be desired, the copper with its deposited mercury and any precipitate that may form being kept within the limits of a small detachable tube.

ANALYTICAL PROCESS.—The details of the process, as finally worked out, are as follows:—A small filter tube of convenient dimensions (say, length 5 cm. and bore 0·6 cm.), is prepared, one end being slightly constricted, and it is packed in the following manner:—A pad of glass wool is pressed into the tube up to the constricted end, which is then covered to the required depth (say, 1 inch) with copper filings which have been passed through a 60-mesh sieve. The copper is cleaned with dilute nitric acid and washed with water, which may readily be done by attaching the tube to a filter pump and sucking the liquids through.

The filter tube is attached to the bottom of the main tapped funnel of the apparatus with rubber tubing so that it hangs down inside the flask; it is desirable to set it at an angle, so that the drops fall quite clear of the return tube, thus ensuring efficient circulation.

The solution to be tested should be made acid with either sulphuric or nitric acid (hydrochloric acid, if present in any considerable amount, must be eliminated); it is neutralised with ammonia and treated with about 2 c.c. of nitric acid per 100 c.c. of liquid; the total volume must naturally be such as can conveniently be dealt with in the apparatus. The prepared solution is run into the main funnel (the tap of which is closed) through the small funnel at the top, which is afterwards rinsed; the small funnel tap is then closed, the main tap opened, and the whole amount of liquid allowed to filter into the flask. This can be considerably hastened by attaching a filter pump to the lower exit tube, the tap of the return tube being closed (it is desirable, however, that filtration should not be too rapid).

When all the solution has been filtered the main tap is closed, the return tube tap opened, and the liquid re-transferred to the main funnel by detaching the filter pump and attaching it to the top exit tube; if the main tap is now opened the liquid will continue to percolate until it is considered desirable to stop it.

Deposition should be complete in 1½ to 2 hours, and at the end of this time the pump is detached from the top exit tube and attached to the lower one, and the

solution is allowed to filter into the flask. So long as the tap of the return tube is fully open no suction is applied to the liquid filtering. As filtration is often slow, however, it may be advisable to hasten it with a slight suction, but this must be done very cautiously, as the mercury is readily drawn through the filter.

A sensitive means of regulating the suction is available in the tap of the return tube; if the tap is partly closed any desired degree of suction may be applied to the filter, the remainder of the suction being "shunted" through the return tube. When the solution has all been filtered the filter is washed twice with water run in through the top funnel.

The filter tube is now detached, placed in a hole in the rubber stopper of a filter flask, and water removed by washing two or three times with small quantities of acetone, the excess of which is drawn off by very gentle suction. The filter, together with the copper carrying the mercury, is next transferred to a small porcelain crucible. This is effected by pushing a stout wire through the constricted end of the filter tube, when it will be found that the filter wipes any trace of mercury from the walls of the tube in its passage.

The crucible is then placed in a warm spot (e.g. on an asbestos plate on a steam bath) for a few minutes until all smell of acetone has disappeared. Care must be taken that the crucible does not become more than warm to the hand during this operation.

Sublimation of the Mercury.—The sublimation of the mercury can be carried out by a modification of the Eschka and Holloway process. The crucible is fitted tightly into a round hole in a piece of asbestos board, about 5 inches square, which rests on a tripod; a weighed platinum dish (about 50 c.c. in capacity) is placed on the mouth of the crucible, which it should fit fairly closely; the dish is filled with cold water, and a small bunsen flame is applied to the crucible, sufficient to keep the bottom at dull red heat; it is desirable to shield the flame by plates of asbestos board placed round the tripod. When the water in the dish becomes warm it is run out through a small siphon, cold water at the same time being run in; it may be necessary to do this four or five times during the sublimation. At the end of twenty minutes, which should be sufficient for the sublimation of quantities up to 0·1 grm., the gas is turned off and the crucible allowed to cool, the water being kept cold as before.

When the crucible is no more than warm to the hand the water is siphoned out of the dish as completely as possible, and the dish carefully lifted off the crucible and immediately turned upside down. The mercury will be found adhering to the bottom. The inside of the dish is rinsed with acetone, which is then poured out. The inside and sides of the dish are dried as completely as possible with a cloth, great care being taken not to touch the patch covered with mercury; the dish is placed upside down in a desiccator for half an hour and then weighed still upside down. With larger quantities of mercury it is desirable to place a fresh dish on the crucible and re-heat as before, to be sure that all mercury has been collected.

RESULTS OF TEST EXPERIMENTS.—The following results were obtained with solutions of mercuric nitrate to which copper nitrate had been added.

Weight of mercury taken. Grm.	Weight of cupric nitrate added. Grms.	Weight of mercury recovered. Grm.
0·0053	5·0	0·0053
0·0106	5·0	0·0107
0·0264	5·0	0·0266
0·0528	5·0	0·0525
0·0759	5·0	0·0758
0·1056	5·0	0·1050
0·5046	5·0	0·5024

The details of the last experiment are of interest. Re-distilled mercury (0·5046 grm.) was dissolved in nitric acid and treated as described above. The solution was percolated for $1\frac{1}{2}$ hours through a bed of copper about an inch deep and weighing about 3·7 grms. (this may be compared with Gordon's 4 to 7 grm. coil for 5 mgrms. of mercury). Care was taken not to let the liquid filter too fast, as the finely divided mercury is rather easy to wash through the filter. The filtrate was quite bright and, on being tested with a copper strip, showed no trace of mercury.

The mercury was sublimed for half-an-hour at a fair heat, the first weight obtained being 0·5011 grm.; the crucible was then reheated and yielded a further 0·0013 grm., a total of 0·5024 grm., this being a total yield of 99·6 per cent. It would seem that the loss of two mgrms. occurred during sublimation, and that a more cautious application of heat might have given a higher result.

A few experiments were carried out in sulphuric acid solutions; the mercury was in the form of mercuric sulphate, the copper as cupric sulphate, and the solutions were made strongly acid with sulphuric acid.

	Weight of mercury taken. Grm.	Weight of cupric sulphate added. Grms.	Strength of H_2SO_4. Per Cent.	Time of percolation. Hours.	Weight of mercury recovered. Grm.
(a)	0·0250	5·0	25	$1\frac{1}{2}$	0·0245
(b)	0·0500	5·0	25	$1\frac{1}{2}$	0·0496
(c)	0·1000	5·0	25	$1\frac{1}{2}$	0·0993

Experiments (a) and (c) suffered slight mishaps which may have made the results a trifle low.

RESULTS WITH HYDROCHLORIC ACID.—The method was tested with hydrochloric acid solutions, with entirely hopeless results, e.g.

Mercury taken. Grm.	Mercury recovered. Grm.
0·050	0·008
0·025	0·020

This was the more unexpected, as one of the authors had shown (Evans, "Chemistry of the Reinsch Test," ANALYST, 1923, 48, 357 and 417) that the exact contrary

holds good for arsenic, antimony and bismuth. It would seem that the cause of the trouble is cuprous chloride which, depositing on the copper, prevents the access of the mercury. It is necessary, therefore, to eliminate hydrochloric acid if present; this should not be difficult, as Winkler (*Zeitsch. anal. Chem.*, 1924, 262) has shewn that mercury salts may be evaporated on the water bath without loss in the presence of sulphuric acid and potassium chloride.

EFFECT OF OTHER METALS.—It follows from the results given above that Gordon's fear (*loc. cit.*) of the interference of arsenic, antimony and bismuth is without foundation, for three reasons (Evans, *loc. cit.*):—

(*a*) Arsenic, antimony and bismuth do not deposit on copper in the absence of hydrochloric acid. (*b*) They are inhibited from depositing by the presence of nitric acid. (*c*) They are inhibited also by cupric salts.

It would seem therefore that, with the doubtful exceptions of silver, gold, etc., which are hardly likely to sublime at the temperature used, the reaction is specific for mercury.

DETERMINATION IN VERY DILUTE SOLUTIONS.—To illustrate the great dilutions at which determinations can be made, the following experiments may be cited:—

Volume of solution. C.c.	Weight of mercury added. Grm.	Mercury found. Grm.	Parts per million.	
			Added,	Found.
1500	0·00505	0·0052	3·4	3·5
7000	0·00505	0·0053	0·72	0·76

In these cases the flask of the apparatus was replaced by a large bottle of the required capacity, and a tube was attached to the free end of the filter tube leading to the bottom of the bottle. The liquid was allowed to circulate for several hours; otherwise the determinations were carried out as usual.

An Apparatus for Continuous Percolation and for Filtration in Neutral Atmospheres.*

BY B. S. EVANS, Ph.D., F.I.C.

(*Read at the Meeting, March 3, 1926.*)

IT frequently happens in analytical chemistry that it is desirable to bring a liquid into intimate contact with the surface of a solid, and the greater the surface the higher its efficiency for the purpose required. A case in point is the "Jones Reductor." It so happens, however, that precisely that form of solid which exposes the greatest surface, *i.e.* a powder, is often the one which is most difficult to bring into intimate connection with the whole of the liquid or else, for various reasons,

to deal with afterwards. Thus lead powder would, presumably, be more effectiv
than foil for the reduction of tin, but one is precluded from filtering owing t
danger of oxidation by the air. The ideal case would be one where the liqui
could be continuously filtered through a bed of the powder in question. Th
labour involved in doing this by hand for any length of time being very great, th
apparatus described below was devised for accomplishing it automatically.
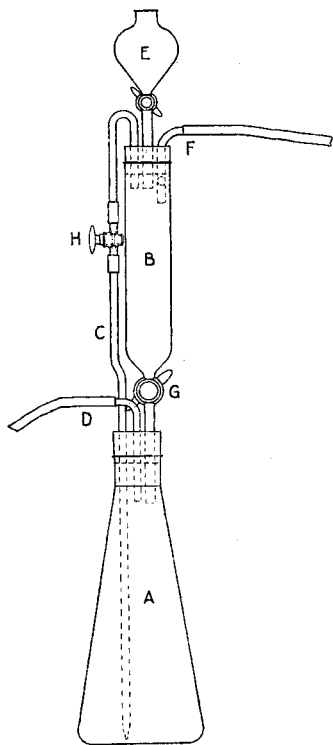
CONSTRUCTION OF APPARATUS.—The apparatu
in its simplest form consists of:

(1) A flask A carrying a rubber stopper pierce
with three holes. Passing through these three hole
are:—

(2) The main funnel B, a cylindrical funne
(*e.g.* a separating funnel with a wide mouth or on
with the top cut off). The stem of this funnel reache
an inch or two below the stopper.

(3) The "return tube," C, a glass tube (the lowe
end of which is drawn out into a jet), passing from th
bottom of the flask, bent in such a way as to ru
parallel and close to the side of the main funnel, agai
bent over at the top and passing through the rubbe
stopper closing the mouth of the main funnel. And

(4) The "lower exit tube" D, a glass tube bent
at right angles, each limb being about 3 inches long

The mouth of the main funnel is closed with a
rubber stopper, also pierced with three holes, throug
which pass:—

(5) The upper bent back end of the return tube,

(6) A small tapped funnel E, serving to admit
any required liquid to the apparatus, and

(7) The "Upper Exit Tube" F. This is similar to the lower exit tube,
but has a hole blown in the limb which passes through the stopper about $\frac{1}{2}$ in.
from the end and facing the inner side of the right angle made by the tube; *i.e.*
it faces the wall of the main funnel and away from the end of the return tube.

A short glass tube open at both ends and with one end constricted is packed
• with the material which it is desired to percolate, held in position by a wad of glass
wool or similar material, which rests against the constricted end. This tube is
attached with rubber tubing to the end of the stem of the main funnel in such a
way that it hangs down into the flask, the constricted end downwards.

If the upper exit tube is now attached to a filter pump and the liquid which
it is proposed to use for percolating is run into the main funnel through the top
funnel, the tap of the latter being then closed, the following sequence of events
will be noted:—

(a) The liquid will filter through the packed tube into the flask, since the open return tube ensures that the pressure in the flask and in the main funnel are the same.

(b) As soon as enough liquid has collected in the flask to block the drawn-out end of the return tube it is drawn up that tube, since the closing of it establishes a difference of pressure.

(c) The amount of liquid collected having been only just sufficient to touch the return tube, it does not fill that tube, but is immediately followed by air bubbles which drive it right up the tube and back into the main funnel.

DRAWBACKS AND THEIR REMEDY.—This apparatus works perfectly, so far as percolation goes, but it has one or two drawbacks which can be eliminated by some simple additions.

(a) It is very useful to be able to control the flow from the main funnel into the flask. The working of the apparatus depends on the provision of a resistance in the downward branch of the circuit sufficient to counteract the weight and friction of the liquid in the return tube; this resistance in the simpler modification is provided by the packing in the filter tube, but, should this be insufficient, it is desirable to be able to put in more resistance. A tap G between the body and stem of the main funnel serves this purpose admirably. If a separating funnel is used for the main funnel, this tap is already there.

(b) The simple apparatus is difficult to connect and disconnect, owing to the return tube being held by stoppers at both ends; also a tap in the return tube is a very great advantage for a variety of reasons. If therefore, a short length be cut out of the middle of the return tube and an equal length carrying a tap, H, be inserted in its place by rubber connections, it serves both these purposes, as simple disconnection of the joint above the tap will allow the upper rubber stopper to be withdrawn.

(c) As flasks vary in size, it is best that the return tube should end some inches from the bottom, the lower part terminating in a jet being attached to this by rubber tubing.

The suction applied to the upper exit tube may be replaced by pressure applied to the lower one; in this way percolations may be conducted entirely in an inert atmosphere, the source of pressure being a cylinder of the gas desired. When it is required to stop the action and filter off the liquid the source of gas is disconnected from the lower, and immediately connected with the upper exit tube, thus keeping the inert atmosphere unchanged. If the filter acts too slowly, the tap of the return tube can be turned off, thus causing the liquid to filter under pressure, and, if this pressure is too great, it can be very exactly adjusted by partly turning this tap, thus using the return tube as a "shunt" for excess pressure.

It sometimes happens that the volume of solution which has to be dealt with is greater than the capacity of the main funnel; in this case a portion of the liquid is placed in the main funnel and the remainder in the flask, which must be large

enough to hold it conveniently, and the return tube is adjusted so as just to clear the liquid in the flask. Doubts arose whether, under these conditions, the liquid would circulate properly or whether the drop falling in would be immediately caught up again by the return tube. The following experiments were therefor. made:—About 300 c.c. of approximately 0·1 N potassium dichromate were placed in the flask and 100 c.c. of water in the main funnel with a filter tube of glass wool to introduce the necessary resistance; the apparatus was then started; at the end of half an hour the main funnel tap was turned off, and 25 c.c. were pipetted from the main funnel and the flask into two Nessler tubes; on comparison, the colours were found to be identical, thus showing that mixing had been complete. The experiment was reversed, with dichromate solution in the main funnel and water in the flask, with the same result; in this second experiment the cause of the thorough mixing became evident; each dichromate drop, when it struck the surface, formed a vortex ring which went straight to the bottom of the flask and then spread out. An illustration of the use of the apparatus as a percolator is given in the preceding paper (Evans and Clarke, *supra*, p. 224).

FILTRATION IN AN INERT ATMOSPHERE.—The apparatus adapts itself readily to filtration of a precipitate in an inert atmosphere. Instances will readily occur where a precipitate requires to be protected from moisture, carbon dioxide, oxidation by the oxygen of the air, etc.; in these cases considerable manipulative skill is required to prevent contamination by the air. For this purpose an ordinary pulp filter is placed in the bottom of the main funnel; the apparatus is swept out by a current of the required gas or purified air passed in through the upper exit tube.

The reagents are then added through the top funnel, care being taken not to allow the entrance of any air, each being passed through the filter into the flask beneath, and the filter being washed once or twice with a little of the solvent before the admission of the next reagent. The reagents having thus been brought together in the flask in a neutral atmosphere they can then be heated or treated in any required manner.

When precipitation is complete the gas inlet tube is transferred from the upper to the lower exit tube, and, the tap of the main funnel being turned off, and that of the return tube turned on, the liquid and precipitate are driven, by the gas pressure, over into the main funnel. By again transferring the gas pressure to the top, the liquid can be filtered, and, the cycle being repeated, the remains of the precipitate can be transferred to the main funnel; two or three alternate filtrations and returns suffice to transfer the whole of the precipitate. It now remains simply to filter the precipitate and wash it with liquid added through the top funnel as before.

Two or three points need emphasising:—(a) To add liquid through the top funnel it is necessary for the tap of the return tube to be open. (b) The gas supply must be kept running throughout the operations. (c) It is desirable, in running

liquid into the main funnel to shake gently with a rotatory motion to prevent the stream boring into the pulp.

APPLICATION TO WATER ANALYSIS.—An example of the use of the apparatus for filtration in this manner is the following:—

Determinations of the carbon dioxide content of sodium carbonate and bicarbonate were made by sweeping out the apparatus with air passing through caustic soda, running in known amounts of a standard solution of the carbonates, and precipitating the carbonate ion with barium hydroxide solution filtered straight into the main funnel. The precipitate, after being washed with cold boiled-out water, was dissolved in excess of standard acid and back-titrated. The following results were obtained:—

Amount of carbonate added.	HCl used for solution (0·1 N). c.c.	Titration with NaOH (0·1 N) c.c.	Amount of carbonate found. c.c.
9·7 c.c. 0·05N NaHCO$_3$ +5·0 c.c. N/10 Na$_2$CO$_3$	20·0	5·3	14·7
9·7 c.c. 0·05N NaHCO$_3$	25·0	15·3	9·7
Difference (should equal 5·0 c.c. of 0·1 N Na$_2$CO$_3$) ..			5·0

This method should provide an accurate means of determining carbon dioxide in water.

DISCUSSION.

The PRESIDENT complimented the author on having evolved a method for dealing with such comparatively large amounts of mercury as 0·5 grm. The apparatus itself he regarded as extremely useful, and he expressed the opinion that it would find a wide application; for example, its possible use for the extraction and hydrogenation of oils occurred to him. Its great advantage was that it could be constructed in the laboratory. As in the case of all perfectly simple inventions, one was left wondering why nobody had thought of it before. He suggested that an arrangement might be devised for effecting a continuous stream of cold water in the platinum basin. He enquired as to what extent the volatility of mercury would affect the author's method?

