

THE ANALYST.

OBITUARY.

SIDNEY HARVEY.

SIDNEY HARVEY was born at Diss, Norfolk, on October 28, 1834. He was educated at a private school at Woolwich and at King's College School, London. Articled at an early age to an operative chemist, he studied chemistry during 1854-5 at the Royal College of Chemistry, and later at the Royal School of Mines. His liking for analytical chemistry was largely due to the influence of A. W. Hofmann, who was Professor at the Royal College of Chemistry at the time, and of whom Harvey always spoke with a regard that was almost reverence.

After some years' experience as assistant in several businesses in London, he took over an old-established pharmacy in the High Street, Canterbury, in 1861, and passed his examination as a Pharmaceutical Chemist in 1864.

About 1865 he began to devote a good deal of his time to analytical work, which branch of his business so increased that he was well known as an analyst in East Kent when the Food and Drugs Act of 1875 came into operation. He was immediately appointed Public Analyst for the City of Canterbury, and soon afterwards for the Boroughs of Dover, Folkestone, Margate, and Ramsgate. He relinquished his pharmaceutical chemist's business in 1893, removing his laboratory to Watling Street, where he practised as an analytical chemist till his retirement in 1915.

He was elected a member of the Society of Public Analysts in 1879, and served on the Council, either as ordinary member or Vice-President, from 1886 to 1899, and again as Vice-President in 1903-4. Seven papers stand to his name in the volumes of the ANALYST. He attended the meetings frequently until about twelve years ago, when advancing age made travelling difficult, and he retained his interest in the proceedings of the Society almost to the end of his life.

Nothing gave him more pleasure in his later years than to entertain any of his professional brethren, who might be visiting Canterbury, at the old-fashioned house, part of a manor house dating to Stuart times, that he had made his home.

Somewhat reserved and retiring in disposition, Sidney Harvey was a man of a deeply religious nature and a staunch and loyal friend. He was a wide reader, and was possessed of a singularly retentive memory. He had many interests apart from chemistry, but geology and the antiquities of Canterbury and its neighbourhood fascinated him most.

While never a robust man, he had scarcely known serious illness, but after 1915 he gradually failed, and for the past three years had been unable to leave his room. He died on March 27, 1919, leaving a widow, one son, and five daughters.

ERNEST M. HAWKINS.

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

ORDINARY MEETING, MAY 7, 1919.

HELD at the Chemical Society's Rooms, Burlington House. Dr. S. Rideal, F.I.C., President, in the chair.

Certificates were read for the first time in favour of Messrs. Andrew More, F.I.C., John Haworth, F.I.C., and H. T. Lee, M.Sc., A.I.C.

Certificates were read for the second time in favour of Messrs. Charles Frederick Lee Barber, A.I.C., George Stanley Withers Marlow, B.Sc., F.I.C., and Frederick William Read.

The following were elected Members of the Society : Messrs. Madanlal Jekisandas Gajjar and James Sorley, F.I.C.

The following papers were read : "The Analysis of Mercury and Zinc Cyanide and of Double Cyanide Gauze," by T. F. Harvey, F.I.C., and F. Mackley ; "The Estimation of Small Quantities of Antimony," by W. Beam, M.D., F.I.C., and G. A. Freak, B.Sc., F.I.C. ; "Studies in Steam Distillation: Part VI.," by H. Droop Richmond, F.I.C. ; "The Detection of Cocaine, Heroine, and Veronal in Viscera," by P. A. Ellis Richards, F.I.C.

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THE DETECTION AND ESTIMATION OF COCAINE, HEROINE, AND VERONAL IN VISCERA.

By P. A. ELLIS RICHARDS, F.I.C.

(Read at the Meeting, May 7, 1919.)

THE viscera usually examined in cases of suspected narcotic poisoning are the liver, spleen, kidney, brain, stomach, and intestines, together with the contents of the last two, the urine, and, occasionally, the blood. In cases where a portion only of any organ, such as the liver, is submitted, the total weight of the latter should be ascertained from the pathologist carrying out the post-mortem examination, in order that in the event of a poison being found the quantity present in the whole organ may be calculated. The same remark naturally applies to the urine when a portion only has been reserved for analysis.

The various organs and fluids are weighed, and the former reduced to a suitable state of sub-division. This is best effected, after a little preliminary dissection, by passing the material twice through a mincing-machine, when aliquot portions can be taken for analysis. At this stage it should not be forgotten that, although medical

evidence may have suggested death from a narcotic, the analyst in most cases must satisfy himself that no other poison is present.

A weighed portion of the material, acidulated with tartaric acid, should in the first place be distilled in a current of steam and the distillate reserved for examination with a view to the detection of volatile poisons—*e.g.*, chloral, chloroform, etc.

A further weighed quantity of viscera, rendered acid with acetic acid, is warmed with double its volume of alcohol (90 to 95 per cent.), allowed to stand for some hours, the alcohol decanted, and the residue again extracted with the same solvent. The various portions of alcohol are mixed and filtered through cloth, using the filter pump if necessary, concentrated, and again filtered—this time through paper. If the solution be still too deeply coloured, lead acetate may be used as a clearing agent, the liquid being again raised to the boiling-point, filtered, and the lead removed by hydrogen sulphide. The filtrate, after concentration to small bulk at a low temperature, is reserved for the extraction of alkaloids, veronal, etc.

The urine, rendered faintly acid with acetic acid, is raised to the boiling-point, allowed to simmer, small portions of finely powdered lead acetate being added from time to time until precipitation ceases. After filtration and removal of the lead by hydrogen sulphide the liquid is concentrated to small bulk and reserved for examination as before.

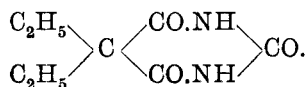
Each of the various concentrates acidulated with acetic acid is extracted in a separating funnel with successive small quantities of ether. The various portions of solvent are mixed, the ether evaporated, the residue, if any, dried in the water-oven, weighed and examined for veronal, sulphonal, trional, etc.

The aqueous solution, after the ethereal extraction just described, is rendered alkaline with ammonia and shaken with chloroform, this operation being repeated three times, the various portions of the solvent mixed and shaken with two successive portions (10 c.c.) of $\frac{N}{10}$ hydrochloric acid. The aqueous solution, after rendering alkaline with ammonia, is re-extracted with chloroform. The residue obtained after distilling off the chloroform may, where cocaine is suspected, be re-extracted with benzene, in which this alkaloid is distinctly soluble.

The final residues obtained are dried, weighed, redissolved in suitable solvents, and aliquot portions evaporated in small flat-bottomed porcelain basins. One residue in each case is treated with a few drops of a 2 per cent. acetic acid solution and the special alkaloidal group-tests applied, the precipitates, if any, being reserved for microscopical examination and comparison with those obtained from known alkaloids.

The following notes on the identification of certain narcotics may be of interest:

Veronal (B.P. Barbitone, Diethyl barbituric acid),



This is a white crystalline substance with a slightly bitter taste, sparingly soluble in water (1 part in 150), and, when pure, has a melting-point of 191° C. After extraction from viscera the melting-point is frequently slightly lower, about 186° C.

The medicinal dose given in the B.P. is 5 to 10 grains, whilst it is suggested by W. H. Willcox that 50 grains may be regarded as the minimum fatal dose for a healthy adult.

In cases of death from veronal poisoning the organs frequently contain a fair amount of the substance, and, as the latter is excreted by the kidneys, one usually finds a distinct proportion in the urine. Sometimes, however, a considerable period elapses between the taking of the fatal dose and the resulting death, and under such circumstances much of the drug may have been eliminated.

Veronal dissolves very readily in alkaline solutions and is easily extracted from an acid solution by means of ether. The crystalline character of the ethereal extract is of help in its detection, as also is the melting-point and the fact that it sublimes completely if carefully heated. The crystalline sublimate, if any, yielded by the extract may be compared microscopically with that obtained from actual veronal. Confirmation of the presence of veronal may be obtained by adding a small portion of the extract to a little fused potassium hydroxide, when ammonia should be evolved and the residue yield effervescence of carbon dioxide and a curious fatty odour on treatment with dilute sulphuric acid. The Prussian-blue test with ferrous sulphate and the pink colour with copper sulphate mentioned in the B.P. are not so satisfactory in toxicological tests. Millon's reagent (mercury dissolved in dilute nitric acid) gives a white gelatinous precipitate which dissolves in excess of the reagent.

Ammoniacal copper sulphate + veronal evaporated to dryness on a microscope slide gives pink to violet crystals that are fairly definite when compared with control slides (Tunmann, *Apoth. Zeit.*, 1917, **32**, 289-299; and *ANALYST*, 1918, **43**, 67). I find that a solution of veronal in dilute ammonia, when evaporated, yields long crystals with serrated edges, markedly differing from those yielded by trional and sulphonal under the same conditions.

Sulphonal, $(\text{CH}_3)_2\text{C}(\text{C}_2\text{H}_5\text{SO}_2)_2$;

Trional, $\text{CH}_3\text{C}_2\text{H}_5\text{C}(\text{C}_2\text{H}_5\text{SO}_2)_2$;

Tetronal, $(\text{C}_2\text{H}_5)_2\text{C}(\text{C}_2\text{H}_5\text{SO}_2)_2$.

These are all white crystalline substances of similar type and reactions, and are much less toxic than veronal, being frequently employed as hypnotics. The B.P. gives the doses as from 10 to 20 grains for trional and tetronal, and from 10 to 30 grains for sulphonal, whilst considerably larger amounts would be required to produce fatal results.

They are best identified by their melting-points, these being: sulphonal, 125° C.; trional, 75° C.; and tetronal, 85° C. As a class these sulphones are sparingly soluble in water, but much more so in alcohol. When heated strongly they yield sulphur dioxide, with fused potassium cyanide they give an odour of mercaptan, whilst with fused sodium acetate they evolve hydrogen sulphide.

Heroine, di-acetyl morphine, $\text{C}_{21}\text{N}_2\text{NO}_5$, formed by the action of acetic anhydride on morphine.

Originally introduced as a substitute for morphine, it has recently gained notoriety on account of its use by certain drug-takers. The hydrochloride of the alkaloid is the form in which it is most frequently employed, and, like the corresponding cocaine salt, it is usually taken as a snuff. Consequently, where this poison or

cocaine is suspected, swabs should be taken from the mucous membrane of the nose, and submitted to chemical examination. One-sixth of a grain is stated to have produced fatal results, and one-thirtieth of a grain has produced dangerous symptoms.

A fatal case of poisoning, described by W. R. Boyd in the *Medical Journal of Australia*, is quoted in the *Lancet* of May 3rd of this year. In this case, 6.97 grains of heroine were administered in mistake for veronal, death ensuing seventy hours later, only $\frac{1}{84}$ grain of morphine being found in the organs.

Heroine hydrochloride is a white crystalline substance with a bitter taste, easily soluble in water, and having a melting-point of 230° C., differing in this respect from morphine hydrochloride, which chars without melting.

It resembles morphine in its reactions with Frohde's solution, ferric chloride, and also with iodic acid and starch, but the colours produced are slightly slower in appearing. A 2 per cent. solution of hexamethylene tetramine in strong sulphuric acid gives a fine purple colour very slowly turning blue, but in this instance also a very similar reaction is yielded by morphine and its salts. The sodium phospho-molybdate precipitate dissolves in ammonia to a blue colour practically identical with that given by salts of morphine under similar conditions.

Cocaine, $C_{17}H_{21}NO_4$.—The hydrochloride, the usual form in which this alkaloid is found in commerce, occurs in white prismatic crystals, strikingly soluble in water (2 in 1) with a melting-point of 186° C.

