

THE ANALYST.

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

ORDINARY MEETING, NOVEMBER 3, 1920.

HELD at the Chemical Society's Rooms, Burlington House, Mr. Alfred Smetham, President, in the chair.

Certificates were read for the first time in favour of Messrs. Urban Aspey, Herbert Corner Reynard, B.Sc. (London), A.I.C., Edwin Burnhope Hughes, B.Sc. (London), A.I.C., Harry Jephcott, M.Sc. (London), A.I.C., Arnold Lees, A.I.C.

A Certificate was read for the second time in favour of Mr. T. K. Ghose, B.A.

The following was elected a Member of the Society: Mr. Udolphus Aylmer Coates.

The following papers were read: "The Gravimetric Estimation of Bismuth as Phosphate and its Applications in Ore Analysis," by W. R. Schoeller, Ph.D., and E. F. Waterhouse; "The Time Factor in Saponification," by Percival J. Fryer, F.I.C.; "The Position of Analytical Chemistry in France," by Victor Cofman, B.Sc.; "Apparatus for Collecting Samples of Water at Great Depths," by W. T. Burgess, F.I.C.



THE GRAVIMETRIC ESTIMATION OF BISMUTH AS PHOSPHATE AND ITS APPLICATION IN ORE ANALYSIS.

BY W. R. SCHOELLER, PH.D., AND E. F. WATERHOUSE.

(Read at the Meeting, November 3, 1920.)

THIS paper is the result of an investigation into the analytical chemistry of bismuth, in the course of which we elaborated a slightly modified method for estimating the metal as phosphate. As pointed out by Moser in his monograph, "*Die Bestimmungsmethoden des Wismuths*" (Stuttgart, 1909), the phosphate process is the most advantageous form of bismuth estimation; bismuth phosphate, BiPO_4 , is of definite composition; it forms a white, heavy, crystalline precipitate resembling lead sulphate, quite insoluble in water and in very dilute nitric acid, depositing and filtering quickly,

unchanged by ignition, and not readily reduced. In spite of these advantages, the method does not appear to have found a place in any English textbook. It has been studied by Stähler and Scharfenberg (*Ber.*, 1905, **38**, 3862), Salkowski (*ibid.*, **38**, 3943), Moser (*Zeitsch. anal. Chem.*, 1906, **45**, 19), and Stähler (*Chem. Zeit.*, 1907, **31**, 615). Salkowski adds 5 c.c. of 25 per cent. phosphoric acid to the hot, moderately acid nitrate solution free from hydrochloric acid or chlorides, which have a solvent effect on bismuth phosphate. Stähler and Scharfenberg use a 10 per cent. solution of trisodium phosphate, enough of which is added to neutralise nearly all the nitric acid present in the solution; they state that disodium phosphate may also be used, but in this case the final volume of the liquid, if rather acid at the start, may become inconveniently large. These directions make it plain that a very large excess of precipitant is used, especially as chlorides are stated not to interfere provided sufficient sodium phosphate is added. Moser, on the other hand, adds only a very slight excess of $\frac{N}{5}$ diammonium phosphate solution to the liquid containing no chlorides and as little free nitric acid as possible. Stähler almost neutralises the nitrate solution with trisodium phosphate, using rosolic acid as an indicator. This very brief survey will suffice to show a marked variation in the procedure adopted by different investigators for the precipitation of bismuth phosphate; moreover, their views as to the solubility of the precipitate in dilute nitric acid would appear not to be the same, and some of the directions for adjusting the acidity of the solution are sufficiently vague to make experimentation necessary before confidence in the accuracy of the process is finally gained. We felt, therefore, that it might be useful to repeat the earlier work and, if possible, improve upon it. Working with pure bismuth nitrate solutions, we soon arrived at the conclusion that the quantitative precipitation of the phosphate was a matter of rather delicate adjustment: if the acidity was kept low, the liquid on being heated to boiling usually deposited a heavy crystalline precipitate of oxynitrate, to re-dissolve which it was necessary either to add a disproportionate amount of nitric acid or evaporate to small bulk. Again, the phosphate obtained from solutions containing but little nitric acid was rather bulky and flocculent, and appeared to lead to slightly high results. If, on the other hand, the acidity was a little higher than necessary to prevent deposition of the subnitrate, a small fraction of the bismuth failed to precipitate as phosphate unless an excessive quantity of alkaline phosphate was added. This fact would explain the use of trisodium phosphate by Stähler and Scharfenberg. As regards Moser's practice—*i.e.*, addition of a slight excess of ammonium phosphate over that required to react with the bismuth nitrate—we were unable, as a rule, thereby to effect complete precipitation. Further work led to the adoption of our modified process, which is free from the drawbacks indicated above, and has given consistently good results; the directions about to be given are precise enough to enable those unfamiliar with the method to apply it successfully.

Authors' Method for Estimating Bismuth as Phosphate.—The cold bismuth solution, containing no chlorides and an indefinite amount of nitric acid in a total bulk of less than 100 c.c., is carefully treated with strong ammonia until a slight permanent precipitate is obtained. The precipitate, being formed in the cold, is not the crystalline oxynitrate, but a flocculent basic nitrate which dissolves at once on addi-

tion of 2 c.c. of strong nitric acid. The clear liquid is now heated to boiling and precipitated whilst boiling with a 10 per cent. diammonium phosphate solution contained in a burette. The addition should proceed very slowly at first (about 80 drops per minute) to favour the formation of a coarsely-crystalline precipitate; once the bismuth phosphate is thrown down, the remainder of the precipitant may be added more rapidly. It is advisable to stir the solution, as the beaker is liable to bump badly. A considerable excess of ammonium phosphate should be used, viz.:

20	c.c. of 10 per cent. solution for	0.05	gram. bismuth.
30	"	"	0.1 "
40	"	"	0.2 "
50	"	"	0.3 "
60	"	"	0.4 to 0.5 "

When the required amount of precipitant has been added, the solution is diluted to 300—400 c.c. with boiling water and left to settle for ten to fifteen minutes on a hot plate or water-bath. The clear liquid is then decanted through a 9 cm. No. 40 Whatman filter, or preferably a Gooch crucible; the precipitate is stirred up twice with a hot 3 per cent. ammonium nitrate solution containing a few drops of nitric acid per litre, transferred to the filter, the washing completed, and the precipitate dried and ignited gently. If a filter paper is used, the precipitate is detached as completely as possible, and the paper charred at low temperature in a porcelain crucible. Factor Bi: $\text{BiPO}_4 = 0.6865$.

The method in its present form entirely obviates the formation of crystalline oxynitrate caused by deficiency of nitric acid, while the subsequent dilution of the liquid reduces the concentration of the nitric acid to a point where the last traces of the bismuth are precipitated by the excess of ammonium phosphate. The filtrate from the bismuth phosphate usually remains quite colourless on treatment with hydrogen sulphide; sometimes a faint coloration is produced, but the loss is altogether negligible (*i.e.*, less than 0.0002 gm.).

Authors' Process for Estimating Bismuth in Ores.—This process, which we are using in actual practice, consists in determining bismuth as phosphate after separating it from lead, arsenic, antimony, copper, and iron. Special attention is given to the quantitative separation of lead, which we effect by Clark's method, *i.e.*, precipitation of the bismuth by metallic iron, lead (and any tin present) remaining in solution (*J. Soc. Chem. Ind.*, 1900, 19, 26). This is undoubtedly the most satisfactory practical method for the separation of the two metals. Galletly and Henderson, who tested it, found it to be "not only easily carried out, but also capable of yielding accurate results" (*ANALYST*, 1909, 34, 389); but, like the phosphate method for bismuth, it has not yet found its way into any of the standard textbooks. The separation of arsenic and antimony is carried out, as usual, by digesting the hydrogen sulphide precipitate with alkaline hydroxide and sulphide, any molybdenum present being also extracted. If the ore carries copper, the precipitated sulphides are digested with sodium cyanide, which dissolves copper sulphide; arsenic, antimony, and copper may be removed in one operation. Mercury, a rare constituent, is eliminated when the sulphides are treated with

nitric acid, in which the mercury compound is insoluble. The assay is carried out as follows :

The ore (1 grm. or more, according to the bismuth content) is digested on a hot plate with strong hydrochloric acid ; galena, if present, is thus decomposed without formation of lead sulphate. Nitric acid is then added, and the assay evaporated almost to dryness. The nitrates are again converted into chlorides by evaporation with hydrochloric acid ; the residue is taken up with the same acid, and after moderate dilution with water, the gangue is filtered off and washed with dilute hydrochloric acid (1 : 10 water). The filtrate is precipitated with 1 to 2 grms. of pure, very fine iron wire (one foot of which weighs about 0·1 grm.), the action being allowed to proceed at boiling heat for ten to twenty minutes. The metallic sponge and excess iron are filtered off, washed with boiling water, returned to the beaker, and dissolved in hot hydrochloric acid and a few drops of bromine. When the metals have dissolved the solution is boiled till bromine is expelled, diluted, precipitated with hydrogen sulphide, and the precipitate filtered off and washed with acidulated hydrogen sulphide water. The sulphides are returned to the beaker and treated hot with freshly-made caustic soda and hydrogen sulphide, a freshly-made solution of sodium cyanide being added if copper is present. The residue from this extraction is filtered on the paper previously used, and washed with dilute sodium sulphide solution. The filter is then spread against the side of the beaker, the precipitate rinsed down, and the paper cleaned with hot dilute nitric acid (1 : 1). The washed filter is discarded, more nitric acid added, and the assay evaporated until the sulphur has fused into a transparent globule. Bromine should on no account be used at this stage, or low results may ensue ; if nitric acid alone fails to decompose the sulphide completely, the presence of mercury is indicated. The sulphur is filtered off on a very small filter which is washed with dilute nitric acid ; the filtrate, which should occupy less than 100 c.c., is now ready for the phosphate precipitation.

If a preliminary test proves lead to be absent, the iron precipitation is omitted and the filtrate from the gangue treated with hydrogen sulphide. If copper and the metals of the arsenic sub-group are known to be absent, the hydrogen sulphide precipitate is dissolved at once in nitric acid for the precipitation of the bismuth as phosphate.

Results of Test Analyses.—A number of ores were assayed by us according to the method described above, with the following results :

Ore A contained As, Sb, and a little Pb.

Bi (as BiOCl) : 4·63 ; 4·62 per cent.

Bi (as BiPO₄) : 4·64 ; 4·70 ; 4·72 per cent.

Ore B contained Pb, Cu, and As.

Bi (as BiOCl) : 15·83 ; 15·70 ; 15·71 per cent.

Bi (as BiPO₄) : 15·76 ; 15·82 ; 15·83 ; 15·86 per cent.

Ore C contained wolfram and very little copper. The filtrate from the gangue was treated with hydrogen sulphide, the precipitate dissolved in nitric acid, and the solution precipitated with ammonium phosphate.

Bi (as BiOCl) : 34·15 per cent.

Bi (as BiPO₄) : 34·05 ; 34·09 ; 34·03 ; 34·17 per cent.

Ore D contained small amounts of As, Sb, and Pb.

Bi (as BiOCl) : 20·17 per cent.

Bi (as BiPO₄) : 20·01 ; 20·05 ; 20·21 ; 20·13 per cent.

