AUGUST, 1925. Vol. L., No. 593.

THE ANALYST

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

Obituary.

WILLIAM JOSEPH DIBDIN.

TYPES of mind and character are apt to recur in families. William Joseph Dibdin had much in common with his great-grandfather, Charles, who wrote:

> " Go patter to lubbers and swabs, do you see,
> 'Bout danger and fear and the like.
> A tight-water boat and good sea room give me,
> And it a'nt to a little I'll strike.
> Though the tempest top gallant mast smack smooth should smite,
> And shiver each splinter of wood,
> Clear the deck, stow the yards, and house everything tight,
> And under reefed foresail we'll scud.
> Avast! nor don't think me a milksop so soft
> To be taken for trifles aback;
> For they say there's a providence sits up aloft
> To keep watch for the life of poor Jack!"

These lines, I think, express very fairly W. J. Dibdin's philosophy of life. An ardent, brave, untiring man, he was too straightforward to study diplomacy, and was, without being aggressive, a fighter.

I first met him in 1894, and the first question he put to me was about Frankland's carbon and nitrogen process, in the value of which he believed to his dying day. I satisfied him and was chosen to work in the old laboratory at Craven Street. He at once impressed one as of a genial disposition, with a hearty and, sometimes, dominant manner which, one realised later, was the expression of a kind heart moved by sincere and compelling convictions. Moreover, however unconventional his chemistry seemed, he always had a clear idea as to what he wanted to find out, a keen eye for a likely road to pursue, and a readiness to adopt any hopeful means to an end.

The tendency many of us suffer from, to let learnedness obscure the real point at issue, never showed itself in Dibdin. He was at his best in attacking a technical rather than a theoretical problem. Whether the moral desire to meet some pressing need or the real love of investigation predominated I cannot say, but both motives were present in a marked degree, and inspired him to unwearying effort.

He was an enthusiastic experimenter, clever at designing apparatus and methods. One may mention the ten candle pentane argand, the radial photometer, the micro-filter, and a very good mercury pump for extracting the gases dissolved in water. A keen and able microscopist, his old staff remember with joy how, by an adroit touch of the cover glass, he convinced a rather highly qualified medical microscopist that a supposed desmid was nothing more than an air bubble.

Dibdin entered the service of the London ratepayers in 1877 as a gas examiner; in 1882 he became assistant to Mr. Keates, chemist to the Metropolitan Board of Works, and on the death of that gentleman in 1883, he showed himself so capable as a witness before Lord Bramwell's Commission* that he was appointed his successor. He worked out a scheme for the treatment of London sewage by chemical precipitants in plant designed by Sir Joseph Bazalgette, engineer to the Board. After this had been in use some few years a prolonged examination of the water of the Thames from Teddington to the Nore, conducted in 1893–4, showed its beneficial results. The report on this work is a little-known classic. Dibdin was, with Dupré, an early and vigorous exponent of the value of the determination of dissolved oxygen in water, and was, I believe, the first to study the rate of absorption of that gas by water. He was a pioneer, in this country, of the biological treatment of sewage, and the one-acre coke bed at Barking was of world-wide fame. The variability of standard candles was one of the causes of uncertainty and dispute as to the illuminating power of gas, and Dibdin's 10 candle pentane argand was found by a Departmental Committee, in 1894, to be the most trustworthy and practical standard then available. It was never prescribed for official testings, as Mr. A. Vernon Harcourt, one of the gas referees, brought out an even better lamp some time after this decision. An exhaustive analysis of the London water supply, of the water of the upper Thames, and of the proposed Welsh supply, convinced Dibdin that the water of the Thames could be brought to a much greater degree of purity than the companies then achieved, and that the grandiose Welsh scheme was unnecessary. Time has shown this verdict to be a *vere dictum*. Other smaller pieces of work were taken up as occasion demanded. His slate bed filter, which, he felt, did not receive justice at the hands of the Royal Commission on Sewage Disposal, and an investigation on the strength of mortars, were later outstanding pieces of work, but I think he was at his best when working for London.

Dibdin joined the Society of Public Analysts in 1889; he served twice on the Council, and was a Vice-President from 1895 to 1896. Among his numerous communications to THE ANALYST, the following may be mentioned:—"Alkaline Waters," (**26**, 20); "The Action of Alkaline Waters on Iron" (**30**, 238); "The

* Royal Commission on Metropolitan Sewage Disposal.

Determination of Dissolved Oxygen in Water" (**26**, 147); "Sewage Effluents" (**23**, 206); "Microscopical Examination of Water" (**21**, 2); and "The Analysis of Sewage Débris in Contact Beds" (**32**, 108).

Official life was difficult for Dibdin. A man of strong convictions, marked individuality and direct methods, he was not always *persona grata* at Spring Gardens. He was unable to disguise his dislike for the political element predominating on the L.C.C. in its early days. Throughout life it was fortunate for him that his joy was in the chase rather than in the spoils.

Dibdin died on June 9, 1925, in his 75th year, the accident which accelerated his death being due to an act of kindly thoughtfulness. His wife, who had helped him in much of his work, and all but one of the large happy family of boys and girls whom one remembers in his home at Sutton, survive him. A man of the widest sympathies and interests, he might have said with Terence:

"Homo sum, nil humani a me alienum puto."

J. H. COSTE.

The Adulteration of Conserves, with Special Reference to Pectin and Agar-agar.

BY JOHN KING, F.I.C.

(*Read at the Meeting, May* 6, 1925.)

LEGAL ASPECTS.—There are in this country no legal standards for conserves, and legal proceedings for adulteration are therefore normally instituted under the Sale of Food and Drugs Act, 1875, Section 6.

It is extremely difficult, in the absence of such standards, for an analyst to adopt any representative procedure, or to standardise his methods with any degree of exactness, so as to detect adulteration of an order that will secure the conviction of an offender.

The section mentioned above requires a vendor to sell the substance demanded, and the purchaser not to be prejudiced in regard to its quality.

Authoritative opinions as to the nature of conserves are not very helpful. Webster's dictionary, often quoted in the courts, defines jam as a "conserve of fruit, boiled in mass with sugar and water."

There is a notable paucity of legal cases dealing with conserves, and High Court decisions have, in some important cases, quashed magisterial convictions, as in the glucose case mentioned later.

The Sale of Food Order of August, 1921, No. 1305, Part IV., which dealt with conserves, has been amended by No. 1883. The sub-clauses 9, 10 and 13, being revoked, no longer constitute legal standards, but nevertheless they still serve the useful purpose of having expressed an authoritative opinion. Thus it may be

assumed that a purchaser of marmalade is prejudiced by the inclusion of fruits and vegetables other than citrus fruits, unless these are specifically mentioned on the label. No definition of *citrus* fruit was given in this order, though the American Food Inspection Decision, 182 (ANALYST, 1921, **46**, 479) lays down standards.

Old and well-known trade customs and methods are generally not held to fall under the meaning of the term adulteration. Thus the inclusion of a considerable percentage of glucose has been held by the High Court not to constitute a prejudice to the purchaser, it being a well recognised trade practice. It is certain, however, that the average purchaser, would if he were aware of the fact, be highly indignant at the inclusion of a chemically prepared substance in place of the sugar which he would normally expect to be the sweetening ingredient of jam.

The use of "stiffeners" appears to fall under a different category, as these are added with the direct object of hiding inferiority.

CHEMICAL AND PHYSICAL ASPECTS.—From the manufacturer's point of view, the ideal to be aimed at is the production of the jam of maximum saleability, at the minimum cost. The extent of inclusion of the least costly fruits may be held to be limited by the Order previously mentioned, but the amount of water that can be added is limited by the saleability of the article. The sweetening matters mostly used are refined sugar, glucose, and "sprayed sugars." The last-named, which are very widely used, are refined sugars, sprayed with 5 to 10 per cent. of a 60–70 per cent. solution of artificially prepared invert sugar. They are considered sweeter, and are cheaper than a pure cane sugar of similar polarisation, and jams prepared from them have less tendency to crystallise on storage. Glucose is reputed to reduce the setting power if present in large quantities.

The problem from the adulterator's standpoint resolves itself mainly into (*a*) the inclusion of fruits and vegetables other than those specified in the description; (*b*) the inclusion of the least costly fruits in an unwarrantable proportion; (*c*) the production of a conserve of a reasonable consistence from the minimum of fruit and sugar, as it is obvious that the public will consider firmness as a criterion of the amount of fruit present.

Consistence in a properly made jam depends almost entirely on the pectin content, and the presence of cellular structure. Pectin, being a colloid, should, for the purpose of producing firmness, be brought into the gel form as much as possible, and manufacturers have resorted to many methods for this purpose. The addition of 1 per cent. of phosphoric, tartaric, or citric acid will often do much to promote gelatinisation.

SPECIAL FUNCTIONS OF PECTIN IN RELATION TO CONSERVES, WITH RECENT WORK IN CONNECTION WITH JELLIES, ETC.—A notable amount of work has been done in America on pectin in relation to the conserve industry. Lal Singh (1) has shown that a definite relationship exists between the amount of acid present in fruit juices and the amount of sugar necessary to form jellies. Between certain limits, the greater the acidity of the juice, and the greater the percentage of pectin, the lower the amount of sugar required. It is therefore desirable to increase the acidity

of the juice to the maximum limit compatible with taste in order to save sugar. By increasing the pectin in a juice from 0·9 to 1·5 per cent. a jelly maker can easily save over 15 per cent. of sugar. He has estimated that waste lemon peel from citric acid factories could yield over 90 lbs. of pectin per ton of peel.

F. Hardy (2) has investigated the effect of different processes for the extraction of pectin from lime rind, and has concluded that the H-ion concentration influences widely both the amount extracted, and the jellying power of the extract.

Johnstin and Denton (3) have pointed out the relationship between the viscosity and jellying power applied to citrous extracts, and H. A. Noyes (4) has shown that different processes for the manufacture of jams and jellies have great effects on the jellying power, the "vacuum cook process" being the most efficient from this standpoint. It seems highly probable therefore, in view of the above work, that great changes in the processes employed in the conserve industry may be expected in the future.

A certain amount of information may be obtained as to the nature of the acids present by the electrical method of titration of a filtered solution of the conserve. The neutralisation curves, however, should be interpreted with caution, owing to the presence of substances having a buffer action. Curves obtained by using jams of known constitution might conceivably be used as standards.

The slow decomposition of pectin, even by boiling citric and malic acids in the concentration usually met with in conserves, as described later, shows that the natural acid content of fruit should be carefully taken into account in jam manufacture. Where the natural pectin content is insufficient, the gelatinising power is often augmented by gelatin, agar-agar, or possibly even by pectin itself, obtained from pomace. Crude pectin has already been placed on the market, and a powder described as "pectin jelly," containing apple pectin, on examination, was found to be correctly described as such.

The addition of gelatin is readily detected by the addition of picric acid to a solution of the matter precipitated by alcohol. Where gelatin is added, the acid content of the jam is of extreme importance. Baker found that a jelly made from gelatin, with acidity of 0·35 per cent. as acetic acid, would set easily at ordinary temperatures, but that at 0·71 per cent. acidity the same concentration of gelatin gave no gelatinisation, even at 0° C.

The detection and determination of agar-agar presents such difficulties, and the methods described in the literature are so unreliable, that it seemed worth while to devise a better method.

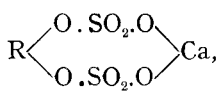
Detection, Determination and Properties of Pectin and Agar-agar.—The methods at present in use for the detection and determination of agar-agar are microscopical and physical. Parkes (5) suggests that, as the solution temperature of agar-agar is 80° C., that of pectin being much lower, the conserve could be freed from its natural pectin by washing with lukewarm water, and filtering. The débris on the filter could then be boiled and refiltered while hot, when all the agar-agar, but none of the pectin, would appear in the filtrate. Recent work by

Schryver, Carré and Haynes, and by Tutin, has shown that it is almost impossible to wash the whole of the pectin from fruit tissue, even on very prolonged treatment. So difficult is this, that the former workers have described a more difficultly soluble form of pectin which they call protopectin, and which could not be washed from fruit tissue by ordinary means (6). Tutin (7) has shown, however, that this is probably merely a question of cell rupture, and that protopectin does not in fact exist as a body separate from pectin.

This work seems to show that provided some unruptured cells exist in a conserve (and microscopical examination almost always shows this to be the case), washing by lukewarm water cannot entirely take out the pectin. Further boiling would inevitably wash out more, and this might prove a source of error in this method.

The microscopical method of detection of agar-agar is discussed later.

Agar-agar is obtained from various closely related seaweeds in the vicinity of Japan, Hongkong and Australia. The purified material has been carefully examined and is known chemically as δ-galactan or δ-gelose. On degradation by acid, arabinose and galactose have been recognised amongst the products. It is readily soluble in water from 80° C. upwards, but it is insoluble in alcohol and acetone. Gelose has never been obtained in a crystalline state, and never quite free from nitrogen, although Klostermann claims to obtain nitrogen-free agar-agar by hydrolysis with alcoholic potash. A somewhat similar substance has been prepared from carrageen moss by Haas, and has been found to contain the radicle



since it gives no precipitate with barium chloride until boiled with concentrated hydrochloric acid (8).

Gelose has been examined by Fairbrother and Mastin and independently by several other workers, and has been found also to contain this radicle (9). The ester has been decomposed by hydrochloric acid in a large number of samples, and the sulphate estimated by precipitation as barium sulphate. The results were fairly constant, varying only from 2·8 to 2·5 per cent. as SO_4. This reaction has been used to determine the agar-agar in a conserve, as described later.

Pectins from such widely separated sources as apple, rhubarb, mangel-wurzel, etc., have been shown by von Fellenburg, Tutin, Tollens, and Carré and Haynes to be identical. Pectin is readily hydrolysed in the cold by dilute sodium hydroxide to the sodium salt of pectic acid, Tutin believing pectin to be the dimethyl-isopropenyl ester of pectic acid (10). Schryver and Haynes have assigned the formula $C_{17}H_{24}O_{16}$ to this acid, and Carré and Haynes have shown that it can be precipitated quantitatively even in very dilute solution from acetic acid solution as the calcium salt (11). It will be seen therefore that, if the pectin from conserves can be precipitated in this way, leaving the agar-agar unchanged, a method for the detection of the latter can easily be devised. In order to test this possibility solutions of agar-agar from several sources were treated in the cold with sodium

hydroxide at varying concentrations, acidified with acetic acid, and calcium chloride added. In no case was there any precipitate.

EXPERIMENTAL.—The completeness of the precipitation of pectin from commercial conserves and its quantitative aspect were studied as follows:—

Fifty grms. of various jams were heated on a water bath in a 500 c.c. beaker, and thoroughly disintegrated by squeezing with a flat-ended glass rod. Where difficulty was experienced owing to the presence of whole fruit, the jam was ground little by little with clean sand in a mortar, until completely broken up. Hot 95 per cent. alcohol, or industrial methylated spirit, was then added, little by little, with constant stirring, until the volume reached 300 c.c. It was then kept at about 50° C., and the mass at the bottom frequently stirred with the rod until inspection showed no gelatinous particles ($\frac{1}{2}$ to 2 hours). By this means the sugary matter was obtained in solution and the pectin precipitated. The contents of the beaker were filtered through coarse filter paper on a Büchner funnel, suction being used only towards the end of the filtration. The filter and its contents were well washed with warm 95 per cent. alcohol, and washed back into the beaker, re-treated with 300 c.c. of hot alcohol and refiltered. The pectin was dissolved from the residue by boiling water, the solution being filtered and cooled. Dilute sodium hydroxide solution was added until an approximate concentration of 0·02 *N* was obtained, and the mixture was allowed to stand one hour in order to complete hydrolysis. Sufficient acetic acid was added to give a concentration of 0·1 *N* free acid, and then 20 c.c. of 10 per cent. calcium chloride solution. After standing one hour the precipitate was boiled for a few minutes, and filtered through a tared, fluted filter paper, the filtrate being treated as below. The gelatinous precipitate was well washed with boiling water, washed back into the beaker, re-boiled with 200 c.c. of water and refiltered through the same filter paper. This paper had been thoroughly washed with boiling water, dilute acetic acid, and more water and dried till constant in weight. The precipitate was dried and weighed.

The amounts of pectin, insoluble in 95 per cent. of alcohol and precipitated by calcium chloride, in some common jams were as follows:—Plum and apple, 0·13; blackberry and apple, 0·25; plum, 0·46; damson, 0·53; raspberry, 0·31; and gooseberry, 0·45 per cent. (expressed as calcium pectate). It will be seen by these results that jams differ widely as regards pectin content, and this suggests a method of quantitative determination in mixtures. In the absence of standard jams of this type, the author has not been able to pursue this possibility.

The filtrate from the above described precipitation was neutralised, sodium hydroxide added until the alkalinity was 0·02 *N*, and the former process repeated. An addition of calcium chloride gave no further precipitate, indicating absence of pectin. The solution, after being neutralised, was evaporated to very small bulk and cooled at intervals during the concentration to test gelatinisation. None could be obtained.

THE HYDROLYSIS OF AGAR-AGAR BY ACIDS.—Many jams known to be free from agar-agar were mixed with a known quantity of a solution of agar-agar in

water. The process described above was repeated with these adulterated jams. The filtrate from the calcium pectate, on evaporation, gave typical gels in some, but not in all cases. It became necessary, therefore, to study the effect of acetic and other organic acids on agar-agar. This was done by boiling 0·5 grm. at various dilutions under a reflux condenser for various times, cooling and diluting to 200 c.c. and titrating the reducing sugars formed with Fehling's solution, the percentage of reducing sugar formed being a measure of decomposition. An acetic acid solution of potassium ferrocyanide was used as an external indicator. The results are embodied in the following table:—

	Time of hydrolysis.	Concentration and acid used.	Reducing sugars formed by hydrolysis, expressed as hexose. Per Cent.
Agar 1.	30 mins.	3 per cent. HCl	49·6
2.	,,	,,	46·3
3.	,,	,,	47·0
,,	15 mins.	,,	44·4
,,	1 hour	,,	47·0
,,	2 hours	,,	47·0
,,	4 hours	5 per cent. citric	47·2
,,	2 ,,	,,	47·2
,,	1 hour	,,	43·5
,,	2 hours	1 per cent. citric	41·3
,,	4 ,,	1 per cent. malic	46·6
,,	4 ,,	1 per cent. tartaric	46·6
,,	2 ,,	5 per cent. acetic	23·2
,,	3 ,,	1 per cent. citric	44·4

It is to be inferred from these results that agar-agar is completely decomposed by tartaric, citric, and malic acids in four hours at a concentration of 1 per cent. acid, and that higher concentrations very rapidly decompose this substance. The hydrolysis of agar-agar by organic acids at a concentration likely to be found in jams and conserves generally, makes the addition unprofitable unless added immediately before the mass is cooled.

In view of these results, it became necessary to modify the procedure in order to obtain satisfactory results with small quantities of agar-agar. The filtrate from the calcium pectate was therefore immediately neutralised, before concentration which was continued only to 50 c.c., after which 400 c.c. of cold 95 per cent. alcohol were added, and the whole allowed to stand overnight in order to precipitate the agar-agar. It was then filtered through a fluted paper, washed with 50 per cent., and then with 25 per cent. of cold alcohol. The filter and contents were then boiled with water, filtered and concentrated. No difficulty was experienced in obtaining satisfactory gelatinisation even from 0·1 per cent. agar-agar.

It was hoped that the original residue precipitated by alcohol from a conserve would be sufficiently free from sugar to merit investigation as to the comparative hydrolysis of agar-agar and pectin by acids. After a short trial the attempt was

abandoned, as such a method would often be misleading, since the difficulty of testing for commercial sweetening agents, possibly occluded under such circumstances, was found to be very great. Not only did presumably pure pectin reduce Fehling's solution to an appreciable extent, but it was found that pectin was hydrolysed by organic acids of a strength previously found to decompose agar-agar.

