

# THE ANALYST.

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

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### OBITUARY NOTICE.

#### EDWARD JOHN BEVAN.

EDWARD JOHN BEVAN was the son of Edward Bevan, of Birkenhead. He was born in 1856, and was educated at private schools in Birkenhead and Birmingham. As he had shown the boy-amateur's predilection for experimental "science," his parents secured for him, at the age of seventeen, a post as assistant in the laboratories of the Runcorn Soap and Alkali Company, and the opportunity of a career as chemist.

At this early stage he came under the influence of pioneer workers in the alkali industry, the Brothers Hargreaves, Davis, and Weldon, who wisely advised systematic academic training and the "genial" atmosphere of Owens College, at that date (1876) the recognised head centre of chemical science in this country.

Here he was associated with C. F. Cross and a group of research workers, under the influence of Roscoe and Schorlemmer, and the student friendship which resulted was so cemented that on leaving the college, each to engage on technical research work in connection with "cellulose" industries, active correspondence and in fact collaboration ensued. Bevan's position was that of chemist at the Mosselburgh Paper Mill (A. Cowan and Co.), where he enjoyed the sympathetic collaboration of the director-manager, R. C. Menzies, which contributed much to the consolidation of his technical-scientific grasp of manufacturing industry. Further, his three years' residence in the bracing atmosphere of the East of Scotland changed Bevan in point of health from the "rather delicate" youth to a man of relatively robust constitution.

The collaboration with C. F. Cross then resulted in the momentous decision for a joint adventure in the field of cellulose research, pure and simple, and, coming to London, they were able, through the influence of such friends as Thiselton Dyer, Hugo Müller, and H. E. Armstrong, to secure an entrée to the Jodrell Research Laboratory, Kew Gardens (1883). The problem of "ways and means" asserting itself on the prospective exhaustion of the slender funds accumulated in the previous period of salaried appointments, it was necessary to supplement the research programme by work in the technical-industrial field. There resulted the more formal partnership of "Cross and Bevan," an engagement with the firm of Thomson, Bonar, and Co. for research work in developing the "Ekman" bisulphite pulping process (wood cellulose), and the finding of laboratory accommodation, which they owed to R. E. H. Goffin and the Council of the Westminster (Secondary) Schools.

This was the early phase of long years of struggle which has resulted in the achievements by which "Cross and Bevan" is or are known.

But it is due to Edward John Bevan, as a Past-President of the Society, to notice more particularly that phase of his career in which he has made his special personal reputation—that is, in filling the position of Public Analyst under appointment by the Middlesex County Council.

Apart from his official duties, he took, from the first, a keen interest in the work of our Society. He was an abstractor of the ANALYST in its early days, and was a frequent contributor to its pages. For ten years (1894–1904) he was one of the Secretaries, in which position his enthusiasm and unflinching courtesy endeared him to all the members. In the following year he was elected President.

His professional reputation is so well established, generally, and, as Bevan would say, generously recognised, that the writer can add nothing to the estimates of his achievement in this field, often and fully expressed by his brother chemists, who, from their training and the exacting discipline of their professional work, are of necessity rigorous in their judgment of capacity.

The writer knows that Bevan derived the greatest encouragement from this ungrudging recognition by his peers of his actual professional work and its associated activities for the general welfare, of which mention should also be made of his devoted work as Fellow, Member of Council, and Vice-President of the Institute of Chemistry.

What the writer feels competent to add is a word on the moral qualities which Bevan brought to bear on his work—a grasp of order and method necessarily; a finished skill in manipulation, with great rapidity in routine operations; and, above all, a sensitive conscientiousness in regard to accuracy and the rigorous discipline, self-imposed, in taking all possible trouble to exclude every error outside the inevitable but measurable error of all laboratory estimations.

Such qualities may no doubt, and in a measure, be acquired; in Bevan's case it was original endowment or genius.

That he was "genial," in the ordinary acceptation of the term, is especially known to his wide circle of intimate friends. His was an attractive personality; he had much charm of manner, which made him popular in many charmed circles of club and of family, and his fondness for outdoor sports of all kinds was an added link of interest with many friends both inside and outside his profession. In his youth he was a keen cyclist, and later actively welcomed the coming of the motor-car, whilst still more recently he became an ardent golfer. He was also particularly interested in theatrical matters, and was for many years a supporter of the Playgoers Club. He was not married.

Those who may be interested in the records of research work in which the names of C. F. Cross and E. J. Bevan are associated, are referred to an article in the *World's Paper Trade Review* for January 28, 1921, from which it will be seen that the association has weathered the struggles and storms of forty-two years.

As a last word, Bevan's death, on October 17, at the early age of sixty-five, was directly connected with his personal war service—the conscientious discharge of his duties as "special" in his home district (Watford).

C. F. CROSS.

NOTE.—A portrait of Mr. Bevan will be published in a forthcoming number.—EDITOR.

## THE SEPARATION OF ALUMINIUM FROM BERYLLIUM.—PART II.\*

BY HUBERT T. S. BRITTON, B.Sc., A.I.C.

## AMMONIUM CARBONATE METHOD.

THE separation of beryllium from aluminium depending on the insolubility of aluminium hydroxide and the solubility of beryllium hydroxide in concentrated solutions of ammonium carbonate was discovered by Vauquelin in 1798 (*Ann. Chim. Phys.*, 1798, **26**, 155). Damour (*ibid.*, 1843, [iii.], **7**, 173) regarded the method as unreliable, as he found that it gave too low results for beryllium. It was first described in detail by Rose (*Handbuch der Analyt. Chem.*, 1851, II., 59). Two procedures were given: treating either (a) the hydroxides, or (b) the solutions of the two metals, with a sufficiently large quantity of ammonium carbonate solution. He admits that a little aluminium is afterwards found in solution. Wöhler, however (*Handbook of Inorg. Analysis*, English edition, 1854, 114), states that aluminium hydroxide can be completely precipitated by slowly pouring a solution of the two earths into an excess of a warm concentrated solution of ammonium carbonate. Weeren (*Pogg. Ann. der Physik.*, 1854, **92**, 91) found that the method yielded an incomplete separation, inasmuch as some alumina passed into the ammonium carbonate solution. Lewy (*Comptes rend.*, 1857, **45**, 881) used the method for the analysis of beryl. Hofmeister (*J. prakt. Chem.*, 1859, **76**, 1) endorsed Weeren's observation that the ammonium carbonate solution dissolved some aluminium hydroxide, and endeavoured to remove it by a series of fractional precipitations of the hydroxides contained in the filtrate. By such a treatment he obtained a slightly higher figure for alumina than that obtained by Lewy for the same type of beryl. Joy (*Sill. Amer. J. Sci.*, 1863 [ii.], **36**, 83) agreed with previous investigators that considerable amounts of alumina were dissolved by ammonium carbonate solutions. He carried out his experiments by pouring the solution of aluminium and beryllium salts into a warm saturated solution of ammonium carbonate and allowing it to stand, with occasional stirring, for some days. He investigated the solubility of beryllium hydroxide in a saturated solution of ammonium carbonate when allowed to stand, with occasional shaking, in stoppered bottles for varying times, and found that the maximum solubility was reached after ten days, and, further, that the solubility then decreased by as much as 15 per cent. after sixteen days. Williams (*Proc. Roy. Soc.*, 1877, **26**, 165) thoroughly investigated the method, and established the fact that beryllium hydroxide was "permanently soluble in a saturated solution of carbonate of ammonium," his solubility experiments extending over three years at room temperature. When the same solution (4 grms. BeO per litre) was heated in a sealed tube at 100° C. for two days, he observed that no precipitate formed. Williams also was not able to confirm the observations of Weeren, Hofmeister, and Joy that some alumina passed into solution, but, on the contrary, found that some beryllia was always precipitated with the alumina when allowed to stand for periods varying

\* See ANALYST, 1921, p. 359.

from five minutes to forty-eight hours. In the separation, in which he tried to remove the beryllia from the first aluminium hydroxide precipitate, he found that no less than seven extractions were necessary to reduce it to as little as  $\frac{1}{2}$  per cent. Wünder and Chéladzé (*Ann. Chim. anal.*, 1911, **16**, 205) examined the method by treating the precipitated hydroxides with a 5 per cent. ammonium carbonate solution for periods ranging from fifteen minutes to fourteen hours, and in every case found that much beryllium hydroxide remained undissolved.

The literature on this subject is thus somewhat confusing. On account of the results of some preliminary experiments, it was thought that the method might be made satisfactory, especially if carried out by gradually pouring a solution of aluminium and beryllium salts into a *cold* concentrated solution of ammonium carbonate, with continuous stirring. In this way it was hoped to minimise as much as possible any adsorption effects, it being thought that the failure of Wünder and Chéladzé was due to some beryllium hydroxide being occluded by the aluminium hydroxide, and consequently not being acted on by the ammonium carbonate solution. The aluminium hydroxide which is precipitated by means of ammonium carbonate is flocculent, and is therefore easily filtered and washed.

#### EXPERIMENTAL.

The behaviour of ammonium carbonate solutions towards solutions of aluminium sulphate and beryllium sulphate was tested in the following manner: To 100 c.c. of saturated ammonium carbonate solution 0.5 *N*-beryllium sulphate solution was added gradually from a burette, with shaking, until a precipitate was just produced; 200 c.c. of the 0.5 *N*-beryllium sulphate solution were required—in other words, 100 c.c. of saturated ammonium carbonate solution, even when diluted to 300 c.c., are capable of maintaining 1.255 grms. of beryllium oxide in solution. The experiment was repeated with a boiling ammonium carbonate solution. Nothing happened until 60 c.c. of 0.5 *N*-beryllium sulphate solution had been added, when an intense precipitate was immediately produced, which slowly dissolved on the addition of another 100 c.c. of ammonium carbonate solution.

In the case of aluminium, a distinct precipitate was formed by the addition of 0.1 c.c. of 0.5 *N*-aluminium sulphate solution to 100 c.c. of ammonium carbonate solution at room temperature. With boiling ammonium carbonate, some alumina passed into solution, no precipitate being produced until 2.8 c.c. of 0.5 *N*-aluminium sulphate solution had been added (*i.e.*, = 0.024 gm.  $\text{Al}_2\text{O}_3$ ).

Whereas the effect of heating the ammonium carbonate solution was to cause the solution of some aluminium hydroxide sufficient to render unreliable any separation involving the use of *warm* ammonium carbonate, separations in the *cold*, if no other complications arose, should be quantitative.

For the following separations, solutions were made up containing weighed amounts of beryllium sulphate and aluminium sulphate.

*Experiment 1.*—To 100 c.c. of a solution of ammonium carbonate a solution of beryllium sulphate was slowly added, with continuous shaking, the beryllium passing completely into solution; then a solution of aluminium sulphate was slowly added with shaking. The precipitate of aluminium hydroxide appeared from the

first. It was filtered immediately by suction, washed with ammonium carbonate solution, and ignited.

*Experiment 2.*—To a solution of beryllium sulphate and aluminium sulphate 100 c.c. of saturated ammonium carbonate solution were added. This caused a precipitate, which, on shaking, dissolved to almost a clear solution. After further shaking and vigorous stirring the solution became opalescent in appearance, and in about five minutes a heavy precipitate settled out. When no further precipitate appeared to separate out, the precipitate was filtered, washed, and estimated.

*Experiment 3.*—A solution of the two sulphates was slowly added, with stirring, to 100 c.c. of a saturated solution of ammonium carbonate. This caused no apparent change, but on stirring the solution gradually became opalescent, and a precipitate finally settled out. It was allowed to stand for ten minutes, filtered, washed, and ignited.

*Experiment 4.*—As it was believed that the delayed formation of the colloidal precipitate observed in Experiments 2 and 3 was probably the cause of the beryllia being found in the aluminium hydroxide precipitate, this experiment was carried out as follows: To a solution of aluminium and beryllium salts 100 c.c. of the saturated ammonium carbonate solution were added drop by drop, with stirring, in order that the two hydroxides might first be precipitated in extremely small amounts, and that the addition of more ammonium carbonate might dissolve the beryllium hydroxide. After the ammonium carbonate was all added, the solution was vigorously shaken for five minutes, and then filtered by suction. The filtrate at first was clear, but after a time it became opalescent, finally gave a precipitate, and was filtered. Both precipitates were washed with ammonium carbonate solution and estimated. First precipitate = 0.0997 grm.; second precipitate = 0.0547 grm.

Experiment Number.	Grm. Taken.		Grm. $\text{Al}_2\text{O}_3$ Found.	Extra Grm.
	BeO.	$\text{Al}_2\text{O}_3$ .		
1	0.1278	0.1744	0.2224	0.0480
2	0.1805	0.2406	0.3070	0.0664
3	0.1579	0.2117	0.3020	0.0903
4	0.1261	0.1278	0.1544	0.0266

From these results it will be seen that in every case the amount of beryllia carried down by the precipitated aluminium hydroxide was appreciable, no matter how the process was carried out. Nor could the beryllia be removed from the alumina precipitate, although as much as 200 c.c. of saturated ammonium carbonate solution were employed in washing. There seems no doubt that the delayed precipitation was the cause of the failure to effect a quantitative separation. Experiments were tried, having either the sulphate or chloride of potassium present in solution, in the hope that such an electrolyte would promote immediate precipitation of the aluminium hydroxide, but the results were negative.

It was now left to be decided whether ammonium carbonate might be used under the conditions of the previous experiments as a means of obtaining beryllium hydroxide free from alumina. It seemed reasonable to regard the aluminium hydroxide precipitate as containing all the alumina, together with the beryllia which it had carried down, and thereby leaving only beryllia in solution. As the presence of aluminium hydroxide renders some beryllia insoluble, it was thought just possible that the converse, beryllia rendering some alumina soluble, might also be true. Two separations were therefore carried out in the manner described in Experiment 4. The filtrates were evaporated to dryness, during the course of which the beryllium hydroxide was precipitated. The residues, which had not been heated too strongly, were dissolved in dilute hydrochloric acid, and heated to drive off carbon dioxide; then excess of about 6 *N*-sodium hydroxide solution was added, and the solution again boiled to drive off ammonia. The solutions, having been evaporated to about 20 c.c., were cooled, and sufficient hydrochloric acid added to produce the faintest precipitates, the hydroxides thus being dissolved in the minimum amounts of sodium hydroxide solution. Each of the solutions was then diluted to 500 c.c., boiled for forty minutes, and filtered. Under these conditions no beryllia should remain in the mother liquor. On acidification with hydrochloric acid and treatment with ammonium chloride and ammonium hydroxide a gelatinous precipitate was produced in both filtrates. These precipitates were proved to be aluminium hydroxide by dissolving one in a little potassium hydroxide solution, and, after having driven off any occluded ammonia by boiling, acidifying the solution with sulphuric acid and leaving it to crystallise; and the other, by suspending it in a saturated solution of ammonium chloride and boiling the liquid for several hours until all free ammonia had been driven off. In the first case crystals of alum were obtained, and in the second the precipitate did not dissolve. Hence, all alumina is not precipitated by the addition of ammonium carbonate in the cold when beryllium salts are present in solution.

In conclusion, the ammonium carbonate method is unsatisfactory both quantitatively and qualitatively.

#### AMMONIUM SULPHITE METHOD.

Two methods of separation of aluminium from beryllium were described by Berthier (*Ann. Chim. Phys.*, 1843, [iii.], 7, 74), which depend upon the fact that the concentration of the hydrogen ions in a weak solution of sulphurous acid from which no more sulphur dioxide can be expelled by boiling is sufficient to maintain beryllium hydroxide in solution, but insufficient to prevent the precipitation of aluminium hydroxide. One method was to dissolve the hydroxides of aluminium and beryllium in sulphurous acid, and then boil until the evolution of sulphur dioxide had ceased; whilst the other was to treat a solution of the two hydroxides with excess of a strong solution of ammonium sulphite, and boil until all sulphur dioxide had been driven off. Böttiger (*Liebig Ann.*, 1844, 51, 397) investigated these methods, and found that in both cases some beryllia was invariably precipitated with the alumina. Weeren (*Pogg. Ann. der Physik.*, 1854, 92, 91) and Joy (*Sill. Amer. J. Sci.*, 1863, [ii.], 36, 83) arrived at the same conclusion.

The two methods are in reality almost identical, for in the case where the

precipitated hydroxides are employed much ammonia is retained in the precipitates. It was therefore decided, first of all, to ascertain under what conditions the aluminium hydroxide was completely precipitated and the beryllium hydroxide completely soluble.