Summary and Conclusion.—A modification of the method for the gravimetric estimation of bismuth as phosphate is described, the directions being made sufficiently explicit to render the method easily applicable even if the operator has only an occasional determination to make. A process for the estimation of bismuth in ores is also described in detail ; in this the bismuth is removed by iron wire, copper, arsenic, and antimony by extraction of the sulphides with sodium cyanide and sulphide, and the bismuth sulphide converted into and weighed as phosphate.

As a result of our experience with the above process we endorse Moser's opinion of the phosphate method—viz., that in the great majority of cases it should prove the most advantageous method, and be accorded first place in the textbooks.

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DISCUSSION.

Mr. C. E. BARRS said he had considerable experience in the analysis of bismuth in metals and ores, usually carried out by weighing on a tared filter paper as bismuth oxychloride.

In the first place, it should be remembered that bismuth ores are often very complex, containing most of the common metals, and sometimes silver, gold, tungsten, molybdenum, tellurium, etc. He had had an opportunity of trying the separation of the bismuth with iron wire, and found that if carried out as directed, it is very useful as a satisfactory separation from lead and tin, both of which may lead to inaccuracies, whether the bismuth is estimated as phosphate or oxychloride.

The estimation of small quantities of bismuth—say, under 0·25 per cent.—may be made colorimetrically by precipitation as oxychloride, with subsequent solution of the precipitate in sulphuric acid, and dilution to known volume, *small* portions being taken, with the usual addition of potassium iodide, for the colour test in order that the tint shall not be too strong.

He asked the authors what their experience had been when treating the sulphuretted hydrogen precipitate with cyanide. He had found that in some cases, and more particularly if tin is present, a tendency for the bismuth sulphide to wash through the filter, but this seems to be prevented if the precipitate is given a final wash with hot water before washing back into the beaker for digestion with cyanide.



THE POSITION OF ANALYTICAL CHEMISTRY IN FRANCE.

By L. COFMAN, B.Sc.

(Read at the Meeting, November 3, 1920.)

LET me say at once that the present article deals solely with the professional side of the subject. Information regarding recent development and progress in Analytical Chemistry can be gathered easily from periodicals devoted to it, but one looks in vain for enlightenment concerning the conditions under which the profession is carried on. Such conditions are, nevertheless, important, even from a purely scientific point of view, for they react on the quantity and quality of original work done, especially in a branch of science where the bulk of the research is done by the rank and file.

EDUCATION.

One of the chief drawbacks to a satisfactory organisation of the analytical profession in France is the lack of an institution devoted entirely, or mainly, to its teaching. Chemistry is taught in the Science Departments and in a number of Institutes of Applied Chemistry attached to various Universities throughout the country. Such are the Institutes of Applied Chemistry of Paris, Lyon, Nancy, Lille, Toulouse, Clermont-Ferrand, the Electrochemical Institute of Grenoble, and the School of Chemistry of Bordeaux. Training in Analytical Chemistry is also given in the Higher Schools of Pharmacy and in the Municipal School of Physics and Chemistry of Paris. The Pasteur Institute, the National Agronomic Institute, and various other bodies have courses devoted to more or less specialised branches of Chemistry.

The training received by the student naturally varies greatly from place to place. At the Institute de Chimie Appliquée of Paris, a three years' course is provided, at the end of which a degree of "ingénieur-chimiste" is conferred on the candidate who has successfully passed the examinations, one at the end of each year. This Institute will no doubt play in future a more important part than it has done so far in chemical education. The premises which it occupies at present bear a not very flattering testimony to the esteem in which Applied Chemistry was held in France before the war. Students and apparatus are crowded together in a set of irregular buildings entirely unsuited for such use. Fortunately the lessons of the great war have not been all unheeded. The University of Paris, realising the great importance of Applied Chemistry in modern life, has decided to move the Institute from its old site at 3, Rue Michelet, to more spacious premises at the centre of the University grounds, Rue Pierre Curie. The new building is rapidly being completed, and provided with all necessary modern appliances and with every comfort for the students.

THE TITLES OF "CHIMISTE-ANALYSTE," "CHIMISTE-EXPERT," AND
"PHARMACIEN-CHIMISTE."

None of the educational bodies so far mentioned confers a degree in analytical chemistry, and the title of "chimiste-analyste" is fully as much abused in France as its equivalent is in England. The profession includes :

(i.) Chemists employed in the various laboratories in connection with the repression of frauds, or who occupy other official positions.

(ii.) Those placed on the list of experts to the Courts of Justice, who may be called as advisers in chemical matters.

(iii.) Analytical and consulting chemists with private laboratories, of whom there are relatively few.

(iv.) Pharmacists in business who undertake certain kinds of analytical work, especially of a biological character.

Following the insistent efforts of M. Cazeneuve, a law was passed in 1913* creating a title of "chimiste-expert du Government." The jury examining for this qualification is composed of teachers from the higher colleges which impart chemical training, and of members of a Permanent Technical Committee attached to the Ministry of Industry and Commerce. The law provides that special courses in analytical chemistry shall be instituted at certain Universities for training candidates already possessing an advanced knowledge of chemistry. The "chimistes-experts" exercising the profession previous to the legislation could obtain the new title on showing that they possessed an adequate qualification.

A recent bye-law (June, 1920) creates a post of "pharmacien-chimiste" in the Army, obtained by competition before an examining jury. The duties of the pharmacien-chimiste in peace-time include the analysis of foods and water supplies, and the assay of drugs and chemicals intended for military hospitals. In time of war they would be placed in charge of the Army Chemical and Toxicological Laboratories.

PROFESSIONAL ASSOCIATIONS, PERIODICALS.

There is much similarity between the French and the English chemical associations. Thus the Société des Chimistes-Experts de France corresponds to the Society of Public Analysts and other Analytical Chemists, and the Société des Chimistes Français has more than one point in common with the Institute of Chemistry as modified of late.

The first-mentioned Society aims at bringing together all those who are, or intend to be, nominated as "chimistes-experts" to the Courts of Law. Its official organ is the *Annales des Falsifications*. No special qualifications are required for membership. The Society was formed in 1912, and two years later had acquired over 500 members (the total number of chemists inscribed on the expert-lists is about 1,000). The following list gives the occupation of the members, and is interesting as showing approximately the proportion of different kinds of "analytical chemists."

* This Act, pending the promulgation of further bye-laws and regulations, has remained without practical application so far.

Government officials (chemists employed in municipal laboratories, etc.)	187
Military (pharmacists in the Army, Navy, and Colonies)...	66
Chemists, free	79
Pharmacists, civil	153
Various (industrials, doctors, engineers)	26
	Total 511

The other and more recent Association, the Société des Chimistes Français, was intended to include all chemists of French nationality. It is divided into an industrial and an analytical section, the members being further classed in different categories according to qualification and experience possessed. The *Annales de Chimie Analytique* has become the organ of the Society, which has at present some 460 members. Both the above societies are making numerous efforts to improve the social and material conditions of the analytical chemists.

THE ORGANISATION IN CONNECTION WITH THE REPRESSION OF FRAUDS.

Special legislation for the inspection of pharmaceutical and allied products existed in France at an early period (law of 21 Germinal, year XI.), but the first important modern legislation for the protection of the public from adulterated food was passed in 1905. This and subsequent bye-laws instituted various services for the purpose. The inspection of foodstuffs, beverages, and products for agricultural use is entrusted to officials nominated by the prefect (the county representative of the Home Office). These officials, of which there are some 1,000 throughout the country, occupy the ranks of police inspector, veterinary, county, or municipal agents. Their payment consists of a fixed small fee per sample taken in addition to travelling expenses. A certain number of Syndicates provide the authorities with experienced agents at their own expense. A chief inspector and fourteen county inspectors are charged with the control of this organisation.

Of the four identical samples sealed by the agent at an inspection, three are retained for subsequent reference and one is forwarded to an official laboratory. There are forty such laboratories maintained by different towns or counties. The Government also contributes to their upkeep by an amount depending on the number of samples analysed annually. This is fixed at 1.75 analyses per 1,000 inhabitants, which gives a total of 70,000 analyses carried out yearly in the whole of France.

The most important of the above-mentioned laboratories is the Laboratoire Centrale d'Analyse des Matières Alimentaires, of Paris. It is almost elegantly maintained, and consists of seven principal laboratories: two of these are devoted to the analysis of wine, carried out in a very thorough manner; two others are allotted to oils and fats, and the rest to other articles of food. An important part of the work consists in the testing of new analytical methods. If these are found more reliable than the ones already in use, circulars are sent out rendering their employment compulsory in all official analyses.

For the analysis of certain classes of products there exist special laboratories; for instance, the Laboratoire des Produits Résineux at the University of Bordeaux and

the *Laboratoire des Conservees de l'Armée*, the latter for the supervision of preserved meat and fish.

The surveyance of drugs and similar products is quite distinct from that of food. Pharmacies, dispensaries, and mineral water factories must be inspected yearly by a pharmacist nominated by the prefect on the recommendation of the local Faculty, or School, of Pharmacy. Drug stores, perfumeries, and other premises where medicinal or hygienic preparations are sold, are also subject to inspection. The analysis of suspected materials is carried out in laboratories attached to the Schools of Pharmacy, but placed under the supervision of the Ministry of Agriculture. Twenty such laboratories exist in France. The one in Paris is naturally the better organised. In addition to the analysis of drugs and chemicals there is a bacteriological section for the testing of the germicidal power of disinfectants, the adequate sterilisation of surgical dressings, etc. Further, a large amount of research is always in progress.

OTHER LABORATORIES.

The Municipal Laboratory, attached to the Prefecture de Police of Paris, devotes much of its activity to food analysis, biological examinations, and physiological testing for private individuals, at a nominal fee. It possesses more extensive accommodation, but is less conveniently arranged than either of the previously described laboratories.

At the Institute des Arts et Métiers there is a Department corresponding to the National Physical Laboratory of Teddington; its Chemical Section undertakes the testing and assaying of lubricating oils, alloys, and other industrial products.

Finally may be mentioned the *Laboratoire d'Essais de Semences* for the testing of seeds, and the Institute Pasteur for biological examinations.

It will be seen from the above-outlined description of French institutions that the work carried out in England by the Public Analyst is divided in France amongst several organisations. This division of labour has its disadvantages, and overlapping cannot be avoided, for instance, in the case of substances, such as olive oil, which are used both as food and for pharmaceutical purposes. But one undoubtedly good point is the official testing and unification of analytical methods, which would render impossible the promulgation of orders or regulations which cannot be carried out such as the recent Cocoa Order of the Food Controller, which set a limit to the amount of shell allowable, without indicating the method to be followed for its estimation.

DISCUSSION.

Mr. CRIBB said that the Report of the Eighth International Congress of Applied Chemistry contained a series of papers on the methods adopted for dealing with adulteration in Canada, the United States, and most of the Western European nations. From Mr. Cofman's paper it would appear that no noteworthy advances had been made in France, either in legislation or administration, since the date of that Congress.

The most important of the French laws relating to adulteration were, however, passed as recently as 1905, and, as there was a central executive, more likely to be

better and more uniformly administered than was possible with the worn-out laws in force in this country.

In two directions the French system was ahead of ours. They could take samples from the wholesale dealer and manufacturer, and the judge was allowed the assistance of a scientific expert in difficult cases. There was some reason for hoping that before long our more or less obsolete legislation would receive the attention of the Government, and such papers as that of Mr. Cofman and those to which he referred, would afford very useful suggestions.



STATUTORY STANDARD FOR GHEE.

By T. K. GHOSE.