THE HYDROLYSIS OF PECTIN BY ACIDS NORMALLY OCCURRING IN FRUITS.—Crude pectin was dissolved in water, hydrolysed in the cold by 0·02 *N* NaOH for one hour and acetic acid added until neutral to litmus. It was boiled, filtered and cooled, acetic acid added until 0·1 *N*, and 20 c.c. of 10 per cent. calcium chloride added. After standing one hour, the precipitate was boiled, filtered and weighed in the usual way as calcium pectate. A similar weight of the same crude pectin was next boiled under a reflux condenser with malic and citric acids and the residual pectin determined as above.

Original weight of pure pectin taken, expressed as calcium pectate, = 0·1155 grm.

Concentration and acid used.	Time of boiling. Hours.	Pectin not decomposed expressed as calcium pectate. Grm.	Pectin decomposed (by difference). Grm.	Per Cent.
Citric, 2 per cent.	3	0·0730	0·0425	37·0
Malic, 2 per cent.	,,	0·0745	0·0410	35·5
Malic, 1 per cent.	,,	0·0895	0·0260	22·5
Citric, 1 per cent.	,,	0·0865	0·0290	25·1

These results show that prolonged boiling of jam is most undesirable owing to the decomposition of pectin. It explains the great difficulty experienced by the housewife in obtaining crab-apple jelly of good setting quality at times, the pectin content being low, and the acidity often 2 per cent. and over.

In the presence of liquid glucose and gelatin it was found necessary to make a slight modification of the procedure, as the dextrin and gelatin precipitated by alcohol treatment congealed with the pectin to a sticky mass. In the case of gelatin, the precipitate produced by alcohol was dissolved in hot water, the solution cooled, the gelatin removed by precipitation by picric acid, and the excess of this by white of egg, which was then precipitated by boiling. Dextrin was removed by treatment with cold water, but the subsequent filtration proved troublesome.

DETERMINATION OF AGAR-AGAR.—It will be obvious from the foregoing that any methods for determining agar-agar in a conserve must be limited to evaluating that which remains undecomposed by the acids present, and this may not, under certain circumstances, be a correct criterion of the amount originally added.

A method for the determination of this undecomposed agar-agar was devised by taking advantage of the presence of the group $\begin{matrix} SO_2O \\ \\ SO_2O \end{matrix}\!\!>\!Ca$, which is completely

hydrolysed on prolonged boiling with hydrochloric acid into the ionised SO_4 radicle, which can be precipitated in the normal way by barium chloride.

If the amount of jam available is limited, the agar-agar precipitated by alcohol for the qualitative examination may be utilised, but it is preferable to take a fresh quantity and proceed as follows:—

One hundred grms. of jam, or more, if only slightly adulterated, are carefully freed from sugar as previously described. The residue on the Büchner funnel is boiled for some minutes in 200 c.c. of water, filtered, washed, re-boiled and re-filtered, pressure being used when required. It is essential to keep the fluid over 80° C. during filtration to ensure that all the agar-agar passes into the filtrate. This is concentrated to 300 c.c., and an aliquot portion (say 100 c.c.) taken for the determination of free sulphate by the usual method. Care should be taken by deferring the addition of hydrochloric acid until the last possible moment, not to decompose any agar-agar. The remaining 200 c.c. are concentrated to 100 c.c., and an equal bulk of concentrated hydrochloric acid is added. The mixture is boiled for 6 hours, more acid being added when necessary. Finally it is concentrated to 25 c.c., then diluted to 300–400 c.c., and filtered hot. Barium chloride is added and the whole allowed to cool and stand overnight, as the barium sulphate does not precipitate quickly. Washing, filtering and incinerating may then be carried out as usual, and the weight of agar-agar found from the formula:— $15\left[\frac{3}{2}(a-2b)\right]$, where a represents weight of barium sulphate from the total sulphate from the 200 c.c., and b the weight of barium sulphate from the free sulphate in the 100 c.c.

Fifteen is chosen as the factor which represents slightly less than the maximum of barium sulphate obtained by hydrolysis and precipitation of sulphate from agar-agar. The difficulty of effecting rapid precipitation from the hydrolysed agar-agar and pectin was found to be due to the degradation products of the pectin. On boiling pectin for some hours with concentrated hydrochloric acid, diluting and filtering, the resulting liquid was found to delay, for many minutes, the precipitation of barium sulphate, even from concentrations far greater than those encountered in agar-agar determinations.

Typical results obtained were as follows:—

1. 0·5 grm. of agar-agar in 100 grms. of gooseberry jam gave 0·0092 grm. of $BaSO_4$ for (b) and 0·0448 grm. for (a), hence agar-agar = 0·59 grm.
2. 0·5 grm. of agar-agar in 100 grms. of strawberry jam gave 0·0078 grm. of $BaSO_4$ for (b) and 0·0380 grm. for (a), hence, agar-agar = 0·504 grm.
3. 0·5 grm. agar-agar in 50 grms. of gooseberry jam, on hydrolysis of both (a) and (b), gave 0·0520 grm. of $BaSO_4$.* Thus the sulphate given by hydrolysis of the agar-agar = 0·0342 grm., which is equivalent to 0·51 grm. of agar-agar.

* Fifty grms. of jam treated in the same way before hydrolysis gave 0·0178 grm. of $BaSO_4$.

Analytical figures are given by F. Hartel and J. Solling (*Zeitsch. Untersuch. Nahr. Genussm.*, 1911, **21**, 168–196) for most common jams for insoluble matter, soluble solids, acidity, sucrose, sugar-free extractives, mineral matter and alkalinity of the mineral matters.

MICROSCOPICAL EXAMINATION OF CONSERVES.—The microscopical examination of a conserve for the identification of fruit and vegetables is of extreme importance, and it very often furnishes the only evidence of adulteration.

Many books of reference, such as those of Winton, König, Schneider, Moeller, and Schimper, are available, which illustrate and describe the microscopical characteristics of a fair number of fruits and vegetables, but these are often misleading, and comparison with known specimens actually isolated from jams is far preferable.

The conserve should be thoroughly mixed in a tall beaker, with about 50 times its volume of hot water, and stirred from time to time until the gelatinous matter is in solution. The fruit débris should then be allowed to settle, and the clear fluid drawn away through a glass tube by applying gentle suction from a water-pump. The washing and decanting should be repeated, if necessary, and the whole of the débris thrown into a large flat-bottomed porcelain dish. It will then be possible to examine with a hand lens various distinctive objects. These will usually be clear enough for microscopical examination as a water preparation, though, occasionally, epidermis will need further treatment by chloral hydrate in order to obtain the required transparency.

Permanent mounts of specimens required for future reference are best made in glycerin jelly. Specimens isolated by the above treatment will usually require further treatment by washing in hot water. They should then be transferred to a 50 per cent. solution of glycerin in water, allowed to drain, and mounted in jelly in the usual way. Ringing by gold size on Japan black is essential for permanence.

EPIDERMIS.—The arrangement, size and shape of cells should be noted, together with presence or absence of stomata, hairs, etc. It should be remembered that the epidermis from fresh fruit is often very different from that in the finished conserve. One should be cautious in assuming epidermis to be glabrous, as hairs easily become detached by boiling. Very often a clue is given by a circular or rosette-like arrangement of cells around the base of a hair which may be missing.

The best method is that of comparison with epidermis obtained from known jams. As there are not many fruits normally used in jam making whose epidermis will withstand the process of long-continued boiling, a suitable collection of permanent slides is readily made.

CELLULAR STRUCTURE.—A little of the conserve itself may be examined directly or after suitable dilution with water or glycerin. The presence of cells, vessels, seeds, hairs, styles, etc., should be noted. The large parenchymatous cells of the apple should not be confused with those of the turnip, vegetable marrow or banana. These distinctive cells of the apple are sometimes, but by no means invariably, stained violet with iodine solution. Stone cells arranged in large

groups may indicate pear or quince. Curious bundles of narrow stone cells in bundles of 6 to 12 occur in the currant.

Vessels may indicate apricot, peach, mangel-wurzel, carrot, ginger, cherry, or certain vegetables. Reticulated vessels do not necessarily indicate roots, as they are present in cherry, apricot, and peach. Size is a good guide, but comparison is practically essential.

SEEDS.—The seeds should be isolated, and, by the aid of a lens, separated into obviously different kinds and dried. These are best examined by an inch objective with a binocular microscope when available, and with dark ground illumination and a top light. The differences are very striking, and varieties can be easily recognised by reference to standards. These can be prepared by isolating seeds from known jams, washing, drying, and mounting loosely in a circular black-backed recess cut in a hard wood slide, and protected by a coverslip. It is often stated that the seed content of jams prepared from aggregate fruits is sufficiently constant for a count to be of great value in quantitative work. In recent years much has been done by hybridisation to increase the weight of the fleshy portion of the whole fruit compared with that of the actual seeds, so that in modern practice a seed count is of very doubtful value.

HAIRS.—The water-washed residue previously examined for epidermis should be separated by centrifuging, and studied for detached hairs. In most fruits these are fairly distinctive in size and shape, but the general characteristics are sometimes so altered in the process of jam making as to make them unreliable as a means of identification. Prolonged search is often necessary to obtain a complete specimen, and it is advisable to secure this, as the terminal point often provides valuable information. Care should be taken not to mistake a broken style for a hair.

STYLES.—Those of the strawberry, raspberry and blackberry are distinctive, and are easily picked out with forceps, being 2 to 4 mm. in length.

The original quality of the fruit may be questionable; in this case, search should be made for fungus growths, including yeast cells, spores, etc. Many of these show up well with Gram's stain, being Gram positive.

THE IDENTIFICATION OF AGAR-AGAR BY THE PRESENCE OF DIATOMS.—Agar-agar as normally prepared, always contains diatoms, sponge spicules, and other forms of marine origin. The most easily recognised parts of these objects, being composed of silica, will persist in any conserve in which agar-agar is used. They are usually somewhat distorted and fragmentary, and their isolation and identification is attended by some little difficulty. As little as 0·25 per cent. of agar-agar may produce a marked improvement in the consistence of a conserve, so that a large bulk, say 100 grms., should be taken for the test.

The direct ignition method of Macara, and the sulphuric acid method have proved troublesome with large bulks, and the following is to be preferred when a slight amount of adulteration makes it necessary for every diatom to be carefully preserved.

One hundred grms. are warmed in a large beaker with concentrated nitric acid on a water bath, the acid being added in small portions at intervals, in order to avoid excessive frothing. When the sugary matter is decomposed, the acid is evaporated to small bulk, and the whole of the contents of the beaker washed into a platinum dish. The mass is evaporated to dryness, gently ignited until a perfectly white ash is left, and this is boiled for a few minutes in dilute nitric acid. The contents of the dish are then washed into a boiling tube, and the sediment washed by decantation, great care being taken not to lose any diatoms. This is best done by drawing the clear fluid away by means of a pipette attached to a suction pump, and applying gentle and steady suction. A few crystals of potassium chlorate are added to the sediment, and about 10 c.c. of concentrated sulphuric acid. This is heated for a few minutes in order to decompose any remaining organic matter, the presence of which might hide small but important diatoms.

The final washing of the diatoms may be done in a small test tube, the base of which has been drawn out to about 1 mm. The diatoms should then be concentrated in a few drops of water only, and removed by means of a small dropping pipette to a microscope slide. Any remaining diatoms may be removed by the addition of a few drops more water, which is agitated briskly before being drawn into the pipette. The liquid on the slide should be evaporated to dryness, and diatoms permanently mounted *in situ* in styrax, as prolonged examination and reference to standards are often necessary. Such standards may be purchased from professional diatomists, or may be prepared by igniting shredded agar-agar and washing as above, removing individual diatoms from the dry slide by means of a hair to a gummed coverslip and finally mounting in styrax. The natural silica content of certain conserves may be high enough to make the mounting *in situ* inadvisable, in which case selected diatoms may be transferred to a gummed coverslip by means of a hair, as mentioned above.

Many of the diatoms and spicules described as diagnostic by text-books and various papers are, on examination, found to exist in non-agar-agar producing localities. Some, indeed, are quite plentiful round the British coast. Sanft (12) and Marpmann (13) have examined agar-agar from the standpoint of the diatoms which are characteristic, but unfortunately neither has considered the possibility of confusion with diatoms from commercial diatomites. Differences are to be expected, as the diatoms from agar-agar are essentially of marine formation, whereas commercial diatomites are prepared from diatomaceous deposits from old fresh water lakes or inland seas.

The author was fortunate in obtaining the help of Mr. F. W. Payne, B.A., who has been kind enough to review the whole subject, with the object of laying down characteristics which could be considered specifically diagnostic. He has prepared a list, given below, of the diatoms found in many specimens of agar-agar. The presence of one or two diatoms included in the list is not necessarily diagnostic, but, nevertheless, if taken in conjunction with the table of localities, quite a small number may afford strong presumptive evidence. Most of these listed diatoms are illustrated in the photomicrograph, or reference may be had to Schmidt's *Atlas of the Diatomaceae.*

As mentioned in the chemical section, an attempt to improve a conserve by the addition of agar-agar may fail if the acidity is high, and the agar-agar is added at too early a stage in the boiling. In such circumstances chemical tests might fail, whereas a microscopic examination should always succeed, unless the agar-agar has been ground so finely before use that it is impossible to recognise individual diatoms.

Should a manufacturer claim that diatoms found in his conserve are derived from his filters, it is suggested that he be called upon to produce a sample. Comparison of the diatoms would then easily detect similarity or otherwise, provided that sufficient diatoms from the conserve are available.

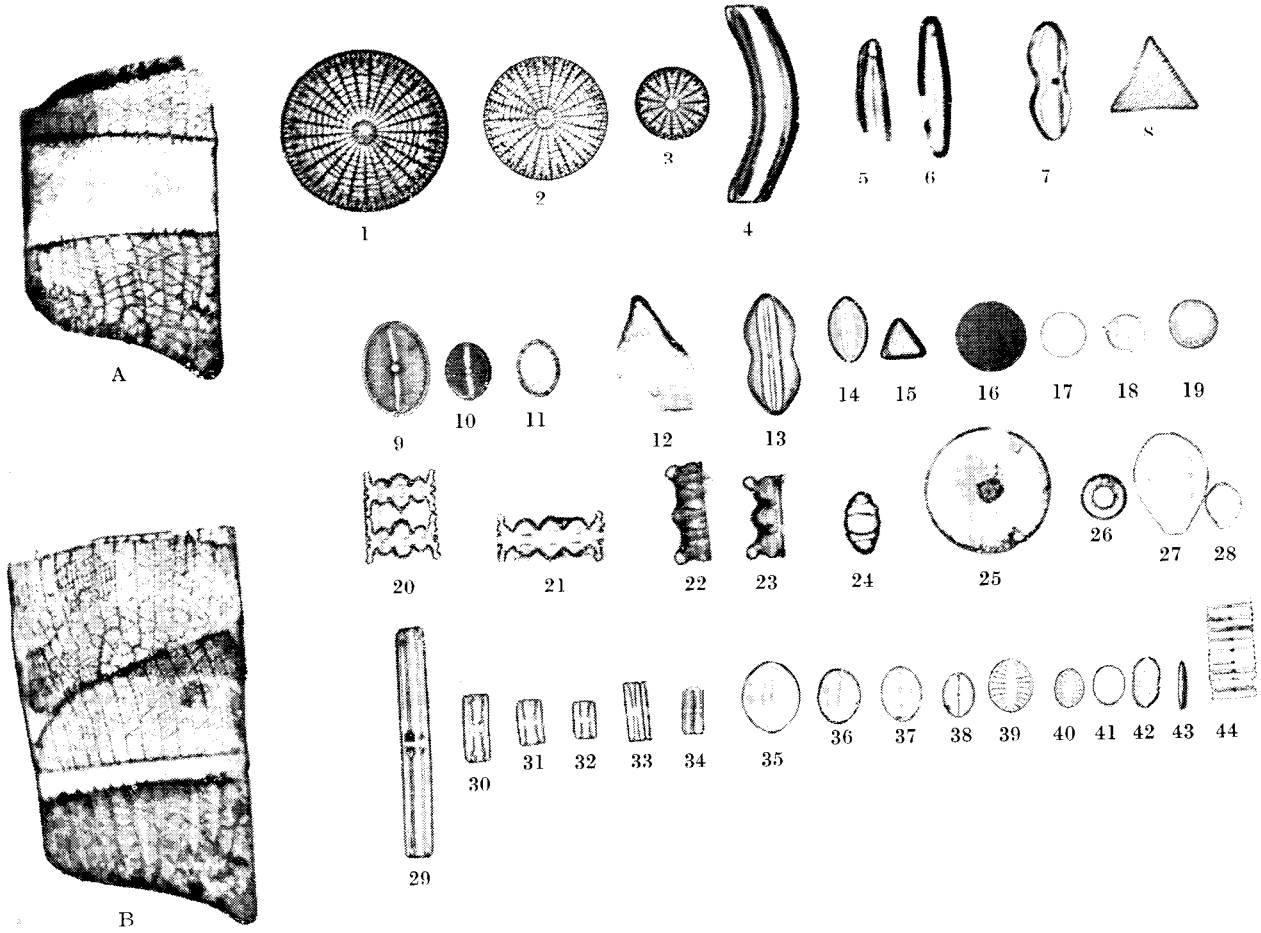
DIATOMS IN AGAR-AGAR.

Name of Diatom.	*Other localities in which found.*
ACTINOCYCLUS RALFS, VOR. GRUN	Manila, Cuba, Society Is., Smyrna.
***ARACHNOIDISCUS ORNATUS, E.	Africa, West Coast of America.
**BIDDULPHIA PULCHELLA, GRAY.	Campeachy Bay, Brazil, Mediterranean, Australia.
*BIDDULPHIA, TUOMEYI, BAIL.	Guano, Brazil, Madagascar, West Coast of Africa.
***CAMPYLONEIS GREVILLEI (W Sm) GRUN.	California, Australia, Arabia, etc.
CLIMACOSPHENIA MONILIGERA, E.	Cuba, W. Africa, California.
COCCONEIS CORONATA, E.	W. Indies, Smyrna, Corsica.
,, FIMBRIATA. E.	Corsica, W. Coast of America.
*** ,, HETEROIDEA, HANTZSCH	Australia, New Zealand, Colon, E. Indies.
* ,, SCUTELLUM, E.	Ubiquitous.
*** ,, SPLENDIDA (Greg) GRUN.	Cuba, Scotland, California, etc.
*COSCINODISCUS NITIDUS, Greg.	Scotland, Colon, Alexandria, etc.
,, 2 undetermined SP	
**GRAMMATOPHORA MACILLENTA, E.	Britain, The Levant, etc.
*GLYPHODESMIS WILLIAMSONI (Greg) GRUN	Scotland, California.
**GRAMMATOPHORA MARINA, E.	Ubiquitous.
** ,, SERPENTINA, E.	Ubiquitous.
*GEPHYRIA MEDIA, ARN	Sandwich Is., California.
*HYALODISCUS STELLIGER, BAIL.	Ubiquitous.
,, SP	
**ISTHMIA NERVOSA, K.	North of Europe and America.
NAVICULA CRABRO, E.	Common.
,, LACRIMANS, A.S. (GEMMATULA, VAR. CL.)	Gulf of Mexico.
,, LYRA, VAR. E	Common.
**PODOCYSTIS SPATHULATA (SHADB.) V. vH°	Society Is., Mauritius, Central American Coasts, etc.
SURIRELLA DIVES, CSTR	Carpentaria Bay.
TRICERATIUM FAVUS, E.	Ubiquitous.
,, PARALLELUM, GREV. VAR.	?