## EXPERIMENTAL.

A solution of ammonia (sp. gr. 0.880) was saturated with sulphur dioxide. On adding 0.5 *N*-beryllium sulphate solution in considerable quantity to 100 c.c. of the boiling solution no precipitation occurred, even after boiling for several hours until all sulphur dioxide had been given off. The solution remained clear, even when evaporated to the point of crystallisation. When 0.5 *N*-aluminium sulphate solution was added to 100 c.c. of the boiling sulphite solution, no precipitation took place until 0.5 c.c. had been added, when a distinct turbidity was produced, more aluminium sulphate producing a precipitate. After boiling a solution containing much suspended aluminium hydroxide until the evolution of sulphur dioxide had ceased, no aluminium hydroxide could be found in the filtrate. The aluminium hydroxide precipitate was not gelatinous, and could be filtered easily. As the fundamental principles of this process were satisfactory, two separations were carried out thus: Solutions containing weighed quantities of the two sulphates were each added to 100 c.c. of ammonium sulphite solution. In each case no precipitation occurred at first, but after a few seconds the solutions gradually became cloudy, and precipitates ultimately settled out. Boiling was carried on, keeping the solutions up to their original volume by occasionally adding water until no sulphur dioxide could be detected in the issuing steam, after which the precipitates were filtered off, ignited, and weighed.

Number.	Grm. Taken.		Grm. Al <sub>2</sub> O <sub>3</sub> Found.	Extra Grm.
	BeO.	Al <sub>2</sub> O <sub>3</sub> .		
1	0.1081	0.1695	0.1811	0.0116
2	0.0706	0.0766	0.0911	0.0145

As the weights of alumina in both cases were much too high, due to the precipitation of the beryllium hydroxide under the influence of the aluminium hydroxide, the mother liquors were examined for the presence of alumina in a similar way to that described in the previous section, but none was found.

Further analyses were considered unnecessary, as the method appeared to give analogous results with the ammonium carbonate one, due in all probability to the same cause—viz., the delayed formation of the aluminium hydroxide precipitate. Although it has not been possible to render this separation quantitative, it can be employed for freeing beryllium hydroxide from alumina. The latter procedure, however, involves much loss of beryllia and is tedious, many hours' boiling being required to drive off all the free sulphur dioxide.

## SODIUM CARBONATE AND SODIUM BICARBONATE METHODS.

Hart (*J. Amer. Chem. Soc.*, 1895, **17**, 604) stated that adding sodium carbonate to a solution of aluminium and beryllium salts caused the precipitation of nearly all the alumina, the beryllia remaining in solution. The suggestion was improved upon by Parsons and Barnes (*J. Amer. Chem. Soc.*, 1906, **28**, 1589), who recommended the use of a 10 per cent. solution of sodium bicarbonate, rapidly raising the temperature of the solution to boiling, and keeping it at that temperature for not more than half a minute. Their method was to dissolve convenient quantities of the hydroxides in hydrochloric acid, neutralise as nearly as possible with ammonium hydroxide, make up to 100 c.c., and then add 10 grms. of sodium bicarbonate. The mixture was heated rapidly to boiling, and kept boiling for half a minute, cooled, and filtered. The aluminium hydroxide precipitate contained some beryllia, which, according to Parsons and Barnes, can be removed by another re-solution and re-extraction. Noyes, Bray, and Spear (*J. Amer. Chem. Soc.*, 1908, **30**, 481) agree that the method is satisfactory when the amount of alumina present is not large. When the alumina present was between 0.1 and 0.5 gm., they found that the aluminium hydroxide precipitate retained from 0.002 to 0.005 gm. of beryllium oxide. As the author did not obtain good separations by this method in several instances, it was decided to investigate it in order to find what conditions were necessary to make it give accurate results.

## EXPERIMENTAL.

The following Table I. gives the number of c.c. of 0.5 *N*-beryllium sulphate solution and 0.5 *N*-aluminium sulphate solution which can in each case be added to 100 c.c. of sodium carbonate solutions before a precipitate is formed :

TABLE I.

	Temperature.	Sodium Carbonate saturated at 15° C.	0.502 <i>N</i> -Na <sub>2</sub> CO <sub>3</sub> .
0.5 <i>N</i> -BeSO <sub>4</sub> ...	15° C.	150 c.c. = 0.9412 gm. BeO.	0.1 c.c. = 0.0006 gm. BeO.
	100° C.	150 c.c. = 0.9412 gm. BeO.	0.25 c.c. = 0.0015 gm. BeO.
0.5 <i>N</i> -Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	15° C.	7.0 c.c. = 0.0596 gm. Al <sub>2</sub> O <sub>3</sub> .	5.3 c.c. = 0.0451 gm. Al <sub>2</sub> O <sub>3</sub> .
	100° C.	6.5 c.c. = 0.0554 gm. Al <sub>2</sub> O <sub>3</sub> .	5.0 c.c. = 0.0426 gm. Al <sub>2</sub> O <sub>3</sub> .

From these data it will be seen that the solubility of aluminium hydroxide in a saturated solution of sodium carbonate is sufficient to render a method depending upon it futile. The results obtained from 0.502 *N*-sodium carbonate solution show that the effect of diluting sodium carbonate solutions of alumina and beryllia is to precipitate both the hydroxides:

Table II. gives similar results for sodium bicarbonate solutions.



TABLE II.

		Temperature.	Sodium Bicarbonate.			
			0·942 <i>N.</i> (Saturated at Room Temp.).	0·687 <i>N.</i>	0·502 <i>N.</i>	0·251 <i>N.</i>
0·5 <i>N.</i> -BeSO <sub>4</sub> .	c.c. = grms. BeO	15° C. {	23·2	16·5	10·0	2·7
			0·1456	0·1035	0·0628	0·0017
0·5 <i>N.</i> -BeSO <sub>4</sub> .	c.c. = grms. BeO	100° C. {	35·4	22·0	14·5	2·7
			0·2221	0·1381	0·0910	0·0017
0·5 <i>N.</i> -Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .	c.c. = grms. Al <sub>2</sub> O <sub>3</sub>	15° C. {	0·05	0·05	0·05	0·05
			0·0004	0·0004	0·0004	0·0004
0·5 <i>N.</i> -Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .	c.c. = grms. Al <sub>2</sub> O <sub>3</sub>	100° C. {	0·05	0·05	0·05	0·05
			0·0004	0·0004	0·0004	0·0004

In the case of experiments carried out at 100° C., it should be stated that some beryllium hydroxide separated out on cooling. Table II. shows that Parsons and Barnes's method is theoretically possible when the amounts of beryllia present are within its limits of solubility in the amount and concentration of the sodium bicarbonate solution employed. If the separation is carried out by taking a neutral solution of about 20 c.c. of the two earths, to which 100 c.c. of saturated sodium bicarbonate solution are to be added, it appears that the beryllia should not exceed 0·15 gm. When the neutral solution is made up to 100 c.c., and 10 grms. of sodium bicarbonate are added, a little more than 0·15 gm. of beryllium oxide may be present. However, as a safe working rule, the amount of beryllia present should be less than 0·15 gm.

Experiments were carried out to ascertain what was the effect of boiling saturated sodium bicarbonate solutions in which (*a*) aluminium hydroxide was suspended, and (*b*) beryllium hydroxide was dissolved. After boiling for ten minutes, not a trace of alumina could be found in the mother liquor, but in the case of the beryllium hydroxide solution a slight precipitate began to deposit. From Table I. it will be observed that alumina is slightly soluble in boiling sodium carbonate solutions; hence it is possible that the decomposition produced by prolonged boiling of a bicarbonate solution may cause the solution of a little alumina. Solution of beryllium hydroxide in sodium bicarbonate solutions takes place much more quickly on warming; consequently, as Parsons and Barnes suggested, it is advisable to carry out the separation by rapidly raising the temperature to boiling.

The following separations were carried out with a view to finding the amount of beryllia which is carried down with the aluminium hydroxide in a single precipitation. Solutions were made up of weighed amounts of beryllium sulphate and aluminium sulphate in the minimum amount of water, and treated with saturated sodium bicarbonate solutions in various ways. The resulting mixtures were immediately filtered by suction, and the alumina estimated, after it had been freed from adsorbed

sodium salts, either by solution in hydrochloric acid and reprecipitation with ammonium chloride and ammonium hydroxide, or by extracting the ignited precipitate (having been ignited in a platinum crucible) with hydrochloric acid, decomposing any aluminate with ammonium chloride and ammonium hydroxide, and boiling the liquid.

TABLE III.

No.	BeO Taken.	Al <sub>2</sub> O <sub>3</sub> Taken.	Al <sub>2</sub> O <sub>3</sub> Found.	Amount BeO Adsorbed.	BeO in Alumina Precipitate.
	Grm.	Grm.	Grm.	Grm.	Per Cent.
1	0·1007	0·0262	0·0284	0·0022	7·7
2	0·0124	0·1176	0·1250	0·0075	6·0
3	0·0929	0·1644	0·1730	0·0086	5·0
4	0·1003	0·0259	0·0279	0·0020	7·2
5	0·1010	0·0512	0·0575	0·0063	11·0
6	0·1004	0·1020	0·1160	0·0140	12·0
7	0·0125	0·1175	0·1260	0·0085	6·7
8	0·0560	0·1025	0·1045	0·0020	1·9
9	0·1103	0·0794	0·0837	0·0043	5·1
10	0·0132	0·1087	0·1090	0·0003	0·3

All these separations, except Nos. 1 and 2, having been mixed in the cold, were carried out by pouring a solution of the two sulphates drop by drop into 100 c.c. of warm, saturated sodium bicarbonate solution, and then quickly raising the temperature to boiling, keeping it at that point for not more than a minute, filtering the liquid, and making the estimation. In each case the aluminium hydroxide precipitation occurred from the beginning. It will be seen that the amount of beryllium hydroxide carried down with the aluminium hydroxide depended on (a) the bulk of the aluminium hydroxide precipitate, and (b) the amount of beryllia in solution. It was thought that the beryllium hydroxide which was brought down was mechanically adsorbed by the aluminium hydroxide, and consequently vigorous stirring should have the effect of forcing much of the adsorbed beryllia back into solution. This was borne out in separation No. 8, where, the mixture having been kept thoroughly stirred, the amount of beryllium oxide adsorbed was only 1·9 per cent. Stirring in the case of No. 9 did not have as great an effect, as the amount of beryllia present was proportionately greater. As the ratios of the two oxides taken in separations Nos. 1 to 9 are quite general, it was inferred that with efficient stirring the amount of adsorbed beryllia under the same conditions was never likely to be more than 10 per cent. of the total precipitated oxides. The success of the method, involving a second precipitation from a neutral solution of the first precipitate, therefore depended upon whether a mixture of the two oxides, 10 per cent. of which was beryllia, could be quantitatively separated. Such a mixture was satisfactorily separated—viz., No. 10—the amount of beryllia being actually 10·8 per cent.

The following separations were carried out, employing two precipitations, by dissolving the first aluminium hydroxide precipitate in the minimum amount of

dilute hydrochloric acid, neutralising the solution with ammonia, and adding it drop by drop, with stirring, to 100 c.c. of warm saturated sodium bicarbonate solution, rapidly heating the liquid to boiling-point and allowing it to boil for half a minute, and completing the estimation of alumina as previously described. The beryllia was estimated as usual, after having combined the two filtrates and boiled the liquid with excess of concentrated hydrochloric acid until all carbon dioxide had been driven off.

TABLE IV.

No.	Grm. Al <sub>2</sub> O <sub>3</sub> .		Grm. BeO.	
	Taken.	Found.	Taken.	Found.
1	0·0226	0·0226	0·1409	0·1406
2	0·0792	0·0795	0·1106	0·1106
3	0·0852	0·0855	0·0941	0·0940
4	0·1537	0·1542	0·1421	0·1417
5*	0·1087	0·1090	0·0132	0·0129

If the necessary precautions are taken, the method is therefore capable of giving satisfactory results.

## SUMMARY.

(1) Ammonium carbonate is of no use for the quantitative analysis of solutions of aluminium and beryllium salts; some beryllia is carried down with the alumina, and also a little alumina remains in solution.

(2) Ammonium sulphite does not give a quantitative separation, as some beryllia is absorbed by the alumina. All the alumina is, however, precipitated.

(3) A saturated solution of sodium carbonate dissolves an appreciable quantity of beryllia, but also much alumina.

(4) The method employing two precipitations by means of saturated sodium bicarbonate solution is satisfactory, provided that (a) neither the beryllia nor the alumina in 100 c.c. of solution saturated with sodium bicarbonate is more than 0·15 gm.; (b) adsorption in both precipitations is kept at a minimum by thorough stirring.

The author desires to take this further opportunity to express his appreciation of the interest taken by Professor A. J. Allmand in this work.

KING'S COLLEGE, UNIVERSITY OF LONDON,  
W.C. 2.

\* These data give the complete analysis of mixture No. 10 in Table III., and were therefore obtained by one precipitation only.



## SUGAR CALCULATIONS.

By J. F. LIVERSEEGE, F.I.C.

A PUBLIC ANALYST in the examination of substances containing sugars has to use constants and equations. The following notes are the results of an attempt, by selection and calculation, to obtain a set of data that is clear and consistent.

**SPECIFIC ROTATORY POWER.**—There are ambiguities in published specific rotatory powers owing to authors failing to state the temperature and concentration of the solutions used, and sometimes it is not clear whether the sugar was anhydrous or contained water of crystallisation. In some cases results given by various authorities differ considerably, and the selection of the best value has been difficult. This particularly applies to lævulose and invert sugar. The value for invert sugar should be half the algebraic sum of those of lævulose and dextrose. The selected values for sugars are given in the table on p. 450. In practice it is a convenience to apply the term "specific rotatory power" not only to pure sugars, but also to mixed substances such as treacle.

**CORRECTION FOR TEMPERATURE.**—In factors given for the correction of specific rotatory power there often is doubt as to whether in the equations given the sign for left-handed rotation (–) is to be used as well as any algebraical sign. As the temperature correction will never alter the rotation from "+" to "–," it is better to ignore the optical signs in making this correction.

Equation (1), given below, is to be used when the specific rotatory power of a pure sugar is being determined; equation (2) when the actual angle of determination is being corrected for temperature. In this case,  $\alpha^{20}$  means the angle at 20° C. of a solution examined in a 200 mm. tube;  $[\alpha]_D$  may be substituted for  $\alpha$  in this equation. The values for "D" and "d" are given in the table, and "t" represents degrees C.

$$(1) [\alpha]_D^{20} = [\alpha]_d + D(t - 20). \quad (2) \alpha^{20} = \frac{\alpha^t}{1.0 + d(20 - t)}$$

The application of equation (2) may be illustrated by the correction for temperature of a solution of invert sugar, which gave a rotation of  $-7.7^\circ$  at  $24.4^\circ$  C.:

$$\alpha^{20} = \frac{7.7}{1.0 + 0.0149(20 - 24.4)} = 8.24^\circ.$$

Solutions should be made up to volume at 20° C., and polarised as near this standard temperature as is convenient.

**CORRECTION FOR CONCENTRATION.**—The standard concentration is 10 w/v (*i.e.*, 10 grms. per 100 c.c.). When a sugar solution is diluted, in addition to the primary reduction of the angle according to the equation,

$$(3) \alpha = \frac{w/v \times [\alpha]_D}{50},$$

there is with some sugars a secondary reduction, owing to the fact that in dilute solutions the rotatory power *per unit of sugar* is less than in stronger solutions.

The sign  $[\alpha]_D^{20}$  is often used to indicate the specific rotatory power of a sugar at

20° C. when a sodium flame is used. A useful extension of it is to add the figure for the  $w/v$  of the solution to the left-hand side of the bracket. The sign  $^{10}[\alpha]^{20}$  would therefore mean that the specific rotatory power was corrected to 20° C., and that the figure was also corrected for this secondary alteration to 10  $w/v$ . The corresponding sign for the angle actually observed in a 200 mm. tube is  $^{10}\alpha^{20}$ . Again, to avoid ambiguities, the optical signs of the sugar solutions are ignored in making the correction.

Equation (4) is to be used for correcting the specific rotatory power of sugar to the standard of 10  $w/v$  when the concentration is known. In many cases, however, the concentration of the solution has to be determined, and therefore cannot be used. In such cases an approximation may be made by using the determined angle in place of the  $w/v$ , as in equation (5). The values for "C," "c," and "B," are taken from the table :

$$(4) \quad ^{10}[\alpha]_D = {}^x[\alpha]_D + C(10 - w/v).$$

$$(5) \quad ^{10}\alpha^{20} = {}^x\alpha^{20} + c(B - \alpha^{20})^x\alpha^{20}.$$

The above-mentioned solution of invert sugar may be taken as an example of equation (5) :

$$^{10}\alpha = 8.24 + 0.0080(4.2 - 8.24)8.24 = 7.98^\circ.$$

CALCULATION OF  $w/v$  WHEN ONE SUGAR ONLY IS PRESENT.—Either of the following equations may be used :

$$(6) \quad \text{Sugar } w/v \text{ in the solution} = \frac{50 \times ^{10}\alpha^{20}}{^{10}[\alpha]_D^{20} \text{ of pure sugar}}.$$

$$(7) \quad \text{Sugar } w/v \text{ in the solution} = \frac{10 \times ^{10}\alpha^{20}}{B}.$$

Applying this to the above-mentioned solution of invert sugar,

$$\frac{50 \times 7.98}{21} \text{ or } \frac{10 \times 7.98}{4.2} = 19.0 \text{ } w/v;$$

and as 100 c.c. contained 19.2 grms. of the substance, the percentage of invert sugar present =  $\frac{19.0 \times 100}{19.2} = 99$  per cent.