Mr. G. N. HUNTLY, commenting on the use of the apparatus for precipitation and filtration in an inert gas, suggested that a three-way tap in the return tube would dispense with the rather clumsy method of transferring the suction tube from the lower to the upper exit tubes. In the case of mercury determinations he thought that either imperfect condensation or loss on drying might account for low results.

Mr. C. E. SAGE called attention to the need of a method for determining mercury when in the form of the iodide and double iodide of potassium. From the point of view of economy he wondered whether condensation could be effected equally well on a gold dish.

Mr. P. A. ELLIS RICHARDS asked whether gold or silver plates could be used.

Mr. E. HINKS was under the impression that Hehner had used the Reinsch process for mercury determinations, and that he did not find hydrochloric acid inhibitive.

Mr. E. T. Brewis observed that the quantitative condensation of mercury presented difficulties. Some years ago he had devised an apparently efficient apparatus, consisting of a crucible embedded in an iron block and covered with a silver plate pressed into position by means of a bath containing cold water; but, even with this, mercury vapour escaped, and satisfactory results could not be obtained.

Mr. T. Rendle asked whether the presence of organic matter in solution would affect the determination in any way.

Dr. B. S. Evans, in his reply, said he hoped the apparatus would find many applications. It could only be applied to extraction, however, when a solvent with low vapour pressure was used. A more obvious use was in the rapid preparation of saturated solutions. He agreed that a system of automatic cooling would be advantageous, but so far he had been working against time with a view to obtaining results, and he had not found himself unduly hampered by having to change the water three or four times in the course of the twenty minutes required for complete condensation.

The evidence of various speakers certainly seemed to indicate that loss of mercury by volatilisation could occur, but he could only point to the figures quoted in the paper as evidence that, under the conditions given, one recovered substantially the weight one had put in. After all, a thin dish fitted quite well on to the smooth top of a crucible, and it also overlapped considerably. With small amounts of mercury he had found no appreciable loss; but with larger amounts it had to be remembered that the vapour was heavily charged. This meant that heat had to be applied cautiously, and it was possible that some mercury remained in the crucible. It was always desirable to examine the crucible edge carefully for droplets of mercury after sublimation.

He had not made determinations in the presence of organic matter, but with regard to the presence of iodides, Winkler (*loc. cit.*) had said that when a solution of mercury salts was evaporated in the presence of potassium salts and sulphuric acid there was no loss. He had every reason to suppose that gold or silver dishes or plates would give equally satisfactory results. He did not know the conditions under which Hehner's work was done, but the interference of hydrochloric acid when percolated through copper seemed to be largely mechanical; cuprous chloride was formed in such quantities that it coated the copper and clogged the filter. He had considered the insertion of a three-way tap, but it would have to be one of special design, as not only would the gas inlet have to be reversed, but the other exit tube would have to be opened to the air.

Addendum on the Determination of Mercuric Iodide.—Two or three experiments on the determination of mercuric iodide were made. The iodide was dissolved in a hot mixture of nitric acid, hydrochloric acid, and water in the proportions 1:1:2. The solution, after being heated to boiling, was made alkaline with ammonia, one or two grms. of ammonium nitrate added, and hydrogen sulphide passed through it. The mercuric sulphide was filtered off, washed with hot dilute ammonium nitrate solution, and dissolved in 20 c.c. of the same (boiling) acid mixture as was used for the original sample. The paper was well washed with hot water, the filtrate neutralised with ammonia, 4 c.c. of strong nitric acid added, and the liquid then percolated through copper and finished as usual. It was found that the small amount of hydrochloric acid thus added had no appreciable effect on the results.

	Mercuric iodide taken. Grm.	Mercury taken. Grm.	Mercury found. Grm.
(a)	0·3994	0·1703	0·1778
(b)	0·2000	0·0883	0·0882

In (a) the sublimate was accidentally slightly contaminated, which accounts for the rather high result.

In answer to questions received as to the applicability of the method to the B.P. red iodide ointment, it should be sufficient to shake the sample with two or three quantities of hot acid mixture in a separating funnel, filter, and treat the filtrate as described above.

A word of caution is necessary against dissolving the mercuric iodide in potassium iodide, as it seems impossible to wash all the iodide out of the sulphide precipitate; even so, fairly good results are obtained, but the colour of the sublimate is apt to be alarming (scarlet and yellow), as any trace of iodide left and sublimed is forced into the free surface of the sublimate.

The Hot Springs at Nasavusavu.

By C. H. WRIGHT, M.A., F.I.C.

(Read at the Meeting, February 3, 1926.)

OF the many hot springs in Fiji, the best known are those at Nasavusavu, (generally, but incorrectly, spelt Savusavu,) on the south coast of the island of Vanualevu. These hot springs have undergone considerable changes since 1840, when they were visited by the United States Exploring Expedition under Charles Wilkes, who gave the first description of them.[1]   In this he mentioned that there were five hot springs, at some distance* from the beach, occupying a basin forty feet in diameter, and that these springs were used by the natives to boil their food.

Miss Gordon Cumming, who visited Nasavusavu in August, 1876, states[2] that there were then three principal springs which "bubble up in circle like a small crater.   They are intermittent, and the highest makes a fountain about two or three feet high."   John Horne, who was there in 1878, refers[3] to "the principal boiling springs, three or four in number," and states that "the water in these boils up to a height of about a foot, with a gurgling sound."   Dr. Guppy[4] states that about this time the springs assumed the form of geysers.   From information, collected by him from old residents in the neighbourhood, it appears that "the waters spouted up to a height of from 40 to 60 feet, not vertically, but at an angle.   Each outburst, which lasted for ten or twenty minutes, was followed by a similar interval of repose, during which the springs dried up.   This continued for a month or two, after which the springs gradually resumed their normal level." When Guppy visited the springs in July and November, 1898, "they were boiling

* About 150 yards.   C. H. W.

briskly, attaining a height of a few inches, and showed no signs of intermittent action."

When the author was at Nasavusavu in September, 1921, there were three springs. In two of them the water was boiling briskly, attaining a height of about one foot, but in the spring in the centre the temperature was 79° C. A thermometer held by means of a long stick in the centre of the largest spring recorded 100° C. This is of interest, because Wilkes and Guppy did not measure a temperature higher than 210° F. At low tide steam rose from the beach, many patches of which were so hot that they were painful to a bare-footed Fijian.

A sample of the water taken by the author from the largest of the three hot springs had the following composition, stated as ions in parts per 100,000:— Calcium, 177·5; magnesium, 0·4; sodium, 129·9; potassium, 19·2; chlorine, 496·0; sulphate (SO_4), 24·2; silicate (SiO_3), 19·9; and difference, 4·8. Total solids (dried at 120° C.), 871·9.

The waters from the hot springs at Nasavusavu have been analysed four times previously; and it was in the hope of reconciling some of the discrepancies in these analyses that the present sample was analysed. The first analysis was made with the sample of water obtained by the United States Exploring Expedition in 1840; but since the methods of chemical analysis were then somewhat imperfect, this analysis[1] will not be considered further. In 1876 a sample of the water was obtained by Kleinschmidt, which was analysed by Dr. Oscar Pieper of Hamburg.[5] The next sample was collected by H. S. Cooper in 1877 or 1878, and was analysed by H. Rocholl.[6] The fourth analysis was made by A. Liversidge[7] on a sample obtained by Dr. T. D. Bromlow, of H.M.S. *Sapphire*, 1879.

The results of these analyses are given in the table. It should be added, however, that Pieper detected in the water potassium and silicic and sulphuric acids, but the quantity of water was insufficient for their determination. It will be seen from the table that Pieper's and Rocholl's results agree fairly closely with the author's. Liversidge's analysis shows less calcium chloride than Rocholl's and the author's, and differs from all the others in that it includes alumina, aluminium chloride and traces of phosphoric acid. The author found that when the original sample without dilution was made alkaline with ammonia, there was formed a white precipitate of calcium hydroxide. But when it was diluted with an equal volume of water and made alkaline it remained perfectly clear, even after long standing. Both Pieper and Liversidge detected iron in the water; and the former states that after long standing hydrated iron oxide separated out. On the other hand, the author, on testing comparatively large quantities of the water, found it to be free from iron, and his sample remained perfectly clear on standing.

It may thus be concluded that the mineral matter in the water from the hot springs of Nasavusavu consists chiefly of the chlorides of calcium and sodium, together with smaller quantities of magnesium, potassium, sulphate and silicate; and that it has maintained this general composition since 1876.

ANALYSIS OF NASAVUSAVU WATER.

PARTS PER 100,000.

(Conventionally expressed.)

	Pieper (1876)	Rocholl (1877 or 1878)	Liversidge (1879)	Wright (1921)
Total solids	848	879·6*	832·0‡	871·9§
	—	772·6†	781·3†	—
Lime	231	—	—	248·4
Chlorine	479	—	—	496·0
Free sulphuric acid	—	4·9	—	—
Calcium sulphate	—	26·0	37·27	34·3
Calcium chloride	—	435·5	365·29	463·8
Magnesium chloride	—	2·1	1·20	1·7
Sodium chloride	—	264·1	329·48	299·7
Potassium chloride	—	41·5	13·72	36·6
Sodium silicate	—	—	—	31·9
Silica	—	—	13·91	15·7
Iron	present	—	—	absent
Phosphoric acid	—	—	traces	,,
Alumina and traces of ferric oxide	—	—	4·17	—
Aluminium chloride	—	—	12·86	—
Iodine and bromine	absent	—	absent	—
Nitrate	,,	—	—	—
Borate	,,	—	—	—

* Dried at 212° F.; † After ignition; ‡ Dried at 110° C.; § Dried at 120° C.

BIBLIOGRAPHY.

1. WILKES, C. *Narrative of the United States Exploring Expedition.* Philadelphia, 1845, Vol. III., pp. 196–8.
2. CUMMING, C. F. GORDON. *At Home in Fiji.* London, 6th edition, 1901, p. 217.
3. HORNE, J. *A Year in Fiji.* London, 1881, p. 163.
4. GUPPY, H. B. *Observations of a Naturalist in the Pacific between* 1896 *and* 1899. London, 1903, Vol. I., pp. 25–9.
5. *Journal des Museum Godeffroy.* 14, Hamburg, 1879. Ref. given by Guppy.
6. COOPER, H. S. *The Islands of the Pacific.* London, 1888, p. 378.
7. LIVERSIDGE, A. *Journ. and Proc. R.S.N.S.W.*, 1880, XIV., pp. 147–8.

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.

LIME PIP OIL.

IN the preparation of essential oil of limes in the West Indies, there is a considerable accumulation of the "pips," and efforts are now being made to utilise these by crushing them to express their fixed oil.

We have recently examined a sample of this oil, and, as we have been unable to find any published data on lime pip oil, a record of its characteristics may be of interest. They were as follows:—

Specific gravity at 15·5° C.	0·925
Refractive index, n_D^{40}	1·465
Free acidity (as oleic acid)	2·8 per cent.
Saponification value	193·5
Iodine value (Wijs, 1 hour)	109
Unsaponifiable matter	0·8 per cent.
Clouding point in Bellier's test	17·5° C.

Fatty Acids.

Refractive index, n_D^{40}	1·4565
Neutralisation value	198
Titre	35·8° C.

The crude oil on which these figures were determined was reddish-brown in colour, and had a distinctly bitter flavour, but when refined with a little alkali and decolorising carbon, it yielded an oil pale yellow in colour, quite free from any bitter taste, and of bland flavour.

This refined oil gave the following results:

Specific gravity at 15·5° C.	0·921
Refractive index, n_D^{40}	1·4645
Clouding point in Bellier's test	18·4° C.

<div align="right">

S. C. MARSHALL.
M. S. SALAMON.

</div>

DETERMINATION OF THE STRENGTH OF GLACIAL ACETIC ACID.

IN determining the percentage purity of samples of glacial acetic acid different results are frequently obtained according to the method employed.

The direct titration method almost invariably gives higher figures than those given by the freezing point method, owing to the presence of traces of formic acid and to the difficulty frequently experienced in obtaining a definite "end point."

For general purposes the freezing point method is the more satisfactory, and gives very consistent results, provided the necessary precautions are taken. The following procedure has been found to be satisfactory from the points of view both of convenience and of accuracy:—

The approximate freezing point of the acid is determined by cooling in a freezing mixture, the thermometer being then transferred to another test tube containing some of the acid. The second quantity of acid is cooled in a water-bath at a temperature about 5° C. below that registered as the approximate freezing point, and supercooling is allowed to take place to the extent of about 1° C. The thermometer is now rapidly dipped into the first tube containing the frozen acid, and is returned, together with a few adhering crystals, to the tube containing the supercooled acid. The maximum temperature recorded is taken as the freezing point of the acid.

In Fresenius' *Chemical Analysis* (7th edition, Vol. 2, p. 204) a table is given showing the freezing points of various strengths of acetic acid. A curve, based upon these figures, has been drawn, and it has been found that, for concentrations

of acid ·between 97 per cent. and 100 per cent. by weight, the relationship between concentration and freezing point is practically a rectilinear one. Accordingly, the following expression has been arrived at for determining the concentration of the acid:—

When x = per cent. of anhydrous acetic acid by weight, and t = freezing point (°C.), $x = 0.64t + 89.5$.

The table below shows the figures obtained by using this expression, as compared with those given by Fresenius.

Freezing point. °C.	Calculated. Per Cent.	Determined. (Fresenius) Per Cent.
11·95	97·2	97·1
13·25	98·0	98·0
14·00	98·5	98·5
14·80	98·9	99·0
15·65	99·5	99·5
16·70	100·2	100·0

CECIL O. HARVEY.

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.

FLOWERS OF SULPHUR.

REX v. ROBINSON BROTHERS, LTD.

ON December 16, 1925, a Birmingham firm was summoned at Croydon Police Court, at the instance of the London Chamber of Commerce, for having sold goods to which a false trade description, "Flowers of Sulphur," had been applied, contrary to the Merchandise Marks Act, 1887.

Mr. H. D. Roome, for the prosecution, stated that growers of hops were willing to pay a higher price for flowers of sulphur, produced by sublimation, than for ground sulphur. In May, when the transaction took place, the difference in price between the two kinds of sulphur was about £6 per ton. An official of the London Chamber of Commerce had ordered 2 cwt. of "Fortress brand flowers of sulphur," as advertised by the defendants in a Herefordshire paper. Microscopical examination and solubility tests indicated that the substance supplied consisted entirely of ground sulphur.

Chemical evidence was given that the sulphur was over 99 per cent. in purity, contained no arsenic or selenium, and that the whole of it would pass through an 80-mesh sieve, but that it was not flowers of sulphur.

Evidence was also given for the prosecution by the director of a firm of chemical manufacturers who made both kinds of sulphur. In the hop market ground sulphur was sold to hop growers and others as flowers of sulphur, but witness had always protested against the practice.

Mr. Willis, for the defence, urged that there was no case for a jury. The definition of flowers of sulphur in the British Pharmaceutical Codex included both ground and sublimed sulphur.

The Bench held that there was a *prima facie* case for trial by indictment, and bound the defendants over to appear at the next Surrey Quarter Sessions.

On March 3, the case was tried at the Guildford Assizes, before Mr. Justice Horridge and a jury, Sir Travers Humphreys leading for the prosecution, and Sir Arthur Colefax for the defence.

Mr. W. R. Smith said that he had analysed the sample, and had found it to consist of crystalline particles, and to contain no amorphous globules. It was almost completely soluble in carbon disulphide, not more than 1 per cent. remaining undissolved. In his opinion, flowers of sulphur should consist largely of opaque amorphous globules, and from 12 to 30 per cent. of it should be insoluble in carbon disulphide. The sample contained no acid, whereas the British Pharmacopoeia fixed a limit for the amount of acid that should be present. He regarded the term "flowers of sulphur" as synonymous with sublimed sulphur. He did not agree with the statement in the Pharmaceutical Codex that flowers of sulphur might also be obtained by grinding sulphur.

Prof. H. G. Greenish, joint editor of the British Pharmacopoeia, said that the statement in the Pharmaceutical Codex that "sublimed sulphur is readily soluble in carbon disulphide" was not correct. It was true of ground sulphur. In his opinion, flowers of sulphur could only be obtained by sublimation.

Trade evidence in support of the prosecution was given by Mr. H. T. Brandram Jones, director of Brandram Bros. & Co., makers of sulphur products, by representatives of chemical dealers, and by a hop grower.

Mr. D. Lloyd Howard said that, in his experience, the term "flowers of sulphur" always meant sublimed sulphur. It was analogous to "flowers of camphor" and "flowers of benzoin," also obtained solely by sublimation.

The evidence for the defence included other representatives of firms manufacturing sulphur products, chemical manufacturers and dealers, and hop growers. It was to the effect that the trade description, "flowers of sulphur," had for many years been applied to sulphur other than that obtained by sublimation.

Prof. E. S. Salmon, Advisory Officer to the Ministry of Agriculture (present on subpoena), said that, for the purpose of use by the hop grower, pure sulphur in any fine form would have the same effect, however obtained. Sublimation was not the important thing, but purity and fineness.

Mr. Calder, Managing Director of Messrs. Chance & Hunt (manufacturers of the product in dispute), stated that the process of manufacture was to carry the sulphur in the form of a very fine dust into a collecting apparatus, by means of a current of an inert gas, after which the deposit was again passed through a sieve. He could not see anything wrong in applying the expression "flowers of sulphur" as a trade description of this product. He agreed with the statement in the Pharmaceutical Codex that flowers of sulphur could be made either by sublimation or by grinding.

Mr. W. J. U. Woolcock, President of the Society of Chemical Industry, said that he was one of those responsible for the preparation of the British Pharmaceutical Codex. Since 1903 onwards this reference to the manufacture of flowers of sulphur by a grinding process had been continued. In his opinion, the term was properly applicable to a product not obtained by sublimation.