*** denotes occurring very frequently, ** denotes frequently, * denotes sparingly, and the unstarred diatoms only occurred once in agar-agar. The actual numbers found by F. W. Payne. in examining a large number of samples were approximately *** 100 to 150, ** 10 to 20, * 2 to 6 (in 5 grms. of agar-agar).

The author wishes to express his thanks to Mr. F. W. Payne, B.A., whose great experience regarding diatoms, and whose helpful suggestions have been most valuable.

In conclusion, he desires to thank Mr. F. J. Osborn for the many excellent photomicrographs of diatoms which he has been good enough to prepare from time to time, and the Government Chemist for permission to publish this paper.



Easily recognisable are the huge *Isthmiæ* (A and B); the *Arachnoidiscus* (1–3); *Gephyria* (4); *Triceratium favus* (8); *Cocconeis splendida* (9); *Navicula gemmatula* (13), and *Lyra* (14); *Biddulphia Tuomeyi* (20, 21), and *Pulchella* (22–24); *Podocystis* (27, 28); the *Grammatophoræ* (29–34); and *Campyloneis* (39, 40).

REFERENCES.

(1) *Ind. Eng. Chem.*, 1922, p. 710.
(2) *Biochem. J.*, 1924, 283.
(3) *Ind. Eng. Chem.*, 1923, p. 780.
(4) *Ind. Eng. Chem.*, 1923, 15, News Edition, March 10, p. 3.
(5) ANALYST, 1921, 46, 239.
(6) *Biochem. J.*, 1922, 704.
(7) *Biochem. J.*, 1923, 510.
(8) *Biochem. J.*, 1921, 469.
(9) *J. Chem. Soc.*, 1923, 1414.
(10) *Biochem. J.*, 1923, 83.
(11) *Biochem. J.*, 1922, 60.
(12) *Zeitsch. allgem. Osterr. Apoth.-Vereine, Wien*, 1902, No. 9, pp. 229–232.
(13) *Zeitsch. angew. Mikroskopie*, Bd. 2, 1887.

GOVERNMENT LABORATORY,
CUSTOMS HOUSE, E.C.3.

DISCUSSION.

Mr. L. K. BOSELEY said that acids could increase the gelatinising effect of pectin, but they also caused the inversion of sugar; so that it was not possible to increase the percentage of acid without running the risk of inversion. Thus the set of the pectin could not be considered alone. Agar-agar was not used in large quantities on account of its price; the cheaper apple-pectin from the manufacture of cider was probably used in preference. Moreover, agar-agar was affected by boiling; 0.7 per cent. of acid at a temperature of 223 F. was sufficient to destroy it.

Mr. T. MACARA disputed the statement that it was possible, as stated by Tutin, to dissolve out the pectin in the ordinary way. He agreed with Schryver, Sucharipa and others that a considerable proportion of the pectin existed, in some fruits at least, in an insoluble form. He considered that none of the formulae so far proposed for pectin should be accepted until it had been shown that a single substance had been isolated. The pectins so far described appeared to be mixtures; consequently analytical methods based on them should only be accepted temporarily. The alcohol precipitation method, as advocated by American chemists, gave an acid hydrolysis product of the mixed substances, whilst the Carré and Haynes method gave a lime salt of the acids liberated by saponification of the esters. Neither method gave the actual pectin present in the solution. With regard to agar, he had found Parke's method decidedly useful.

Mr. J. KING, in his reply, states that the value of high acidity as an accessory factor to gelatinisation lies in its application to the jelly rather than to the jam industry. The acidity of a jam can, however, be increased without very serious sugar inversion, by deferring to the last possible moment the addition of a fruit juice of high acidity to the previously boiled body of a jam. Data on pectin-sugar-acid gels are given in the *J. Assoc. Off. Agric. Chemists*, 1923, **7**, 57. The home market for agar-agar is based mainly on bacteriological requirements. Its use in the Malay States for jelly-making indicates possibilities elsewhere.

In reply to Mr. Macara, it should be mentioned that the present uncertainty regarding the chemistry of the substances grouped under the name "pectin," makes it advisable to remove these entirely before testing for other gelatinising agents by a method, such as that of Carré and Haynes, which is not open to criticism as regards completeness of precipitation. The accuracy of the existing methods for determination of pectin is doubtful (*cf. J. Assoc. Off. Agric. Chemists* 1923, **7**, 107; 1924, 281; and *J. Soc. Chem. Ind.*, 1925, **44**, 257T.; ANALYST, 1923, **48**, 223).

The Detection of Plant Phenols by the Use of Nitrites or Nitric Acid.

By ALAN H. WARE.

With the exception of Griessmayer's test for ellagic acid, the method used by Procter for detecting ellagitannins, and Histed's employment of nitric acid to distinguish between different kinds of aloins, very little use appears to have been made of sodium nitrite, nitrous acid or nitric acid for the detection of plant phenols in vegetable substances. The present paper describes a series of new or improved tests for certain plant phenols in which one or other of the reagents named are used.

(*a*) The extractive is heated with sodium nitrite without any added acid. Distinctive results are yielded by phloridzin and ellagitannins.

(*b*) The method is similar to the last, but a few drops of very dilute acid are also used, and heat is applied only if necessary. Very distinctive results are given by iso-barbaloin (see Curacao aloes, etc.) and ellagitannins.

(*c*) Liebermann's test (concentrated sulphuric acid in a dry tube with a minute crystal of the nitrite are heated with phenolic body) is applied to the pure or crude phenol. Distinctive results are given by the catechins and chrysarobin (purified Goa powder).

(*d*) The method used is similar to the last, but a wet tube is used and the nitrite is added in excess of the phenolic body. See catechins and "tobacco-tannin."

(*e*) The method is that of Griessmayer's test for ellagic acid. The test was applied to a large number of plant-phenols, but a positive result is apparently only given by ellagic acid. For a result which appears at first to be positive, but is due to the action of the nitric acid alone, see the next paragraph.

(*f*) The solid substance is dissolved in strong nitric acid and heated to about 100° C. It is then diluted with water as in Griessmayer's test. A very distinctive result is obtained with phloridzin.

No distinctive results of any special value were obtained in these tests with any of the following:—Gallic acid, gallotannin, quercetrin, brazilin, hæmatoxylin, salicylic acid or partly purified phlobatannins; and, although a very large number of vegetable extractives were tested, no other results of much value, except those recorded, were obtained on testing aqueous extractives by any one of the methods described. It would be tedious to tabulate all the individual negative results given by the last class indicated, but they included those obtained with samples containing anthraquinone, benzophenone, xanthone, flavone and flavonol derivatives, and anthocyanins.

A description of the more useful results obtained is described under the following heads and in the order given, *viz.*:—(1) Curacao aloes and commercial aloin; (2) phloridzin; (3) ellagitannins in extractives; (4) the catechins, (5) commercial chrysarobin, and (6) "Tobacco-Tannin."

(1) Curacao (so-called Barbadoes) Aloes and Commercial Aloin.—A very distinctive and specific reaction, due to iso-barbaloin, can be obtained by dissolving a little Curacao aloes, or of the commercial aloin made from this, in sufficient water to give to the solution only a straw-colour, and then adding to this solution a few crystals of sodium nitrite and one drop of acetic acid (33 per cent.). If this mixture be shaken in the cold for about one minute, a rich pink colour, becoming purplish-red, is produced. If dilute alkali (1 per cent. aqueous potassium hydroxide solution) be now rapidly added in sufficient quantity, the colour changes to a very pronounced green, which remains permanent for a considerable time.

Discussion.—The reaction must be due to iso-barbaloin, because it is not given by Socotrine or Zanzibar aloes, each of which contains both barbaloin and beta-barbaloin, but no iso-barbaloin. Cape aloes and Natal aloes give a poor pink coloration, but do not yield a definite green on the addition of alkali. Cape aloes is known to contain a little iso-barbaloin. It is therefore probable that Natal aloes also contains a trace. The reaction is not given in neutral solution, either in the cold or on warming (see phloridzin and ellagitannins). It is more convenient and more delicate than the cupraloin test, which also often fails unless careful adjustment of the quantities of the reacting substances is made. A few ellagitannin bodies (containing also gallic-acid tannin) give somewhat similar results, but can readily be distinguished by the transiency of the colours given, for, with these bodies, both the colour given on the addition of acetic acid and that given on the subsequent addition of *excess* of alkali rapidly become brown; whereas the colours with the aloes and aloin are much more permanent.

(2) Phloridzin.—Phloridzin is a glucoside of phloretin (a phloroglucinol body). It is found in the root-bark of many of the *Rosaceæ*, particularly that of apple, pear and plum trees. It is employed in physiological research to produce glycosuria, and is sometimes given in medicine as a substitute for quinine. It is said to yield a fine purple colour with ammonia on exposure to the air, but the author finds the following method a much more ready way of identifying it:—

Test.—A small quantity of phloridzin and a few crystals of sodium nitrite are shaken with a few c.c. of water in a test-tube, and the mixture is then heated and finally boiled for at least a minute. A clear pink colour first appears, which becomes almost wine-red, and this remains unaltered for a long time. On adding alkali the colour changes to yellow.

Another Test.—A little phloridzin is dissolved in strong nitric acid, and the solution is then heated to about 100° C., and diluted with water. A pink or wine-red colour is thereby given. Under certain conditions a purplish precipitate may fall. In either case, if the mixture be shaken with ether, the colouring matter is abstracted and the ether becomes intensely red. The colouring matter

produced by this method is not the same as that given in the first test; for that cannot be shaken out from an aqueous solution by means of ether, and it is also destroyed by dilute nitric acid.

Discussion.—The fact that in the first test the colour can be obtained without the addition of acid, and changes to yellow and not to green on the addition of alkali, sharply differentiates phloridzin from iso-barbaloin. Ellagitannins under the same conditions give a green colour reaction changed to brown by the addition of alkali. It is probable that the root-barks containing phloridzin would, in extractive, give the first reaction described, but the author has not yet had the opportunity of testing them. Both tests appear to be unusually specific in relation to other plant substances.

(3) ELLAGITANNINS IN AQUEOUS EXTRACTIVES.—The use of nitrous acid to detect ellagitannins appears to be due to Procter (*Leather Industries Laboratory Book*). It was probably suggested by Griessmayer's reaction for ellagic acid (finally established by A. G. Perkin and Nierenstein (*Trans. Chem., Soc.*, 1905, **87**, 1412). Very useful results are obtained by Procter's method under suitable conditions, but the method is by no means fool-proof, sometimes yields uncertain results, and does not always distinguish between bodies which contain but little ellagitannin and those which contain much. The present author finds that the following modifications of Procter's method give more satisfactory results:

(*a*) *Test with Nitrite without added Acid.*—The extractive is heated in a test-tube with a few crystals of sodium nitrite, the heat being applied gradually by holding the tube a little distance above a small flame. In no case should the liquid be boiled. If the original extractive is strongly coloured, it should be diluted with water to leave a distinct but not a marked colour. Under these conditions a very definite green colour reaction is usually readily obtained.

(*b*) *Test in the presence of added Acid.*—The method is similar to that described under (*a*) except that about one grm. of powdered *acid* phosphate of sodium and one drop of 33 per cent. acetic acid or of 10 per cent. hydrochloric acid are added before heating the mixture. The acid phosphate (NaH_2PO_4) acts as an efficient buffer, and its use does away with the necessity for very careful adjustment of relative strength of acid, sodium nitrite or extractive used. It will be sufficient if the following readily applied precautions are adopted:—The extractive, if necessary, must be diluted so that its colour is not too pronounced, the quantity of sodium nitrite added in this method should not be more, as a rule, than twice the bulk of a pin's head, and the acid used should not be far removed from the strength indicated. Usually a beautiful blue or violet colour reaction is given, but if the mixture is heated to too high a temperature a green coloration may be produced. After the blue is obtained the colour is often intensified by cooling the tube under the tap. A better blue or violet is sometimes obtained either by adding another drop of acetic acid, after cooling, and re-heating gradually as before; or by repeating the test with the acid phosphate, but without the acetic or hydrochloric acids. If an intense red colour is given either before heating or

in the very early stages of heating (brown should be given at first), too much sodium nitrite has been used. This can sometimes be remedied by thorough dilution; if not, the experiment must be repeated with less nitrite. The appearance of such a red colour, however, is an indication of ellagitannin, and the author often finds it convenient to make a preliminary test for ellagitannins by dissolving a few crystals of sodium nitrite in a little of the extractive on a slab, and then placing in the solution some broken crystals of acid phosphate of sodium. If ellagitannin be present, an evanescent pink to purple colour appears. In some cases the colour is given by the nitrite alone.

Discussion and Results.—There is not the slightest difficulty in getting a beautiful blue or violet colour reaction with a typical ellagitannin extractive, even by the merest tyro at such work. The tests also afford a very convenient method of distinguishing between two different classes of ellagitannin compounds, *viz.*:—(*a*) Typical ellagitannin compounds which yield a really good blue or violet colour reaction in the second method; and (*b*) compounds in which the ellagitannin is either very little in total amount, or is relatively little, in relation to interfering substances, and thus do not give a really good blue or violet colour-reaction. This last class of substance gives, however, an unmistakable green colour-reaction in one or both of the two methods which have been described; and they often yield a purplish colour in the method in which acetic acid is used. The following tannin-containing compounds fall into class (*a*), *viz.*:—Divi-divi, valonia, *Eucalyptus globulus* leaves, the kinos from *Eucalyptus microcorys* and *E. maculata*, Knoppern galls, pomegranate root-bark and fruit-rind, and myrobalans. In class (*b*) are Basra and Aleppo galls, algarobilla and pimento fruits. Negative or negligible reactions, with respect to the particular colour reactions under discussion, are given by gallotannin, Chinese galls, sumach, red rose petals, the bark and leaves of *Hamamelis virginica*, bearberry (*Arctostaphyllos uva-ursi*) leaves, cloves and logwood; all of which substances are especially characterised by the presence of typical gallic acid derivatives. It should be noted, however, that many of the typical ellagitannin-bodies contain also gallic-acid tannins, sometimes in considerable quantity. The presence of gallic-acid tannins does not materially interfere with the specific reaction given by ellagitannins, which appear to interact with the nitrite preferentially. This is not the case, however, with gallic acid; for should this be present in relatively large quantity, as in Aleppo and Basra galls, it will prevent the obtaining of a good blue or violet colour-reaction with the ellagitannin also present. Some similar phenol (which appears, however, to be iron-browning) present in Algarobilla renders it difficult or impossible to get the typical blue or violet colour reaction with that body, notwithstanding the fact that the tannin present is supposed to be mainly of the ellagitannin class. Pimento berries appear to contain both a little phlobatannin and gallic-acid tannin, in addition to the ellagitannin.

(4) The Catechins.—The presence of the catechins, in such vegetable substances as contain them, is of course very easy to detect, owing to the readiness with which they may be shaken out from aqueous admixtures by means of ether,

and the phloroglucin reaction obtained by means of the deal shaving and hydrochloric acid test (applied to the ethereal solution). It does not, however, appear to have been recorded that the Liebermann reaction is also readily given by catechins, even in the crude condition as obtained by evaporating an ethereal solution made as above described. In this way the presence of catechin in Acacia cutch, gambier or guarana can readily be confirmed. The colour given is an intense violet, as in the case of phloroglucinol itself.

(5) TOBACCO-TANNIN.—The author has recently had the opportunity of examining some samples of the partially purified "tobacco-tannin," so-called, obtained by Bell's process. This was kindly sent him by Mr. Ernest Paul from the Chemical Laboratory of the Imperial Tobacco Co. Tobacco-tannin appears to be one of the many pseudo-tannins which in some respects resemble the catechins, but do not give the phloroglucinol reactions referred to above. If, however, the Liebermann method be varied, as described under (*d*) in the earlier part of this paper, a carmine-purple colour-reaction is given. This appears to be a purely oxidising reaction, as an apparently exactly similar reaction is given by dissolving the tannin in sulphuric acid and adding a drop or two of test-solution of ferric chloride. Both phloroglucinol and the catechins resemble tobacco-tannin in giving the carmine-red colour reaction with sulphuric acid and sodium nitrite (excess) in a wet tube; and catechins also give a similar reaction, if the ferric chloride be substituted for the nitrite.

It should be noted, however, that all of these reactions given by the catechins in the presence of sulphuric acid may be partly due to the action of the sulphuric acid alone. Procter states in his *Laboratory Manual* that gambier-catechin dissolves in sulphuric acid to give an intense purple colour. This is somewhat misleading, for this catechin dissolves in cold concentrated acid, if water be excluded, to give a yellow colour; and it is not until a relatively high temperature is reached that the non-distinctive red, first given on heating, changes to the characteristic rich crimson-purple. This latter reaction is not given by phloroglucinol, neither is it given by "tobacco-tannin," so that there is no danger of ascribing results to wrong causes in the case of these two compounds. Since phloridzin shows a transient tendency to give a similar reaction under the conditions just described, it is possible that other phloroglucinol compounds may be found to give the same result; but the author was unable to obtain the reaction with quercetrin, which yields only a non-distinctive red or brown, of the kind so frequently given to this kind of test.

(6) COMMERCIAL CHRYSAROBIN (PURIFIED GOA POWDER).—This gives a purplish colour-reaction in Liebermann's test. On adding a little water and then diluting with alcohol (90 per cent.), the colour changes to green, which becomes a characteristic wine-red on adding ammonia. The series of colour changes given make this test one of the most specific for chrysarobin, but it should be noted that the *first* colour given may not always be very marked, and consequently is liable to be overlooked.

In conclusion, the writer desires to thank Dr. Nierenstein, who very kindly sent him authentic specimens of several catechins, and from whom most of the tanning materials were also obtained.

UNIVERSITY COLLEGE OF THE S. WEST,
EXETER.

A New Colorimetric Method for the Determination of Cobalt in the Presence of Nickel.*

BY B. S. EVANS, M.C., M.B.E., Ph.D., B.Sc., F.I.C.

(*Read at the Meeting, May* 6, 1925.)

THE ordinary processes for the determination of cobalt involve separations, usually more or less complicated, from other elements and especially nickel, which take up much time and sometimes necessitate the use of expensive chemicals.

Of these, the one apparently in most general use is that based upon the precipitation of cobalt by α-nitroso-β-naphthol. This is an excellent process giving good results, but it has serious drawbacks; the reagent is costly, the time required is considerable, and, if there is doubt as to whether cobalt is present at all, the bulky precipitate of the reagent itself entirely obscures any small trace of cobalt that may be present, and requires further treatment to make it available as a qualitative indication.

The phosphate method requires rigorous separation of the cobalt from any of the numerous metals which are precipitated as phosphate.

The volumetric cyanide method requires separation of the cobalt from, at any rate, nickel.

In the circumstances, therefore, it would seem that there is room for a method which is cheap and rapid, which gives a definite qualitative reaction for cobalt, which can be carried out in the presence of nickel, and which gives as accurate results as can be reasonably expected from a colorimetric method.

The method is based on the quantitative production of highly coloured cobaltammine when cobalt in ammoniacal solution is treated with an oxidising agent.

If a solution of cobalt is treated with ammonium chloride and excess of ammonia, the colour changes from the original faint pink to a brownish yellow; if a small quantity of sodium peroxide is now added the yellow colour slowly

* Communication from the Research Department, Woolwich.

changes to an intense rose pink, the change being usually complete in a minute or two. This pink colour is proportional to the amount of cobalt present, and may be used as the basis of a colorimetric determination.