It is employed medicinally as a local anæsthetic in minor operations of the eye, throat, and mouth, but it has recently come into prominence from its illegitimate employment as a snuff. Its physiological effect appears at first to be stimulating, but this is sooner or later followed by lassitude and, in excessive doses, by a state of coma. It causes dilatation of the pupil and usually disturbances of the nervous system. The body soon becomes tolerant to the drug, and, in the case of habitual takers, little or none may be found in the organs after death. In this respect it would appear to resemble morphine (*cf.* Webster, *ANALYST*, 1917, **42**, 226).

In addition to its distinctive melting-point of 98° C., this alkaloid is characterised by the following reactions:

It possesses a bitter taste followed by a somewhat prolonged numbness of the tongue. When evaporated to dryness with a few drops of nitric acid, and the residue moistened with a little alcoholic solution of caustic potash, it yields the characteristic odour of benzoic ether (meadowsweet).

Pisani (*Rend. Soc. Chem. Ital.*, 1914, **6**, 132) states that with a 2 per cent. solution of hexamethylene tetramine in strong sulphuric acid a wine-red colour is produced, becoming more intense as the temperature rises. I am unable to confirm this, as, under the conditions specified, no reaction is obtained beyond a slight charring produced by the rise of temperature.

The resorcinol and strong sulphuric acid test proposed originally by M. Goeldner (*Zeitsch. anal. Chem.*, 1901, **40**, 820) is quite fallacious, as shown by L. A. Ryan (*J. Amer. Chem. Soc.*, 1915, **37**, 1960), the lavender-blue colour supposed to be indicative of cocaine being caused by traces of nitrous or nitric acid in the sulphuric acid employed.

Cocaine gives with a permanganate solution, under certain conditions, distinct and characteristic crystals, but special precautions are needed to get a satisfactory result. The modification of the test proposed by E. H. Hankin (*ANALYST*, 1911, **36**, 2), where the alkaloid is dissolved in a saturated alum solution and added to a dried film of potassium permanganate on a microscope slide, gives excellent results and the crystals are quite definite. The concentration of cocaine in the alum solution should not be less than 1 part in 10,000.

Although the salts of many alkaloids yield a precipitate with potassium chromate, cocaine hydrochloride gives no precipitate until after the addition of a few drops of concentrated hydrochloric acid. Morphine and heroine give no reaction with potassium chromate solution in either neutral or acid solution.

Wagner's solution (iodine in potassium iodide) throws down a brownish-red precipitate with salts of this alkaloid that appear as dark brown oily drops when examined microscopically. The same result was obtained with this reagent when cocaine hydrochloride was dissolved in saturated alum. Sodium phospho-molybdate gives a curdy yellowish-white precipitate soluble in ammonia to a very pale bluish-green solution.

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THE ESTIMATION OF SMALL QUANTITIES OF ANTIMONY.

By W. BEAM, M.D., F.I.C., AND G. A. FREAK, B.Sc., F.I.C.

(*Read at the Meeting, May 7, 1919.*)

HAVING occasion to make a series of determinations of small quantities of antimony in the urine of patients treated by intravenous injection of tartar emetic, we attempted to apply the method described by Schidrowitz and Goldsbrough (*ANALYST*, 1911, **36**, 101), which is based upon the extraction of the antimony by boiling with copper and hydrochloric acid, as in Reinsch's test, and the subsequent solution of the antimony and conversion into sulphide which is estimated colorimetrically. Schidrowitz and Goldsbrough only claim to extract from 70 to 80 per cent. of the amount originally present, but in our hands the method was not found capable of even this degree of accuracy without modification.

Their procedure is as follows :

"The copper with the antimony deposit was warmed to about 70° C. for a few minutes with 5 c.c. of a 5 per cent. potassium hydroxide solution, 10 c.c. of distilled water, and an excess of permanganate. As a rule, 1 c.c. of a permanganate solution, of which 1 c.c. was equal to 0.01 grm. iron, was sufficient. The solution was then poured off the copper, the latter washed with a little distilled water, and the solution then boiled for a few minutes and subsequently filtered from the precipitated manganese dioxide. The excess of permanganate in the filtrate was then destroyed by adding a 1 per cent. solution of tartaric acid to the boiling liquid until the latter was colourless. The solution was then made up to a known volume, an appropriate aliquot

part taken and made up to 10 c.c., after adding the requisite amount of gum solution and hydrochloric acid, after which the liquid was saturated with hydrogen sulphide, as described above."

The above method is unsound in that the antimony in the standard for comparison exists as antimonious sulphide, whereas that in the test solution is in a higher state of oxidation. This is to be expected from the treatment to which the copper strip is subjected, and is shown by the contrast in the effect of the passage of hydrogen sulphide through the test and standard liquids. In the latter case the yellow colour of the antimonious sulphide is struck at once, whereas in the former it invariably requires an appreciable time to make its appearance. The method is also objectionable inasmuch as the test solution has been found almost invariably to be opalescent, which renders comparison with the clear standard impossible. This opalescence is in part due to the separation of free sulphur resulting from the decomposition of antimonious sulphide. It has been found essential to work with absolutely limpid solutions if any high degree of accuracy is to be attained.

Reduction of the test solutions with sulphur dioxide, together with other modifications mentioned below, renders it possible to reach a very high degree of accuracy, having regard to the small quantities of antimony dealt with.

Our procedure is as follows:

A strip of copper foil of suitable size (working with quantities of 0.1 mgrm. to 1.0 mgrm. of antimony, about 1.5 cms. by 10 cms. will suffice) is made into a coil so that, after the deposition, it is ready for immersion in the alkaline permanganate solution, contained in a small beaker, without further handling. When deposition is complete the coil is taken out of the acid liquid by means of glass forceps, dropped into a large beaker filled with water, then without delay transferred to a boiling alkaline permanganate solution consisting of 15 c.c. of 1 per cent. potassium hydroxide solution and an amount of potassium permanganate solution (1 c.c. = 0.01 gm. of iron) depending on the quantity of antimony on the strip, which is approximately indicated by the appearance of the deposit. (See note below.) For deposits of less than 0.3 mgrm. of antimony, 0.5 c.c. of permanganate solution will suffice, and the boiling should be continued for not more than one minute; for quantities of 0.5 mgrm. of antimony, 1 c.c. of permanganate solution should be used, and the boiling continued for five minutes or until the solution is decolourised; for quantities of about 1 mgrm. of antimony, 2 c.c. of permanganate solution may be used, and the boiling carried out as above. With very thin deposits, if the boiling is too long continued there is a danger of solution of copper from the strip.

In all cases it is advisable to check the complete solution of the antimony by treating the copper strip with hydrochloric acid (1 : 5). If any black stain remains, the strip is quickly washed with water and given a second treatment with alkaline permanganate. The liquid is poured off the copper strip, which is washed twice with distilled water, and the solution filtered through a small thick paper filter. A sufficient quantity of dilute hydrochloric acid is then added to neutralise the alkali and to provide an excess of 0.2 c.c. of strong acid. Sulphur dioxide is then passed through the solution for about three to five minutes, and the liquid gently boiled until every trace of sulphur dioxide is eliminated. A small piece of cotton thread is useful to

regulate the boiling, and it is advisable to carry the evaporation until not more than 10 c.c. of liquid remain. The solution is then cooled, 0.5 c.c. of 10 per cent. gum solution added, the liquid made up to 25 c.c., and hydrogen sulphide passed through it until the colour is fully developed. As noted above, this takes place in a few seconds. Comparison is made with a standard solution of antimony (in the form of potassium antimonyl tartrate) treated in the same manner. Extremely consistent readings may be obtained by the use of a Dubosq colorimeter.

It is obvious that the success of the process depends, in the first place, on complete extraction of the antimony by the copper strip. It is, of course, impossible to give definite directions as to the time of boiling necessary in all cases, but the following figures will serve as a rough guide: In a solution of 50 c.c. volume containing 10 c.c. of concentrated hydrochloric acid and 0.5 mgrm. of antimony, deposition is complete in one and a quarter hours, the strip of copper being of the size given above. Deposition takes place more slowly if less acid be present.

It has been found that if the antimony-covered strip is allowed to stand before treatment with the alkaline permanganate solution, the deposit becomes progressively converted into a passive form which is less readily attacked and dissolved by the reagent. It may be reconverted into its original form by short immersion in dilute hydrochloric acid, but this is objectionable in that more or less of the antimony is lost. For this reason it is recommended that the alkaline permanganate solution should be prepared so that the coated strip may be dropped into it immediately the deposition is complete. The above condition appears to be analogous to that observed by Vaubel and Knocke (*Chem. Zeit.*, 1916, **40**, 209-210; *ANALYST*, 1916, **41**, 147), who found that antimony stains on porcelain, which, when fresh, were soluble in hypochlorite solution, became more or less insoluble after standing for twenty-four hours.

Experiments were tried with various concentrations of potassium hydroxide in the alkaline permanganate solution, with the result that 1 per cent. was found to be the most suitable. With greater concentrations there is a liability of solution of traces of copper, which is marked by the appearance of a brown tint after the passage of hydrogen sulphide. Lower concentrations fail to remove the whole of the antimony from the strip.

It will be noted that we have recommended the use of a limited amount of permanganate solution, and more especially the boiling of the liquid until it is completely decolourised. This is in order to avoid the presence of manganese chloride in the final test liquid, since it was found that the latter is more readily kept clear in its absence. Even in its presence, however, the liquid will remain limpid a sufficient length of time to enable the colour comparison to be made.

The following figures will give some indication of the accuracy which may be attained:

Antimony taken.					Antimony found.		
0.3 mgrm.	0.31 mgrm.	103	per cent.
0.5 "	0.51 "	102	"
0.5 "	0.50 "	100	"
0.5 "	0.49 "	98	"
1.0 "	1.00 "	100	"
1.0 "	1.03 "	103	"

NOTE.—As a clinical method, where great accuracy is not required, and in the absence of interfering metals, it is found that the Reinsch test is capable of giving quantitative results with fair approximation to the truth. It is only necessary to prepare a series of standard strips and to compare the appearance of the test strip, made under similar conditions as regards strength of acid and time of boiling, with these. In the case of urine, it will be found advisable to dilute with an equal bulk of water before adding the acid in order to avoid discoloration of the deposit by organic matter. The strip should be dried by pressing between filter-paper, since the stain is rapidly affected by air and moisture.

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ESTIMATION OF SMALL QUANTITIES OF LEAD IN FOODS AND SUBSTANCES CONTAINING CALCIUM PHOSPHATE.

By B. W. J. WARREN, F.I.C.

IN the B.P. method for the estimation of lead, the substance to be tested is dissolved either in water or a dilute solution of ammonia.

With foods it is necessary to first destroy the organic matter and estimate the lead in the ash. The ash can be dissolved in dilute nitric acid, and the solution rendered alkaline with ammonia, a precipitate of calcium and magnesium phosphate being obtained. If the filtrate is used for the estimation of lead (using the B.P. method) considerable quantities of lead may sometimes be overlooked, as the lead is occluded with the precipitated phosphates (or as an insoluble double phosphate of calcium and lead).

Wilkie (*J. Soc. Chem. Ind.*, 1909, 28, 636) has pointed out that ferric hydroxide will remove lead from a tartrate solution.

If calcium phosphate containing traces of lead and copper is dissolved in dilute nitric acid and ammonia be added the precipitate contains all the lead, while the copper is in solution. If this precipitated phosphate (containing lead) is dissolved in dilute nitric acid the lead can be estimated colorimetrically.

In the absence of iron, lead and copper can be easily and accurately estimated in a food. Iron, however, presents some little difficulty: if a phosphate precipitate containing lead and iron (copper being eliminated as shown above) is dissolved in dilute acetic acid the solution is turbid owing to the presence of phosphate of iron. If this precipitate is filtered off some of the lead is removed with the precipitate (with the material with which I was working about two-thirds were removed).