Dr. J. A. Voelcker said that if he were asked to report whether a sample had been properly described as "flowers of sulphur," he would first ask whether it had been sold for medicinal purposes or for agricultural and horticultural purposes. If it had been sold for medicinal purposes he would hold that it was necessary for it to comply with the definitions laid down in the British Pharmacopoeia, but for agricultural and horticultural purposes he would consider that in the term "flowers

of sulphur" the word "flowers" had application primarily to the condition of the material. The nature of it and the form in which it occurred and the way in which it had been prepared would be quite immaterial to him, so long as it did its work. From the standpoint of purity and fineness he would not hesitate to call the sample "flowers of sulphur," and from the standpoint of the hop grower, he considered it in every way a satisfactory material.

Dr. Bernard Dyer said that the sample was exceedingly pure (99·9 per cent.) and exceedingly fine (passing through a 150-mesh sieve). In his opinion, such a product was quite properly described as "flowers of sulphur."

The Judge, in his summing up, pointed out that no question of fraud was involved in the issue; under the Merchandise Marks Act, 1887, the motives of the person who applied a false trade description were immaterial. To come within the Act a description must not only be false, but must also be a false trade description. People in the trade would naturally know what a trade description was. It seemed to him a large order to say that a description was false when so many witnesses for the defendants, composing a great variety of different members of the trade, had said that it was a true and not a false description.

The jury found the defendants guilty, and the judge imposed a fine of £50 and costs. Notice of appeal was given.

Shortly after the hearing of this case, the London Chamber of Commerce issued the following explanatory notice:—

FLOWERS OF SULPHUR.

In order to avoid misconception, the London Chamber of Commerce has issued the following statement regarding the recent prosecution under the Merchandise Marks Act, 1887, relative to the trade use of the description "Flowers of Sulphur."

At the outset unquestionably the term was applied to sublimed sulphur.

Some years ago it was found possible to produce, by other processes, a sulphur composed of particles of a purity and fineness equal to sublimed sulphur. The new product, for this reason, was sold by some manufacturers and merchants as "Flowers of Sulphur."

Whether the expression "Flowers" implied the product of sublimation or highly refined sulphur of a similar fineness of particle became a question, there being considerable difference of opinion in the trade.

So far as the largest market for finely divided sulphur, the rubber industry, was concerned, the point was of no importance. But in the hopfields sublimed sulphur had been in general use as the specific against the disease known as hop "mould."

In these circumstances, the London Chamber of Commerce was urged to take legal action in order that the precise meaning of the trade term "Flowers of Sulphur" might be finally settled by the Courts.

The jury found that "Flowers" could only be applied to sublimed sulphur.

The Chamber is anxious to make it clear that this result does not imply any reflection upon those manufacturers and merchants who have been in the habit, for years, of applying the word "Flowers" to a sulphur which was not, in fact, sublimed. It desires, therefore, to state categorically that, whilst the trade was divided in opinion, all parties acted in perfect good faith, and the attitude of the Chamber in the matter having now been explained, the trade interests concerned have abandoned their intention to proceed with the appeal which had been lodged. In the circumstances, the Chamber of Commerce is meeting the defence in the matter of costs.

PROHIBITION OF MILK SUPPLY BY LOCAL AUTHORITY.

REX *v.* HALE U.D.C. (*ex parte* WRIGHT).

IN this case, heard in the King's Bench division on February 23rd, before the Lord Chief Justice and Justices Salter and Branson, the Hale Urban District Council, Cheshire, appeared to show cause against rules *nisi* for *mandamus* or

certiorari, which had been obtained by Mr. J. Wright for quashing an order made by that Council in December, 1924.

In that month an outbreak of diphtheria had been traced to milk supplied by a farm in an adjoining district, on which farm six, out of seven persons, were suffering from diphtheria. The patients were sent to the hospital, and the Hale Council served a notice (under Sec. 4 of the Infectious Diseases (Prevention) Act, 1890), requiring the farmer to show cause why an order should not be made prohibiting the further supply of milk from his farm. The farmer was then in hospital, but his father-in-law consented, on his behalf, to the order being made, and no complaint was made until a year later, when the Court was asked to grant a *mandamus* to compel the Hale Council to withdraw the order on the ground that it was void because the conditions of the Section had not been complied with.

If, said counsel, there were any irregularities in the making of the original order, strict compliance with any of the requirements of the Section was waived because of the urgency of the matter. The farmer relied upon the technical irregularity in the serving of the notice, and, in asking the Council to withdraw it, he pointed out that the cow had recovered. To this the Council replied that they would remove the embargo if the cow were slaughtered. They did not claim the right to order a cow to be slaughtered, but they did claim the right to keep the order in force so long as the cow remained alive in the district. For the sake of what counsel described as "bovine obstinacy," law costs greatly in excess of the value of the cow had been incurred.

The Lord Chief Justice, in giving judgment, said that, although there had been some irregularities, they were not of a large or comprehensive character, and the applicant had disentitled himself to a writ of *certiorari*. The question was whether it was safe that milk should be distributed from this farm while there remained on the farm a certain cow which had a lesion on one of its udders exhibiting diphtheritic germs—a cow which was a sort of reservoir of potential diphtheria. It was quite clear that the Council had taken up the position that they were willing that the order should be withdrawn when the cause of infection had been removed. They were not satisfied that this cause of infection had been removed, but they offered to take a different view if the cow were taken away. That was quite a different thing from an order for the slaughter of the cow. He could not help thinking that an unedifying degree of obstinacy had been displayed. This was not a case for a writ of *mandamus* any more than it was a case for a writ of *certiorari*, and the rules should be discharged.

The other Justices concurred, and the rules were discharged with costs.

ARSENICAL APPLES: INSPECTOR'S RIGHT OF SELECTION.

On March 24th a Hornsey tradesman was summoned at the Highgate Police Court for having refused to sell apples, exposed for sale, to an authorised inspector under the Sale of Food and Drugs Acts. It was proved that when the inspector insisted that certain apples should be sold to him, the defendant declined to serve him.

For the defence it was contended that, as the inspector wanted to select the apples, the defendant was justified in refusing to allow him a privilege denied to other customers.

The Bench imposed a fine of £10 and costs.

At the same Court another tradesman, of N. Finchley, was summoned for selling apples containing the equivalent of 1/12th grain of arsenic per lb.

In cross-examination the inspector admitted that he had picked out the apples which he wanted.

The solicitor for the defence urged that the inspector had no right to select the bad apples, and that if he insisted that he would "have those," it was not open to him afterwards to argue that he had been supplied with an article not of the nature, substance and quality demanded.

The Bench did not accept this contention, but fined the defendant £5 and costs.

LEMON CHEESE: A DEFECTIVE CERTIFICATE.

IN the Legal Notes of the February issue of THE ANALYST there appears an account of certain proceedings instituted at Birkenhead, which terminated in favour of the defendants, on a technical point, as indicated by the above title.

In view of the fact that my name appears as having analysed one portion of the official sample referred to, I should like, in fairness, to correct the impression which the unfortunate wording of the second paragraph of the report is calculated to convey.

Actually, what happened was that I was asked to examine the vendor's portion of the sample, *with special reference to the starch-content,* to confirm, or otherwise, the results set out in the following official certification of the Public Analyst:

	Per Cent.
Lemon Cheese	58
Starch	24
Added water	18
	100

"Lemon cheese is understood to consist of egg, butter, sugar, and lemon, and, although most manufacturers add some starch and water, the above amounts are, in my opinion, excessive."

My analysis ("Water, 32; starch, 4; and sugar and other 'lemon cheese' solids, 64 per cent.") was at such variance with the foregoing that an adjournment of the case became necessary, in order to refer the third portion of the sample to the Government Laboratory.

The certificate subsequently issued by the Government Chemist disclosed the following percentage composition:

	Per Cent.
Fat	2·8
Proteins	1·2
Solids from liquid glucose	31·0
Invert sugar	12·7
Cane sugar	11·9
Starch	3·0
Acidity (as citric acid)	0·7
Insoluble solids (mainly from lemon)	2·9
Water (by difference)	33·8
	100·0

Loss in 5 hours at 100° C.   34·6 per cent.

The opinion based upon these results was to the effect that "the sample contains three per cent. of starch, calculated as free from water, and, in addition, about nine per cent. of water in excess of the water associated with proteins, liquid glucose, starch, lemon products, and fat contained therein."

The statement of the prosecution that, of the four ingredients—sugar, butter, eggs, and lemon—the Government Analyst had only found 49 to 50 per cent., is quite incorrect. Any criticism on his part was directed to the presence of about nine per cent. of water extraneous to that associated with proteins, glucose, etc., *i.e.* water incorporated in the manufacturing process.

Apart from the substantial agreement in respect of starch-content between the Government certificate of analysis and my own, the point I wish to emphasise is that the "Sugar and other 'lemon cheese' solids" (exclusive of the starch) certified by the Government Chemist—on a simple arithmetical basis, of course—amounts to either 63·2 or 62·4 per cent., according to whether the percentage of water is based upon the "difference" figure or the "Loss in 5 hours at 100° C."

S. ERNEST MELLING.

Parliamentary Notes.

TUBERCULIN TESTED MILK.—The Minister of Health, replying to a question by Mr. Alfred Williams, on March 18th, said that there were approximately 200 herds producing Certified Grade A (Tuberculin Tested) milk, but that he had no information as to how many of these were pedigree herds. Calves were not usually kept in contact with these cows, and there was therefore no necessity for young stock to be tested until they were introduced for milking purpose. If in any case calves were kept in contact with the cows, they must be tested at the time the general tests of the herd were made.

BAKERS' ITCH.—On March 23rd, the Minister of Health replied to a question by Mr. Harland that he was aware of the prevalence of this disease. It was investigated by the medical department of the Ministry of Health in 1922, and no reasons were then disclosed why it should be made notifiable. There was no evidence showing that this disease was due to the modern use of chemicals in the bleaching of flour.

ARSENIC IN APPLES.—Mr. Neville Chamberlain, replying to a question by Col. Day, said that he had received representations from the Surbiton Council with regard to fruit exposed for sale, and found to contain arsenic. He said that the present Imported Food Regulations enabled port and riparian sanitary authorities to seize and destroy any imported food which was unfit for human consumption, and he did not think it would be practicable to strengthen these regulations without imposing an absolute prohibition on the importation of apples—a measure which he did not consider would be justified at present. He was not aware of any case in which an excessive amount of arsenic had been found on home-grown or Empire-grown apples.

"Artificial Wools": Misuse of Terms.

IN the annual report of the British Research Association for the Woollen and Worsted Industries reference is made to the question of "artificial wools." It is recommended that careful consideration should be given to the name "artificial wool" before any trade custom is allowed to be established by which goods consisting wholly or partly of "artificial wool" might be described as "wool" or

"all wool." There are already several customary terms that are misused or are even deceptive, such as "cashmere," "merino" (a blend of cotton and wool in hosiery), and "woollen" (often containing much cotton). The most recent example however, is "artificial silk," for which a change of name to "rayon" has already been adopted in the United States, and is recommended by the Silk Association in this country.

"Artificial silk," or "rayon," is, in fact, a very close imitation of silk in its principle characteristic—lustre; but so-called "artificial wool" does not reproduce the essential characteristics of wool, and, at best, has but an outward resemblance to it. Some day, perhaps, artificial wool may be made which has several, at least, of these characteristics, but of present substitutes, adds the report, none of those examined has shown more than an outward resemblance to wool, and this often disappears on wetting. Wool substitutes are many, and may include almost any fibre, but synthetic wool is non-existent at present.

Queensland.

REPORT OF THE GOVERNMENT ANALYST FOR 1925.

ACCORDING to the report of Dr. J. B. Henderson, the Government Analyst, the number of samples examined during the year ended June 30, 1925, was 5904, of which 1657 were for the Customs, 1370 for the Health Department, 71 for the Police, and the remainder for other Government departments.

FOOD AND DRUGS.—Of the 1069 samples examined, 783 were taken by inspectors in accordance with the provisions of the Health Acts, and 110 of these were condemned. These samples included 697 of milk (68 condemned), 29 of spirituous liquors (16 condemned), 16 of sausages and mincemeat (7 condemned), and 9 of cordials, all of which were condemned.

The seven samples of sausages and mincemeat condemned contained excessive quantities of sulphite preservative.

MILK.—Of the 697 legal samples of milk, 4 were genuine but below standard, 27 were deficient in fat, and 37 were adulterated with water.

BACTERIAL CONTAMINATION OF MILK.—The Reductase Test, incorporated in the 1924 Food and Drug Regulations for New Zealand (ANALYST, 1925, 50, 141), was applied to Brisbane milk. According to this standard, the milk, when subjected to the reductase test, shall not completely decolorise a methylene blue solution of definite strength in less than 3 hours. This time limit has been confirmed by a number of observers as indicative of milk containing less than 500,000 bacteria per c.c.

For the purpose of the test, milk may be divided into the following classes:

		Organisms per c.c.
Class I.	Decolorised in $5\frac{1}{2}$ hours or longer	Under 500,000
Class II.	Decolorised in less than $5\frac{1}{2}$ hours, not less than 2 hours	$\frac{1}{2}$ to 4 millions
Class III.	Decolorised in less than 2 hours, not less than 20 minutes	4 to 20 millions
Class IV.	Decolorised in less than 20 minutes	Over 20 millions

The reductase test was applied to 234 samples of milk taken from carts, most in early winter, and the following results were obtained:—Class I., 90 per cent.; Class II., 5 per cent.; Class III., 3 per cent.; and Class IV., 2 per cent.

FRUIT CORDIALS.—The samples condemned contained an excessive amount of preservative, or were deficient in sugar, or were misdescribed as to their origin.

Recently preparations of southern origin, purporting to be fruit extractives, have appeared on the market. On analysis they proved to be merely aqueous emulsions containing a small proportion of the oil of the fruit named on the label. Practically all the fruit cordials sold contain sulphite preservative, which destroys the vitamin constituent. In the light of recent work on vitamins, it would seem advisable to consider the re-drafting of the standards for non-alcoholic cordials.

MINES DEPARTMENT.—The decrease in the number of samples examined was mainly due to fewer samples from the State arsenic mines being submitted. The fact that fewer samples were also submitted by prospectors is probably an indication of a continued decrease in prospecting, since, for many years, samples from prospectors with mining rights have been assayed free of charge.

The great bulk of the assaying work was for gold and silver. There was a fair proportion of lead and copper ores, and cobalt was looked for, but tungsten and molybdenum ores are now rarely submitted.

CAVE DEPOSIT.—A sample of brown organic material with an odour suggestive of guano was submitted as natural bitumen. It was said to be an exudation on a sandstone rock in a cave in West Queensland. It consisted of 40·4 per cent. of organic matter, the remainder being mainly sand. On analysis it gave the following results:—Soluble in water, 31·8; soluble in petroleum spirit, trace; total phosphates, 0·98; total nitrogen, 4·2; organic nitrogen, 3·9; nitric nitrogen, 0·3; potassium salts, present. The material is not bitumen, but is evidently an evaporation product of the aqueous leachings of a guano or cave deposit. This is the third instance of this kind of product being submitted for examination.

ALGAE GROWING IN HOT WATER.—A sample of water containing an organic constituent, suspected to be petroleum, was taken from the overflow pool of a hot bore. The organic matter proved to be one of the fresh water algae which grow in hot water. They have been recorded elsewhere as growing abundantly up to 68° C., and scantily up to 80° C., and, whilst well-known in countries where natural hot springs abound, this is the first instance in which they have been noted in Queensland.

POLICE DEPARTMENT.—Seventy-one exhibits were submitted, as compared with 54 in the previous year. Of 18 cases of deaths in which poisoning was suspected, strychnine was found in 5 cases, arsenic in 2, cresols in 2, cresols and chlorodyne in 1, and in the other 8 cases no poison was found. Other samples submitted included abortifacient medicines, shampoo powder, well-water for poison (none present), smoking opium, and 2 specimens of writing.

Department of Scientific and Industrial Research.

FOOD INVESTIGATION. Special Report No. 26.

THE STORAGE OF EGGS.*

PART I.—PRACTICAL.—In 1922, 4332 million eggs in shell were consumed in Great Britain, and, of these, 38 per cent. were imported from abroad and 22 per cent. from the Irish Free State.

STORAGE CONDITIONS.—Eggs for storage should preferably be not more than 1 week old and should not have been kept at a temperature above 60° F. Storage temperature should be between 0° and 0·6° C., and the relative humidity of the air kept constant at 80 per cent. in order to counteract loss of water from the eggs by evaporation, and at the same time not exceed the safety limit for development of mould growth. (Actual loss in weight of eggs in seven months with 80 per cent. humidity was 2·68 per cent.) In order to insure these conditions, circulation of the air in the store is necessary. This also removes accumulating odours and reduces storage taste.

The loss of water from eggs is due to the white of the egg being a solution of salts and colloids freezing at –0·45° C., so that at 0° C. the vapour pressure is 0·016 mm. below that of water. Air will therefore tend to take the place of water in the egg if the humidity of the surrounding atmosphere is below 99·6 per cent. Packing material is thus liable to become damp.

If the pores of the shell are closed (and aluminium soap in gasoline or, better, pentane solution was found suitable for the purpose), evaporation is checked, but the appearance of the eggs is affected. Oiled or waxed wrapping paper has been suggested, as for apples (ANALYST, 1924, 49, 138).

Moulds.—Among the moulds observed in eggs were representatives of the following genera and species:—*Sporotrichum, Penicillium, Cladosporum herbarum, Alternaria, Thamnidium,* and *Mucor.*

Surface sterilisation in a 1 in 1000 mercuric chloride solution resulted in only a slight mould growth inside and adjoining the air chamber after storage for 7 months in a 100 per cent. humidity. Sealing, by placing the eggs in a vacuum and then passing through a paraffin wax bath had much the same effect. Dipping for a few seconds in boiling water was found to delay mould growth. For a discussion of the various ways of measuring humidity in the air reference should be made to Special Report No. 8 of the Food Investigation Board (ANALYST, 1926, 51, 35–36).

Grading.—Eggs should be graded to size. Imported Irish and Danish eggs are graded according to weight per great hundred (10 doz.) Russian eggs as prime (average 14·7 lb. per great hundred), average or secondary, and small (passing through a ½ inch hole), and Canadian eggs according to the specification of April, 1924. Eggs with blood spots should not be stored.