With specific rotatory powers the following equation may be used :

$$(8) \quad \text{Sugar per cent. (by weight) in the substance} = \frac{100 (^{10}[\alpha]_D^{20} \text{ of substance})}{^{10}[\alpha]_D^{20} \text{ of pure sugar}}.$$

MIXTURE OF TWO SUGARS.—Solutions of sucrose which is partly inverted often require examination. On inversion, 100 parts of sucrose yield 105 parts of invert sugar. In many cases it is more convenient to express invert sugar in terms of the sucrose from which it was derived, and I have used the term "inverted sucrose" to indicate this result. Unless it is known that only two sugars are present, it is necessary to prove by the determination of the reduction and total organic solids that such is the case. A mixture of sucrose and invert sugar in suitable proportions is optically inactive.

SUCROSE AND INVERTED SUCROSE.—When the substance consists of sucrose which has been partly inverted, the following equations will give its composition. "I" is used for the angle after inversion, corrected for temperature and concentra-

tion by equations (2) and (5), and  $\alpha$  for the original angle of polarisation *at the same dilution as "I"*:

- (9) Sucrose  $w/v$  in solution = 0.565 (change of angle of rotation).  
 (10) Inverted sucrose  $w/v$  originally present = 0.565 ( $3.023 I \pm \alpha$ ).  
 (11) Angle due to sucrose = 0.75 (change of angle of rotation).  
 (12) Angle due to inverted sucrose originally present = 0.249 ( $3.023 I \pm \alpha$ ).

In equations (9) and (11) the change of rotation will be the sum of the angles if the sign of rotation is altered by inversion, and their difference if their signs are the same.

In equations (10) and (12) "—" is used if the sign of the optical rotation is changed by inversion. If these equations give a negative result, dextrose, and not inverted sucrose, is present.

SUCROSE AND DEXTROSE.—The corresponding equations for mixtures of sucrose and dextrose are:

- (13) Dextrose  $w/v$  = 0.235 ( $\alpha \pm 3.023 I$ ).  
 (14) Angle due to dextrose = 0.249 ( $\alpha \pm 3.023 I$ ).

The "—" sign is used if inversion changes the optical sign.

DEXTROSE AND LÆVULOSE: INVERT SUGAR.—If dextrose and lævulose are present in equal quantities they become invert sugar. If the quantities are dissimilar, a mixture of invert sugar with excess of lævulose or dextrose results, and the composition can be calculated in either form by the following equations:

- (15) Per cent. of invert sugar = 1.35 ( $52.8 - {}^{10}[\alpha]^{20}$ ).  
 (16) Per cent. of lævulose = 0.678 ( $52.8 - {}^{10}[\alpha]^{20}$ ).

In these equations the optical signs must be included as well as the algebraic ones.

CORRECTION FOR TEMPERATURE AND CONCENTRATION OF A MIXTURE OF TWO SUGARS.—As sucrose and inverted sucrose polarise in opposite directions, the angle of a mixture of them is not a measure of the amount of inverted sucrose that is present. To correct the original angle, it is necessary to make an approximate calculation of the amount of inverted sucrose present in the solution before inversion.

The figures obtained with a sample of syrup of rhubarb may be taken as an example of this correction. A 25.5  $w/v$  solution of it gave  ${}^x\alpha^{16} = +6.3^\circ$  and  ${}^xI^{12} = -7.0^\circ$ . The latter, corrected by equations (2) and (5), became  ${}^{10}I^{20} = -6.16$ .

The approximate angle of the original inverted sucrose calculated by equation (12) =  $0.249 (3.023 \times 6.16 - 6.63) = 3.1^\circ$ .

This angle ( $-3.1$ ) is therefore a measure of the inverted sucrose originally present, and can be corrected as usual by equations (2) and (5), which give the value  $-2.95^\circ$ . The original angle can then be corrected by the result of this calculation, as the sucrose angle would not be affected by temperature and concentration:

$${}^{10}\alpha^{20} = 6.3 + (3.1 - 2.95) = 6.45^\circ.$$

The composition of the original solution can then be calculated by equations (9) and (10):

$$\text{Sucrose} = 0.565 (6.45 + 6.16) = 7.14 \text{ } w/v.$$

$$\text{Original inverted sucrose} = 0.565 (3.023 \times 6.16 - 6.45) = 6.88 \text{ } w/v.$$

As these were 25.5 *w/v* solutions, the syrup contained 28.0 per cent. of sucrose and 26.7 per cent. of inverted sucrose. The latter could be changed to invert sugar by multiplication by 1.05.

REDUCTION.—The following general equation for the calculation of *K*—*i.e.*, reduction expressed as dextrose and lævulose—is sometimes useful :

If “*a*” grm. of sugar or syrup is diluted to “*b*” c.c. = (1),  
and “*c*” c.c. of (1) is diluted to “*d*” c.c. by inversion = (2),  
and “*e*” c.c. of (2) is diluted to “*f*” c.c. = (3),  
and “*g*” c.c. of (3) = 10 c.c. of Fehling solution.

$$(17) K = \frac{5 b d f}{a c e g}$$

MIXTURES OF SEVERAL SUGARS.—When, in addition to the presence of sucrose, which is more or less inverted, glucose syrup is present, it is necessary to supplement the polarisation figures by a determination of the *K* and the organic solids both before and after fermentation. In such cases it is better to polarise at 20° C. instead of making the complicated temperature correction.

The fermentation may conveniently be performed as follows : Twenty grms. of the substance are weighed and mixed with 220 c.c. of water and 1 grm. of yeast, and the mixture is sterilised by boiling for a short time. After cooling, 3 grms. of yeast are added, and the flask is plugged with cotton-wool and incubated at about 28° C. for three or four days ; 100 c.c. of almost clear liquid are then decanted, and the remainder measured (= “*a*” c.c.). The 100 c.c. are evaporated down to less than one-half, alumina cream added, and diluted to  $\frac{10,000}{100+a}$  c.c., so as to give a 20 *w/v* solution. After filtration the solution is analysed. It is a convenience to start two fermentations at once, so that if the *K* of the first indicated that fermentation is incomplete, the second flask can be incubated for a day or two longer, or the turbid liquid residue of the first fermentation may be returned to the incubator.

The following analysis of a sample of war-time “high-class stoneless damson and apple jam” shows the results obtained with such a mixture. The specific rotatory power before inversion was +47.3°, and after inversion +29.3°. These values, calculated by equations (11) and (8), give the specific rotatory power due to sucrose as 13.5°, and showed that 20.3 per cent. of sucrose was present.

	Organic Solids.	<i>K</i> .	[ $\alpha$ ] <sub>D</sub> .
	Per Cent.		
Original ... ..	59.3	26	47.3
Subtracting sucrose ... ..	20.3	—	13.5
	39.0	26	33.8
After fermentation ... ..	16.0	3	24.6
Lost by fermentation ... ..	23.0	23	9.2

As the *K* of the fermented portion equalled the organic solids, the latter consisted of dextrose and lævulose. For this fermented sugar [ $\alpha$ ]<sub>D</sub> =  $\frac{9.2 \times 100}{23} = 40.4^\circ$ .

The percentage composition is then calculated by equation (15) :

$$\text{Invert sugar} = 1.35 (52.8 - 40.4) = 16.8 \text{ per cent.}$$

This shows that 16.8 per cent. of the 23 per cent. (= 3.9 per cent.) of the sugar fermented was invert sugar. The remainder of the *K* (26 - 3.9) may be taken to be dextrose. The optically active part of the 16.0 per cent. of residue after fermentation may be taken to be a mixture of dextrose and dextrin. The specific rotatory power due to the 3 per cent. of dextrose is subtracted.

			Organic Solids.	[ $\alpha$ ] <sub>D</sub> .
			Per Cent.	
Unfermented	...	...	16	24.6
Dextrose	...	...	3	1.6
Dextrin, etc.	...	...	13	23.0

$$\text{Dextrin [by equation (8)]} = \frac{100 \times 23}{200} = 11.5 \text{ per cent. This leaves } (13.0 - 11.5 =)$$

1.5 per cent. of other unfermented solids.

The complete analysis of the jam was as follows : Sucrose, 20.3; invert sugar, 3.9; dextrose, 22.1; dextrin, 11.5; other soluble organic solids, 1.5; ash, 0.8; insoluble matter, 1.2; and water (by difference), 38.7 = 100.0.

A careful examination of the insoluble matter of the jam only detected 0.04 per cent. of damson skin; so, in addition to being adulterated with glucose syrup, very little damson was present. There was some evidence that the jam had been dyed.

#### CONSTANTS AND FACTORS FOR SUGARS.

Sugar.	Molecular Weight.	$^{10}[\alpha]_D^{20}$ .	<i>B</i> .	<i>C</i> .	<i>c</i> .	<i>D</i> .	<i>d</i> .
Lactose :							
$C_{12}H_{22}O_{11}$ ...	342.24	55.3°	11.06°	—	—	0.079	0.0015
$C_{12}H_{22}O_{11} \cdot H_2O$ ...	360.26	52.5°	10.5°	—	—	0.075	0.0014
Maltose :							
$C_{12}H_{22}O_{11}$ ...	342.24	138°	27.6°	—	—	0.095	0.0007
$C_{12}H_{22}O_{11} \cdot H_2O$ ...	360.26	131°	26.2°	—	—	0.090	0.0007
Sucrose :							
$C_{12}H_{22}O_{11}$ ...	342.24	66.5°	13.3°	—	—	0.010	0.0001
Dextrose :							
$C_6H_{12}O_6$ ...	180.13	52.8°	10.56°	0.030	0.0005	—	—
Lævulose* :							
$C_6H_{12}O_6$ ...	180.13	95°	19.0°	0.110	0.0006	0.630	0.0066
Invert sugar* :							
$2(C_6H_{12}O_6)$ ...	360.26	21°	4.2°	0.070	0.0080	0.312	0.0149
Sucrose (inverted)* :							
$C_{12}H_{22}O_{11}$ ...	342.24	22°	4.4°	0.074	0.0077	0.327	0.0149
Dextrin :							
$n(C_6H_{10}O_5)$ ...	—	200°	40.0°	—	—	—	—



$^{10}[\alpha]_D^{20}$  = specific rotatory power of 10 w/v solution at 20° C. with sodium flame;  $B$  = rotation of 200 mm. 10 w/v solution at 20° C.;  $C$  = increase in numerical value of  $^{10}[\alpha]_D^{20}$  for increase of 1 w/v of sugar;  $c$  = concentration coefficient;  $D$  = decrease in numerical value of  $^{10}[\alpha]_D^{20}$  for increase of 1° C.;  $d$  = temperature coefficient; \* = left-hand rotation.



#### 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.*

#### APPROXIMATE ESTIMATION OF COMMERCIAL CRESOL IN LYSOL.

THE process described in an abstract in the ANALYST (1921, 375) can be accurate only where the soap used is free from volatile fatty acids. Serious mistakes have been made by allowing volatile fatty acids (such as occur in coconut oil, etc.) to distil over and augment the layer of tar acids in the distillate. To obtain accurate results when there is any doubt of the quality of the soap, the following method may be employed:

Dissolve the sample in hot water in a separator, add a piece of stick sodium hydroxide, and shake the liquid until the alkali has dissolved. Add excess of brine and separate the precipitate. Redissolve the residue in hot water, add a small piece of stick sodium hydroxide, and precipitate the soap again with brine; repeat the process once more to make sure of extracting all the tar acids. Acidify the united alkaline solutions and extract them with benzene. The benzene can then be extracted with a small quantity of strong sodium hydroxide solution, and the latter can be acidified in a burette and the tar acids read off in the usual way.

G. F. WESLEY MARTIN.



#### NOTES FROM THE REPORTS OF PUBLIC ANALYSTS.

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

#### ANNUAL REPORT OF THE BIRMINGHAM CITY ANALYST, 1920.

EGG POWDER.—Four of twelve informal samples were composed of tartaric acid, sodium bicarbonate, and rice starch. The other eight samples contained calcium hydrogen phosphate instead of tartaric acid, seven contained rice starch, and one a mixture of starches. One sample had been made with impure calcium hydrogen phosphate containing 55 per cent. of calcium sulphate, which is much in excess of the limit of 10 per cent. recommended for calcium hydrogen phosphate in a Local Government Board Report. The result was that the egg powder contained about 9 per cent. of calcium sulphate in excess.

The labels of some of the samples were very misleading, such as, for instance, "a perfect substitute for eggs." No action can be taken, however, for such misleading labels under the Food and Drugs Acts, although possibly the vendors may

be liable under the Merchandise Marks Act. The prices charged for the powders varied from 1d. to 2d. per ounce.\*

**CUSTARD POWDER.**—Sixteen of nineteen informal samples consisted of coloured and flavoured starch, the starches found being maize, arrowroot, potato, tapioca, and rice. One sample, stated to contain 24 per cent. of proteid nourishment, or forty times as much as ordinary custard powder, contained only 5·4 per cent. of proteins. Another sample labelled "Sweetened with pure sugar only" contained about 50 per cent. of sugar; but two samples of the same make, marked "Sweetened, no sugar required," contained no sugar, but about 0·2 per cent. of saccharin. These two samples were marked as adulterated, and particulars of the case have been sent to the Ministry of Health.

#### REPORT FOR THE FIRST QUARTER, 1921.

**NATIONAL HEALTH INSURANCE DISPENSING—*Quinine Mixture.***—An informal sample contained 21 grains of quinine sulphate and 183 minims of dilute hydrobromic acid, whereas the prescription ordered 32 grains and 240 minims respectively. A subsequent formal sample from the same vendor contained 19 grains of quinine sulphate and 206 minims of diluted hydrobromic acid. The explanation given was that the correct quantities had been dissolved in a Winchester quart bottle which held 100 ounces, whilst the compounder had thought that it held only 80 ounces. The arrangement between the Birmingham Insurance Committee and panel chemists does not recognise the use of such stock mixtures, and requires that each prescription shall be separately dispensed. The vendor was fined. J. F. LIVERSEGE.

#### ANNUAL REPORT OF THE BOROUGH ANALYST FOR THE METROPOLITAN BOROUGH OF STEPNEY FOR THE YEAR 1920.

**MILK.**—Roughly, one sample in seventeen was adulterated, compared with one in six in 1918, and one in twelve in 1919. Notwithstanding this improvement, a simple calculation shows that there is still a great deal of tampering with milk in the borough.

The average composition of the 858 samples of milk analysed during the year was: Total solids, 12·40; solids, not fat, 8·81; and fat, 3·59 per cent. These figures, which include those of grossly adulterated samples, entirely refute the statement, frequently made in court and elsewhere, that farmers have difficulty in producing milk to satisfy the minimum limits prescribed by the Sale of Milk Regulations. One of the samples contained 35 grains per gallon of boric acid. Proceedings were taken under the Public Health (Milk and Cream) Regulations, and the vendor was fined £100.

**BORIC ACID IN BUTTER AND MARGARINE.**—Of 89 samples of butter analysed, 46·3 per cent. were free from preservative, 26·2 per cent. contained 0·1 per cent. of boric acid, 17·5 per cent. contained 0·2 per cent., 5 per cent. contained 0·3 per cent., and 5 per cent. contained 0·4 per cent. These figures show that butter can be sold free from preservative, and I consider that preserved butter should be required to have a declaratory label similar to that required in the case of preserved cream.

Seventy-three samples of margarine were analysed, 10·9 per cent. of which were free from preservative, whilst 8·7 per cent. contained 0·1 per cent. of boric acid, 43·5 per cent. contained 0·2 per cent., 32·5 per cent. contained 0·3 per cent., and 4·4 per cent. contained 0·4 per cent. HERBERT HAWLEY.

\* Cf. Beach, Needs, and Russell, ANALYST, 1921, 279.

## DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH.

## REPORT OF THE FOOD INVESTIGATION BOARD FOR THE YEAR 1920.\*

**FISH PRESERVATION COMMITTEE.**—An interim report on the method of preserving fish by freezing them in brine has been published. Further results obtained in the investigation of the freezing process, and in the rate of penetration of salt from brine of definite concentrations, have also been published (Adair, *Biochem. J.*, 1920, 14, 762).

**MEAT COMMITTEE.**—It is commonly accepted that the formation of lactic acid plays an important part in the ripening of meat and in the changes undergone by flesh on prolonged storage. Experiments have shown that continuous exposure to the freezing-point will produce a non-irritable condition in frog's muscles, and that too without the production of lactic acid, if the muscles be kept in air; but that, if kept in nitrogen, the loss of irritability is much less rapid, and lactic acid is formed. Slow autolysis takes place during the storage of meat, but whether lactic acid is formed has not yet been determined (Foster and Moyle, *Biochem. J.*, 1921, 15, 334).

A simple technique for the cultivation of anaerobic bacteria has been devised, and the spore formation of these bacteria investigated. The conditions under which mould fungi develop upon meat have also been studied (Brooks and Kidd, *ANALYST*, 1921, 292).