It is, however, possible to match the colour with the slightly turbid solution and thus estimate the lead.

The method adopted is as follows:

Ten grms. of foodstuff are incinerated in a silica dish, dissolved in a small quantity of water with the addition of 1 c.c. of nitric acid, filtered, and washed. To the filtrate, which should be colourless, a slight excess of ammonia is added, the precipitate filtered and washed well. (The filtrate can be tested for lead by B.P.

method.) The copper will be in the solution, while the lead (most, if not all) will be in the precipitate.

The precipitate is washed into a Nessler cylinder with water, 5 c.c. of dilute acetic acid are added, followed by an aqueous solution of hydrogen sulphide, and the colour matched in the usual manner.

The control solution is prepared as follows :

A quantity of dilute lead solution (B.P.) diluted with water is rendered faintly alkaline with ammonia, and to this is added 5 c.c. dilute acetic acid and finally hydrogen sulphide solution.

The following results were obtained by the above method on samples prepared by adding lead to a food containing copper and iron :

Lead added.				Lead found.
10 parts per million	8, 9 parts
20 " " "	20, 16 parts

SUTTON ROAD, SOUTHBEND.

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MILK CALCULATIONS: A CRITICISM.

By H. DROOP RICHMOND, F.I.C.

A SERIES of four papers by Leslie J. Harris have lately appeared in the ANALYST (1918, 43, 263, 435, 375, and 1919, 43); of these only the first was read before the Society, but unfortunately provoked no discussion. I have studied the series with much interest, as the formulæ deduced are admittedly based on my work, and I am pleased to find that there are new ideas in them; the more I study these papers, however, the more I am convinced that the assumptions on which they are based are not wholly true.

Mr. Harris gives as reasons for calculating his table of specific gravity factors—first, that “the Gerber method cannot be considered accurate to more than 0·1 per cent., and that it is liable to other errors (the errors enumerated)”; second, that “the estimation of total solids by direct evaporation on porcelain is rapid, gives very consistent results, and duplicates agree closely,” and he implies, without a definite claim, that the calculation of the fat from total solids and specific gravity by my formula is more accurate than a Gerber fat estimation; third, that for calculating the fat a milk scale is hardly suitable for working out a long sequence, and will be found tedious in use, and the same is true of tables worked out by me and others. (Incidentally I may mention that the footnote saying that my original table is intended for the calculation of total solids from fat is not true, as the original tables were devised to give fat from total solids.)

From the result of many thousands of careful comparisons, some of which have been published, I have no hesitation in saying—first, that the “other errors” enumerated are just those errors which prevent the Gerber method being accurate to more than 0·1 per cent. of fat; second, the calculation of fat from total solids

estimated gravimetrically involves greater inaccuracy than the estimation by the Gerber method, and that when the total solids are determined by one of the comparatively rough methods which have to be adopted when several hundred samples a week are examined, the order of accuracy is distinctly inferior to that of the Gerber method: this statement is made from a very large experience of both methods; third, while I have never had any commercial interest in the "Milk Scale," I have lately received information from one firm who makes and lists this which shows that there is a very large demand for it, and it is a distinct commercial success, and evidently it is not found tedious in use. The tables I have published vary from a very large one, in which the calculations were pushed to three places of decimals (in "Dairy Chemistry," Table CXXXII.), which I frankly admit is not suited for rapid use, to the smaller one in the same work, and those in "Dairy Analysis" (pp. 89 and 90), which are at least as convenient as the more cumbersome table of Mr. Harris, as they avoid turning over a page (a great consideration for rapid work) and do not necessitate searching about the page. Mr. Harris's idea of additive factors is new, and if the table were on one page and read directly down the page it would be admirable.

With reference to the new formula for the calculation of added water (ANALYST, 1918, 43, 345), the rule on which it is based is too arbitrary for use by the public analyst, as it involves the assumption that the limits fixed for the composition of milk depend upon each other, and I am of opinion that a public analyst using the formula might have considerable difficulty in convincing a magistrate of its accuracy. For practical use my G+F formula serves every purpose, has a scientific basis, and does not involve a laborious calculation.

Mr. Harris's third paper (ANALYST, 1918, 43, 375) gives a table and a chart which cannot be of great use to the public analyst, as he must know the actual composition of his milk, and a calculation of the fat and solids-not-fat will show at once if the milk is sufficiently below standard to warrant further determinations, while the dairy analyst will soon learn to judge infallibly by inspection.

Mr. Harris appears to have realised that his formula for added water is too long, and in his latest paper (ANALYST, 1919, 45) he has endeavoured to avoid it by a shorter calculation plus a reference to a table which may require interpolation, or by a chart and a slide rule, but objection can be taken to the basic formula, and it gives erroneous results, as may be illustrated by an exaggerated example: if to 50 grms. of milk containing 3.0 per cent. of fat and 8.5 per cent. solids-not-fat 50 grms. of fat are added, we get a milk with fat 51.5 per cent. and solids-not-fat 4.25 per cent.; on adding an equal quantity of water to this we calculate from the table that the solids-not-fat in the fat-free milk is 2.86 per cent., and, by interpolation in the table, the amount of added water is 67 per cent., which is not a percentage on the sample examined, but on a hypothetical sample free from excess of fat. A rational method of calculation is this:

$$\text{Water added} \quad \dots \quad 100 - \frac{2.125}{8.5} \times 100 = 75 \text{ per cent. ;}$$

but, as the fat is 25.75 per cent., it is evident that 25 per cent. of the apparent dilution is due to excess of fat, leaving a 50 per cent. dilution with water.

NOTES.

The Editor desires 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.

CALCULATION OF THE COMPOSITION OF THE ORIGINAL MILK
FROM THE ANALYSIS OF CHEESE.

The formulæ below may be used to calculate the fat and solids-not-fat in the original milk. (F = percentage of fat in cheese, P = percentage of proteins in cheese.)

$$\text{Fat in original milk} = \frac{100 F}{35.4 P + F} + 0.25.$$

$$\text{Solids-not-fat in original milk} = \frac{333 P}{35.4 P + F}$$

H. DROOP RICHMOND.

BOOT'S PURE DRUG CO., LTD.,
ISLAND STREET, NOTTINGHAM.

NOTE ON THE PERCENTAGE OF OIL IN MALT EXTRACT AND COD-LIVER OIL.

It is usual to express the percentage of oil obtained during analysis as percentage by weight, and the formula used by Hitchman and myself (*J. Soc. Chem. Ind.*, 1917, 36, 273) gives the percentage by weight. The percentage (15) given in the British Pharmaceutical Codex, 1911, which is the semi-official standard, is, however, a percentage by volume; and 15 per cent. by volume corresponds to about 10.4 per cent. by weight.

The relation between percentages by weight (W) and volume (V) is:

$$W = \frac{V \times \text{S.G. Oil}}{V \times \text{S.G. Oil} + (100 - V) \text{S.G. Ext.}}, \text{ and } V = \frac{W \times \text{S.G. Ext.}}{W \times \text{S.G. Ext.} + (100 - W) \text{S.G. Oil}}$$

Without any appreciable error, the S.G. of the oil may be taken as 0.927, and that of the extract as 1.410.

As the figure of the B.P.C. has also been adopted for National Health Insurance purposes, it may be taken that 15 per cent. by volume, or 10.4 (or, in round figures, 10) per cent. by weight, of cod-liver oil is the minimum standard to which malt and oil preparations, if no definite percentage is notified on the label, should conform.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Technical Coumarone Resins. J. Marcusson. (*Chem. Zeit.*, 1919, 43, 109-111; 122-123.)—Coumarone resins, which are prepared by treating solvent naphtha with sulphuric acid, are almost completely soluble in acetone, whereas coal-tar pitch, lignite pitch, and petroleum pitch, are only sparingly soluble in that solvent. They may be distinguished from the condensation products of phenols with formaldehyde by their ready solubility in petroleum spirit and by not yielding phenol when heated with soda-lime in an oil-bath at 260° C. Natural resins, again, have a higher melting-point, saponification value, and iodine value, than coumarone resins, and are also, unlike coumarone resins, optically active. About thirty different grades, classified in accordance with their colour and hardness, are on the market. The degree of hardness is determined by the softening point, samples softening above 50° C. being termed "very hard," those at 40° to 50° C., "hard," and those at 30° to 40° C., of "medium hardness." The consistence is determined by measuring the time taken by a wire nail (130 mm. long, and 23 to 24 grms. in weight) to sink vertically up to its head into the resin at 20° C. A resin which softens below 30° C., and into which the nail sinks in at least 500 seconds is regarded as "soft"; whilst one which with the nail test shows at least 100 seconds is "viscous," and below that value is "fluid." Coumarone residues are products which, when distilled in a current of steam, yield less than 35 per cent. of coumarone resin, and with which the nail test shows a value of five seconds or less. The colour is recorded on the basis of comparisons made with a solution of 1.5 gm. of potassium bichromate in 100 c.c. of 50 per cent. sulphuric acid, the resin being dissolved in benzene. Estimation of free sulphuric acid and sulpho-acids is of importance in view of the fact that some of the poorer qualities of resin corrode instead of protecting metal surfaces. In Wolff's method (*Farben Zeit.*, 1917, 919) 10 to 20 grms. of the resin are dissolved in 50 c.c. of neutral benzene or xylene, and the solution repeatedly washed with hot water. The aqueous extracts are acidified, and the sulphuric acid precipitated at 80° C. as barium sulphate. The filtrate is evaporated with a few c.c. of strong potassium hydroxide solution; the dry mass fused, cooled, and taken up with dilute hydrochloric acid. The barium sulphate precipitated from this solution corresponds to the soluble combined sulphuric acid. Three modifications of polymerised indene occur in technical coumarone resins. The first of these (m.-pt. 210° C.) is only very sparingly soluble in ether; the second (m.-pt. about 165° C.) is readily soluble in ether, but sparingly soluble in alcohol-ether; whilst the third, which melts below 100° C., is readily soluble in alcohol-ether. Three grms. of the resin are dissolved in 15 c.c. of ether, and the solution treated with 45 c.c. of 96 per cent. alcohol, which is added, drop by drop, from a burette. The beaker is chilled for half an hour in ice water, and its contents filtered with the aid of suction. The precipitate is washed with

15 c.c. of chilled 96 per cent. alcohol, the filtrate and washings evaporated, and the residue weighed. The precipitate is dried and washed with acetone, and the acetone solution evaporated to obtain the resin, whilst the insoluble asphaltic substances on the filter are dissolved in chloroform or benzene, the solution evaporated, and the residue weighed. As a rule, technical "very hard" coumarone resins contain from 52 to 53 per cent. of constituents insoluble in ether-alcohol but soluble in acetone; "hard" resins, 42 to 44 per cent.; those of "medium hardness," 32 to 34 per cent.; "soft" resins, 15 to 30 per cent.; and "fluid" resins, 5 to 7 per cent. Two kinds of oily products are also present in coumarone resins—those which are readily attacked and those not attacked by sulphuric acid. For this test 5 grms. of the resin are dissolved in 50 c.c. of carbon tetrachloride, and the solution shaken with 3 c.c. of strong sulphuric acid. After separation of the acid layer, the solution is shaken with aqueous potassium hydroxide solution and evaporated, and the residue dried and weighed. In the case of "very hard" resins the amount of resin recovered ranged from 96 to 100 per cent.; in "soft" resins 71 to 89 per cent.; and in "fluid" resins from 61 to 70 per cent. But even when a large excess of sulphuric acid was used only a portion of the oil was sulphonated and separated with the acid.