IN view of the fact that ghee is an important article in the Indian dietary, and that it is difficult to purchase good ghee at the present time, it has become necessary to fix a standard for the genuine article. Ghee may be described as clarified butter—*i.e.*, butter fat from which moisture, casein, and other constituents, except butter fat, have been removed. The literature regarding the composition of ghee is very meagre. European textbooks deal with butter fat, whereas Indian ghee consists essentially of fat from buffalo milk, though there may be a slight admixture of butter from cow's milk. Richmond, Bolton, Revis, Trimen, and others have published work on ghee (*Analyst*, 1910, **35**, 343; 1911, **36**, 392; 1913, **38**, 242).

Investigations into the composition of Indian ghee have been carried on in the Corporation Laboratory for a number of years. Genuine samples of ghee, derived from individual animals as well as from herds, have been analysed, and the results recorded in Tables I. and II. Altogether 67 samples from individual buffaloes and 66 samples from herds were analysed. The minimum Reichert-Wollny value obtained was 29.2 and the maximum 42.0, the average being 34.5. The minimum of 29.2 was obtained in only one case out of 233, the next minimum being 30. In judging the quality of a commercial sample of ghee the Reichert-Wollny value 30 was adopted as the standard for purity. In October, 1918, a conference of the chemists of Calcutta was held, and the question of a standard for ghee discussed. It was decided that a sample of ghee would be declared as pure when the Reichert-Wollny value was 30 and above, and no sample would be regarded as adulterated under the Act unless it was below 28, and provided the other tests corroborated. The Corporation standard of 30 was reduced to 28, at the suggestion of one of the members of the conference, in consideration of accidental admixture with cows' ghee. It may be stated here that the English standard for butter fat, adopted by the Ministry of Agriculture and the Society of Public Analysts, is 24, though no statutory standard has been laid down.

TABLE I.

Results of Analysis of Samples of Buffalo Ghee made in the Laboratory from Milk of single Buffaloes.

Serial Number.	M.-pt.	Reichert-Wollny Value.	Saponification Value.	Serial Number.	M.-pt.	Reichert-Wollny Value.	Saponification Value.
1	35.5	38.6	237	35	36	35.8	236
2	35.5	39.3	238	36	37	30	230
3	36.5	37.5	235	37	37	33	235
4	36.8	31.8	230	38	36	35.8	237
5	34	31.5	230	39	36	38.8	237
6	35.3	34.5	234	40	36	38.9	239
7	36	34.6	233	41	37	37	236
8	37.3	35.4	234	42	36	35.6	235
9	36	30.5	229	43	36.5	34.3	232
10	35.5	33.1	231	44	37	32.7	231
11	38	33.4	232	45	36	36.2	235
12	36	42	239	46	36	35.9	236
13	37	36.2	236	47	35	40.6	240
14	35	39.9	239	48	35	39.9	240
15	35	41.9	238	49	35	39.2	238
16	34	40.6	238	50	35.5	39.5	239
17	34	39.1	237	51	36	37	238
18	35	30	229	52	36	36.9	234
19	36	34.8	234	53	36	34.3	233
20	35	42	240	54	37	34.6	233
21	35	36.2	236	55	36	34.5	234
22	36	31.3	230	56	35	36.5	235
23	36	33	231	57	35	35.2	235
24	37	34.6	232	58	35.5	36.1	236
25	37	34	233	59	36	34.1	235
26	36	35.7	235	60	34.5	38.7	236
27	36	35.7	234	61	36.5	31.7	230
28	37	30.4	227	62	36	34.5	233
29	35	32	233	63	36	34.8	232
30	36	31.8	231	64	37	30.1	227
31	36	31	231	65	37	30.2	226
32	36	32.7	232	66	36	30.8	229
33	37	36.1	235	67	36	30.8	227
34	36	37.2	237				

TABLE II.

Results of Analysis of Samples of Buffalo Ghee made from Butter produced from Mixed Milk of several Buffaloes. The Samples were collected from Dairies in different parts of India.

Serial Number.	Butyro Refractometer Reading at 40° C.	Reichert-Wollny Value.	Saponification Value.	Serial Number.	Butyro Refractometer Reading at 40° C.	Reichert-Wollny Value.	Saponification Value.
1	—	36.1	234	43	42	34.7	233
2	—	38	236	44	42	36.3	233
3	—	35	233	45	42	32.5	230
4	—	39	238	46	42	33.9	231
5	—	36	236	47	41	34.9	231
6	42	30.1	228	48	41	34.1	230
7	42	32.3	233	49	41.5	34.4	232
8	42	36.1	236	50	41	34.5	234
9	42	33.2	230	51	42	33.2	232
10	42	34.9	232	52	41	35.1	234
11	42	33	233	53	41	35	230
12	42	31.9	230	54	41.5	35	235
13	42	36.3	235	55	42	32.6	230
14	42	32.8	230	56	42	33.7	231
15	41.5	36.2	234	57	41	36.1	232
16	41.5	35.1	236	58	41	35.9	230
17	41	34.6	234	59	41	36	233
18	41.5	35	233	60	42	31.1	230
19	42	31.1	229	61	42	31.2	229
20	42	32.6	230	62	42	30.4	229
21	41.5	35.7	232	63	42	30.6	228
22	42	33.6	231	64	42	32.6	230
23	42	33.7	230	65	42	31.1	229
24	42	32.7	230	66	42	30	230
25	42	29.8	228	67	42	33.8	231
26	42	31.5	229	68	42	33.2	231
27	42	34.8	231	69	41	36.7	234
28	41.5	34.9	232	70	41	33.9	232
29	42	34	232	71	41	35.6	234
30	42	34	230	72	41	33.9	231
31	42	35.1	230	73	42	32.5	231
32	42	36	233	74	42	33.6	232
33	42	34.9	231	75	42	30.5	229
34	42	32.8	229	76	42	35.4	231
35	41.5	36.1	234	77	42	32.1	230
36	42	32.1	230	78	42	30	228
37	41.5	34.4	230	79	42	33.4	230
38	41.5	35.6	231	80	42	35.2	230
39	42	34.4	230	81	42	31.7	231
40	42	34.4	232	82	42	35.7	233
41	42	33.5	230	83	42	37.8	236
42	42	34	236	84	41	35.6	233

TABLE II.—*continued.*

Serial Number.	Butyro Refractometer Reading at 40° C.	Reichert-Wollny Value.	Saponification Value.	Serial Number.	Butyro Refractometer Reading at 40° C.	Reichert-Wollny Value.	Saponification Value.
85	41	35·3	235	126	42	32·9	230
86	41	36	233	127	41·5	30·5	229
87	41	33	230	128	41	30·9	228
88	42	34·1	231	129	42	30·5	227
89	42	36·2	232	130	41	32·1	230
90	42	32·6	230	131	42	31·3	230
91	42	35	234	132	42	29	227
92	42	30·1	229	133	42	30	229
93	40	30·7	230	134	41	31·2	230
94	42	36·3	235	135	42	31	230
95	41	35	232	136	42	31	229
96	41	37·6	236	137	42	30·3	227
97	41	36·7	236	138	40	31	230
98	42	32·8	230	139	42	31	220
99	42	32	230	140	42	31	230
100	42	38·2	236	141	42	30·9	227
101	40	29·9	228	142	41·5	31·5	229
102	42	29·6	227	143	42	31·5	228
103	42	29·4	228	144	42	30·5	229
104	42	29	227	145	42	32	230
105	40	32·6	230	146	40	31	229
106	42	33·3	232	147	41	32·3	230
107	41	29·2	230	148	41	34·6	231
108	42	29·1	227	149	41	36	231
109	40	31·3	229	150	42	30	227
110	42	29·6	228	151	42	31	230
111	42	32·1	231	152	42	34·8	233
112	42	32·2	231	153	40	31·9	230
113	42	31	230	154	42	32·3	231
114	42	32·8	230	155	42	32·8	230
115	42	34·5	232	156	42	33	233
116	42	35·4	234	157	41·5	30·4	227
117	42	33·9	233	158	42	29·2	226
118	42	33·3	232	159	42	30	226
119	42	32	230	160	42	30·6	228
120	40	32·3	230	161	42	30·5	227
121	42	30·1	229	162	42	31·8	227
122	42	32·6	230	163	42	30	226
123	42	32	229	164	41·5	31·8	228
124	41·5	31	230	165	41·5	30·5	226
125	42	32·6	231	166	42	30·7	226

NOTE.

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.

LOSS OF SPIRIT STRENGTH DUE TO EVAPORATION.

The following figures were obtained by measuring the loss of alcohol in spirit kept in a bottle lightly covered with a watch-glass, and having an exposed surface area of approximately 1 square inch. The determinations were made with spirit kept at 65° F. and 80° F. respectively, and the sp. gr. determined in each case at 60° F., the total volume of spirit being about 200 c.c.

65° F.				80° F.			
Date.	Sp. Gr.	Proof Spirit.	Loss.	Sp. Gr.	Proof Spirit.	Loss.	
		Per Cent.	Per Cent.		Per Cent.	Per Cent.	
16/4/20	954.05	= 67.42	—	954.05	= 67.42	—	
17/4/20	954.35	= 67.07	0.35	954.6	= 66.77	0.65	
19/4/20	954.4	= 67.01	0.06	954.7	= 66.65	0.12	
20/4/20	954.5	= 66.89	0.12	955.0	= 66.30	0.35	
21/4/20	954.6	= 66.77	0.12	955.3	= 65.94	0.36	
22/4/20	954.9	= 66.42	0.35	955.6	= 65.58	0.36	
23/4/20	955.1	= 66.18	0.24	955.9	= 65.21	0.37	
26/4/20	955.5	= 65.70	0.48	956.2	= 64.85	0.36	
27/4/20	955.7	= 65.46	0.24	956.5	= 64.48	0.37	
28/4/20	955.75	= 65.40	0.06	957.0	= 63.86	0.62	
29/4/20	955.8	= 65.33	0.07	957.5	= 63.23	0.63	
30/4/20	955.9	= 65.21	0.12	957.7	= 62.98	0.25	
1/5/20	956.2	= 64.85	0.36	958.2	= 62.35	0.63	
3/5/20	956.6	= 64.36	0.49	958.7	= 61.71	0.64	
4/5/20	956.8	= 64.11	0.25	959.2	= 61.07	0.64	
5/5/20	957.15	= 63.67	0.44	959.5	= 60.68	0.39	
6/5/20	957.5	= 63.23	0.44	959.8	= 60.29	0.39	
Total loss in twenty days			4.19	Total loss in twenty days			7.13

L. BRIANT.
H. W. HARMAN.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.**FOOD AND DRUGS ANALYSIS.**

Chemical Method for the Detection of a Previously Frozen Condition in Fruit. W. M. Dehn and M. C. Taylor. (*J. Ind. and Eng. Chem.*, 1920, 12, 977-979.)—Sugars exercise a protective action in fruit against freezing; cane sugar disappears in rotting fruit, and is transformed into invert sugar during the stages of ripening and over-ripening; cane and invert sugar are differently localised in the tissues of the fruit. The method described below makes use of the fact that freezing brings about a very rapid transformation of cane to invert sugar, resulting from the free mingling of cane sugar with inverting acids or invertase, or both. One half of the fruit to be examined is analysed directly, and the other half is frozen with ice and salt. Each sample of the fruit may be cut into halves, or, with very small fruit, two approximately equal samples may be set aside for the analysis. The directly analysed portion involves the estimation of invert sugar before and after hydrolysis; the frozen portion is also analysed for invert sugar before and after hydrolysis. In other words, the ratios of invert sugar to cane sugar before and after freezing are compared. In all cases the estimations are made by weighing the cuprous oxide formed after reduction. In the case of some peaches it was found that the freezing and thawing produced in fifteen hours three times as much inversion as storage for two weeks. By inspection of the values found for the percentage of inversion obtainable from the fruit before and after laboratory freezing, reliable conclusions may be drawn as to whether the fruit has already been subjected to cold storage or not, since the change brought about by freezing and storage will not be repeated by laboratory freezing, provided the first freezing was thorough and the laboratory freezing was not delayed too long. It will also be observed that not only cane sugar is inverted during rotting, over-ripening, rotting and freezing, but inverted sugar previously present or formed by inversion may be lost by chemical decomposition, or by metabolism of the plant, or by yeasts and moulds. The method takes advantage of divergencies in the largest components of the fruit, since inversion subtracts from the cane sugar concentration and adds to the invert sugar concentration.