**ENGINEERING COMMITTEE.**—Investigations of the thermal conductivity and specific heat of insulating materials, on the thermodynamics of liquid carbonic acid (*Proc. Roy. Soc.*, 1920, 98A), and on the question of the measurement of humidity have been carried out.

**FRUIT AND VEGETABLE COMMITTEE.**—Experiments on the storage of English apples have been made with a view to determining the most suitable conditions. The method of "gas storage" approximately doubled the storage life of the varieties tested. Experiments on the preservation of fruit by freezing for jam-making have shown that the only fruits which can be satisfactorily kept in this way are those which only contain a peroxidase, but no oxygenase or catechol substance (Onslow, *ANALYST*, 1921, 292). The following fruits could therefore be kept in a frozen condition for several months in air for jam-making: Strawberries, raspberries, black currants, red currants, and gooseberries; whilst cherries and plums could only be kept unaltered in a frozen condition in an atmosphere free from oxygen. The effect of an excess of carbon dioxide on stored apples is very similar to that of low temperature. The amount of sucrose and pectin present in the juice decreases as the concentration of carbon dioxide increases; a similar increase also takes place in the ratio of fructose to dextrose, but the acidity is but little affected.

Mangin's observations on the existence of pectic compounds in three forms have been confirmed, and methods have been worked out for the extraction of each form of pectin. A new method of estimating pectin, based on its precipitation from an aqueous extract by means of alcohol, has been devised, and it has been established that more pectin can be obtained from fruit tissue by the action of heat and acid than is originally present in the soluble form. At a certain stage of over-ripeness the pectic acid seems to disappear. Experiments are being made to ascertain whether the enzyme pectase is concerned in the production of pectic acid in the fruit.

**OILS AND FATS COMMITTEE.**—Investigations of the syntheses of isomeric oleic acids have been continued, and a further examination has been made of the unsaturated fatty acids of reduced linseed oil (iodine value, 96). It has been found that the main constituent of the unsaturated fatty acids is oleic acid, and evidence

\* Copies of the Report can be obtained from H.M. Stationery Office, Imperial House, Kingsway, W.C. 2. Price 1s. net.

has been obtained that an olein results from the reduction of the glycerides of linolic acid.

The constitution of the synthetic fats obtained by the interaction of methyl glucoside or mannitol and olive oil has been studied. In the case of the methyl glucoside compounds condensation, resulting in the union of one oleyl residue with the carbohydrate chain, takes place first, and, later, the carbohydrate chain loses one molecule of water, and the synthetic fat anhydromethyl glucoside mono-oleate is obtained. In the case of the compounds of mannitol and olive oil two oleyl residues take part in the reaction.

*Sterols and the Iodine Value.*—The abnormally high iodine value and low saponification value of yeast fat have been proved to be due to the presence of ergosterol (a sterol believed by Tanret to be characteristic of the lower plants). In determining the iodine value of sterols by means of Wijs' reagent a considerable amount of substitution occurs, owing to the influence of the glacial acetic acid. This substitution effect is particularly noticeable when a condensed ring nucleus containing one double bond reacts with the Wijs' solution. Hence, in the case of a fat which contains an appreciable amount of sterol, the Hübl and Wijs' iodine values will differ to some extent, and the former value will more accurately estimate the degree of unsaturation of the fat. An attempt is being made to apply the difference between the iodine values obtained by the two methods under controlled conditions as a means of estimating the sterol in a fat (MacLean and Thomas *Biochem. J.*, 1921, 15, 319).

*CANNED FOODS COMMITTEE.*—Results obtained by W. Savage have proved that putrefying meat has little obvious effect upon the health of animals, and the conclusion is drawn that cases of poisoning from tinned meat are due neither to putrefactive bacteria nor to their products, but to the presence of some specific and highly toxic organism, such as Gaertner's bacillus.

The chemical changes occurring in untreated and canned herrings have been studied. Although it is improbable that autolytic enzymes ever act alone in untreated herrings, there is reason to believe that without the intervention of bacteria they might bring about an improvement in quality. On the other hand, deterioration is probably due to the further action of bacteria. In the case of tinned herrings, the bacterial product might arise before or after canning. Since trimethylamine is not, so far as is known, produced by enzymic action, and appears at an early stage in decomposing herrings, its estimation should throw light upon the problem. A new process of estimating it in the presence of other volatile bases has been based on Foreman's alcohol titration method (*ANALYST*, 1920, 45, 380), and is being used in investigating the question.

Estimations of the total acid radicles, volatile organic bases, and amino-acids (Foreman, *loc. cit.*) in canned and fresh herrings have shown that the former contain about twice the quantity of amino-acids, sixteen times the volatile acids, six to seven times the organic acid radicles, and five times the "net acidity" present in the latter. The small increase in amino-acids indicates that proteolysis only proceeds to a small extent in the canning and maturation. It seems probable that one or more highly nitrogenous constituents of the herring is destroyed, with the production of volatile bases.

Fresh herrings are distinctly acid in reaction, and it is probable that the changes produced in canning are largely due to the action of the acids under the influence of heat on the complex compounds present. Tinned herrings contain appreciable amounts of tin, as well as a small amount of iron, and it is possible that compounds of these metals may have a catalytic effect in continuing at the ordinary temperature the changes occurring in the canning process.

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## U.S.A. SERVICE AND REGULATORY ANNOUNCEMENTS.

MISBRANDING OF EGG SUBSTITUTE (*U.S. Dept. Agric. Bureau of Chemistry, Service and Regulatory Announcements*, 1921, N.J. 9290).—Analysis of a sample of "Eggno," stated on the label to be "an excellent substitute for eggs . . . to be used for baking and cooking purposes," showed that it consisted of rice starch, skim milk powder, and milk casein or albumin, artificially coloured with a coal-tar dye, and possibly containing a small amount of egg powder. Baking tests made by the Bureau showed that cakes made with the product were no better in volume or quality than cakes made with water, and were in no measure comparable in volume or quality with cakes made with an equivalent amount of eggs, as claimed on the label. The case was heard on December 9, 1920, in a District Court of the United States, and the defendants were convicted and fined. Notice of appeal was given, and the case is pending.



## ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

## FOOD AND DRUGS ANALYSIS.

Proportion of Fat in Casein. D. M. Gangolli and A. N. Meldrum. (*Dept. Industries, Bombay Presidency, Bull. No 4*, 1921.)—A method recommended for the estimation of fat in casein consists in placing 15 c.c. of  $\frac{N}{2}$  sodium hydroxide solution in a separating funnel, adding 1 gm. of the powdered casein gradually, and shaking the mixture until the casein is dissolved completely. The mixture is then extracted with ether, the ethereal solution evaporated, and the residue of fat weighed. Solution of the casein in hydrochloric acid tends to give high results for the fat, owing to partial hydrolysis of the casein, with the formation of substances which are soluble in ether. The average quantity of fat in casein is 0.25 per cent., but is usually more than this in the case of Indian casein. The colour of casein depends largely on the amount of fat present; pure casein may be prepared by washing the crude product with alcohol, and then extracting it repeatedly with petroleum spirit. Ether should not be used, as it is liable to contain peroxide, which may contaminate the casein.

W. P. S.

Separation and Identification of "Sandy" Crystals in Ice Cream. H. F. Zoller and O. E. Williams. (*J. Agric. Research*, 1921, 21, 791-795).—It is generally assumed that the "sandiness" of ice cream is due to the separation of sucrose crystals, although no scientific evidence is given in support of this statement. A number of "sandy" ice creams were allowed to melt at the ordinary temperature, the "sand" was separated by means of the centrifuge, washed with water saturated with sucrose and lactose, and with this solution containing 50 per cent. of acetone, then dried on a Buchner funnel, and, finally, by heat in a water oven. Microscopic examination, crystallographic measurements, and analysis by the Munson and Walker method identified the "sand" as  $\alpha$ -lactose (lactose monohydrate).

T. J. W.

**Estimation of Starch by means of Taka-Diastase. E. Horton.** (*J. Agric. Science*, 1921, **11**, 240-257.)—The results obtained in the estimation of starch in wheat by the use of taka-diastase varied according to the sample and quantity of the enzyme employed. These variations led to an extensive investigation into the action of this enzyme upon pure potato starch. In the method applied, with modifications, the starch, gelatinised in boiling water, was cooled to 38° C., when toluene and the enzyme were added, and the temperature maintained at 38° C. for sixteen to twenty-four hours. After the mixture had been heated to 100° C. for fifteen minutes, sodium fluoride was added, and the solution was cooled, treated with basic lead acetate solution, diluted to definite volume, and filtered. Excess of lead was then removed by the gradual addition of powdered sodium carbonate, and, after filtration, the rotatory and reducing powers were determined, and the starch calculated from the dextrose and maltose found. The purity of the starch employed was ascertained by the use of the same method with malt diastase instead of taka-diastase. The factors studied included the following: Source of enzyme, age of enzyme, age of *Aspergillus oryzae* culture from which the enzyme was prepared, variation in amount of enzyme used, time of hydrolysis, addition of yeast extract or of malt diastase to the enzyme, and variation in the clarifying agents used. The results obtained varied between 86 and 97·8 per cent. of starch, and, although great differences in the dextrose-maltose ratio were found, a large proportion of the results was within the limits of 91 and 96 per cent. The conclusion is drawn that the taka-diastase method is unreliable, and test experiments should be made upon pure starch with every sample of enzyme before and during use. It is possible that the discrepancy observed is due to the persistence of dextrin, but this has not been proved.

T. J. W.

**Examination of Sugar Crystals by Projection. G. P. Meade.** (*J. Ind. Eng. Chem.*, 1921, **13**, 712.)—A small amount of the sugar to be examined is placed in a 40 mm. Petri dish, a few c.c. of alcohol saturated with sugar are added, and the crystals are separated and distributed over the dish. The latter is then fitted in the holder of the projection apparatus. The author uses a Bausch and Lomb balopticon with a lens of 8-inch focus, the projection lens having a diameter of 1·6 inches; the instrument is provided with a vertical attachment, in order that the object to be projected may be placed in a horizontal position. A magnification of ten diameters is obtained when the centre line of the projection lens is 7 feet from the screen. The latter consists of a smooth plaque of plaster of Paris about 18 inches square, and ruled into squares of various sizes (for example, 4, 6, 8, 10, and 12 mm. squares), so that, allowing for the magnification, the size of the crystals may be measured.

W. P. S.

**Revision of the Optical Method for Analysing Mixtures of Sucrose and Raffinose. C. A. Browne and C. A. Gamble.** (*J. Ind. Eng. Chem.*, 1921, **13**, 793-797.)—The change in the Herzfeld divisor of the Clerget formula for estimating sucrose from 142·66 to 143·0 has necessitated a revision of the Creydt formula for analysing mixtures of sucrose and raffinose. On the basis of the invert factor—0·33 for sucrose and +0·514 for raffinose, and 1·852 as the ratio of the specific rotation of sucrose to that of raffinose, the following formulæ give the percentages of sucrose (S)

and raffinose (*R*) in a mixture when the sucrose normal weight of the sample is taken for the estimation :

$$S = \frac{0.514P - P'}{0.844} \text{ at } 20^\circ \text{ C., and } R = \frac{0.33P + P'}{1.563} \text{ at } 20^\circ \text{ C.}$$

*P* is the direct polarisation, and *P'* the invert polarisation of the solution. General formulæ applicable at all temperatures are :

$$S = \frac{P(0.478 \times 0.0018t) - P'(1.006 - 0.0003T)}{(0.908 - 0.0032t)(1.006 - 0.0003T)}, \text{ and}$$

$$R = \frac{P(0.43 - 0.005t) + P'(1.006 - 0.0003T)}{(1.681 - 0.0059t)(1.006 - 0.0003T)},$$

where *T* is the temperature of the direct polarisation, and *t* that of the invert polarisation. W. P. S.

**Detection of Tartaric Acid in Wines.** L. Mathieu. (*Ann. Chim. anal.*, 1921, 3, 270-271.)—Free tartaric acid in wine may be separated from potassium hydrogen tartrate and calcium tartrate by means of amyl alcohol. Twenty c.c. of wine are shaken with about an equal volume of amyl alcohol, and, after separation, the clear layer of amyl alcohol is removed and shaken with an equal volume of water. The aqueous layer is then separated and evaporated to dryness to remove the last traces of amyl alcohol, which causes the precipitation of calcium lævo-tartrate, and the residue is dissolved in water (5 to 6 c.c.) and treated with a drop of calcium chloride solution (150 grms. CaCl<sub>2</sub>.6H<sub>2</sub>O and 40 grms. NH<sub>4</sub>Cl per litre) and 2 c.c. of ammonium lævo-tartrate free from dextro-tartrate (20 grms. per litre of 20 per cent. alcohol). In the presence of tartaric acid a precipitate of calcium racemate is formed immediately; 0.5 gm. of free tartaric acid per litre of wine may be detected by the above method, and smaller amounts by using a larger sample of the wine.

R. G. P.

**Chemical Composition of Arachis Oil.** G. S. Jamieson, W. F. Baughman, and D. H. Brauns. (*J. Amer. Chem. Soc.*, 1921, 43, 1372-1381.)—Two specimens of arachis oil expressed in the laboratory from nuts of the white Spanish type and the Virginian type had iodine values of 90.1 and 94.8 respectively. The mixed fatty acids yielded no linolenic hexabromide, but a linolic tetrabromide. The proportions of linolic and oleic acids were calculated from the percentages of bromine in the bromides. The saturated fatty acids were separated by the lead-ether method, and fractionated by distillation of their methyl esters under reduced pressure, corrections being made from the iodine and saponification values for the amounts of unsaturated fatty acids in each fraction. By these means the two oils were found to have the following percentage composition :

	Oleic Acid.	Linolic Acid.	Palmitic Acid.	Stearic Acid.	Arachidic Acid.	Lignoceric Acid.	Unsaponifiable Matter.
Oil from Spanish nuts ...	52.9	24.7	8.2	6.2	4.0	3.1	0.2
Oil from Virginia nuts ...	60.6	21.6	6.3	4.9	3.3	2.6	0.3

No hypogæic acid or behenic acid was present.

**Detection of Tea-Seed Oil in Olive Oil.\*** J. Dybowsky and E. Millia. (*Comptes rend. Acad. Agric. de France*, 1920, **6**, 577-581; *Inter. Rev. Sci. Agric.*, 1921, **11**, 1193-1194.)—The presence of 10 to 20 per cent. of tea-seed oil in olive oil may be detected by shaking 20 grms. of the oil for one minute with 6 drops of sulphuric acid; the mixture is then treated with 9 drops of nitric acid, shaken for one minute, heated at 100° C. for five minutes, and finally cooled to 10° C. After two hours at the latter temperature the mixture remains liquid if 20 per cent. of tea-seed oil is present; if the original oil contained 10 per cent. of tea-seed oil the mixture solidifies, but the mass is not as hard as that obtained with pure olive oil. A characteristic reaction, which will detect as little as 5 per cent. of tea-seed oil, consists in shaking 4 c.c. of the oil for thirty seconds with a mixture of 5 c.c. of pure sulphuric acid, 3 c.c. of nitric acid, and 3 c.c. of water; the whole mixture is kept at 5° C. for five minutes and observed after a further fifteen minutes. Pure olive oil gives a straw coloration and remains clear, pure tea-seed oil becomes turbid and sooty-black, whilst olive oil containing 5 per cent. of tea-seed oil gives a dark straw-coloured, turbid mixture. These colorations are observed in the oily layer; the acid layer is colourless in all cases.

W. P. S.

**Soya-Bean Oil.** C. R. Fellers. (*J. Ind. Eng. Chem.*, 1921, **13**, 689-691.)—The quantity of oil in soya beans varies considerably; in twenty-six varieties of the bean examined, the amount ranged from 14.6 to 25.6 per cent.; the quantity of protein varied from 35.7 to 44.3 per cent. The sp. gr. of the oil is practically constant at 0.925, and the refractive index at 25° C. varies but little from 1.474; the iodine value ranges from 123.2 to 132.3, and the saponification value from 190 to 195. There appears to be some relation between the colour of the beans and the drying property of the oil, dark beans yielding oil of rather poor drying power.

W. P. S.

**Chemical Investigation of Mutton-Bird Oil.—I.** C. L. Carter. (*J. Soc. Chem. Ind.*, 1921, **40**, 220T.)—The oil extracted from the stomach of the Australasian petrel (*Estrelata lessona*) is a fairly thin, straw-coloured, non-drying oil, closely resembling sperm oil in chemical characteristics. The oil contains 38 per cent. of alcohols, chiefly cetyl alcohol, and 61 per cent. of fatty acids, which are chiefly oleic acid with some acids of the linolic or linolenic series. At 6° C. spermaceti separates out, and at 0° C. the oil solidifies. The chief constants are: Sp. gr. at 15° C., 0.884; free fatty acids, 2.23 per cent.; alcohols, 38.4 per cent.; saponification value, 119.6; iodine value, 130 (Wijs), 108 (Hübl); viscosity, 0.3724; m.-pt. of alcohols, 31° to 31.5°; saponification value of acetate, 189; and iodine value of fatty acids, 150 (Hübl), 178 (Wijs). (Cf. *ANALYST*, 1911, **36**, 290.)