In the case of a sample containing no interfering element a convenient amount is brought into solution, if necessary with the aid of acid, and the solution placed in a Nessler glass; 2 c.c. of 20 per cent. ammonium chloride solution are added, and then ammonia until a slight excess (say 2 to 5 c.c. of 1:1 solution) is present. The second Nessler tube contains 2 c.c. of ammonium chloride solution and 2 to 5 c.c. of ammonia made up to approximately the same level with water. About 0·6 grm. of sodium peroxide is added to each tube, standard cobalt solution is run into the standard tube, a small quantity at a time, and the tubes are allowed to stand for about two minutes before the colours are compared. A convenient standard to use is one containing 6·69 grms. of cobalt ammonium sulphate per litre, 1·0 c.c. corresponding to 0·0010 grm. of cobalt. As little as 0·01 grm. of cobalt gives as intense a colour as may be readily matched.

The following results show the degree of accuracy that may be obtained:— Varying proportions of cobalt were added to solutions of iron in hydrochloric acid, the liquid was nearly neutralised with ammonia, the iron thrown out with zinc oxide made into a cream with water;* and the whole made up to a known volume, filtered and the cobalt determined as described above in an aliquot part of the filtrate.

Weight of Iron. Grms.	Cobalt added. Per Cent. (on iron).	Cobalt found. Per Cent. (on iron).
1·0	4·0	3·90 4·00
1·0	3·0	3·00
1·0	2·0	2·05 2·00
1·0	1·0	0·90 1·00
5·0	0·5	0·49
5·0	0·1	0·11

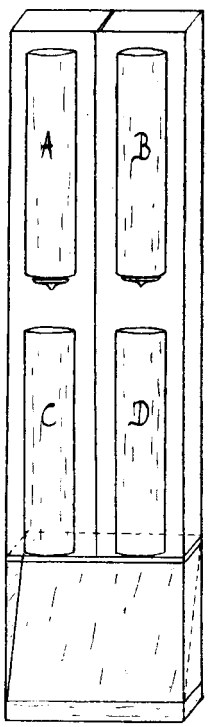
Where several estimations of cobalt have to be made, it is considerably quicker to prepare the solution of cobaltammine beforehand; this may readily be done by placing 20 c.c. each of 20 per cent. ammonium chloride solution and 1:1 ammonia in a 200 c.c. measuring flask, adding about 0·6 grm. of sodium peroxide, running in 50·0 c.c. of standard cobalt solution from a burette in quantities of about 1 c.c. at a time, and allowing the pink colour to develop after each addition before adding the next; more sodium peroxide should be added after every 3 or 4 c.c. Finally, the volume is made up to 200 c.c. and the liquid shaken and allowed to stand some hours before use. In making a determination with this

* It has been found necessary to have the iron in hydrochloric acid solution; with other acids loss of cobalt may occur.

solution, the test liquid is allowed to stand for a few minutes after addition of the sodium peroxide, to develop the colour, and the standard solution is then run into the other tube from a burette till a match is obtained, the volume being made up with water before the final matching. This solution of cobaltammine, made as described above, will keep without appreciable change for a month; the standard solution of cobalt ammonium sulphate is, of course, perfectly stable.

In presence of nickel a disturbing factor is introduced by the blue colour given by nickel with excess of ammonia, and still more by the fact that the shade of this colour may vary from a purple blue to a greenish blue with varying concentrations of ammonia. It was found possible to stabilise this blue colour by keeping the excess of ammonia low and adding sodium citrate. By these means a blue colour was obtained free from any violet shade, and its tint remained constant at any concentration. To prove this, a series of colorimetric determinations of nickel was made on solutions of varying strength, about 2·0 c.c. excess of ammonia being used and 5 grms. of sodium citrate added. The solution of nickel used contained 0·089 grm. of nickel per 100 c.c.

Nickel solution added. c.c.	Nickel. Grm.	C.c. taken to match colour.
50	0·0444	49·8
40	0·0355	39·8
30	0·0266	29·7
20	0·0778	19·6
10	0·0089	9·75
5	0·0044	4·70
2	0·0018	2·20



DETERMINATION OF COBALT IN PRESENCE OF NICKEL.—Varying amounts of cobalt and nickel were taken, and the cobalt determined in the mixture in the following way:—The solution was nearly neutralised with ammonia and divided into two equal parts which were placed in two Nessler glasses, A and B. Two c.c. of 20 per cent. ammonium chloride solution, 2 c.c. of ammonia (1:1), and about 5 grms. of sodium citrate were added to each and about 0·6 grm. of sodium peroxide to one of them, A. These two tubes, A and B, were placed in the two top compartments of a Walpole colorimeter. The bottom pair of tubes, C and D, each contained 5 c.c. of 20 per cent. ammonium chloride solution, 5 c.c. of ammonia (1:1) and about 5 grms. of sodium citrate, and about 0·6 grm. of sodium peroxide was added to D. The contents of th is tube, D, were then titrated with cobalt ammonium sulphate solution until the colours matched. When a match was obtained a second quantity of sodium citrate was added to the two top tubes, A and B, and of sodium peroxide to A and D; this was merely to ensure that the colour reactions were complete.

Taken.			Found.	
Nickel. Grm.	Cobalt Grm.	= Per cent. of Cobalt (on Nickel).	Cobalt Grm.	= Per cent. of (Cobalt on Nickel).
0·02	0·0010	5·0	0·00095	4·7
0·032	0·0016	5·0	0·0015	4·7
0·032	0·0032	10·0	0·00325	10·1
0·032	0·0008	2·5	0·0007	2·2
0·032	0·0004	1·25	0·00045	1·4
0·032	0·0064	20·0	0·0065	20·3

STEEL.—The process for steels is as follows:—Five grms. are dissolved in *aqua regia*, nearly neutralised with ammonia, precipitated with a cream of zinc oxide in water, made up to 500 c.c., mixed and filtered through dry paper into a dry beaker. Of this filtrate, 150 c.c. are placed in a dry beaker, 22·5 c.c. of ammonia (1:1), 7·5 c.c. of 20 per cent. ammonium chloride solution, and about 0·6 grm. of sodium peroxide added, and the solution allowed to stand for a few minutes till the manganese dioxide precipitate is sufficiently separated to allow of filtration. It is then filtered, and 120 c.c. (representing 1 grm. of sample) are placed in tube A of the colorimeter. The other three tubes contain 5 c.c. of 20 per cent. ammonium chloride, 5 c.c. of ammonia (1:1), and 20 c.c. of water; to tubes A and B, 5 grms. of sodium citrate are added, and to tube D about 0·6 grm. of sodium peroxide. The colour of the sample can now be exactly matched by additions of nickel solution to tube B, and standard cobalt solution to tube D, the volume required of the latter being a measure of the cobalt in 1 grm. of sample. If nickel is not present the determination can, of course, be carried out with two tubes only, as in ordinary colorimetric work.

The following results were obtained on steels with and without nickel:—

Weight of steel taken. Grms.	Cobalt added. Per Cent.	Nickel. Per Cent.	Manganese. Per Cent.	Cobalt found. Per Cent.
5·0	1·00	—	0·4	0·99
5·0	0·50	—	0·4	0·49
5·0	0·10	—	0·4	0·098
5·0	0·05	—	0·4	0·054
5·0	1·00	2·0	0·4	1·00
5·0	0·70	2·0	0·4	0·75
5·0	0·50	2·0	0·4	0·49
5·0	0·20	2·0	0·4	0·20
5·0	0·10	2·0	0·4	0·09

The colorimeter used for this work was home-made, as the Walpole colorimeters sold are not nearly large enough; it consists merely of a long flat box without ends and with one side hinged on, and a partition runs longitudinally down the middle. One end is closed by a piece of wood having two circular holes to admit light to the tubes; this forms a bottom to the box, and on it rest the lower two tubes; two small metal ledges are screwed to the wall half way up and support the upper tubes without interfering with the passage of light. Three of the sides (one long and two short), prolonged beyond the bottom, form a space into

which is fixed a white surface (porcelain, etc.) at an angle of 45° to the axis of the box, which serves to reflect light up through the two tubes.

As will be seen above, it is quite possible to make an accurate colorimetric determination of nickel with ammonia if sodium citrate is added. It has not, however, been found practicable to apply this method to the determination of nickel, as well as cobalt, in steels, because zinc oxide removes an appreciable proportion of the nickel; the same drawback has been found to apply to other reagents used for the removal of the iron.

The Occurrence of Glass Particles in Foodstuffs.

By ARNOLD R. TANKARD, F.I.C., and C. J. H. STOCK, B.Sc., F.I.C.

During recent months there have appeared in various journals statements concerning the presence of glass particles in jams and other foods. We have been engaged for some considerable time past in examining foods for fragments of glass, and the following tables give a summary of our main results:—

1. (A.R.T.)

Article.	Number examined.	Number free from glass.	Number containing glass fragments.	Character of glass particles.
Aerated waters and beverages	12	9	3	Minute fragments.
Bottled fruit	1	1	0	—
Confectionery (sweets)	6	6	0	—
Jams and marmalade	24	18	6	Splinters and minute particles.
Meat and fish pastes, etc.	12	11	1	Minute particles.
Pickles and sauces	12	4	8	Minute particles.
Coffee essence	6	3	3	Minute particles.
Lemonade crystals and powders	6	2	4	Large flakes and particles of appreciable size.
Totals	79	54	25	= 31·6 per cent.

The largest particles of glass were found in the lemonade crystals and powders ($\frac{1}{4}''$; 1/6″ in length); other samples contained particles 1/25″ long, and many of much smaller dimensions (average 1/4000th sq. inch).

2. (C.J.H.S.)

Article.	Number examined.	Number free from glass.	Number containing glass fragments.	Character of glass particles.
Aerated waters and beverages	31	10	21	Mainly minute fragments.
Bottled fruit	1	0	1	Mainly fragments.
Calves' foot jelly	1	1	0	—
Jam, fruit jelly, and marmalade	66	20	46	Some particles of appreciable size.
Lemon cheese and curd	8	5	3	Minute particles.
Mincemeat	3	0	3	Minute particles.
Orange cream	1	1	0	—
Fish and meat pastes	31	2	29	Some particles of appreciable size.
Meat in jars (tongue)	3	2	1	Minute particles.
Pickles, relish and sauce	11	2	9	Minute particles.
Totals	156	43	113	= 72·4 per cent.

The largest particles of glass were found in the jams (3/8″ × 5/16″, and smaller particles; pickles, ¼″ × 1/8″; ¼″ × 1/7″; 1/5″ × 1/7″, &c.); one particle 1/8″ long was found in an aerated water, but, in the main, these beverages contained minute glass fragments only (1/500th to 1/1000th or 1/2000th inch).

Whatever may be the actual result of the occasional ingestion of foods contaminated with these glass particles, it must be evident that small splinters and spicules of glass are potentially dangerous, and therefore undesirable impurities in food. At the present time the cheap glass jar has largely ousted the safer but more costly stoneware jar for the packing of jams and similar articles of diet. These glass jars are in some instances faultily made and contain air-bubbles which easily break down when the jar is filled with a hot liquid. Other glass containers, such as the bottles in which "lemonade crystals," sauces and coffee essences are sold, show the same defect, and are, moreover, sometimes so badly made that at the joint of the moulded bottle thin plates of glass, readily detachable, are evident to the eye. We have, during the last four years, come across several samples of "lemonade crystals" which were appreciably contaminated with glass particles in this way. The bottles showed many air-bubbles, some of which readily broke down internally on lightly rubbing them with a glass rod.

It seems probable that jam manufacturers do not always reject the jam in glass jars which have cracked and splintered when filled, but, after sieving this jam, re-pack it in glass or stoneware jars. Glass jars splintered, rough and sharp-edged on the top lip, have been seen by us in the case of samples received under the Sale of Food and Drugs Acts.

There is no doubt that the common glass jar at present made is often unsuitable for the reception of foods, especially for food such as jam, which is poured in hot. There are, of course, glass jam jars of superior make, but these are costly and not commonly used. Glass frequently contains lead, arsenic, and boric acid, and jars of this material may yield up any of these constituents, the amount and nature of which will vary with the contained food. Our examinations have revealed the fact that many foods contain also quite appreciable quantities of siliceous matter, some of the particles of which were sharp-edged and pointed.

If the quality of the glass containers used for packing many foods to-day can be improved, so as to remove the obvious objections, well and good; if not, it would be better for the purity of our food supply if we could get away from such containers altogether.

It is interesting to note that with the vogue in glass jars, jams and similar products are now almost invariably coloured with a coal-tar dye, whereas formerly they were frequently prepared without any added colouring matter.

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.

ZINC IN TINNED AND COPPERED PEAS.

OWING to an oversight in the note on the above in the last number of this journal it was stated that the zinc was determined nephelometrically as *sulphide*. We should have written *ferrocyanide*, which in our experience has proved more satisfactory.

CECIL H. CRIBB and A. L. STILL.

THE DETECTION OF ARACHIS OIL IN OLIVE OIL.

THE occurrence on the market of edible olive oil which, although free from arachis oil, gives a precipitate with Bellier's test in its various modifications, has been mentioned in previous notes (ANALYST, 1925, 182, 285). Several samples examined recently have been found to give a precipitate with Lüers' modification of the test (ANALYST, 1913, **38**, 58) at temperatures of 17° C. or over, requiring at least a further 0·5 c.c. of glacial acetic acid to lower the temperature of precipitation below 17° C. This is an excess of acid which renders the test considerably less delicate.

The precipitate appears to consist mainly of acid potassium palmitate, accompanied by unsaponifiable matter. It is well known that "saponified" oils give a precipitate in Bellier's test, due to their excessive proportion of unsaponifiable matter, but the effect of the smaller proportions normally present in edible oils, although not generally apparent, is not negligible. As an instance, the unsaponifiable matter separated from one of the oils in question, with an original melting point of 55° C., when crystallised once from 70 per cent. alcohol, gave a precipitate melting at 70° C., a figure which may have led to erroneous conclusions being drawn as to the presence of arachidic acid.

The following modified test, designed to eliminate these causes of precipitation, and capable of detecting the presence of 5 per cent. of arachis oil in olive oil, has therefore been worked out:—One grm. of olive oil is saponified with 5 c.c. of 2*N* alcoholic potassium hydroxide solution (made with 90 per cent. alcohol), the mixture being treated under a reflux condenser for 5 minutes. After dilution with 25 c.c. of water, the solution is well shaken with 30 c.c. of ether to remove the greater portion of the unsaponifiable matter; the separated aqueous solution is acidified with 2 c.c. of concentrated hydrochloric acid, and the fatty acids extracted by shaking with 20 c.c. of ether. This ethereal solution, after being washed once with 10 c.c. of water, is evaporated, and the fatty acids heated in the water oven for about 10 minutes. The separated fatty acids are dissolved in 50 c.c. of 70 per cent. alcohol, and the solution cooled to 14° to 15° C., and maintained at that temperature for an hour, or longer if necessary. In the greater proportion of oils tested 5 per cent. of arachis oil caused a precipitate at 15° C. in about half an hour, but in the case of an oil containing a high proportion of liquid fatty acids, cooling to 14° C., may be necessary to induce precipitation. Most oils which are free from arachis oil remain clear at 12° C.; in exceptional cases a precipitate may occur at 14° to 15° C. The melting point of the precipitate should therefore be taken in all cases where precipitation occurs above 14° C.

In the absence of arachidic acid, the original precipitate, after being well washed with cold 70 per cent. alcohol to remove soluble fatty acids, melts at about 52° to 54° C., and, after recrystallisation from 90 per cent. alcohol, at about 60–61° C. The precipitate from oils containing from 5 to 10 per cent. of arachis oil melts between 60° and 65° C., and one recrystallisation from 90 per cent. alcohol is usually sufficient to raise the melting point above 70° C.

As modern methods of olive oil extraction may involve the crushing of the fruit with sufficient pressure to break the kernel, it is probable that many olive oils contain a small proportion of the kernel oil, and the abnormal behaviour of certain oils in Bellier's test is, no doubt, due to the proportion of kernel oil being greater than usual. A sample of commercial olive kernel oil, consisting of a mixture of kernel oil with olive oil, gave a precipitate in Lüers' test similar to, but heavier than, that from the abnormal oils, the unsaponifiable matter (2·5 per cent.) yielding a heavy precipitate at 30° C.; when the unsaponifiable matter was removed, and Lüers' test then completed, a precipitate consisting mainly of acid potassium palmitate came down at 19° C. When the test was carried out on the separated fatty acids, it was necessary to keep the solution at 14° C. for several hours before a precipitate was formed, and the melting point of this was sufficiently low to indicate the absence of arachis oil.

As olive kernel oil probably contains stearic acid, as stated by Lewkowitsch (*Oils, Fats and Waxes*, 3rd Edition, Vol. 2, 631), the occurrence of a small proportion of this acid in olive oil is not unlikely; the presence of arachis oil should therefore not be assumed unless a fractionated acid melting at over 70° C. is obtained.

A. D. POWELL.

THE COMPOSITION OF LEMON CHEESE.

THE Standard for Lemon Cheese, put forward by Elsdon (ANALYST, 1925, 230) cannot be allowed to pass without comment. In so far as it is an attempt to prevent the sale of mixtures made according to the formulæ on p. 232 under this name, it is of course excellent, and it must command the support of every reputable manufacturer; but it goes far beyond this, and attempts to set up a standard, with which at least 95 per cent. of the lemon cheese on the market could not comply —not because such lemon cheese is in any way adulterated or misdescribed, but simply because such qualities, however desirable, are not what is generally known as "Lemon Cheese."

The first thing noticeable about the standard is that it fails to apply several important decisions of the High Court of Justice, and also that it takes no account of a very important section of the Sale of Food and Drugs Act. Two of the judgments of the High Court, which have been overlooked are *Sandys* v. *Rhodes* (67 J.P. 352) and *Anderson* v. *Britcher* (78 J.P. 65). It is true that neither of these cases refers specifically to lemon cheese, but in both cases a rule is laid down which would be followed in this case; the rule is that if a substance has been sold under a particular name, and that name is well known to the general public as a name for the substance sold, then it is no offence to sell the substance under that name, even though the name itself may mean pedantically something quite different from the article actually purchased. In fact, as the result of the above ruling, the question must be asked: "Is the article supplied under a particular name the same as the article known commercially under that name?" If the answer to this question is in the affirmative, then no offence has been committed; this is the legal position, and quite rightly so, and I am afraid that it will require much more evidence than that produced to alter it.

"Lemon Cheese" is a generic name for a substance known to the public as consisting of a mixture of sugar, edible fat, and egg substance, flavoured with lemon; during the manufacture of this substance there is added, under the provisions of Section 6, Sub-section 1 of the Sale of Food and Drugs Act, 1875 (which section Elsdon has apparently overlooked or ignored) a proportion of starch, generally cornflour; there is also added under the provisions of *Smith* v. *Wisden* (66 J.P. 150) and of *Wilson* v. *McCutcheon* (40 Sc.L.R. 31), a small proportion of glucose for the purpose of preventing the crystallisation of the cane sugar.

The writer has had the opportunity of seeing and supervising the manufacture of many tons of lemon cheese, and there is no question that the addition of these substances, on the manufacturing scale, is necessary, and therefore legal; it is quite true that lemon cheese can be made in the laboratory or the kitchen with edible fat, sugar, eggs and lemon, but this is because the cheese can be continuously and intimately mixed during the whole operation; but, on the manufacturing scale, where a couple of hundredweights, or more, are being made at once, this mixing, though it may be, and is, continuous, cannot be carried out in such a manner as to mix the constituents intimately, ; this has been tried and it is found that the fat will not mix with the sugar, but rises to the surface in the jars. A small quantity of water and cornflour is therefore added, when the fat becomes intimately mixed with the other ingredients.