H. F. E. H.

Detection of Incipient Putrefaction in Meat. R. Strohecker. (*Chem. Zeit.*, 1920, 44, 744.)—Six 300 c.c. Winkler oxygen flasks are charged each with 5 grms. of sample and filled up with water at 40° C. They are incubated respectively for 0, 2, 4, 6, 8 and 10 hours at 23° C. Absence of oxygen after four to six hours, as shown by the Winkler method, indicates incipient putrefaction. Meat beginning to decompose (5 grms. in a 60 c.c. oxygen flask filled up with a nitrate solution containing 3 mgrms. N_2O_5 per litre) completely destroys the nitrate in two to four hours of incubation. Fresh meat requires much longer. Nitrite solution gives similar results. A method depending on the detection of amino acids by the "ninhydrin" reaction gave

less satisfactory results; methods based on the presence of other decomposition-products, as also on the concentration of hydrogen ions in fresh and decomposed meat, were unsuccessful. For the detection of advanced putrefaction 10 grms. of the sample, with 1 c.c. of Schardner's methylene-blue solution, are incubated at 45° C. in a 60 c.c. oxygen flask, completely filled with water and sealed. Distinct decolorisation in forty-five minutes indicates putrefaction. O. E. M.

New Dye for the Recognition of Abnormal Milk. I. J. O. de Vries. (*Nederl. Tijdschr. voor Melk-hygiene*, 1920, 83; through *Chem. Zeit.*, 1920, 44, *Rep.* 245.)—V. Slyke and Baker suggest the use of bromocresol purple (dibromo-*o*-cresol-sulphonphthalein) for the rapid detection of abnormal milk. The author has investigated their statements. On the addition of 1 drop ($\frac{1}{20}$ c.c.) of the saturated aqueous solution of the dye to 3 c.c. of the milk, a coloration is developed: greyish-blue with normal milk, dark blue with a lower content of acid, and light green to light yellow with a higher content. The higher content of acid may be due to formation or addition of acids, acid salts, or formaldehyde, or too great a degree of pasteurisation. If the colour is bluish, the milk is from an inflamed udder, or is watered or deprived of cream, or alkali has been added. O. E. M.

Concentration of Hydrogen Ions in Milk. J. Tillmans. (*Chem. Zeit.*, 1920, 44, 744.)—Although a normal raw milk may show increasing acidity by the Soxhlet and Hempel methods, its PH' remains constant. A normal milk will not pass the alcohol test if its PH' reaches 6 on the Sørensen scale. The alizarine test of Morres is nothing more than the alcohol test, with simultaneous estimation of PH'. It was shown that the acidity of fresh milk is due not to lactic acid, but to primary and secondary phosphates. The behaviour of boiled milk differs from that of fresh; either it undergoes a delayed lactic acid fermentation, or putrefies with production of alkaline bodies. Measurement of PH' was useless as a means of detecting neutralised milk, but specific conductivity determinations revealed addition of bicarbonate. Preservatives did not affect the PH', nor did removal of cream or addition of water up to 60 per cent. The reductase of Schardinger was the most sensitive to increase of PH', catalase the least, while the Storch enzyme occupied an intermediate position. Tubercular and other diseases were not revealed by abnormal figures, and milk from goats and sheep gave the same results as cows' milk. O. E. M.

Estimation of Moisture in Beet-Sugar Factory Products. V. L. Aikin. (*J. Ind. and Eng. Chem.*, 1920, 12, 979-981.)—A large number of results for moisture estimations of beet-sugar products are recorded, in which the method of mixing with sand is adopted. It is recommended that only sand be used which will pass a screen with 0.25 mm. perforations, the sand being previously digested in hot hydrochloric acid, washed and ignited. Twenty to twenty-five grms. of the sand are dried and weighed just previous to the estimation. Not more than 1 gm. of the material to be dried is then added, together with 1 c.c. of water; the dish is placed on top of the oven until warm, and the contents then mixed for three minutes until a perfectly homogeneous mixture is obtained. The whole is dried at a temperature of 105° C.

for six hours, and re-weighed until the loss in weight, after heating for a period of one hour, is less than 0.1 per cent. All weighings are made as soon as the temperature of the desiccator is within 2° C. of the temperature of the balance. Any determination which does not check within 0.2 per cent. should be repeated. H. F. E. H.

Chemical Composition of Natural Rice and of Italian Polished Rice.

G. Issoglio. (*Estratto dagli Atti della Reale Accademia delle Scienze di Torino*, 1919, **53**, 731-744; through *Int. Rev. Sci. and Prac. of Agric.*, 1919, **10**, 877-8.)—The author, with a view to contributing to the study of the most rational utilisation of cereals, has undertaken numerous investigations on the qualities of the following kinds of rice, specially with regard to their phosphatic constituents :

I. *Riso svestito rosso* (red hulled rice), composed of homogeneous grains covered with a red skin and perfectly white inside ; the red colour is fairly resistant to acids, but is entirely removed by alkalis, specially ammonia. II. *Riso svestito bianco* (white hulled rice), which stands cooking well, giving out the characteristic smell of this cereal. III. *Riso brillato perla* (polished pearl rice), formed of small, transparent, heavy grains, slipping easily over each other ; stands cooking well, but does not give off any agreeable odour. IV. *Riso mercantile raffinato* (refined commercial rice), white, homogeneous, stands cooking badly, gives off no agreeable odour, insipid. V. *Riso cremonese bianco* (white Cremona rice), similar to IV. VI. *Riso cremonese striato di rosso* (red striped Cremona rice), used with V. in experiments by Professor Guareschi, in the course of which pigeons and fowls preferred white Cremona rice. VII. *Riso bianco mercantile* (white commercial rice), kind placed on the market in 1918 ; has the organoleptic characters of milled rices with negative characters of appetibility.

The following table shows the results of the analysis of each of these 7 qualities :

	I	II.	III.	IV.	V.	VI.	VII.
Specific gravity ...	1.444	1.426	1.458	1.430	1.428	1.438	1.435
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Water ...	12.74	13.22	13.65	13.28	12.88	12.65	13.37
Ash ...	1.43	1.24	0.87	0.53	0.59	0.68	0.57
Fats ...	7.15	2.08	0.64	0.58	0.65	0.77	0.68
Nitrogen ...	1.53	1.37	1.25	1.27	1.30	1.51	1.28
Crude albuminoids ...	8.57	7.81	7.12	7.23	7.41	8.55	7.29
Cellulose ...	1.21	1.15	0.18	0.22	0.25	0.32	0.24
Pentosans ...	0.35	0.30	0.05	0.08	0.12	0.18	0.10
Starch ...	73.55	73.20	77.19	78.08	78.10	76.95	77.75
Total phosphoric anhydride P ₂ O ₅ ...	0.84	0.68	0.26	0.24	0.38	0.46	0.29
Phytinic phosphoric acid ...	0.485	0.435	0.058	0.055	0.152	0.188	0.082
Lecithinic phosphoric acid ...	0.010	0.008	traces	traces	—	—	—
Phosphoric acid proportional to 100 parts of ash ...	58	55	30	45	62	67	50

The difference between hulled rice and milled rice is evident: the former is fairly rich in both mineral and organo-mineral phosphatic compounds, while the latter is poor in these constituents. Thus the amount of phosphoric pentoxide may be as high as 5 grms. per kgrm. in hulled rice, while it does not reach 1 grm. per kgrm. in refined polished rice; lecithinic phosphoric pentoxide is entirely absent from the milled rice, while in hulled rice, in the form of phospholecithin, it is as much as 0.1 grm. of lipoid per kgrm.

It is also desirable to draw attention to the chemical affinity which exists between the phosphate derivatives and albuminous matter, from which comes the constant fact that rice rich in phosphates is also rich in albuminoids.

COMPARATIVE ANALYSIS OF 1 GRM. OF WHOLE RICE AND OF 1 GRM. OF POLISHED RICE.

	Whole Rice.	Polished Rice.
	Per 1,000.	Per 1,000.
Phosphorus	2.678	0.890
Potassium	2.469	0.690
Magnesium	0.865	0.270
Sodium	0.434	0.210
Calcium	0.266	0.080
Iron	0.144	0.070
Chlorine	0.115	0.150
	6.871	2.287

All this confirms the argument of Professor Guareschi that polishing decreases the valuable nutritive elements in rice.

Estimation of Sweetening Power. R. Pauli. (*Chem.-Zeit.*, 1920, **44**, 743.)

—This estimation has not hitherto been based on accurate methods, and the author outlines an improved experimental psychological procedure, including the interpretation of the results obtained. The method depends on the comparison by tasting of a number of pairs of solutions, one member of each pair being a sugar solution of unvarying concentration, the other member one of a series of saccharin solutions varying by equal stages within fixed limits above and below the sugar solution in sweetness. The conditions of test must be rigidly laid down and observed, and a number of observers is necessary to eliminate personal error. O. E. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Aminoethanol and of Choline appearing in the Hydrolysis of Phosphatides. P. A. Levene and T. Ingvaldsen. (*J. Biol. Chem.*, 1920, **43**, 355.)—The following method is a modification of the one devised by Thierfelder and Schulze (*Zeitsch. physiol. Chem.*, 1916, **96**, 296). The ether-soluble acetone-insoluble egg lipoids are freed from white matter and hydrolysed by boiling with 3 per cent. sulphuric acid for eight hours. After filtration the sulphuric acid is precipitated by barium hydroxide, the filtrate concentrated in vacuo and basic lead acetate added

until no further precipitation occurs. The precipitate is filtered off, and excess of lead removed from the filtrate by sulphuretted hydrogen, when hydrochloric acid is added, and the solution evaporated with water repeatedly to remove acetic acid. The residue is then extracted with absolute alcohol, filtered, evaporated, and the residue of mixed bases dissolved in water and diluted to 25 c.c. Of this solution 15 c.c. are evaporated on a water-bath to a small volume, mixed with excess of calcium oxide, and extracted by boiling for a few minutes with three 75 c.c. portions of dry acetone. After filtration the combined extracts are acidified with hydrochloric acid and evaporated in vacuo, the residue being dissolved in water and evaporated to 10 c.c. Excess of strong hydrochloric acid is then added to 9 c.c. of the aqueous solution, followed by 1.9 grms. of gold chloride. On standing in a desiccator over sulphuric acid large crystals separate, which, after recrystallisation from dilute hydrochloric acid, melt at 190-192° C., and contain 49.11 per cent. of metallic gold, the theoretical amount for aminoethanol gold chloride being 49.17 per cent. The residue from the acetone extract is treated with water, filtered, the excess of hydrochloric acid and of calcium being removed by silver oxide and carbon dioxide respectively, treated with alcoholic picric acid solution, concentrated in vacuo, and allowed to cool in an ice chest. The precipitate of choline picrate is filtered off, and recrystallised from water, yielding long needles, having a melting point of 241-242° C., and containing 16.75 per cent. of nitrogen, comparing well with the theoretical value of 16.85 per cent.