C. A. M.

Chemical Assay of Foxglove. Tschirch and Wolter. (*Schweiz. Apoth. Zeit.*, 1918, 56, 469; through *Pharm. J.*, 1919, 102, 219.)—The authors propose the following method for the chemical assay of foxglove leaves: The leaves are first exhausted with ether, by which chlorophyll, fat, and resin are removed. They are then treated according to Keller's method, but, after precipitating with lead, the glucosides are shaken out with acetone, the acetone solution being made to separate by adding sodium chloride. In this way a mixture of all the active constituents of the leaves is obtained, for which the authors propose the name of *pandigiton*. The physiological activity of the *pandigiton* obtained is, however, less than that of the corresponding quantity of the solution before its removal, although the solution left after its removal is quite inactive. Two explanations of this are possible—viz., either the separation has involved some change, or the inactive substances in the liquid exert some favourable influence. Moreover, the weight of *pandigiton* obtained is not proportional to the physiological activity of the leaves. Nevertheless, the authors claim that the acetone method is a chemical method of assay, inasmuch as by it all the active constituents, and not the digitoxin alone, are separated and weighed.

H. F. E. H.

Rancidity of Palm-Kernel and other Feeding-Cakes. J. R. Furlong. (*J. Agr. Sci.*, 1919, 9, 137-142.)—Palm kernels, palm-kernel cake, and meal contain a lipase, which in the presence of moisture and warmth acts upon the oil present, liberating fatty acids of which the volatile members have a strong sweat-like odour, and a very small amount of these acids is sufficient to impart to the cake a peculiar odour. This change does not occur if the cake is kept dry, a condition which is necessary to the preservation of all feeding-cakes. Freshly prepared palm-kernel cake has a characteristic smell, which is probably due to a trace

of these acids produced during manufacture. The fatty acids of palm-kernel oil include 13 per cent. of the volatile fatty acids—caproic, caprylic, and capric, all of which have an unpleasant odour. Of these the chief is caprylic acid, which has an intense odour of sweat. Consequently the presence of a very small amount of these volatile acids in the cake, due to a slight decomposition of the oil, apart from any production of rancidity, is sufficient to impart to the cake an unpleasant smell. Palm-kernel cake does not decompose more readily than cotton-seed, linseed, and ground-nut cakes, these latter developing more acidity under similar conditions. These cakes differ from palm-kernel cake in not yielding volatile fatty acids on decomposition. Palm-kernel cake has been kept for eighteen months and was at the end of the time in good fresh condition and showed no increase in acidity. The only precaution taken was to keep the cake dry.

H. F. E. H.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

German Seaweeds. R. Albert and M. Krause. (*Chem. Zeit.*, 1919, 43, 97-99.)—The following quantities of iodine and ash were found in various seaweeds collected around Heligoland :

Plant.	Ash. Per Cent.	Iodine in Ash. Per Cent.	Iodine in Dry Substance. Per Cent.
<i>Laminaria hyperborea</i> , roots	52.37	1.095	0.574
" " stalks	34.43	2.962	1.02
" " leaves	27.00	4.247	1.148
" <i>saccharina</i> , "	34.8	0.322	0.147
<i>Halidrys siliquosa</i>	25.68	0.977	0.229
<i>Ascophyllum nodosum</i>	21.81	0.793	0.198
<i>Himantalia lorea</i>	27.47	0.004	trace
<i>Fucus serratus</i>	17.7	0.537	0.095
<i>Laminaria digitata</i> , leaves	30.00	2.551	0.765
<i>Fucus vesiculosus</i> ,	20.40	0.187	0.038
<i>Chondrus crispus</i> , leaves	25.00	0.3	0.076
<i>Ceramium rubrum</i> , stalks	35.00	trace	trace

W. P. S.

"Perocide." F. Mach and P. Lederle. (*Chem. Zeit.*, 1919, 43, 117-118.)—A fungicide sold as "perocide" consists essentially of the sulphates of the cerite earths (cerium, lanthanum, neodymium), and is a by-product of the manufacture of incandescent gas mantles. It usually contains from 43 to 47 per cent. of cerite earth oxides. The amount of these oxides present may be approximately estimated as follows: Ten grms. of the sample are boiled with 500 c.c. of water for fifteen minutes, cooled, water is added to make the volume 500 c.c., and the solution is filtered. Fifty c.c. of the filtrate are treated with 5 c.c. of 10 per cent. (by weight) sulphuric and 5 c.c. of 5 per cent. ammonium acetate solution, heated to boiling, and

an excess of saturated oxalic acid solution is added. The mixture is again boiled, placed aside overnight, and the precipitate collected, washed with hot water, ignited, and weighed. The constituents insoluble in water are estimated by shaking 5 grms. of the sample (ground previously to pass a 2 mm. sieve) with 200 c.c. of water for one hour, filtering the mixture, washing the insoluble portion with warm water, drying it at 105° C. for one hour, and weighing. W. P. S.

ORGANIC ANALYSIS.

Estimation of Benzene, Toluene, etc., in Coal Tar and Similar Products, and in the Intermediate Products of Tar Distillation. H. G. Colman and E. W. Yeoman. (*J. Soc. Chem. Ind.*, 1919, **38**, 57-68 T.)—The principles underlying the methods employed are those described by Colman in earlier papers (*e.g.*, *ANALYST*, 1915, **40**, 166, 170). The present paper describes various modifications and additions to the methods formerly described, extends the use of the methods to a wider range of distillates, and includes graphs. G. C. J.

NOTE BY ABTRACTOR.—This highly important paper cannot be satisfactorily abstracted. The graphs, which in the original paper are too small, would be useless if further reduced for reproduction in the *ANALYST*.

Determination of Aromatic Hydrocarbons in Petrol. H. G. Colman and E. W. Yeoman. (*J. Soc. Chem. Ind.*, 1919, **38**, 82-83 T.)—The authors have approved of the method recently described by Thole (*ANALYST*, 1919, 144) and have extended the investigation of the correction necessary on account of the expansion which occurs on mixing aromatic with aliphatic hydrocarbons. For toluene and xylene the same deviation curve may be employed, but with benzene the expansions, on mixing with paraffin hydrocarbons of the same boiling-point, are almost double those which occur with xylene and toluene. An abnormally high proportionate expansion is noted when small quantities of paraffin are mixed with large quantities of benzene. In the case of a mixture containing 50 per cent. of benzene and 50 per cent. of petrol, the correction necessary for the expansion is in the neighbourhood of 2 per cent. The following formulæ show the method of applying the data of the deviation curves to the calculation of the results: If a^1 , a^2 , and a^3 are the respective sp. grs. of the fractions after sulphonation, and b^1 , b^2 , and b^3 the sp. grs. of the fractions before sulphonation, all at 15°/15° C., then:

$$\begin{aligned} (1) \text{ Benzene} &= 100 \times \frac{\{(b^1 + k^1) - a^1\}}{(0.885 - a^1)} \\ (2) \text{ Toluene} &= 100 \times \frac{\{(b^2 + k) - a^2\}}{(0.872 - a^2)} \\ (3) \text{ Xylene} &= 100 \times \frac{\{(b^3 + k) - a^3\}}{(0.868 - a^3)} \end{aligned}$$

where k^1 and k , derived from the deviation curves, represent the amounts by which the sp. gr. of a mixture of aromatic hydrocarbon and petrol has been lowered by the expansion which occurs on mixing them. The results obtained give the volumes occupied by the respective constituents in 100 volumes of the mixture, but, since contraction takes place on separation, 100 c.c. of a mixture of equal volumes of benzene and petrol would only yield 49.75 c.c. instead of 50 c.c. of each. Hence to convert

the volume percentages found by the formula into volume percentage of the separated hydrocarbons the figures must be multiplied by $b \div (b + k)$. To convert the volume percentages found by the formula into percentages by weight, these must be multiplied by $c \div (b + k)$ where c is the sp. gr. of the aromatic hydrocarbon concerned.

J. F. B.

Detection and Estimation of Carbonyl Chloride (Phosgene). A. Kling and R. Schmutz. (*Comptes rend.*, 1919, 168, 773-775.)—Phosgene may be detected by absorption in cold saturated aniline water, with which it forms diphenylurea, which is extremely insoluble in water and can be recognised microscopically by the formation of long needles. For the detection of traces of phosgene in the air, the gas is aspirated through a few c.c. of cold saturated aniline water. The precipitate forms at once, and may be identified by microscopical examination and determination of its melting-point. Working with 5 litres of gas, passed through the aniline solution at the rate of 1 litre in five minutes, phosgene can be distinctly recognised at a dilution of 1 in 100,000. If other gases which are capable of reacting with aniline be present—for instance, chlorine or bromine—these must first be removed. This may be effected without action upon the carbonyl chloride by aspirating the gas through a filter tube containing cotton-wool which has been steeped in a concentrated solution of potassium iodide and then completely dried. For the estimation of carbonyl chloride in commercial phosgene, quantities ranging from 0.2 to 0.35 gm. are weighed out in sealed glass bulbs which are then introduced into screw-stoppered bottles containing 150 c.c. of an aqueous solution of aniline at a strength of 26 grms. per litre. The bottles are closed, the bulbs are broken by shaking, and the precipitate of diphenylurea is filtered off in a Gooch crucible after standing for two hours. The precipitate is washed with 50 to 70 c.c. of cold water, dried at 70° C., and weighed. Since small pieces of glass may be present, the diphenylurea is dissolved by boiling acetone and the crucible again weighed after igniting at about 400° C. If the commercial phosgene is in solution in an organic solvent which might act upon the diphenylurea, the precipitate should be digested on the water-bath for an hour to drive off the solvent and allowed to remain until the next day before filtration. The reaction takes place according to the equation—



provided there is an excess of free aniline. The results may be confirmed by an estimation of the chloride in the filtrate. If the quantity of carbonyl chloride exceeds 0.5 gm. for 150 c.c. of aniline water, the formation of diphenylurea is not quantitative and phenylisocyanate is also produced; in that case the experiment must be repeated.

J. F. B.

Precautions necessary in Grinding Coke for Analysis. A. E. Findley. (*J. Soc. Chem. Ind.*, 1919, 38, 93-94 T.)—When coke is ground in an iron mill or with an iron pestle and mortar the percentage of ash may be appreciably increased by iron derived from the grinding tools. The amount of this contamination will vary according to the conditions and time of grinding and the hardness of the coke.

Comparative investigations with an iron pestle and mortar and an agate pestle and mortar showed that no very appreciable error was introduced during the conversion of a coarse powder to a fine powder; the contamination must therefore occur during the conversion of the small pieces to coarse powder. A piece of hard crucible-steel coke, large enough to require considerable treatment before grinding to a powder, was divided into four parts and these were separately analysed: (a) Broken up by hammering in a cloth and then reduced to powder in an agate mortar, ash 7.0 per cent.; (b) broken up in an iron mill and powdered with an iron pestle and mortar, ash 8.2 and 8.4 per cent.; (c) broken up and powdered in a stoneware mortar, ash 8.5 per cent.; (d) control sample ground in an agate mortar, ash 7.15 per cent. The iron oxide in samples (a), (c), and (d) was found to be 1.875 to 1.885; that in sample (b) was 2.915 and 3.125. Hence it follows that contamination by grinding in a stoneware mortar is at least as great as when an iron mortar is used, but the contaminating particles in the former case are composed of stoneware and not of iron. Probably the best method for practical use is to break down the sample to coarse powder in a linen bag, and once the coke is well broken down, grinding in a mortar does not appreciably increase the ash.