H. E. C.

**Sweet Fennel Oil.** C. E. Sage. (*Perf. Essent. Oil Rec.*, 1921, **12**, 46; *Chem. Abstracts*, 1921, **15**, 1597.)—Samples of this oil obtained on the market are in some cases apparently derived from the whole Spanish plant instead of from the fruit only. They are distinguished from the genuine oil by the almost entire absence of anethole,

\* The tea-seed oil giving these reactions had probably not been refined.—EDITOR.



and are suitable for flavouring purposes only. Two samples examined showed the following characteristics: Sp. gr. at 15.5° C., 0.9203 and 0.9340;  $n_D^{25}$ , 1.4945 and 1.4980;  $[\alpha]_D$ , +46 and +24; solubility in 90 per cent. alcohol, 1 in 1; and both remained liquid on cooling to -15° C. It is suggested that such products should be designated "plant oils," in contradistinction to the "fruit oils" usually manufactured.

T. J. W.

**Identification of Ouabain and Strophanthin.** A. Richaud. (*J. Pharm. Chim.*, 1921, [vii.], 24, 161-166.)—For the identification of these glucosides the following properties serve: Ouabain forms a pure white powder of nacreous appearance, and crystallises in rectangular plates. It dissolves in 150 parts of water at 15° C., giving an absolutely colourless, transparent solution, which has a slight bitter taste, and gives no lasting froth when shaken. If a few crystals of resorcinol and then a few of ouabain are added to 4 to 5 c.c. of concentrated hydrochloric acid in a test-tube, and the latter is heated to 60° to 70° C. in a water-bath for a few moments, no coloration appears. Strophanthin is usually a dirty white or very pale yellow powder, and is amorphous or crystallised in spangles, often arranged radially; 1 part dissolves in 40 to 43 parts of water at 15° C., giving a solution which is not absolutely colourless or transparent, has a very marked bitter taste, and yields a persistent froth when shaken. When treated with hydrochloric acid and resorcinol, as described above, strophanthin gives a pink coloration.

T. H. P.

**Microchemical Tests for Saccharin and its Salts.** Denigès. (*Bull. Soc. Pharm. Bordeaux*, 1921, No. 2; *Ann. Chim. anal.*, 1921, 3, 273-275.)—The silver salt of saccharin is produced by treating on a microscope slide less than 1 mgrm. of a soluble salt of saccharin, such as the ammonium salt ("sucramine") or the sodium salt ("sucrose"), with a drop of ammoniacal silver nitrate solution (3 per cent.). The crystals are of characteristic appearance, and serve for the microchemical identification of saccharin salts. Saccharin itself, which is almost insoluble in water, is first dissolved in a drop of ammonia, and the solution evaporated to dryness on the microscope slide. The crystals of saccharin obtained by the treatment of the sodium or ammonium salt with dilute sulphuric acid (10 per cent.), or with concentrated hydrochloric acid, are also of characteristic appearance. The sodium salt of saccharin may be detected by the formation of cubic crystals of sodium chloride produced on acidification with hydrochloric acid, whilst the ammonium salt yields prismatic crystals of hydrogen ammonium tartrate when treated with sodium hydrogen tartrate.

R. G. P.

**Derivatives of Sulphur in Commercial Salvarsan.**—II. H. King. (*J. Chem. Soc.*, 1921, 119, 1415-1420.)—The substance  $C_6H_8O_6NSAs$ , obtained as a by-product in the reduction of 3-nitro-4-hydroxy-5-sulphinophenylarsinic acid with sodium hyposulphite, has now been proved to be the sulphonic acid of phenylarsenious acid, and not the sulphinic acid of the phenylarsinic acid. Hence, the amorphous arsenobenzenes containing the sulphinic acid group in commercial salvarsan (ANALYST, 1921, 407) are in reality identical with the corresponding sulphonic acids,

and the slight differences in analytical figures and toxicity are those of amorphous substances prepared under different conditions. The principal sulphur-containing impurity in commercial salvarsan is 3 : 3'-diamino-4 : 4'-dihydroxy-5-sulpharseno-benzene monohydrochlorides associated with varying proportions of "salvarsan sulphate."

### BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

**Detection of Secretion Disturbances in Milk.** G. Koestler. (*Mitt. Lebensm. Hyg.*, 1920, **11**, 154-169; *Chem. Abstracts*, 1921, **15**, 1581.)—Very slight pathological changes affect the milk, causing an increase in the serum-soluble nitrogen, chlorine, and sodium, and a decrease in the lactose, potassium, and phosphoric acid. The extent of the disturbance may be measured by calculation of the "chlorine-sugar" number from the formula  $100 \left( \frac{\text{chlorine per cent.}}{\text{lactose per cent.}} \right)$ . For normal milks this value was found to be 0.49, whilst for a pathological milk having a normal appearance the value was 15. The ratio of chlorine to phosphoric acid in the ash may be employed in a similar manner for the detection of pathologically abnormal milk. The abnormal milks examined showed a normal depression of the freezing-point.

T. J. W.

**Estimation of Small Quantities of Phosphates in Biological Products.** G. Denigès. (*Comptes rend. Soc. Biol.*, 1921, **84**, 825; *J. Pharm. Chim.*, 1921, [vii.], **24**, 184-186.)—The reagents employed in this method are: (a) Sulphomolybdic reagent, prepared by mixing equal volumes of 10 per cent. ammonium molybdate solution and concentrated sulphuric acid; (b) stannous chloride: 0.1 gm. of thin tinfoil is treated in a test-tube with 2 c.c. of hydrochloric acid and 1 drop of 3 to 4 per cent. copper sulphate solution, the liquid being warmed gently until the metal dissolves, and the solution diluted to 10 c.c. with water, allowed to cool, and decanted from any deposit. Before the test is applied, the organic matter must be destroyed in either of the two following ways: (1) The substance is incinerated with one-third or one-fourth of its weight of calcined magnesia free from phosphates, the residue taken up in water containing a little sulphuric acid, and the liquid diluted to a known volume after prolonged boiling to transform pyro- or meta-phosphoric ions into phosphoric ions. (2) By means of nitric and sulphuric acids: With blood, for instance, 1 gm. or 1 c.c. is mixed in a porcelain dish about 8 cm. wide with 1 c.c. of nitric acid, 1 c.c. of sulphuric acid, and, drop by drop, 30 to 40 drops of 90 to 95 per cent. alcohol. When effervescence ceases, the liquid is covered with a funnel fitting inside the dish, which is heated gently on gauze or asbestos card. If the liquid darkens when white sulphuric acid fumes appear, it is heated again with a few drops of nitric acid, these operations being continued until the solution remains uncoloured, when all the nitric acid is expelled. The liquid is then cooled, diluted with 10 to 12 c.c. of water, boiled to one-half its volume, and finally made up to 200 c.c. To 5 c.c. of the solution obtained by either of these methods are added 4 drops of the above sulphomolybdic reagent, and, after mixing, 2 to 4 drops of the stannous chloride solution, which must be freshly prepared. After further mixing, the tube is left for ten

minutes, and the blue coloration of its contents then compared with those of the liquids in a series of tubes similarly prepared from 5 c.c. of solutions containing from 0.001 to 0.012 gm. of  $P_2O_5$  per litre. This reaction is most sensitive with solutions containing between 0.0005 and 0.010 gm. of phosphorus per litre. T. H. P.

**Rose Bengal as a General Bacterial Stain.** H. J. Conn. (*N.Y. Agric. Expt. Stat. J. Bact.*, 1921, 6, 253-254; *Chem. Abstracts*, 1921, 15, 1551.)—Rose bengal readily stains bacterial protoplasm, but has little affinity for slime and debris surrounding the organism. It is of particular value for staining slime-forming bacteria, such as *B. radicumicola*. The stain, which remains in good condition for several months, is prepared by dissolving 1 gm. of rose bengal in 100 c.c. of 5 per cent. phenol solution. T. J. W.

**Detection of Manurial Pollution in Milk by the Anaerobic Spore Test.** J. Weinzirl. (*Amer. J. Public Health*, 1921, 11, 149-152; *Chem. Abstracts*, 1921, 15, 1581-1582.)—From 0.5 to 1 gm. of melted paraffin wax is poured into a test-tube, which is then plugged with cotton-wool and sterilised. Five c.c. of the milk under examination are run from a sterile pipette into each of five tubes containing the wax, and the whole are heated to 80° C. for ten or fifteen minutes. The tubes are then incubated at 37° C. for three days, when the presence of anaerobes is indicated by the evolution of gas, which lifts the wax plugs. Two positive results out of five indicate excessive pollution. The results obtained suggest that the number of infected samples detected is probably too low, owing to caramelisation of the lactose present, but a higher proportion is found by the addition of lactose to the milk. The method is considered to yield results of equal value to those obtained by the *B. coli* test. T. J. W.

**Notes on *B. coli* and *B. aerogenes*.** M. Levine. (*Amer. J. Public Health*, 1921, 11, 21-23; *Chem. Abstracts*, 1921, 15, 1740.)—The growth of *B. coli* is inhibited in 0.5 per cent. peptone medium containing 1 part per 200,000 of crystal violet, or 1 part per 1,000,000 of brilliant green. The concentration of the peptone in culture media has a decided influence upon the inhibiting action of dyes upon bacterial growth. The growth of *B. coli* is stimulated by a bile-salt concentration of less than 0.5 per cent., but is partially inhibited when the concentration is increased to 0.7 or 1.0 per cent. The production of both liquid and solid media which will inhibit the growth of *B. coli*, and not that of *B. aerogenes*, appears practicable. T. J. W.

**Souring of Beef caused by *Bacillus megatherium*.** H. Bunyca. (*J. Agric. Research*, 1921, 21, 689-698.)—The condition known as "sour beef" is due to an acid fermentation, which occurs when the meat is stored, while still warm, under conditions which do not allow it to cool. An organism was isolated from the interior portion of a sample of such meat, and was found to correspond closely in morphology and cultural characteristics with *B. megatherium* var. *Ravenellii* (from soil) and *B. megatherium* var. *de Bary* (from cabbage infusions). It was a round-ended, chain-forming, spore-bearing, aerobic, slightly motile rod, from 2 to 5 $\mu$  long and 2 $\mu$  wide

During sporulation the organisms became symmetrically swollen; the spores were equatorial. Colonies of twenty-four hours' growth were densely opaque at the centre, whilst the periphery was granular, and had the appearance of irregular circles. When grown on agar and potato, the organisms formed yellowish growths, resembling those of *B. subtilis*. This bacillus, like *B. megatherium* var. *de Bary*, produced the "sour beef" condition in sterile specimens of raw beef, the optimum temperature being about 37° C. The offensive putrefactive odour was due to propionic acid, which could be isolated by extracting the sour beef with 5 per cent. sulphuric acid and distillation of the filtered extract with steam. The organism was not pathogenic to guinea-pigs, and did not produce any appreciable amount of toxin in ordinary media or raw beef under laboratory conditions.

**Serological Method for Detection of Infected Foods.** J. Bronfenbrenner and M. J. Schlesinger. (*Proc. Soc. Exp. Biol. Med.*, 1921, 17, 24-25; *Chem. Abstracts*, 1921, 15, 1766.)—The whole sample is finely chopped, and an extract prepared, concentrated to a small volume, and tested against a set of specific immune sera. The presence of *B. botulinus* protein in 20 gm. samples of inoculated food was detected when the proportion of toxin was so small that a mouse weighing 15 to 20 grms. required 7 grms. administered by the mouth, or 1.3 grms. by injection, to show definite symptoms. If a collection of specific sera of high titre is available, the above procedure may be completed within twenty-four hours. T. J. W.

**Colour Reaction Common to Antiscorbutic Extracts and Hydroquinone.** M. Bezssonoff. (*Comptes rend.*, 1921, 173, 466-468.)—Folin's reagent for phenols is prepared by boiling for two hours a mixture of 100 grms. of sodium tungstate, 20 grms. of phospho-molybdic acid, and 50 c.c. of concentrated phosphoric acid, in a litre of water. When used in the presence of an excess of sodium carbonate it gives a blue coloration with mono-, di-, and triphenols and their derivatives, and with many vegetable substances containing or not containing vitamin C. A modified reagent, in which the quantity of phosphoric acid is reduced to a third, and to which, without boiling, an equal volume of *N*-sulphuric acid is added, gives a slate-grey coloration, changing to blue, with vegetable juices (orange, lemon, tomato, grape, cabbage) known to have pronounced antiscorbutic power. On the other hand, extracts of seeds (oats, turnip) without antiscorbutic power do not give the coloration. The plum, which is an anomaly among fresh vegetable products by reason of its being devoid of antiscorbutic power, does not give a blue coloration, but a yellowish-brown coloration, persisting for three hours; whereas the peach, belonging to the same genus as the plum, gives the blue coloration. Causes which diminish or destroy the antiscorbutic power, such as exposure to the air or boiling, also reduce or inhibit the blue coloration. Potato juice extracted in the presence of acid gives a distinct blue coloration; whereas the juice extracted without acid (weak antiscorbutic power) gives no coloration, or only a faint one. Cow's milk, which has only a slight antiscorbutic power, does not give the reaction, except a fugitive coloration at the moment of adding the reagent in the case of chilled milk. Urine from a man whose diet has been sufficiently rich in the antiscorbutic principle

gives a positive reaction. It is suggested that the reaction is probably due to an unstable radicle, probably a polyphenol, which readily becomes detached from vitamin C in its solutions. The reactions given by this reagent with phenols are essentially different from those obtained with Folin's reagent. Monophenol, salicylic acid, and tyrosin do not give any reaction; whilst tannin and guaiacol give a brownish-yellow coloration. The only compound tried which gives the blue coloration of Folin's reaction is hydroquinone. Pyrocatechol and catechol give a reddish violet coloration changing to blue, whilst phloroglucinol gives no coloration. The complex giving the reaction can be separated in soluble monoclinic crystals having a composition corresponding with the formula— $(17\text{WO}_3)(\text{MoO}_3)(\text{P}_2\text{O}_5\cdot\text{H}_2\text{O})21\text{H}_2\text{O}$ . This differs from the complex in Folin's reagent in having a lower proportion of molybdenum trioxide to that of tungsten trioxide.

**AGRICULTURAL ANALYSIS.**

**Estimation of Clay in Heavy Soils. A. F. Joseph and F. J. Martin.**

(*J. Agric. Science*, 1921, 11, 293-303.)—The Sudan method, as devised by Beam (*Cairo Scientific J.*, 1911, 107-119), is recommended as preferable to the English or American methods generally used. One hundred grms. of soil are dried at 100° C., and sifted through a 2 mm. circular mesh. The portion passing through is again sifted through a 1 mm. mesh. Five grms., or less, of the portion passing through are shaken for two hours with 100 c.c. of 0.2 per cent. sodium carbonate solution. The mixture is transferred to a beaker, diluted with water to a height of 10 cm., well mixed, and allowed to stand for sixteen hours, when the clay suspension is decanted, and the residue treated with 0.05 per cent. sodium carbonate solution, left to stand, and again poured off, this being repeated six to nine times. A final decantation is made with water, and the residue dried at 100° C. and weighed. The silt is removed by washing the dried residue into a beaker, and after leaving it for seven and a half minutes in a 10 cm. column of water, decanting the liquid, washing the remainder back into the original beaker, drying and weighing it, subsequently separating it into "coarse sand" and "fine sand" by sifting it through a mesh of 0.2 mm. The "coarse sand" is added to the portion already obtained not passing the 1 mm. sieve. A centrifugal method is also described which requires constant attention, but allows of results being obtained in much less time. In both cases the time required is considerably less than with either the American or English methods. The soil particles are classified in the three methods according to the following table :

	METHOD.		
	English.	American.	Sudan.
Gravel and stones ...	Above 1 mm.	1 to 2 mm.	Above 2 mm.
Coarse sand ...	1 to 0.2 mm.	1 to 0.05 mm.	2.0 to 0.2 mm. 0.2 to 0.02 mm.
Fine sand ...	0.1 to 0.04 mm.		
Coarse silt ...	0.04 to 0.01 mm.	0.05 to 0.005 mm.	0.02 to 0.002 mm.
Fine silt ...	0.01 to 0.002 mm.		
Clay ...	Below 0.002 mm.	Below 0.005 mm.	Below 0.002 mm.

T. J. W.

## ORGANIC ANALYSIS.

**Detection of Oxalic and Citric Acids. Polonovski.** (*J. Pharm. Chim.*, 1921, [vii.], 24, 167-168.)—Oxalic acid may be detected as follows: The mixture having been decomposed in the usual way by means of sodium carbonate, 2 to 3 c.c. of the alkaline filtrate are acidified with acetic acid, and the solution treated with 1 c.c. of saturated calcium fluoride solution; under these conditions oxalates alone are precipitated. If the mixture is free from fluorides, the calcium fluoride added may be replaced by calcium sulphate solution. To detect citric acid, the dissolved or powdered substance is treated at 50° to 60° C. with potassium permanganate solution until it remains pink, and is then heated gently with 1 c.c. of bromine water until it becomes decolorised; in presence of citric acid, perbromoacetone is formed, this being easily recognisable by its pungent action on the mucous membranes of the eyes.