Candling.—Candling is useful for detecting quality. A new-laid egg appears clear and transparent, with the yolk (vaguely seen in the middle) tinted rosy red with a slightly darker fringe towards the centre, the air chamber about 3 mm. high and with the diameter of a 6d. The yolk flattens little when the egg is broken on a plate. On ageing, the yolk becomes redder and slightly marbled and moveable along the longitudinal axis, and the air chamber increases in size; the white gets

* By T. Moran and R. Piqué. 80 pp. 7 plates. H.M. Stationery Office. 1926. Price 1s. 3d. net.

thinner and yellower, and the yolk whitish yellow, flattening on breaking the egg on a plate. Moveable air chambers occur in old eggs exposed to dampness, and are seen as transparent rings on twisting the eggs. Bloodspots are only seen on twisting the egg. Rotten eggs appear opaque, some with streaks or patches; mould inside the shell appears as spots or patches ranging in colour from reddish brown to black. Eggs with brown shells are more difficult to judge by candling than white shelled eggs. The best results were obtained with the light . a gas-filled electric lamp filtered through a blue filter (Wratten No. 43).

The density test, as usually practised, by putting the eggs into a salt solution of 2 oz. to 1 pint, is shown to be only an approximation, as the density of eggs varies from at least 1·01 to 1·1, which is enough to mask a loss by evaporation of 8 per cent.

Changes during Storage.—Changes occurring during storage allow of a chemical method of grading edible eggs. Ammoniacal nitrogen (as mgrms. per 100 grms. of material) is given by Houghton and Weber by the Folin titration method as: Seconds, 11·4; spots, 14·1; light rots, 17·3; rots, 26·2; black rots, 169·6; and by the Folin Nesslerization method as 12·4, 20, 21·5, 29·9, and 148·6 respectively. Physical changes are: loss of bloom, greater homogeneity of the white (which becomes yellower), greater delicacy of the yolk membrane, loss of water by evaporation, and consequent increase in size of the air chamber, and, after about 6 months, acquisition of a "Storage" taste. The average viscosity (relative to water) of the filtered white of new-laid eggs is 3·02, and of stored eggs 3·77. Evaporation of water from eggs, and passage of water from the white to the yolk are contributing causes of the differences observable after storage.

Defrosting.—Removal of eggs from cold store will usually result in condensation of moisture on their surfaces, since otherwise the air would have to contain less than 4·876 grms. of water per cb. metre. Artificial defrosting may be brought about by gradually raising the temperature of the store before moving the eggs, from 16 to 24 hours being taken for the process, with a good air circulation. If defrosting is incomplete, the eggs will become damp and mould will rapidly develop.

Storage in Water-glass and Lime Water.—Eggs stored in water-glass have their pores sealed by deposition in 3 to 7 days, and thus must be pierced before boiling. With lime water the egg does not appear to be completely sealed, and water passes through the shell. Lime water is definitely an antiseptic and less likely to suffer from mould and bacterial contamination than water glass.

PART II.—This gives the theoretical data from the paper published in the *Proc. Royal Soc.* (ANALYST, 1926, 39). D. G. H.

Dangerous Drugs Act, 1920.

(10 and 11 Geo. 5, c. 46).

SCHEDULING OF VERONAL AND THE BARBITONE GROUP.

The Home Secretary gives notice that it is proposed to apply for an Order of the King in Council in pursuance of Sec. 8(2) of the Dangerous Drugs Act, 1920, declaring that Part III. of that Act shall apply to veronal and the other drugs of the barbitone group in the same manner as it applies to the drugs mentioned in Sub-Sec. (1) of Sec. 8 (morphine, cocaine, etc.), and in pursuance of Sec. 7 of the Act to make regulations, limiting the supply of the drugs to authorised persons or institutions or to persons for whom the drugs have been prescribed by a duly qualified medical practitioner.

The drugs to which these Regulations apply are diethyl barbituric acid and other alkyl, aryl, or metallic derivatives of barbituric acid, whether described as veronal, proponal, medinal, luminal, dial, or by any other trade name, mark, or designation, and any preparation, admixture, or other substance containing any of them.

Copies of the draft regulations may be obtained on application to the Under-Secretary of State, Home Office, Whitehall, London, S.W.1.

National Physical Laboratory.

TESTS ON VOLUMETRIC GLASSWARE USED IN DAIRY CHEMISTRY.

LABORATORY MARKS.—Butyrometers, milk test bottles and milk pipettes submitted for test, and found to comply with the above regulations will be etched with the Laboratory mark

NP B
'26

This mark is a combination of the Laboratory monogram, and the date of test with the letter B, to indicate that the apparatus meets with the Class B regulations.

The National Physical Laboratory is also authorised under section fourteen of the schedule of the Dairy Industry Act (Act No. 16 of 1918) of the Government of the Union of South Africa, to test glassware used in the testing of milk and cream, and required to fulfil the Regulations under Section Twenty of the above Act, and to etch the mark ⟋U|G⟍ on each vessel found to comply with the requirements of the Union of South Africa.

FEES.—When 12 or more instruments of the same type and capacity are sent together for test a reduction of $33\frac{1}{3}$ per cent. is made on the fees given below:

Butyrometers or Test Bottles ..   .. , each vessel   1s. 6d.
Milk Pipettes   ..   ..   each vessel   2s. 6d.

(*This leaf is intended to replace p. 14 of the Pamphlet " Tests on Volumetric Glassware used in Dairy Chemistry," issued April 30th, 1921*).

March 1st, 1926.   J. E. PETAVEL (*Director*).

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Composition of Marrow of Fresh and Cured Hams. W. F. Schroeder and D. Edelman. (*J. Agric. Res.*, 1926, 31, 1015–1016.)—Analyses of the marrow from femur bones of fresh and cured hams (by the usual potassium nitrate process and with sodium nitrite) showed a higher proportion of ash, sodium chloride, and nitrites, in the cured hams, so that curing agents actually penetrate the marrow. The amounts of nitrites (as $NaNO_2$) in the marrows ranged from 15 to 55 parts per million.   D. G. H.

Chemical Study of the Flesh of Emaciated Cattle. R. Hoagland and W. C. Powick. (*J. Agric. Res.*, 1926, 31, 1001–1013.)—Analysis of the flesh of cattle with no recognised disease and classified as " extremely emaciated " (flesh, connective tissue and bone marrow abnormal in appearance) and " very thin " (shrunken musculature, but no apparently abnormal condition) showed that the

most important variations from the normal were the higher moisture and lower protein contents, and it is suggested that the ratio between protein and moisture may prove of value in classifying animals for food purposes. The average ratio for fat cattle was found to be 1 : 3·5, and for emaciated cattle 1 : 4·2. In many respects the flesh of " very thin " cattle showed an intermediate condition between that of normal and extremely emaciated cattle. Percentages of purine and creatinines were always within normal limits. The proportion of fat was very low, and dextrose was only found in two out of 8 samples of emaciated flesh.

D. G. H.

Detection of Incipient Putrefaction in Meat and Meat Products. E. Arbenz. (*Mitt. Lebensm. Hyg.*, 1925, **16**, 84–95 ; *Chem. Abstr.*, 1925, **19**, 2988.)— The beginning of putrefaction and an approximate idea of its extent may be discovered by measurement of the oxygen consumed and of the amount of methylene blue reduced :—Five grms. of chopped meat are digested with 300 to 400 c.c. of water (previously heated to 22°–23° C.) for 2 hours at 22°–23° C. The oxygen is then determined by adding 1 c.c. of 80 per cent. manganese chloride solution and 1 c.c. of 33 per cent. sodium hydroxide solution, followed, after a few minutes, by a few crystals of potassium iodide and 5 c.c. of concentrated hydrochloric acid, titrating the liberated iodine with thiosulphate, and calculating the result into the equivalent of oxygen. When there is no further precipitation of iodine after 2 hours the meat is unfit for food. In applying the methylene blue test 5 grms. of the chopped meat are mixed with 60 c.c. of water at 40° C., and 1 c.c. of methylene blue solution (5 c.c. of saturated solution with 195 c.c. of water), the flask placed in water at 45° C., and time required for reduction noted. If reduction takes place in less than 1 hour incipient putrefaction is indicated. Satisfactory results were thus obtained with beef, pork, veal, mutton, horseflesh, mincemeat, sausage, pigeon, and several kinds of fish.

Foods made from Yeast. E. Stern and H. Becker. (*Chem. Zeit.*, 1926, **50**, 185–186.)—A preparation containing about 66 per cent. of sugar carbohydrates and 33 per cent. of yeast constituents has recently been put upon the market under the name of " Katazymans." It is made in Munich by the plasmolysis of washed and pressed yeast at the ordinary temperature by means of Soxhlet's nutrient sugar. The normal water content of the yeast cells (75 per cent.) is sufficient for the plasmolysis. The cell contents mainly diffuse out of the cells, and liquefaction takes place without any addition of water. The product is then heated for an hour at 45° to 50° C. to destroy the zymase, and is finally completely dried *in vacuo* at a temperature not exceeding 60° C. An analogous preparation has been described by Willstätter and Sobotka, who mix the yeast with a sufficient quantity of sugar to prevent fermentation, and heat the mixture for an hour at 55° C., after which the temperature is raised to 100° C., and the liquefied mass cooled for about an hour. Finally the product is dried and, if desired, mixed with a cereal flour, cocoa, etc.

Commercial Apple Juice and Pectin Products. H. Eckart. (*Zeitsch. Nahr. Genussm.*, 1925, **50**, 405–415.)—The pectin number proposed by Mehlitz (*Konserven-Industrie*, 1925, **12**, 73, 607) furnishes a valuable aid in the evaluation of pectin products, but cannot be employed alone for the comparison of the jellifying action of different pectin solutions. In the first place it has not yet been definitely explained whether, and, if so, to what extent, the pectin content represents a measure of the ability to gelatinise. The composition of the extract also requires attention, as this may be of more value in one preparation than in another. As is shown by the results given in the appended table, the concentration gives no indication of the purity quotient. With the Nollco commercial preparations, the different concentrations alone determine the differences in pectin content. On the other hand, the Pomosin and Douglas (American) products show increased pectin contents which are not the result of mere concentration. The following table gives a selection of the analytical results obtained:

	Percentage of			Pectin number.
	Calcium pectate.	Pectin.	Extract.	$\dfrac{\text{Pectin} \times 100}{\text{Extract}}$
Boiled apple juice	0·57	0·52	5·1	10·2
,, ,, ,, concentrated 1:2	1·15	1·06	10·6	10·0
,, ,, ,, ,, 1:4	2·30	2·13	21·0	10·1
Nollco apple juice	1·37	1·26	11·3	11·2
,, ,, ,, ...	2·54	2·34	25·8	9·1
Pomosin extract	2·30	2·12	28·6	7·4
,, ,, ...	3·21	2·95	7·6	38·8
,, dried extract ..	32·40	29·81	88·2	33·9

T. H. P.

Determination of the Age of Bread. J. P. Peper. (*Chem. Weekblad,* 1926, **23**, 163–168.)—A Dutch labour regulation forbids the sale of new or hot bread, and all bread delivered before ten o'clock in the evening must be at least 12 hours old. To ascertain whether bread complies with this regulation the method devised by Katz has been applied. This is based on the fact that as bread becomes old there is a synhaeresis, or reversal of the gelatinisation of the starch granules which takes place during baking, while small amounts of the absorbed water are continually lost by the granules. In applying the test 20 grms. of the bread crumbs are mixed with water and kneaded through the meshes of fine silk gauze, and the resulting suspension is diluted to 250 c.c. with water in a measuring cylinder, after which 0·5 c.c. of toluene and 0·5 c.c. of acetaldehyde are added. The height of the column of sediment formed after 24 hours stands in close relationship to the specific gravity of the starch granules. The progressive synhaeresis can thus be followed by measuring the height of the sediment of the sifted crumbs 0, 12, 24, 36, and 48 hours after the baking of the bread. The results obtained with a large number of samples have been tabulated and plotted as curves, and from them the following deductions have been drawn. With normal good bread, when the height of the initial sediment exceeds 96 c.c. the bread is new, whilst when it is less

than 85 c.c. the bread is old.   But as new bread of poor quality may show an initial figure as low as 85 c.c., it is necessary to take as a second criterion the difference between the initial reading and that obtained after the bread has been kept for 24 hours.   If this difference exceeds 15 the bread is new ; if it is less than 15, the bread is old.   Old bread which has been re-heated may sometimes give results similar to those obtained with new bread ; in other cases it behaves like old bread.   This difference depends upon the extent of the chemical action promoted by the heating.

Phytin Content of Foodstuffs.   H. P. Averill and C. G. King.   (*J. Amer. Chem. Soc.*, 1926, 48, 724–728.)—The following modification of Heubner and Stadler's method of determining phytin (*Biochem. Zeitsch.*, 1914, 64, 422) is recommended.   Eight grms. of the finely ground foodstuff are extracted with 200 c.c. of 2 per cent. hydrochloric acid solution for 3 hours, 50 c.c. of the filtered extract being diluted so as to contain 0·6 per cent. of the acid and the phytic acid determined by titration, in presence of 10 c.c. of 0·3 per cent. ammonium thiocyanate solution, with ferric chloride solution containing 0·00195 grm. of iron per c.c. From the iron equivalent thus obtained, the equivalent of phytin phosphorus is calculated by means of the factor 1·19 ; further multiplication by 3·55 gives the amount of phytin.   With soya beans or nuts, extraction with ether prior to that with acid is necessary to avoid disturbing colour and turbidity in the extract.

Some of the percentages of phytin obtained, calculated as $C_6H_{18}O_{24}P_6$ on the air-dried materials, were as follows :—Barley, 1·07 to 1·19 ; buckwheat, 1·25 to 2·39 ; buckwheat flour, 1·86 ; oats, 0·77 ; rye, 1·04 to 1·87 ; rye flour, 0·74 to 1·46 ; rye middlings, 3·33 ; wheat, 1·16 to 1·36 ; wheat bran, 4·53 ; wheat flour, 0·66 to 1·28 ; hempseed, 2·75 ; millet seed, 1·12 ; rape seed, 2·63 ; soya beans, 1·79 to 2·58 ; almonds, 2·41 to 2·74 ; Brazil nuts, 2·62 to 3·30 ; filberts, 1·60 to 1·72 ; hickory nuts, 1·48 to 1·67 ; pecan nuts, 1·40 to 1·52 ; peanuts, 1·77 to 2·17 ; roasted peanuts, 1·34 to 1·66 ; walnuts, 1·42 to 2·04.

In general, hard winter wheat flours are of higher phytin content than soft and spring wheat flours.   Heating, soaking, or steaming of the materials causes very appreciable decrease in the amount of phytin present, and this, in conjunction with the ease with which enzymic hydrolysis of the phytin takes place is probably the reason for the divergent proportions of phytin found by different investigators, and for the fact that few have succeeded in isolating significant amounts of the hexaphosphoric acid compound.   T. H. P.

Detection of Methyl Alcohol in Alcoholic Beverages.   F. R. Georgia and R. Morales.   (*Ind. Eng. Chem.*, 1926, 18, 304–306.)—The following procedure is recommended :—Five c.c. of the alcoholic solution (distillate) diluted previously so as to contain 5 per cent. by volume of total alcohol, are treated with 2 c.c. of a solution containing 3 grms. of potassium permanganate and 15 c.c. of 85 per cent. phosphoric acid per 100 c.c.   The excess of permanganate is then destroyed by adding 2 c.c. of a solution of 5 grms. of oxalic acid in 100 c.c. of dilute (1 : 1)

sulphuric acid.   As soon as the pink coloration has disappeared, 5 c.c. of Schiff's reagent are added ; the characteristic colour due to formaldehyde develops within ten minutes if the sample contained methyl alcohol.   The Schiff reagent is prepared by dissolving 0·2 grm. of rosaniline hydrochloride in 120 c.c. of hot water, treating the cooled solution with 2 grms. of anhydrous sodium sulphite dissolved in 20 c.c. of water, adding 2 c.c. of concentrated hydrochloric acid and diluting the mixture to 200 c.c.   Certain substances interfere with the detection of the methyl alcohol, and methods are given for their removal before the test is applied. *Formaldehyde*, if present originally in the sample, is removed by treating 10 c.c. of the diluted sample with 1 grm. of pyrogallol, 10 c.c. of water, and 5 c.c. of concentrated sulphuric acid in a closed flask, and distilling the mixture after the lapse of ten minutes.   *Amyl alcohol, essential oils (except oil of angelica), fluid extracts of arnica and ipecacuanha* :—Ten c.c. of the diluted sample are mixed with 40 c.c. of water and 14 grms. of sodium chloride and extracted with 25 c.c. of petroleum spirit, and the aqueous portion is then separated and distilled. *Cinchonine, glycerol, liquorice, methyl violet, quinine sulphate, strychnine sulphate, tannin* :—By distillation.   *Aniline* :—By distillation from sulphuric acid solution. *Phenols* :—By distillation from sodium hydroxide solution.   *Acetaldehyde, benzaldehyde* :—By addition of sulphuric acid before Schiff's reagent is added. Satisfactory methods for removing methyl acetate, oil of angelica and pectinous substances have not been discovered. W. P. S.

Determination of Moisture in Cloves.   P. May.   (*Perfum. and Essential Oil Rec.*, 1926, **17**, 65.)—The use of a special apparatus, such as the Brown-Duvel tester (ANALYST, 1926, 152), is unnecessary, since the Dean and Stark method may be satisfactorily adapted to the determination of moisture in cloves.   A condenser is fixed to the mouth of a tapering graduated tube which is connected by a side arm with a round bottomed distilling flask.   The cloves and a colourless petroleum oil of somewhat high boiling point (one boiling from 165°–175° C., with 50 per cent. distilling at 178° C. was found satisfactory), are heated in the round-bottomed flask, when the water carried over accumulates in the graduated tube, whilst the excess of condensed petroleum oil flows back into the flask by the side arm.   Apparently dry samples of Zanzibar and Penang cloves of good quality contained, respectively, 13·0 and 12·8 per cent. of moisture. D. G. H.

