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The Quantitative Determination of Cotton, Linen and Wood Fibres in Paper Pulp.

By W. DICKSON, F.I.C.

(Under the Analytical Investigation Scheme.)

(*Read at the Meeting, April* 1, 1925.)

Introduction.—In a previous paper (Analyst, 1921, **47**, 373) the use of polarised light in distinguishing and determining hemp fibres in presence of wood fibres was dealt with.

It was anticipated that cotton and linen fibres could be distinguished and determined in a similar manner. A case came to the author's notice in which the content of chemical wood fibre present in a rag and wood paper was required. This determination was carried out in precisely the same way as that of hemp previously mentioned. For preliminary tests, however, a few synthetic mixtures of cotton, linen and wood fibres were examined with the following results:—

	Taken. Per Cent.	Found. Per Cent.	Percentage Error.
Chemical wood in rag and wood mixtures	16·6	17·4	+4·8
,, ,, ,, ,, ,, ,, ,,	33·3	32·8	-1·5
,, ,, ,, ,, ,, ,, ,,	12·0	11·1	-7·0

The results obtained for the unknown samples of paper were 13·5 and 13·7 per cent. respectively. These results were stated by the makers to correspond very closely with the furnish employed by them.

In this work no attempt had been made to distinguish between cotton and linen in the rag. It is, however, usual for furnish analyses to show each constituent separately. From photo-micrographs previously taken by the author, it was thought that there would be little difficulty in distinguishing between cotton and

linen by means of polarised light. When, however, synthetic mixtures were taken, it was at once seen that the problem was not so simple. It is true that a sample of pure cotton fibre gives quite a characteristic appearance under polarised light, and also that a sample of pure linen fibre gives another characteristic appearance. If, however, the two fibres are beaten together, the characteristics are much more difficult to observe, and good counts are not easily obtained.

Acting on a suggestion thrown out during the discussion of the previous paper, attempts were made to reduce metals on the fibres in such a way as to bring out sharp distinctions.

Copper, gold, and silver were used under various conditions. The first two metals did not seem to be of service for the particular problem in hand, but reduction of silver from ammoniacal silver nitrate and subsequent clearing with dilute nitric acid yielded promising results.

Distinction between Cotton and Linen Fibres by Staining with Silver Nitrate.—A study of this method was therefore made. Samples of pure cotton were stained and cleared. Under the microscope with polarised light these appeared bright and showed all the characterictics of cotton fibres as seen by this method of illumination. Samples of linen fibre, on the other hand, retained their dark silver stain even after clearing, and, when viewed under the microscope, failed to transmit light. They appeared practically black. By turning the cross nicols attached to the substage of the microscope one can cut out the linen and cause the cotton to appear, or cut out the cotton and cause the linen to appear. Under ordinary light the cotton fibres, being very transparent, are scarcely seen, whilst the linen fibres, filled with reduced silver, appear almost black. Viewed under polarised light, they are merged into the black background. Figure 1 shows linen fibres under ordinary illumination. Figure 2 shows cotton fibres viewed under polarised light. The corresponding views with the nicols turned would be, for linen, a perfectly black background (polarised light); for cotton, almost a white background (ordinary illumination). Figure 3 shows a mixture of cotton and linen under ordinary illumination. It will be seen that the linen is very black, whilst the cotton is practically invisible. Figure 4 shows the same mixture under polarised light. The cotton now shows up very brightly, whilst the linen is merged into the black background.

It will thus be seen that extremely good differentiation between cotton and linen has been obtained.

The differentiation is nearly as great as that between black and white, and it is one which readily lends itself to photographic reproduction. This is a great advantage from the standpoint of disputed furnishes.

It should be noted that many other fibres, for example, hemp, manilla, and esparto, stain like linen, and that it is the behaviour of cotton which is exceptional. This is perhaps to be expected, since the function of the cotton hair on the plant is quite different from the function, in their respective plants, of the other fibres mentioned.

Fig. 1

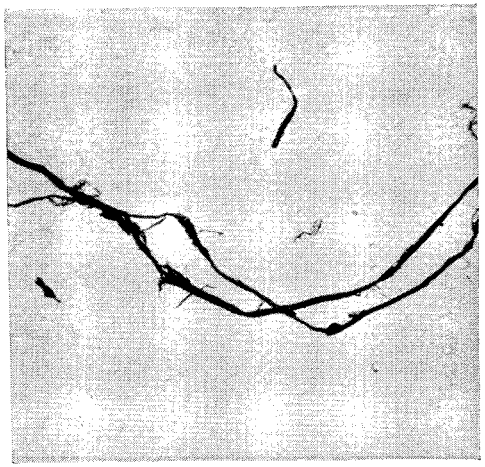


Fig. 3

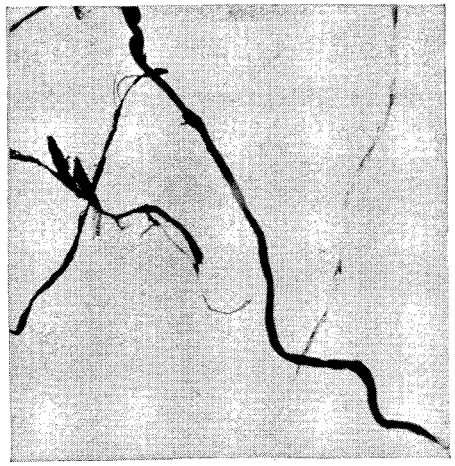
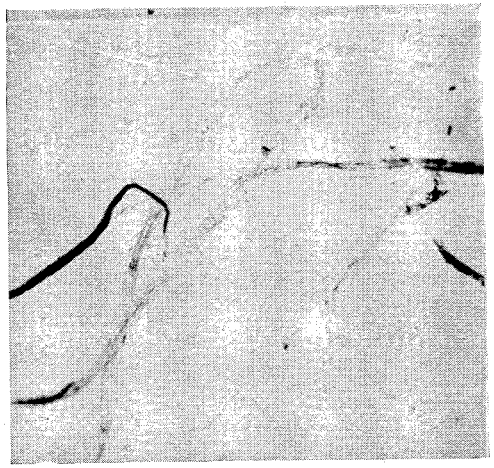


Fig. 5



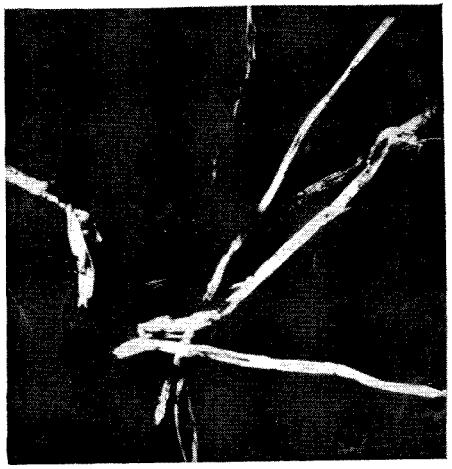


Fig. 2

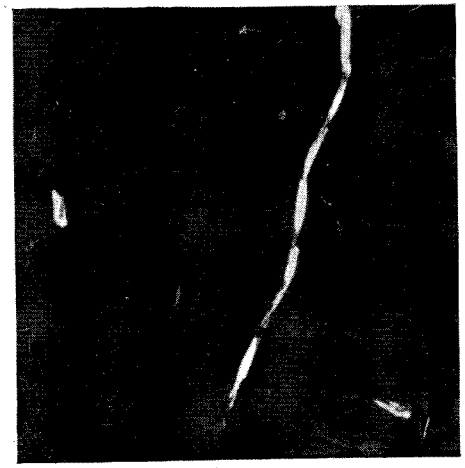


Fig. 4

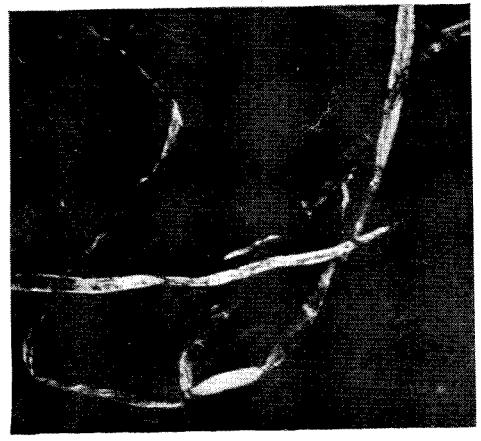


Fig. 6

Fig. 1. Linen fibres under ordinary illumination. Fig. 2. Cotton fibres under polarised light. Fig. 3. Mixture of cotton and linen under ordinary illumination. Fig. 4. The same mixture under polarised light. Fig. 5. Wood fibres under ordinary illumination. Fig. 6. The same wood fibres under polarised light.

RATIONALE OF THE METHOD.—It has long been known that cellulose fibres, cotton wool, etc., have reducing properties. As an example, cotton waste for use in explosives manufacture is generally specified to give not more than such and such a copper reduction value. This is usually determined by the reduction of Fehling solution. The material causing reduction is considered as an impurity.

In order, therefore, to show that a method based on the capacity of the fibres themselves to reduce silver was sound, it became necessary to show that it would work, even on finely bleached rags. It is evident that so long as the reduction takes place on the linen the cotton does not matter. Pulp was therefore prepared from an old linen handkerchief which had been frequently washed and bleached, and which yielded a perfectly white pulp. This pulp was put through the staining and clearing processes, and appeared in a satisfactory state under the microscope. Thus the cleanest of linen rags will show up quite well.

It was, of course, evident that the crux of the method was the clearing process. For some reason stained linen resists the action of dilute nitric acid much better than does cotton. This fact was brought out by a series of quantitative determinations of reduced silver in the fibres after clearing.

TABLE 1.

Silver Reduction Values.

	Reduced silver. Per Cent.	Silver remaining after clearing. Per Cent.
Linen handkerchief	9·09	5·15
Sample of flax	11·70	9·44
,, ,, cotton	18·69	0·51

Transverse sections of cotton and linen fibres were stained with silver and examined. The silver appeared chiefly in the canals, but also in the tissue of the walls. The canals were bigger in the case of cotton. The cell walls of the flax appeared stronger, and are therefore probably less easily permeated by the acid.

A paper by Ball (*The Empire Cotton Growing Review*, No. 2, 1924, p. 94) throws some light on the subject. This paper shows that cotton fibres have a most complex structure, consisting of two series of spirals. Ball sums up his ideas of the structure of the cotton fibre by comparing it to a "sponge." The cotton sections showed small granular points throughout the cell wall. These were doubtless sections of Ball's spirals stained with silver.

This conception seemed to fit in with the rapid way in which the clearing solution permeates the cotton fibre. So far as is known, the linen fibre has not been subjected to such a searching examination as that given by Ball to the cotton fibre. It is to be expected, however, that if this were carried out, it would be found that the flax fibre is less of a "sponge" than the cotton fibre. It was considered that such an investigation was outside the scope of such a paper as the present one.

Whatever the actual explanation of the reaction may be, the main thing from a practical point of view is to be able to bring it about with certainty when required. In some of the earlier experiments this did not happen; at least the differentiation was not so good as expected. When this took place it was evident that the conditions were not properly understood. Variations in the concentration of silver solution were tried and found to have some effect. This work had of necessity to be done in an intermittent manner, and it was suspected that drying of the fibre between staining and clearing had some effect. This was investigated, and it was found that drying the fibre between staining and clearing had a profound effect on the retention of silver by the linen. The cotton was not affected to any great extent.

Obviously, therefore, the mixtures should be dried after staining. This, however, raised a difficulty. If the stained pulp is allowed to dry spontaneously on the filter paper, it mixes up fairly well with the clearing solution. If, however, it is dried quickly in the water oven it forms a clot which is very difficult to tease out. Solid masses of fibres appear in the clearing solution, prevent uniform clearing, and spoil the result. It was desired to avoid spontaneous drying, as this takes too much time. Eventually it was found best to cover the fibre with alcohol, mix up and drain, and repeat the process. After the alcohol had drained the second time, the filter was removed to the drying oven, where drying took place in a short time. The fibre so dried does not clot and is easily teased out. Efforts were made to tease out in water and so get extremely uniform pulp. This proceeding completely ruined the result, inasmuch as both kinds of fibre came out of the solution more or less uniformly stained. The differentiation had been lost. It is thus essential that the dry fibre in a well teased out state should go direct into the clearing solution.

Determination of Cotton and Linen Fibres.—Several synthetic mixtures were made up and examined by the method indicated. The results were as follows:—

Table II.

Determination of Cotton and Linen Fibres in Mixtures of the two.

	Taken. Per Cent.	Found. Per Cent.	Percentage Error.
Linen	54·0	53·4	−1·1
,,	50·0	50·2	+0·4
,,	26·0	26·3	+1·1
,,	24·5	24·8	+1·2
,,	11·2	10·9	−2·6
Cotton	14·4	14·6	+1·4

It will be seen that, on the whole, the results are within the limits of error permissible in many ordinary gravimetric methods of analysis.

Determination of Cotton, Linen and Wood.—When wood is stained with silver and cleared by the method used for cotton and linen, most of the cells are

comparatively little stained, and these stand out well under polarised light. There are, however, some which retain silver strongly, and these are difficult to distinguish from linen. Generally the dark woody fibres show some characteristics of wood by which they can be distinguished, but, even if this is not the case, the size of the fibres will enable them to be distinguished from the linen. The wood fibres, but little stained, show all the usual characteristics of the ribbon type of fibres in chemical wood pulp, and the small pit-like markings show up well, owing to their retention of silver. They are consequently easily distinguished from cotton.

Figure 5 shows wood fibres under ordinary illumination, and Figure 6 the same fibres under polarised light.

These figures will serve to illustrate what has just been said regarding the appearance of wood fibres when treated by this method.

A series of synthetic mixtures containing cotton, linen and wood was made up and examined by the method under consideration. The results were as follows:

DETERMINATION OF COTTON, LINEN AND WOOD FIBRES IN MIXTURES OF THE THREE.

TABLE III.