T. J. W.

Estimation of Diastatic Power. E. I. Rosenblum. (*J. Soc. Chem. Ind.*, 1920, 39, 311-313T.)—The difference in hydrogen ion concentration of starches due to the presence of acid salts renders the estimation of diastatic power untrustworthy. The diastatic power is at a maximum when the digesting solution has a faintly acid reaction towards methyl-red but is alkaline towards methyl-orange. This condition may be secured by the addition of 0.5 per cent. of ammonium dihydrogen phosphate to the solution. The diastatic power is estimated as described by Harrison and Gair (*Pharm. J.*, 1906, 94, 6). Twenty c.c. of a solution, containing 0.1 gm. of malt extract, are added to a starch solution, containing 1 gm. of dry starch per 80 c.c. of water, which has been prepared by boiling and cooling to 46° C.; the mixture is kept at 46° C. for thirty minutes, a few drops of 20 per cent. sodium hydroxide solution are then added, the mixture diluted to 100 c.c., and titrated with Fehling's solution. This titration is termed T_D ; a further titration of a 1 per cent. malt extract alone gives T_M , a measure of the sugar originally contained in the extract. The formula $9531 \left(\frac{1}{T_D} - \frac{1}{T_M} \right)$ gives the diastatic power.

W. P. S.

Estimation of the Saccharogenic Power of Diastatic Preparations. K. Oshima. (*J. Ind. and Eng. Chem.*, 1920, 12, 991-993.)—The author criticises adversely the Lintner method for the estimation of diastatic power, together with the Ling modification as adopted by the Institute of Brewing. He states that in the Ling modification there is no accurate agreement with the Lintner scale, since the quantity of enzymes and production of sugar do not give an exact proportion; par-

ticularly is there a noticeable difference when the production of sugar is too much or too little. To obviate these difficulties, Sherman and his associates proposed a method which depends upon the gravimetric estimation of copper oxide. The method is, however, for that reason too troublesome for routine factory work. The author then describes his own method as follows: In the case of a solid sample, it must be first extracted with distilled water for three hours at room temperature. From 1 to 10 grms. of the sample are usually extracted in 100 c.c. of water. If the enzyme is too concentrated, the solution is further diluted with a known amount of distilled water. Ten c.c. of the extract or the diluted extract are put into a flask containing 100 c.c. of 2 per cent. soluble starch solution, which has been placed in a water-bath kept at 40° C., the contents of flask are mixed, and the flask is allowed to remain in the bath for thirty minutes. At the end of that period, 10 c.c. of $\frac{N}{8}$ sodium hydroxide are added at once to stop further enzymatic action. Graduated quantities of the digest are then poured into a series of test tubes, each containing 5 c.c. of Fehling's solution; and the tubes are placed in a boiling water-bath for ten minutes, the tubes being shaken once during that period. The smallest quantity of the digested starch solution which just reduces 5 c.c. Fehling's solution is determined. It may be more convenient to use at first only two or three tubes containing quantities of the digest within wide regions of variations, so as to determine the approximate concentration of the digest to be used. The Lintner value according to tables and formulæ appended was then calculated from the amount of digested starch solution which will just reduce 5 c.c. of Fehling's solution. Experiments are described from which a culture of *Aspergillus oryzae* and an extract of malt were used, and it is claimed that the method is very convenient for experimental studies on various diastatic products, taka diastase, etc. It may also be used for the study of the influence of alkalinity, acidity, antiseptics, salts, etc. Results of experiments are also described in which a temperature of 50° C. is used for the enzyme action.

H. F. E. H.

NOTE BY ABTRACTOR.—The above method appears to possess no advantages whatever over the Lintner "tube" method or the Ling modification thereof, nor does the author appear to be aware that the amount of maltose formed is only a linear function of the diastase, when means are taken to keep the starch conversion within the limits of Kjeldahl's law of proportionality. This is provided for both in the Lintner method and in Ling's modification.

Colorimetric Estimation of Histidine in Protein. M. T. Hanke and K. K. Koessler. (*J. Biol. Chem.*, 1920, **43**, 527.)—From 1 to 3 grms. of protein are mixed with 60 c.c. of 20 per cent. hydrochloric acid and hydrolysed by boiling for twenty-eight hours. Volatile matters are removed by distillation *in vacuo* at 60° C., and the residue finally dried *in vacuo* at 80° C. for one hour. The residue dissolved in 100 c.c. of water is treated with excess of calcium oxide and 50 c.c. of 90 per cent. alcohol, and then distilled *in vacuo* at 40° C. to remove ammonia, alcohol, etc. The residual solution is filtered and the precipitate washed with large volumes of hot water. To the alkaline filtrate is added a slight excess of hydrochloric acid, the liquid evaporated to dryness, dissolved in 18 c.c. of 37 per cent. hydrochloric acid,

diluted to 100 c.c., and heated on a water-bath; 100 c.c. of hot aqueous 15 per cent. phosphotungstic acid are added, and the mixture allowed to digest for thirty minutes. It is then left to cool slowly, and finally for three days in an ice-bath. The cold solution is filtered rapidly, the residue well washed with 200 c.c. of an ice-cold solution containing 3.3 grms. of hydrochloric acid, 15 grms. of phosphotungstic acid, and previously saturated with histidine phosphotungstate. The precipitate is dissolved in just sufficient 3*N* sodium hydroxide, filtered, and diluted to 1,000 c.c. This solution is used for the colorimetric estimation as previously described (*J. Biol. Chem.*, 1919, **39**, 497).

Histamine and tyramine both yield a similar pink colour with *p*-phenyldiazonium-sulphonate, but are never present in sufficient quantity in non-putrid protein matter to affect the estimation of histidine.

Several tables are furnished giving results obtained by the above method with various proteins, and the effect of the presence of other amino-acids upon the estimation.

T. J. W.

Colorimetric Estimation of Histamine in Proteins. M. T. Hanke and K. K. Koessler. (*J. Biol. Chem.*, 1920, **43**, 543.)—The dry solid is hydrolysed without preliminary treatment, but hydrated tissues are mixed with sufficient alcohol to give a final alcoholic strength of 75 per cent., a few drops of acetic acid added, and the mixture heated on a water-bath for two hours. After cooling and filtering, the residue is washed with 95 per cent. alcohol, and the alcohol evaporated from both residue and filtrate. The dry solid is hydrolysed, purified, and converted into histamine phosphotungstate by the methods described for histidine (see previous abstract). The phosphotungstate precipitate is suspended in a large volume of hot water, treated with excess of saturated barium hydroxide solution, and digested for one hour on a water-bath. After cooling, the solution is filtered, the filtrate freed from excess of barium by the careful addition of $\frac{N}{2}$ sulphuric acid, the mixture filtered hot, and evaporated to dryness. From the residue, by a complicated series of reactions and colorimetric determinations, for which the original paper must be consulted, the percentage of histamine is determined. Experiments are described, showing that histamine is quantitatively precipitated by phosphotungstic acid, by silver nitrate and by baryta, and is extracted to the extent of 96 per cent. by a mixture of chloroform and methyl alcohol.

T. J. W.

Adamkiewicz Protein Reaction and a New Colour Test for Glyoxylic Acid. W. R. Fearon. (*Biochem. J.*, 1920, **14**, 548.)—The work described in this paper was designed to find a satisfactory method for the colorimetric estimation of tryptophane, and the nature of the aldehydes concerned in the Hopkins-Cole and Rosenheim reactions in order to decide the glyoxylic acid-formaldehyde controversy.

The reagent used for the glyoxylic acid test is a 1 per cent. solution of pyrogallol in pure concentrated sulphuric acid free from nitrous contamination. This will keep in satisfactory condition for a few days only. On adding a trace of glyoxylic acid and gently warming, a deep blue colour gradually develops, which

becomes carmine on dilution with water, and again blue on the addition of sulphuric acid in excess. With the aliphatic aldehydes the reagent gives various shades of red.

The author concludes as a result of this work that the Hopkins-Cole test is due to glyoxylic acid, while the Rosenheim reaction depends upon the presence of formaldehyde.

The following qualitative test for tryptophane and the indoles is given: the reagent is a 10 per cent. solution of salicyl aldehyde in alcohol free from acetone. To the liquid under examination is added excess of strong hydrochloric acid, 4 or 5 drops of the reagent, and the tube is gently warmed for a few minutes. One drop of 10 per cent. hydrogen peroxide is then added and the warming continued, when tryptophane yields an intense blue colour, skatole gives a deep purple, while indole produces a bright carmine. Care must be taken not to add excess of hydrogen peroxide or the colours will be destroyed.

T. J. W.

Proximate Analysis of Hardwoods: Studies on *Quercus Agrifolia*.

W. H. Dore. (*J. Ind. and Eng. Chem.*, 1920, **12**, 984-987.)—In a previous paper (*ANALYST*, 1919, **44**, 299) the author has described methods for the analysis of wood, and given the results of three coniferous woods and two hardwoods. It was then shown that the procedure succeeded with coniferous woods, but not with hardwoods, owing to the fact that König's 72 per cent. sulphuric acid method for lignin does not give reliable results for the woods of the broad-leaved trees. In the present paper the author employs the gaseous hydrochloric acid method of König and Becker (*J. Soc. Chem. Ind.*, 1919, **38**, 530A). This method is described in detail, for which the original paper should be consulted. The composition of oak wood was found to be that given in the following table, the figures in all cases being the average of two or more determinations.

COMPOSITION OF LIVE OAK WOOD (*Quercus Agrifolia*).

Constituent.	Air-dry Basis. Per Cent.
Loss on drying	4.20
Benzene extract	0.50
Alcohol extract	4.33
Soluble in cold water	3.66
Soluble in cold 5 per cent. sodium hydroxide	18.71
Cellulose	45.48
Lignin	20.25
Pentosans not otherwise accounted for	1.89
Mannan (residual)	—
Galactan (residual)	1.49
Total	100.51

In this table the first five groups of substances represent extraneous matter removed from the wood in the course of the purification process, while the last five groups represent substances contained in the residual purified tissue. The amounts obtained for each of the first five groups vary considerably with the manner in which the determinations are carried out, but the weight of purified residue remaining is in all

cases about the same. The purification treatment employed for the tissue of coniferous woods (successive extraction with benzene and alcohol) was supplemented with digestion in cold water and cold 5 per cent. sodium hydroxide solution. These treatments remove all adventitious substances, but do not injure either the cellulose or the lignin. The modified procedure conforms to the requirements of a satisfactory analytical scheme, in that it accounts for all of the material of the wood, yields the maximum amount of main constituents free from impurities or degradation products, avoids overlapping of constituents, and overlooks no constituents of importance.

H. F. E. H.

ORGANIC ANALYSIS.