Analysis of Commercial Vanilla Oleoresins.   J. B. Wilson and J. W. Sale.   (*Ind. Eng. Chem.*, 1926, **18**, 283–285.)—These flavouring preparations are obtained by extracting vanilla beans with dilute alcohol, or dilute alcohol and glycerin, and evaporating the solvent under reduced pressure.   Analyses are given of commercial samples and also of samples prepared in the laboratory.   The following determinations yield useful data in valuing the preparations :—Ash, usually less than 7·2 per cent., unless alkali has been used in the extraction ; the total alkalinity of the ash, expressed as c.c. of N acid per 100 grms. of oleoresin, is less than 50 unless added alkali is present.   *Substances soluble in 95 per cent.*

alcohol. The total solids of this solution vary from 50 to 70 per cent. Vanillin is also determined in this solution, and varies from 2 to 6 per cent. ; the solution also contains the glycerin. In the case of samples prepared without the use of glycerin small amounts of a residue may be obtained, but this is obviously not glycerin and the quantity is insignificant when compared with the 11 to 37 per cent. present in the extracts prepared with alcohol-glycerin. *Caramel* is determined in the aqueous extract of the oleoresin after the extraction with 95 per cent. alcohol. For this purpose the extracted residue is dissolved in water, the solution filtered, evaporated to a syrup, and cooled ; 50 c.c. of paraldehyde and sufficient alcohol to cause the water and aldehyde to mix, are added, the precipitate is collected after eighteen hours, washed with a few c.c. of alcohol, dissolved in water, and the solution is evaporated and the residue weighed. The amount of " caramel " thus obtained is usually less than 10 per cent. except from samples containing added caramel. *Vanilla resin* is determined on an extract of the oleoresin in 47·5 per cent. alcohol, and the same extract serves for the determination of the lead number. The vanilla resin amounts to about 2 per cent., whilst the lead number is about 15 per cent., but varies with the amount of glycerin, etc., present. All the results should be calculated as percentages of the oleoresin. W. P. S.

Colour Reaction distinguishing between Aniseed Oil and Star Anise Oil. W. van den Driessen Mareeuw. (*Pharm. Weekblad*, 1926, **63**, 390–391.)— An oil supplied as pure aniseed oil by a well-known firm answered to the requirements of the Dutch Pharmacopoeia for that oil, but did not give the right colour reaction in Zimmerman's test (*Apoth. Z.*, 1925, 1344). In this test 2 to 3 drops of the oil are mixed with 5 drops of a solution of vanillin (0·4 grm. in 5 grms. of alcohol and 5 c.c. of water), and the mixture is made up to 1 c.c. with concentrated hydrochloric acid. After standing for 5 minutes at the ordinary temperature the tube is immersed in a water-bath at 50° C., and the temperature gradually raised to 100° C. The tube is then withdrawn from the bath and the colour of its contents noted after cooling. Pure aniseed oils give reddish colorations, ranging from pink to yellowish red, red or reddish brown, according to the age of the oil and the temperature of the reaction. Star anise oils, on the other hand, give green colorations in the test. As the oil in question gave a green coloration and no indication of red, the conclusion was drawn that it was derived from star anise (*Illicium verum*), or was a mixture of anethol and star anise oil.

Determination of Formaldehyde in Formalin Tablets. I. M. Kolthoff. (*Pharm. Weekblad*, 1926, **63**, 389–390.)—Iselin's method of determining formaldehyde in formalin tablets by means of aniline, the excess of which is afterwards determined bromometrically, is involved and does not yield accurate results, Romijn's iodimetric method can be used in the presence of sucrose, but not of lactose. The acidimetric method (in which the formaldehyde is converted by means of ammonia into urotropin) is trustworthy, even in the presence of sugar, and in certain cases the bisulphite method may be advantageously used.

Iodeosin Indicator in Alkaloidal Determinations. D. B. Dott. (*J. Pharm.*, 1926, **116**, 357.)—Unsatisfactory results were obtained with iodeosin from two sources used in very dilute solution (1 in 10,000) as indicator in strychnine titrations, where the strychnine was dissolved in excess of 0·1 *N* acid and titrated with 0·1 *N* sodium hydroxide solution. D. G. H.

Alleged Deterioration of Indian Opium. D. B. Dott. (*Pharm. J.*, 1926, **116**, 356.)—A sample of Indian opium, powdered and dried at 95°–99° C., and kept in the dark in a stoppered bottle from February, 1924, till March, 1926, lost only 0·24 per cent. of morphine from an initial content of 10·92 per cent. D. G. H.

Analysis of Mercuric Iodides. J. Sandilands. (*J. Pharm.*, 1926, **116**, 357–358.)—Cold sodium thiosulphate solution is added to the weighed mercuric iodide, the salt dissolved, the solution diluted with an equal volume of cold water, and the mercury precipitated by means of hydrogen sulphide. The precipitate is washed, collected, dried, and weighed. To determine the iodine, the mercuric iodide is moistened with water, and an amount of zinc dust equal to 10–20 times the weight of salt used is added, followed by a few drops of dilute sulphuric acid, and cold water. The mixture is shaken vigorously at intervals for 15 minutes, after which the zinc amalgam is filtered off, washed with water, and the filtrate acidified with dilute nitric acid, and titrated with 0·1 *N* silver nitrate solution and thiocyanate. Aluminium or magnesium may be used in place of the zinc, and in the case of magnesium the sludge of mercury and magnesium on the filter paper may be dissolved in nitric acid, and the solution of mercuric nitrate titrated, and results obtained both for the mercury and for the iodine. D. G. H.

Biochemical, Bacteriological, etc.

Secretion of Nitrates with Milk. H. Krause. (*Arch. Hyg.*, 1925, **85**, 271–279 ; *Chem. Abstr.*, 1925, **19**, 2977.)—Experiments to determine under what conditions cows will secrete nitrates with the milk have shown that milk from cows that have drunk water containing 80 mgrms. of nitrate (as N_2O_5) per 1000 c.c. is free from nitrate. This is also true of the milk of cows which have been drinking for 30 days a water containing 500 mgrms. of nitrate per 1000 c.c., or of cows to the food of which has been added not more than 7 grms. of potassium nitrate. But doses in excess of this amount lead to nitrate excretion. After a dose of 15 grms. of potassium nitrate given in 6 litres of water, only 0·5 mgrm. of nitrate (as N_2O_5) was found in 1000 c.c. of milk. After a single large dose the maximum excretion appeared in 9 hours.

Sweetness of Sugars. Beister, Wood and Wahlin. (*Amer. J. Physiol.*, 1925, **73**, 387–400 ; *J. Inst. Brew.*, 1926, **32**, 38.)—The relative sweetness of sugar solutions has been determined by the " minimum concentration " method. With cane sugar as 100, laevulose was 173·3 ; dextrose, 74·3 ; maltose, 32·5 ; and lactose, 16·0. The value found for invert sugar made from sucrose with invertase

was 127·4, whilst that of the invert sugar made by mixing equal weights of dextrose and laevulose was 130 ; in each case allowance was made for the fact that 100 parts of cane sugar yield 105 parts of invert sugar (cf. ANALYST, 1922, **47**, 261).

Oxygen and Carbon Dioxide Limits in Respiratory Air. A. Grögli. (*Arch. Hyg.*, 1925, **95**, 160–173 ; *Chem. Abstr.*, 1925, **19**, 2677.)—Experiments were made to establish the limits of carbon dioxide and oxygen concentration compatible with life in resting guinea-pigs, rats and mice under different conditions of temperature. In the first series of experiments the air in the glass box containing the animals was frequently changed, so as to have natural conditions. In the second series the carbon dioxide was artificially absorbed, in order to discover the influence of a reduced quantity of oxygen. In the third series carbon dioxide was admitted in excess of the normal quantity in the air, and in the fourth series oxygen and carbon dioxide were admitted together. In the first series of experiments the animals remained alive for 45 to 70 hours at 15° to 31° C., during which time the carbon dioxide rose to 14·6–15·9 per cent., and the oxygen sank to 4·5–5·2 per cent. When the animals were kept at 32° to 34° C., or at 2° to 5° C., most of them died at a concentration of oxygen of 7–8 per cent., and of carbon dioxide of 11·8–12·7 per cent. If the carbon dioxide was absorbed the animal remained alive longer. On the reduced oxygen concentration—at most from 58 to 83 hours with the oxygen concentration reduced to 3·1–3·4 per cent. At 30° to 31° C. the ability to resist was less and the animals died at an oxygen concentration of 4·9–5·8 per cent. If the temperature was increased further to 32° to 34° C., or lowered to 3° to 5° C., the demand for oxygen was greater, and death occurred at an oxygen concentration of 6–7 per cent. When the carbon dioxide content was increased to 12·6–17·2 per cent. at room temperature the animals lived 20 hours, during which time the carbon dioxide rose to 20·8–22·7 per cent., and the oxygen fell to 9·6–12·8 per cent. With the simultaneous artificial increase in oxygen and carbon dioxide the animal tolerated more carbon dioxide. All experiments were carried out with and without partial absorption of the water vapour produced. The results show that the minimum limit of tolerance to reduced oxygen and the maximum tolerance to carbon dioxide is not constant, but depends upon the temperature.

Fate of Creatine when Administered to Man. A. Chanutin. (*J. Biol. Chem.*, 1926, **67**, 29–41.)—A discussion of the creatine-creatinine metabolism must be of a somewhat hypothetical nature because of the number of unknown factors to be considered. Feeding experiments were carried out with creatine on man under fairly constant conditions by introducing a large excess of creatine. Total nitrogen in the urines was determined by the Kjeldahl-Gunning method, creatinine by the Folin method, and creatine by the autoclave method of Folin. Creatinine zinc chloride was used as a standard for the creatine and creatinine determinations. Ammonia nitrogen was determined by the procedure of Van Slyke and Cullen. Tables give detailed results of the experiments, which show that creatine and creatinine are not independent of one another in the body. Creatine appears to

be absorbed completely from the alimentary tract, carried to the tissues, and either stored there or rejected at once, and eliminated through the kidneys. There is no evidence of its bacterial destruction in the alimentary tract. The creatinine content of the urine in man increases after ingestion of large doses of creatine. Extra creatinine excretion is derived directly from the creatine taken by the mouth. There is evidence that creatine has an indirect action on nitrogen metabolism.   P. H. P.

Concentrations of Certain Constituents in Normal Beef Plasma. C. S. Robinson and C. F. Huffman. (*J. Biol. Chem.*, 1926, **67**, 245–255.)— Experiments were carried out to determine : (1) The variations in the composition of the blood of normal cattle, and (2) the magnitude of these variations taking place in 24 hours. The components studied were inorganic phosphorus, chlorine, carbon dioxide and calcium. Some determinations were also made of potassium and magnesium. The blood samples were taken from animals of various ages and sexes, all of which were on ordinary herd rations except in a few cases, when calcium phosphate was added. The methods used for the determinations are outlined, and charts and tables show the results. An analysis of more than 100 samples of blood from normal mature cattle showed the following values :

	Range.	Average.	Maximum 24 hr. variation.
	Mgrms. per 100 c.c.	Mgrms. per 100 c.c.	Mgrms. per 100 c.c.
Inorganic phosphorus	3·00–8·99	5·87	1·87
Chlorine	294–357	329	24
Calcium	7·7–14·7	11·0	2·0
Carbon dioxide	41·4–75·8	59·2	14·0
Potassium	16·4–41·3	27·3	—
Magnesium	0·31–3·08	2·16	—

Increased intake of calcium phosphate produced a slight increase in the inorganic phosphorus and calcium in the blood, but the values were still within the normal range. Other factors produced greater changes in these two constituents.
                                                        P. H. P.

New Sulphur-containing Compound (Thiasine) in the Blood. S. R. Benedict, E. B. Newton and J. A. Behre. (*J. Biol. Chem.*, 1926, **67**, 267–277.)— The preparation, composition and some of the properties are described of a new compound, isolated in crystalline form, which is responsible for the interference in the direct methods for the determination of uric acid in blood. The substance has been called thiasine, and melts sharply at 262°–263° C. Elementary analyses yielded the following results :—

	Carbon.	Nitrogen.	Hydrogen.	Oxygen.	Sulphur.
Found, per cent.	47·35	18·20	6·55	17·10	10·30
Calculated for $C_{12}H_{20}N_4O_3S$, per cent.	48·0	18·6	6·6	17·2	10·6

Hydrochloric acid content of thiasine hydrochloride : calculated 10·8 per cent. ; found 10·9 to 11·8 per cent. On this basis the molecular weight of thiasine appears

to be 300, as required by the above formula. Thiasine is strongly dextrorotatory, $[\alpha]_D = +116°$, and it occurs in human bloods in sufficient quantity to account for much of the rotation observed in filtrates from whole blood. The only positive reaction of thiasine, so far found, is the reduction of the complex tungstic acid reagents commonly used for the determination of uric acid. In the presence of cyanide as the only alkali, and with the use of the reagent and technique of Benedict (*J. Biol. Chem.*, 1922, **51**, 187), 1 part of uric acid yields a colour equal to that given by 7·5 parts of thiasine. Mixtures of uric acid and thiasine in any proportion yield a colour equal to the sum of the two when determined separately. So far only pig and human bloods have been examined for thiasine, and in both of these the thiasine is contained wholly in the corpuscles. A procedure for its determination in blood is outlined. Colorimetric determinations showed an average thiasine content for pig's blood of about 25 to 30 mgrms. per 100 c.c. and for human blood of about 14 to 15 mgrms. per 100 c.c. of blood. The molecular structure of thiasine is still being studied. P. H. P.

Correction of Folin-Wu Blood Sugar Values. B. L. Oser and W. G. Karr. (*J. Biol. Chem.*, 1926, **67**, 319–323.)—Folin and Wu (*J. Biol. Chem.*, 1920, **41**, 367) included two standard solutions in their method for the determination of blood sugar because the blue colour obtained does not vary directly as the concentration, but this is not always satisfactory. Since the deviation of the observed from the actual values for given concentrations of glucose is constant, it is possible to correct the values obtained by the original Folin-Wu method by reference to a curve constructed from readings of solutions of known concentrations. Determinations were made on solutions of pure glucose (99·6 per cent. by polarimetric and gravimetric copper reduction methods) in 0·2 per cent. benzoic acid. The colorimetric ratios were plotted as abscissae against the actual concentrations as ordinates. The values may be assumed to fall along straight lines (almost on the 45° line), and the equations for the lines are :— $y_1 = 0·88x_1 + 12$ and $y_2 = 0·855x_2 + 29$, in which x_1 and x_2 are the values obtained when using the weak and strong acids, respectively, and y_1 and y_2 are the corresponding actual concentrations. A figure of the curve is given ; also tables of values calculated from these equations. P. H. P.

Quantitative Methods for the Determination of Iron in Biological Materials. C. A. Elvehjem and E. B. Hart. (*J. Biol. Chem.*, 1926, **67**, 43–51). Three methods are discussed for the accurate determination of iron in biological materials, (1) the method of Thomson (*J. Chem. Soc.*, 1885, **47**, 493), which may be used for materials relatively high in iron and low in phosphorus, such as lettuce ; (2) the modification of Walker (ANALYST, 1925, **59**, 279), which may be used for materials somewhat rich in phosphorus, such as cabbage ; and (3) a new method which may be used for materials relatively low in iron and high in phosphorus, such as milk. In Thomson's method the material is ashed, and the ash dissolved in hydrochloric acid. A small amount of potassium permanganate is added to

insure oxidation of all the iron to the ferric state, and ferric thiocyanate is formed through the addition of potassium thiocyanate. A colorimetric determination is then made against a standard iron solution. With materials such as milk the thiocyanate colour faded immediately, owing to the presence of phosphates. The authors tried various modifications, and finally devised a satisfactory method for the determination of iron in such substances. The phosphorus was completely removed with ammonium molybdate, leaving excess of molybdate, iron, calcium and magnesium in the filtrate. Upon addition of potassium hydroxide solution the molybdate dissolved as potassium molybdate, and the iron was precipitated as iron hydroxide, together with the calcium and magnesium hydroxides, which were filtered off on an asbestos Gooch crucible. The iron was dissolved in hydrochloric acid and determined colorimetrically. Full experimental details are given. Walker's method involves the use of nitric acid instead of hydrochloric acid.

P. H. P.

Benzidine as a Reagent for Living Plants. C. Rouppert. (*Comptes Rend.*, 1926, **8**, 533–535.)—Axial sections of the hypocotyledonary portions of various types of haricot bean (*Phaseolus vulgaris*) give colour reactions with benzidine on oxidation with hydrogen peroxide, the colours varying according to the degree of oxidation. On cultivating the beans in weak (0·01 per cent.) benzidine solutions, the Mangin-Raciborski reaction for lignified tissue was obtained, and in the case of the red varieties of bean containing tannin substances, the presence of an oxidase was demonstrated in the pectin walls of the tannin ducts. D. G. H.

Inactivation of Vitamin *A* by Rancid Fat. W. C. Powick. (*J. Agric. Res.*, 1926, **31**, 1017–1026.)—Pronouncedly rancid lard did not appear to be actually toxic to rats, but its inferiority to sweet lard appeared to lie in its destruction of vitamin *A* due to oxidation by organic peroxides. Destruction was more significant the more intimate and prolonged the contact of the rancid fat with the vitamin, and appeared to be gradual over a period of several days. D. G. H.