T. H. P.

**Greasiness of Fatty Substances. P. Woog.** (*Comptes rend.*, 1921, 173, 303-306.)—The author considers that the greasiness of fatty substances depends to some extent on the tendency of the molecules to coalesce (*coincement*). As this tendency should become more pronounced as the molecules increase in dimensions, the molecular volumes have been determined microscopically for a number of fatty and mineral oils and for glycerol. It is found that the molecular volumes of the fatty oils are distinctly higher than those of the mineral oils of corresponding viscosity. For mineral oils, the greasiness runs closely parallel with the molecular volume; the dictum stating that, for equal viscosities, the most greasy oil is that of lowest density, is thus explained, since in a homologous series the viscosity increases with the molecular volume. For example, the following results were obtained:

	Sp. gr. at 15° C. ( <i>D</i> ).	Mole- cular Weight ( <i>M</i> ).	$\frac{M}{D \cdot N}$ ( $N=61 \cdot 10^{22}$ ).	<i>K</i> .
c-c.				
<i>Fatty Oils:</i>				
Colza ... ..	0.9149	912	1634.14.10 <sup>-24</sup>	5.77
Arachis ... ..	0.9162	862	1542.36	5.64
Castor ... ..	0.9626	890	1515.75	5.47
Lard ... ..	0.9169	831	1485.76	5.21
Olive ... ..	0.9152	805	1441.94	5.05
Lanoline (anhydrous)	0.9556	793	1360.43	4.4
<i>Petroleum Oils:</i>				
Locomotive ... ..	0.9056	853	1544.12.10 <sup>-24</sup>	7.32
Cylinder ... ..	0.8946	748	1370.70	6.76
Spindle ... ..	0.8918	296	544.11	3.32
French neutral ... ..	0.8529	270	518.96	3.27

The temperature coefficient *K* of superficial molecular energy, determined by the formula of Eotvos, Ramsay, and Shields, is never lower than 2.12 for true lubricants.

T. H. P.

**Oil-film Measurement of Fatty Oils, and Certain Phenomena of Molecular Solutions.** P. Woog. (*Comptes rend.*, 1921, **173**, 387-390.)—Measurement of films of various fatty oils (see preceding abstract) extended on the surface of water in a layer one molecule in thickness shows that, except in the case of castor oil, the molecules of oils appear to exhibit only slight asymmetry. Similar measurements made on the principal constituents of the oils give numbers differing little from those published by Langmuir (*J. Amer. Chem. Soc.*, 1917, **39**, 1848) in support of his theory of surface tension.

T. H. P.

**Aschmann's Method of Determining the Iodine Value.** B. M. Margosehes and R. Baru. (*Chem. Zeit.*, 1921, **45**, 898.)—Aschmann's method of determining the iodine value of oils by means of an aqueous solution of iodine monochloride (*Chem. Zeit.*, 1898, **59**, 71) has been condemned on the ground of the reagent not being miscible with solutions of fat. Comparative experiments here described, however, show that the iodine values thus obtained after an absorption of twenty-four hours, during which the mixture is shaken at least six times, agree closely with the Hübl and Wijs values in the case of non-drying oils, and come between the Hübl and Wijs values in the case of drying oils (hemp and niger seed oils). The reagent has the advantage of stability of "titre."

**Quantitative Separation of the Lead Salts of Saturated and of Less Saturated Fatty Acids.** A. Seidenberg. (*J. Amer. Chem. Soc.*, 1921, **43**, 1323-1336.)—A more accurate and sharp method of separating the lead salts of fatty acids than has hitherto been possible is based on their solution in three solvents—alcohol, chloroform, and ether—two of which are more volatile than alcohol and have also a greater solvent action—and in then volatilising the mixed solution until all the more insoluble salts are precipitated. In this way it is possible to remove completely the whole of the salts of the saturated fatty acids, and concordant results are obtained independently of the quantities used. The method is not applicable to oils, such as linseed oil, which contain linolenic acid, since the saturated fatty acids will contain more or less of that acid; but good results are obtained in the case of oils, such as cottonseed oil, which contain the less unsaturated fatty acids, oleic and linolic acids. The lead salts prepared from 2 to 5 grms. of mixed fatty acids are dehydrated by exposure to the air, and finally by washing with several portions (20 c.c. in all) of anhydrous alcohol, which are then decanted into a cylinder. The residual lead salts are dissolved in 20 c.c. of boiling chloroform, the solution evaporated to about 8 c.c., and, after the addition of about 60 c.c. of ether, boiled for a moment and poured into the cylinder containing the alcohol. The flask is rinsed out with the minimum quantity of hot chloroform and ether, and the total contents of the cylinder made up to 120 c.c. with ether. Air is now aspirated through this solution until the volume has been reduced to 65 or 70 c.c., after which the liquid is filtered through a Gooch crucible. The precipitate on the filter is redissolved in the smallest possible quantity of boiling chloroform, about 50 c.c. of ether added, and the solution boiled for a moment and allowed to cool. The mixture of solution and precipitate is filtered, and the precipitate washed three times with

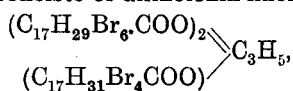
10 c.c. of ether. This process is repeated until a sufficiently pure precipitate is obtained. The filtrates are united and treated with 20 c.c. of 95 per cent. alcohol, and the volatile solvents removed by aspiration until the volume has been reduced to about 30 c.c. If the precipitate separated by filtration is not greatly discoloured when heated at 100° C., it is weighed; otherwise it is redissolved in 3 c.c. of boiling chloroform, about 20 c.c. of boiling ether added, the solution cooled and filtered, and the residue weighed. The amount of this residue, added to that of the main residue, gives the amount of the lead salts of the saturated fatty acids in the sample.

**Precipitation of Solid Fatty Acids with Lead Acetate in Alcoholic Solution.** E. Twitchell. (*J. Ind. Eng. Chem.*, 1921, **13**, 806-807.)—The following process is recommended as being more satisfactory than the lead-salt-ether method for the separation and estimation of solid fatty acids: About 2 grms. of fatty acids from a solid fat, or 10 grms. from an oil, are dissolved in hot alcohol, and a boiling solution of 1.5 grms. of lead acetate in alcohol is added; the total quantity of alcohol used should be 100 c.c. The mixture is allowed to cool slowly, maintained at 15° C., for eighteen hours, and the precipitate then collected on a filter and washed with 95 per cent. alcohol until the filtrate remains clear when diluted with water. The precipitate is next transferred to a beaker, boiled with 100 c.c. of alcohol and 0.5 c.c. of acetic acid, the solution allowed to cool as before, and the precipitate again collected and washed, and then washed into a beaker with ether. Nitric acid is added to decompose the lead salts, the mixture is poured into a separating funnel, and the acid solution drawn off; the ethereal solution is washed thoroughly with water, then evaporated, and the residue of fatty acids dried and weighed. The amounts of solid fatty acids yielded by various oils were as follows: Cottonseed oil, 23.13; soya-bean oil, 17.01; olive oil, 10.93; earthenut oil, 16.58; lard, 40.02; tallow, 53.62; partly hydrogenated cottonseed oil, 50.62 per cent. In each case the iodine value of the solid fatty acids was below 1, except with those of the hydrogenated cottonseed oil (42.21) and lard (4.38).  
W. P. S.

**The "Oil Bromide" Test of Linseed Oil.** T. A. Davidson. (*J. Ind. Eng. Chem.*, 1921, **13**, 801-806.)—Of late years it has been the usual practice to use the fatty acids of linseed oil, and not the oil itself, for the estimation of the hexabromide value. As the drying properties of the oil are due to the glycerides of linolenic acid, and not to linolenic acid, and as there is good reason to assume that the acid exists in linseed oil as a mixed glyceride, the author considers that it is desirable to apply the test directly to the oil, as proposed originally by Hehner and Mitchell (*ANALYST*, 1898, **23**, 310). The term "oil bromide" is used for the insoluble brominated glycerides, and "hexabromides" for the insoluble brominated acids. The following procedure is recommended for the estimation of the oil bromide value; the final washing with alcohol yields a product which remains a powder when dried: Four grms. of the oil are dissolved in 35 c.c. of ether in a centrifuge tube, the solution is cooled at 0° C., and 1 gm. of bromine is added slowly while the mixture is stirred. The top of the tube is rinsed down with 5 c.c. of ether, and the whole mixture maintained at 0° C. for two hours, and then submitted to centrifugal action for three



minutes at 2,000 revolutions per minute. The liquid is decanted from the precipitate, and the latter is washed twice with ether (10 c.c. each time) at 0° C. and once with alcohol, the precipitate being separated by centrifugal action after each washing. After the final washing the precipitate is dried, first in air and then at 100° C. for thirty minutes. All the ether used should be saturated previously at 0° C. with oil bromide (obtained from a former estimation). The yield of oil bromide from genuine linseed oils varies from 50 to 72 per cent.—average 60·9 per cent., a figure higher than those so far recorded. Oil bromide is an amorphous, slightly yellow solid, melting at 140° to 145° C. It contains 57 to 58 per cent. of bromine; is fairly soluble in chloroform, carbon tetrachloride, acetone, and acetic acid; slightly soluble in ether, benzene, and petroleum spirit; and practically insoluble in alcohol. It is probable that the substance consists of dilinolenin-linolin bromide,



mixed with some brominated glyceride of lower bromine content. To estimate the bromine content, a quantity of the oil bromide is mixed with 68 times its weight of alcohol, and 6·5 times its weight of sodium is added; the mixture is heated under a reflux condenser for one hour, and the resulting sodium bromide then titrated by Volhard's method. Ignition with lime yields low results for the bromine, and this method is considered to be untrustworthy for the purpose. W. P. S.

**True Oxygen Absorption of Oils. II. Linolenic and Linolic Acids.**  
**S. Coffey.** (*J. Chem. Soc.*, 1921, 119, 1408-1415.)—The hexabromostearic acid obtained directly from linseed oil yields, when reduced with zinc, a mixture of  $\alpha$ - and  $\beta$ -linolenic acids, from which the  $\alpha$ -acid can be separated as hexabromide by rebromination. The mixture of the two acids was oxidised as previously described (*ANALYST*, 1921, 418); another portion of the mixture was brominated by the method of Hehner and Mitchell (*ANALYST*, 1898, 23, 310), and, after removal of the insoluble hexabromostearic acid, the residual  $\beta$ -acid was debrominated, and the  $\beta$ -linolenic acid oxidised under identical conditions. The results due to the oxidation of the separated  $\alpha$ -linolenic acid were then obtained by difference. In applying the oxidation method to the quantitative estimation of the constituents of linseed oil the amount of  $\alpha$ -linolenic acid is calculated from the quantity of carbon dioxide in the volatile products by multiplying by the ratio  $\text{C}_{18}\text{H}_{30}\text{O}_2/\text{CO}_2$ . The total oxygen absorption is due to oxidation of linolic and linolenic acids, and, since the amount of the latter is known, that of the linolic acid can be calculated. The oleic acid is calculated by difference from the total iodine value, glycerol is estimated directly, and saturated fatty acids and unsaponifiable matter found by difference. In this way a sample of linseed oil was found to have the following composition: Saturated fatty acids and unsaponifiable matter, 8·1; glyceryl radicle, 4·3; oleic acid, 5·0; linolic acid, 48·5; and linolenic acid, 34·1 per cent.

**Estimation of Sodium Chloride in Soap.** **H. C. Bennett.** (*J. Ind. Eng. Chem.*, 1921, 13, 813.)—A weighed quantity of about 5 grms. of the soap is dissolved

in 150 c.c. of hot water, and the soap and soluble fillers are precipitated by the addition of 25 c.c. of 20 per cent. magnesium nitrate solution; the hot, unfiltered solution is then titrated with standard silver nitrate solution, potassium chromate being used as indicator.

W. P. S.

**Consistence of Starch and Dextrin Pastes.** W. H. Herschel and C. Bergquist. (*J. Ind. Eng. Chem.*, 1921, **13**, 703-706.)—The viscosity apparatus described previously by Bingham (*ANALYST*, 1914, **39**, 284) is modified in its application as a plastometer; by plasticity is meant a measure of the consistence at a given time. Two side strips are fitted to the cover of the apparatus, and aid in unscrewing the cover; the plug is made in two pieces, so that the centre of a long capillary is accessible and readily cemented into position, and a ring attached to the upper part of the plug holds the latter in position. Capillaries of 1 to 1.5 mm. diameter are used, and have a length of 25 to 40 mm. Adhesives in the concentration at which they are used are mostly plastic, but differences in shear value and rigidity of different samples of the same concentration are striking and significant. Although further work on the subject is necessary, the results so far obtained indicate that the values obtained by the plastometer will show body, spreading power, and gelatinising power of starch and dextrin pastes.

W. P. S.

**Effects of Chemical Reagents on the Microstructure of Wood.** A. Abrams. (*J. Ind. Eng. Chem.*, 1921, **13**, 786-790.)—Cross, radial, and tangential sections are cut from the wood, and these sections are placed in the depressions of a microscope cell slide; the cells of the slide are filled with the reagent to be studied, and a plain slide is placed over the cell slide, but separated from it about 1 mm. by pieces of thin cover-glass. The two are wired together (in dealing with sodium bisulphite or other substance which attacks iron wire, the slides are sealed into glass tubes with the reagent) and placed in an autoclave containing a considerable quantity of the reagent. The autoclave is heated for various lengths of time at the required pressures, and at stated intervals is cooled quickly, and a slide removed for microscopic examination. Photomicrographs are given, showing the action of various substances on wood. In general, cellulose solvents, such as copper-ammonium reagent and 75 per cent. sulphuric acid, act strongly and about equally on both the middle lamella and the cell wall. Oxidising substances (chromic acid, bleach solutions, and potassium chlorate in nitric acid) act on the cell wall, but have comparatively little effect on the middle lamella. The ordinary paper-making reagents, such as dilute sodium hydroxide solution and sodium bisulphite solution, act strongly on the middle lamella, and have much less effect on the cell wall.

W. P. S.

**Smokeless Fuels.** F. S. Sinnatt. (*J. Roy. Sanit. Inst.*, 1921, **42**, 22-26.)—In modern gasworks there is a tendency to produce increasingly large volumes of gas at the expense of the coke, with the result that the percentage of inorganic constituents in the residue becomes excessively high. For example, cases may be cited of coke yielding 20 to 30 per cent. of ash and containing a considerable proportion of water. Smokeless fuel should have some of the following characteristics: It should

be smokeless and ignite with ease; should not be too fragile or too bulky; should not absorb water, and, if it becomes wet, should dry in a reasonable time. It should have a low ash content, and the ash should not be powdery; it must contain only a small percentage of sulphur; must be sufficiently porous for the combustion to proceed internally as well as externally, in order that the ash produced does not cover the surface and prevent combustion, but must not be too porous, or it will be fragile. No convenient method of determining the relative values of different smokeless fuels has yet been developed, but it is advisable to ascertain the proportion of volatile matter and the texture or micro-texture of the coke. The following results were obtained in the analyses of typical varieties of such fuels:

Percentage.	Lignite, Low Tempera- ture.	Carbo- coal.	Briquette Sun Fuel Company.	Hargreaves Coke.	Special Briquette.	Low Tem- perature Carbon Coke.
Water ... ..	6.44	2.03	3.10	0.60	2.60	2.42
Ash ... ..	16.85	15.66	10.48	10.70	9.00	4.70
Volatile matter ...	22.43	5.94	10.08	7.80	13.00	15.45
Volatile organic matter	15.99	3.91	6.98	7.20	10.40	13.03
Coke ... ..	77.57	94.06	89.92	92.20	87.00	84.55
Fixed carbon ... ..	60.72	78.40	79.44	81.50	78.00	79.85
Calorific value (British thermal unit per lb.)	10,280	11,570	12,740	13,180	12,400	13,550

**Detection and Estimation of Coal Tar Oils in Turpentine.** V. E. Grotlisch and W. C. Smith. (*J. Ind. Eng. Chem.*, 1921, 13, 791-793).—A method for the detection of small quantities (1 to 2 per cent.) of coal tar oils, such as benzene, toluene and commercial solvent naphtha, in turpentine, consists essentially in concentrating these oils by converting the turpentine into pinene hydrochloride, and separating the latter. The oils are then sulphonated, distilled, and identified as described below. One hundred c.c. of the turpentine are cooled in a mixture of ice and salt, and then saturated with dry hydrogen chloride; when absorption of the latter is complete, the mixture is kept for one hour in a closed vessel in the freezing mixture, to obtain maximum crystallisation of the pinene hydrochloride. This compound is very soluble in both mineral and coal tar oils, and if more than 10 per cent. of the oils is present, scarcely any crystals are obtained. The crystals are separated by filtration, and the filtrate is distilled under reduced pressure (10 inches of mercury) until crystals separate out in the condenser, or until 25 c.c. of distillate are obtained. The distillate is added drop by drop to four times its volume of fuming sulphuric acid (containing 4 per cent. of free sulphur trioxide), and the mixture is then heated at about 70° C. for twenty minutes, with frequent agitation. The sulphonated mixture is next diluted with its own volume of water and steam distilled. In the case of pure turpentine, a very small quantity (not more than 0.5 c.c.) of yellow oil, having a refractive index at 20° C. above 1.500, is obtained. If mineral

oil is present in the sample, it is recovered at this point, and may be identified by its characteristic odour and refractive index, which is usually below 1.400. When oil ceases to distil the steam supply is disconnected, and the mixture distilled by direct heat. As soon as oily drops appear in the distillate, hot water is added slowly to the flask from a dropping funnel, and at a slightly lower rate than the liquid is distilling, so that the temperature rises gradually. The distillation is run at about 5 or 8 c.c. per minute. In the case of adulteration with solvent naphtha, hydrolysis of the sulphonated compound begins at about 115° C.; when the temperature reaches about 170° C. excessive frothing occurs, and the distillation must be stopped. The volume of oil recovered in the distillate is multiplied by 2.2 to obtain the percentage of coal tar oil in the sample. The oil may be identified as a benzene hydrocarbon by the usual tests (nitration, etc.). The method does not give reliable results when applied to wood turpentine, whether obtained by distillation with steam or by destructive distillation, probably because the high temperatures used in the methods of manufacture break down the terpenes and resins partially, with the formation of ring hydrocarbons; if, however, more than 4 per cent. of oil is recovered from a wood turpentine, the purity of the sample is doubtful.