Estimation of Cineol Essential Oils. C. Kleber and W. F. von Rechenberg. (*J. prak. Chem.*, 1920, 101, 171-176.)—A method which may be used for the estimation of cineol in eucalyptus oil depends on the determination of the solidifying point of the oil. Pure cineol solidifies at 1.2° C. The following table gives the solidifying points of eucalyptus oil containing various amounts of cineol :

Cineol, per Cent.	Solidifying Point.	Cineol, per Cent.	Solidifying Point.
100	1.2°	82	- 8.0°
98	0.3°	80	- 9.2°
96	- 0.7°	78	- 10.3°
94	- 1.7°	76	- 11.5°
92	- 2.7°	74	- 12.8°
90	- 3.8°	72	- 14.1°
88	- 4.8°	70	- 15.4°
86	- 5.8°	68	- 16.7°
84	- 6.9°		

Normal eucalyptus oils do not contain less than 70 per cent. of cineol; in the case of oils containing less than this quantity it is advisable to add 50 per cent. of pure cineol before determining the solidifying point, and to make a correction subsequently for the quantity added.

W. P. S.

Estimation of Creatinine. E. Vautier. (*Ann. Chim. anal.*, 1920, 2, 300-305.)—The following modification of the picric acid method is suitable for the estimation of creatinine in "concentrated bouillon" and similar preparations. From 0.25 to 3 grms. of the substance, according to the probable amount of meat extract present, is evaporated with hydrochloric acid, and the residue taken up with boiling water, and filtered through a moistened filter to remove fat, etc. The filtrate is again evaporated to dryness on the water-bath, with the addition of a few drops of concentrated hydrochloric acid, and the mass frequently stirred to prevent the crystals of sodium chloride which separate enveloping some of the liquid containing creatinine. The dry or nearly dry residue is cooled, treated with 10 c.c. of water, and then with 15 c.c. of picric acid solution (1.2 per cent.) and 8 c.c. of 10 per cent.

sodium hydroxide solution, shaken, and, after five minutes, made up to 500 c.c., and the creatinine estimated colorimetrically. For this purpose the colour may be compared with that of a $\frac{N}{2}$ solution of potassium dichromate in a layer of 8 cm. in depth. This coloration is matched approximately by the following amounts of creatinine :

Creatinine,																			
mgrms. ...	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	8.0	9.0	10.0	12.0	15.0	20.0			
Depth of																			
liquid, cms.	18.0	14.5	11.5	9.9	9.5	8.6	7.7	7.2	6.7	6.3	5.5	4.8	4.2	3.6	2.8	2.2			

The intermediate values may be obtained by interpolation.

C. A. M.

Microchemical Identification of Cyanic Acid. R. Fosse. (*Comptes rend.*, 1920, 171, 635-637; 722-723.)—Cyanates, when heated with water and ammonium chloride, yield urea, which may be identified by means of xanthhydrol. Characteristic crystals are obtained when 0.005 gm. of silver cyanate is boiled with 5 c.c. of water and the solution then cooled. A blue coloration is obtained when 1 mgrm. each of silver cyanate, potassium chloride, and crystallised cobalt acetate are ground together in a porcelain basin; the colour is destroyed by water, but re-appears when alcohol is added. Cobalt thiocyanate has also a blue colour, but it is insoluble in amyl alcohol, is not decolourised by dilute acids, and gives a red coloration with ferric salts, and may thus be distinguished from the blue potassium cobalticyanate.

W. P. S.

Sensitive Form of the Lieben Iodoform Reaction. R. Kunz. (*Zeitsch. anal. Chem.*, 1920, 59, 302-303.)—The test consists in warming 10 c.c. of the solution between 50°-60° C. with 1.5 to 2 c.c. of 1 in 10 sodium hydroxide solution, about 0.15 gm. of potassium iodide, and 0.2 gm. potassium persulphate. A solution containing 1 drop of alcohol in 100 c.c. water gives a turbidity after ten minutes.

O. E. M.

Distillation of Petroleum, Petroleum Residues, or other Oils immiscible with Water. H. Offermann. (*Chem. Zeit.*, 1920, 44, 773-774.)—Wet steam is used at ordinary pressure instead of the superheated steam, with or without reduction of pressure, prescribed by Engler. Cracking is thus avoided, and laboratory and refinery results are brought to the same level. Steam from a conical flask is conveyed upwards, with partial condensation, to a dip tube passing through the tubulure to the bottom of a weighed retort, the neck of which rises to an adapter attached to a Liebig condenser. The charge of oil, 500-1,000 grms., having been heated to boiling, the steam is passed through at such a rate that at the beginning the distillate consists of 25 per cent. water, and 75 per cent. oil, in the middle 50 per cent. of each, and towards the end 75 per cent. water and 25 per cent. oil. Comparative results from a petroleum residue show that lower sp. gr. and flash-point of the fractions produced, a higher proportion of pitch and loss, and production of pitch of higher melting-point and greater insolubility in benzene, accompany the use of steam superheated to 250-400° C.

O. E. M.

INORGANIC ANALYSIS.

Estimation of Carbon Dioxide in Alkali Bicarbonate containing Carbonate. W. Hartmann. (*Zeitsch. anal. Chem.*, 1920, **59**, 289-297.)—The substance equivalent to 0.1-0.3 gm. bicarbonate, together with small pieces of pumice and short lengths of 2 mm. bore glass tubing, is placed in a boiling-tube, 15 × 3 cm., closed by a 4-hole rubber stopper. This carries a 200° C. thermometer and an inlet tube, both reaching the bottom, and also a tap-funnel and an outlet tube. Air freed from carbon dioxide by bubbling it through potash solution is drawn through the apparatus, and thence in succession through cooling bulbs, strong sulphuric acid in a washing bottle surmounted by a water-jacketed tube, a calcium chloride tube, a weighed potash absorption apparatus, and a calcium chloride tube, to a laboratory pump. After admission through the tap funnel of 7 c.c. of 70 per cent. glycerol for each 0.1 gm. of carbonate taken, the contents of the tube are heated for ten minutes at 117° C. for sodium bicarbonate or 114° C. for potassium bicarbonate. Air is now drawn through for thirty minutes at 90-100° C., and the potash bulb weighed. For the normal carbonate the temperature is raised to 180°-190° C. for five minutes, 2 drops of water are added, and heating continued for five minutes. Air is drawn through for thirty minutes at 150° C. before weighing. Any ammonia present can be estimated by rendering the sulphuric acid alkaline and distilling. Anhydrous normal carbonate, especially sodium carbonate, requires heating at 180-190° C. for forty minutes, with addition of 2 drops of water every two minutes. The table of results shows that although normal carbonate, especially potassium carbonate, gives off considerable quantities of carbon dioxide at 115-120° C., the bicarbonates at these temperatures give results agreeing with theory. With bicarbonates, high and low results respectively are produced by the admixture of salts which promote dissociation, and an excess of normal carbonate. The method is only applicable to bicarbonates alone or in admixture with non-electrolytes.

O. E. M.

Determination of Carbon Monoxide in Air contaminated with Motor Exhaust Gas. M. C. Teague. (*J. Ind. and Eng. Chem.*, 1920, **12**, 964-968.)—Motor exhaust gas is found to contain the following constituents: carbon dioxide, carbon monoxide, oxygen, hydrogen, methane, traces of unsaturated hydrocarbons, and some unburned gasoline; there are also present water and varying amounts of oil-smoke particles. The iodine pentoxide method for determining low concentrations of carbon monoxide was investigated with reference to its use with dilute motor exhaust gas. The form of apparatus hitherto used was found to give appreciably higher results, owing to the presence of small amounts of unburned gasoline. A new apparatus was therefore devised, and is fully described and illustrated. All the interfering gases are first removed at the temperature of liquid air (-190° C.); at this temperature liquid gasoline becomes a glass-like solid, while all the water, carbon dioxide, and probably all the unburned hydrocarbons are also condensed. The method is found quite satisfactory for determining carbon monoxide in small quantities in

the presence of gasoline vapour. A portable apparatus is also described and illustrated, which permits a determination to be made in fifteen minutes with an accuracy of 0.003 to 0.005 per cent. carbon monoxide; this may be of use in exploring vehicular tunnels, garages, and other enclosed spaces. H. F. E. H.

Separation of Iron from Aluminium by Precipitation as Prussian Blue. H. Hale and G. O. Burr. (*J. Amer. Chem. Soc.*, 1920, **42**, 2056-2058.)—The quantitative separation of iron from aluminium by precipitation as Prussian blue is not practicable, owing to the aluminium interacting with the excess of ferrocyanide to form a jelly which cannot be manipulated. The addition of acid retards, but does not prevent the formation of the jelly. Coagulation of the Prussian blue is effected by the addition of various salts, especially aluminium chloride; but, even after coagulation, quantitative filtration is impossible, although good results were obtained by centrifuging. C. A. M.

Volumetric Estimation of Mercury, Iron, and Vanadium in the Same Solution. G. Hinard. (*Ann. Chim. anal.*, 1920, **2**, 297-299.)—I. *In the Highest Stage of Valency.*—The solution of the three metals in dilute sulphuric acid is treated with hydrogen sulphide, the mercuric sulphide separated and oxidised with bromine-water, the mercury precipitated as hydroxide, which is dissolved, without filtration, in potassium cyanide solution, and the excess of cyanide titrated with standard silver nitrate. The filtrate from the mercuric sulphide is boiled, and oxidised with bromine-water, the iron precipitated with potassium hydroxide, redissolved in hydrochloric acid, and reprecipitated. It is then dissolved in warm sulphuric acid, the solution cooled, treated with excess of potassium iodide, and the iron estimated by titrating the liberated iodine with standard thiosulphate solution. Another portion of the original solution is rendered just alkaline, the incipient precipitate redissolved in a slight excess of sulphuric acid, and the iron and vanadium estimated together iodimetrically, the titration with thiosulphate being continued in this case until the liquid becomes blue. The vanadium is then found by difference. II. *In Different Stages of Valency.*—The total quantities of the three metals are estimated as described above. Another portion of the original solution is treated with sodium chloride, which precipitates mercurous salts, and the mercuric salts are then precipitated with hydrogen sulphide from the filtrate. *Iron and Vanadium.*—(a) *Absence of ferrous salts:* Vanadic acid is estimated iodimetrically as described, and a deduction made for the amount of thiosulphate corresponding to the iron already estimated. The vanadium, present as a vanadyl salt, is obtained by difference. (b) *Presence of ferrous salts, absence of vanadates:* The solution is treated, without prior oxidation with bromine-water, with potassium iodide. In this case the liberated iodine corresponds to the ferric iron. The ferrous iron is found by difference, whilst the total vanadium has been already determined. C. A. M.