Relative Effectiveness of Disinfectants. H. Lüers and F. Weinfurtner. (*Woch. Bran.*, 1926, **43**, 35–39, 45–49 ; *J. Inst. Brewing*, 1926, **32**, 121.)—The relative toxicities towards yeast of a number of disinfectants have been determined by a modification of the Rideal-Walker method, a 1 per cent. solution of phenol being used as the standard. The relative values obtained were as follows :—

Ammonium bifluoride	0·4
Hydrofluosilic acid	0·4
"Pyrizite" (a sodium borofluoride preparation)	0·6
Formaldehyde	0·9
Phenol	1·0
Formic acid	1·6
"Antiformin" (sodium hypochlorite and caustic soda)	3·3
"Radoform" (similar to antiformin) ..	3·3
"Mianin" (sodium salt of paratoluene sulphochloramide)	5
Benzoic acid	5
"Aktivin" (similar to mianin).. ..	8
"Chloramin" (similar to mianin) ..	9
"Pantosept" (sodium salt of dichlorylparasulphamino benzoic acid) ..	11–13
"Novocit"	12–13
Sulphurous acid	25
"Magnocid" (basic magnesium hypochlorite)	30
Salicylic acid	35
Bleaching powder	35
Active chlorine	65
Cyclo-telluro-dimethylpentane diketone (probably impure) ..	80
Mercuric chloride	110
"Caporite" (crystallised calcium hypochlorite)	120

Water-soluble Vitamin Content of the Velvet Bean. W. D. Salmon and E. R. Miller. (*J. Agric. Res.*, 1925, 31, 793–799.)—Pigeons suffering from pronounced polyneuritis on a diet of polished rice were restored to an apparently normal condition by 1 to 2 grms. of finely ground velvet beans (*Stizolobium deeringianum*). Alcohol and acetic acid extracts were also successfully used. Two grms. of raw velvet beans, *i.e.* 9·5 to 13 per cent. of the total diet, acted as a preventive against polyneuritis, but when thoroughly extracted with alcohol 2 to 4 grms. of beans had no protective action. Large amounts of beans, however, were found to be harmful both to pigeons and rats. Cooking or autoclaving at 15 lbs. pressure did not lessen their harmful effect on rats.   D. G. H.

Vitamins in Canned Peaches. E. F. Kohman, W. H. Eddy, V. Carlsson, and N. Halliday. (*Ind. Eng. Chem.*, 1926, 18, 302–303.)—The usual canning process has no great effect on the vitamin C content of peaches; the removal of oxygen from the peaches may afford some protection to the vitamin, but the extent of the protection is insignificant. With regard to the vitamins A and B, the results of the experiments were inconclusive.   W. P. S.

Toxicological and Forensic.

Toxicity of Carbon Tetrachloride. M. Khalil. (*Lancet*, 1926, 210, 547–548.)—Some supplies of so-called pure carbon tetrachloride have been found to be toxic to experimental animals, and were probably toxic to man. The toxicity cannot be attributed to carbon disulphide, since when quantities varying from 1 to 5 c.c. of carbon disulphide were added to one dose (5 c.c.) of pure carbon tetrachloride, and the mixture administered to experimental animals, including monkeys, no ill-effects were produced. On the other hand, the first fraction of a distillate of sulphur-free carbon tetrachloride proved non-toxic. The toxic impurities are probably sulphur compounds distilling at a lower temperature than carbon tetrachloride (76° C.). They may be eliminated by distilling the carbon tetrachloride and throwing away the first portion of the distillate (about 1 per cent. of the total quantity). The most effective dose of carbon tetrachloride (5 c.c.) for treating *Ankylostoma duodenale* is perfectly safe if the purified drug is used.

Haemolysins of Fungi. V. Pettinari. (*Arch. Farm. Sper.*, 1925, 39, 162–172; *Chem. Abstr.*, 1925, 19, 2353.)——The *Amanita* haemolysin is not a specific toxic principle of any particular species, but a substance widely distributed among diverse species of both toxic and edible fungi. The amount varies in different parts of the fungus, and is found in the cuticle, gills and stalk in decreasing order. In general, the amount is so small that Ferri's method of demonstrating haemolysins is, for all practical purposes, specific for *Amanita phalloides* and the highly toxic fungi. The test can be made much more delicate by using 5 per cent. of defibrinated

blood in physiological salt solution instead of entire blood. The haemolytic substance is partly volatile, and the amount diminishes when the fungus is dried. When injected or administered orally in the amount present in fungi no appreciable toxic action is observed, and even in the poisoning of human subjects it plays only a secondary rôle. In the toxicological classification of fungi the group containing a labile haemolytic principle in Ferri's sense should be omitted. The only haemolytic toxic principle capable in itself of causing poisoning and known to occur in fungi is helvellic acid. The distillate from *Amanita phalloides* is non-toxic ; hence the common belief that the fungus contains a volatile poison is erroneous.

Agricultural Analysis.

Determination of Ammoniacal Nitrogen in Manures. F. Chastellain. (*Helv. Chim. Acta.*, 1926, **9**, 205–216.)—The methods available for this determination are discussed in detail, with special reference to manures containing urea and dicyandiamidine. (1) When neutral formaldehyde and ammonium salt solutions are mixed, the acid of the salt is liberated, and may be detected by an indicator. The drawbacks to this method are : (*a*) The tendency of the formaldehyde to liberate acid from amino-substances present, and (*b*) the lack of a suitable indicator. Phenolphthalein cannot be used in the presence of cyanamide unless dicyandiamidine is absent. Rosolic acid, however, may then be used, if any phosphates are removed by milk of lime. If used with caution, this and the following method give reliable results. (2) Magnesia liberates ammoniacal nitrogen from solutions at the boiling-point, but does not attack the other nitrogenous compounds appreciably unless the urea content is above 25 per cent. The distillation should not be carried too far, and the use of reduced pressure is to be preferred. (3) Sodium hydroxide displaces ammonia from solutions of its salts in the cold. The usual method, which depends on the quantity of ammonia liberated after various periods of time, is replaced by a determination of the ratio of the amounts evolved from two different samples, one of known composition. This is equal to the ratio of their ammoniacal nitrogen contents. Under the conditions specified this provides an exact method, and is recommended for general use. The determinations involving precipitation as magnesium ammonium phosphate and the use of phosphotungstic acid are not discussed. J. G.

Age of Wheat determined from Temperature of Germination. O. Munerati. (*Comptes Rend.*, 1926, **8**, 535–537.)—Wheat grains of the current year germinate best at temperatures of 12°–14° C., few germinating at 22°–24° C. or at 5°–7° C. Those of the previous year germinate well over the whole range of temperature, but they tend to get ahead of the current year's grains at 12°–14° C., and to be behind at 5°–7° C. Older grains do best at 30°–32° C., and 22°–24° C., and are slow at 12°–14° C. and slower still at 5°–7° C. Other cereals behave similarly. D. G. H.

Possibilities and Limitations of Chloropicrin as a Fumigant for Cereal Products. R. N. Chapman. (*J. Agric. Res.*, 1925, **31**, 745–760.)—Chloropicrin has unusually good penetrating power, but at the same time tends to escape through the walls of the fumigating chamber, so that, unless it is used as a spray and a high concentration is built up in the atmosphere before it escapes, the concentration may fall below lethal point. The confused flour beetle (*Trilobium confusum*) was killed in the centre of a 98 lb. sack of flour in less than 24 hours with a concentration of about 32 grms. per cb. metre in a wooden box. This insect required from 30 to 50 per cent. greater dosage than the granary weevil (*Calendra granaria*). Indian-meal moths (*Plodia interpunctella*) and their pupae in the cracks of the wall of a room, 80 by 18 feet, were destroyed by spraying the walls, ceiling and floor, with chloropicrin (32 grms. per cb. metre for the volume of the room). Chloropicrin is highly toxic to insects, and with a concentration between 1 and 125 grms. per cb. metre the factors of time and temperature bear an inverse ratio to each other, and concentration being constant, time to kill bears a linear relationship to temperature down to 0° C. Chloropicrin was found to have a retarding effect on yeast activity, and a deleterious effect on the properties of gluten. Bread-making tests showed that, given sufficient aeration or exposure to the atmosphere, treated flours or wheats can recover from the effects of the chloropicrin.   D. G. H.

Differentiation of Ravison Rape Seed and Black Mustard Seed. Vizern and Guillot. (*Ann. Falsif.*, **206**, 1926, 87–88.)—Mere visual examination, even with a lens, is not to be relied upon. If examined under a microscope at a magnification of 200 diameters, mustard seed shows a reticulated skin traversed by ribs which divide its surface into small hexagonal hollows. Ravison rape seed, on the contrary, is almost smooth, slightly dotted and devoid of hexagonal figures. In section, the most characteristic distinction is the appearance of the sclerotic layer of integument. That of mustard seed is composed of a single stratum of cells, the smallest having a square section. On both sides the height of the cells increases continuously, forming a salient in the mucilaginous layer, and then decreases. This effect is reproduced at almost regular intervals, giving the section the appearance of a festoon. With ravison rape seed, however, the sclerotic layer is composed of cells taller than they are broad and having all the same height. The brownish-red grains, considered by some to be mustard seed not fully developed, are shown by the above method of examination to be identical with ravison rape seed. The presence or absence of these hexagons may be taken as being specific for distinguishing mustard seed and ravison rape seed. The proportion of essential oil of mustard in the black grains and red grains of ravison rape seed grown in Roumania was the same, *viz.* 0·13 per cent.   R. F. I.

Organic Analysis.

Determination of Nitro-Compounds by Reduction with Titanous Chloride. I. M. Kolthoff and C. Robinson. (*Rec. Trav. Chim.*, 1926, **45**, 169–176 ; *cf. id.*, 1924, **43**, 775).—In order to eliminate decomposition of the titanous chloride, the authors carry out the reduction at a low hydrogen ion concentration. Sodium citrate is used as the buffer substance, and in its presence nitro-compounds are rapidly reduced by titanous chloride at room temperature. To 25 c.c. of the nitro-compound and 30 c.c. of 20 per cent. sodium citrate solution, are added about 0·2 grm. of solid sodium bicarbonate. The carbon dioxide evolved removes the last traces of air from the solution, and a stream of this gas is passed through the solution during the whole procedure. The 0·05 N titanous chloride solution is then added till the intensity of the violet colour indicates an excess of 1 to 3 c.c. Reduction occurs within two minutes, after which the solution may be back-titrated with 0·025 N iron alum solution either potentiometrically or, after acidification, with ammonium thiocyanate as indicator. The titanous chloride is standardised against a 0·05 N potassium dichromate solution, since the objections to this method raised by Zintl and Rauch (*Z. anorg. Chem.*, 1925, **146**, 281) have not been confirmed. A blank of 0·2 c.c., due to the sodium citrate, must be subtracted from the amount of titanous chloride solution used, but that due to the sodium bicarbonate is negligible. The method has been tested and found satisfactory for six nitro-compounds. J. G.

Formaldehyde Titration of Certain Amino-Acids. S. L. Jodidi. (*J. Amer. Chem. Soc.*, 1926, **48**, 751–753.)—Cystine may be accurately determined by " formol" titration, and so may hippuric acid ; with the latter, however, ordinary titration with alkali hydroxide gives equally exact results. Aceturic acid probably gives results similar to those with hippuric acid. Tryptophan behaves similarly to histidine and proline towards formol titration, which indicates about 87 per cent. of the true quantity. T. H. P.

α-Naphthyl Isocyanate as a Reagent for Alcohols. V. T. Bickel and H. E. French. (*J. Amer. Chem. Soc.*, 1926, **48**, 747–751).—a-Naphthyl isocyanate reacts readily in the cold with primary alcohols, giving good yields of urethanes which crystallise well and melt sharply. Citronellol does not react, but both hydroxyls of ethylene- and trimethylene-glycols, and all three of glycerol react. Ethylene- and trimethylene-chlorohydrins give good yields, and the corresponding bromohydrins slightly lower yields, of urethanes. Secondary alcohols react well when heated with the reagent, but tertiary alcohols give unsatisfactory results. The presence of moisture is not detrimental to the reaction with alcohols of low molecular weight or with those which form urethanes in the cold, but it interferes seriously in cases where heating is necessary. The urethanes are easily separated from dinaphthylcarbamide, which is the only by-product, owing to their ready solubility in hot petroleum spirit. The formation of ethers from sodium sym-

tribromophenoxide and the chlorides of tertiary alcohols is not a satisfactory means of identifying such alcohols, as the chlorides of the purely aliphatic tertiary alcohols do not react in this way.   T. H. P.

Standardisation of the Sandmeyer Reaction, with Special Applications. **H. S. Fry and I. W. Grote.** (*J. Amer. Chem. Soc.*, 1926, **48**, 710–714.)—A standard method of carrying out the Sandmeyer reaction is proposed, the reagents being used in the proportion of 10 mols. of acid to 1 mol. of cuprous salt and 1 mol. of amine. Possible variations due to catalytic effect and changes of concentration are avoided by the use of a uniform amount of sodium nitrite slightly in excess of that required theoretically for diazotisation. The results obtained show that the introduction of chlorine, bromine, and iodine into para-halogenated amines gives practically identical average yields of the dihalogenated benzenes when the diazonium salt is added to either a hot (100° to 105° C.) or a cold (0° to 5° C.) cuprous halide solution. Hence the conflicting statements published concerning the relative merits of the hot and cold methods are probably due to the varying conditions employed.

The character of the halogen atom already in the para-halogenated aniline or the presence of a nitro group appears to have little if any effect on the yield of para-halogenated derivative. In the standardised cold method as applied to para-halogenated anilines, cuprous chloride and bromide may be replaced satisfactorily by the cupric salts.   T. H. P.

Thiocyanogen Absorption of Oils and Fats. H. P. Kaufmann. (*Z. Unters. Lebensm.*, 1926, **51**, 15–27.)—Continuing his work on the application of the method of thiocyanogen absorption (ANALYST, 1926, 157), the author gives the following results obtained with other oils and fats :—*Cacao butter* :—The iodine value (37·53) agreed with that calculated from the thiocyanogen absorption. Hence this sample did not contain the triglyceride of linolic acid. *Coconut oil* :— The iodine and thiocyanogen absorptions agreed, so that here, again, the fat did not contain linolic acid. *Poppyseed oil* had an iodine value of 78·7, calculated from the thiocyanogen absorption, as compared with the ordinary iodine value of 133·6. These results correspond with 27·48 per cent. of triolein and 63·81 per cent. of trilinolin. The corresponding quantities found by Hazura and Grüssner were 30 per cent. and 65 per cent. *Arachis oil* :—Five specimens of different origin gave iodine values ranging from 87·1 to 89·3, whilst the iodine values calculated from the thiocyanogen absorption varied from 68·1 to 78·5. From these values it was calculated that the oils contained the following amounts of oleic and linolic acids :—I. oleic 80·7, linolic 10·0 ; II. oleic 58·55, linolic 22·45 ; III. oleic 55·66, linolic 23·14 ; IV. oleic 63·86, linolic 18·6 ; V. oleic 61·32, linolic 19·76 per cent. *Sesame oil* :—Three different oils had iodine values of 108·9, 109·8 and 107·1, and iodine values corresponding to the thiocyanogen absorption of 75·7, 76·6 and 77·02. These values correspond to : I. oleic acid 48·6, linolic acid 38·84 ; II. oleic acid 51·7, linolic acid 37·78 ; III. oleic acid 54·2, linolic acid 34·96 per cent.

Analysis of Mixtures.—It is shown by the results obtained with experimental mixtures that it is possible, from the different values, to calculate the proportions of a mixture of two or three oils and fats. For instance, a mixture of poppyseed oil, arachis oil and coconut oil had an iodine value of 74·19, whilst the iodine value calculated from the thiocyanogen absorption was 51·94. The constants of the three fats used were as follows :—Poppyseed oil : iodine value, 133·6, and iodine value from thiocyanogen absorption, 78·7 ; arachis oil : iodine value 88·0, iodine value from thiocyanogen value, 68·1 ; coconut oil : iodine value by each method, 9·4. The percentage composition of the mixture was therefore as follows :

	Taken.	Found.
Poppyseed oil	25·06	24·51
Arachis oil	42·96	43·06
Coconut oil	31·98	32·43

The following equations were used for the calculation :

$$x + y + z = 100$$
$$133{\cdot}6x + 88y + 9{\cdot}4z = 74{\cdot}19 \,.\, 100$$
$$78{\cdot}7x + 68{\cdot}1y + 9{\cdot}4z = 51{\cdot}94 \,.\, 100.$$

Elaeostearic Acid.—Experiments with elaeostearin have shown that the iodine value calculated from the thiocyanogen value is exactly half of the iodine value, as in the case of linolic acid ; hence the method can be used to determine the composition of tung oil.

Differentiation of Cotton and Kapok. A. Lejeune. (*Bull. Soc. Chim. Belg.*, 1925, **4**, 419–421).—Kapok is coloured yellow and cotton is unaffected by an aqueous solution of aniline sulphate. After immersion in a 10 per cent. solution of potassium iodide containing 5 per cent. of iodine, and subsequently in concentrated sulphuric acid, or better, in a cooled mixture of 4 vol. sulphuric acid, 1 of water and 1 of glycerin, cotton is coloured blue-black and kapok yellow-brown. Iodine in potassium iodide solution to which zinc chloride has been added produces similar colorations. These tests cannot be used for the approximate determination of the two kinds of fibres in mixtures, and the coloration in the aniline sulphate test is not very pronounced. Since basic triphenylmethane colours have a selective preference for kapok, it is possible to distinguish kapok and cotton in a mixture by placing the material in a boiling neutral bath of malachite green, followed by a slightly ammoniacal bath of oxamine red which is at boiling point, but is not boiled after immersion of the fabric. Under these conditions kapok is stained dark green and cotton bright red, and an approximate determination of their proportions is practicable. D. G. H.

Detection and Determination of Glycerol in Cotton Cloth and Sized Yarns. G. Smith. (*J. Textile Inst.*, 1926, **17**, T.187–191).—*Qualitative.*—Five to twenty grms. of the cloth, depending on the glycerol content, are extracted for 3 hours in a Soxhlet apparatus with 95 per cent. alcohol. When cold, the

solution is filtered, evaporated to about 2 c.c., and shaken with 20 c.c. of warm very dilute hydrochloric acid. This solution is filtered from precipitated free fatty acids, boiled with slight excess of sodium carbonate, to remove any zinc, and after all carbon dioxide has been boiled off, magnesium is precipitated with a few drops of 10 per cent. sodium hydroxide solution. The liquid is again filtered, and the filtrate neutralised with hydrochloric acid and evaporated to about 5 c.c. This solution is now " benzoylated " by shaking it with 1 c.c. of benzoyl chloride and 5 c.c. of approximately 2 per cent. sodium hydroxide solution (methyl red as indicator), sufficient alkali being used to dispel the odour of benzoyl chloride and turn the indicator yellow. The somewhat oily product is filtered off, washed with water, dissolved in 2 c.c. of alcohol and allowed to evaporate slowly, preferably in a small test-tube. This glyceryl dibenzoate should melt sharply at 72°–73° C. An alternative (colorimetric) method is that of Denigès :—The cloth is extracted, as described above, with alcohol, and the solution acidified with warm very dilute hydrochloric acid, filtered, and evaporated to about 1 c.c. This is heated on the water-bath with 10 c.c. of a 0·3 per cent. solution of bromine for 20 minutes, or until the yellow colour of the bromine has disappeared. This solution may then be concentrated to 1 c.c., if necessary. On heating, say, 0·2 c.c. of it on a water bath with 2 c.c. of sulphuric acid and 0·1 c.c. of a 5 per cent. alcoholic solution of phenyl salicylate, an intense violet colour is obtained which is said to be specific for glycerol. Codeine gives a greenish-blue coloration, which is also specific for glycerol, but this colour reaction is masked if the solution contains a trace of organic matter which is charred by sulphuric acid.