Further, it is quite pedantic in these times to expect lemon cheese to be made with butter; if the same principle (namely, that because the formulæ given by the cookery books consulted demand butter, therefore any food product not following such directions is not of the nature, substance and quality demanded) is applied to all food products, then the majority of confectioners will have to be brought to the police courts, since most cookery books require cakes to be made with butter, but quite 99 per cent. of the cakes sold by retail are not so made. In the same way lemon cheese is generally made with margarine, and I contend that there is no offence in this, and, in support, I find that the *Daily Mail* published a cookery book in 1919 (after the War), which contains a formula for lemon cheese, and it is only to be expected that the formula given therein is correct; this formula supports my contention that lemon cheese is made with margarine.

Unlike Elsdon, I do not propose to give a formula, since these vary considerably, but the formula given by Elsdon on p. 231 should be so altered as to agree with the legal position—namely, that lemon cheese should consist essentially of cane sugar, butter or margarine, and eggs; that the presence of glucose and cornflour in reasonable proportions does not constitute an adulteration, and that lemon oil is generally used in place of lemon.

The use of tartaric acid or gum tragacanth is undoubtedly an adulteration, as is also the presence of salicylic acid. The presence of boric acid is due to the use of liquid eggs; if, and when, the importation of liquid eggs containing this preservative is prohibited, the presence of boric acid should be regarded as an adulteration, and even now there should only be a very small quantity present.

The actual quantities of each ingredient vary considerably, but at least 75 per cent. of the sample should consist of sugar (including glucose), butter or margarine, and eggs; the cornflour should not exceed 10 per cent.; the remainder should be water, citric acid and lemon oil.

There is also one statement in the paper to which exception must be taken, as it exemplifies the total ignorance of trade conditions which is very often displayed by the highly trained scientific mind; the statement is made that "The general public do not always consider that a lower price means an inferior article . . . they put variations down to keen competition, believing that traders will not be allowed to sell widely varying products under the same description." Unfortunately this is not in accordance with the facts; the general public has, since the

War, a most reprehensible habit of considering that the highest priced article is necessarily the best; also, whilst it is true that, in the wholesale trade, competition is keen and regulates the wholesale prices, yet in the retail trade, there is apparently an almost total absence of competition, except to find the highest price which the general public will pay for any article, however good or however poor. It is this obviously easy method of obtaining excessive prices for inferior articles, which has induced the production of the adulterated article made according to formula quoted by Elsdon on p. 232.

T. R. HODGSON.

THE SAFE-GUARDING OF SEALS.

MR. HODGSON'S note (ANALYST, 1925, 236), criticising my suggestion as to the sealing of samples (and incidentally of other things as well), constitutes in itself a strong proof that the original paper, of which an abridged note was published (ANALYST, 1925, 130), was not without its justification. Mr. Hodgson presumably was not present at the meeting at which the paper was read; if he had been, I think he would agree that the method of sealing which he mentions as being in use in the County Borough of Wallasey is absolutely no protection whatever; in fact, its very elaborateness is a weakness, in that it gives a false sense of security. As far as uncertainty as to the particular seal used goes, that is automatically eliminated, and, with regard to cost, I suppose that 2d. would be an extravagant estimate for copying the whole number. My suggestion was put forward tentatively in the hope that someone interested would adopt it and make it workable. With regard to the possibility of mixing the inks, I admit the difficulty; but surely if an inspector can take out one seal on a day's round without danger of getting it confused with its sisters he could also, and equally safely, take out one bottle of ink (with its attendant pen if necessary tied on to it), and that is all that is required. With regard to the difficulty of letting the referee analyst know, I am not competent to speak. Even if no key substance is used in the ink at all, I maintain that a signature on a seal is a greater protection than a raised device, inasmuch as the one requires at least some artistic ability to copy, whilst the other requires practically no ability at all.

B. S. EVANS.

THE DETERMINATION OF LEAD.

A METHOD which may frequently be of use depends upon the precipitation of lead from faintly acid solutions, by means of sulphur dioxide.

The lead is dissolved by any convenient method; free acid is removed by a slight excess of ammonia, and the solution is then rendered faintly acid with acetic acid. A brisk stream of sulphur dioxide is bubbled through the liquid until all the lead is precipitated and the solution is saturated with the gas. (With about 0·2 grms. of lead in 150 c.c. of solution this point is reached in about 10 minutes, and may generally be recognised by the supernatant liquid turning slightly yellow.) The precipitate is filtered off (Whatman No. 40 paper is suitable), washed free from sulphurous acid and rinsed into an excess of 0·1 *N* iodine solution. Concentrated hydrochloric acid is now added until all the solid is dissolved, and no trace of cloudiness remains. When this state is reached the filter paper is added to the liquid, stirred round, and the excess of iodine determined by titration with sodium thiosulphate.

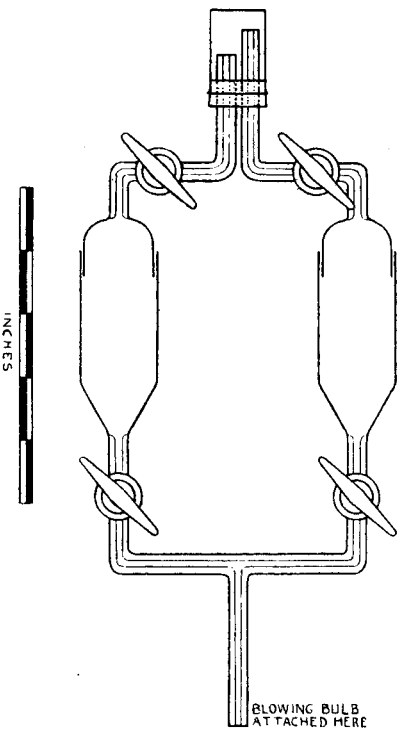
In order to obtain accurate results two conditions must be observed:—(1) The liquid must be saturated with sulphur dioxide; (2) sufficient hydrochloric acid must be added to dissolve the precipitate completely, as otherwise the end-point is very indistinct.

This method cannot be used in the presence of tin.

C. E. RICHARDS.

AN APPARATUS FOR THE DETECTION OF AIR MOVEMENT.

IN a previous communication (ANALYST, 1925, 213) attention was drawn to the extreme importance of air movement in the investigation of ventilation conditions, and passing reference was made to an apparatus designed by the author to detect this; some details may be of interest. The apparatus will detect very slight air currents, and is useful, not only for determining the general air movement in a space, but also for discovering sources of entry and exit; it also furnishes some idea of the velocity of these movements.



The principle is simple, but difficulties were encountered in utilising it in practice; these were overcome by modifications embodied in the apparatus illustrated. The apparatus is entirely of glass, and may be conveniently mounted on a board. A T-piece of thick-walled tubing is bent at right angles at each end, and the extension of the attached stopcocks opens out in each case into a wide tube or container. The containers are fitted with hollow stoppers which, again, are attached to stopcocks in tubing bent in the manner shown. The ends of these tubes, the right-hand one of which projects 0·4 inch beyond the other, are held together by being passed through a cork which, again, supports a cylindrical shield. Each container is about half filled with asbestos (a brand which has been found suitable is "Asbestos for Gooch crucibles B.D.H."), and this may be previously dyed with litmus solution and dried.

To prepare the apparatus for use all stopcocks are shut off, and it is then disconnected by removing the right-hand stopper and tube together with the shield; to facilitate this, the cork carrying the shield fits this tube tightly, but is made so as to slip readily off the left tube. The left container having been unstoppered, a small quantity of ammonia is added for absorption by the asbestos and the stopper replaced. The right container is now unstoppered, and after the addition of concentrated hydrochloric acid, it is re-sealed by the stopper carrying the shield, which is slipped over the left tube. A blowing bulb is attached, as indicated in the diagram.

In using the apparatus the four stopcocks are opened, and by simply exerting gentle pressure on the blowing bulb a continuous cloud of artificial smoke is

projected. It is hardly necessary to explain the action. The air passing through the apparatus is divided into two parts; to one gaseous ammonia, and to the other gaseous hydrochloric acid, is added; these come together within the shield, with consequent production of ammonium chloride. It will be seen that no carbon dioxide or other material which would vitiate the results in an investigation of the ventilation conditions in a space is added to the air by the action of the apparatus.

The apparatus is obtainable from Messrs. Casella & Co., Ltd.

ROBERT C. FREDERICK.

ROYAL NAVAL MEDICAL SCHOOL,
R.N. COLLEGE, GREENWICH, S.E.10.

Notes from the Reports of Public Analysts.

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

COUNTY OF KENT.

THE total number of samples examined under the Fertilisers and Feeding Stuffs Act was 1395, representing an increase of about 300 samples over the previous year. In only one instance was it found possible to institute proceedings against a vendor for a false warranty. In nearly every other instance the vendor could not be convicted for giving a false warranty, as he had obtained from the manufacturers a guarantee, which, presumably, he had no reason to believe to be false. This particularly applies to the shoddy manufacturers, who sell through local agricultural agents who do not handle or see the material itself.

SHODDIES.—Of the 294 samples examined, 169 were sold at "unit value," and 116 of the remainder with a definite guarantee. Of these, no less than 70 contained less ammonia than guaranteed, and 60 per cent. of all the samples were below the guarantee. The deficiencies in ammonia were mainly in the grades containing between 5 and 9 per cent.; and in no case was there a material deficiency in shoddy guaranteed to contain more than 14 per cent. of ammonia. The low quality was caused either by excessive quantities of water or of mineral matter. The highest water content found was in felt cuttings, which contained 72 per cent. The mineral matter had sometimes been added knowingly to give weight.

FEATHER WASTES.—These were very variable in composition. Clean feathers, as sold commercially, should contain from 14 to 16 per cent. of ammonia, but, owing to the presence of admixed dirt, the ammonia content is liable to be low. In one case, for instance, 47 per cent. of mineral matter was present, and the waste contained only 6·5 per cent. of ammonia. *Wings of fowls*, sold whole, contained: Water, 15·6; ammonia, 12·92; phosphates, 11·3; and mineral matter, 14·7 per cent. Whole wings of birds decompose very slowly in the soil, and should only be used when a slow supply of nitrogen is required.

STEAMED BONE FLOURS.—Four samples contained less than the guaranteed amount of phosphates. In 3 instances the guaranteed phosphates were abnormally high, exceeding 70 per cent., the flours having been well dried to obtain a minimum of moisture. On analysis, however, the phosphates were 2·6, 2·3 and 4·0 per cent., respectively, below the guaranteed amount.

BLOOD AND SKIN WASTE.—Samples examined had the following composition: Water, 68·8; ammonia, 1·0; phosphates, 0·2; and lime, 1·5 per cent.

"LOOSE MANURE."—A fertiliser sold under this name contained about 46 per cent. of water, about 1 per cent. of ammonia, and 14 per cent. of organic matter, and could be considered as little else than rich soil. There is little doubt that considerable quantities of these doubtful products are sold annually, buyers being attracted by the low price asked for them.

MEAT AND BONE MEALS.—A considerable number were found to be deficient in phosphates or ammonia. In 9 samples a guarantee of 8 per cent. of ammonia and 35 per cent. of phosphates had been given, and in every instance the phosphates were in excess of the guarantee (sometimes as much as 5 per cent.), whereas the ammonia was deficient.

PHOSPHATIC GUANO.—A new type, which will probably soon reach the English market, should prove a valuable fertiliser, since its phosphates are almost entirely soluble in citric acid solution. It contained nearly 3 per cent. of ammonia, about 40 per cent. of total phosphates, and a little potash.

BARLEY MEAL.—A sample was found to contain at least 30 per cent. of wheat. Another sample sold by the same firm contained not only a proportion of wheat, but also a considerable amount of ground cereal husk, so that the meal contained no less than 19·5 per cent. of crude fibre. In each case the farmer had doubted the meal owing to the condition of his pigs after he had used it for a few days.

MEAT MEAL.—A sample of otherwise satisfactory composition contained 4 per cent. of salt, which was much more than is usual.

BRACKEN POISONING.—A case of poisoning among cattle was found to be caused by bracken. Large quantities were separated from the stomachs, and the condition of the viscera afforded confirmation of the action of the bracken in causing the death of the cattle.

F. W. F. ARNAUD.

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.

CITRIC-SOLUBLE PHOSPHATE IN FERTILISERS.

ON June 20 a company was prosecuted at Torrington Police Court by the Devon County Council with the consent of the Ministry of Agriculture, in respect of ten tons of fertiliser, called 'Plutophos," supplied to an agriculturist.

There were three summonses. The first alleged that on the sale of the fertiliser the defendant failed to give the invoice required by Section 1 and 10 (1) of the Fertilisers and Feeding Stuffs Act. Mr. H. A. Davis, assistant solicitor to the Council, stated that the seller of a fertiliser containing soluble phosphates must state either the amount of phosphates soluble in water or the amount soluble in a solution of citric acid. If the latter alternative is taken, the citric acid solution must be of the strength prescribed by the Regulations made under the Act, and

must be operated for the limited period also prescribed. The defendants stated on their invoice and on their advertisements that 95 per cent. of the total phosphates in "Plutophos" was soluble in a solution of 0·2 per cent. citric acid, when 1 grm. of plutophos was shaken in 5 litres for two hours. The amount of acid used was the same as in the official test, but the period of shaking was four times as long, and the official test prescribed 5 grms. of phosphate not 1 grm., as stated on the invoice. The results obtained by the County Analyst were 17·6 per cent. dissolved by the official test and 57·2 per cent. by the method described by the defendants.

The defendants substituted their own test for the standard test prescribed in the Regulations, thus enabling them to state a higher degree of solubility than would have been produced by the standard test.

After hearing the solicitor for the defence, the Bench decided to fine the defendants £20.

The Bench then dealt with the second summons, which alleged "that the defendants on the sale of a fertiliser known as 'Plutophos' did cause or permit a certain invoice of the said fertiliser to be false in a material particular, *viz.*, by stating that 95 per cent. of the soluble phosphates contained in the said fertiliser were soluble in the solution of citric acid specified in the invoice, whereas in fact only 57·2 per cent. were so soluble, there being a deficiency of 14·05 per centum to the prejudice of the purchaser thereof, contrary to Section 6(1)(B) of the Fertilisers and Feeding Stuffs Act, 1906."

Mr. Davis stated that the invoice was false, even if the analysis of the fertiliser was carried out in accordance with the method laid down by the defendants' invoice, and was false to a far greater degree if the analysis was made by the method prescribed by the Act.

Mr. Dutton, the County Analyst, gave evidence as to the result of his analysis. For the defence it was argued that the defendants had used all reasonable care in respect of the quality of the fertiliser, and the defendants produced a certificate of Dr. Bernard Dyer that "Plutophos" had been analysed and found as stated in the invoice. Mr. Davis contended that there was nothing proved to connect this certificate with the consignment to the purchaser. The Bench considered the case proved and imposed a fine of £20.

The third summons also alleged that the invoice was false inasmuch as it stated the "Plutophos" contained 75 per cent. of pure phosphates. Mr. Dutton stated that the phosphates amounted to 72·9 per cent., a deficiency of 2·1 per cent. The Bench considered the deficiency less serious in this case, and imposed a penalty of £5.

"CREAM CHESHIRE CHEESE" AND "CREAM CHEESE."

ON June 10 a dairy company was summoned at Salford for selling "Cream Cheshire Cheese" and "Cream Cheese" not of the nature, substance and quality demanded.

Mr. G. D. Elsdon, the Public Analyst, certified that the first sample contained 32 per cent. of fat, 42 per cent. of water, 22 per cent. of proteins, and 4 per cent. of mineral matter, milk sugar, etc. It was an ordinary whole milk cheese.

The second sample contained 27 per cent. of fat, 44 per cent. of water, 24 per cent. of proteins, and 5 per cent. of mineral matter, etc. It, too, was an ordinary whole milk cheese.

Although there was no standard fixed, the analyses of hard cheese over a number of years in Salford had shown, on the average, 30 to 33 per cent. of fat. Cream cheese should contain approximately 70 per cent. of fat. The proportion

of fat to proteins should be not less than 3:1. In the first sample the proportion was as 1·4 to 1, and in the second sample as 1·1 to 1.

In cross-examination he said that he had no standard in mind when issuing his certificate. The samples were so much below the lowest possible standard. He admitted that there was no standard for unpreserved cream, but suggested that it should contain at least 35 per cent. of fat.

Mr. W. H. Roberts, City Analyst for Liverpool, agreed with Mr. Elsdon's certificate, and stated that he considered the samples to be whole milk cheeses and not cream cheese. In his opinion cream cheese should be made from cream only, with or without the addition of rennet. An ordinary whole milk cheese should contain 45 to 60 per cent. of fat, calculated on the dry cheese.

Mr. Ricketts, for the defence, contended that there was no legal standard for cheese and no legal standard for cream. He took exception to the Analyst's certificate on the ground that the Analyst ought to have given his reasons for not calling the first sample a cream cheese, and he quoted several cases in which the prosecution had failed owing to the vagueness of the certificate. The cheese sold was not a new article; it was not made with the intention of defrauding the public, and no person would dream of thinking it an ordinary cheese. It was made from whole cream milk as distinct from the skimmed or separated article.

The Magistrate, who had previously over-ruled the legal objection to the certificate, imposed a fine of £10, with 10 guineas costs.

The second summons, relating to a Gruyere cheese, was withdrawn on the technical ground that there was no contract for the sale.

SALE OF AN "INVALID WINE."

ON June 24th a grocer was summoned at Salford for selling "Liebig's Invalid Wine," which was not of the nature, substance and quality demanded, and a trading company was summoned as the vendors for aiding and abetting. Mr. F. Ray, barrister, pleaded guilty for both parties.

Mr. R. H. Wright, for the Salford Health Committee, said that the sample, when analysed by the Public Analyst, was found to contain:—Water, 80; total sugars, 18; alcohol, 1·5; and other solid matter, 0·5 per cent. The last item (0·5 per cent.) contained:—Nitrogen, 5 parts in 100,000; quinine, 3 parts in 100,000; phosphorus, 4 parts in 100,000; and salicylic acid, 5 parts in 10,000. The preparation in question had not been made by the Liebig Company, and, while the prosecution did not complain that the defendant company was using somebody else's name, they submitted that the name "Liebig" suggested that there was extract of meat in the article.

The prosecution, continued Mr. Wright, did not agree with the description "wine," and the analyst would say that "wine" was the fermented juice of the grape. The comment of the analyst was: "This is not an invalid wine. Its composition is similar to that of a flavoured artificial cordial, except for the presence of a trace of quinine, which only amounts to about a quarter of a grain per pint."

On the bottle was a small label, which said: "This beverage is prepared in accordance with the requirements of the Food and Drugs Act, and contains a small quantity of salicylic acid as a preservative." Another label said: "Recommended by the medical profession. Health, strength, vigour. Liebig's invalid wine. Strengthening and nutritious. Take a wineglassful three times a day. Non-excisable."

Mr. Ray, for the defence, said that the prosecution would be unable to bring any evidence that the preparation had injured anyone; it was simply that it did not comply with the requirements of the law. The company's explanation of having broken the law was that the wine had been manufactured for them according to a formula, and that the formula did not indicate the amount of salicylic acid being put into the preparation. Had it not been for the fact that an excess of salicylic acid was present, the allegations and inferences of the Public Analyst would have been disputed. Acting on his (counsel's) advice, the Company decided to accept full responsibility for having broken the law. He suggested that what constituted an "invalid wine" was purely a matter of opinion.

In reply to the Stipendiary, counsel said he must admit that a decent wine must necessarily be the product of the grape. Questioned about the use of the word "Liebig," he replied that the Company was not charged with false representation, but with selling something to the prejudice of the consumer.