		Taken. Per Cent.	Found. Per Cent.	Percentage Error.
Cotton		59·6	60·2	+1·0
Linen	(1)	20·2	19·3	-4·4
Wood		20·2	20·5	+1·4
Cotton		32·8	33·1	+0·9
Linen	(2)	39·1	40·5	+3·5
Wood		28·1	26·4	-6·0
Cotton		79·2	77·3	-2·4
Linen	(3)	10·9	12·3	+12·8
Wood		9·9	10·4	+6·0

The results are good. In No. 3 mixture, where the quantities of linen and wood are small, the percentage error in the former is high. This is in agreement with the experience noted in the previous paper, where it was shown to be useless to attempt to determine quantities of fibre below about 10 per cent., and to expect to get a low percentage error. The details of the method are as follows:—

METHOD.—The paper is brought into the form of pulp in exactly the same manner as in the previous paper, ANALYST, 1923, **48**, 377. The pulp is collected in the Gooch crucible as before, but, when ready for staining, it is transferred to a test tube containing 10 c.c. of 5 per cent. silver nitrate solution which has been converted into ammoniacal reagent by adding from a burette, immediately before use, the necessary quantity of 20 per cent. ammonia solution just to dissolve the silver hydroxide first precipitated.

The pulp is stained by heating it in a boiling water bath for 30 minutes. It is filtered off through a small tough filter paper, washed thoroughly with distilled

water, 10 c.c. of alcohol are added to the filter, and the pulp is gently stirred, care being taken not to injure the filter paper. The filter is allowed to drain, a further quantity of 10 c.c. of alcohol is allowed to drain through, and the filter is dried in the water oven. When dry, the pulp is separated from the filter paper and thoroughly teased out by means of a pair of fine forceps and a small scalpel. It is most important that this teasing shall be thorough, as imperfect teasing leads to unequal clearing in the next operation. Twenty c.c. of 2 per cent. nitric acid, made by diluting 2 c.c. of nitric acid (sp. gr. 1·42) to 100 c.c. with distilled water, are placed in a crystallising dish and heated to 70–80° C. on a water bath. The teased pulp is added and stirred frequently during a period of five minutes. The mixture is filtered quickly and washed thoroughly with distilled water. The pulp is now diluted to the same concentration as it was to begin with, and slides are made and mounted. This and subsequent operations are the same as described in the previous paper.

Known mixtures should be used for practice until the staining method has been mastered and good results obtained with the mixtures. In making the counts doubtful fibres may be encountered, and in diagnosing these, consideration should be given to the micro-structure of the fibre, as well as to the depth of silver observed. Slides containing pure fibres, stained by the method used, should be at hand for purposes of comparison.

The author desires to express his thanks to the Society for a grant from its research fund towards the cost of this work.

Discussion.

The President asked whether the author had tried the action of nickel salts on cotton and wood fibres with a view to differentiation, and whether he had used a selenite plate in addition to polarised light. Later, he mentioned that good results had been obtained with a mixture of nickel sulphate (5 per cent.) by means of the selenite plate.

Mr. Chaston Chapman said that he was glad that the author had found time to act upon the suggestion which he (Mr. Chapman) had made to him sometime ago—that he should study the relative reducibility of certain metals, such as silver, gold and palladium, as a means of distinguishing between various fibres. He desired to congratulate Mr. Dickson on the results he had obtained with silver, and would further suggest that useful indications might be obtained by the employment of other kinds of illumination than ordinary white or polarised light. He referred, for example, to certain monochromatic lights, or even ultra-violet rays might not be out of the question, although their use was a little troublesome and involved somewhat expensive apparatus.

Mr. R. L. Collett congratulated the speaker on his technique, but doubted whether the majority of analysts could hope to imitate the accuracy of his counting under the microscope.

Dr. H. P. Stevens asked whether loose, untreated fibres or beaten fibres were used in the experiments. As the results were expressed in percentages, the respective densities and lengths of the different fibres would have to be known. He was slightly sceptical of the accuracy of the counting method and enumerated difficulties.

Mr. DICKSON, replying, said that he had tried other metals, but, owing to lack of time, had determined to concentrate on silver. He had not used nickel, but thought it might prove useful in differentiating between cotton and wood fibres. With regard to other sources of illumination, he had tried various monochromatic lights, but, as his apparatus was designed for polarised light, he decided that these would not justify the cost of installation. He admitted the relative inaccuracy of the counting method as usually applied, but affirmed that inaccuracy could be much minimised by teasing the fibres apart as far as possible. In addition, he maintained that good differentiation by staining with silver, and the use of polarised light helped greatly. He used beaten fibres in his experiments, thus imitating as far as possible the conditions of the paper maker. In many commercial mills, he explained, the beating was destructive, and corrections had to be applied to make counts correspond to the quantities of materials known to be present. His own results, however, had not shown any correction to be necessary. He referred Dr. H. P. Stevens to the work of Spence and Krauss (*World's Paper Trade Review*, Dec. 18, 1917) on the correction for differences in density of the various fibres. He stated that neither in the case of his experiments on hemp and wood mixtures (ANALYST, 1923, **48**, 73) nor in the present work did corrections appear necessary, although the mixtures were made up on the basis of dry weight of fibre.

The Influence of Palm Kernel Meal on the Composition of Bacon Fat.

BY J. S. WILLCOX AND H. T. CRANFIELD.

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

THE utilisation of the residues from the oil extraction of palm kernels for pig feeding has, in this country, increased very largely in recent years. During the time of scarcity of feeding stuffs in the war period, ground palm kernel cake and palm kernel meal were introduced into many proprietary pig meals, and since that time these meals have become quite common in the dietary of the pig. A distinction between these two types of palm kernel residues is that palm kernel cake meal contains about 6 per cent. of oil, whereas palm kernel meal obtained by the solvent process of oil extraction contains only about 1½ per cent. of oil.

The introduction of a new substance into the rations of pigs is always watched carefully by bacon curers, who, in some cases, are only too ready to attribute inferior quality in bacon to the new foods. How far such prejudice is justified can only be determined by carefully conducted feeding experiments.

Such an experiment has recently been carried out by Messrs. Frank Rayns and John Duncan, members of the Agricultural Staff of the Midland Agricultural and Dairy College, the object of this experiment being to ascertain the influence, if any, of palm kernel meals on the keeping qualities of bacon and on the consistence

of bacon fat. A full report of this work has recently been published in the form of a College Bulletin.*

Samples of the bacon produced from this experiment were sent to the College Laboratories, and the fat submitted to analysis. It is considered desirable that the results obtained should be placed on record, since they may be of some interest to analytical chemists generally.

Brief details of the experiment are as follows:—Four pens of six pigs each were fed on the following rations:—

Ration for first six weeks.

	Pen 1. Per Cent.	Pen 2. Per Cent.	Pen 3. Per Cent.	Pen 4. Per Cent.
Palm kernel cake meal	20	—	40	—
Palm kernel meal (extr'd.)	—	—	—	40
Sharps	70	70	50	50
Barley meal	10	10	10	10
Bean meal	—	20	—	—
Whey	One gallon per pig per day to all pens.			

Ration for last six weeks.

Palm kernel cake meal	20	—	40	—
Palm kernel meal (extr'd.)	—	—	—	40
Sharps	20	20	10	10
Barley meal	60	60	50	50
Bean meal	—	20	—	—
Whey	Two gallons per pig per day to all pens.			

The age of the pigs at the outset was 16 weeks. At the end of the twelve weeks' period the pigs were sold to a well known local firm of pork butchers and bacon curers, who gave an expert opinion on the pork from the merchant's point of view. They stated:—"For a porker or cutting pig you could not find any difference in any of the pigs."

The most suitable bacon pig was selected from each pen and cured for bacon. The curers reported on the bacon as follows:—

Pen 1. (Receiving 20 per cent. of palm kernel meal, containing 6 per cent. of oil.) "Soft in the fat."

Pen 2. (Receiving no palm kernel meal.) "Fat firm and the best of the lot."

Pen 3. (Receiving 40 per cent. of palm kernel meal, containing 6 per cent. of oil.) "Softer than No. 2, and will, no doubt, be very soft and tallowy in one or two months."

Pen 4. (Receiving 40 per cent. of palm kernel meal, 1½ per cent. of oil.) "Soft but not so bad as Nos. 1 and 3.

From these comments it appears that the palm kernel meal had some influence on the consistence of the bacon fat, tending to make it soft.

* "Report on the use of Palm Kernel Bye Products in the Fattening of Pigs, and their influence on Pork and Bacon." The Midland Agric. and Dairy College Bulletin. January, 1925.

Samples of the back bacon fat were taken for analysis. Two methods of extraction of the pure fat were used:—(*a*) The crude fat was cut into thin strips and rendered at a temperature not exceeding 70° C. (*b*) The crude fat was passed through a mincing machine and then extracted with petroleum spirit in a Soxhlet extractor. The samples of pure fat gave the following results on analysis:—

	From Pen No.	Iodine value.	Saponification value.	Melting point °C.	Reichert-Meissl value.	Polenske value.	Zeiss Butyro-refractometer No. at 40° C.
A. Rendered fat.	1.	56·5	205·8	29·5	0·44	0·6	48·5
	2.	59·7	198·0	28·0	0·49	0·9	48·5
	3.	57·2	198·5	29·0	0·55	0·6	48·0
	4.	56·1	200·5	29·0	0·44	0·6	48·0
B. Fat extracted with petroleum spirit.	1.	56·5	199·5	30·0	0·60	0·7	48·5
	2.	56·1	193·4	28·0	0·66	0·7	49·5
	3.	56·8	197·9	30·0	0·44	0·7	48·0
	4.	58·6	202·3	30·5	0·55	0·7	48·5

For comparison, the following figures for pure lard are given:—

Authority.						
Bolton and Revis	57 to 66	193 to 199	35 to 46	—	—	49 to 52
Wynter Blyth	59	196	—	0·5	—	48·6 to 51·2
Wiley	62·5	—	40·7	—	—	—

Discussion of Results.—On comparing the two sets of figures, A and B, there appears to be no consistent variation in composition due to the method of extraction.

Comparing the figures obtained from the different pens, the following points are observed:—*Iodine value*: No definite variation noticeable. *Saponification value*: Pen A2 and B2 (no palm kernel) give a slightly lower value than the other three pens. *Melting point*: Pen A2 and B2 produce a fat with a slightly lower melting point than the other pens. *Reichert-Meissl and Polenske values*: There are no significant variations which follow the differences in feeding. *Refractometer number*: This figure is very consistent throughout.

The only indications of possible influence due to palm kernel feeding are a slight rise in the saponification value and also in the melting point. These, however, do not follow the increase in palm kernel oil given in the rations, since the highest saponification value figures (A1 and B4) and the highest melting points (A1 and B4) are not from the pen receiving the largest amount of palm kernel oil (A3 and B3).

Judging from these figures, one must arrive at the conclusion that the analytical data do not explain the experts' comments on the quality of the bacon and consistence of the fat.

It is suggested either that the hardness or softness of bacon fat is not indicated by marked differences in chemical composition of the pure fat, or that small differences in chemical composition, within experimental variation, are sufficient to produce a marked influence on the physical condition of the bacon fat.

The authors wish to express their thanks to Messrs. Rayns and Duncan for permission to quote experimental details and comments from their report.

THE MIDLAND AGRICULTURAL AND DAIRY COLLEGE,
SUTTON BONINGTON, LOUGHBOROUGH.

DISCUSSION.

Mr. OSMAN JONES suggested that a soft fat might also be due to a constitutional defect in the pig as distinct from the better known cause of the use of unsuitable food. From his own experience he had found that variations in the quality of the fat might result under identical conditions of feeding. That is to say, that out of a litter of ten pigs all fed alike, nine would perhaps be normal, the tenth soft in the fat. The use of palm kernel meal for pigs ought not to be encouraged where the object was to produce prime quality bacon, as it had been found that by using even 15 per cent. of the total ration, it had a tendency to produce a thin bellied pig with hard lean. Irrespective of feeding he had always found that a soft fat gave a higher iodine value and a lower melting and setting point than a firm fat.

Mr. E. R. BOLTON thought the quality of the pig-fat might be influenced by other ingredients than the fat in the feeding-stuff, as the latter did not retain its identity throughout.

The PRESIDENT compared the case with that of certain metals and suggested that a physical factor was probably responsible for differences.

Mr. T. MACARA reminded the authors that "fat" in animals was not only fat, but also moisture and tissue; the percentage of moisture should be determined, and the size of cells and general structure should be subjected to microscopical examination, as these might have a greater influence on the apparent hardness or softness of the "fat" than the actual composition of the true fat.

Dr. J. A. VOELCKER mentioned that maize meal always gives a soft fat, and as maize was present in such very small quantities, it was hardly likely that the softness was due to this.

Mr. R. L. COLLETT said that it was well known that maize protein was deficient in tryptophane, and this deficiency might affect the fat metabolism in the body.

Mr. OSMAN JONES remarked that, while whole maize or maize meal produced a soft fat, it would appear that flaked maize did not have this tendency. He also said that when cotton seed cake was used for feeding, cases were known where the resulting fat gave a positive reaction to the Halphen test.

Mr. E. R. BOLTON said that cotton seed gave a hard fat though, like maize, it contained a liquid oil.

Mr. C. L. CLAREMONT pointed out that it was a well known physiological fact that whatever fat was present in the food had to be digested and broken up before passing into the body.

Mr. G. D. ELSDON said it was necessary to examine the fat rather than the fatty acids. The Valenta test, he thought, might show differences; also the form of the crystals separated from ether.

Mr. M. S. SALAMON asked whether a record of the free fatty acid in the original fat had been kept, as acid cakes had a bad effect on pigs.

Mr. E. T. BREWIS said that, judging by the evidence obtained with 150 to 200 pigs, almond cake gave a fat so soft that it all boiled away.

Mr. H. T. CRANFIELD in his reply, said that he was merely trying to investigate why the analytical data did not correspond with the experts' opinion. Fat in the body, he continued, was formed from carbohydrates, but also from fat eaten, though this latter was emulsified, saponified and probably, in part, reconstituted. It was well known that coconut had a marked influence on milk fat—but, as subsequently pointed out by Mr. Partridge, milk fat came through the glands and was not stored; pig fat was fatty tissue, and the analogous case would be pig's milk. The acidity of the cakes had not been determined, but good commercial samples had been used.

A Modified Electrometric Method for the Determination of Hydrogen Ion Concentration.

BY S. GLASSTONE, M.Sc., PH.D., F.I.C.