J. F. B.

Colorimetric Estimation of Organic Substances. H. Heidenhain. (*J. Ind. and Eng. Chem.*, 1919, 11, 297-299.)—The method depends on the different shades of colour obtained when varying quantities of a substance (*e.g.*, sugars, glycerol, alcohol, organic acids, etc.) are heated with an excess of potassium dichromate solution and sulphuric acid; the colours are compared with those produced by known amounts of the substances under the same conditions.

W. P. S.

Estimation of Eugenol, Triacetin, and Benzyl Alcohol in Presence of Each Other. H. Perperot. (*Ann. Chim. anal.*, 1919, 1, 112-114.)—In analysing a mixture containing triacetin, eugenol, and benzyl alcohol, the triacetin is first estimated by saponification with $\frac{N}{1}$ -sodium hydroxide and titration of the excess of alkali with $\frac{N}{1}$ -sulphuric acid, turmeric being used as indicator. Under these conditions the benzyl alcohol is not affected, whilst the sodium eugenate is decomposed by the sulphuric acid. The eugenol may be estimated by determining the iodine value of the mixture by Hübl's method. If 0.5 grm. of the sample be taken and its chloroform solution treated with 10 c.c. each of the two portions of Hübl's solution, and the uncombined iodine titrated, after two hours' absorption, with a standard solution of sodium thiosulphate (24 grms. per litre), the number of c.c. of thio-sulphate required multiplied by 1.64 gives the percentage of eugenol. For the estimation of the benzyl alcohol 2 grms. of the mixture are acetylated, the excess of acetic anhydride converted into acetic acid by the addition of water, and the liquid extracted with petroleum spirit. The extract is washed with water and with sodium carbonate solution, and the washings are extracted three times with petroleum spirit. The united petroleum spirit extracts are saponified with alcoholic $\frac{N}{1}$ -sodium hydroxide solution, and the amount of benzyl alcohol calculated from the saponification value.

C. A. M.

Analysis of Natural Gas and the Calculation and Application of Results.

R. P. Anderson. (*J. Ind. and Eng. Chem.*, 1919, **11**, 299-306.)—The combustion method for the analysis of natural gas consists in passing the gas slowly into oxygen in which an electrically heated platinum spiral is glowing, the calculations of the composition of the gas being made from observations of the contraction in the volume of the gas and of the volume of carbon dioxide formed. To determine the point at which contraction no longer takes place, the authors make use of a narrow glass tube arranged vertically near the combustion pipette; the lower end of this tube is attached to a T-piece fitted in the rubber tubing connecting the pipette with the levelling bulb. When the gas has been forced over into the pipette, the rubber tube is clamped just below the levelling bulb; under these conditions contraction of the gases in the pipette will cause the level of the mercury in the narrow tube to fall, and when no further fall is observed combustion of the gas is complete. The combustion pipette should be constructed of Pyrex glass. As regards the calculation of the results of the combustion, it is considered to be more logical to state the average number of carbon atoms per molecule of paraffin hydrocarbon and the percentage of paraffin hydrocarbon in the natural gas than to give a statement of the percentages of two hydrocarbons forming a mixture equivalent, as regards combustion data, to the one actually analysed. Tables of correction factors are given and the calculation of specific gravity and heating value is discussed from the point of view of the theoretical and actual values.

W. P. S.

Indophenine Reaction. E. Wray. (*J. Soc. Chem. Ind.*, 1919, **38**, 83-84 T.)—

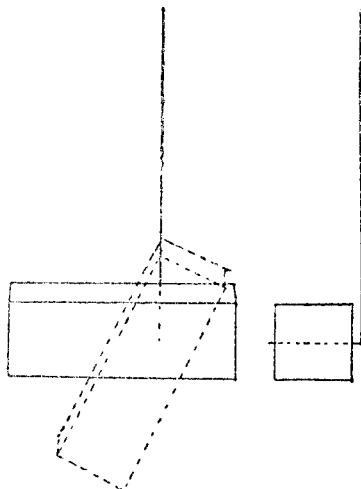
When the isatin test for thiophen in commercial benzol is carried out with pure sulphuric acid, the blue coloration may not occur but a green coloration may be obtained. Nevertheless, when this occurs, the blue colour will gradually form at the points where the liquid is exposed to the air, and is immediately developed if an oxidising agent be added to the mixture. In this observation the author confirms the statement of Bauer (*Ber.*, 1904, **37**, 1244, 3128), and he points out that, in view of the superior purity of commercial sulphuric acid at the present time, it is not sufficient to rely on the accidental presence of nitric oxides for carrying out the test. The following modification of the procedure is proposed: 25 c.c. of a solution of 0.5 gm. of isatin in 1,000 grms. of pure sulphuric acid are placed in a stoppered cylinder of 100 c.c. capacity; 1 c.c. of the benzol to be tested is added, followed by 25 c.c. of pure sulphuric acid to which one drop of concentrated nitric acid has been added. The cylinder is closed and the whole well shaken, when if thiophen be present the blue indophenine reaction is immediately obtained. The test may also be used for the quantitative colorimetric estimation of thiophen, but more accurate results are obtained by the gravimetric mercuric acetate method.

J. F. B.

Softening-Point of Pitch. P. E. Spielmann and G. C. Petrie. (*J. Soc.*

Chem. Ind., 1919, **38**, 68-70 T.)—The authors discuss older methods for determining the so-called softening-point of pitch, and recommend the following as better calculated to yield concordant results when two or more workers are concerned. A pitch block, $1\frac{1}{2}$ inches long by $\frac{1}{2}$ inch square, is cut from the sample, or moulded.

A piece of copper wire of No. 17 standard guage is bent in the form of an L, the foot of which is $\frac{3}{4}$ inch long. The shaft is heated in a flame until the foot is hot enough to penetrate the block. The wire is inserted in the side of the block at a point $\frac{1}{2}$ inch from one end. After cooling for three hours, the foot of the L exterior to the block is scraped free from pitch, and the block suspended about 2 inches below the surface of



some freshly boiled cold water contained in a 750 c.c. beaker. A thermometer with its bulb on a level with the block, and a stirrer, complete the apparatus. The temperature of the water is raised about 1°C . per minute, and the flame removed when the upper horizontal edge of the block comes in line with the upright copper wire. Stirring is continued until the temperature begins to fall, the maximum temperature observed being taken as the softening-point of the sample. G. C. J.

Quantitative Testing of Rainproof and Waterproof Cloth. G. Martin and J. Wood. (*J. Soc. Chem. Ind.*, 1919, **38**, 84-87 T.)—The most convenient rapid method for testing waterproof cloth is the drop method. A sheet of cloth is placed over a piece of white blotting-paper on a plate of glass, fixed by means of lead strips and supported at an angle of 45° . Water is then allowed to fall on the cloth from a burette placed at a height of 5 feet, and adjusted at the rate of twenty drops per minute. The flow is continued until the water penetrates and stains the blotting-paper; the number of drops is then recorded. The results fluctuate within wide limits on the same sample of cloth, so that the maximum and minimum results should be noted as well as the average. A drop test of 15 to 18 drops (average) represents a good quality of cloth, but the War Office specification requires an average of 60 drops. The test may be repeated after subjecting the cloth to various mechanical treatments such as steeping in water, pressing, ironing, or steaming, in order to ascertain the amount of deterioration produced thereby. The filter test, in which the time is noted for water to penetrate when supported in a body on the cloth, has been applied in various modifications. This test is inferior to the drop test, but is useful for detecting

pinholes. For testing the relative values of goods which are so thoroughly water-proofed as to allow very little moisture to come through after many hours, Gawalowski's apparatus is sometimes employed, consisting of a graduated tube over the lower end of which a disc of cloth is clamped. The tube is filled with water to a height of 30 cm., and the water which comes through is collected and measured. If no water has passed through in twenty-four hours, but a little moisture has collected on the underside of the cloth, this is estimated by means of a wet and dry bulb hygrometer, the apparatus being enclosed in a glass box for this purpose. J. F. B.

Volumetric Method for Estimating Salicylic Acid in the Presence of Salicylic Aldehyde. R. Berg. (*Chem. Zeit.*, 1919, 43, 129.)—The colorimetric estimation of salicylic acid by means of ferric chloride cannot be performed when salicylic aldehyde is present. A volumetric method may, however, be employed, in which the salicylic aldehyde acts as an indicator. A few drops of a 2 per cent. alcoholic solution of salicylic aldehyde when added to 100 c.c. of water show a distinct yellow colour on the addition of one drop of $\frac{N}{20}$ sodium hydroxide. The colour disappears after about half a minute, owing to the action of carbon dioxide, but returns after boiling. The procedure, as applied to 10-15 c.c. of an ethereal solution of salicylic acid, containing also salicylic aldehyde, is as follows: The solution is shaken in the separating funnel three times with 10 c.c. of a freshly prepared $\frac{N}{20}$ solution of sodium bicarbonate each time. This method of extraction is effective for quantities not exceeding 0.15 gm. of salicylic acid and 0.5 gm. of the aldehyde. The ether is then washed four or five times with 5 to 10 c.c. of water. The last wash-water must show immediately a permanent yellow colour with one drop of $\frac{N}{20}$ sodium hydroxide. The united extracts and washings, which are yellow in colour, are titrated with $\frac{N}{20}$ sulphuric acid until decolourised, and the solution is heated. The yellow colour returns as the carbon dioxide is expelled, and the end point is arrived at by repeated heating and addition of acid. It may be more convenient to acidify with excess of acid, boil, cool, and titrate back with $\frac{N}{20}$ sodium hydroxide. The method shows a satisfactory degree of accuracy. J. F. B.

INORGANIC ANALYSIS.

Analysis of Brass Ingots from Swarf. R. H. Deakin. (*Chem. News*, 1919, 118, 193-194.)—Methods are recommended for the estimation of copper, zinc, lead, tin, iron, aluminium, and nickel, any of which may be present in brass made from swarf. Copper is estimated by the iodimetric thiosulphate process on 0.5 gm. sample, adding 2 c.c. of 20 per cent. sodium phosphate to the acetic acid solution to prevent iron from reacting. Zinc is estimated by the volumetric ferrocyanide method after removal of the copper as thiocyanate. The sample (1 gm.) is dissolved in 10 c.c. hydrochloric and 2 c.c. nitric acid, the solution boiled to decompose nitric acid, diluted to 100 c.c., made ammoniacal, and then just acid with hydrochloric acid. Sulphur dioxide is passed in until the colour pales, and 7 c.c. of 20 per cent. ammonium thiocyanate added. Sulphur dioxide is passed in once more, and after half an hour the precipitate is filtered off through double hardened filters. To the

filtrate and washings 10 c.c. sulphuric acid are added, and, after a few minutes, 5 c.c. of nitric acid which completes the destruction of the excess thiocyanate. The solution is evaporated until it fumes in order to separate lead, which need not, however, be filtered off at this stage. The solution is diluted, 2 grms. ammonium chloride and excess ammonia added, and the hydroxides of iron, tin, and lead filtered off and washed with 5 per cent. ammonia containing ammonium chloride. Such lead as passes into solution at this stage does not interfere with the titration of the zinc. The filtrate is boiled to expel ammonia, and made acid with hydrochloric acid, of which 5 c.c. excess is added. It is then titrated with potassium ferrocyanide solution (1 c.c. = 0.01 gm. Zn), using uranium indicator. The ferrocyanide is, of course, standardised under identical conditions as regards acidity, bulk of solution (250 c.c.), etc. Nickel is looked for after removal of iron and excess of ammonia in above process. If present, it is precipitated as nickel glyoxime, filtered off, and estimated with $\frac{N}{50}$ cyanide solution. When nickel has to be separated in this way, the glyoxime in the filtrate must be destroyed by boiling down with hydrochloric acid before proceeding with the estimation of zinc. Lead is estimated by separating as sulphate, dissolving in ammonium acetate, and precipitating by the addition of 5 per cent. ammonium molybdate solution, drop by drop, to the boiling solution; drop by drop addition avoids the formation of acid lead molybdates of uncertain composition. In presence of iron, tin is overestimated by the usual method of treating the alloy with nitric acid, the metastannic acid being always contaminated with iron. Tin can be estimated in such material as follows: The sample (5 grms.) is dissolved in 30 c.c. hydrochloric and 10 c.c. nitric acid, the solution diluted to 200 c.c., and 2 gm. ammonium chloride and 40 c.c. ammonia added. The solution is boiled and filtered through pulp, which is washed well with hot 5 per cent. ammonia. The pulp is extracted with dilute (1:1) hydrochloric acid and cold water, and the extracts returned to the original beaker, where they are reprecipitated with ammonia, filtered off, and the pulp-filter washed. A second extraction of the pulp follows, this time with cold dilute (1:3) sulphuric acid, which leaves on the pad as sulphate any lead present. About 5 c.c. concentrated sulphuric acid are added to the extract, which is made up to 300 c.c. and saturated hot with hydrogen sulphide. The tin sulphide is filtered off on double hardened fluted filters and washed with hydrogen sulphide water containing a little sulphuric acid and ammonium acetate. It is then dried, ignited, treated with a little ammonium carbonate, ignited again, and weighed as SnO_2 . Iron is estimated in the filtrate from the tin sulphide. This filtrate is boiled to expel hydrogen sulphide, cooled, and titrated with $\frac{N}{10}$ permanganate. When it is required to examine the brass for aluminium, the filtrate from the tin sulphide is boiled until free from hydrogen sulphide, oxidised with nitric acid, iron and aluminium precipitated as hydroxides and separated in the usual way. G. C. J.