A nominal fine of 5s. was imposed on the actual vendor, and the Company was fined £20 and 50 guineas costs.

Sources of Industrial Alcohol.*

THE Report is supplementary to that published in the *Kew Bulletin*, 1912, 113–130, and calculated and determined yields of alcohol with the sources of information (here mostly omitted) are given under similar headings.

FRUITS.—Carob or Locust Bean (*Ceratonia Siliqua* Linn.), 18–24 litres per 100 kilos. Smyrna fig (*Ficus Carica* Linn.), the alcohol yield from ripe figs is about the same as that from plums, *viz.* 30 to 33 litres of 54 per cent. alcohol from 100 kilos of dried figs, one hectare of fig trees producing 800 to 900 litres of 90 per cent. alcohol. Prickly pear (*Opuntia spp.*): Areas infested by these species in 1919 were estimated at over 20 million acres in Queensland, and over 2 million acres in New South Wales. For profitable collection there should be at least 10 tons of fruit per acre, capable of producing about 110 gals. of spirit. Motor spirit of this origin from the Orange Free State is called "Springbok," and is mixed with certain chemicals (not disclosed). Coffee Pulp (*Coffea arabica, C. liberica, C. robusta*): 127 litres from 100 kgrms. Horse Chestnuts (*Aesculus Hippocastanum* Linn.): 27–28 litres from 100 kilos. of dried fruits (*cf.* Baker and Hulton, ANALYST, 1917, **42**, 500). Acorns of the common and evergreen oak: From 100 kilos of dry whole acorns, 8·58 to 20·16 litres, or 28 to 31 litres of alcohol from the kernels. Persian lilac (*Melia Azedarach* Linn.): Nearly 10 per cent. on the air-dried fruit.

ROOTS, TUBEROUS ROOTS AND ROOTSTOCKS.—Beetroot (including sugar beet), mangold and mangel. Experiments in 1920–1923 at the Royal Naval Cordite Factory, Wareham, with French seed yielded 16–32 tons of roots per acre, calculated to give 10 gals. of 95 per cent. alcohol per ton (*Fuel Res. Brd.*, 2nd Memo., 1921; 3rd Memo., 1925). Bitter and sweet Cassava (*Manihot utilissima* Pohl.): Freshly dug roots, pulped and boiled and treated with malt, yielded 18·9 gals. per ton, or 75·6 gals. per ton on the dried material. Roots purchased in the market and treated with taka-diastase gave 81·5 gals. per ton on the dried material. Potato (*Solanum tuberosum* Linn. var.): In Germany the alcohol industry has been under State regulation since 1919, and both the price of alcohol and that which manufacturers

* *Kew Bulletin of Miscellaneous Information*, 1925, 5, 193–216. Obtainable at H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 1s. net.

may pay for potatoes used in its production are controlled. The production of alcohol in 1921–1922 was estimated at 1,100,000 hectolitres, and, to make up for the scarcity of potatoes, maize was imported. Arrowroot (*Zamia floridana* DC.) is restricted to Florida and not to be recommended for cultivation. Burrawong of Australia (*Macrozamia spiralis*): The yield of alcohol from both inner and outer cores is about 14 gals. per ton, which, with the butts being delivered at 12s. per ton, would make the price of the raw material (alcohol) 10d. per gal. This source would only be valuable while the wild resources last. Artichoke (*Helianthus tuberosus* Linn.): A ton of roots are estimated to yield 19 gals. of 95 per cent. alcohol. Sweet Potato (*Ipomoea batatas* Linn.). A liquid fuel from this source, "Acetol," has been patented in South Africa.

GRAIN.—Eddoes (*Colocasia antiquorum* Schott.) and tannias (*Xanthosoma sagittifolium* Schott), and yams (*Dioscorea spp.*), being native food products, are not likely to pay for alcohol production, and have a longer period of growth than sweet potatoes. Maize (*Zea mais* Linn.): One ton yields 83 gals. of 95 per cent. alcohol, and one 200 lb. bag of "mealies" 8 gals. "Penrol" is a South African motor spirit described as alcohol from "mealies," and dissolved acetylene. Sorghum, Guinea Corn or great millet (*Sorghum spp.*) yields about 85 gals. of alcohol per ton; rice (*Oryza sativa* Linn. var.), 88 gals. of 95 per cent. alcohol.

STEMS.—Grass trees or "black boys" (*Xanthorrhoea preisii* Endl., *etc.*): One ton of core gives an average yield equivalent to 19 gals. of 95 per cent. alcohol; sago palms (*Metroxylon Rumphii* Mart.) give approximately 550–650 lbs. of raw starch per tree. Sorghum stems (*Sorghum saccharatum*), cleaned, yield about 12 gals. of 95 per cent. alcohol per ton. Sugar cane (*Saccharum officinarum* Linn.): In general, one ton of sugar will give 16 gals. of first class alcohol. "Espiritu Mortor" is an example of a denatured alcohol from sugar, and is used in Havana for motors, 2000 of which used it in 1922.

LEAVES.—Sisal hemp (*Agave sisalana* Perr.): The sap obtained by pressing the leaves varies in density according to the season, and 1000 medium leaves may yield from approximately 21·6 kilos. of sugar (13 litres of absolute alcohol) to 36·5 kilos (22 litres of alcohol). It is possible that by application of a recently discovered electrolytic method of purification for cane juice (*Inter. Sugar J.*,1921, 418, 470) to agave leaf juice, which may contain 10–15 per cent. of sucrose, it will become possible to overcome the fermentation difficulties.

INFLORESCENCES.—Nipa Palm (*Nipa fructicans* Thunb.): About 250 gals. of spirit per annum (estimating 100 gals. of sap to produce 6–7 gals. of alcohol) should be produced by each acre under management. Mowrah (*Bassia latifolia* Roxb.): It has been estimated that in Hyderabad State alone 700,000 gals. of proof spirit would be given by the mowrah trees found there, at a cost of 30s. per ton for collecting, drying and delivery of the flowers at a factory in the area of growth. By native methods 82 lbs. of dried flowers yield 2·8 gals. of proof-spirit, but in England over 6 gals. could be obtained from the same quantity.

CELLULOSIC MATERIALS.—Peat. The average yield of 95 per cent. alcohol per ton of peat is 15 gals. (*Fuel Res. Bd.*, 2nd Memo.) *Seaweed*: 100 lbs. of red wrack (*Laminaria Cloustoni* Edm.), dried to a moisture content of 10 per cent., and heated for a short time with weak sulphuric acid, and the acidity further reduced after cooling, are capable of yielding, after fermentation with brewers' yeast, about 6 litres of alcohol on distillation (Thorpe). Prickly Pear Stems (*Opuntia stricta*, Haw.). The green parts of the Queensland plant contain 0·64 per cent. of total sugar as dextrose, and, when prepared under good conditions, the yield of alcohol per cent. of weight of plant used was 0·5.

DENATURANTS.—Among the suggested denaturants of alcohol of vegetable origin are "tobacco oil," rape, linseed or castor oil fatty acids with petroleum distillate, "resin spirit," or pine oil with benzol and nitrobenzol (allowed by the Canadian Inland Revenue instead of methyl alcohol), citronella oil (in Ceylon), and eucalyptus oil (in New South Wales).

An extensive bibliography is added, and also an "Index to Plants and Subjects," which refers both to the 1912 Report and to the present one.

D. G. H.

Sale of Paris Green.*

OWING to the prevalence in some districts of an epidemic of crane fly or "daddy long legs" (*Tipula oleraceae*), there has been an abnormal demand for Paris green as an effective agent for the destruction of the larvæ ("leather jackets"), which had caused extensive destruction of cereal crops. Varying opinions have been expressed as to whether or not Paris green comes within Part I. of the Poisons Schedule (*cf.* ANALYST, 1925, 291), but from the definition given by the Arsenic Act of 1851 it seems clear that it does come within Part I. and must therefore be registered and signed for, whether sold by a qualified pharmacist or by an agricultural or horticultural licensed poison vendor.

* *Pharm. J.*, 1925, **114**, 719.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Rapid Routine Method for the Determination of Total Solids in Eggs. R. Hertwig and L. H. Bailey. (*J. Assoc. Off. Agric. Chem.*, 1925, **8**, 451–454.)—The proportions of total solids found by drying commercial dried egg, previously ground and sifted through a household flour sifter, in a ventilated air-oven for about 1 hour at 110° to 119° C., or by drying liquid egg at 115° C. for about 3 hours after the bulk of the moisture has been expelled by heating on a steam-bath for about 30 minutes, agree well with the results obtained by the "official" vacuum method, and are not affected by slight variation in the time or temperature.

T. H. P.

Delicate Test for Benzoperoxide (Benzoyl peroxide) in Flour. F. Kirchhof. (*Chem. Zeit.*, 1925, **77**, 535.)—Benzoperoxide (benzoyl peroxide) can be detected in a solution of 1 part in 100,000 by means of titanium-sulphuric acid. This is prepared by dissolving 0·1 to 0·2 grm. of titanium oxide (TiO_2) in hot concentrated sulphuric acid and allowing it to cool. A minute crystal of benzoperoxide (de Haën's "Luzidol") in this solution gives an intense orange-yellow colour, and a larger amount a cherry-red colour which persists for a long time. Five c.c.

of a 1:20,000 solution of benzoperoxide shaken with 1 c.c. of the above titanium-sulphuric acid give an intense orange colour; 5 c.c. of a solution of 1 part in 200,000, after prolonged shaking, still give a distinct yellow colour. The reaction is less sensitive in the presence of moisture, so that it is better to dissolve the benzoperoxide in carbon disulphide rather than in benzene. On the addition of water the colour given by the benzoperoxide completely disappears, probably as a result of hydrolysis of the coloured compound produced, thus differing from the colour known to be produced by hydrogen peroxide in *dilute* titanium-sulphuric acid. The colour reaction is probably due to the formation of perbenzoic acid and its combination to form a complex pertitanic acid. R. F. I.

Studies on Glycogen, Part I. The Nature of Yeast Glycogen, its Preparation, Estimation, and Rôle in Yeast Metabolism. A. R. Ling, D. R. Nanji and F. J. Paton. (*J. Inst. Brewing*, 1925, **31**, 316–321.)—The method used for preparing glycogen consists essentially in extraction of dried yeast with boiling 2 per cent. sodium hydroxide solution, precipitation with alcohol, and removal of the mannan extracted with the glycogen by addition of Fehling's solution to the aqueous solution of the mixture. In estimating the glycogen and mannan in yeast during various stages of fermentation, the yeast was skimmed off, filtered and washed with water on a Buchner funnel, and 50 grms. of the yeast of known moisture-content heated with 110 c.c. of 85 per cent. potassium hydroxide solution for 2 hours on a water-bath. The cold liquid was made up to 500 c.c., and filtered, and each of two 100 c.c. portions of the filtrate precipitated by addition of alcohol to bring the concentration of the latter in the liquid to 55 per cent. The precipitates were allowed to settle overnight and were then washed with 60 per cent. and afterwards with 95 per cent. alcohol, dissolved in hot water and neutralised. One of the solutions was treated with sufficient sulphuric acid to make the final concentration of the acid 8 per cent. The second portion was treated in warm 2 per cent. sodium hydroxide solution with Fehling's solution, and the precipitated mannan allowed to settle; the clear supernatant liquid was decanted and the precipitate transferred to a small filter-paper, washed with 2 per cent. sodium hydroxide solution, the mannan compound on the filter being dissolved in cold 8 per cent. sulphuric acid, and the filter-paper washed with similar acid. The two solutions were then heated for three hours under a reflux condenser, cooled, neutralised carefully with sodium hydroxide, made up to 200 c.c., and the solution containing mannan filtered through a fine filter-paper from the precipitated copper hydroxide. The dextrose and mannose were then determined iodimetrically in aliquot parts of the two solutions. T. H. P.

Relative reducing Powers of Sugars. A. W. Rowe and B. S. Wiener. (*J. Amer. Chem. Soc.*, 1925, **47**, 1698.)—The following sugars have been tested for velocity of reduction and found to be widely different:—Mannose, laevulose, galactose, maltose, and lactose. By the Folin and Wu method, after six minutes' boiling, the reduction velocity of maltose is only 40 per cent., and that of mannose 55 per cent. of that of glucose. By the Lewis and Benedict method, after ten

minutes' boiling, the reduction velocity is quite different, that of mannose being equal to glucose, and that of maltose being 82 per cent. of that of glucose. A chart is given showing the equivalent reductions of various mixtures of glucose with each of the above sugars. The reduction velocity is proportional to the relative amounts of the several compounds and to the reducing power of each. A table is also given showing that the effect of the time factor is highly variable. Reduction values in aqueous solutions are shown to be the same as in sheep-blood plasma. R. F. I.

"Neutralising Value" of Monocalcium Phosphate in Baking Powders. L. H. Bailey. (*J. Assoc. Off. Agric. Chem.*, 1925, **8**, 444–447.)—Accurate determination of the neutralising value of monocalcium phosphate, that is, the number of parts of sodium bicarbonate reacting with 100 parts of the phosphate at the boiling point, is not possible by titrimetric methods, but may be effected as follows:—A quantity of 26·73 grms. of sodium bicarbonate is mixed with 41·77 grms. of the monocalcium phosphate and, if desired, 31·50 grms. of neutral starch, the mixture being kept dry until used. One grm. of this test baking powder is boiled in a 250 c.c. Pyrex beaker with 100 c.c. of distilled water until free from carbon dioxide, the P_H value of the cold supernatant liquid being measured electrometrically or colorimetrically. If a value other than 7·0 is obtained, other test powders containing either more or less of the phosphate are similarly tested. The results may be checked by determining the residual carbon dioxide, which should be absent if the value of P_H is 7 or less. T. H. P.

Sunflower and Safflower. Distinctive Features of the Seeds and Cakes. Vizern and Guillot. (*Ann. Falsif.*, 1925, **18**, 284–286.)—Oils of the seeds of the sunflower (*Helianthus annuus*) and safflower (*Carthamus tinctorius*) are difficult to distinguish. Cold extracted oils had the following characteristics:—Sp. gr. at 15°C., 0·925 and 0·926; saponification values, 190 and 190; iodine values, 129 and 141; "titer" test, 17° and 16° C. Carthamus seeds are white, about 9 mm. long and 4–5 mm. wide with 4 longitudinal ridges. Sunflower seeds are irregularly coloured from black to white and are about 12–16 mm. long by 6–9 mm. wide, and flattened. The cakes may be distinguished by boiling the separated pericarp débris with dilute potassium hydroxide solution so as to allow transverse sections to be cut; these are stained in the usual way. In carthamus seeds the outer 6–10 layers of mesocarp cells (immediately under the epicarp) are separated from the middle layers by a row of black cells, and often the outer layers are found broken away, leaving these black cells on the periphery. The middle mesocarp layer consists of strongly sclerified cells, the smaller cells being on the outside, and this zone extends uniformly through the preparation. In the sunflower no black cells are present, and the sclerified cells of the middle mesocarp are larger on the outside, but the outstanding feature is the presence in the middle mesocarp of regularly placed longitudinal radial bands of cells which, although sclerified, have thin walls and give rise to well marked partitions in the mesocarp. D. G. H.

Determination of Yohimbine in Barks and Galenic Preparations of "Yohimbehe." Raymond-Hamet. (*Bull. Sc. Pharm.*, 1925, **32**, 21–27; *J. Pharm. Chim.*, 1925, **117**, 493.)—Since the toxicity and therapeutic value of yohimbine is widely different from that of other alkaloids present in yohimbehe, yohimbine should be determined separately, The total alkaloids are extracted with an alkaline mixture of ether and chloroform, the solution extracted with dilute sulphuric acid, and the alkaloids again dissolved in ether and rendered alkaline with sodium carbonate. The residue left after distilling the ether is dissolved in absolute alcohol, the solution slightly acidified with hydrochloric acid in absolute alcohol, evaporated to dryness, and taken up with absolute alcohol. Yohimbine hydrochloride remains insoluble and may be separated and weighed.

D. G. H.

New Principle for Analysing Organo-therapeutic Powders. M. Javillier, H. Allaire and M. Groc. (*J. Pharm. Chim.*, 1925, **117**, 513–525.)—Since the composition of the various organs is shown to be characteristic with regard to certain constituents, a determination of these constituents will afford information as to the purity of the preparation. A summary of some of the tables of figures given in the paper for the organs of the horse, pig, cow, calf, and sheep is as follows:—*Lipoids.*—40 to over 80 per cent. on the dry material are present in nerve tissue; 25–40 per cent. in the suprarenal glands, liver, pancreas and testicles; less than 25 per cent. in the lungs, heart, kidneys, muscle, spleen, thyroid, and ovary (variable). *Total Phosphorus.*—1·3 to over 2 per cent. on the dry material in thymus, nerve tissue, pancreas, suprarenal glands, and testicles; 1 to 1·3 per cent. in spleen, kidney, liver, and lungs; less than 1 per cent. in heart, ovary, muscle, and thyroid. *Lipoid Phosphorus.*—0·6 to 1·4 per cent. on dry material in nerve tissue, hypophysis and suprarenals; 0·35 to 0·6 per cent. in testicles, liver, pancreas, lung, heart, kidneys, and ovaries (variable); less than 0·35 per cent. in placenta, spleen, thymus, thyroid, breast and muscle. *Nucleic Phosphorus.*—Over 1 per cent. in thymus; 0·3 to 0·6 per cent. in pancreas, spleen, placenta, lungs and testicles; 0·1 to 0·3 per cent. in prostate, liver, kidney, and ovaries; less than 0·1 per cent. in nerve tissue, heart and muscle. The ratios of certain figures are also significant, and it is possible to establish, for example, the following values for certain preparations:—

	Lipoid P. / Total P.	Nucleic P. / Total P.
Muscle powder	15–22 per cent.	4 per cent.
Splenic powder	20–26 ,,	30–31 ,,
Cerebral white matter powder	70–75 ,,	2–3 ,,
Thymus powder	8–10 ,,	over 60 ,,

D. G. H.

Biochemical, Bacteriological, etc.

Copper, Manganese, Zinc, Nickel and Cobalt as Vital Factors in Soils, Plants, and Animals. J. S. McHargue. (*J. Agric. Res.*, 1925, **30**, 193–196.)—Further evidence is adduced in support of the thesis that small amounts of these metals are necessary for vital metabolic processes (*cf.* ANALYST, 1924, **49**, 445).

A number of different animal and plant materials have been examined and found to contain small amounts of these metals. A few of the results in parts per million on the dry material are:—

	Cu.	Fe.	Mn.	Zn.	Ni.	Co.
Blue grass	7·5	336·0	30·0	28·0	trace	trace
Soya bean leaves	8·0	336·0	160·0	110·0	—	,,
Wheat bran	16·0	210·0	125·0	75·0	—	—
Patent flour	trace	24·0	10·0	trace	—	—
Rice polishings	7·0	168·0	100·0	70·0	—	—
Lean ox meat	0·4	225·0	trace	15·0	—	—
Blood of calf	8·0	1720·0	trace	32·0	—	—
Egg yolks	2·5	100·0	1·5	67·0	—	—

H. E. C.

Decomposition of Proteins and Amino Acids by Micro-organisms. S. A. Waksman and S. Lomanitz. (*J. Agric. Res.*, 1925, **30**, 263–281.)—The mode of decomposition of amino-acids and casein by certain mould fungi, bacteria and actinomyces has been followed by measuring the residual amino nitrogen, the formation of ammonia, and the disappearance of dextrose. The organisms do not act alike. The mould fungi utilised the amino-acids and protein as sources of carbon and nitrogen, producing ammonia in quantity depending upon the carbon content of the particular amino-acid molecule. The bacteria (*B. cereus* and *fluorescens*) were unable to attack glycocoll, alanine or phenylalanine, but rapidly decomposed casein, with the formation of ammonia. The actinomyces utilised amino-acids and accumulated ammonia. Ammonia accumulation cannot be used as an index of proteolytic activity except when co-related with the carbon content of the amino-acids, since its formation depends upon the ratio of carbon to nitrogen.