THEORY.—In the ordinary electrometric measurement of hydrogen ion concentrations a platinised platinum electrode is immersed, either completely or partly, in the solution which is to be examined, and a stream of purified hydrogen gas is bubbled through the liquid; when a steady state is reached the potential of the electrode is measured against that of a standard half-element, usually the calomel electrode. From the result obtained the potential, e, of the electrode on the normal hydrogen scale may be calculated, and the hydrogen ion concentration of the solution, $[H^{\cdot}]$, is then given by the equation:

$$e = RT/F \log_e [H^{\cdot}],$$

that is,

$$e = 0{\cdot}058 \log_{10} [H^{\cdot}] \text{ at } 18^{\circ} \text{ C.}$$

This potential, e, may be regarded as a measure of the tendency of hydrogen gas at a pressure of one atmosphere to go into solution and form hydrogen ions at the particular concentration of the solution under examination; consequently, if the process were truly reversible, in the thermodynamic sense, this same potential would have to be applied to a cathode in the given solution before hydrogen evolution at atmospheric pressure could commence. It has been found, however, that the cathodic potential required depends on the nature of the electrode (Caspari, *Zeitsch. physikal. Chem.*, 1899, **30**, 89); the difference between the actual potential which must be applied before bubble evolution commences at atmospheric pressure and the theoretical reversible potential is called the "overvoltage." In the case of a platinised platinum cathode several authors (Caspari, *loc. cit.*, Coehn and Dannenberg, *ibid.*, 1901, **38**, 609, etc.) have found that in acid solution the overvoltage is practically zero, and consequently visible hydrogen evolution

commences at the same potential as that assumed by a platinised platinum electrode placed in the same solution and around which hydrogen gas at a pressure of one atmosphere is bubbled. The present author has extended these observations to various electrolytes other than acids (*e.g.* solutions of sodium hydroxide, sodium sulphate, potassium chloride, and various Sörensen buffer mixtures made up according to the directions given by Clark in *The Determination of Hydrogen Ions*, 1922, p. 107 *et seq.*), and it has been found that, as long as the solution is well buffered, the overvoltage at the platinised platinum cathode is zero, independent of the hydrogen ion concentration, for all solutions between acid and alkaline. It appeared that this fact might be made use of in a modified form of hydrogen electrode, and so used for the determination of hydrogen ion concentrations. According to the results that have been obtained in the overvoltage measurements, if a small platinised platinum cathode is placed in the solution the hydrogen ion concentration of which is to be determined, and then a small polarising current applied until bubble evolution just commences, then the potential of the cathode will be the same as that of an ordinary hydrogen electrode in the same solution, and so the hydrogen ion concentration may be calculated from it by means of the equation given above. The advantage of the method would lie in the elimination of the apparatus necessary for the preparation and purification of hydrogen, and also in a more rapid attainment of equilibrium, particularly in alkaline solutions, in which the ordinary hydrogen electrode is known to be very sluggish. It also appeared possible that in solutions containing reducible substances the results might be more accurate than with the gas electrode, since much smaller amounts of hydrogen gas would be brought into contact with the electrolyte before the attainment of equilibrium. A number of experiments has therefore been made in which the hydrogen ion concentration of a series of solutions (*e.g.* various buffer solutions, partly neutralised strong and weak acids, acid solutions containing oxidising agents, and aqueous-alcoholic solutions of alkali) was determined by the polarised platinum cathode as outlined above, and the results compared with those given by the ordinary hydrogen electrode or obtained by calculation.

Experimental Method.—The cathode used was either a small piece of platinum foil or a length of about 1 cm. of platinum wire, lightly platinised in the usual manner by the electrolysis of a solution of platinic chloride containing a little lead acetate; the anode was a short piece of smooth platinum wire. These electrodes were placed in the liquid under examination, and a gradually increasing polarising current applied from a battery of accumulators connected across a variable resistance. When bubbles just commenced to be evolved at the cathode, that is at the rate of not more than one bubble per minute, the potential of this electrode was measured against that of a saturated calomel electrode; the tip of the latter was packed with cotton wool to prevent the rapid diffusion of the solution under examination into the standard electrode solution. In order to prevent any appreciable error in the potential due to the resistance of the electrolyte (*cf.* Glasstone, *J. Chem. Soc.*, 1923, **123**, 2926), the tip of the standard

electrode was placed as close as possible to the polarised electrode the potential of which was being measured. As far as potential measurement was concerned the anode was, of course, neglected. From the known value of the potential of the saturated calomel electrode the potential, on the hydrogen scale, of the polarised platinum cathode at which bubble evolution just commenced was determined, and from this the hydrogen ion concentration of the solution was calculated. A number of observations on different solutions was made, and some determinations were also made with the hydrogen electrode in the ordinary way; the general results obtained are described below.

GENERAL RESULTS.—In general, it has been found that, provided the solution is fairly acid or alkaline, that is, if the P_H is less than 3 or more than 11, the new method is more rapid than the ordinary electrometric method and also fairly accurate; in alkaline solutions its superiority is very marked. Between the limits of P_H stated above, however, the method is only of value if the solution is well buffered; otherwise it is quite useless. An attempt was made to follow an electrometric titration of acid by an alkali with the polarised platinum cathode; the shape of the titration potential curve was quite normal, but the sudden rise of cathodic potential occurred before the solution was really neutral. The explanation of this curious behaviour is probably as follows:—In the vicinity of the neutral point, even at the low current densities required to cause a very slow evolution of bubbles, the rate of removal of hydrogen ions from the vicinity of the platinised platinum cathode by discharge is greater than the rate of replacement by diffusion; probably the presence of a large excess of another positive ion retards the rate of diffusion to some extent, and hence there is a local diminution of hydrogen ion concentration, and the potential of the polarised electrode becomes more negative. During the electrometric titration the solution in the vicinity of the cathode, if it was originally acid, will be neutralised before the bulk of the solution, and consequently the sudden rise in the titration curve will occur before the bulk of solution reaches the neutral point. This view has been confirmed by making up an unbuffered solution of partly neutralised sulphuric acid of P_H 6, and adding a drop of phenolphthalein solution; the solution was, of course, colourless. A platinum wire cathode was then polarised in this solution with a current of 0·00005 amp., which was hardly sufficient to cause evolution of bubbles; after a short time a distinct pink colour was observed in the vicinity of the electrode, and the potential measured was much more negative than that given by an ordinary hydrogen electrode, in the same solution. In the case of a well buffered solution (*e.g.* partly neutralised acetic acid), however, a slight diminution of the hydrogen ion concentration in the vicinity of the electrode is at once rectified, and the results obtained with the polarised platinum cathode are identical with those given by the ordinary hydrogen electrode.

The method had also been tried in the case of solutions containing an oxidising agent; in the case of chromic acid results have been obtained which are in agreement with those of Britton (*J. Chem. Soc.*, **1924**, **125**, 1572), provided the solution of oxidising agent is fairly dilute. Similarly, solutions of nitric acid of 0·1 *N*

concentration and below gave results in agreement with those calculated from the concentration and the degree of dissociation.

The polarised platinum cathode will probably be of value in the determination of the hydrogen ion concentrations of alkaline solutions containing organic substances like alcohol or acetone; in these cases the ordinary hydrogen electrode reaches its equilibrium very slowly, whereas the platinised platinum cathode appears to come to equilibrium rapidly. Some observations of this kind have already been made in alkaline solutions containing large proportions of ethyl alcohol, and the hydrogen ion concentrations determined were in agreement with those calculated from the values of the dissociation product of water in alcoholic mixtures determined by Löwenherz (*Zeitsch. physikal. Chem.*, 1896, **20**, 283).

CONCLUSION.—A modified electrometric method, in which a polarised platinum cathode is used, for the determination of the hydrogen ion concentration of solutions has been devised. The new method does not require a supply of hydrogen gas, and equilibrium is attained rapidly; it can only be applied, however, to solutions of P_H less than 3 or more than 11, unless they are well buffered. In alkaline solutions the new method is much superior to the ordinary hydrogen electrode method.

UNIVERSITY COLLEGE, EXETER.

A New Method for the Separation and Determination of Tin in Alloys.*

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

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

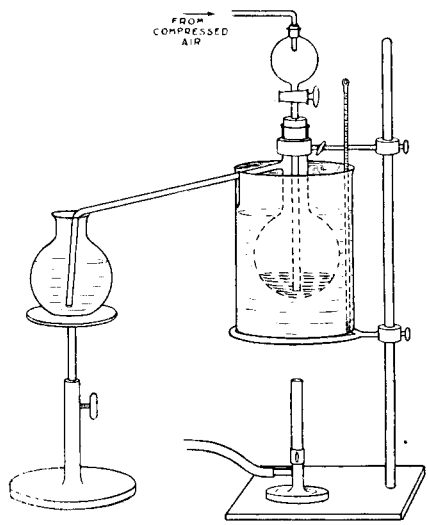
THE presence of tin interferes with the determination of a large number of other metals. The removal of the tin as metastannic acid, though complete, is undesirable on account of the amount of other metals dragged down, which may amount to 5, or even 10 per cent. of the precipitate. Precipitation of sulphides (of, say, lead, copper, etc.) in alkaline tartrate solution yields a bulky, colloidal precipitate very difficult to wash free from tin; other methods involve such drawbacks as the use of large quantities of organic reagents, of hydrofluoric acid with its attendant troubles, etc. The following method was devised to remedy these defects.

PRINCIPLE OF SEPARATION.—The method is based on the fact that the majority of ordinary metals and alloys dissolve, often with considerable violence, in the liquid formed by the addition of bromine to alcohol; the products of the reaction would appear to be the lower bromides (*i.e.* cuprous, ferrous, etc.). If this liquid

* Communication from the Research Department, Woolwich.

is now heated in a suitable apparatus, the volatile bromides can be completely distilled off from the non-volatile ones. In this way a separation can be effected between tin, antimony and arsenic, on the one hand, and copper, iron, lead, zinc, manganese, nickel, and cadmium, on the other. Bismuth appears to distribute itself between the distillate and the residue, and the same is probably true for aluminium, but this metal requires further investigation. Details of the method are as follows:—

APPARATUS.—A Würtz flask (capacity 100 c.c.), having the side tube bent down almost parallel with the neck, is fitted with a cork through which passes



the stem of a tapped funnel (capacity about 50 c.c.), having a narrow mouth, and a stem long enough to reach the bottom of the flask. The mouth of the funnel is closed with a cork through which passes a short tube connected with a source of compressed air (*i.e.* air under sufficient pressure to drive it through the apparatus), so that, when the apparatus is connected, air can be passed through it. The Würtz flask is held by a clamp on a retort stand, and a ring on the same stand carries a bath of carnauba wax in such a way that it can be raised, immersing the flask in the bath, or lowered to allow the flask to cool; it is desirable that a slot should be cut in the mouth of the bath to take the side tube of the flask, thus ensuring more complete immersion.

PROCESS.—The whole apparatus is thoroughly dried and disconnected. A convenient amount (up to 4 grms.) of the alloy, in as finely divided a condition as is practicable, is weighed into the Würtz flask, and the required amount of alcohol poured in through the neck. This varies according to the amount of alloy taken; for 4 grms. 30 c.c. has usually been found sufficient, but the amount can be altered if found desirable. The cork, carrying the tapped funnel, is then pressed firmly into position, the tap turned off, the flask attached to its clamp,

and bromine to the amount of one-third the volume of the alcohol is poured into the funnel. A flask containing about 250 c.c. of water is placed in position beneath the side tube of the Würtz flask, so that the level of the water is just below the mouth of the tube without touching it; this flask should be of sufficiently squat form to allow the side tube subsequently to be immersed as far as possible in the water. The bromine is now run into the Würtz flask *very gradually*, a drop or two at a time; with the first two or three additions nothing appears to happen, but when the action is once started it becomes extremely violent, unless great care is taken. Distillation always takes place to a certain extent, but the rate of admission of bromine should be regulated so as to prevent this becoming excessive. When all the bromine has been added the apparatus is allowed to stand until all action has ceased, when the tap of the funnel is opened and the receiving flask raised so that the side tube dips under the surface of the water as far as possible. The cork, carrying the tube from the compressed air supply, is inserted in the neck of the funnel, and the air turned on, so that a slow stream bubbles through the apparatus. The bath of wax, which has meanwhile been melted, is now placed on its ring and raised so that the Würtz flask is immersed as far as possible; a thermometer is placed in it and a burner lighted underneath it. If the alloy contains any notable amount of tin or antimony, say, upwards of 1·0 per cent., 20 c.c. of strong hydrochloric acid are added to the water in the receiving flask (if this is not done, the mouth of the side tube becomes stopped up with hydrolysed tin or antimony bromide). The temperature is taken up gradually to 300° C., care being taken to avoid loss of fumes above the water in the receiving flask due to excessive air supply; on the other hand, the air current must be sufficient, otherwise there is a danger of the lower end of the funnel stem being choked by the solidification of the salts in the Würtz flask; also the volatile bromides are very heavy, and a fairly rapid stream of air is necessary to sweep them out. Towards the end of the distillation the air current can be somewhat increased; a good plan is to keep it just below the point at which fumes are carried off from the surface of the receiving liquid. When the neck and cork of the Würtz flask have been quite dry for about 10 minutes the burner is turned out, the thermometer removed, and the wax bath lowered till clear of the flask, the air current being allowed to continue. When the Würtz flask has cooled sufficiently the receiving flask is lowered till the liquid in it is clear of the side tube, the air turned off, and the cork and tube removed from the mouth of the funnel; 20 c.c. strong hydrochloric acid are now run in through the funnel, the tap turned off, the wax bath raised round the flask again, and the hydrochloric acid allowed to distil for a short time; the object of this is to clear out traces of tin bromide from the side tube. The contents of the Würtz flask should now be entirely free from tin, antimony and arsenic, and consist of bromides of the other metals present, together with a small amount of organic matter; they can be dissolved, rinsed out of the flask and determined by any suitable processes. The liquid in the receiving flask should contain the whole of the tin, antimony and arsenic, free from other metals; the only notable exception to the above seems to be bismuth, which sublimes and

distributes itself over the whole apparatus including both distillation and receiving flasks.

TIN.—To determine tin in the distillate, the contents of the receiving flask are rinsed into a flask (capacity 800 c.c.) and boiled until the brown organic compound at the bottom has been driven off; this must be done in the fume cupboard, as the solution contains an intensely lachrymatory organic compound which will speedily make the laboratory uninhabitable; the initial distillation, on the other hand, by observing the precautions outlined above, can be carried out in the open laboratory without discomfort. When the distillate is clear and colourless 50 c.c. of hydrochloric acid are added, and the liquid is oxidised with potassium chlorate, added in small quantities at a time, until all the bromine has been driven off. After all chlorous fumes have been dispelled the liquid is filtered, if necessary, and the tin is reduced and titrated with iodine in the ordinary manner. If a considerable amount of antimony is present, it must be removed; this may conveniently be done by the method of Järvinen (*Zeitsch. anal. Chem.*, 1923, **63**, 184), which, for ready reference, is abstracted below:—

The solution, which if necessary is concentrated to about 70 c.c., is rinsed with 30 c.c. of concentrated hydrochloric acid into an Erlenmeyer flask; 0·5 grm. of pure powdered iron is added, and the flask is closed with a Bunsen valve and heated for one hour on the water bath; another 0·5 grm. of iron is then added and, after 10 minutes, a little more; the liquid is then at once filtered into a flask, and the precipitate washed with hot water. As traces of tin are dragged down by this precipitated antimony, the precipitate is rinsed off the paper back into the Erlenmeyer flask with 40 c.c. of water, and dissolved by adding 20 c.c. of concentrated hydrochloric acid and boiling, with subsequent addition of small quantities of potassium chlorate. An addition of 0·5 grm. of iron is made to the cooled solution, and it is allowed to stand for half an hour, after which it is heated to about 60° C., another 0·5 grm. of iron added, and it is filtered, the filtrate being mixed with that obtained in the first separation. The tin, free from antimony, is now all contained in the filtrate and may be reduced and determined in the ordinary manner.