Determination of Nitrates in Soil. A. L. Whiting, T. E. Richmond, and W. R. Schoonover. (*J. Ind. and Eng. Chem.*, 1920, **12**, 982-984.)—The method

suggests modifications in the Devarda method, and depends upon the oxidising action of sodium peroxide and the completeness of extraction of the nitrate with hydrochloric acid. The water-free samples from moisture determinations are placed in 400 c.c. shaker bottles, and 300 c.c. of approximately 0.5 per cent. hydrochloric acid are added. The mixture is then shaken in a mechanical shaker for one to three hours, and allowed to settle overnight. Five grms. of sodium peroxide are placed in a 800 c.c. Kjeldahl flask. A 200 c.c. aliquot part of the acid soil extract is blown off into a graduated flask, then poured on the peroxide, and the contents of the flask boiled down to 20 to 25 c.c., or, if urea is present, to complete dryness. Two hundred c.c. of nitrogen-free distilled water are now added, together with 0.5 gr. of Devarda's alloy (50 per cent. aluminium, 45 per cent. copper, and 5 per cent. zinc), and the mixture distilled for forty minutes, and collected in standard sulphuric acid. Sodium hydroxide of a strength such that 1 c.c. = 0.5 mg. of nitrogen is used for titration. Rosolic acid or methyl red is the best indicator. Hydrochloric acid was selected as the best acid for extraction, a solution of 0.2 per cent. strength being very satisfactory where soils are not heavily treated with carbonates. Between the limits 0.2 and 1 per cent. the strength of the acid was found not to affect results. By the use of sodium peroxide an alkaline solution is left which expels the ammonia and leaves the proper conditions for reduction with Devarda's alloy. The method determines only nitrate nitrogen, as confirmed by precipitation of the nitrate in the soil extracts with 10 per cent. nitron solution in 5 per cent. acetic acid, followed by an analysis of the crystals of nitron nitrate and the filtrate from the same. Slightly lower results are given by the Devarda method, when modified as described, than by the nitron method. The method has been successfully used on thousands of soil samples, and also been applied with satisfactory results to crop samples.

H. F. E. H.

Examination of the Methods used in Estimating Phosphorus in Iron and Steel, Ores and Slags. H. Kinder. (*Chem. Zeit.*, 1920, 44, 692-693.)—The magnesia method is slow, and for small quantities of phosphorus, inaccurate. The molybdate method was examined, and the results of Finkener, who dries the precipitate at 105° C., and of Meinecke, who ignites to about 450° C., were confirmed. The presence of 1 per cent. of copper, 20 of nickel, or 5 of cobalt, is without effect, while 5 per cent. of chromium was doubtful. Tungsten takes down phosphorus, and the precipitate may be dried, dissolved in hydrochloric acid, oxidised with potassium chlorate, and the phosphorus determined as usual in the diluted filtrate. Molybdenum precipitates must be examined for phosphorus. Solutions containing vanadium should be reduced with ferrous sulphate or sodium sulphite before precipitation. Titanium causes the precipitation of some phosphorus; the precipitate is fused with soda, and phosphorus estimated in the extract. Arsenic is partially precipitated as molybdate, unless the concentration of the nitric acid or of the solution is increased, or the precipitation carried out below 65° C. Reduction and volatilisation of arsenious acid by means of hydrobromic acid is the best method. For ores or slags the weight taken for analysis should be 5 or 10 grms. for 0.1 per cent. phosphorus, down to 0.3 or 0.5 gm. for 2 per cent. The liquid for precipitation should not exceed 150 c.c., and

contain not over 5 c.c. strong nitric acid. The temperature should be 60-70° C., and 50-60 c.c. molybdate solution should be used. Settling, hastened by shaking, must be complete before filtering. The reagents should be measured, and a control performed with them in glass vessels similar to those used in the analysis, while fusion of residues must not be carried out in platinum crucibles used for igniting magnesium pyrophosphate. For iron and steel, volumetric estimation is necessary in the works on account of its quickness, and is sufficiently accurate if based on gravimetric estimations of samples similar to those under test. O. E. M.

Estimation of Potassium as Perchlorate. G. P. Baxter and F. E. Rupert. (*J. Amer. Chem. Soc.*, 1920, **42**, 2046-2048.)—Experiments described in detail show that in Baxter and Kobayashi's method of estimating potassium as perchlorate (ANALYST, 1920, 238), ethyl alcohol, denatured with 5 per cent. of methyl alcohol, may be used in place of absolute ethyl alcohol as the washing liquid, whilst the temperature is unimportant, if the washing liquid is saturated with the potassium perchlorate after the initial extraction, and the following modification of the method is used: The solution of the mixed potassium and sodium perchlorates is evaporated to dryness with a small quantity of perchloric acid, and the residue extracted with 20 c.c. of denatured alcohol containing 0.2 per cent. of perchloric acid, and washed with two portions of 5 c.c. of the same solution. The first extract and washings are filtered through a tared platinum sponge crucible. The potassium chlorate is then dissolved in water, the solution evaporated to dryness with a small quantity of perchloric acid, and the residue transferred to the platinum sponge crucible by means of denatured alcohol which contains 0.2 per cent. of perchloric acid and is saturated with potassium perchlorate. This solution is made by dissolving the necessary quantity of potassium perchlorate in warm concentrated perchloric acid, and adding the solution to the alcohol. From 75 to 95 c.c. are used for washing the residue in the crucible at a temperature up to 20° C. C. A. M.

Volumetric Estimation of Potassium. G. Ajin. (*Chem. Trade J.*, 1920, **67**, 501.)—To 25 c.c. of a 2 per cent. solution of potassium sulphate or chloride, 50 c.c. of 2*N* tartaric acid are added slowly, followed by 25 c.c. of $\frac{N}{2}$ aqueous sodium hydroxide, the flask being well shaken during the addition and for five minutes after. Twenty-five c.c. of 96 per cent. alcohol are run in very slowly with constant agitation, and the whole allowed to stand eight hours. The precipitate is filtered off, washed nine times with neutral 96 per cent. alcohol, transferred to a flask and exactly neutralised to phenolphthalein by the addition of $\frac{N}{10}$ sodium hydroxide in 96 per cent. alcohol. A known volume of $\frac{N}{10}$ aqueous sodium hydroxide is added until the precipitate is completely dissolved and the excess of the sodium hydroxide titrated with $\frac{N}{10}$ hydrochloric acid. The volume in c.c. of $\frac{N}{10}$ sodium hydroxide used in dissolving the precipitate, multiplied by 0.00471, gives the quantity of potassium oxide (K_2O) present. T. J. W.

Differentiation of Radium from Barium by Means of Iodic Acid. G. Denigès. (*Comptes rend.*, 1920, **171**, 633-635.)—The crystals obtained when a drop of barium bromide solution is treated with a drop of iodic acid solution are quite

different from those obtained under similar conditions from radium bromide solution. The difference is particularly marked when the concentration of the bromide solutions does not exceed 0.03 per cent.

W. P. S.

Volumetric Methods for Estimating Tin. J. G. F. Druce. (*Chem. News*, 1920, 44, 173.)—The known methods were investigated. Titration of stannous salts in hydrochloric acid with standard iodine solution gave results agreeing well with the theory, and can be used for the inorganic stannochlorides and those of the aliphatic amines; most of the aromatic amines darken and obscure the end-point. Permanganate is satisfactory for use on fresh stannous solutions in sulphuric acid; a small quantity of sodium carbonate is added to prevent oxidation.

O. E. M.

APPARATUS, ETC.

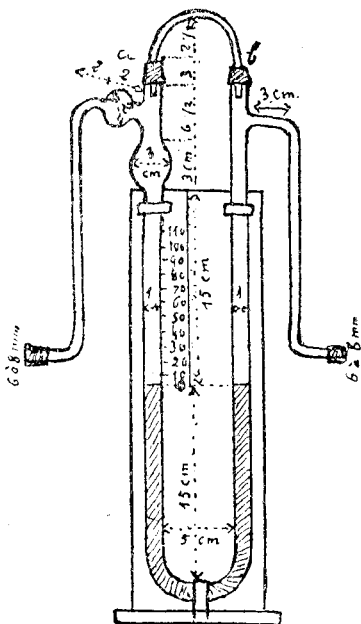
New Method of Measuring the Velocity of a Current of Gas. J. Erlich. (*Ann. Chim. anal.*, 1920, 2, 289-297.—When a liquid flows under a pressure h , through a capillary tube, its velocity, v , may be represented by Poiseuille's formula—

$$V = Kh \frac{d^4}{l},$$

where d represents the diameter of the

tube, l its length, and k a constant depending upon the nature of the glass of the tube and of the liquid. This law also extends to gases, but above a certain velocity (70 to 100 litres per hour) h increases more rapidly than the law indicates. When a gas escapes from a capillary tube at a velocity, v , it causes a difference of pressure h , apart from the tube. Since k is constant for a given gas and apparatus each value for v corresponds with a value for h which is always the same. An apparatus embodying this principle is shown in the accompanying diagram.

A capillary tube ab is connected at its extremities with the limbs of a differential manometer m , the bulbs on which are intended to prevent the liquid reaching the capillary tube. The deviations of the level in the manometer are measured upon a scale graduated in tenths of a cm. In practice it is convenient to construct a graph for a given apparatus and gas, showing directly the values of v corresponding with the deviations in level h . It is essential that the gas should be dry. Changes of temperature are corrected by means of the formula—



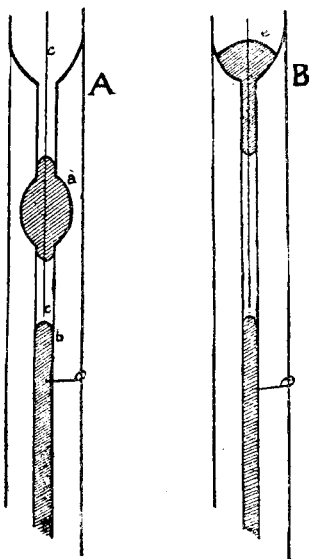
$$V_r = \frac{V}{1 + k(t - t^0)} \left(\frac{273 + t^0}{273 + t} \right)^3,$$

where t represents the temperature at which the gas was measured— t^0 the temperature of the determination, V the velocity read on the graph, V_r the true velocity, and k the coefficient of dilatation of the manometer liquid.

C. A. M.

Laboratory Apparatus for Preparing Electrolytic Hydrogen. W. G. Palmer. (*J. Soc. Chem. Ind.*, 1920, 39, 316r.)—An unglazed earthenware pot, 3 by 7 inches, has three holes drilled in its closed end; vertical rows of small holes are also drilled in the sides, and the pot is soaked in melted paraffin wax. A large sheet of parchment paper is wrapped round the pot so as to cover the rows of holes and is held in position with rubber bands. A cylindrical cathode of sheet nickel is fixed in the pot, the leads passing through glass tubes cemented in two of the holes in the closed end, and the pot is then placed in an inverted position in a vessel containing 10 per cent. potassium hydroxide solution. A cylindrical anode of sheet nickel, previously immersed in concentrated nitric acid for thirty minutes is fitted in the vessel containing the electrolyte, a distance of about 0.3 inch being left between the anode and the parchment paper cover of the pot. The third hole in the end of the pot is fitted with a rubber bung and a delivery tube for the hydrogen. A current of about 10 ampères may be used.

W. P. S.



Thermo-Regulator. J. F. King. (*J. Amer. Chem. Soc.*, 1920, 42, 2058.)—A simple device is shown to prevent the oxidation of mercury in a thermo-regulator owing to the formation of the small arc when the contact is broken between the mercury column and the adjustable wire contact. In the accompanying diagram A represents the capillary of a regulator with a small bulb at *a*. A capillary pipette is passed down to *b*, and a stream of an inert gas is introduced, to displace the air in the capillary, which is then sealed with a few drops of mercury at *a*. This mercury rises and falls with the movement of *b*, but never falls down on to *b*. Any arc then formed between the wire and the mercury column at *b* will then be in an inert gas. In the case of an ordinary thermo-regulator, B, the same result may be attained by sealing the capillary with a drop of mercury at *c*.

C. A. M.

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GOVERNMENT REPORT.