H. E. C.

Papain Lipase. M. Sandberg and E. Brand. (*J. Biol. Chem.*, 1925, **64**, 59–70.)—This investigation was carried out since practically nothing was known of the presence of a lipase in crude papain. Crude papain contains a lipase of considerable activity. Papain lipase splits both higher glycerides and lower esters. It is insoluble in water and has its optimum effect at 35–40° C. at a P_H of 5·8 to 6·2 in acetate buffer. As to the kinetics, the law of Schütz is followed to a certain extent. Hydrogen cyanide and bile salts have no effect at all on the activity of the lipase. Calcium chloride, however, activates papain lipase in an alkaline medium (changing P_H). Papain lipase differs from castor lipase in its P_H optimum and its susceptibility to activation by calcium chloride in an alkaline medium (changing P_H), but, so far, sufficiently definite investigations to decide whether they are really two different enzymes have not been made. Purification of papain lipase is possible by removing the papain with water. Full details of the experiments carried out and their results are given. P. H. P.

Resistance of the Antirachitic Substance in Cod Liver Oil to Reagents. C. E. Bills. (*J. Biol. Chem.*, 1925, **64**, 1–9.)—Experiments are described which were carried out on unfrozen, unadulterated, Newfoundland cod liver oil when

starting a chemical examination of antirachitic substances. Rachitic rats were given the oil crude and after it had been treated with various reagents. The antirachitic vitamin, vitamin *D*, in cod liver oil is not destroyed by hydrogen dioxide, hydrogen sulphide, sulphur dioxide or formaldehyde. It is readily destroyed however, by nitrous fumes, and slowly by direct steam or contact with mineral acids. In these experiments the non-active constituents of cod liver oil may, in some cases, have protected the vitamin against the reagents employed; in other cases they may have accelerated its destruction. A table shows the results. P. H. P.

Antiscorbutic Properties of Eggs. S. M. Hauge and C. W. Carrick. (*J. Biol. Chem.*, 1925, **64**, 111–112.)—In an experiment to test the potency of the egg in relation to its prophylactic and therapeutic value in scurvy, eggs were used from hens in an experimental lot which had been fed on an antiscorbutic ration. Egg whites and egg yolk were ineffective in delaying the symptoms of scurvy in guinea pigs. The therapeutic action of egg portions was also negative. The presence of the antiscorbutic substance was not demonstrated. The authors do not consider that this justifies the supposition of Plimmer, Rosedale and Raymond (*Biochem. J.*, 1923, **17**, 787) that "The growth of the chick in the shell to the day of hatching is one of the best indications that *C*-vitamin is not needed by the growing bird," because possibly this factor is synthesised by the chick. Similarly, their findings do not support the statement of McCollum in *The Newer Knowledge of Nutrition* (New York, 2nd revised edition, 1923, 152), that "Egg yolk is at least comparatively rich . . . in water-soluble *C*." P. H. P.

Antirachitic Value of Irradiated Cholesterol and Phytosterol. III. Evidence of Chemical Change as shown by Absorption Spectra. A. F. Hess and M. Weinstock. (*J. Biol. Cheml.* 1925, **64**, 193–201.)—Since, under any fixed set of conditions the absorption spectrum of a substance is constant and reproducible, absorption spectrum measurements may be employed as evidence of chemical change. By means of spectrum absorption tests a chemical change was demonstrated in cholesterol which had been endowed with antirachitic potency by ultra-violet irradiation. This activated cholesterol absorbs ultra-violet radiations to a less degree than does ordinary cholesterol, an effect which is intensified with increasing degrees of irradiation. Prolonged irradiation by means of the mercury vapour lamp leads to an inactivation of cholesterol instead of an activation. After irradiation for many hours the activated cholesterol becomes less transparent than even non-irradiated cholesterol, and is a comparatively opaque product of a yellowish colour with a lowered melting point. The absorption spectra of cholesterol is not altered by visible light, radiant heat and Röntgen rays, Neither dihydrocholesterol nor dihydrophytosterol undergoes a spectral change on irradiation. When activated cholesterol is kept in a watery suspension or in a dry state its spectral transmission becomes gradually diminished until it reaches a point where it transmits less than it did originally. These spectrographic results were tested and confirmed quantitatively by means of a thermo-pile galvanometer set. Figures of the spectra obtained are given. P. H. P.

Antirachitic Value of Irradiated Cholesterol and Phytosterol. II. Further Evidence of Change in Biological Activity. A. F. Hess and M. Weinstock. (*J. Biol. Chem.*, 1925, **64**, 181–191.)—Dry milk, flour and spinach can be rendered antirachitic by radiations from the quartz mercury vapour lamp. This potency was maintained by the spinach after it had been boiled for ½ hour. Preliminary tests have shown that irradiated vegetables and dry milk possess curative value in the rickets of infants. Oleic acid and egg phosphatide cannot be activated by the radiations. Irradiated cholesterol was found to prevent rickets in rats when given subcutaneously. A series of tests with selective filters showed that the radiations which render cholesterol active biologically are ultra-violet radiations similar in their wave-lengths to those which have been found to protect animals against rickets when they are directly exposed to the rays. Irradiated cholesterol is effective in preventing the rickets brought about by a diet either low in phosphorus or low in calcium. Skin which ordinarily possesses no antirachitic potency develops this quality after irradiation with ultra-violet rays. Rats were fed with samples. The saturated reduction products of cholesterol and phytosterol (dihydrocholesterol and dihydrophytosterol) were not activated by irradiation, and did not acquire antirachitic properties. It seemed as if the ultra-violet rays acted at the site of the double bond in the unsaturated cholesterol. Two other unsaturated compounds, however, cymene and citronella oil, did not acquire antirachitic properties on irradiation. P. H. P.

The Combination of Phenol Red and Proteins. A. Grollman. (*J. Biol. Chem.*, 1925, **64**, 141–160.)—The purpose of this investigation was to study the combination of phenolsulphonephthalein (phenol red) and the blood proteins. The elimination of this dye by the kidney has been extensively studied and used as a means of throwing light on the mechanism of renal function and, hence, a knowledge of its combinations with proteins seemed pre-requisite for a complete exploitation of this mode of attack of the problem. The adsorption of phenol red on blood charcoal and a number of proteins has been quantitatively studied. Adsorption curves for blood serum and hydrophilic systems are given. The P_H effect on the adsorption by these substances has been studied, and the P_H effect on adsorption by proteins is discussed and compared with the results obtained on charcoal. Adsorption by blood sera of various animal species, *viz.* rabbit, dog, pig, hen, duck, and frog, has been determined and compared. Indications are given of the applications of the results to studies in renal function.
P. H. P.

Modification of Bloor's Method for the Determination of Cholesterol in Whole Blood or Blood Serum. G. E. Sackett. (*J. Biol. Chem.*, 1925, **64**, 203–205.)—A modification of the method of Bloor (*J. Biol. Chem.*, 1916, **24**, 227) is described for the determination of cholesterol in blood; it takes less time, requires less blood, less alcohol and ether for precipitation of the protein, and eliminates the brown colour which may develop. The method is as follows:—Put 9 c.c. of alcohol in a 15 c.c. graduated centrifuge tube, add 3 c.c. of ether, mix

by inverting, run in 0·2 c.c. of whole blood or plasma, cork tightly and shake vigorously for 1 minute. Let lie horizontally with the sediment evenly distributed along the tube for ½ hour, rapidly centrifuge for 3 minutes and decant into a beaker. Evaporate to dryness on a water bath. Extract the cholesterol twice for about 2 minutes with 2 to 2·5 c.c. of chloroform and decant into a 10 c.c. glass-stoppered graduated cylinder. Let cool and make up to 5 c.c. Measure 5 c.c. of a standard cholesterol solution in chloroform (containing 0·4 mgrm. of cholesterol) into a similar 10 c.c. cylinder. Add to each solution 2 c.c. of acetic anhydride and 0·1 c.c. of concentrated sulphuric acid. Mix by inverting several times, leave in dark for 10 minutes, then transfer to the colorimeter cups and compare as usual, setting the standard at 12 or 15 mm.

$$\text{Calculations:—} \frac{\text{Reading of Standard}}{\text{Reading of Unknown}} \times 200 = \text{mgrm. per 100 c.c. of blood.}$$

A table shows results found by the author's method and that of Bloor. P. H. P.

Determination of Small Amounts of Protein Nitrogen. E. R. Main and A. P. Locke. (*J. Biol. Chem.*, 1925, **64**, 75–80.)—A spectrometric extension of the method of Folin and Denis (*J. Biol. Chem.*, 1916, **26**, 473) for the determination of small quantities of ammonia nitrogen is proposed. The Keuffel and Esser colour analyser was used by the writers. Through it, parallel beams of monochromatic light reach the eye; one has passed through the Nesslerised solution, and the other through distilled water. The intensity of the beam from the distilled water can be diminished to a value exactly equal to that coming from the unknown solution. This transmission value is recorded and subtracted from a value obtained when the Nesslerised sample is replaced by a Nesslerised blank solution. The difference represents light absorbed by the mercury-ammonium complex, and is proportional to the concentration of ammonia in the sample. The light absorption of a series of Nesslerised solutions of ammonium sulphate was determined at wave-lengths: 500, 520, 540, 560, 580, and 600 *mu*. The values are recorded in a figure and a table. Digestion and Nesslerisation are carried out as suggested by Folin and Denis. The Nesslerised solution is centrifuged for 10 minutes, decanted into a 10 cm. colour tube, and the colour absorption read at once as indicated. The absorption value is converted to a nitrogen value by reference to the given table. Samples used should not contain more than 0·02 to 0·03 mgrm. of nitrogen per 100 c.c., and the digestion mixture should be diluted to 6 c.c. before evaporation and digestion are begun. This method permits the use of samples containing as little as 0·005 mgrm. of protein nitrogen with fair accuracy and with great facility, and is useful when only minute quantities of nitrogenous material are available for analysis. P. H. P.

Indole Content of Canned Crustacea. D. B. Dill and P. B. Clark. (*J. Assoc. Off. Agric. Chem.*, 1925, **8**, 449–451.)—The indole often found in canned crustacean meat appears to be derived from the alimentary tract, whence it escapes into the flesh during cooking. T. H. P.

Electrolytic Modification of the Gutzeit Method for the Determination of Arsenic in Body Tissues. W. E. Lawson and W. O. Scott. (*J. Biol. Chem.*, 1925, **64**, 23–28.)—Modifications in the electrolytic Gutzeit method, as described by Klein (*J. Assn. Off. Agric. Chem.*, 1920, **3**, 512), for the determination of arsenic, are proposed, which will do away with certain objections to the zinc-acid method of producing hydrogen for this determination. The apparatus required, and how it should be set up, are carefully described. To 10 grm. of the tissue to be tested, finely chopped and in a 500 c.c. Pyrex Kjeldahl flask, are added 20 c.c. of arsenic-free sulphuric acid, about 5 grm. of potassium sulphate and a crystal of copper sulphate. This is heated cautiously until the material no longer foams, then more strongly until the solution is colourless. More acid must be added occasionally. The solution is cooled, diluted in a volumetric flask to 100 c.c., and 20 c.c. are pipetted into a 100 c.c. Erlenmeyer flask. From 0·5 to 1 c.c. of stannous chloride solution (80 grms. of stannous chloride in 100 c.c. of water to which has been added 5 c.c. of arsenic-free hydrochloric acid) is then added and the mixture heated to boiling. After cooling the flask, the contents are diluted to 50 c.c. and poured into the porous cup. Seventy-five c.c. of 4 per cent. sulphuric acid are pipetted into the beaker outside of the cup, the stopper is replaced, the whole apparatus standing in a cooling bath, and the solution is electrolysed with a current of 0·9 ampère at 5 volts, over a period of 1 hour. The cathode is of pure sheet lead and the anode is of platinum foil. The length of stain on the mercuric bromide paper is compared with standards made with pure arsenious oxide, and the amount of arsenic per grm. is determined from the aliquot part taken for analysis. The method of preparation of the standards, lead acetate paper and mercuric bromide paper is given. A table shows the accuracy obtained when following the procedure and using the apparatus recommended. The addition of stannous chloride to the solution is required in order to reduce all the arsenic to the trivalent form. Oxidation of part of the arsenic takes place when the tissue is destroyed with sulphuric acid and arsenic salts are not reduced to arsine by nascent hydrogen. P. H. P.

Determination of Blood Sugar. S. R. Benedict. (*J. Biol. Chem.*, 1925, **64**, 207–213.)—Known methods for the determination of blood sugar are discussed, and attention is directed to the need for an accurate measurement of the actual glucose content of the blood. The author has devised a method which is based on the Folin-Wu method, but yields results about 20 per cent. lower. It indicates that the normal blood sugar content averages about 75 mgrms. per 100 c.c., but probably 60 mgrms. per 100 c.c. is the actual content. The copper reagent employed is a modification of the qualitative citrate carbonate solution, in which the concentration of citrate has been increased very materially, whilst the quantity of copper and carbonate has been reduced. A small amount of sodium bisulphite has been added to the reagent, which results in a remarkable increase in the quantity of cuprous oxide obtained from the small amount of sugar contained in the dilute blood filtrate. The sulphite has no reducing action upon the copper solution.

The tungstic-arsenic-phosphoric acid reagent (*J. Biol. Chem.*, 1922, **51**, 187), with 5 per cent. of commercial formalin is used for the development of colour by cuprous oxide. The final solutions have slightly more colour than the Folin-Wu method gives, and the colour shows little tendency to change on standing. The new process is nearly identical in quantity of reagents used and general technique with that of Folin and Wu. P. H. P.

Determination of Uric Acid in the Blood. S. R. Benedict. (*J. Biol. Chem.*, 1925, **64**, 215–219.)—Bulmer, Eagles and Hunter (*J. Biol. Chem.*, 1925, **63**, 17) published a study of the direct determination of uric acid in the blood as proposed by Benedict (*J. Biol. Chem.*, 1922, **51**, 187), stating that figures obtained by the direct method are "unreliable." In this present paper the author justifies his position. He had stated that the method could not be applied to animal bloods, and had proposed two precipitation methods for use as checks in unusual cases (*e,g.* nickel rash) when the figures for uric acid were found to be high. A small proportion of human bloods show high uric acid by the direct method, but it is a simple and safe procedure. It is still an open question whether it should be abandoned. Certain statements made by Bulmer, Eagles and Hunter are said to be incorrect. The uric acid-reacting interfering compound precipitated by the silver lactate (which they state is not precipitated by silver lactate) has been isolated within the last few months, after two years of research. Over a grm. of the compound in pure crystalline form has been obtained from the blood of one species, and the presence of the substance has been proved in the blood of every species (including human). Its composition and properties are being studied. A simple quantitative method for its determination in blood, tissues and urine has been devised, by which its distribution and variation in health and disease are being studied. P. H. P.

Toxicological, and Forensic.

Two Ptomaines met with in Toxicological Work. L. van Itallie and A. J. Steenhauer. (*J. Pharm. Chim.*, 1925, **117**, 532–535.)—A substance giving most of the reactions of veratrine was met with in the course of examination of the residue obtained by the Stas-Otto method from a mixture of uterus, blood, stomach and intestines. No red coloration, however, was obtained on warming with sulphuric acid, nor any red-violet colour with Vitali's reaction; neither could the slow muscle contraction typical of the action of veratrine, be obtained. A substance giving the reaction of, and apparently identical with, *p*-oxy-phenylethylamine (tyramine) was isolated from a sample of fat two months after death. D. G. H.

A Case of Poisoning by Nitrobenzene. R. Frossard. (*J. Pharm. Chim.*, 1925, **117**, 478–480.)—A dessert spoonful of an insecticide consisting of nitrobenzene (identified by the sp. gr., b. pt., and reduction to aniline) produced acute signs of poisoning in a child of 14. Cyanosed extremities, livid face, rapid and

thread-like pulse and convulsive movements rapidly followed violent digestive disturbances. Cyanosis improved in 24 hours, when the pulse rate was 120, temperature 38·2° C., red blood corpuscles 4,824,000, white corpuscles 178,000. Urine was dark brown, non-albuminous and contained small quantities of nitrobenzene. It was scanty in quantity, increasing in volume on the third day, and contained large proportions of bile acids and pigments. The stools contained blood, and jaundice lasted 10 days. Recovery was complete in 21 days. The toxic dose of nitrobenzene appears to be large. D. G. H.

Organic Analysis.

The Fat of Algae in relation to Petroleum. J. Marcusson. (*Chem. Zeit.*, 1925, **49**, 455–456.)—By extraction with ether and chloroform 6·8 per cent. of oil was obtained from dried green algae slime (*Microcystis flos aquae*). It had the following constants:—Acid value, 135; saponification value, 190; iodine value, 91; iodine value of fatty acids, 151; and unsaponifiable matter, 12·4 per cent. The fat is considerably hydrolysed, containing about 70 per cent. of free fatty acids, consisting of approximately equal quantities of solid and highly unsaturated liquid acids which formed octobromides; the unsaponifiable matter comprised sterol and higher alcohols. In agreement with the views of Engler, it is suggested that this oil is a possible source of natural petroleum, since the decomposition of cellulose gives rise to nascent hydrogen which, in the presence of contact substances, generates solid saturated acids and alcohols from the fatty acids present. H. E. C.

Determination of Pentosans. F. W. Klingstedt. (*Zeitsch. anal. Chem.*, 1925, **66**, 129–160.)—This paper contains a critical study of the methods of determining pentosans. It is shown that oxymethylfurfural and other compounds forming condensation products with phloroglucinol are formed by the distillation, with 12 per cent. hydrochloric acid, of hexoses and any substances, such as sucrose and various celluloses, which yield hexoses on hydrolysis. The solubility of the precipitate in alcohol is used as a basis for the approximate determination of pentosans in the presence of substances yielding oxymethylfurfural, but there is at present no satisfactory method available for separating methyl-pentosans. The undried phloroglucide of oxymethylfurfural is partly soluble in alcohol, but, when dried or precipitated hot, it is almost completely insoluble. The phloroglucide of methylfurfural is soluble in alcohol, but that of furfural is insoluble after drying. In the course of the distillation the major part of the oxymethylfurfural comes over in the early stages, and usually all is over when 360 c.c. have been collected, but too high results are always obtained, even when the phloroglucide is dried and extracted with alcohol. A fairly accurate determination can be made by distillation for only such time as is necessary for the decomposition of the pentosans. This happens with cellulosic substances when 150 to 180 c.c. have passed over. The end point may be recognised by testing with phloroglucinol and hydrochloric acid; when more than a trace of furfural is present the

precipitate is of a greenish colour. The condensation should take place at room temperature, and the dry precipitate should be extracted with alcohol.