The following results were obtained with synthetic mixtures by the alcohol-bromine method:—

Weight of copper.	Weight of Tin.		Percentage of tin.	
added. Grms.	added. Grm.	found. Grm.	added.	found.
3·80	0·2000	0·1983	5·00	4·96
3·84	0·1600	0·1574	4·00	3·93
3·88	0·1200	0·1196	3·00	2·99
3·92	0·0800	0·0760	2·00	1·90
3·96	0·0400	0·0395	1·00	0·99

Where the percentage of tin lies below the limit (say, 0·5 or possibly 0·2) which can be accurately determined volumetrically, the method has no advantage over the ordinary metastannic acid process.

OTHER METALS.—One of the chief points of difficulty in the analysis of alloys containing tin or antimony lies in the determination of the other metals present; a series of experiments was therefore carried out to prove that there is no loss by volatilisation of the bromides of these metals. In all cases they were mixed with a large excess of tin and the metals were determined in the solution of the residue in the flask by ordinary methods. The following results were obtained:—

Weight of tin taken. Grms.	Metal added.	Weight taken. Grm.	Weight found. Grm.
4·0	Copper	0·0458	0·0461
,,	,,	0·0840	0·0842
,,	,,	0·2266	0·2254
,,	Iron	0·0440	0·0442
,,	,,	0·0800	0·0795
,,	Lead	0·0420	0·0414
,,	,,	0·0800	0·0813
,,	Nickel	0·0441	0·0428
,,	,,	0·0801	0·0786
,,	Cadmium	0·0413	0·0429
,,	,,	0·0806	0·0829
,,	Manganese	0·0977	0·0971
,,	Zinc	0·0800	0·0800

The method has proved invaluable in the analysis of tin-zinc alloys; it allows of a clean separation of tin and antimony from any of the metals mentioned above in little over an hour, and tin can be determined in a bronze in well under a day. A curious point to note is that the alcohol need not be absolute; all the experiments mentioned above were carried out with 96 per cent. alcohol. An obvious drawback to the method is that it can only be used on the metals themselves, not on their salts. The principle of the method would appear to be of wide application, and further work on it is in progress; partial success has been obtained in determining antimony in antimony-copper alloys and vanadium in steel, but not sufficient to justify publication.

The method has been tried for the determination of sulphur in steel and lead, but has proved unsatisfactory.

It should be unnecessary to point out that anhydrous bromine is an unpleasant, not to say dangerous, substance, and that special care should be taken with this part of the process.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE DETECTION OF ANNATTO IN MILK.

As it is now illegal to add colouring matter to milk, a search for annatto, which is the colour usually employed, has become necessary in milk samples taken under the Food and Drugs Acts.

Five c.c. of milk are treated as in the Gottlieb-Röse process for fat by adding 0·5 c.c. of ammonia solution (sp. gr. 0·88), followed by 5 c.c. of alcohol, 12·5 c.c. of methylated ether, and finally 12·5 c.c. of petroleum spirit. The annatto remains in the aqueous layer, colouring it yellow. The ether is blown off, and a piece of filter paper is placed in the solution containing the annatto. With an amount of annatto equivalent to 0·35 grains per gallon of milk the aqueous solution showed a distinct yellow colour; the filter paper, after standing in the solution overnight, was dyed a distinct brown. When tested (i) with stannous chloride it gave a pink coloration; (ii) with citric acid, a pink coloration; and (iii) with nitric acid, a greenish-blue coloration; in each test the reactions were very distinctive.

I have found this method to give much more conclusive results than that of adding a strip of filter paper to the milk direct, after addition of sodium bicarbonate.

HAROLD LOWE.

MODIFICATIONS OF THE FERRIC CITRATE PRECIPITATION TEST FOR TANNINS.

THE experience gained by a further use of the test for tannins described by the writer (ANALYST, 1924, **49**, 467) has led to the adoption, for certain purposes, of two useful modifications of the test. The first of these is employed in testing commercial extractives and mixtures. It consists in the addition of normal sodium sulphite, to prevent excessive precipitation, in the preliminary stages of the test, of dehydrated or oxidised tannin in the form of phlobaphene. It is more particularly intended to supersede the second method described in the original paper, when the purpose is that of detecting "tannin" in a preparation which may contain but little of that class of substance in soluble form. It is particularly useful, therefore, in the analysis or identification of pharmaceutical "mixtures" or extractives.

Method.—Dissolve 0·5 grm. of freshly powdered normal sodium sulphite in a few c.c. of water. Boil, and drop in the concentrated extractive until the colour is sufficiently marked. In the case of a weak pharmaceutical "mixture" add the sulphite to a little of this and boil. In either case, next add 1 grm. of ammonium acetate to the boiling mixture, cool a little, and filter. Lastly boil the mixture with sufficient of the ammonium ferric citrate solution as usual. The filtration prior to the addition of the ferric citrate solution will remove interfering substances, but only the less soluble portion of the tannin-bodies, the sodium sulphite preventing the precipitation of most of the more soluble part.

If the precipitate given is purple-violet or violet-blue, as is most frequently the case, the presence of tannin may be assumed with certainty (in one case, only, namely, logwood, the coloured precipitate given is only partly due to tannin, hæmatoxylin also being partially precipitated as a violet-blue compound). Should the precipitate be brown-black, however, another modification of the test must be applied to distinguish between phloroglucinol-catechol flavones or flavonols and certain phlobatannins. These anthoxanthins are seldom, if ever, precipitated by the original method of applying the test, if the usual weak aqueous extractives prepared for testing crude vegetable substances be employed. Further experience, however, has shown that even that method, when used with stronger extractives or solutions, may partly precipitate quercetrin and luteolin, and possibly other related flavone derivatives, as brown iron compounds, unless the admixture is kept distinctly on the acid side of P_H 7. The use of sodium sulphite, of course, increases the tendency to the precipitation of these anthoxanthins. The following method has therefore been devised to distinguish between these bodies and tannins in plant extractives:—

Differentiation of Anthoxanthins and Tannins.—Boil 5 c.c. of the extractive, or water-diluted extractive, with the iron and ammonium citrate solution. Cool and filter. Add 1 grm. of ammonium chloride and again boil. All tannins and phlobaphenes are precipitated by this means, some completely and some less completely, but no anthoxanthins appear to be thus precipitated. In the absence of tannins, shown as just described, the presence of anthoxanthins may often be demonstrated by next adding a little solution of ammonia and again boiling. A deep brown precipitate, often copious, indicates flavone or flavonol. If tannins have been indicated in "neutral" solution, and the precipitate removed by filtration, it is not safe to assume that a further precipitate given on boiling with ammonia is due to an anthoxanthin, since some phlobatannins are incompletely precipitated until the admixture is made alkaline.

With other phenolic bodies than those named in this note the results given by the new modifications of the test are similar to those given by the older method, except that the coloured solutions that are yielded when sodium sulphite is used are blue-violet, rather than purple-violet, in the case of phenols which give such colour-reactions (see list in original paper).

It should be noted also that the ammonium chloride method, although a little more specific for tannins than the other methods, is intended to supplement these, when necessary, and not to supersede them, because it is less delicate than these, the precipitate given being less bulky and less characteristically coloured.

ALAN H. WARE.

UNIVERSITY COLLEGE, EXETER.

PREPARATION OF NESSLER SOLUTION.

IN my note (ANALYST, 1925, 67) on this subject, I stated "The sensitiveness of Nessler's solution . . . usually increases with age." Mr. R. C. Frederick (ANALYST, 1925, 183) gives some good evidence to show that this statement, which is commonly encountered in literature, is not correct. I made the statement, however, not only because it was a current one, but because it accorded with my experience.

Carrying my mind back, I feel sure that the solutions with which I had noticed an increase with age, were those which, probably from having been made at too high a temperature, were very little sensitive when freshly made, and these solutions did increase in sensitiveness with age. It is evident from Mr. Frederick's

experiments, and also from my experience with the modified solution I proposed, that a well prepared Nessler solution does not increase in sensitiveness with age.

While I feel that the time-honoured statement has certainly a well-founded justification, I did not realise that its limitations were so narrow as Mr. Frederick has shown.

H. DROOP RICHMOND.

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.

CITY OF BIRMINGHAM.

ANNUAL REPORT OF THE CITY ANALYST FOR 1924.

DURING the year 4067 samples were bought informally and 646 formally, being at the rate of 499 per 100,000 inhabitants. These included 2512 samples of milk (152 adulterated), 234 of butter (2 adulterated), 511 of margarine (1 preserved), 43 of ground ginger, 73 of mustard, 40 of vinegar, 95 of coffee, and 20 of chicory.

Of the 206 samples adulterated, 183 were foods, 2 beverages, and 21 were drugs. The percentage of adulteration was 4·4, as compared with 4·8 in 1923.

MILK.—The number of samples taken (2512) was at the rate of 266 per 100,000 inhabitants.

FLOUR.—Nine of 44 samples of flour and 4 of 13 samples of self-raising flour contained persulphate or peroxide.

ROCHELLE SALT.—A vendor was fined £2 for the sale of Rochelle salt which contained about 23 per cent. of sodium bicarbonate, and about 0·6 per cent. of cream of tartar. Four other samples were genuine.

For particulars of other foods and drugs examined during the year see the Quarterly Reports (ANALYST, 1924, **49**, 380, 581; 1925, 67, 185).

J. F. LIVERSEEGE.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

THE QUESTION OF "REASONABLE DILIGENCE."

ON May 19, a Stores society was summoned at Westminster Police Court for having, as retailers, sold tinned peas under a warranty from the vendor, and not having exercised all reasonable diligence to comply with the obligations of the Food and Drugs Act.

Mr. Rotton (for the Westminster City Council) said that the summons was under Sec. 3 of the Act, for selling an article injurious to health, inasmuch as the peas contained the equivalent of 3 grains of crystallised copper sulphate per lb.

Mr. Beck, for the defence, said that it was not contested that the quantity of copper sulphate was injurious, but the Stores had no knowledge of the extent of the addition, and that, by making test analyses over a period of years, they had taken every precaution to ascertain that the article they sold was within the limit allowed.

The manager of the Stores department said that the peas had been bought with a warranty that not more than 2 grains of copper sulphate per lb. had been used, and that all the tins sold had a notice to this effect on the label. The Stores had obtained their peas from the same firm for 25 years, independent analyses of them had been made, and in every instance from 1910 the results of the analysis had been satisfactory.

In cross-examination, witness admitted that the analyses were dropped after 1923.

The Magistrate (Mr. Boyd) said that, in his view, mere belief in the genuineness of an article was not sufficient in itself to say that all diligent reasonable steps had been taken to see that there had not been a sale of an article injurious to health. No one would suggest that a sample of every lot should be analysed, but the evidence was that the test analyses, which had been made periodically over so many years, were dropped. The onus was on the defendants to satisfy the Court, and while it was clear that they had taken a lot of trouble and precaution, it just fell short of "all reasonable diligence" to find out the condition of this particular article sold. He was sure that the society had acted in perfect good faith, and, as they were so careful and had done so much, he did not intend to take a serious view of the case. A very small fine of £2 with 5 guineas costs would be imposed.

CONNOTATION OF THE TERM "SHERRY."

On April 3, a firm of grocers and wine merchants was summoned, under the Merchandise Marks Act, 1887, at Marylebone Police Court, at the instance of the Sherry Shippers' Association, for selling certain bottles of wine to which there was applied a false trade description, to wit, "Corona Pale Sherry."

Mr. Monier Williams, for the prosecution, said that he should call evidence to prove that the description on the label was false in various respects. Firstly, it obviously indicated that the goods in question were manufactured in Spain; and, secondly, that the contents of the bottles were natural wine, and that they were made from Spanish grapes. The bottles each bore a label, upon which were the words: "Corona pale sherry" in prominent type, and underneath, in much smaller and less conspicuous lettering, were the words: "Produced in England from the juice of selected foreign grapes." The prosecution would contend (and that would give rise to the question as to what sherry was) that sherry was a natural wine of a particular type and of a particular style and character, grown in a particular district in Spain, and that the word "sherry" could not properly be applied to any other wine.

When the hearing was resumed on May 7 the Magistrate said that it seemed to him that the defendants did not contend that this wine came from Spain. They only claimed it to be a wine in a sherry category. The question was, therefore, a legal one, and he suggested that the prosecution should call just one witness; and then, if necessary, call rebutting evidence later.

Evidence was then given by the representatives of two firms of sherry importers to the effect that sherry was a "natural" wine coming from the Xeres district of Spain, and that it could not be produced elsewhere. They suggested that all the cheap sherry sold in England before the war came from that particular district, and said that sherry was a wine that was never adulterated.

Sir Henry Curtis Bennett, K.C., for the defence, said that the matter was important, for it concerned not only British sherry as produced in the British isles, but also a wider trade which included Colonial sherry coming from at least four different Colonies. He proposed to call evidence to prove that the term sherry had been applied to wine produced in this country for very nearly 200 years. He would produce a registered label showing that the name was used in 1851, and would show by references to dictionaries and text-books that the title "sherry" had been generally and lawfully applied to wines not coming from Spain, but from other parts of the world, since 1805. All this, he submitted, was overwhelming evidence that "sherry" was a generic term.

Sherry, said Sir Henry, was the description of a class of wine just as much as Australian burgundy was the description of a wine which was a class of burgundy. There had never been a prosecution in connection with Australian burgundy. There had been a prosecution in respect of port—*Hooper* v. *Riddle & Co.*, one of the cases mentioned by the prosecution. That was in respect of Tarragona, and the present case was a very much stronger one for the defence than that. The label in that case read: "Stower's Tarragona Port, blended with wine produced from finest foreign grapes." That label not only described the port, but had the territorial description of Tarragona. The "Corona Pale Sherry" label had no territorial description, but in the strongest terms described exactly what the contents of the bottle were composed of. The wine was produced in England from selected foreign grapes; it was, therefore, an absolutely true description.