W. P. S.

**The Gold Number of Commercial Gelatins.** F. A. Elliott and S. E. Sheppard. (*J. Ind. Eng. Chem.*, 1921, **13**, 699-700.)—The gold number of colloids indicates the number of mgrms. of colloid necessary just to prevent the precipitation of 10 c.c. of standard gold solution by 1 c.c. of 10 per cent. sodium chloride solution. The gold hydrosol is prepared by heating a mixture of 120 c.c. of conductivity water, 2.5 c.c. of 0.6 per cent. gold chloride solution, and 3.5 c.c. of 0.18 *N*-potassium carbonate solution, nearly to boiling, and adding 2 c.c. of 0.3 per cent. formaldehyde solution. Commercial gelatins differ but little as regards their gold value, and the latter does not bear any simple relation to those properties which are of chief interest to users of gelatins.

W. P. S.

### INORGANIC ANALYSIS.

**Phenol Red as Indicator of Acidity.** A. Massink. (*Pharm. Weekblad*, 1921, **58**, 1133-1136.)—For the colorimetric estimation of hydrogen-ion concentration in the region of  $P_H$  7 to 8, neutral red is a more suitable indicator than phenol red, the latter being considerably influenced by the presence of salts. For example, in the case of a water with an electrical conductivity of  $K_{18} \cdot 10^6 = 33,350$ , the  $P_H$  value with neutral red was 7.10, whilst with phenol red it was 7.35. But the same water diluted 1:10, so that its conductivity became  $K_{18} \cdot 10^6 = 4,250$ , still gave the same  $P_H$  value (7.10) with neutral red, but only 7.05 with phenol red.

**Detection of Carbon Monoxide.** C. R. Hoover. (*J. Ind. Eng. Chem.*, 1921, **13**, 770-772.)—A mixture of iodine pentoxide 11.0, granular pumice-stone 34.0, and fuming sulphuric acid (60 to 80 per cent. SO) 55.0 per cent. yields a green to black coloration when brought into contact with carbon monoxide. Under favourable conditions as little as 0.005 per cent. of carbon monoxide in air may be detected by

passing 500 c.c. of the air through a glass tube containing a 50 mm. by 5 mm. column of the mixture. The green colour indicating the presence of carbon monoxide disappears in a few minutes, and the tube may be used again for several successive tests. Colour standards may be prepared with pumice-stone and basic copper acetate or chromium oxide, and the test thus rendered approximately quantitative. Other unsaturated gases, such as hydrocarbons, hydrogen sulphide, hydrocyanic acid, etc., interfere, but may be removed by passing the air previously through dry active charcoal; hydrogen, methane, sulphur dioxide, nitric acid, and ammonia, are without action on the mixture.

W. P. S.

**Sensitive Reaction for Copper. P. Falcicola.** (*Giorn. Chim. Applic.*, 1921, **3**, 354-355.)—Copper may be detected in very dilute solution by means of a solution of alkali thiocyanate mixed with a cold saturated solution of gallic acid. With this reagent a  $\frac{N}{100000}$  copper sulphate solution gives a decided turbidity, whilst a  $\frac{N}{1000000}$  solution, if shaken with the reagent, soon develops perceptible whitish cloudiness. The cupric solution must be neutral, since the presence of mineral acid hinders the appearance of the precipitate, although the latter, after its formation, is virtually insoluble in acids; the ions  $Ag^+$ ,  $Pb^{++}$ , and  $Bi^{+++}$ , should also be absent, as they yield precipitates under the above conditions.

T. H. P.

**Rapid Estimation of Copper and Iron in Aluminium Alloys.\* P. Hulot.** (*Bull. Soc. Chim.*, 1921, **29**, 836-837.)—The alloy (2 to 5 grms.) in the form of wire, sheet, or turnings is dissolved in 20 per cent. sodium or potassium hydroxide solution, the liquid being warmed gently towards the end of the reaction and diluted with water; the iron and copper remain insoluble, and may be washed, collected, and weighed. If greater accuracy is required, the copper may be dissolved and deposited electrolytically, and iron may be dissolved and reprecipitated as hydroxide. The method avoids the filtration of gelatinous alumina, and is therefore useful for rapid work in the foundry.

R. G. P.

**Estimation of Aluminium and of Aluminium Oxide in Commercial Aluminium. J. H. Capps.** (*J. Ind. Eng. Chem.*, 1921, **13**, 808-812.)—A weighed amount of the metal is boiled with water in a flask to expel dissolved and absorbed gases; the flask is then filled with water (previously boiled) and closed with a stopper carrying a tapped funnel, a short side tube, and a capillary delivery tube connected with a gas measuring burette. A quantity of reagent (sulphuric acid or sodium hydroxide solution) is admitted from the funnel, the side tube being opened during this operation and then closed. As hydrogen is evolved, a portion of the liquid in the flask is forced back into the funnel, a tap on the capillary being closed, thus forming a gas space in the flask. Connection with the burette is then made, the contents of the flask are heated, and, when evolution of gas ceases, the flask is filled with liquid from the funnel, and all gas driven over into the burette. The latter is provided with a water jacket and is filled with water saturated with hydrogen. If

\* A similar method was described by Gemmell in the discussion on a paper by Withey (*ANALYST*, 1916, **41**, 181).—EDITOR.

sulphuric acid is used to decompose the metal, allowance must be made for the quantity of hydrogen formed by the iron present in the aluminium; when sodium hydroxide is used, a similar allowance must be made for the hydrogen resulting from the action of the alkali on the silicon present. The volume of the hydrogen obtained is a measure of the metallic aluminium present in the sample; the aluminium oxide is obtained by difference.

W. P. S.

**Estimation of Vanadium in Ores and Metallurgical Products. R. B. Schaal.** (*J. Ind. Chem. Soc.*, 1921, **13**, 698-699.)—The method depends on the reduction of vanadium pentoxide to the dioxide by prolonged boiling with hydrochloric acid, and subsequent titration of the dioxide with permanganate solution— $V_2O_5 + 2HCl = V_2O_4 + Cl_2 + H_2O$ , and  $5V_2O_4 + 2KMnO_4 + 6HCl = 5V_2O_5 + 2KCl + 2MnCl_2 + 3H_2O$ . In a nearly neutral solution containing an excess of ammonium phosphate the end-point of the titration is sharp, and relatively large amounts of iron, chromium, molybdenum, cobalt, nickel, titanium, and zirconium do not interfere; small amounts of manganese are also without effect. In the case of steels, 2 grms. of the sample are dissolved in 50 c.c. of hydrochloric acid (1 : 1), 5 c.c. of nitric acid are added, and the solution is evaporated to 10 c.c. For ores, from 1 to 5 grms. of the sample are digested with 60 c.c. of concentrated hydrochloric acid, 5 c.c. of nitric acid and 2 c.c. of hydrofluoric acid are added, and the mixture is evaporated to dryness. With ferrovandium, 0.5 gm. is treated with 10 c.c. of nitric acid, 2 c.c. of hydrofluoric acid are added after a time, the mixture is heated, and, when violent action has ceased, 40 c.c. of concentrated hydrochloric acid are added, and the mixture is evaporated to dryness. To the solution or residue, as the case may be, are added 40 c.c. of hydrochloric acid, the mixture evaporated to dryness, this addition of acid is repeated, and the mixture evaporated to 10 c.c. The residue is mixed with 50 c.c. of water, filtered, and the insoluble portion washed five times with cold water, 15 c.c. being used each time. The filtrate is treated with 60 c.c. of 15 per cent. ammonium phosphate solution, ammonia is added until a precipitate forms, and this is dissolved by the addition of a slight excess of hydrochloric acid. The solution is then diluted to 300 c.c., and titrated with standardised potassium permanganate solution.

W. P. S.

**Electrometric Estimation of Bromate, Dichromate, Nitrite, and Chloride Ions. W. S. Hendrixson.** (*J. Amer. Chem. Soc.*, 1921, **43**, 1309-1317.)—Methods are described for the direct electrometric titration of iodide with bromate and dichromate, and the indirect titration of bromate with permanganate. The direct titration of iodide can be made in 2 *N*-sulphuric acid solution, but much time is required to reach the true end-points, and the indirect titration, finishing with permanganate, is preferred. For the electrometric estimation of nitrite, a weighed quantity of standard permanganate solution is placed in the titration vessel, diluted, and 10 *N*-sulphuric acid is added in amount sufficient to make the final solution about 1.5 *N*. The mechanical stirrer is started, and the nitrite solution added slowly in such quantity that 5 to 10 per cent. of the permanganate remains unchanged. After five minutes an excess of standard iodide solution is added, and the titration completed with permanganate solution.

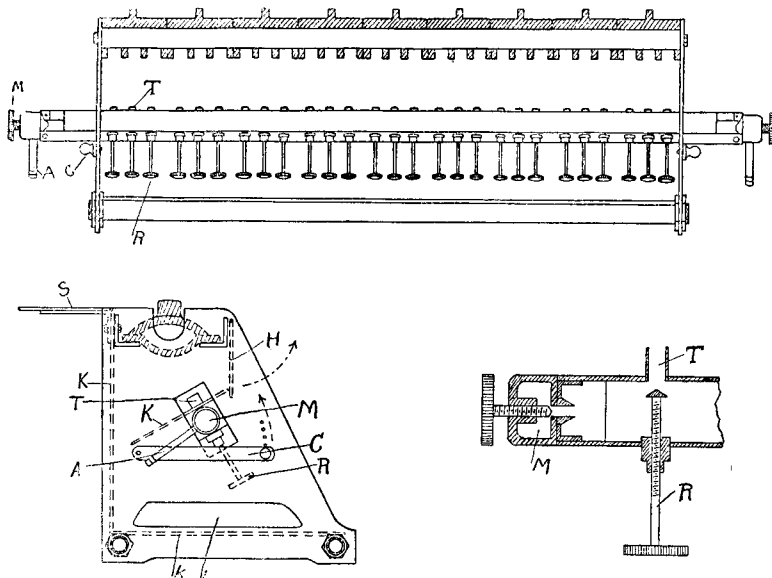
W. P. S.

**Estimation of Sodium Hyposulphite.** J. H. Smith. (*J. Amer. Chem. Soc.*, 1921, 43, 1307-1308.)—About 0.4 grm. of the solid sodium hyposulphite (usually, but wrongly, termed sodium hydrosulphite) is placed in a beaker, and treated with a large (100 per cent.) excess of ammoniacal silver nitrate solution; the mixture must not be warmed. As the salt dissolves it is oxidised, and metallic silver is precipitated. The precipitate is collected on a Gooch crucible, washed with ammoniacal ammonium nitrate solution, the crucible then placed in the same beaker, and the silver dissolved in warm nitric acid. The solution is boiled to expel nitrous acid, the crucible is removed, and the solution is diluted and titrated with  $\frac{N}{10}$  potassium thiocyanate solution, ferric alum being used as indicator. The oxidation proceeds according to the equation:  $\text{Na}_2\text{S}_2\text{O}_4 + 2\text{AgNO}_3 + 4\text{NH}_4\text{OH} = 2(\text{NH}_4)_2\text{SO}_4 + 2\text{NaNO}_3 + 2\text{H}_2\text{O} + \text{Ag}_2$ ; one molecule of sodium hyposulphite is, therefore, equivalent to two molecules of potassium thiocyanate.

W. P. S.

### PHYSICAL METHODS, APPARATUS, ETC.

**Improved Gas Combustion Furnace for Use in Organic Analysis.** T. J. Hedley. (*J. Chem. Soc.*, 1921, 119, 1242-1243.)—A light steel framework similar to that employed in the usual form of furnace, but covered at the back and base with uralite, and fitted with a horizontal door of the same material in front, carries a row of trough tiles and covers at the top. The multiple burner consists of a large metal tube, closed in the centre, and fitted at each end with a gas

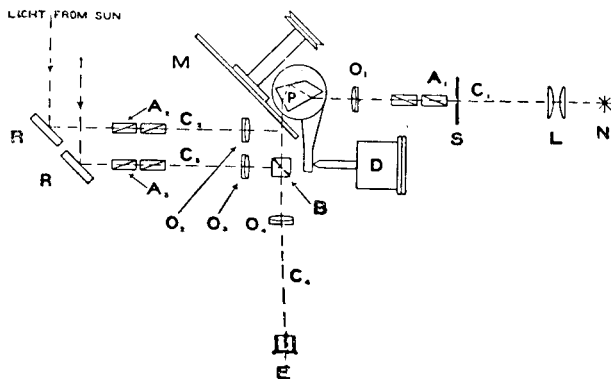


inlet and a screw valve. The separate burners fitted on one side of this tube are controlled by screw cone valves projecting from the opposite side of the tube. The whole burner is supported by spring arms fixed upon the end plates of the furnace, and the burners can be adjusted at different distances from the tiles. The construc-

tion is shown in the diagram: *A*, gas inlet; *C*, spring arms; *H*, hinged uralite screen; *K*, fixed uralite screens; *M*, main gas regulator; *R*, cone valves; *S*, hot tile shelf; *T*, burner tubes. The advantages claimed for this type of furnace are: Portability, the weight being less than 30 pounds; small gas consumption; protection from excessive radiation; rapid control of temperature by adjustment of the gas; and economy in cost of construction. T. J. W.

**Rapid Method of Determining the Density of Air.** G. P. Baxter. (*J. Amer. Chem. Soc.*, 1921, **43**, 1317-1318.)—A sealed globe of known exterior volume is weighed under accurately known conditions of temperature, pressure, and humidity; from these conditions the density of the atmosphere at the time of weighing and the buoyant effect of the air on the globe and weights are computed. By adding the difference between the buoyant effect on the globe and on the weights to the weight of the globe in air, the weight of the globe *in vacuo* is found. If the globe be weighed in the air under any conditions, the weight of the air displaced by the globe in excess of that displaced by the weights is found by subtraction from the weight *in vacuo*; this difference, divided by the volume of the globe, less the volume of the weights, gives the atmospheric density. W. P. S.

**Measurement of Colour.** C. E. K. Mees. (*J. Ind. Eng. Chem.*, 1921, **13**, 729-731.)—The most convenient and satisfactory colorimeter for reading the colour of an object in absolute units—namely, the wave length of the dominant hue and the percentage—is the monochromatic analyser. In this instrument the colour to be measured is balanced by means of a mixture of spectrum colour and white light. For instance, to measure the colour of a green tinted paper, the paper is placed so that the light reflected from it falls into the colorimeter (see illustration). In the photometric field at *B* is seen one part of the field filled by the green light from the coloured paper, whilst the other part of the field can be illuminated by means of a spectrum, to which can be added any proportion of white light that is required. The



wave length of the spectrum light is changed by means of the drum until the tint appears to be approximately right; white light is then added from the beam  $C_2$ , reflected from the glass surface *M*, until the two tints are about the same, the intensity



of the two fields being kept equal to each other. The tint may now require a little correction, and when this is almost exactly right the admixed white light may require another correction. By this method the colour may be matched rapidly, and the dominant tint is then read off on the spectrum drum, and the percentage of admixed white light is measured photometrically in the instrument. A red may be specified as being of dominant tint wave length 650, corresponding in tint with the red hydrogen line; a yellow might be of dominant tint 580; a greenish-yellow would be 570, and a yellowish-green 560.