Quantitative.—The glycerol is isolated, freed from substances likely to interfere (as for the benzoyl process above), and determined by the acetin method. This is only successful when the amount of glycerol in the cloth exceeds 1 per cent.

R. F. I.

Inorganic Analysis.

Absorption of Carbon Monoxide by Cuprous Chloride Solution. **L. Moser and F. Hanika.** (*Zeitsch. anal. Chem.*, 1926, **67**, 448–456).—The effectiveness of the absorption was ascertained in a large number of test determinations. The most effective acid absorbent contains cuprous chloride, 16 to 18 ; hydrogen chloride, 24 to 27 ; and water, 60 to 55 parts by weight. The presence of stannous chloride (0·2 to 0·3 parts of $SnCl_2.2H_2O$) favours the absorption, but larger quantities act unfavourably. The tests prove that ammoniacal cuprous chloride solution is distinctly superior to the acid solution ; the best results were obtained with a solution of the following composition :—Cuprous chloride, 11 to 12 ; ammonia, 13 to 14 ; and water, 76 to 74 parts by weight. One c.c. of this liquor absorbs 31 c.c. of carbon monoxide at 20° C.   W. R. S.

Colorimetric Method for the Determination of Hydroxylamine. **G. W. Pucher and H. A. Day.** (*J. Amer. Chem. Soc.*, 1926, **48**, 672–676).—Hydroxyl-amine may be determined accurately by means of its colour reaction with benzoyl

chloride and ferric chloride. The stock standard solution, which is stable for at least ten months, is prepared by diluting 5 grms. of pure hydroxylamine hydrochloride to 500 c.c. with water. Twenty-five c.c. of this are diluted to 500 c.c. to give a dilute standard, which keeps for two to three months ; 2 c.c. correspond with 1 mgrm. of the hydrochloride or 0·475 mgrm. of the base.·

To 4 c.c. of a solution containing 0·8 mgrm. to 3·5 mgrms. of hydroxylamine hydrochloride, and placed in a 200 × 25 mm. cylinder, marked at 25 and at 50 c.c., are added 2 drops of benzoyl chloride, 4 c.c. of ordinary 95 per cent. alcohol, and 2 c.c. of a solution containing 2 grms. of trihydrated sodium acetate in 100 c.c. and preserved with chloroform. The liquid is shaken for 20 to 30 seconds, left for 2 to 3 minutes, then treated with 2 c.c. of a solution containing 0·5 grm. of hexahydrated ferric chloride, and 2 c.c. of concentrated hydrochloric acid per 100 c.c., and diluted to 25 or 50 c.c. After a few minutes' rest, any unchanged benzoyl chloride remaining on the surface is made to settle by gentle shaking, and the colour of the liquid is then read in the colorimeter ; the presence of a small amount of benzoyl chloride does not impair the reading.

Before the development of the colour, the solution should be rendered neutral or very slightly acid to phenolphthalein, but heating or excess of alkali must be avoided. The colour is stable towards sunlight and electric light, and dilution of the coloured liquid to any desired volume is permissible. The method is applicable in presence of dextrose provided that the ratio, hydroxylamine : dextrose, is not less than 1 : 20. T. H. P.

Air Oxidation of Titanous Sulphate Solution. Vanadous Sulphate, a New and Powerful Reducing Agent. A. S. Russell.

(*J. Chem. Soc.*, 1926, 497).—A solution of titanous sulphate in 4 N sulphuric acid, prepared from titanic sulphate by reduction for 1 minute with zinc amalgam, decantation into a flask and thence to a burette, shows a constant titre with 0·1 N permanganate solution for 12 hours, so that precautions for excluding air are not necessary. Vanadous sulphate is a more powerful reducing agent than titanous sulphate, coming between cadmium and tin in a 2 N sulphuric acid solution. A 0·1 N solution (prepared in the same way as titanous sulphate) may be kept in 10 N sulphuric acid for one hour without special precautions being necessary to prevent oxidation, and it may therefore be used witout excluding air for volumetric determinations which can be completed within that time. D. G. H.

Method for the Electrolytic Separation of Metals. D. J. Brown.

(*J. Amer. Chem. Soc.*, 1926, 48, 582–583).—In electrolytic separations of metals in which the cathode potential is controlled, a wire of the metal that is being deposited may be substituted for the auxiliary electrode used by Sand, being connected with the cell, voltmeter, etc., in the same manner. During the deposition of the metal on the cathode, the observed potential of the cathode is kept at − 0·1 to − 0·2 volt in relation to the wire. The electrolysing current is maintained until it reaches a residual value as in the deposition by Sand's method. For

metals like bismuth with a tendency to form a powdery deposit, the cathode potential is kept somewhat less negative during the early stages of the deposition. The wire and cathode are always weighed together.

When no wire of the metal is available, a platinum wire or, in the case of tin, a copper wire is placed in parallel with the cathode and the current broken a moment before the wire is used as auxiliary cathode. When platinum wire is used in parallel, the potential difference must be low until the wire is covered with deposited metal.   T. H. P.

Volumetric Determination of Uranium, Vanadium, Copper, and Iron in Uranium Ores. A. S. Russell. (*J. Soc. Chem. Ind.*, 1926, **45**, 57–60T.)— The volumetric determination of these four elements in presence of each other can be accomplished by means of $0 \cdot 1$ N solutions of permanganate and thiosulphate, ferrous sulphate in 2 N and uranous sulphate in 4 N sulphuric acid (ANALYST, 1926, 158), and liquid zinc amalgam. 1. (*a*) The first of three portions of the solution in sulphuric acid, free from chloride and nitrate, and containing the elements in the highest state of oxidation attainable by permanganate, is acidified with sulphuric acid to 6 N concentration and shaken in a reagent bottle with 3 per cent. zinc amalgam, without exclusion of air, until the lavender colour of the vanadous salt remains unchanged ; the reduction is extremely rapid. Copper sulphate is reduced to metal, which dissolves in the amalgam. The decanted solution is exposed to the air for half a minute, when any tervalent uranium is converted into the quadrivalent state. The solution and the washings from the amalgam are poured into excess of $0 \cdot 1$ N permanganate ; this is heated to 80° C., and the excess is titrated with uranous sulphate. The permanganate re-oxidises V_2O_2, UO_2 and FeO in the following order : $V^{II} \longrightarrow V^{III}$; $V^{III} \longrightarrow V^{IV}$ and $U^{IV} \longrightarrow U^{VI}$; $Fe^{II} \longrightarrow Fe^{III}$; $V^{IV} \longrightarrow V^{V}$ (the last at 80° C.). Alternatively, the reduced solution may be titrated in the cold till permanently pink, then heated to 80° C., and the titration concluded. (*b*) The solution is now treated with ammonia till a precipitate just does not form, and, when cold, with 5 grms. of potassium iodide and a paste of freshly-made cuprous iodide ; the liberated iodine, after 3 minutes' standing, measured by thiosulphate, gives $Fe^{III} \longrightarrow Fe^{II} + V^{V} \longrightarrow V^{IV}$. Uranyl salt is unaffected by potassium iodide. (2) The second portion is treated with iodide in exactly the same manner as the first ; the liberated iodine in this case measures the changes $Fe^{III} \longrightarrow Fe^{II} + V^{V} \longrightarrow V^{IV} + Cu^{II} \longrightarrow Cu^{I}$. (3) The third portion is treated as the first ; when fully oxidised it is treated with phosphoric acid and a few drops of a saturated solution of diphenylamine in strong sulphuric acid, and titrated with ferrous sulphate till the blue colour disappears. This marks the end of the reaction $V^{V} \longrightarrow V^{IV}$. The ferrous sulphate titration for vanadium is more accurate than that with vanadous, titanous, or uranous sulphate, high results being obtained with these reagents on account of partial reduction of ferric ion. Thus the constituents are determined in 4 titrations: (1*a*) U + V + Fe ; (1*b*) V + Fe ; (2) V + Fe + Cu ; and (3) V. In the analysis of uranium minerals, the finely-ground material is dissolved in *aqua regia* ; silica

and tungstic acid are removed after evaporation. The filtrate is evaporated with sulphuric acid until it fumes, which eliminates lead and barium. If arsenic and molybdenum are present, they are precipitated by hydrogen sulphide, in which case copper is also removed. The filtrate is boiled to expel hydrogen sulphide, and titrated with permanganate till a pink colour just appears ; it is then ready for reduction, etc., as described. Arsenic, molybdenum, and tungsten are the interfering elements that must be removed, whilst chromium is not found in uranium ores. If arsenic and molybdenum are absent, the hydrogen sulphide treatment is unnecessary. W. R. S.

Iodimetric Determination of Arsenic Acid. B. Ormont. (*Zeitsch. anal. Chem.*, 1926, **67**, 417–426.)—The procedure is based upon Rosenthaler's iodimetric method (ANALYST, 1922, **47**, 367), with this modification, that the iodine is boiled off in an open flask, and the resultant arsenious acid titrated. The arsenate solution should contain 15 to 20 per cent. of sulphuric acid or 10 to 12 of hydrogen chloride ; it is treated with a slight excess of potassium iodide and, while kept at 90° to 95° C., it is subjected during 20 to 30 minutes to a current of the gases evolved by the action of strong sulphuric on oxalic acid. The liquid is then diluted to its original volume. The very slight amount of iodine still present is not boiled off, but removed by careful titration with $0.05\ N$ sodium sulphite solution. A slight excess of strong sodium hydroxide solution is added (methyl orange as indicator); the liquid is just re-acidified with sulphuric acid, cooled, treated with the usual amount of bicarbonate, and titrated with iodine. The process is claimed to be expeditious and accurate ; it avoids the possibility of any reversion in the reaction
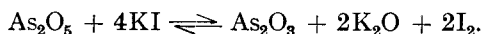
$$As_2O_5 + 4KI \rightleftharpoons As_2O_3 + 2K_2O + 2I_2.\qquad \text{W. R. S.}$$

Determination of Calcium by Conversion of the Oxalate into the Carbonate. H. W. Foote and W. M. Bradley. (*J. Amer. Chem. Soc.*, 1926, **48**, 676–678.)—Calcium may be determined accurately as the carbonate by igniting the oxalate in a current of carbon dioxide at 675° to 800°. The porcelain Gooch crucible (30 c.c.) containing the precipitated oxalate is covered with a Rose crucible lid and supported in a 15 c.c. porcelain crucible, which, after the gradual heating required to dry the precipitate, may be heated with the full flame of a bunsen burner. The current of carbon dioxide is continued until the Gooch crucible is cold. T. H. P.

Physical Methods, Apparatus, etc.

Extension of the Method of Critical Solution Temperatures for the Analysis of Petroleum Spirit. M. Aubert and E. Aubrée. (*Compt. rend.*, 1926, **182**, 577–579.)—For solutions of petroleum spirit in aniline Simon and Chavanne (ANALYST, 1919, **44**, 348) have formulated two expressions connecting the percentages of aromatic, saturated cyclic, and saturated acyclic hydrocarbons with their respective critical solution temperatures and those of the mixture before

and after separation of the aromatics by nitration. These equations may then be combined by elimination of the last-named quantity, and an expression obtained in which the percentages of aromatic and saturated cyclic hydrocarbons are the only unknowns. Similar data for a number of pure and mixed hydrocarbons have been determined with benzyl alcohol as solvent, and it is suggested that from the pair of analogous equations the two unknowns may be calculated. This means the elimination of the nitration process, with consequent economy of time and material. It is important, however, to keep control over the solvents by means of a standardised stock hydrocarbon.   J. G.

Turbidimeter for Measurement of Low Turbidities. J. R. Bayliss. (*Ind. Eng. Chem.*, 1926, **18**, 311–312.)—A sectional diagram of the apparatus is shown in the illustration. The object is to obtain a perfectly blue light below the long glass tubes, so that the observer will see only a blue light when there is no trace of turbidity in the liquid in the tubes. Any matter in suspension reflects white light rays and cuts out some of the blue rays, and a turbidity of " 2 " cuts out almost all the blue light. Standards are prepared as follows :—About 3 grms.

SECTION AA   SECTIONAL   ELEVATION
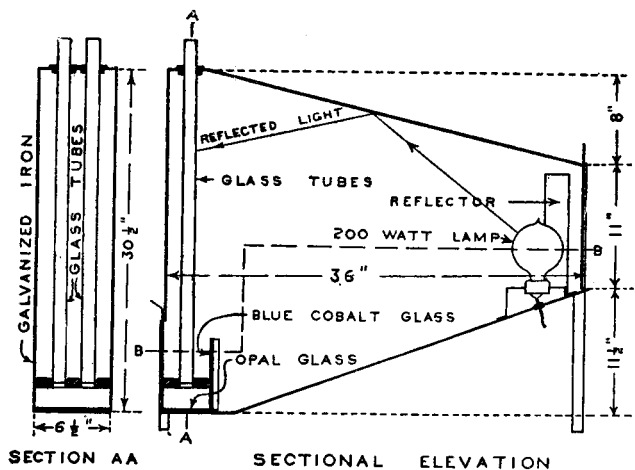
of Fullers' earth are shaken with 1 litre of water, allowed to settle for twenty-four hours, and the liquid is then drawn off without disturbing the sediment, and observed in a Jackson candle turbidimeter. A turbidity between " 25 " and " 40 " is usually observed. This solution keeps fairly constant for some months, and portions are diluted with clear water, as required, to obtain standards ranging from " 0·2 " to " 2."   W. P. S.

New Melting-point Apparatus. J. R. Hosking and W. F. Short. (*J. Soc. Chem. Ind.*, 1926, **45**, 89T.)—In this form of apparatus, the numerous disadvantages of using a liquid heating bath are met by heating the capillary-tube and thermometer in a hard glass tube (A), through which a current of hot air can be drawn by aspiration from a heating unit. This consists of two concentric spiral

copper tubes mounted in a sheet iron case heated by a Bunsen rose burner. One end of the spiral is provided with a gas cock which can be closed, thus regulating the inlet of cold air. The hard glass tube, bent at right angles, is fitted to the copper spiral by packing it into a brass cup with asbestos string and a cement of
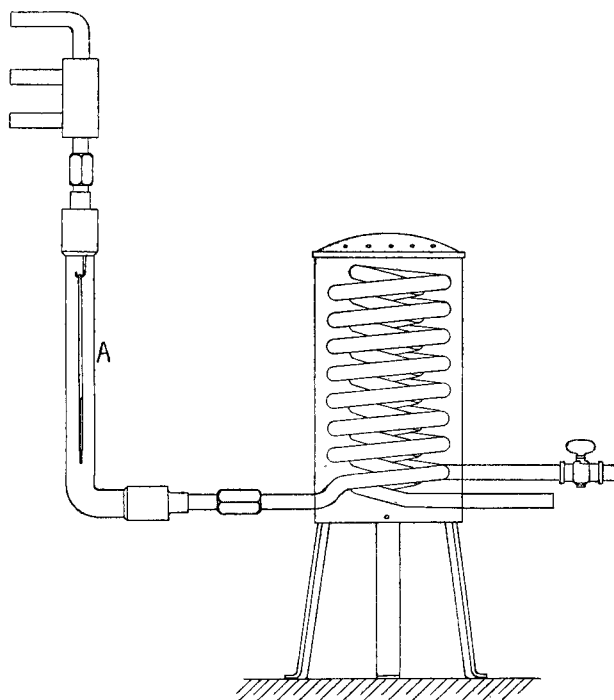
sodium silicate and plaster of Paris. To the upper end is attached a similar brass cup, into which is screwed the hook from which the thermometer is suspended. Above the cup the copper tube is water-cooled in order to protect the rubber connections leading to the water pump. By regulating the gas flame, the rate of suction, and the volume of cold air admitted the rise of temperature can be controlled to almost any desired speed. The higher the temperature required in the glass tube, the greater should be the volume of the air aspirated. Temperatures up to 360° C. are easily attained, and the apparatus quickly cools, when desired, so that a number of melting-points can be determined in a short time.   R. F. I.

Potentiometric Titration of some Oxidising Acids.   M. L. Malaprade.
(*Bull. Soc. Chim.*, 1926, 39–40, 325–336.)—An oxygen electrode, which consists of a platinum wire covered with a coating of gold, has been used in conjunction with a potassium nitrate " bridge," and a saturated calomel electrode for the determination of the P_H of solutions containing oxidising agents. Such an electrode has a potential given by the expression $E = 0.0002\ T\ (K' - P_H)$, where T is the absolute temperature, and K' a constant dependent on the oxidising power of the

solution.   It differs from the ordinary oxygen electrode in that K' is independent of the pressure of oxygen, and it may therefore be used for solutions containing powerful oxidising agents.   The E.M.F. of such a cell is a linear function of the P_H, but varies according to the nature of the solution for a particular P_H.   This involves the elimination of K' by a colorimetric determination of the P_H of a given solution of an oxidising agent.   The determinations may be carried out with an accuracy of the order of unity.   The electrode is used to fix the choice of a suitable indicator, and to investigate the neutralisation-curves of chromic, iodic, and periodic acids.   The existence of the compounds H_4NaIO_6, and $H_3Na_2IO_6$ is recorded, but $H_2Na_3IO_6$ is probably too strongly hydrolysed to be detected.   In the presence of slight traces of oxidising agents, the electrode may also be used for the titration of non-oxidising acids, and yields results comparable with those obtained with the quinhydrone electrode.   J. G.