Referring to the case of *Holmes* v. *Piper (Ltd.)*, 1913, quoted by the prosecution, he submitted that it did not bind the magistrate in the present case at all. The label there was "Fine British Tarragona Wine." The terms "British" and "Tarragona" were contradictory terms. The label did not read, "Finest British port wine," and, furthermore, it could not be, and was not, argued that Tarragona was a generic term. "Tarragona" had not been used for a 100 years or more to describe a certain type of wine as the term "sherry" had, and the label did not give details as to where the wine was produced and from what it was produced, as the label in the present case did. That case had nothing whatever to do with the facts in this. He submitted that the defendants had committed no offence; that Section 18 did not arise; and that, in fact, the term "sherry" was a generic term which had been in use in this country for a very long period of time.

Mr. C. A. Mitchell gave evidence that he had analysed a sample of wine handed to him by the defendants. It had the following composition:—Proof spirit, 27·7; total solids, 5·99; total acidity, 0·39; volatile acidity, 0·027; sugar, 4·2; mineral matter, 0·31; and phosphoric acid (P_2O_5), 0·05 per cent. This analysis agreed with that of a wine made from pure grape juice. The wine itself agreed in general characteristics with wine that had long been sold as "British sherry."

In cross-examination he said that he was not in any way connected with the manufacturers of the wine in this case. He agreed that the climate and soil of the Xeres district might help to give Spanish sherry its peculiar flavour, but considered that it was much more due to the type of grape. He would apply the term "natural" to a wine made, as this wine was said to have been, from concentrated grape juice, diluted and fermented with a wine yeast.

Evidence was given by representatives of several firms manufacturing British wines, and all agreed that the term "British Sherry" had long been used and was

well recognised in the trade. Such labels were not used with the intention of suggesting that the wine was a Spanish wine, but were meant to describe a wine of the sherry type.

The Magistrate (Mr. Hay Halkett) gave his decision on May 25th. At one time, he said, the case seemed to turn on Sec. 18 of the Merchandise Marks Act, but the more he had listened to the witnesses called for the defence, the more it was borne in upon him that "sherry" was not a generic term at all. Whatever the dictionaries might say, it seemed to him that sherry was a wine derived from a place in Spain, and if "sherry" was asked for, Spanish sherry was expected to be supplied. He found, as a fact, that the word "sherry" meant the produce of a certain part of Spain. If, he said, he held it was a generic term, it would be an erroneous term.* He imposed a penalty of £20, with 75 guineas costs.

Mr. Monier Williams recalled that at a previous hearing Sir Henry Curtis Bennett had stated that there had not been any similar prosecutions in connection with burgundy. He (Mr. Williams) had found that there had been prosecutions for selling Australian burgundy under the title of "burgundy" unqualified.

Sir H. Curtis Bennett gave notice of an appeal "on a question of fact."

* Counsel's opinion on this decision is that it does not preclude the sale of British sherry if the label has the qualifying adjective "British."—EDITOR.

New Zealand.

FIFTY-SEVENTH ANNUAL REPORT OF THE DOMINION LABORATORY.*

THE total number of samples examined for various Government departments during the year 1923 was 4581, of which 2965 were milks, foods and drugs for the Public Health Department.

MILK.—Of the 2325 samples examined, 1763 were taken in and around Wellington, and only 53 of these failed to comply with the regulations; more than half of the condemned samples consisted of good milk that had been allowed to become stale. Eight prosecutions were undertaken for fat deficiency, 7 for added water, and 6 for decided staleness. Since 1916, when the City Inspector was first appointed, the percentage of condemned samples has fallen from 23 to 3. The samples taken in the country districts showed a marked improvement in cleanliness.

BUTTER.—Of the 121 samples examined, 13 showed a decided excess of water, the highest amount found being 21·9 per cent.

PRESERVATIVES.—Preservatives were detected in tomato sauce, beer and whitebait, and artificial colouring matters in jams, cordials and other foods in which their use is contrary to regulations.

The report also contains a detailed report of analyses of (1) coal and carbonaceous minerals; (2) limestones, marls and clays; (3) rocks, minerals and ores; (4) waters; and (5) miscellaneous samples, including sands for glass-making, natural gas, and oil from coal.

Numerous examinations were also made for the Customs, Post and Telegraph, Railways, Public Works, and other departments. There are two appendices, one giving an account of the administration of the Explosive and Dangerous Goods Act, and the other a detailed classification of New Zealand clays.

* By J. S. Maclaurin, D.Sc., Dominion Analyst. Published 1925. Wellington, N.Z.

Medical Research Council.

FOOD POISONING—A STUDY OF 100 RECENT OUTBREAKS.*

THE cause of an outbreak of food poisoning may be:—

I. *Infection of the food with members of the Salmonella (or Gaertner) group of bacilli.*—This is the most common cause of all, and a special investigation of the group has been made in this connection (see Medical Research Council Report by the same authors, ANALYST, 1925, **50**, 239–241). These bacilli are particularly associated with the more severe and definite outbreaks, and the more fully outbreaks are investigated, the larger the proportion of cases referable to this cause. A living Salmonella strain was isolated in 20 outbreaks of the present series, and in 14 cases it was *B. aertrycke*, but in the absence of the living bacilli the undestroyed toxins are an important source of poisoning, particularly in the case of canned food. In order to prove the complicity of the toxins, evidence should be sought along the following three lines:—(*a*) Demonstration of the toxicity of the food by feeding animals with it or by injecting extracts of the suspected food into animals. In this connection it must be borne in mind that laboratory animals have a comparatively low susceptibility to infection by the Salmonella group. (*b*) Demonstration of the production of specific agglutinins in the blood of patients suffering from food poisoning. This method, although most valuable in the case of the living organisms, implies that in toxin outbreaks the heated bacilli, introduced by way of the alimentary canal, can cause development of specific agglutinins, and when the type of disease is acute gastro-intestinal irritation with quick recovery after elimination of the irritant, very little production of agglutinins is usually to be anticipated. (*c*) Demonstration of the production of specific agglutinins in animals through the injection into them of suitable incriminated food. This method assumes the same premise as (*b*), but has the advantage over (*b*) of direct introduction into the animal system.

By the use of one or more of these methods some positive evidence for infection by Salmonella toxins was obtained in 16 cases, 12 of which were from canned foods. Another 17 cases (all canned foods) are ascribed to the same cause without bacteriological proof. It may be noted that canned foods are prepared under conditions that will kill living bacilli but not of necessity destroy the toxins.

II. *Organisms of dysentery or closely allied type.*—Such organisms have been definitely found in four cases associated with the production of symptoms indistinguishable clinically from Salmonella group outbreaks, and they may be the cause of some of the unidentified outbreaks.

III. *Botulism group.*—Three cases (one the Loch Maree outbreak, ANALYST, 1923, **48**, 118–120), are referred to this cause. Two of these cannot definitely be accepted in the absence of corroborative bacteriological evidence. This is an extremely rare form of food poisoning in this country, but one in which the toxin is extremely potent, it being an almost universal rule that every person partaking of the food is affected.

IV. *Cheese.*—Eight outbreaks were attributable to this food. In one, Salmonella toxin was present, but in the others the causes were obscure. Probably in none of these cases was a living organism the toxic agent. Tyrotoxicon was looked for in every case but one, but was not found, and, indeed, the evidence of its being the causative agent of earlier outbreaks appears to be doubtful; nor were poisonous

* By W. G. Savage, M.D., B.Sc., and P. Bruce White, B.Sc. Pp. 112. Obtainable at Adastral House, Kingsway, W.C.2. Price 2s. 6d. net.

metals present. The toxic body acts for the most part as a gastro-intestinal irritant, but can be absorbed (altered or unaltered), giving rise to more remote symptoms, and may be assumed to be the product of bacterial activity. The evidence appears to be against toxic degradation products of non-bacterial origin such as poisonous amines, which, even if proved present, would have to be in sufficient amount to overcome the protective defensive mechanism of normal deaminisation and oxidation in the liver. It is probable that, whilst the toxic agent is not the same in all cases of cheese poisoning, the commonest cause is undestroyed toxin of some specific bacillus, belonging, in some cases at least, to the Salmonella group.

V. *Chemical origin (i.e. due to substances of known molecular grouping).*—Two outbreaks (one due to zinc and one to belladonna) are noted. No case of tin poisoning is recorded, nor do there appear to be any reports of such cases in recent years.

VI. *Undetermined causes.*—Twenty-four cases were from undetermined causes, but in many of these there was lack of material for examination and inadequate investigation, and only 7 outbreaks are of quite obscure origin.

The causes of the 100 outbreaks may be summarised as follows: Probably not true food poisoning outbreaks, 3; those due to members of the Salmonella group, 66; to members of the dysentery group, 4; to *B. botulinus*, 1; of definite chemical origin, 2; cheese-poisoning, 8; mild evanescent outbreaks, 9; those of undetected bacterial origin, 7.

Paths of infection of Salmonella group bacilli.—Whilst in many cases the toxic agent is determinable, and is most usually of the Salmonella group, the means by which it has gained access to the food is usually obscure.

(*a*) A habitat outside the animal body as a reservoir of Salmonella bacilli (such as manure). No data at present support such a hypothesis.

(*b*) Infection from a human case. Although claims are made for infection from a human source for Salmonella bacilli, it is extremely rarely that human carriers are found responsible for outbreaks.

(*c*) Infection from an animal source, due (i) either to the meat or milk of the infected animal being used as human food, or (ii) to infection of sound food from bacilli derived from an animal suffering from, or a carrier of, a Salmonella infection. One or other of these causes cannot be excluded in some 14 cases, exclusive of canned food.

A consideration of all available facts strongly suggests that, in the majority of cases, the food was originally bacterially sound and becomes contaminated from outside. Specific infection may be sometimes carried by rats or mice, and a virulent *B. enteritidis* was isolated from rats caught in slaughter houses. No positive evidence for infection by flies has been adduced, apart from the seasonal prevalences being rather similar.

Prevention of Outbreaks of food poisoning.—This will largely be brought about by control and eradication of the Salmonella and, possibly, the dysentery types of poisoning, and information as to the reservoirs of these bacilli is required. In the first instance, it is necessary that all outbreaks of food poisoning should come under official cognisance and be adequately investigated by experienced bacteriologists. Secondly, made-up meat and milk food products need special supervision and control by licensing to ensure suitable premises, cleanly procedure, no proximity to undesirable trades, and particularly protection of the products during cooling processes. In addition, in the case of canned foods much might be done by a properly developed system of coding, so that consignments might be traced. D. G. H.

Ministry of Health.

DRIED MILK*

STATUTORY RULES AND ORDERS, 1923, No. 1323.

PUBLIC HEALTH, ENGLAND.

The Public Health (Dried Milk) Regulations, 1923, dated November 5, 1923, made by the Minister of Health.

68576.

The Minister of Health in the exercise of the powers conferred upon him by the Public Health Act, 1875, (**a**) the Public Health (London) Act, 1891, (**b**) the Public Health Act, 1896, (**c**) the Public Health (Regulations as to Food) Act, 1907, (**d**) and by Section 8 of the Milk and Dairies (Amendment) Act, 1922, (**e**) and of every other power enabling him in that behalf, hereby makes the following Regulations, that is to say:—

1. These Regulations may be cited as the Public Health (Dried Milk) Regulations, 1923, and shall come into operation on the 1st day of May, 1924.

2.—(1) In these Regulations unless the context otherwise requires:—

"The Minister" means the Minister of Health;

"Local Authority" means any Local Authority authorised to appoint an analyst for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907, and "public analyst" means an analyst so appointed;

"Dried Milk" means milk, partly skimmed milk, or skimmed milk, which has been concentrated to the form of powder or solid by the removal of water;

"Skimmed Milk" includes separated or machine-skimmed milk;

"Gross weight" of a tin or other receptacle means the weight of the tin or other receptacle and of its contents;

"Label" includes a mark.

Percentages shall be calculated by weight.

(2) These Regulations apply to dried milk to which no other substance has been added and to the dried milk contained in any powder or solid of which not less than 70 per cent. consists of dried milk.

(3) The Interpretation Act, 1889, (**f**) applies to the interpretation of these Regulations as it applies to the interpretation of an Act of Parliament.

3. The Local Authority shall enforce and execute these Regulations, and for this purpose shall make such enquiries and take such other steps as may seem to them to be necessary for securing the due observance of the Regulations in their district.

4. No person shall sell or expose for sale or deposit in any place for the purposes of sale, or despatch or deliver to any purchaser, broker, or agent any dried milk intended for human consumption unless the dried milk—

(1) is contained in a tin or other receptacle which is labelled in the manner prescribed in the Schedule to these Regulations; and

(2) contains not less than the following percentages of milk fat, namely,—

In the case of milk described as dried full cream milk not less than 26 per cent.;

In the case of milk described as dried three-quarter cream milk not less than 20 per cent.;

(**a**) 38–9 V. c. 55. (**b**) 54–5 V. c. 76. (**c**) 59–60 V. c. 20.
(**d**) 7 E. 7. c. 32. (**e**) 12–3 G. 5. c. 54. (**f**) 52–3 V. c. 63.

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* In view of the frequent references to these Regulations of 1923, the Publication Committee has decided to print them in full.—EDITOR.

In the case of milk described as dried half cream milk not less than 14 per cent.; and

In the case of milk described as dried quarter-cream milk not less than 8 per cent.: Provided that—

(*a*) The provisions of this Article shall not apply in any case where the dried milk is contained in a tin or other receptacle whose gross weight exceeds ten pounds; and

(*b*) Where dried milk is sold by weight and is not placed in the tin or other receptacle in which it is delivered to the purchaser until immediately before such delivery, the provisions of Rules 1 to 4 of the Schedule shall be deemed to be satisfied if the matter therein required to appear on a label affixed to the tin or other receptacle is printed on a separate label or notice delivered to the purchaser, and the last sentence of the declaration required by Rule 1 of the Schedule may be varied so as to relate to one pound or to any other specified weight of the article sold instead of the contents of the actual tin or other receptacle.

5.—(1) The Medical Officer of Health, and any person authorised by him or by the Local Authority in writing, may procure any sample of dried milk, and where an analysis is required for the purposes of these Regulations shall submit the sample to the public analyst and shall forthwith notify to the seller or his agent selling the dried milk his intention to have the same analysed by the public analyst.