Separation of Lead in the Analysis of Metallic Calcium and Barium. J. König. (*Chem. Zeit.*, 1919, 43, 135.)—About 2 grms. of the metal are dissolved in 40 c.c. of dilute (1:1) nitric acid. After boiling for a short time, 100 c.c. of water are added, the greater part of the separated nitrates being thus brought into solution. The solution is neutralised with ammonia and then treated with 300 c.c. of bromine

water which causes the lead hydroxide to pass into solution. The solution is now made alkaline with ammonia, allowed to cool, made up to 500 c.c., mixed, filtered through a double folded filter and 250 c.c. collected, the first cloudy runnings being rejected. The lead is completely precipitated and does not occlude lime or baryta. In the filtrate, barium is precipitated as sulphate by the addition of 20 c.c. of dilute (1:1) sulphuric acid to the boiling solution, and in the filtrate from the barium sulphate calcium is precipitated as oxalate after rendering alkaline with ammonia.

G. C. J.

Gravimetric and Volumetric Estimation of Mercury Precipitated as Mercury Zinc Thiocyanate. G. S. Jamieson. (*J. Ind. and Eng. Chem.*, 1919, 11, 296-297.)—The method described recently (*ANALYST*, 1918, 43, 338) for the estimation of zinc by precipitation as mercury zinc thiocyanate may be applied, conversely, to the estimation of mercury in soluble mercuric compounds. In this case the reagent contains 39 grms. of ammonium thiocyanate and 29 grms. of zinc sulphate per litre.

W. P. S.

Estimation of Nitrous Acid and Nitrites. J. S. Laird and T. C. Simpson. (*J. Amer. Chem. Soc.*, 1919, 41, 524-531.)—The authors having tried a number of volumetric methods for the estimation of nitrites and found none of them satisfactory, have devised the following method, which they recommend. The method consists in oxidation of the nitrite in acid solution with excess of permanganate, reduction of the excess permanganate with excess of ferrous solution, oxalate, or hydrogen peroxide, and titration of the excess reducing agent with permanganate.

An excess of permanganate ($\frac{N}{10}$) is placed in a flask and acidified with 10 to 20 c.c. dilute (1:4) sulphuric acid. The nitrite solution is run into this mixture slowly with constant shaking. Excess of $\frac{N}{10}$ ferrous sulphate, hydrogen peroxide, or sodium oxalate solution is then added, and the clear solution is finally titrated with permanganate. Of the reducing agents recommended, ferrous sulphate is preferred for general work, notwithstanding its instability. The use of oxalates necessitates titration at high temperature, and peroxide solutions are even less stable than those of ferrous sulphate. Hydrogen peroxide, however, can be used in nitric acid solutions, an advantage where nitrates have to be estimated in presence of barium. Chlorides do not interfere unless the concentration of chloride exceeds $\frac{N}{2}$. It is shown that silver nitrite is not a satisfactory standard in nitrite estimations. A satisfactory standard may be made by titrating sodium nitrite solution with potassium permanganate as described above, or sodium nitrite solution may be standardised gravimetrically by reduction of silver bromate to silver bromide by Busvold's method (*Chem. Zeit.*, 1914, 38, 28).

G. C. J.

Estimation of Uranium in Alloy Steels and Ferro-Uranium. G. L. Kelley, F. B. Myers, and C. B. Illingworth. (*J. Ind. and Eng. Chem.*, 1919, 11, 316-317.)—The method outlined below is intended for the estimation of uranium in high-speed steels where it might be associated with chromium, molybdenum, vanadium, tungsten, cobalt, and nickel; in addition to these, carbon, manganese, silicon,

phosphorus, and sulphur are always present, and possibly also traces of aluminium and titanium. Two grms. of the sample are dissolved in 75 c.c. of hydrochloric acid (1 : 1) and the solution is oxidised by the addition, drop by drop, of nitric acid. If tungsten is present, the solution is diluted to 300 c.c., boiled for fifteen minutes, the tungstic oxide collected on a filter, washed, and the filtrate evaporated to dryness and baked; the dry residue is treated with 50 c.c. of hydrochloric acid (1 : 1), diluted with hot water, and filtered. The silica and the remaining traces of tungstic oxide are thus separated; the two precipitates are available for the estimation of silicon and tungsten. In the absence of tungsten, the original solution is evaporated and the residue baked to separate silica. The filtrate is evaporated to a syrup, the greater part of the ferric chloride is extracted with ether, the aqueous solution again evaporated to expel excess of acid, diluted with hot water to 150 c.c., and treated with an excess of saturated sodium carbonate solution. The mixture is boiled and filtered; in this way chromium, iron, manganese, cobalt, nickel, copper, and aluminium hydroxides are separated, together with traces of silica, titanate, phosphorus, and vanadium compounds. The filtrate contains uranium, molybdenum, and vanadium, with traces of the elements just mentioned. If the hydroxide precipitate is large it should be dissolved in hydrochloric acid and reprecipitated. The filtrate is acidified with sulphuric acid, boiled to expel all carbon dioxide, treated with a slight excess of ammonia free from carbonate, and boiled; the uranium, much of the vanadium, and traces of impurities are precipitated. If a large quantity of phosphorus was present in the original metal the precipitate must be dissolved in nitric acid, oxidised with permanganate, and the phosphoric acid precipitated and separated as ammonium phosphomolybdate; the filtrate from the latter is treated with a few drops of sulphuric acid, a small amount of ammonium persulphate, and a slight excess of ammonia, and boiled. Uranium and vanadium are precipitated; the precipitate is collected, transferred to a beaker with a small quantity of water, solid ammonium carbonate is added, and the mixture heated until a part of the ammonium carbonate is decomposed. The uranium and vanadium go into solution, and this is filtered to remove traces of iron, manganese, etc.; the filtrate is acidified with sulphuric acid, boiled to expel carbon dioxide, treated with a slight excess of ammonia, and the precipitated oxides of uranium and vanadium collected and ignited at dull-redness in a platinum crucible. The ignited precipitate consists of U_3O_8 and V_2O_5 . The weight of vanadic oxide thus precipitated with the uranium oxide must be estimated in order to obtain the amount of the uranium oxide. For this purpose the precipitate is dissolved in 50 c.c. of concentrated hydrochloric acid, the solution evaporated with 30 c.c. of sulphuric acid until fumes appear, then diluted to 250 c.c. with hot water, and titrated at 80° C. with permanganate solution, or, the diluted sulphuric acid solution may be oxidised with ammonium persulphate and silver nitrate and then titrated with ferrous sulphate solution as described by Kelley and Conant (*ANALYST*, 1916, 41, 108).

W. P. S.

Estimation of Zinc and Copper in Gelatin. G. S. Jamieson. (*J. Ind. and Eng. Chem.*, 1919, 11, 323-325.)—From 20 to 50 grms. of the gelatin, according to the amounts of copper and zinc present, are heated on a water-bath for two

hours with 100 c.c. of water and 15 to 30 c.c. of hydrochloric acid; during the first part of the heating the mixture is shaken from time to time to loosen any lumps adhering to the bottom of the vessel. The solution is then rendered slightly ammoniacal, cooled to about 40° C., and treated with a current of hydrogen sulphide for two minutes. The precipitated sulphides are collected and washed with very dilute ammonium sulphide solution; in the case of high-grade gelatin, 2.5 mgrms. of iron (as ferrous sulphate) should be added to the hydrolysed gelatin before making the solution ammoniacal. The sulphides are dissolved in hot dilute nitric acid (1 : 1), the solution evaporated with the addition of 10 c.c. of sulphuric acid (1 : 3) until all nitric acid has been expelled, then diluted with 30 c.c. of water, filtered, diluted to 100 c.c., heated at 50° C., and treated with hydrogen sulphide for five minutes. The copper sulphide is collected on a Gooch crucible, washed with warm water saturated with hydrogen sulphide, dried, ignited to oxide, and weighed. The filtrate containing the zinc is heated to expel all hydrogen sulphide, then rendered slightly ammoniacal, 15 c.c. of 50 per cent. formic acid are added, the mixture treated with hydrogen sulphide for five minutes, heated on a water-bath for thirty minutes, and the zinc sulphide collected on a Gooch crucible, washed with 2 per cent. ammonium thiocyanate solution, dried, ignited to oxide, and weighed.

W. P. S.

Conditions affecting the Precise Estimation of Zinc as Sulphide.

H. A. Fales and G. M. Ware. (*J. Amer. Chem. Soc.*, 1919, **41**, 487-499.)—The precipitation of zinc from solutions of formic acid by means of hydrogen sulphide has been studied with special reference to the conditions affecting the accuracy of the estimation. It is shown that the range of hydrogen ion concentration of between 10^{-2} and 10^{-3} is the most favourable for quantitative precipitation of zinc sulphide in a form suitable for rapid filtration and washing. The concentration of hydrogen ions may be kept within this range by the use of ammonium citrate solution and a mixture of ammonium sulphate, ammonium formate, and formic acid. A high concentration of an ammonium salt of a strong acid also favours the formation of a granular precipitate, as does a volume of at least 100 c.c. for every 0.1 gm. zinc present and a temperature of 95° to 100° C. The passing of hydrogen sulphide under pressure is desirable for rapid saturation and for avoidance of loss of formic acid by evaporation.