W. P. S.

**Repair of Crystallised Glass Apparatus.** H. Bailey. (*J. Amer. Chem. Soc.*, 1921, 43, 1319.)—The method consists in holding the nearly-melted glass in a sodium flame for a few seconds. A piece of Pyrex tubing wrapped with a few layers of asbestos paper saturated with sodium chloride solution is held in the blow-pipe flame in such a position that the sodium vapours envelope the devitrified glass; all signs of crystallisation usually disappear by the time the glass is soft enough to work. (*Cf. ANALYST*, 1921, 164.)

W. P. S.



### SAFEGUARDING OF INDUSTRIES ACT, 1921.

#### THE LIST OF ARTICLES CHARGEABLE WITH DUTY UNDER PART I. OF THE ACT.

IN pursuance of Sec. 1 (5) of the Act, the Board of Trade have prepared a list defining the articles which are to be taken as falling under the following general description set out in the Schedule—viz.: Optical glass and optical elements, whether finished or not (List A); optical instruments (List B); scientific glass ware (List C); laboratory porcelain (List D); scientific instruments (List E); gauges and measuring instruments of precision of the type used in engineering machine shops (List F); compounds (not including ores or minerals) of thorium, cerium, and other rare earth metals (List G); synthetic organic chemicals (other than synthetic organic dyestuffs, colours, and colouring matters imported for use as such, and organic intermediate products imported for their manufacture), analytical reagents, all other fine chemicals (except sulphate of quinine of vegetable origin), and chemicals manufactured by fermentation processes (List H).

Articles falling under any of the general descriptions in paragraph 3, or included in the Lists A to H, will be chargeable with duty under Part I. of the Act on and after October 1, 1921.

According to Sec. 1 (4) of the Act, no duty shall be charged under this section in respect of a compound article containing an article subject to duty if the compound is of such a nature that the article liable to duty has lost its identity, and any dispute on this point shall be determined in like manner as disputes as to whether goods are goods specified in the Schedule to the Act.

Sec. 1 (5) provides that if within three months after the publication of any list of articles according to Sec. 1 of the Act any person appearing to the Board to be interested delivers to the Board a written notice complaining that any article has been improperly included in or excluded from the list, the Board shall refer the complaint to the arbitration of a referee to be appointed by the Lord Chancellor,

who shall not be an official of any Government department, and the decision of the referee shall be final and conclusive, and the list shall be amended so far as is necessary in order to give effect to the decision, without prejudice, however, to the validity of anything previously done thereunder.

LIST A—OPTICAL GLASS AND OPTICAL ELEMENTS.—Lenses, photographic (unmounted); lenses, spectacle (mounted or unmounted).

LIST B—OPTICAL INSTRUMENTS.—Binoculars, prismatic; field glasses; microscopes, with or without lenses; opera glasses; ophthalmometers; photographic cameras, with or without lenses; sight-testing apparatus; oculists' trial sets and cases.

LIST C—SCIENTIFIC GLASS-WARE.—Ampoules; barometers, mercury, in glass; beakers; bottles, culture, weighing, and Woolff's; funnels, separating, thistle, and filtering; flasks, various; jars, various; Kipp's apparatus; lecture and experimental apparatus; mortars and pestles; percolators; pipettes; pyknometers; retorts (laboratory); syringes, glass; specific gravity apparatus; thermometers; tubes.

LIST D—LABORATORY PORCELAIN.—Baths, air and water, and cover plates; burners, Bunsen; centrifuges; crucibles; dishes, evaporating; filter funnels; funnels, plain and filtration, including Buchner's funnels; retorts; rings; spatulas; spoons; tubes, combustion; trays; water baths.

LIST E—SCIENTIFIC INSTRUMENTS.—Autoclaves, laboratory; balances, analytical, assay, chemical, and other precision; hydrometers; hygrometers; influence machines (electric) and other static machines; microtomes; physical lecture and experimental apparatus.

LIST G—COMPOUNDS OF CERIUM, THORIUM, AND OTHER RARE EARTH METALS.—The list includes 72 compounds of thorium, 68 of cerium, and 16 of other rare earth metals.

LIST H—ANALYTICAL REAGENTS, FINE CHEMICALS, ETC.—The list includes over 4,000 articles. Those to which the letter D is attached are chemicals covered by the Dyestuffs (Import Regulation) Act, 1920, and come within the scope of the present Act only when importation has been permitted for purposes other than those with which the Dyestuffs Act is concerned.

The letter R signifies that the chemical is included only when it is "pure," "puriss.," "extra pure," "B.P.," "Ph.G.," "A.R.," "for analysis," "reagent," or when it is of special quality for meeting special tests of purity, and not when it is the crude product.

The analytical reagents specifically named include, *inter alia*, the following: Acetanilide (D); acetic anhydride, acetone (R); Acid, acetic (80 per cent. grade or higher); arsenious (R), benzoic (D), boric, carbolic (synthetic) (D); carbolic (R); chromic, citric, formic, gallic, hydriodic, hydrobromic, hydrochloric (R), hydrocyanic, hypophosphorous, iodic, lactic, phosphoric, phosphorous, picric (D), pyrogallic (D), salicylic (D), sulphuric (R); tannic (R); tartaric. Alcohol, absolute; amyl (R), methyl (R). Alum, ammonia (R); chrome; potash (R); ammonium nitrate (R) and 115 compounds of ammonium; aniline (D); antimony chloride (R); barium carbonate (R) and chloride (R), and 75 other barium compounds; bismuth, 64 compounds; bromine; cadmium salts; calcium carbonate (R), phosphate (R), sulphate (R), and 95 other compounds; carbon disulphide (R); carbon tetrachloride; cellulose acetate; chloroform; copper sulphate (R), and 68 copper compounds; cresols; exalgin (D); formaldehyde; guaiacol compounds; hæmatoxylin, heliotropine, iodine (R); iodoform; iron (R), iron perchloride (R), iron sulphate (R), and 80 other iron compounds; lactose (R); lead carbonate (R), oxide (R), subacetate (R), and 54 other lead compounds; lithium, bromide, and 47 other lithium compounds; magnesium sulphate, and 77 other compounds; maltose (R); mercury chloride, bichloride, and 54 other mercury compounds; nicotine (R); perhydrol, phenazone; phenol (D); phenolphthalein; potassium bichromate (R), carbonate (R), hydroxide (R), ferricyanide; ferrocyanide (R), and 145 other salts; quinine, 76 salts; resorcinol; Rochelle salt; saccharin;

salicin ; silver nitrate, oxide, and 48 other salts; sodium bicarbonate (R), hydroxide (R), hyposulphite (R), sulphate (R), sulphite (R), thiosulphate (R), and 225 other salts; strontium bromide, and 34 other strontium salts; strychnine, 23 compounds; thallium acetate; thymol, tin oxide, and other salts; uranium nitrate, veronal, xylene, zinc carbonate (R), chloride (R), oxide (R), sulphate (R), and 71 other zinc salts.

Among the alkaloids in the list are: Epinephrine, ergamine, ergotoxine, novocaine, pellieterine, scopolamine, solanine, and yohimbine, and their salts.

Copies of the list (price 9d.) may be obtained from H.M. Stationery Office at the following addresses: Imperial House, Kingsway, W.C. 2; 28, Abingdon Street, S.W. 1; 37, Peter Street, Manchester; 1, St. Andrew's Crescent, Cardiff; 23, Forth Street, Edinburgh; or from E. Ponsonby, 116, Grafton Street, Dublin.



### NEW CHEMICAL STANDARDS.

(ANALYTICALLY STANDARDISED SAMPLES.)

ANOTHER new plain carbon steel is now ready for issue—viz., "R," which fills the vacancy in the series for a carbon standard between "I" (carbon 0.521 per cent.) and "SI" (carbon 0.921 per cent.). It is also intended to serve as a high manganese standard and a sulphur standard in high carbon steel.

The analyses have been undertaken as usual by a number of experienced chemists representing the following interests: British Government Department; U.S. Bureau of Standards; Referee Analysts, independent; Railway Analysts, representing users issuing specifications; Works Analysts, representing makers and users.

The standard figures are as follows—CARBON STEEL "R": Carbon (combustion), 0.786; carbon (colorimetric), 0.790; silicon, 0.22\*; sulphur, total by oxidation, 0.053; sulphur, evolved as sulphide, 0.50; phosphorus, 0.058\*; manganese, 0.914; arsenic, 0.03\*; copper, 0.02\*; Ni, Cr, W, Mo, none detected by qualitative tests.

The standards may be obtained in 500, 100, or 50 gm. bottles, either direct from Organising Headquarters, 3, Wilson Street, Middlesbrough, or through any of the leading laboratory furnishers, at a price just sufficient to cover the cost. A certificate, giving the names of the analysts co-operating, the types of methods used, and a detailed list of the results, will be supplied with each bottle.

### REVIEWS.

AIDS TO CHEMISTRY. By WILLIAM PARTRIDGE, F.I.C. Pp. viii + 280. London: Baillière, Tindall and Cox. 1921. Price 6s. net.

That our present system of examinations is far from ideal is almost a commonplace, though no alternative method of deciding a person's qualifications has, so far, met with general approval. There are many, both teachers and pupils, to whom the passing of examinations is wellnigh the sole aim of study. From this state of affairs it arises that a class of books has come into existence whose only object seems to be just to push the student through his next examination in the shortest possible space of time. Such books are, as a rule, soulless collections of information, which, though doubtless answering this purpose, can hardly be expected to inspire the

\* Approximate.

learner with enthusiasm or to provide him with a real scientific education. Unable to stand the strain of the unromantic "grind" necessary to acquire the requisite knowledge of detail, his energies begin to flag, and, with a sigh of relief, he drops the unwelcome subject as soon as the examination has been "successfully passed."

The small volume under consideration is somewhat of this type, attempting to cover almost the whole range of chemistry in less than 300 pages. Roughly speaking, 50 pages are allotted to general theory, 50 to the non-metals, and another 50 to the metals; after a short general account of the special features of organic chemistry, aliphatic compounds receive about 70, and aromatic compounds 15 pages; there are also shorter sections dealing with cyanogen and heterocyclic compounds. The book, on the whole, is clearly written, and a large amount of information is condensed into a small compass. The author's choice of material is decidedly open to question, and the volume would be improved by devoting increased space to the more important portions of the subject, omitting reference to various compounds of little or no value to the medical and pharmaceutical students for whom the book is intended.

As a kind of summary for revision purposes, this manual will no doubt be of value, but as a textbook for general study it is not to be recommended.

A. F. KITCHING.

ANALYSES AND ENERGY VALUES OF FOODS. By R. H. A. PLIMMER, D.Sc. Pp 255.  
London: H.M. Stationery Office, 1921. Price 6s. net.

The well-known work of König was for many years practically the only available storehouse of analytical data as to the composition of foods, and much of its information referred to products of Continental origin. The series of analyses made by Atwater and Bryant under the auspices of the U.S. Department of Agriculture was remarkable for its completeness, and when published in 1906 in tabulated form soon gained an unchallenged position as a standard work of reference on the composition and energy values of American foods. Hitherto, however, there has been no similar compilation of systematic analyses of British food products, and this little handbook, therefore, fills a distinct gap.

The investigation was primarily undertaken to obtain trustworthy figures which could be used as a guide by the army medical authorities in supplying the army with the right food under the conditions of rationing prevailing during the war, but it is so full of valuable detail, much of which is new, that it should serve as the foundation upon which definite standards for food values might be based.

The first impression on taking up the book is of the immense amount of work which it embodies, and Dr. Plimmer may be congratulated upon successfully carrying through, almost single-handed, this series of some 900 analyses, involving, as he tells us, over 20,000 weighings.

The work was not intended to supersede the results of Atwater and Bryant, but rather to supplement them and to provide average data for each group of foods.

An outline of the methods of analysis used is given in a short introduction, and the method of calculating the energy values is explained. From the point of view of the chemist, for whom, however, the book is not primarily intended, a little more discussion on the question of the energy value would have been an advantage. It is

not pointed out, for instance, that the calorific values calculated from the composition agree fairly closely with the values actually determined by combustion in the case of certain foods such as meat, milk, white bread, potatoes, and fruits, whereas in other cases, notably peas and many other vegetables, the theoretical values fall far below the actual values. It is only by a compensation of the inherent errors that the calculated and actual values usually agree fairly well when a mixed diet is under examination.

The question of the influence of the digestibility factor is mentioned, but not discussed at any length, although obviously it must have great weight, especially in the case of vegetable products.

Still, whatever method of calculating energy values be ultimately adopted, all the factors used in calculating the proximate composition of the different foods are given, so that it will be possible to recalculate the data upon any other basis.

The tables include not only analyses of all the more common kinds of food, but also, wherever necessary, show the component parts. For example, the average percentages of food products, waste, and bone, in an ox are given, and in the case of the separate joints the fat and skin and lean meat, and analyses of these separate portions are given.

In connection with the analyses of war bread, it is interesting to note that the main difference from pre-war bread, analyses of which by Cribb and Elliot are quoted in full, was in the appearance, the composition being very similar. Parenthetically it might be suggested that this is one of the cases where the digestibility factor might have been taken into consideration.

A useful summary of the food containing the accessory factors, the vitamins A, B, and C, is given in an appendix, so that it can be seen at once whether a particular food is rich or poor in any of these active principles.

In short, as a work of reference this little book is one which every chemist engaged in the examination of foods should have and keep in constant use.

EDITOR.

ANIMAL PROTEINS. By H. G. BENNETT, M.Sc. Pp. xii + 287. London: Baillière, Tindall and Cox. 1921. Price 15s. net.

The title of this book is misleading. It contains a well-written account of the methods employed in the tanning industry, together with a description of the manufacture of gelatin and glue. In this way the industrial chemistry of collagen and gelatin occupy 268 pages, while all other animal proteins are relegated to 14 pages at the end of the book, in which the matter is so condensed as to be practically without value. We are told that the object of the book is to give a general survey of the industries involved, and as far as the leather, gelatin, and glue trades are concerned, it succeeds very well in its purpose. To the young chemist wishing to enter one of these industries the book will be of great value, though he must remember that technical procedure varies so greatly from firm to firm that no account of the details of manufacture can be of universal application. The statement on p. 164, for instance, that "after puering (chrome goat and sheep) it is common to give a low temperature drench" is one which is open to challenge. This, however, is a minor point, detracting little from the value of the whole.

It is with the attempted theoretical explanations of the various stages in leather manufacture that there is more room for criticism. For instance, in the section on "the preparation of pelt" the question as to what actual processes are going on in the lime-pits is taken up at length. One practical result of liming is that the skins can be unhaired at the end of the process. The theoretical question is, How is this brought about? We find the following statements: p. 20, "The function of lime is complex. . . . Its main purpose, however, is that of a partial antiseptic"; on the same page, "all tannery lime liquors are swarming with putrefactive bacteria"; p. 21, "the excess of undissolved lime has an accelerating effect on bacterial activity"; and "the depilating organisms of liquors are probably mostly anaerobes"; "lime plays an essential part in assisting the putrefactive fermentation." Mr. Bennett's theory is evidently that unhairing is brought about by bacterial activity in the lime-pits. Now, although it is true that a number of different species of organisms have been isolated from tanyard lime liquors, it remains to be proved that these bacteria are in a state of functional activity while in the limes. Lime liquors have a hydrogen-ion concentration of  $P_H = 12.4$ , and no evidence has yet been brought forward to show that the number of bacteria increases in the pit during the process of liming. It is now known that many so-called antiseptics—*e.g.*, toluene, ether—act not by killing bacteria, but by inhibiting their activity. The mere isolation of bacteria from a fluid is not proof that the bacteria were playing any active rôle in any chemical processes going on in it. Bacterial activity in the lime-pits has always been assumed, but never proved. In view of the reaction the omission is serious.

Mr. Bennett's theory of the influence of electrolytes on gelatin gels would, we fear, increase rather than decrease the difficulties of any leather chemist anxious to learn the background of his subject. "Lyotrope influence" and "adsorption" are freely invoked as explanations, and in one place (p. 217) we are told "it is sometimes a difficult problem to decide whether an increase or decrease in swelling is due to lyotrope or adsorptive influence." With this statement we can cordially agree. Mr. Bennett's arguments are ingenious, and his theories have doubtless been a guide to him in his own work; but we feel that for the general scientific public, as also for leather chemists, Professor Procter's work and theories will continue to hold the pre-eminent position. It is becoming clearer every day that "adsorption" is a term which describes, but does not explain, concentrations at interfaces, and "lyotrope influence" is a mystery which has happily been dispelled by the recent work of Loeb. Mr. Bennett considers that swelling is due to the repulsion of similarly charged colloidal ions, and refers constantly to gelatin as "primarily a positive colloid" (pp. 45, 213, etc.). What he means by "primarily" is difficult to make out. Gelatin is a positive colloid in acid solutions, a negative colloid in alkaline ones, and neutral at  $P_H = 4.6$ . The nature of the charge, therefore, is not inherent in the gelatin, but depends on the reaction of the surrounding medium. He also states that "pelt is a gel." This seems a somewhat archaic use of the term "gel," and one which is to be avoided.

Mr. Bennett's book, regarded as a treatise on the manufacture of leather, fills an obvious gap in scientific industrial literature, and as such is to be thoroughly commended.

DOROTHY JORDAN LLOYD.