(2) Except where the sample is procured for the purpose of testing the quantity of milk, partly skimmed milk, or skimmed milk of which the contents of a tin or other receptacle are the equivalent, the provisions of Section 14 of the Sale of Food and Drugs Act, 1875, (**a**) as amended by Section 13 of the Sale of Food and Drugs Act, 1899, (**b**) relating to the division of the sample into three parts and the separation, marking and disposal of such parts shall apply.

(3) Where the sample is procured for the purpose of testing the quantity of milk, partly skimmed milk or skimmed milk of which the contents of a tin or other receptacle are the equivalent, the person by whom the sample is procured shall as soon as may be after the net weight of the contents has been ascertained deliver a part of the sample to the seller or his agent.

6. Any officer authorised by the Minister and any officer of the Local Authority duly authorised by the Authority in writing shall have power to enter at all reasonable times any premises where dried milk is prepared, packed, labelled or stored and to inspect any process carried on therein and to take samples of any article used or capable of being used in the preparation of dried milk and of any labels designed to be used for affixing to tins or other receptacles of dried milk.

7. Where the Local Authority on a report to them from the public analyst or otherwise are of opinion that a consignment of dried milk deposited within their district and intended for sale for human consumption does not comply with the requirements of these Regulations they shall endeavour to ascertain where it was manufactured and labelled. If it is ascertained that such dried milk was manufactured or labelled at a place in England or Wales, the Local Authority shall communicate the facts which they have ascertained to the Local Authority for the district in which such place is situated. If it is ascertained that such dried milk was manufactured or labelled at a place not in England or Wales, the Local Authority shall communicate the facts to the Minister.

8. In any proceedings under these Regulations the certificate of the public analyst of the result of the chemical examination of a sample shall be sufficient evidence of the facts therein stated unless the defendant requires that the analyst be called as a witness.

9. A person, in relation to anything within his knowledge, shall truly answer all such questions put to him by the authorities authorised to enforce and execute these Regulations or their officers, or by an officer authorised by the Minister, as may be necessary for the purposes of these Regulations, and shall produce for inspection all such books as the authority or officer may reasonably require for the purposes of ascertaining the persons or places from which dried milk has been obtained and to whom and where it has been consigned or otherwise.

The Schedule.

RULES WITH RESPECT TO THE LABELLING OF DRIED MILK.

1.—(1) Every tin or other receptacle containing dried milk (other than dried milk to which sugar or some other substance has been added) shall bear a label upon which is printed such one

(**a**) 38–9 V. c. 63. (**b**) 62–3 V. c. 51.

of the following declarations as may be applicable or such other declaration substantially to the like effect as may be allowed by the Minister:—

(i) In the case of full cream milk, that is to say, dried milk containing not less than 26 per cent. of milk fat:—

DRIED FULL CREAM MILK.

THIS TIN CONTAINS THE EQUIVALENT OF
(a) PINTS OF MILK.

(ii) In the case of partly skimmed milk, that is to say, dried milk containing not less than 8 per cent. but less than 26 per cent. of milk fat:—

DRIED PARTLY SKIMMED MILK.
((b) CREAM.)

SHOULD NOT BE USED FOR BABIES EXCEPT UNDER MEDICAL ADVICE.

THIS TIN CONTAINS THE EQUIVALENT OF
(a) PINTS OF (b) CREAM MILK.

(iii) In the case of skimmed milk, that is to say, dried milk containing less than 8 per cent. of milk fat:—

DRIED MACHINE-SKIMMED MILK
[or DRIED SKIMMED MILK].

UNFIT FOR BABIES.

THIS TIN CONTAINS THE EQUIVALENT OF
(a) PINTS OF SKIMMED MILK.

(2) The label on any tin or other receptacle containing dried milk to which sugar or some other substance has been added shall be in the appropriate form presceibed in sub-division (1) hereof, with the following modifications:—

(i) There shall be added to the heading the word "Sweetened" if the only substance added to the milk is sugar; the word "Modified" if the only substance added is a constituent of milk, and the word "Compounded" in every other case; and

(ii) The words "with (*c*) added" shall be added to the last sentence in each case, words being inserted at (*c*) to specify the substance or substances added.

(3) The declaration shall be completed as follows:—

(i) There shall be inserted at (a) the appropriate number in words and figures, *e.g.* "one-and-a-half (1½)," any fraction being expressed as eighths, quarters or a half.

(ii) There shall be inserted at (b) the word "Three-quarter" if the percentage of milk fat is not less than 20; "Half" if such percentage is less than 20 but not less than 14; and "Quarter" if such percentage is less than 14 but not less than 8.

(4) For the purposes of this Rule the terms "Milk," "Three-quarter cream milk," "Half cream milk," and "Quarter cream milk" mean milk containing not less than the following percentages of milk fat and milk solids, that is to say:—

	Milk Fat.	Milk Solids (including fat).
Milk	3·6	12·4
Three-quarter cream milk	2·7	11·6
Half cream milk	1·8	10·8
Quarter cream milk	·9	9·9

and "Skimmed milk" means milk which contains not less than 9 per cent. of milk solids other than milk fat.

2. The prescribed declaration shall be printed in dark block type upon a light-coloured ground within a surrounding line, and no other matter shall be printed within such surrounding line.

The type to be used for the heading and the words "unfit for babies" in the declarations set out above shall be not less than one-quarter of an inch in height (or, if the gross weight of the tin or other receptacle does not exceed twelve ounces, one-eighth of an inch in height), and that to be used for the remainder of the said declarations shall be not less than one-eighth of an inch in height (or, if the gross weight of the tin or other receptacle does not exceed twelve ounces, one-sixteenth of an inch in height).

3. The label shall in addition bear the name and address of the manufacturer of the dried milk or of the dealer or merchant in the United Kingdom for whom it is manufactured.*

4. The label shall be securely affixed to the tin or other receptacle so as to be clearly visible. If there is attached to the tin or other receptacle a label bearing the name, trade mark, or design representing the brand of the dried milk, the prescribed declaration shall be printed as part of such label.

5. There shall not be placed on any tin or other receptacle containing dried milk—

(*a*) any comment on, explanation of, or reference to either the statement of equivalence contained in the prescribed declaration or the words "partly skimmed," "machine-skimmed," "skimmed," or "unfit for babies"; or

(*b*) any instructions as to dilution, unless either—

(i) the fluid produced in accordance with such instructions would contain not less milk fat and not less milk solids than milk, partly skimmed milk, or skimmed milk as defined in Rule 1 of this Schedule, as the case may require; or

(ii) such instructions clearly specify that the fluid so produced is not of equivalent composition to milk, partly skimmed milk, or skimmed milk, as the case may be.

6. Wherever the word "Milk" appears on the label of a tin or other receptacle of dried partly skimmed or skimmed milk as the description or part of the description of the contents, it shall be immediately preceded or followed by the words "Partly skimmed," "Machine-skimmed," or "Skimmed," as the case may require.

Given under the Official Seal of the Minister of Health this Fifth day of November, in the year One thousand nine hundred and twenty-three.

(L.S.) A. B. MACLACHLAN,
Assistant Secretary, Ministry of Health.

Note.—The Public Health Act, 1896, provides by sub-section (3) of Section 1 that if any person wilfully neglects or refuses to obey or carry out, or obstructs the execution of any regulations made under any of the enactments mentioned in that Act, he shall be liable to a penalty not exceeding £100, and, in the case of a continuing offence, to a further penalty not exceeding £50 for every day during which the offence continues.

The power of making regulations under the Public Health Act, 1896, and the enactments mentioned in that Act, is enlarged by the Public Health (Regulations as to Food) Act, 1907, as amended by the Milk and Dairies (Amendment) Act, 1922.

* In the case of imported dried milk, the provisions of Section 16 of the Merchandise Marks Act, 1887 (50–1 V. c. 28), must also be complied with if the label bears the name or trade mark of a dealer or merchant in the United Kingdom.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Ratio of Gelatin-Forming Substance to Total Protein in Sausages. O. Luning and S. Gerö. (*Zeitsch. Unters. Nahr. Genussm.*, 1925, **49**, 179–187.)—While the better qualities of sausage are made entirely from flesh or muscle tissue, the inferior qualities contain considerable amounts of sinewy matter, such as skin and waste parts. The proteins of muscle tissue contain much albumin and amino acids, whereas the sinewy tissue yields much gelatin and but little tryptophane or tyrosine. Consequently, separation of the proteins and the evaluation of the proportion of gelatigenous nitrogen to total nitrogen afford an indication of the class of material used in the preparation of the sausage; the greater the gelatin content the less the value. The separation is effected by Striegel's method (*Chem. Zeit.*, 1917, **41**, 313). From 2·5 to 5 grms. of the minced material are boiled with 200 c.c. of water for 5 hours under a reflux condenser; 1 grm. of tartaric acid is added, and the boiling continued for exactly 30 minutes, after which the solution is immediately cooled and almost neutralised with sodium hydroxide solution, 20 c.c. of saturated zinc sulphate solution are added, and the mixture is made up to 500 c.c. and filtered. The nitrogen is determined on 100 c.c. of the clear filtrate. A few figures from the tables of results, showing the percentage of gelatigenous to total nitrogen, are as follows:—Beef (muscle), 33–45; beef (sinews), 88; pork, 26–29; liver, 20–25; and lung, 35–39. H. E. C.

The Problem of Noors Honey. C. F. Juritz. (*Chem. News*, 1925, **130**, 310–312.)—The honey from certain parts of the South-Eastern districts of the Union of South Africa has an unpleasant taste and produces a burning sensation in the throat, even setting up poisoning occasionally. The particular type affected is known as "Noors" or "Noorsdorn," and is derived from various species of *Euphorbia*. Chemical analysis of the honey reveals nothing unusual, except that the non-sugar matter is a little high, *e.g.*:—Water, 15·08; invert sugar, 70·97; sucrose, 3·61; ash, 0·11; non-sugars, 10·34 per cent.; acidity, 100 grm. = 3·65 c.c. *N*. Although no alkaloid could be found, extraction with ether yields about 0·1 per cent. of a yellow oil, to which the burning taste is due. No simple method could be devised for eliminating this active substance on the large scale. H. E. C.

Citrus Pectin. H. D. Poore. (*U. S. Dept. of Agriculture*, 1925, *Bull.* No. 1323.)—In order to extract the pectin from citrus by-products it is necessary either to remove the oils and bitter principles, or to precipitate the pectin from solution. Although dialysis of pectin extracts in an osmogene containing collodion-impregnated cloth membranes removed most of the bitter principles, the method is too slow to be practicable, and the best method of extraction is to heat the

finely ground raw material with acidified water, and to clarify the extract with kieselguhr. The bitter principles may be removed from powdered pectin by means of alcohol, and a process has been devised for removing them from pectin paste by continuous washing with a minimum quantity of denatured alcohol. The composition of apple, lemon and orange pectins, calculated to alcohol precipitate (pure pectin) base affords little help in distinguishing them. It was as follows (in the order named). Acidity (grms. of NaOH per 100 grms.), 3·99, 4·29, 1·22; sodium hydroxide required for cold hydrolysis, 14·57, 15·10, 14·75 per cent.; pectic acid, 66·02, 69·81, 67·02 per cent.; methoxy number, 11·27, 11·41, 10·82 per cent.; araban, 41·02, 43·51, 42·76 per cent.; galactan, 67·78, 65·54, 68·59 per cent.; specific rotation, +217·1, +229·7, +214. Good jellies were produced with as little as 0·2 and 0·25 per cent. of citrus pectin in the finished product, in addition to not less than 0·045 per cent. of citric acid and between 50 and 65 per cent. of sucrose, although if the proportion of pectin be increased to 0·93 per cent., the proportion of sugar necessary for a good jelly may be decreased to between 41 and 52 per cent. D. G. H.

Quercitrin in Tea Leaves. J. J. B. Deuss. (*Rec. Trav. Chim., Pays-Bas.*, 1923, **42**, 623; *J. Pharm. Chim.*, 1925, **117**, 430–431.)—An aqueous extract of tea, heated with 1 to 2 per cent. of hydrochloric acid for 2 hours under a reflux condenser in the presence of a current of carbon dioxide, deposits a brown precipitate which may be filtered off, washed, and dried, and the quercetin extracted with ether and subsequently crystallised from water. Since free quercetin is not present in tea leaves, the author infers that it must have been formed by hydrolysis of quercitrin, but the *J. Pharm. Chim.* points out that rutin is more widely distributed in nature than quercitrin, and also yields quercetin on hydrolysis.

D. G. H.

Nickel and Cobalt in Vegetable Products. G. Bertrand and M. Mokragnatz. (*Bull. Soc. Chim.*, 1925, **37**, 554–558; *cf.* ANALYST, 1925, 84.)—Nickel and cobalt were found in all samples of earths examined from France and other parts of Europe; also in a large proportion of vegetable substances, chiefly the parts used for food. The proportion of nickel, which was separated as the dimethylglyoxime compound, varied from 2·0 mgrms. per kilo of fresh material in ripe peas and 0·175 mgrm. in carrot leaves down to 0·015 mgrm. in decorticated polished rice. The cobalt was present in much smaller proportions, and the precipitate of potassium cobaltinitrite was usually too small to weigh and had to be compared with precipitates from solutions of known strength. The highest proportion found was in buckwheat, 0·3 mgrm., and the lowest 0·005 mgrm. per kilo of fresh material in spinach leaf, tomato, apricot, pericarp grape, haricot pod and polished rice, but its presence in oats was doubtful. D. G. H.

Detection of Organic Cyanides in "Alliols." L. Desvergnes. (*Ann. Chim. analyt.*, 1925, **7**, 129–130.)—Alliols are impure sulphur compounds resulting from the first distillation of light tar oils; they contain about 0·1 per cent. of sulphur

compounds and 0·2 to 0·33 per cent. of nitriles, and are used medicinally. Nitriles, carbylamines and isocyanic esters may all be detected by the formation of amine under suitable conditions, and the isolation of the amine as the picrate in the form of yellow crystals melting at 215–220° C. by the addition of picric acid. The following mixtures are distilled: For *nitriles*, 50 c.c. of the alliol, 5 grms. of dry sodium parings and 50 c.c. of 96 per cent. alcohol; for *carbylamines*, 50 c.c. of the alliol, 25 c.c. of concentrated hydrochloric acid and 25 c.c. of water; for *isocyanic esters*, 50 c.c. of the alliol and 25 c.c. of alcoholic potassium hydroxide solution. *Sulphocyanic esters* may be detected by warming 50 c.c. of the alliol with 5 grms. of zinc powder and 50 c.c. of 10 per cent. sulphuric acid, and collecting the hydrocyanic acid in dilute sodium hydroxide solution; *isothiocyanic esters* by warming 50 c.c. of the alliol with 25 c.c. of concentrated hydrochloric acid and 25 c.c. of water, and collecting the carbon dioxide in baryta water. D. G. H.