Reviews.

THE CHEMISTRY OF DRUGS.   By NORMAN EVERS, B.Sc., F.I.C.   Pp. viii. +247.
    London: Ernest Benn, Ltd.   1926.   Price 32s. 6d.

This work is intended to fill a gap which has hitherto existed between text-books of materia medica and the text-books of chemistry.   The author's endeavour has been to describe medicinal substances from the point of view of pure chemistry, dealing with their chemical constitution, reactions, and the manufacturing processes both of those that are of synthetic origin and those that are obtained from natural substances.

The treatise is divided into two parts, of which the first deals with synthetic drugs and the second with drugs of natural origin.   The former substances are classified as hypnotics, antipyretics, anaesthetics, antiseptics, compounds of arsenic, antimony, and mercury, and miscellaneous synthetic drugs.   Drugs of natural origin are dealt with in two groups—alkaloidal drugs and non-alkaloidal drugs.

The two striking features of Mr. Evers's book are the important place he has given to the chemical constitution of the many substances which he has gathered into his volume, and the concise and clear particulars he has given for the production of them.   It is also noteworthy that he has succeeded in satisfactorily condensing his matter within what must be considered somewhat narrow limits. The great number of drugs comprised under the headings named may be estimated from the index, which contains upwards of 1700 items.   There is an appendix on "Chemical Constitution and Physiological Action," and a second one containing lists of "Trade Names," "Synthetic Drugs," "Drugs containing Alkaloids," and "Drugs containing Glucosides."

There are some very evident typographical errors and a few other small blemishes, such as the two statements, within a few lines, that acetylsalicylic acid is soluble in water to the extent of 1 in 400, and that it is insoluble in water.

The author's desire to be terse has occasionally led him into the use of English which is sometimes ambiguous and sometimes clumsy.

Mr. Evers's volume will receive a welcome from everyone whose business has to do with the chemistry of drugs, especially as the author has provided it with a sufficiency of references to permit the working chemist to trace the original sources of the writer's statements. The book is well printed and well bound.

<div align="right">W. KIRKBY.</div>

PRACTICAL PHYSIOLOGICAL CHEMISTRY. By S. W. COLE, M.A. Seventh edition. Pp. xiv. +481. Cambridge: Heffer & Sons. 1926. Price 16s.

A new edition of "Cole" must always mark a minor epoch for chemists whose work brings them at any point in touch with physiological problems. Mr. Cole's fame as a teacher is certainly national, and possibly, for all we know, international; his volume is, therefore, sure of a very wide circulation among medical and biological students in many universities, colleges and medical schools. It was thus inevitable that his book should be cast in the form most suited to such a circulation, but it would be a thousand pities if any false sense of pride among adult analysts prevented their giving this book a place on their shelves and their benches.

To the pathological chemist there will be little, if anything, new in the book, though the thorough revision, and the considerable enlargement made by the author since the sixth edition appeared in 1920, should make it a useful summary of current analytical practice even to those for whom physiological chemistry is an important part of their daily work. But much more for the analyst in consulting practice or in industry must there be occasions when such a book will be found nearly indispensable. It is just to those for whom a urine analysis, or an estimation of blood-phosphate, or a test for bile-salts, is a rare necessity, that a book which describes all these things is thoroughly useful. Even to the research chemist who may have occasion once in a while to prepare a small specimen of, say, tyrosine or cholesterol, Mr. Cole's book might serve as a convenient stop-gap.

Still, it remains true that this is primarily a text-book, and it is therefore pleasant to record that Mr. Cole has been able to bring to the writing of it that lucidity of exposition for which he is well-known as a lecturer. The accounts of, for instance, hydrogen-ion concentration, of biological oxidations and reductions (a new chapter), of the coagulation of blood, to mention three matters at random, seem wholly admirable, and many of the more middle-aged of us could not fail to profit by reading them. That, at any rate, has been the experience of one whose undergraduate days seem to him to be lost in a remote past.

Amongst the welcome enlargements and additions to be found in this edition are accounts of the quinhydrone and of the double hydrogen electrode (the glass electrode is not mentioned), the Werner theory of the structure of urea, the discovery and nature of glutathione, and many others. It is very interesting to note that Mr. Cole describes and recommends strongly the excellent Hagedorn and Jensen method for determining blood-sugar. This is a ferricyanide-iodide method

similar to that made familiar by Macara and Hinton for milk-sugar. Mr. Cole has followed the authors' original details except in one respect; whereas they insist on the necessity of calculating the "glucose found" separately in test and blank, and subtracting the results, Mr. Cole calculates straight from the net titre, by means of a single constant. The error so introduced is slight in cases of normal blood, but the difference in the methods of calculation leads to appreciable differences in result for highly diabetic bloods. Thus a gross titre of 0·22 c.c. with a "blank" titre of 1·87 c.c. shows 0·304 per cent. of glucose, according to the authors' method of calculation, and only 0·292 per cent. by Cole's method.

The publishers have undoubtedly given us good value—quantitatively as well as qualitatively—and the book, clearly printed on adequate paper, is strongly if not elegantly bound. So excellent, indeed, is this volume in almost every respect, that one cannot help regretting the far from satisfactory proof-reading. (H^2) instead of $(H)^2$ on p. 8, a sentence without a verb at the bottom of p. 36, "phosphorous" for the name of the element on p. 416, " a " instead of " a $_4$ " in the formula for xanthydrol on p. 350, a badly misplaced letter near the top of p. 88,—these are some of the "literals" noticed on a quite casual perusal.

With the matter itself there can be few quarrels. Room might perhaps have been found for a page or two on the Donnan equilibrium and its application by Loeb and others to explain certain phenomena of colloid behaviour. Also, the melting point of cholesterol is certainly above 145° C. Finally, the fifth sentence of p. 6, even when allowance is made for a further "literal" error which renders the statement meaningless, is surely rather more lax in expression than we should expect from Mr. Cole. To state that "Sörensen . . . introduced the symbol pH to denote the hydrogen-ion concentration" is to stretch the meaning of the word "denote" quite illegitimately. Mention should also have been made of the meaning and use of the symbol C_H.

But these are, after all, minor blemishes, and, on the balance, one can have nothing but gratitude to Mr. Cole for a very exhaustive and thoroughly useful piece of work.

A. L. BACHARACH.

THE USE OF SOLVENTS IN SYNTHETIC ORGANIC CHEMISTRY. By DONALD W. McARDLE. Pp. vii. +217. London: Chapman & Hall, Ltd. 1926. Price 15s. net.

The work under review is stated by the author to be the first part of a treatise on organic chemistry on the lines of the well-known works of Lassar Cohn and Weyl; in fact, his original idea was to translate one or other of these books, but, in order to bring the subject matter up to date, it was thought best to write a new volume. In order to do this, some 900 original papers have been consulted (full references being given), the work of the author being that of a compiler. An excellent bibliography and three indexes are provided.

The first part of the book is devoted to "general considerations." This is followed by mention of a large number of solvents. Tables of solubilities and some

useful methods of purifying methyl and ethyl alcohols and ether are given. A large number of little-known facts are presented, and, to a casual observer, the book seems entirely satisfactory; but on second reading it becomes apparent that, while much that is of interest to the academic chemist is to be found in its pages, there is no due proportion between the practical utility of a solvent and the amount of space allotted to it. For instance, such technically important substances as isopropyl alcohol, butyl alcohol, and amyl alcohol are only just referred to, and no mention at all appears to be made of butyl and amyl acetates. From this book, too, one would imagine that such commercially important materials as cyclohexanol, tetralin and hexalin are only chemical curiosities.

On the whole, the book is one of academic rather than technical interest, but can be recommended to the notice of those beginning a course of training in organic research.

HAROLD TOMS.

PERFUMES, COSMETICS AND SOAPS, WITH ESPECIAL REFERENCE TO SYNTHETICS. By W. A. POUCHER. Second edition. Vol. I. Pp. xx. +304, with 24 illustrations. London: Chapman & Hall. 1925. 16s. net.

The early demand for a new edition of this work, first published in 1923, has been taken advantage of to thoroughly revise and extend the book, particularly in the direction of soap and tobacco perfumery, as well as by the inclusion of much further information concerning raw materials. This has led to a very considerable increase in the length of the book, necessitating its division into two volumes, and Vol. I. is described as essentially a dictionary of raw materials of value and interest to the chemist-perfumer.

It is, however, a dictionary of the source and uses of the raw materials rather than of their chemistry and properties, some of the latter being given in only very few cases, and this rather lessens the value of the book to the analyst, though to those engaged in the perfumery and allied industries it should be extremely useful.

The book is well printed and illustrated, and it is difficult to think of any raw material which has been omitted. It contains a very large number of formulae for the reproduction of natural perfumes by mixtures of synthetics.

There are a few statements in the book which require amendment, notably where amyl salicylate is said to be prepared by the action of *chlorine* on a solution of salicylic acid in amyl alcohol, when hydrochloric acid must of course be intended. Hard paraffin is said to be chiefly obtained from shale, and no reference is made to the very large quantities produced from petroleum. Oleic acid is stated to be "prepared by the saponification of oleins (!) (tallow, lard, etc.), the soap formed being decomposed with a mineral acid," but the further separation of stearic acid from oleic acid is not even mentioned, and the description of tallow as an "olein" is unusual and rather misleading. Under sodium hydroxide it is stated that this should contain not less than 90 per cent. NaOH, whereas the quality of caustic soda used for toilet preparations should contain not less than 98 per cent. NaOH. It is rather surprising to find under zinc oxide no reference to its addition to toilet

soaps for the purpose of improving the colour.   Coconut oil is still spelt " cocoanut,"
in spite of the fact that this spelling was dropped in most scientific publications
at least ten years ago, and " safrol " is spelt sometimes with, sometimes without,
a terminal *e*.   The use of the expression " which see," in place of the letters *q.v.*,
is somewhat unusual, and the repeated reference to the " Perfumery and Essential
Oil Record " as the " Perfume Record " is liable to lead to confusion.

                                                                    W. H. SIMMONS.

THE LIFE AND CHEMICAL SERVICES OF FREDERICK ACCUM.   By C..A. BROWNE,
    Chief, Bureau of Chemistry, U.S. Department of Agriculture, Washington,
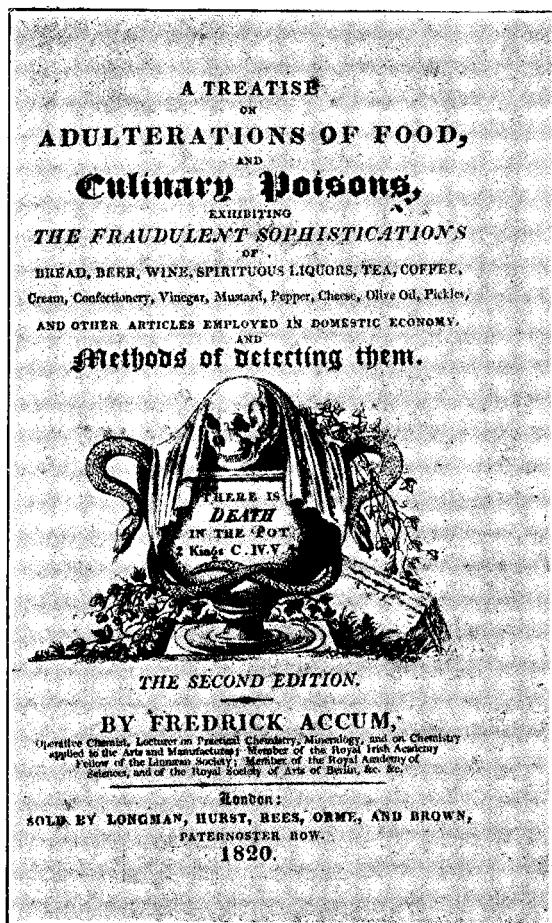   U.S.A.   Pp. 58.   1925.

    The present generation of chemists knows little about Accum beyond the
facts that he had a great deal to do with the introduction of lighting by gas, and
that he wrote a book which gained for him the nickname of " Death in the Pot."
And yet he played such an important part in developing the applications of
chemistry in this country that he has deserved fuller biographical treatment than
is allotted to him in the meagre notice in the *Encyclopaedia Britannica*, which
mainly consists of bald statements of events and dates.   This little monograph
should therefore meet with a widespread welcome, since it not only enables us to
form a mental portrait of Accum himself, but also gives an outline of his chemical
work and a series of pictures of his life in London.   Dr. Browne has been at great
pains to discover every available detail relating to his subject, and, thanks to the
skilful way in which he has handled his material, he has made a permanent addition
to chemical biography.

    From his picturesque narrative we learn that Accum's father was a converted
Jew whose original name, Herz Marcus, became Christian Accum after his baptism
in the Court Church of Bückeburg.   He married a French woman of Huguenot
descent, and Friedrich Christian, the sixth of their seven children, was born in
1769.   The boy was educated in the Bückeburg Gymnasium, and afterwards
studied pharmacy.

    In 1793 he came to London and became an assistant in the pharmacy of
Brande, the father of William Brande, the chemist.   Here he attracted the notice
of William Nicholson, whom he assisted in chemical work, and for whose Journal
he wrote numerous articles, including a report on the production of beet sugar in
Germany.

    About 1800 Accum opened a laboratory and establishment for the supply of
chemicals and apparatus at No. 11 (now No. 33), Old Compton Street, Soho, and
here he gave courses of instruction which it became fashionable to attend, among
those who came to learn a little chemistry being the Dukes of Bedford and
Northumberland.   This school of chemistry also became well known in the United
States, and several Americans who afterwards became famous were trained there,
including Prof. Benjamin Silliman, sen., of Harvard, and Prof. William Peck, of
Yale.   The house in which this early school of chemistry was established might
well be commemorated by one of the L.C.C. mural tablets.

In 1801 Accum became chemical operator at the Royal Institution, where he assisted Davy, and this helped to make him known as a lecturer, and ultimately led to his appointment, in 1809, as Professor of Chemistry in the Surrey Institution, Blackfriars Bridge, where his lectures were so popular that, to quote Silliman's words, he became "a pet chemist to the Londoners."

A TREATISE
ON
ADULTERATIONS OF FOOD,
AND
Culinary Poisons,
EXHIBITING
THE FRAUDULENT SOPHISTICATIONS
OF
BREAD, BEER, WINE, SPIRITUOUS LIQUORS, TEA, COFFEE,
Cream, Confectionery, Vinegar, Mustard, Pepper, Cheese, Olive Oil, Pickles,
AND OTHER ARTICLES EMPLOYED IN DOMESTIC ECONOMY.
AND
Methods of Detecting them.

THERE IS
DEATH
IN THE POT
2 Kings C. IV. V.

THE SECOND EDITION.

BY FREDRICK ACCUM,
Operative Chemist, Lecturer on Practical Chemistry, Mineralogy, and on Chemistry
applied to the Arts and Manufactures; Member of the Royal Irish Academy;
Fellow of the Linnæan Society; Member of the Royal Academy of
Sciences, and of the Royal Society of Arts of Berlin, &c. &c.

London:
SOLD BY LONGMAN, HURST, REES, ORME, AND BROWN,
PATERNOSTER ROW.
1820.

The third chapter of Dr. Browne's biography deals with Accum's work as an industrial chemist, and with his investigations on gas lighting, and in this connection gives an amusing description of his evidence before the House of Commons, where he was sarcastically handled by Brougham (afterwards Lord Chancellor).

It was, however, by his work in exposing the gross adulteration of food and drugs in this country that Accum became most celebrated. As early as 1798 he contributed to *Nicholson's Journal* an article on "An Attempt to discover the Genuineness and Purity of Drugs and Medicinal Preparations," but it was not

until 1820 that his celebrated *Treatise on Adulterations of Food* was published. On the title page of this book he followed the gruesome methods used by Dr. Wilkinson and Dr. Markham in the eighteenth century to arrest the attention of the public, with the result that afterwards the book (and its author) became better known by its motto of "Death in the Pot" than by its proper title. The success of this work was immediate; within two years four editions were published, and it was translated into German and Italian.

It gives an account of the adulteration of bread with such materials as gypsum, alum and pipeclay, of the substitution of sloe, elder and ash leaves for tea, of the thickening of cream with rice and arrowroot, and of cheese made poisonous by annatto adulterated with lead. In the preface we read that "The eager and insatiable greed for gain is proof against prohibitions and penalties; and the possible sacrifice of a fellow creature's life is a secondary consideration of unprincipled dealers."

Not content with thus exposing their practices, Accum also pilloried the offenders by publishing their names, and thereby created powerful enemies who (according to Dr. Browne) were able eventually to drive him out of England.

Apparently Accum had little regard for the lesser moralities. He could see no wrong in palming off old rubbish for the apparatus which Pitt, the Prime Minister, had ordered to be sent to India, and he had no scruples about mutilating the books of other people (as well as his own) if he could thereby collate information required for his work.

Unfortunately he gave way to this evil habit in the library of the Royal Institution, and when some thirty of the missing leaves were discovered in his house, he was at once prosecuted for theft. The magistrate somewhat contemptuously dismissed the charge, but the authorities of the Royal Institution then proceeded against him on the charge of mutilating the books, and he was committed for trial, bail being allowed.

Meanwhile his enemies lost no opportunity of giving the widest publicity to the scandal, and within a month Accum found himself shunned on every side, his career at an end, and his reputation gone. It is little wonder that when his case came up for hearing he did not put in an appearance, having absconded to Germany, leaving his bail to be forfeited.

His last years were spent in Berlin, where, in 1822, he became Professor of Technical Chemistry in the Gewerbe-Institut—a post which he held until his death in 1838.

Such, in brief outline, is the life story of one of the outstanding figures in the chemical world of London at the beginning of the nineteenth century. One can now understand why the name of the chemist who was the first to open a scientific attack upon the stronghold of adulteration should have been allowed almost to die out; and the author of this monograph is to be congratulated on a sympathetic study which must go a long way towards removing that obloquy which has clung to Accum's name for more than a century.

                                                                    EDITOR.