Report of the Government Chemist upon the Work of the Government Laboratory for the Year ended March 31, 1920, with Appendices. Cnd. 881. (Price 3d. net. H.M. Stationery Office.)—The total number of samples examined during the year was 368,898, an increase of nearly 80,000 over the number for the previous year. The principal increases were due to goods liable to Customs duty, such as wines, sugar, tea, and cocoa preparations, and are indicative of the revival of trade; at the same time, there was naturally a considerable decrease in the number of samples submitted by such Departments as the Admiralty, Air Ministry,

and the War Department. Of the above total number of samples, 169,510 were examined at the outport chemical stations.

Customs and Excise : Beer.—The number of samples examined in connection with the duty on beer was 25,489. *Materials used in Brewing :* The samples of malt, corn, brewing sugars, and exhausted grains numbered 323, and the yeast foods and miscellaneous samples 72. *Worts or Beer in the Unfinished Condition :* As a check on the assessment of beer duty 8,890 samples drawn from fermenting vessels were analysed ; 770 samples of beer, submitted as having become sour and unsaleable, were examined, and in most cases the beer was destroyed and the duty remitted or refunded. No evidence of dilution was found in samples of beer taken from the premises of publicans and other retailers. *Non-alcoholic Beers, Herb Beers, and Beer Substitutes :* Of 212 samples analysed, 81 were submitted as non-alcoholic beers, and 22 of these contained more than 2 per cent. of proof spirit. In one case the proof spirit exceeded 5 per cent. Of the remaining 132 samples (herb beers, ginger beers, etc.), 34 contained between 2 and 5 per cent. of proof spirit and 1 sample contained as much as 9·6 per cent. The importation of beer is still insignificant as compared with pre-war times, only 133 samples being examined. *Examination of Beer and Brewing Materials for Arsenic :* The number of samples tested, including beers, worts, malt, sugar, etc., was 969 ; of these, 5 were found to contain arsenic in excess of the limits suggested by the Royal Commission on Arsenical Poisoning. None of the samples of malt and sugar contained arsenic in excess of the limit, but the limit was exceeded in three instances out of 619 samples of beer and wort.

Cider and Perry.—Forty-four samples of imported cider were examined, of which 20 were classed as spirit preparations. Seven samples submitted by Customs and Excise officers were found to be factitious ; 7 samples of sour ciders, submitted for the purpose of obtaining remission of duty, were found to contain sufficient acid to render them unsaleable.

Table Water Duty.—Of 20 samples of mineral waters analysed, 16 proved to be medicinal and 4 were liable to table water duty ; of 333 cordials, syrups, etc., only 10 contained more than 2 per cent. of proof spirit, the highest amount being 11·3 per cent., and none of the 102 samples of so-called non-alcoholic wines contained more than 2 per cent. of proof spirit.

Spirits : Fusel Oil.—Only 1 out of 19 samples of fusel oil from distilleries was found to contain more than the permitted proportion of proof spirit. *Illicitly Distilled Spirits :* The analytical results afforded strong evidence that several of the 16 samples analysed had been distilled illicitly. *Duty-free Spirits, etc. :* Only 16 out of 848 samples of wood and mineral naphtha submitted were found to be unfit for methylating purposes. For the purpose of controlling the use of duty-free methylated and other spirit in connection with manufacturing processes, 539 samples of recovered spirit, etc., were examined. There was a considerable increase in the number of applications in respect of claims for rebate on alcohol used in making medicinal preparations, and for permission to receive methylated spirit and other forms of duty-free alcohol for manufacturing purposes and research.

Wines.—The total number of samples examined in connection with assessment of Wine Duty was 143,544, an increase of over 45 per cent. on the number for 1914.

Sugar, Glucose, and Saccharin.—The number of samples tested was 36,411 of sugar, 1,488 of glucose, and 321 of saccharin. In addition, 121 samples of imported substances likely to contain saccharin were analysed.

Tobacco.—During the year the percentage of moisture was determined in 10,234 samples, and the percentage of oil in 1,140 samples. The other samples examined comprised tobacco for drawback, 13,797; tobacco stalks, 18,893; offal snuff, 12,020; unmanufactured tobacco, 765; miscellaneous samples, 943.

In addition to the above the following number of samples were examined for this Department: Tea, 16,562; coffee and coffee substitutes, 1,527; cocoa and chocolate, 8,922; matches, 72 (no evidence of the presence of white phosphorus was discovered); medicines (under medicine stamp duty), 62; imports by parcel post, 590; etc.

Air Ministry.—Four hundred and ninety-five samples, mainly alloys, were examined for the Department, and special investigations were made on certain deposits found in the cylinders and piston heads of aircraft engines.

Ministry of Agriculture and Fisheries.—The samples examined for the ministry of Agriculture and Fisheries, the Board of Agriculture for Scotland, and the Department of Agriculture and Technical Instruction for Ireland numbered 1,023. *Imported Milk and Cream:* Five samples of fresh milk and 2 of sterilised milk were analysed; all were free from preservatives, but one sample of sterilised milk was deficient in fat. Three hundred and forty samples of condensed milk and dried milk were tested; the percentage of fat in the samples of whole condensed milk varied from 6.4 to 12.2 per cent. All the samples were free from preservatives and there was no evidence of skimming. Eight samples of cream contained from 8 to 10 per cent. of fat; preservatives were absent. *Imported Butter:* Only six samples were received; they were free from foreign fats, but 2 contained more than 16 per cent. of water. Of 267 samples of imported margarine, 5 contained more than 16 per cent. of water. *Imported Cheese:* The 37 samples of cheese analysed were all genuine. This ministry also submitted samples of sheep dips, rat poisons, oysters, fertilisers, insecticides, etc. *River Pollution:* In addition to the examination of 98 samples of river water and effluents, work was carried out in order to determine the nature and proportions of the toxic constituents which might be present in drainage from tarred roads.

Ministry of Munitions.—Amongst the samples examined for this Department were liquid fuel taken from a German tractor; salts and brines for the occurrence of potash and bromine; materials used in the recovery of potash from felspar, and dust in cement works flues. This dust was found to be rich in potash in a form readily available for use. Arrangements were made to extract radium from many thousands of luminous dials, gun sights, compass cards, etc., and practically all the radium present was recovered in a usable form and at comparatively small cost.

Ministry of Food.—During the year 3,073 samples were submitted; these included milk (fresh and condensed), jam, cheese, feeding stuffs, crude and refined oils, and edible fats. Many of the samples were taken in connection with the weekly supervision of factories under the control of the Ministry.

Ministry of Health.—Thirty-six samples of canned meat, condensed milk, and dried egg were examined.

A description is also given of the work done for the Crown Agents for the Colonies, Geological Survey, Home Office, India Office, Inland Revenue, Post Office, Stationery Office, Board of Trade, Corporation of Trinity House, War Department, Office of Woods, Office of Works, London, and Office of Works, Dublin.

Sale of Food and Drugs Acts.—The samples referred by magistrates under the Sale of Food and Drugs Acts of 1875 and 1899, and under Food Control Orders, numbered 121; these included 93 samples of milk, 8 of beer, 5 of whiskey, 3 of margarine, 2 each of butter, vinegar, and cream of tartar, and 1 each of ginger, gin, mustard, canned peas, camphorated oil, and sodium bicarbonate. Four of the samples could not be examined owing to their condition on receipt. The following are the particulars of the most important cases:—*Butter*: One sample contained foreign fat, and 1 excess of water, as alleged by the public analyst. *Vinegar*: Both samples consisted of artificial vinegar, as alleged. *Cream of Tartar*: The results agreed with those of the public analyst, who had found an excessive quantity of lead in the samples. *Beer*: The samples were referred in connection with proceedings under the Beer (Prices and Description) Order, 1919. In 1 case the original gravity differed from that found by the public analyst; in the remaining 7 cases there was no information as to the finding of the public analyst. *Whiskey and Gin*: The point in dispute for all the samples was the alcoholic strength, but it is not known whether or not the results were in agreement with those of the public analyst. In the case of the ginger alleged to contain spent ginger, the results did not agree with those of the public analyst. In the case of canned peas alleged to contain copper, camphorated oil alleged to be deficient in camphor, mustard alleged to contain foreign starch, and sodium bicarbonate alleged to contain arsenic, the results agreed with those of the public analyst. In the 89 cases of milk samples in which certificates were issued the charges were as follows: In 26 samples, added water; 45, abstraction of fat; 6, added water and abstraction of fat; 2, presence of preservatives; and 10, presence of dirt. In 7 cases the results were not in agreement with those of the public analyst.

Fertilisers and Feeding Stuffs Act.—The samples under this Act were submitted by the Ministry of Agriculture and Fisheries in connection with application by local authorities for the consent of the Board to take proceedings against the sellers. The 13 samples of fertilisers received consisted of basic slag, hair and fur waste, guano, and plant foods; the 5 samples of feeding-stuffs consisted of feeding meals and poultry foods.

W. P. S.



REVIEW.

THE CARBOHYDRATES AND ALCOHOL. By S. RIDEAL, D.Sc., F.I.C., and Associate (Industrial Chemistry). Pp. xv+219. London: Baillière, Tindall and Cox, 1920. Price 12s. 6d. net.

The publication of a series of books giving a general survey of the chemistry of industries will commend itself to all chemists, and to none more than to those

engaged in analytical chemistry. This is an age of specialisation, in which no chemical practitioner can possess a knowledge of all branches of chemical technology, and among the problems submitted to him, are frequently those connected with specific industries, so that unless he has some acquaintance with the requirements of these industries his reports may be practically without value to his clients. Dr. Rideal has set himself a heavy task to edit such a series of books, the success of which must depend entirely on securing the best specialists connected with each of the chemical industries.

The principal subjects with which the present volume is concerned are Starch and its Derived Sugars, Manufacture of Sugar from Sugar Cane and Sugar Beet, Sugar Refining, Malting, Brewing, Wine Making, Potable and Industrial Alcohol, Vinegar, Acetic Acid, Acetone, and Glycerin.

The sections contain some useful matters, but the text is marred by many errors and misstatements. This is the more regrettable in view of the fact that the book is written for the guidance of non-experts. Among these the following may be cited: Wheat is stated on one page to contain 55 to 65 per cent. of starch, and on another page its average starch content is said to be 68 per cent. It is not correct that the cheapest form of starch is that derived from potato, and the statement that "wheat starch" is used as a paste for "bill-posting," besides being inaccurate, may have the effect of giving undue prominence to the noble art cited. The title "Cane Sugar" and "Beet Sugar" for the sections dealing with the manufacture of sugar from cane and beet respectively is liable to revive the fallacy that sugar from the two sources differs. Goldthorpe barley is a broad-eared, not narrow-eared, two rowed barley; it belongs to the variety *Hordeum zeocriton*, not *Hordeum distichum*. The statement that by the malting process "the insoluble starch of the grain is converted into soluble fermentable sugar" is one we might expect to find in a book written in the eighteenth century.

Possibly the reason that the production of maltose in Belgium did not prove a commercial success was that the product was not entirely maltose, being made with malt extract. The statement that the percentage of maltose in commercial glucose (understanding thereby liquid glucose) generally exceeds that of dextrose is not the invariable rule.

Our criticism of this book must of necessity be severe, for it will undoubtedly fall into the hands of those who will believe implicitly everything they read in its pages. Whilst admitting, therefore, that some of the text has been carefully compiled, looking at the book as a whole we are forced to the conclusion that it does not fulfil its objects, and we hope the editor may in the near future, with the aid of experts, recast the book, omitting much detail which is not needed, and giving a succinct and accurate epitome of the chemical industries with which it is concerned.

A. R. LING.