Properties of Colloidal Kaolin. W. J. Pope and R. T. M. Haines. (*Lancet*, 1925, **208**, 1123–1124.)—Only the most finely divided kaolin should be used for medicinal purposes. A suspension of 1 grm. of colloidal kaolin, as used for internal administration, (British Colloids, Ltd.) in 100 c.c. of water showed, when examined with the ultra-microscope, numerous particles in vigorous Brownian movement. The average of 52 counts gave the number of particles in 1 grm. as $0{\cdot}9 \times 10^{14}$. When shaken with water the kaolin remained in suspension for more than a week; immediate flocculation was caused by the addition of 0·3 per cent. (by vol.) of strong hydrochloric acid, and the addition of 1·3 per cent. by weight of dissolved sodium chloride effected precipitation within 10 minutes. Moderate amounts of sodium hydroxide did not cause flocculation. The addition of colloidal kaolin to negative colloidal solutions, such as those of arsenic trisulphide, antimony trisulphide, and red colloidal gold, caused no change. But its addition to solutions of positive colloids (*e.g.* colloidal ferric hydroxide solutions) produced immediate flocculation; hence colloidal kaolin is a negative colloid. It removed 5 per cent. of the iodine from a potassium iodide solution containing 1·45 grms. of free iodine. Colloidal kaolin removes a large proportion of basic dyestuffs from aqueous solutions, but does not adsorb acid dyestuffs. Amphoteric dyestuffs behave differently towards colloidal kaolin in acid and alkaline solutions. Basic dyestuffs are distributed between the solution and the kaolin in proportions that can be determined. This distinction is interesting in view of the results obtained by Walker (*Lancet*, 1921, ii., 273) in connection with the action of kaolin on the cholera vibrio and the toxin prepared from the bacilli. The second part of this paper (by J. W. H. Eyre) deals with the absorption of diphtheria toxin by kaolin.

Biochemical, Bacteriological, etc.

Method for the Determination of Hydrogen Sulphide in Proteinaceous Food Products. L. H. Almy. (*J. Amer. Chem. Soc.*, 1925, **47**, 1381–1390.)—As a means of obtaining information concerning the decomposition of food materials,

a method is described for determining small proportions of sulphide sulphur in biological materials. The procedure consists in passing a current of carbon dioxide through an acidified aqueous suspension of the substance, absorbing the liberated hydrogen sulphide in dilute zinc acetate solution, and determining the sulphide content of the absorbing solution by comparison with standards of the "methylene blue" colour produced by interaction of the hydrogen sulphide with *p*-amino-dimethylaniline, hydrochloric acid and ferric chloride, in accordance with Lauth's reaction. Application of the method to beef, pork, and fish kept at temperatures favourable to decomposition indicates the progressive formation of hydrogen sulphide in these materials. T. H. P.

Chemistry of Jaffe's Reaction for Creatinine. II. Effect of Substitution in the Creatinine Molecule and a Possible Formula for the Red Tautomeride. I. Greenwald. (*J. Amer. Chem. Soc.*, 1925, **47**, 1443–1448.)—The red colour obtained when a creatinine solution is treated with picric acid and sodium hydroxide (ANALYST, 1924, **49**, 346) is due to the formation of a red tautomeride of creatinine picrate. Investigation of the behaviour of derivatives of creatinine with picric acid and sodium hydroxide, and of creatinine with various compounds allied to picric acid, indicates that the production of this red tautomeride depends jointly on the formation of a salt, a keto-enolic change within the creatinine molecule, and a change in the picric acid molecule involving the hydrogens in the meta positions, and, probably, all the nitro groups. A provisional formula is given for the tautomeride. T. H. P.

Mechanism of the Interaction of Formaldehyde and Serum Proteins. R. R. Henley. (*J. Agric. Res.*, 1924, **29**, 471–482.)—By reaction velocity experiments on horse serum of known composition it is shown that the formation of euglobulins from serum proteins and their "gelatification" by formaldehyde is a reaction of the second order, proceeding with a definite velocity and having a temperature coefficient of about 3. The process therefore, is slightly different from gelatification by heat, which is a first order reaction. When the proteins are acted upon by formaldehyde there is at first an increase in titratable acidity, but this is followed by a decrease which corresponds to a second stage in the reaction; this is probably a polymerisation involving acid groups and unattached basic groups in the protein molecule. H. E. C.

Enzymatic Synthesis of Protein. IV. Effect of Concentration on Peptic Synthesis. H. Borsook and H. Wasteneys. (*J. Biol. Chem.*, 1925, **63**, 563–574.)—The authors describe their experiments on the effect of concentration of material in solution on peptic synthesis, and give tables and charts to show results. In a solution of the products of peptic hydrolysis of albumin the extent of synthesis with pepsin is in simple inverse proportionality to the dilution. Series of experiments were carried out with constant enzyme concentrations and with varying enzyme concentrations. There is a falling off from the straight line relationship in very concentrated solutions, and in sufficiently high concentrations

the amount of synthesis is actually less than in more dilute solutions. With enzyme concentrations varying between 4·0 and 0·05 per cent., synthesis fails at 38° C. in a solution of products which corresponds to approximately 8 per cent. of protein. No evidence was obtained for the possibility that enzyme disappears in the course of synthesis. Additions of synthesised protein and native protein to a solution of digest and pepsin inhibit the subsequent synthesis to an extent directly proportional to the amount added. Similarly, peptic hydrolysis does not proceed to completion in concentrated protein solutions. P. H. P.

Enzymatic Synthesis of Protein. V. Note on the Synthesising Action of Trypsin. H. Wasteneys and H. Borsook. (*J. Biol. Chem.*, 1925, **63**, 575–578.)—It is shown that the optimum hydrogen ion concentration for tryptic synthesis of protein in a peptic digest of egg albumin is in the neighbourhood of P_H=5·7. From the location of the optimum (shown by a graph) the synthesising enzyme is clearly not pepsin. As had been observed previously by Henriques and Gjaldbak (*Z. physiol. Chem.*, 1912, **81**, 439), in neutral and alkaline reactions hydrolysis also occurs simultaneously with the synthesis. Whether both these reactions are promoted by the same enzyme cannot be decided by these experiments. P. H. P.

Whole and Skimmed Milk Powders as Food. Observations on a New Vitamin for Reproduction. L. T. Anderegg and V. E. Nelson. (*Ind. Eng. Chem.*, 1925, **17**, 451–455.)—Results of experiments with rats showed that whole milk powder, supplemented by iron salts (iron citrate) and carbohydrates or iron salts alone, furnishes everything necessary for growing, reproduction and rearing of young, and that the amount of iron salt added to whole milk powder has a pronounced effect on reproduction. When the animals were fed on skimmed milk powder there was no reproduction, but the addition of yeast or wheat embryo to the skimmed milk powder so changed its food value that young were born and reared by the first generation; the second generation fed on this food grew normally, but did not reproduce. Yeast and wheat embryo must therefore furnish the same supplementing substance. W. P. S.

The Action of Trypsin on Insulin. D. A. Scott. (*J. Biol. Chem.*, 1925, **63**, 641–651.)—It was thought that a study of the action of trypsin on insulin might help to explain the discrepancies in the yields of insulin from the pancreas. At certain acidities insulin is immediately inactivated by trypsin. By suitably adjusting the temperature and acidity, or by the use of alcohol or benzoates, the latent potency may be partly reclaimed. When insulin and trypsin are incubated together (P_H=7·5, 40° C.) for about 12 hours no insulin can be recovered by the procedures referred to above. Trypsin in amounts which inactivate insulin *in vitro* does not inhibit insulin when the substances are injected separately. This is contrary to the results of Epstein and Rosenthal, who found that trypsin inactivates insulin *in vivo* as well as *in vitro*. Experiments, which are described, were carried out on rabbits. P. H. P.

Saponins. I. The Sapogenin obtained from Soapnuts. W. A. Jacobs. (*J. Biol. Chem.*, 1925, **63**, 621–629.)—With but few exceptions, the saponins are glucosides, and the sapogenins are largely responsible for the physical and perhaps biological peculiarities of the saponins. The alcoholic extract of the shells of soapnuts (thought to be the fruit of *Sapindus saponaria* L.) on hydrolysis readily gave an excellent yield of an apparently homogeneous crystalline substance which was easily purified. Several lots of different origin yielded the same sapogenin. This sapogenin has been found to have the formula $C_{31}N_{50}O_4$, and is identical with hederagenin, the dihydroxy acid prepared by van der Haar from *Hedera helix*. Details are given of the preparations of *Sapindus* sapogenin (hederagenin) and some of its derivatives. In certain instances the results of the author differ from those of van der Haar. The fact that the same sapogenin occurs in such widely separated sources as *Sapindus saponaria* and *Hedera helix* is of interest, and other instances of a similar nature may be found. The sapogenin hederagenin appears to differ from the sapogenin obtained from *Sapindus mukorossi* by Winterstein and Blau. P. H. P.

Toxicological and Forensic.

Determination of Lead in Petrol. T. von Fellenberg. (*Zeitsch. Unters. Nahr. Genussm.*, 1925, **49**, 173–178.)—Small quantities of tetraethyl lead are sometimes added to motor spirit (*cf.* ANALYST, 1925, 84). The commercial lead compound boils at about 200° C., with partial decomposition, and may contain about 55 instead of the theoretical 64 per cent. of lead. Although it may be detected qualitatively by burning the petrol in a small jet and absorbing the vapours, the method involves loss and cannot be applied quantitatively. Accurate results are obtainable by wet combustion with sulphuric and nitric acids. To 10 c.c. of the petrol in a Kjeldahl flask are added 0·5 c.c. of sulphuric and 2 c.c. of nitric acid. The mixture is well shaken and warmed over a small flame; after about 10 minutes it is again well shaken, then cooled, and the hydrocarbon is poured off as far as possible. The lead in the residue is determined as lead sulphate in the usual way. The oxidation may also be effected by means of sulphuric acid and permanganate, but this is less satisfactory than the use of nitric acid. Reasonably accurate results are obtained with mixtures containing 0·05 per cent. and upwards of tetraethyl lead. H. E. C.

Water Analysis.

Action of Water on Copper. H. Henstock. (*J. Soc. Chem. Ind.*, 1925, **44**, 219T.)—Water distilled from a tin-lined copper apparatus gave no reaction for copper with ferrocyanide, nor after boiling the water for 100 hours with clean or scaled copper, but after adding the slighest trace of sodium or magnesium chloride the presence of copper was detectable after 1 or 2 hours' boiling with copper.

A series of experiments was carried out by boiling 100 grms. of drawn copper wire with very dilute solutions of various salts at atmospheric pressure.

A solution of	0·0024	pt.	NaCl	per	100,000	showed the presence of copper in				1	hour.
,, ,,	0·0020	,,	$MgCl_2$	,,	,,	,,	,,	,,	,,	2	hours.
,, ,,	0·0031	,,	KCl	,,	,,	,,	,,	,,	,,	2	,,
,, ,,	0·0023	,,	$CaCl_2$	,,	,,	no copper after 48 hours					
,, ,,	0·0020	,,	$MgSO_4$	,,	,,	,,	,,	,,	,,		
,, ,,	0·0023	,,	Na_2SO_4	,,	,,	,,	,,	,,	,,		
,, ,,	0·0022	,,	$CaSO_4$	,,	,,	,,	,,	,,	,,		
,, ,,	0·0020	,,	$MgH_2(CO_3)_2$	,,	,,	,,	,,	,,	,,		
,, ,,	0·0033	,,	Na_2CO_3	,,	,,	,,	,,	,,	,,		
,, ,,	0·0022	,,	$CaH_2(CO_3)_2$	,,	,,	,,	,,	,,	,,		

The same quantities of sodium and magnesium chlorides as above, mixed with equal quantities of calcium bicarbonate or hydroxide, showed no copper after 48 hours' boiling. These results agree with those of Skinner (*J. Amer. Chem. Soc.*, 1906, 361) and Ost (*Chem. Zeit.*, 1902, 845), which were carried out under pressure.

Of two natural soft waters, each containing organic matter, one contained no sodium or magnesium chlorides, and the other contained 0·2 part per 100,000 and no calcium bicarbonate. The former, on heating, had no action on certain copper apparatus, but the latter had considerable action. A Shropshire water used in a private residence had no action on copper pipes while cold, but when heated, it attacked them to such an extent that on the addition of soap the water became green and copper was found in the curds. The composition of the dissolved matter was:—Organic matter, 9·8 per cent.; inorganic matter, 4·2 per cent. (of which 4·1 per cent. was calcium sulphate); sodium and magnesium chloride, 0·1 per cent. The action on the pipes was prevented by the addition of a small quantity of slaked lime. Calcium bicarbonate in waters also prevents the action of any sodium or magnesium chlorides present. In softening waters by the permutite process for subsequent use in copper apparatus all trace of sodium or magnesium chlorides should be removed. (*Cf.* ANALYST, 1925, 248.) R. F. I.

Effect of Air Pollution on Water. B. A. Adams. (*The Medical Officer*, Mar. 21, and May 2, 1925; *Lancet*, 1925, **208**, 1093.)—Water that has been treated with chlorine in minute quantities, as used by the Metropolitan Water Board, is liable, under certain atmospheric conditions, to acquire an odour of iodoform. This is due to the presence in the water of a trace of phenol or allied compound, presumably taken up by the water from the smoke-contaminated atmosphere. Water containing only 1 part of phenol in 1000 millions gives a pronounced taste after the chlorine has been added, and some people have been able to detect the taste when the phenol was present in the proportion of 1 in 5000 millions (=14 grains in 1 million galls. of water). It seems probable that water exposed to the air in an open reservoir could absorb this amount of phenolic substance. When air contaminated with smoke is bubbled through pure water the iodoform taste is obtained on the addition of chlorine, and it is suggested that a method of determining the degree of pollution of the air by smoke may be based on the reaction.