The zinc solution, freed from interfering elements such as copper and cadmium, is evaporated to about 125 c.c. and ammonia added until a slight permanent precipitate results. Twenty-five c.c. of citric acid solution (200 grms. per litre) are next added and the mixture made neutral to methyl orange by addition of moderately strong (15 molar) ammonia. Twenty-five c.c. of a mixture, containing 30 c.c. ammonia (15 molar), 200 c.c. formic acid (23.6 molar), and 250 grms. ammonium sulphate per litre, are next added, and the volume is made up to 200 c.c. or more if more than 0.2 gm. zinc is present. The solution is heated to 60° C. and the flask connected with a hydrogen sulphide generator so that the air is slowly displaced by hydrogen sulphide as the heating continues. At 90° to 100° C., when steam issues from the flask, the exit is closed and the solution, as it cools, is saturated with hydrogen

sulphide under increasing pressure. A Kipp generator provided with a pressure bulb is used for the supply of hydrogen sulphide. The generator is connected in series with a wash bottle which contains a manometer bulb, with a stem of such length that when the liquid level reaches the bottom of this bulb the pressure in the precipitation flask is approximately a third of an atmosphere. The flask is shaken frequently to ensure saturation, which is usually complete, when the precipitate settles rapidly, in about thirty minutes. The precipitate is filtered and washed with a cold, aqueous solution about 0.1 molar with formic acid (4 c.c. of 23.6 molar formic acid per litre) saturated with hydrogen sulphide. The precipitate is transferred to a porcelain crucible and the paper charred, preferably by standing the crucible on a quartz plate heated strongly by a Méker burner. The ignition of the filter-paper is very slowly and carefully completed in an oxidising atmosphere over a Bunsen burner. The reaction should proceed only to the formation of sulphate, not of oxide. After cooling, the precipitate is carefully moistened with concentrated sulphuric acid and the crucible heated in an air-bath until the excess of acid is driven off. If zinc sulphide adheres firmly to the flask, as often happens, it must be dissolved in hot dilute sulphuric acid, the solution neutralised with ammonia, then made faintly acid with formic acid, saturated with hydrogen sulphide under pressure, heated to boiling to coagulate the precipitate, and filtered through the filter containing the already washed zinc sulphide. Further washing is unnecessary. The method as described serves for the precipitation of zinc where only small amounts of the usual interfering metals are present. Not more than 0.2 mgrm. of manganese is carried down even when manganese is present in amount equal to that of zinc. Iron and nickel, however, require more acid to hold them up, if present in concentrations equal to one-tenth that of the zinc; in such cases 20 c.c. excess formic acid (23.6 molar) must be present to secure a clean separation. Cobalt cannot be separated from zinc by one precipitation.

G. C. J.

APPARATUS, ETC.

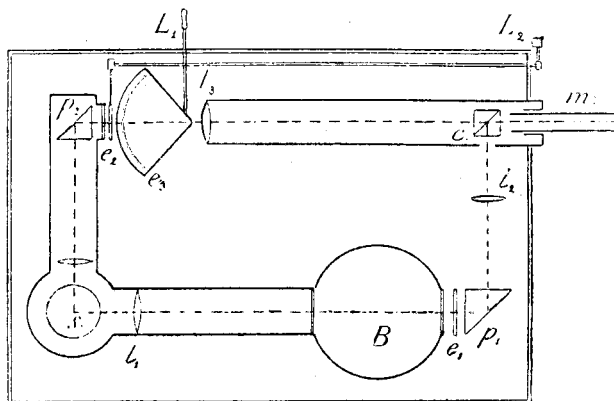
Apparatus for the Automatic Estimation of Small Amounts of Oxygen in Combustible Gas Mixtures or of Combustible Gases in Air. H. C. Greenwood and A. T. S. Zealley. (*J. Soc. Chem. Ind.*, 1919, **38**, 87-90 T.)—The authors have devised an automatic gas analyser which is capable of giving an alarm when the percentage of a certain impurity in the gas mixture exceeds a prescribed value. This form of the apparatus is applicable for the estimation of any impurity which causes a contraction in volume after combustion, this being brought about by contact with a heated platinum wire. For instance, in the production of ammonia from a mixture of nitrogen and hydrogen the apparatus may be used to estimate the oxygen present as an impurity. In the electrolytic production of hydrogen and oxygen it may be used to estimate the contamination of one of the gases with the other owing to perforation of the diaphragm. It may also be used for detecting the presence of hydrocarbon gases in the air of mines or benzol distilleries. The general type of the apparatus is based on the principle already applied for the automatic analysis of furnace gases, but the contraction to be measured is not produced by

absorption in a reagent but by the combustion of the impurity present in the gases. Automatic arrangements provide for making and breaking the required electrical contacts; sealing up a measured quantity of gas in the combustion vessel during the heating period; cooling the gas and measuring the contraction which results; ringing an electric bell when the contraction exceeds a certain amount; flushing out the spent gas by a new charge. The motive power is derived from two water syphons, the second being operated by three discharges of water from the first. By the first discharge of the small syphon the pressure in the limb of the second syphon causes the sealing of the sample of gas in the combustion vessel followed by the switching on of the heating current, these operations being effected by the movement of mercury in the tubes of the apparatus. The second discharge causes a further displacement of the mercury whereby the heating current is cut off. During the next period the gas in the combustion vessel is being cooled by the circulation of water through an arrangement similar to a double surface condenser. The water enclosing the gas which has risen into a compensating bulb during the period of heating will then fall as the gas is cooled, and the amount of fall below the zero point of the instrument is the measure of the contraction. The sensitiveness of the instrument is regulated by the diameter of the tube below the bulb, but it is not desirable to arrange a fall of more than about 7 cm. in this tube. The fall of water in the measuring tube is caused to produce a proportional movement of mercury in an attached vessel, and this movement governs the ringing of an alarm bell when it exceeds the prescribed amount. The third discharge of the small syphon then causes the discharge of the large one, and the cycle of operations is restored to the starting-point, the combustion vessel being opened to the main flow of gas, which sweeps through until the next discharge of the small syphon closes the seals again for another analysis. The time period of the discharge of the small syphon controls the completeness of combustion; the degree of flushing and the degree of cooling after combustion; the period allowed may be five minutes and no sensible error is introduced provided it exceeds one and a half minutes. The heating of the platinum wire should be carried to definitely visible redness, beyond which point no advantage is gained by further heating. The apparatus can conveniently be made sensitive to the presence of 0.2 to 0.3 per cent. of oxygen in a mixture of nitrogen and hydrogen.

J. F. B.

Opacimeter designed for Estimating Bacteria. Lambert, Vles, and De Watteville. (*Comptes rend.*, 1919, 168, 797-799.)—Bacteria suspended in a liquid may be treated as an emulsion, and their quantity estimated photometrically. Certain conditions must be observed in the construction of the apparatus: it is necessary to employ almost monochromatic light of a determined wave length, and the vessels containing the liquids must be capable of sterilisation at high temperatures. In principle the opacimeter is a photometer comprising two juxtaposed rays of light emanating from the same source, one passing through the emulsion and the other through a dimming system of graduated opacity. The apparatus illustrated in the figure is illuminated by a 100 c.p. nitrogen-filled lamp, S, in a metal case, the two pencils of rays issuing at right angles. One pencil is projected by the lens, l, in

parallel rays through two opposite windows in the large copper vessel, *B*, filled with water and capable of receiving the glass cylinder containing the emulsion. The issuing light passes through a colour-screen, *e*₁, average wave length 620 $\mu\mu$, and is totally reflected by a prism, *p*₁, through a lens, *l*₂, on to a cube of glass composed of two cemented prisms, one of which is partially silvered, and reflects the light into



the eyepiece of the microscope. The other pencil of rays from the lamp, *S*, is reflected by the prism, *p*₂, through the colour screen, *e*₂, and the graduated photometric dimming screen, *e*₃, and thence to the microscope, the graduated scale being focussed on the silvered cube. The graduations of the photometric screen are arbitrary, and have to be calibrated in terms of the dry weight, or numbers, of bacteria of the species studied contained in 1 c.c. of the emulsion. This is done by gravimetric centrifugal estimation, or by microscopic counting with a standard emulsion and photometric examination of aliquot dilutions of the same emulsion. J. F. B.

Electrical Precipitator for Analysing Smoke. R. C. Tolman, L. H. Reyerson, A. P. Brooks, and H. D. Smyth. (*J. Amer. Chem. Soc.*, 1919, **41**, 587-589.)—The paper describes and illustrates a modified Cottrell precipitator, which can be used for the analysis of any smoke independent of its chemical nature. It consists essentially of a glass tube $\frac{3}{8}$ inch in diameter and 10 inches long, with a ground glass joint, a central platinum wire, which functions as cathode, and a cylinder of aluminium foil 6 inches long and 0.001 inch thick as anode. The tendency to sparking, which would interrupt the corona discharge, is reduced by threading the wire with a jeweller's die. Direct current in the neighbourhood of 15,000 volts can be used without sparking. In general it is found possible to aspirate a fairly concentrated sample of smoke through the apparatus at a speed of 5 litres a minute and obtain complete precipitation. After precipitation has taken place, the air passes through a flow meter to determine the volume of sample taken. The aluminium foil is weighed at the beginning and end of the experiment. G. C. J.



SCOTTISH BOARD OF HEALTH.

In response to a memorial presented by the Association of Public Analysts of Scotland, the Right Hon. Robert Munro, K.C., M.P., Secretary for Scotland, received a deputation of professional chemists at the Scottish Office, Whitehall, on Saturday, May 3, 1919, when the importance of chemistry in relation to the Scottish Board of Health Bill was discussed. The deputation consisted of:

Mr. R. M. Clark, B.Sc., F.I.C., President, and Dr. J. F. Tocher, F.I.C., representing the Association of Public Analysts of Scotland; Mr. A. Chaston Chapman, F.I.C., Dr. J. T. Dunn, F.I.C., Members of Council, and Mr. Richard B. Pilcher, Registrar, representing the Institute of Chemistry; Mr. B. D. Porritt, B.Sc., F.I.C., representing the Scottish Local Sections of the Institute; and Dr. Samuel Rideal, President, and Mr. P. A. Ellis Richards, Hon. Secretary, representing the Society of Public Analysts.

Mr. R. M. Clark (Glasgow) introduced the deputation. He said that the Institute of Chemistry represented professional chemists, and that its qualifications were formally recognised by the Local Government Board for Scotland in connection with the appointment of public analysts. He explained that the Society of Public Analysts was the representative organisation of public analysts generally. He said that the public analysts were in favour of the Bill as a whole. He referred to the unanimous request of the whole of the public analysts of Scotland, and to the memorial which had been forwarded by the Association of the Public Analysts of Scotland. In view of the importance of chemistry to matters of State, and especially matters of health, it was highly necessary in the public interest that the Secretary for Scotland should consider the inclusion of a qualified professional chemist on the proposed Board of Health. Since the Food and Drugs Acts were not properly administered, it was necessary in any case that a professional chemist should be attached to the Board, and that the profession of chemistry should be represented on the Consultative Councils under the Board. Mr. Clark mentioned many health questions in which chemical advice and assistance were necessary, and gave particulars which evidenced improper administration. Referring to the Sale of Food and Drugs Acts, which would come under the control of the New Board, he showed the lack of closer touch between the Local Government Board and public analysts in the past had resulted in inadequate and uneven administration. Public analysts had no proper security of tenure of office, and were inadequately remunerated for their services. In many places the Acts were entirely ignored.

Dr. J. F. Tocher said that it was against the public interest to overlook the importance of chemistry in public health matters. Chemists were required, not only for the administration of the Sale of Food and Drugs Acts, but also in connection with the administration of the Alkali, etc., Works Regulation Acts, the Rivers Pollution Acts, and other Statutes. He noted, also, that the Scottish Board of Health Bill embodied proposals for the conduct of research which included research in chemical problems. He summarised the qualifications of a chemist as defined by

the Regulations of the Institute of Chemistry, and claimed that the profession of chemistry should be accorded adequate recognition. The administration of the Sale of Food and Drugs Acts in Scotland had never been satisfactory owing to the fact that there had been no chemical officer associated with the Local Government Board. In the public interest it was highly desirable that public analysts should always be consulted by local authorities in matters relating to the administration of the Sale of Food and Drugs Acts, and that they should be properly recognised as officers of the local authorities employing them. A principal chemical officer should be appointed as the adviser of the Board in all chemical matters, including, of course, those connected with food and drugs. There appeared to be an impression in the minds of the authorities that Medical Officers of Health were properly qualified to advise in chemical matters, and that, therefore, they should control the local administration of the Food and Drugs Acts. The position of the Medical Officer of Health under these Acts, however, was that of a sampler, and, in this respect, he was in a similar position to that of a police officer and an Inspector of Weights and Measures. It was highly necessary that a chemical officer should be appointed, either as a member of the Board of Health or as a responsible officer of the Board, to advise on chemical matters relating to the health of the community.