H. E. C.

Action of Yeast on Gallotannin. M. Nierenstein, C. W. Spiers and A. C. Hadley. (*J. Amer. Chem. Soc.*, 1925, **47**, 1726.)—Under the conditions described a gallotannin is obtained free from glucose and optically inactive. A sterilised solution of 20 grms. of purified Chinese gallotannin in 500 c.c. water containing certain mineral salts was inoculated with a *Saccharomyces cerevisiae* and kept at 37° C. for 11 days. A single colony of the yeast from a malt-agar culture of this solution was inoculated into a fresh solution of the gallotannin as above, and the whole process repeated 10 times, when the yeast was found to have reached its maximum intensity. The solution was then filtered, extracted with ethyl acetate, and the extract shaken with a one per cent. solution of potassium bicarbonate saturated with carbonic acid, the aqueous and ethyl acetate layers being examined separately. Starting with 129·6 grms. of anhydrous gallotannin, the aqueous layer produced 22·1 grms. of pure gallic acid (and no *m*-digallic acid), and the ethyl acetate layer yielded 87·2 grms. of a gallotannin optically inactive in water, alcohol, acetone, and ethyl acetate, and giving no glucose on hydrolysis, whereas before fermentation it contained 8 per cent. of glucose. The formula suggested for this modified gallotannin is that of a polydigalloyl-leucodigallic acid anhydride. This does not exclude the existence of a gallotannin *glucoside*.

R. F. I.

Inorganic Analysis.

Test of Ascarite, a Carbon Dioxide Absorbent, as its own Dryer. F. W. Marsh. (*J. Assoc. Off. Agric. Chem.*, 1925, **8**, 442–444.)—Attempts to determine the amounts of carbon dioxide evolved from soils by means of ascarite, which is a special mixture of sodium hydroxide and asbestos and serves well for the determination of carbon in steels, show that this material loses appreciable proportions of moisture when passage of the gases through it is continued beyond about 5 hours.

T. H. P.

Determination of Carbon Monoxide by the Blood Method. Absorption of the Gas by Haemoglobin in Absence of Oxygen. M. Nicloux. (*Comptes Rend.*, 1925, **180**, 1750–1753.)—By slight modification of the apparatus used (*Bull. Soc. Chim.*, 1923, **33**, 818) and by adding to the 6 c.c. of 2 per cent. blood solution 2 drops of 1 per cent. saponin solution and breaking the froth at the exit of the apparatus by means of a trace of octyl alcohol, as little as 0·005 to 0·006 c.c. of carbon monoxide in air from which the oxygen has been removed may be detected by absorption of the gas in the blood and observing the absorption bands of the spectrum. Thus, if the air contains only 1 part of carbon monoxide per 100,000, 500 c.c. of the air are sufficient for the determination. The partial pressure of the carbon monoxide at such dilution appears to be far less than that indicated as necessary by the law of mass action.

T. H. P.

Gravimetric Determination of Cadmium as Ferrocyanide. G. Luff. (*Chem. Zeit.*, 1925, **49**, 513–514.)—The pure sulphate, nitrate, or chloride solution (100 c.c.) is treated at 70° to 80° C. with 10 grms. of ammonium chloride, 20 c.c. of ammonia (sp. gr. 0·92 to 0·93), and a slight excess of powdered potassium ferrocyanide. The precipitate is digested at the precipitation temperature till settled, collected next day on a Gooch crucible, washed with 2·5 per cent. ammonia water, dried at 100° C., and weighed as Cd $(NH_4)_2Fe(CN)_6$ containing 31·16 per cent. of cadmium. The results are good. W. R. S.

Comparison of the Gunning-Arnold and Winkler Boric Acid Modifications of the Kjeldahl Method for the Determination of Nitrogen. K. S. Markley and R. M. Hann. (*J. Assoc. Off. Agric. Chem.*, 1925, **8**, 455–467.)—The results obtained when boric acid solution is used for the absorption of the distilled ammonia are as accurate as those given by the ordinary Gunning-Arnold method, provided that water-cooled condensers are used and the temperature in the receiving flask does not exceed 50° C. The advantages attending the use of boric acid are: the use of standard alkali is eliminated; careful measurement of the fixing medium is unnecessary, and, if any of such medium is sucked back into the distilling flask, more boric acid may be added to the receiving flask and the distillation continued; the boric acid and ammonia solution may be titrated with standard acid in artificial light if bromophenol blue is used as indicator.

As regards the catalyst used in the digestion with acid, the use of copper sulphate and phosphoric anhydride necessitates longer digestion with certain compounds, such as alkaloids, but in other cases it acts as rapidly as mercuric oxide and potassium sulphate. T. H. P.

Determination of Vanadium in Ferrovanadium. Koch. (*Chem. Zeit.*, 1925, **49**, 479–480.)—On account of the labile nature of the oxides of vanadium, its volumetric determination in high-grade ferrovanadium (on only 0·2 grm.) is rather uncertain. The author favours the following gravimetric method. The very finely-powdered alloy (0·5 grm.) is fused in an iron crucible with 6 grms. (accurately weighed) of sodium potassium carbonate. The fused mass is dissolved in water, the residue collected and fused once more with exactly 4 grms. of the fusion mixture. The combined filtrates are evaporated to 300 c.c. and exactly neutralised with the calculated quantity of nitric acid (1:1), the quantity of alkali neutralised by the vanadic acid formed being taken into account. Neutrality is ascertained with litmus paper. The solution is heated to boiling, and the vanadium precipitated with about 50 c.c. of saturated mercurous nitrate solution. The yellow to orange-red precipitate is left to settle, collected, washed with hot dilute mercurous nitrate solution, ignited, and weighed as V_2O_5. W. R. S.

Separation of Molybdenum from Vanadium. A. E. Stoppel, C. F. Sidener, and P. H. M. P. Brinton. (*Chem. News*, 1925, **130**, 353–355.)—Precipitation of the molybdenum by hydrogen sulphide under pressure gave satisfactory results. If a solution containing molybdate and vanadate was made

ammoniacal, saturated with hydrogen sulphide, and acidified, the molybdenum results were 1 to 2 mgrms. high and the vanadium results correspondingly low. W. R. S.

Separation of Celtium (Hafnium) from Zirconium. J. Bardet and C. Toussaint. (*Comptes Rend.*, 1925, **180**, 1936–1938.)—A product rich in celtium (hafnium) is obtained by the fractional precipitation of the phosphates in strong sulphuric acid. The mixed sulphates are dissolved in the strong acid, and the solution is treated with enough 80 per cent. phosphoric acid to precipitate about one-fifth of the oxides. One and a half volumes of water are next added gradually, with vigorous stirring. After being allowed to settle overnight the clear liquor is siphoned off; the remainder is diluted with 3 volumes of water, and the precipitate collected on a filter. The filtrate is added to the siphoned liquid, and the mixture evaporated until it fumes, the treatment of the acid liquor being repeated as long as the product is richer in celtia than the original mixture. This is ascertained by observation of the arc spectrum. The precipitates are united and fused with sodium carbonate; the resulting oxides are converted into sulphates, and the fractionation is repeated. Seven consecutive precipitations gave a product containing nearly 90 per cent. of celtia from a mixture containing 2 to 3 per cent. The method is rapid, but requires large quantities of sulphuric acid, in which zirconium sulphate is sparingly soluble. W. R. S.

Physical Methods, Apparatus, etc.

New Method for the Immediate Detection and Determination of Cobalt by Spectroscopy and Chromoscopy. G. Denigès. (*Comptes Rend.*, 1925, **180**, 1748–1750.)—Addition of 0·1 c.c. of a solution containing 2 grms. of cobalt per litre to 5 c.c. of hydrochloric acid in a test-tube, 15 to 16 mm. in diameter, results in the appearance of a blue coloration which shows characteristic absorption bands when examined by a direct-vision spectroscope; the blue coloration is still perceptible with 0·005 mgrm. of cobalt per c.c. of the acid liquid, but clear vision of the spectrum requires at least 0·02 mgrm. per c.c. By means of standard tubes the amount of cobalt present may be determined in this way. In presence of cupric or ferric ions, which form yellow solutions in hydrochloric acid, the cobalt spectrum is still clearly visible, but the true colour of the cobalt solution is seen only when the copper and iron are reduced to the cuprous and ferrous state by means of stannous chloride. The reaction is not disturbed by the presence of a quantity of nickel 300 times as great as that of the cobalt. T. H. P.

Fused Bisulphate as a Medium for High Temperature Baths. E. Benesch. (*Chem. Zeit.*, 1925, **49**, 509.)—Sodium bisulphate is recommended as a medium for high temperature baths. It is best to prepare it from Glauber's salt and sulphuric acid and drive off the water by subsequent heating. Bisulphate

does not attack glass or porcelain, and iron resists it well if there is excess of normal sulphate. The m.p., b.p., etc., of different mixtures are as under:—

No.	H_2SO_4 Per Cent.	Na_2SO_4 Per Cent.	H_2O Per Cent.	Softens at °C.	Fluid at °C.	Boils (under normal pressure) at °C.
1.	46·4	49·2	4·2	110	145	210
2.	46·5	52·6	0·7	110	156	210
3.	46·3	50·7	2·8	110	140	215
4.	51·8	45·0	3·0	100	145	204
5.	55·2	43·9	0·7	85	125	192
6.	59·0	33·3	7·5	80	105	192
7.	43·5	46·6	9·7	60	95	172
8.	38·7	42·6	18·5	46	56	132
9.	35·5	40·8	23·5	30	40	110
10.	42·2	51·1	6·0	150	170	210
11.	39·4	58·2	1·4	140	175	205
12.	34·3	63·4	0·8	148	180	200
13.	35·7	62·2	1·9	134	182	198

H. E. C.

Generation of Hydrogen Sulphide. Henwood, Garey, Goldberg, and Field. (*J. Franklin Inst.*, April, 1925; *Chem. Trade J.*, 1925, **76**, 644.)—A forgotten method of generating hydrogen sulphide for laboratory purposes has been revived at the Central High School, Pennsylvania. It is based on the interaction of sulphur and paraffin wax, a charge of about 10 grms. yielding about 2000 c.c. of gas. The mixture, which is conveniently incorporated with finely divided ignited asbestos, is heated over a very small Bunsen flame to start the reaction, and the evolution of gas stops within one minute after removal of the flame. The gas is very pure, being free from hydrogen and the spray from metallic salt solutions, so that it does not require washing. The generating apparatus consists of a test tube fitted with a device carrying a delivery tube.

Reviews.

L'Hydrogène et Les Gaz Nobles. By J. J. van Laar. Pp. 79. Leyden, Holland: A. W. Sijthoff. Price 3 G.

This little volume is the first of a series of monographs on Theoretical and Practical Chemistry issued under the auspices of the Société Chimique Néerlandaise, written by authors distinguished in connection with the particular branch treated. It consists of seven chapters dealing respectively with hydrogen, helium, neon, argon, krypton, xenon, and niton, together with an appendix including matter published since 1922. In each chapter is set out a calculation of the atomic weight, normal volume, and other physical constants of these elements, based on the latest

researches on the rare gases with which the name of the author is so closely associated. The treatment is in a simple, though mathematical, style which can be easily followed, and is calculated to convey to the reader a concise summary of the present state of knowledge of these elements.

In these days, when chemical journals are so numerous and voluminous, monographs, such as this, which present a special branch in a readily digestible form are increasingly valuable. The present volume will be sure of a welcome from advanced students and all physical chemists.

H. E. Cox.

THE CHEMISTS' YEAR BOOK, 1925. Edited by F. W. ATACK, D.Sc., F.I.C. Tenth Edition. Pp. 1166 and Index. Manchester: Sherratt & Hughes. Price 21s.

The task of reviewing this volume is rather like that of reviewing an Encyclopædia; it is beyond the compass of one individual accurately to appraise the quality of each section. Such a wide range of data is presented that it is not easy to name an important item which is not included, the only thing necessary being to know just where to look for it, and, as the Index is good, this task is not difficult.

The present volume shows a small increase on that of last year (*cf.* ANALYST, 1924, **49**, 306); the principal alterations noted are some re-arrangements and the re-writing and extension of the tables on the general properties of organic compounds by Dr. E. Hope; this section now provides a full and useful list.

The larger portion of the volume consists of sections on the analysis of different substances; as might be expected, they vary in merit. Perusal of these raises the question whether or not the average chemist would turn to a book of this kind for details of analytical methods, since these sections are necessarily of the nature of abstracts. To the reviewer it seems more reasonable to turn to text books on general analysis or on a special branch. In the absence of such books one usually consults Thorpe's Dictionary or Lunge's "Technical Methods."

There is room for more numerical tables, even if it involves the exclusion of special analytical methods. For instance, one looks in vain for certain Imperial weights and measures, such as Troy weight, or the number of grains in an ounce (480 or 437½). Although standard soap solution is not always reliable for the estimation of hardness in water, a table of equivalents of Clark's soap solution is so useful that it should be given.

The fact that this is the Tenth Edition speaks for the general excellence of the volume, and this edition is an improvement on the last.

H. E. Cox.

AN INTRODUCTION TO THE LITERATURE OF CHEMISTRY. By F. A. MASON, M.A., Ph.D., F.I.C. Pp. 41. Oxford: Clarendon Press. 1925. Price 2s. net.

This useful little pamphlet, which is specially intended for senior students and research chemists, is essentially a guide to the use of chemical literature, and is thus intended to form a link between the theory and practice of the science. The first

part contains a classified list of works, beginning with general dictionaries and encyclopaedias in English, French and German, with short summaries of their scope. This is followed by a list of most of the leading European and American chemical journals. Section 3 gives particulars of journals that publish abstracts, and Section 4 gives a concisely summarised list of text books and special works of reference, classified under appropriate headings. The section on Technical Chemistry admittedly makes no pretence to completeness, except in the case of Dye Chemistry.

The second part gives practical hints on the searching of literature, including patent specifications, whether for the purpose of identifying a substance of unknown composition, or for solving a technical problem, or for discovering whether an investigation has already been forestalled, and it ends with helpful suggestions for making and filing abstracts of papers on special subjects.

So far as it goes, the scheme has been excellently carried out, but on the technical side, at all events, it does not go far enough, for, apart from dyes, only two and a half pages are given to that part of the subject. The value of the book would be greatly enhanced if more of the important chemical industries were treated as fully as the dyestuffs.

In some of his references the compiler is not quite up to date. For instance, the student is referred to the 1916 edition of Treadwell's *Analytical Chemistry*, and there is no mention of the new edition of Allen's *Commercial Organic Analysis*.

Again, there are references to ephemeral trade journals, but apparently the compiler has not yet heard of THE ANALYST. This year our Society is celebrating its jubilee, and it is a chastening thought that the existence of its journal, which for fifty years has circulated all over the world, and is included as an authority in the International Critical Tables, still remains unknown within the University of Oxford, notwithstanding the fact that one of our former Presidents held a professorial post in that University. EDITOR.

THE LABORATORY BOOK OF DAIRY ANALYSIS. By H. DROOP RICHMOND, F.I.C. Third Edition revised. London: Chas. Griffin & Co., Ltd. 1925. Price 5s. net.

The handy little laboratory book compiled by H. Droop Richmond has celebrated its 20th year of usefulness by a third edition, augmented and re-indexed by the author.

Tables of logarithms and antilogarithms have been included, and some new material has been added to bring the book up to date.

In Table I. the mean percentage composition of morning's and evening's milk is given for each month of the year over a period of 20 years, but a short statement as to the source of the milk would have added to the value of the table.

The reduced percentage of fat in separated milk and butter milk given in Table II., compared with figures in previous editions, reveals improvement in methods of dairying.

The gravimetric method for lime in milk on page 15 might well have given place to the volumetric method now more generally used.

As is fitting in such a book, the Gerber test is described at length, but the method of reading recommended would, in the experience of the writer, give slightly low results with the types of centrifuge in common use.

Mention is made of the fact that the test-bottles can be checked at the National Physical Laboratory; the same observation might with advantage have been extended to the 11 c.c. pipettes.

The method of measuring cream with a pipette is still suggested, but Table III. for calculating the fat in cream has been considerably altered in this edition, the figures being as much as 2 per cent. higher in diluted cream, reading 8·5 per cent. on the bottle.

Obsolete methods in which alkaline solutions have been recommended are still mentioned, but no reference is made to their modern prototype, the "Hoyberg" method. The Kjeldahl apparatus shown in Fig. 20 might well have been brought up to date.

A new feature is a brief description of a freezing point determination; this occupies only just over a page, and has no illustration of the apparatus required. In view of the importance of the determination of the depression of the freezing point in milk, a more detailed description would have been welcome. The caution that "the method requires some little skill and practice" is well founded.

As a convenient list of working directions for the analysis of milk and dairy products, this little book can be recommended, but it should not be used as a text book for students, as it is not written from the educational point of view.

JOHN GOLDING.

PRACTICAL CHEMISTRY BY MICRO-METHODS. By E. C. GREY, F.I.C., M.R.C.S. First Edition. Pp. ix. + 124. Cambridge: W. Heffer & Sons. 1925. Price 4s. 6d. net.

The publication of new instructional books for the use of the elementary student still continues at frequent intervals, but it is long since a volume introducing such admirable innovations as the one under review has appeared. The author indicates in the preface the advantages of micro-methods over the usual "test-tubing" from the point of view of economy in chemicals, apparatus and time, and, in addition, might have emphasised the extreme value of such methods in subsequent work when but small amounts of material are available.

The subject matter is suitable for a first year's course in practical chemistry, and includes the work required for the Conjoint Boards of the Royal Colleges of Physicians and Surgeons, but the methods of identification adopted for the metals are unlikely to find favour with some examiners who in the past have been known to refuse a "pass" to an ingenious student, who, although identifying his unknown salt correctly, had done so by methods other than those involving the use

of the usual group reagents and separation methods. The text serves as an admirable introduction not only to qualitative micro-analysis of simple salts and mixtures, but also to practical physical chemistry, volumetric analysis (involving acidimetry, oxidation, reduction and precipitation methods) and the detection of elements and acid radicles in organic compounds.

Some nineteen illustrations of apparatus, etc., are provided, the later ones being incorrectly numbered, but there is no obvious reason why Fig. 15, page 74, should be again reproduced on page 96. In addition, two plates are included, the coloured one illustrating the reactions obtained by ammonium carbonate, potassium iodide and ammonium sulphide with solutions of the salts of eighteen metals. This feature will be of great value to the student, since the colours are in close agreement with those obtained in the actual experiments. In this connection it may be pointed out that, whilst the colour of silver iodide is correctly rendered as yellow, it is stated to be *white* in the text on pages 63 and 65.

The subject-matter is both lucid and easily legible, even in the smaller type, and there are few serious mis-statements,but there is some evidence of hasty proof reading, and a few of the theoretical explanations will hardly find favour with all teachers. Among the loose statements, which, for the benefit of the student (who, by the way, is frequently a severe critic of his text books), should certainly be made clearer, are the following: "a mixture of sodium and ammonium chloride," on page 29, when obviously a mixture of the two chlorides is intended; "action of sodium hydroxide and carbonate," on p. 59, where no indication is given as to whether these are to be used separately or in conjunction; "a decinormal solution of a salt is of the order of 1 per cent.," on p. 75, from which the student will erroneously deduce that any decinormal salt solution is approximately of the concentration given; and on p. 84, a reference is made to "Ex. 4," but the actual experiment is indicated by (*d*). Equations throughout the volume are accurately expressed, and, with two exceptions, formulæ are correctly rendered, but we find on p. 19 lithium chloride given as $LiCl_2$, and on p. 58 chlorine peroxide as Cl_2O. Typographical errors are rare, but in some cases the O in the formulæ of carbonates is printed in small type instead of capitals.

In comparison with the general run of indexes the one provided in this volume is fair, but at the present time a really satisfactory and reliable index is a *rara avis*. The present example contains some items indexed under the wrong letter, thus "Silicic acid" occurs with "Boric acid"; the "charcoal block or match test" under "Micro"; "Vapour pressure" under "Lowering of the vapour pressure," etc. Six page numbers are incorrectly given, and at least twelve references in the text are entirely omitted from this index.

Such defects detract greatly from the value of the book as a treatise for the use of students, and careful revision should be made at the earliest opportunity in order that the volume may be ready for the extensive demand which it certainly deserves in virtue of its many merits and its low price.

T. J. WARD.