Dr. Rideal supported the two previous speakers in their remarks with regard to the lack of proper contact between the Local Government Boards and public analysts. The latter were hardly ever informed of regulations issued by the Board having a direct bearing on their work under the Sale of Food and Drugs Acts. There was a definite need for chemical authority at headquarters. At present the Boards looked to their medical officers for chemical information; but there should be professional chemical officers with a proper knowledge of chemical technique, and there should be proper co-ordination of the work.

Mr. Chaston Chapman said that the war had made everyone realise the increasing importance of chemistry in Governmental affairs. The public was beginning to realise its bearing on all technical processes. Its relation to public health matters was no less important. If the great aim of the Board of Health was to create a healthy environment, it was absolutely necessary that chemists should be intimately associated with this work. All matters relating to the purity of water, air, food, and drugs, came essentially within the function of the chemist. He emphasised, therefore, the necessity of having a chemical officer attached to the Board. If it was found to be impossible, for administrative reasons, to increase the number of members of the Board, he urged the extreme desirability of the representation of chemistry on the Consultative Councils.

Mr. Munro said that he was glad to know that professional chemists supported the measure as a whole. The Bill would be before the House of Lords on Tuesday. He had listened with care and attention to the comprehensive and cogent arguments which had been advanced. The remarks of the deputation might be divided into two sections: (1) The connection of chemists with the Scottish Board of Health, and (2) the amendment of the present Sale of Food and Drugs Acts. In the first, they had concentrated on the importance of the profession of chemistry being represented on the Board of Health. It was obviously impossible to include representatives

of all interests, or of even all important interests, as in that case it would not be a Board, but a mob. Science would be represented—if not adequately represented—by two medical men. The Government was not able to add even one more member without raising further claims. Dentists, veterinary surgeons, pharmacists, sanitary inspectors, and so forth, would all wish representation on the Board. While, however, there was no possible chance of a Government amendment giving effect to the proposal made by the deputation to include a professional chemist on the Board itself, he thought that the objects of the deputation would be attained (1) if a professional chemist was included on the staff as a consultative officer with suitable standing, and (2) by ensuring that chemistry was properly represented on the Consultative Councils. He would confer with his colleague, Dr. Addison, on the whole matter.

With regard to many of the remarks which had been made with reference to the Sale of Food and Drugs Acts, he would be glad to reconsider them when future legislation in that connection was under consideration.

Dr. Dunn, on behalf of the deputation, cordially thanked the Secretary for Scotland for granting the interview. The deputation then withdrew.



INTER-ALLIED FEDERAL COUNCIL OF PURE AND APPLIED CHEMISTRY.

For some time past it has been becoming increasingly clear that of all the problems of reconstruction with which the Allies are faced, one of the most important is the organisation and co-ordination of scientific effort, and the fullest utilisation in the interests of the nations concerned, of all the discoveries and advances which may be made in the domain of natural science. Great as are the advantages of preserving a proper amount of individuality in scientific matters, there is no doubt that the time has arrived when international co-operation is necessary both for the purpose of securing economy of effort, and for preventing that waste of energy due to overlapping, which has for a good many years past exercised the minds of chemists in this, and doubtless in other countries.

The great advance made by the Germans in the application of chemical science to industry during the past half-century has been due quite as much to co-ordination of effort as to hard and unremitting labour. It may fairly be assumed that these qualities and this policy are not likely to disappear as the result of the War, and that the Allies will, in consequence, be faced with the necessity for making every possible effort to meet future German competition. In order to do this, they will have to oppose to the Central Empires a united front.

The recent Inter-Allied Conference in Paris represents a first and very important step in this direction. This Conference had its origin in the visit paid to this country, in November last, by M. Paul Kestner, President of the Société de Chimie Industrielle, and was presided over by Professeur Moureu, Président de la Fédération Nationale des Associations de Chimie Pure et Appliquée, a body analogous to the Federal Council for Pure and Applied Chemistry in this country. Among other French

representatives present were MM. Haller, Poulenc, Béhal, Hanriot, Marquis, representing the French Chemical Society, MM. Paul Kestner, Matignon and Gérard, representing the French Society of Chemical Industry, and a number of chemists representing other French Societies and Technical Associations.

The British delegation consisted of Sir William Pope and Mr. A. Chaston Chapman, representing the Federal Council, and Messrs. Henry Louis, W. F. Reid, Stephen Miall and Edwin Thompson, representing the Society of Chemical Industry.

Among the American representatives were Messrs. Henry Wigglesworth, Lieut-Colonels Bartow, Norris, Zanetti, Major Keys, Dr. Cottrell and Mr. J. C. Pennie.

Italy was represented by Professor Emmanuel Paterno, President of the Italian Chemical Society, Doctors Pomilio, Parodi-Delfino and Giordani; and Belgium by M. Chavanne, President of the Belgian Chemical Society.

It was unanimously resolved to form an Inter-Allied Federal Council for Pure and Applied Chemistry, the Council to consist of not more than six representatives of each of the above-mentioned countries, the members to hold office for three years, and one-third to retire annually and to be eligible for re-election.

The objects of the Federation are to consolidate, as between the different Allied countries, the bonds of esteem and friendship which have already been strengthened during the War; to organise a permanent co-operation between the Chemical Associations of the Allied nations; to co-ordinate their scientific and technical resources, and to contribute to the advancement of chemistry in all its different departments.

It was recognised that none of the delegates present had any authority to bind the Societies and Associations which they represented, and that the resolutions passed at the Conference would have to be submitted for ratification to the various Societies concerned. A Committee which should provisionally act as the Council of the Fédération, and consisting of two representatives of each country, was formed for the purpose of carrying on until the next meeting, which it is suggested should take place in London in July, when it is hoped that the Inter-Allied Federation will be definitely formed. The two British representatives elected as members of this provisional committee were Sir William Pope and Professor Henry Louis.

It may be added that it is proposed, in due course, to admit "neutral" nations to the membership of the Federation.

Although the object of this article is to describe, in the briefest manner possible, the aims of the Conference, and the main results arrived at, it is impossible to omit some reference to the charming hospitality extended by our French colleagues to all the delegates, and to the perfection of the arrangements made for their comfort during their stay in Paris. It is to be hoped that the annual meeting of the Society of Chemical Industry, in July next, will afford us an opportunity of reciprocating the very great kindness shown on every hand to the members of the British delegation.

A reference to the aims of the Federation, as stated above, will suffice to show that the new Inter-Allied Council, which it is to be hoped will come into definite and formal existence in July, will have before it a task of no small magnitude. A good start has been made in that personal contact has now been established, and having regard to the evidences of good feeling and understanding between the Allied repre-

sentatives which was so prominent a feature of the meetings in Paris, it may confidently be hoped that the work of the Federation will proceed harmoniously and effectively. In that event, great good must result, both for chemistry as a science and for those who, as practitioners or as technologists, are interested in any of its various branches.

A. CHASTON CHAPMAN.



REVIEWS.

TECHNICAL HANDBOOK OF OILS, FATS, AND WAXES. By PERCIVAL J. FRYER, F.I.C., and FRANK F. WESTON, B.Sc., F.I.C. Vol. II. Practical and Analytical. Cambridge Technical Series, Cambridge University Press, 1918. 69 Illustrations. Pp. 314. Price 15s. net.

SINCE reviewing Vol. I. of this work (ANALYST, February, 1918) we have awaited with interest the publication of Vol. II., and it is gratifying to note that the utility of the second volume is equal to that of the first.

The present volume has two outstanding features: First, it is a veritable "card index," and written in such a way as to render reference to any part easy and rapid. The greatest care has been taken to set out the printed pages so as to bring the information to the eye of the reader in its order of importance and with the minimum expenditure of mental and physical energy. Secondly, one cannot help being struck by the clearness of the illustrations, particularly the photographs, which show the worker exactly how the authors recommend the various parts of the apparatus to be set up and used. We should not, however, all agree that a shallow evaporating basin supported by a dangerous-looking tripod was the best form of water-bath for heating a saponification value flask. Neither would it be convenient to use two retort stands for each Polenske apparatus; nor would one clamp a Soxhlet apparatus in the dangerous manner indicated.

The book is divided into ten Sections.

Section I. deals with the main principles of general analytical work, including the preparation of standard solutions. In this section the method of preparing $\frac{N}{10}$ sodium carbonate by weighing the chemical on an open watch-glass is very questionable, particularly in the hands of a student.

Section II. gives directions for sampling and preliminary tests.

Section III. is a most important one, and is specially well arranged; it includes a description of the standard analytical determinations.

Section IV. describes special tests and reactions for fats and oils.

Section V. is on the identification and estimation of fatty acids and alcohols, including glycerol.

Section VI. deals with the analysis of hydrocarbons, oils, and waxes.

Section VII. briefly, but adequately, describes methods of testing resin and turpentine.

Section VIII. deals with the interpretation of analytical results.

Section IX. is mainly good advice to the student, with a scheme of analysis and identification.

Section X.—Tables.

Both volumes of this little treatise are to be recommended not only to the student who wishes to familiarise himself with the modern methods of examining oils, fats, and waxes, but also as a useful reference book to the practising analyst.

E. RICHARDS BOLTON.

OSMOTIC PRESSURE. By ALEXANDER FINDLAY. Pp. 116. Longmans, Green and Co., 1919. Price 6s. net.

This volume is one of a set of "Monographs on Inorganic and Physical Chemistry," and is contributed by the editor of the series, Professor Findlay of Aberystwyth. A first edition was published about six years back. Since then some important experimental work has appeared, and there has been a good deal of discussion on the primary cause and mechanism of osmotic pressure. Much of this is incorporated or noticed in the present volume.

The series as a whole is intended for Honours chemistry students and for research workers in the various fields covered; but others will find much that is informative and stimulating in the different volumes, and not least so in this one. The subject cannot be said to make any very immediate appeal to the analytical chemist in general; but, on the other hand, it lies at the root of that modern theory of electrolytes which has shed so much light on the rationale of "wet" methods of inorganic analysis, and it possesses a very direct interest for those who deal with the chemistry of organic tissues and fluids.

Following on a brief historical and introductory chapter, the author discusses the work of van't Hoff, who was the first to correlate the earlier observations by means of a definite theory. An excellent account of the experimental methods of the Earl of Berkeley and Hartley, and of Morse, Frazer, and their collaborators, is next given. Subsequent chapters deal with the theory of ideal solutions, and discuss the available experimental data—practically all for aqueous cane-sugar solutions—in the light of this and of van't Hoff's theory of dilute solutions. The relationships of osmotic pressure to other properties of solutions (vapour pressure, etc.) have a chapter devoted to them, and the monograph closes with a chapter on the fundamental causes of osmotic pressure and the rôle of the semipermeable membrane. Towards these subjects the author adopts a truly cautious and non-committal attitude. We miss an exposition of the views of Tinker on the first of these points; otherwise the book appears to us to give an excellent survey of the field. There are good indexes and a full list of literature references.

A. J. ALLMAND.

ERRATUM.—Page 128, line 16. After weighed, add the words "ignited and the weight of ash subtracted in order to obtain real starch."