Salt Error of Cresol Red. W. D. Ramage and R. C. Miller. (*J. Amer. Chem. Soc.*, 1925, **47**, 1230–1235.)—The best colorimetric method for determining the hydrogen-ion concentration of sea water or other saline media is the double-wedge comparator method of Barnett and Barnett (1920), and the most useful single indicator for use with sea water is *o*-cresol-sulphone-phthalein (cresol red), which has a P_H range of 7·2 to 8·8. The results thus obtained are, however, higher than the true values, owing to the direct effect of the salt present on the colour of the indicator, and the authors find the necessary corrections for salinities varying from 5 to 35 parts of sea salts per 1000 to be as follows:—Salinity 5, correction, 0·11; 6, 0·13; 7, 0·14; 8, 0·15; 9, 0·16; 10, 0·17; 11, 0·18; 12, 0·19; 13, 0·20; 14 to 15, 0·21; 16 to 17, 0·22; 18 to 19, 0·23; 20 to 22, 0·24; 23 to 26, 0·25; 27 to 31, 0·26; 32 to 35, 0·27. T.H.P.

Organic Analysis.

The Carbon Residue Test for Lubricating Oils. W. M. Seaber. (*Industrial Chem.*, 1925, **1**, 79–80.)—The maximum temperature of heating of the oil is the chief controlling factor in the carbon residue test, and for obtaining accurate determinations it is important to be able to control the temperature. For this purpose an electric oven with a temperature control to 5° C. may be used. The rate of increase of temperature from the start need not be rigidly controlled, but 500° C. was found the most suitable maximum temperature for most oils, and it is convenient to reach this temperature in 12 minutes and to take 35 minutes for the whole operation. The rate of change of per cent. of carbon residue with temperature decreases as the temperature rises. Curves are shown for variations in carbon residues with increase of maximum temperature for three classes of oils—light cylinder, superheat cylinder and very high asphaltic cylinder oils. The residues obtained below 444° C. are almost completely soluble in carbon disulphide. Residues obtained at 500° C. are slightly soluble in petroleum spirit, the solubility increasing with decreased temperatures, but not at so quick a rate as their solubility in carbon disulphide. D. G. H.

Oxidation of Chinese Wood Oil. F. H. Rhodes and T. T. Ling. (*Ind. Eng. Chem.*, 1925, **17**, 508–512.)—The drying of Chinese wood oil is due to the spontaneous oxidation of the oil, with the formation of a solid oxidation product; during this oxidation the oil absorbs about 44 per cent. of oxygen in five hundred hours, and at the same time gives off about 36 per cent. of volatile substances. Previous statements that the oil absorbs only 8 to 21 per cent. of oxygen during drying are inaccurate, since allowance was not made for the evolution of volatile substances. When the oil has been treated previously with "driers" (lead, cobalt and manganese soaps) it absorbs about 29·5 per cent. of oxygen in five hundred hours and gives off about 18 per cent. of volatile substances, the net change in weight being, therefore, about the same as for the raw oil, and the treated and raw oils do not absorb the same quantity of oxygen during drying. Cobalt soap (about 0·5 per cent.) appears to be the most effective "drier." If the oil is heated

previously with colophony the oxidation proceeds in two distinct stages; the first begins immediately and continues until about 2·5 per cent. of oxygen has been absorbed, whilst the second and more rapid oxidation begins at this point and continues regularly until the oxidation is completed. Similar, but slower, results are obtained when paracoumarone resin is used instead of colophony. W. P. S.

Test for Comparing Detergent Efficiencies of Soaps. R. M. Chapin. (*Ind. Eng. Chem.*, 1925, **17**, 461–465.)—The test is based on the observation that when dilute soap solutions are shaken with powdered flake graphite in the presence of air the appearance of a white band at the lower edge of the froth indicates the presence of an excess of soap. The first appearance of this band when successive increasing quantities of soap are used constitutes an end-point in the test. The graphite, previously digested with hydrochloric acid, then with ammonia, washed, and dried, is standardised against ammonium palmitate solution containing excess of ammonia; all the tests are made at 45° C., and it is convenient to express the values of different soaps in terms of the "ammonium palmitate coefficient."

W. P. S.

Analysis of Candelilla and other Vegetable Waxes. A. Leys. (*J. Pharm. Chim.*, 1925, **117**, 417–424.)—To 10 grms. of wax are added 25 c.c. of 4·55 per cent. potassium hydroxide solution and 50 c.c. of benzene, and the mixture is boiled under a reflux condenser for 15 minutes. The clear liquid is decanted from the viscous brown residue, 2 grms. of powdered ammonium chloride dissolved in alcohol are added, and after boiling for 30 minutes, 50 c.c. of warm water are added, and the heating slowly continued until the separation of two layers. I.—The lower layer is withdrawn, a boiling solution containing 10 grms. of copper sulphate added, and the mixture heated to boiling. Copper chloride is formed, and organic copper salts are precipitated; these are filtered off, washed with cold water and finally dried over sulphuric acid. On heating these salts with benzene the salts of the *non-saturated organic acids* are dissolved. Twenty to thirty c.c. of fuming hydrochloric acid are added to the separated benzene solution, and, after boiling for a sufficient length of time, copper chloride separates, and the benzene solution of the unsaturated acids is washed and the benzene evaporated. (Candelilla wax contains only traces of such acids.) The copper salts of the *saturated organic acids* are decomposed by warming with hydrochloric acid and benzene, and the liberated saturated acids are dissolved by the benzene and thus separated. II.—The benzene layer is carefully evaporated, and after being heated for at least 24 hours at 110° C., the residue is taken up with 100 c.c. of boiling amyl alcohol, 100 c.c. of fuming hydrochloric acid added, and the mixture boiled for a few minutes and cooled. The solidified cake of hydrocarbons, which overlies the crystalline mass of higher alcohols which separates from the mixture, is removed, cleaned and redissolved in amyl alcohol, a few c.c. of 95 per cent. ethyl alcohol added, and, after warming, a few drops of water. The supernatant amyl alcohol solution is decanted from the lower layer of alcohol and water (which holds all the black material in solution), and, after evaporation, leaves the white *hydrocarbons*. To the mass of

higher alcohols in a porcelain dish is added a large volume of hot water, and the mixture is boiled for a long time on a water bath. The amyl alcohol is evaporated, leaving a mixture of acid-alcohols and higher alcohols which are partly esterified by the hydrochloric acid. After cooling, the acid-water is decanted, and 50 c.c. of boiling benzene added, together with 25 c.c. of boiling alcoholic potassium hydroxide solution. After prolonged boiling, 50 c.c. of warm water are added, and the heating continued until the separation of two layers occurs. The lower alcohol and water layer remains turbid and, on cooling, gives a muddy deposit, which, after being acidified and dissolved in ether, leaves, on evaporation of the ether, a hard brown rather greasy substance melting at 74° C. and having a neutralisation value of 1643. The benzene layer on evaporation leaves the *higher alcohols* contaminated with potassium salts of the acid-alcohols; for these chloroform is the best solvent. A more complete separation may be achieved by prolonged boiling with acetic anhydride, and, on subsequent drying, a mass results, partly liquid and containing the acetic esters of the higher alcohols, and partly solid, consisting of the more or less esterfied potassium salts of the acid-alcohols. III.—The viscous brown residue from the first boiling with potassium hydroxide is dissolved in boiling glacial acetic acid, a few drops of fuming hydrochloric acid and a little benzene added, and the boiling continued. Warm water effects separation of the benzene, and this, on evaporation, leaves a waxy residue melting at 71° C., having a neutralisation value of 102·8 and giving a positive resin reaction with Halphen's reagent. The aqueous layer contains iron, magnesium and a mucilaginous substance.

A commercial sample of candellila wax gave the following analytical figures:—Sp. gr. at 15° C., 0·991; m. pt., 71° C., acid value, 19·4; saponification value, 53·5; iodine value, 12·9; matter volatile at 100° C., 0·52 per cent.; and ash, 0·7 per cent. The figures for 100 grms. of wax deprived of mineral compounds were:—Hydrocarbons, 54·2 grms. of m. pt., 66° C.; iodine value, 6·2; higher alcohols and distinctive acids, 41·0 grms.; saturated acids, 4·2 grms. of m. pt. 69° C.

D. G. H.

Determination of Colophony. J. Davidsohn. (*Chem. Zeit.*, 1925, **49**, 206–207.)—A review is given of the methods for the estimation of colophony. The titrimetric method of Wolff and Scholze (ANALYST, 1914, **39**, 228) is both rapid and accurate, and is preferable to Twitchell's method, but it is essential that methyl alcohol be used, as serious error may arise from the use of ethyl alcohol. From 2 to 5 grms. of the mixed fatty and resin acids are dissolved in 10 c.c. of absolute methyl alcohol, heated for 2 minutes with 5 to 10 c.c. of a mixture of 1 part of sulphuric acid and 4 parts of methyl alcohol, after which 5 to 10 volumes of 7 per cent. sodium chloride solution are added, and the resin acids are shaken out with ether. The mixed ethereal extracts are washed with dilute salt solution, diluted with alcohol, and titrated with alcoholic potassium hydroxide solution. The percentage of resin acids is given by the expression $\frac{17{\cdot}76a}{m} - 1{\cdot}5$, where

a is the number of c.c. of 0·5 N alkali and m the weight of mixed acids taken; the result × 1·07 gives the corresponding quantity of colophony. Double esterification is quite unnecessary. H. E. C.

Determination of Resin Acids. R. Jungkunz. (*Chem. Zeit.*, 1925, **49**, 391.)—Referring to Davidsohn's paper (preceding abstract) it is pointed out that McNicoll's paper (ANALYST, 1921, **46**, 340) has been overlooked. The general accuracy of this process is confirmed, but it appears that the fatty acids are not quite completely esterified. The mean error on known mixtures examined by Wolff and Scholze's process was −1·3 per cent., and by McNicoll's gravimetric method +2·7 per cent. H. E. C.

Solubility of Various Wood Tars in Water. H. N. Calderwood. (*Ind. Eng. Chem.*, 1925, **17**, 455.)—The solubility of various tars was found to be as follows, the figures expressing grms. per 100 c.c. of water:—Hard maple tar, raw settled, 1·05; ditto, boiled, 0·60; pine tar, raw, 0·225. W. P. S.

Rapid Methods for the Analysis of Paper. J. Croland. (*Chim. et Ind.*, March, 1925.)—The composition of a paper may be ascertained by determining the extent to which it absorbs phenol and aromatic bases. A certain weight of the paper is left for a definite time in contact with a solution of the reagent of known concentration, and the amount of the reagent remaining in solution then determined. The following figures represent the absorptive powers of different fibres for phenol and aromatic bases respectively: Pure cotton, 0·2, 1·1; rag pulp, 0·5, 3·0; esparto, 0·7, 3·7; bleached sulphite cellulose, 0·8, 5·0; bleached straw cellulose, 1·1, 6·2; bleached soda cellulose, 1·2, 6·5; unbleached sulphite cellulose, 1·2, 6·6; bleached jute cellulose, 4·1, 10·0; mechanical wood pulp, 8·0, 12·0. Equations are given for calculating the composition of a paper by means of the results of the absorption measurements, and colour reactions are described which permit of the approximate determination of mechanical wood pulp in a paper. When hydrolysed by acid, certain papers yield furfural in amount varying with the origin of the pulp. T. H. P.

Determination of Cyanamide. L. A. Pink. (*Ind. Eng. Chem.*, 1925, **17**, 459–460.)—The following procedure is recommended for the determination of cyanamide in the presence of dicyanodiamide, urea, guanylurea and guanidine. Two grms. of the crude cyanamide are extracted with 400 c.c. of water for two hours, the mixture is filtered, 50 c.c. of the filtrate are treated with 1 c.c. of concentrated ammonia, and ammoniacal silver nitrate solution is added, drop by drop, until no more precipitate forms. After fifteen minutes the precipitate is collected on an asbestos filter, washed eight times with water, then dissolved in dilute nitric acid and titrated with standardised thiocyanate solution, using ferric alum as indicator. If much dicyanodiamide is present in the sample, the silver cyanamide precipitate must be redissolved in about 25 c.c. of N nitric acid, the

filter washed with water, and the solution thus obtained treated with a few c.c. of ammoniacal silver nitrate solution and sufficient ammonia to render it just alkaline. After two hours, the precipitate is collected, washed, dissolved and titrated. In the case of samples also containing carbide, the silver cyanamide precipitate cannot be titrated; the nitrogen in it must be determined by Kjeldahl's method.

W. P. S.

Inorganic Analysis.

New Process for Separating Aliphatic Amines from Ammonia. P. Leone. (*Gazz. Chim. Ital.*, 1925, **55**, 246–252.)—Sodium cobaltinitrite precipitates ammonia quantitatively from its salts, but is without action on amines or alkylammonium salts. To detect ammonia or an ammonium salt in the solution of an amine salt, the liquid is treated with excess of freshly prepared aqueous alcoholic solution of sodium cobaltinitrite and sodium nitrite: the appearance of a turbidity or precipitate indicates the presence of ammonia. The amine contained in a mixture of amine and ammonium salts may be ascertained by first determining the total volatile alkali, and then treating the solution with about double the theoretical quantity of an aqueous alcoholic solution containing 2 to 3 per cent. of sodium cobaltinitrite and 2 to 3 per cent. of sodium nitrite, allowing the mixture to stand for 24 hours, filtering, and determining the volatile alkalinity in an aliquot part of the filtrate. A solution of an amine may be freed from ammonia by neutralisation with acetic acid, treatment with sodium cobaltinitrite and nitrite solution, filtration, and distillation with sodium hydroxide; the distillate is quite free from ammonia.

T. H. P.

New Qualitative Tests for Copper, Iron, and Cobalt. M. L. Nichols and S. R. Cooper. (*J. Amer. Chem. Soc.*, 1925, **47**, 1268–1270.)—With solutions of copper, iron, or cobalt salts, aqueous dinitrosoresorcinol produces either a precipitate or coloration, according to the concentration of the salt: (1) *Copper.* The excess of ammonia in the solution containing copper and cadmium is neutralised with sulphuric acid, a slight excess of which is added. Addition of 2 to 4 c.c. of hot dinitrosoresorcinol solution and a few small crystals of sodium acetate to the liquid results in the formation of a brown precipitate or a dark brown coloration in presence of copper. (2) *Iron.* The ferric hydroxide obtained in the usual way is dissolved in the minimum amount of hydrochloric acid, and the liquid nearly neutralised with sodium hydroxide and treated with 2 to 4 c.c. of the dinitrosoresorcinol solution and a few small crystals of sodium acetate; a green precipitate or coloration indicates the presence of iron. (3) *Cobalt.* The precipitated sulphides of cobalt and nickel are dissolved in *aqua regia*, and the solution nearly neutralised with ammonia solution. A little of the liquid is gently warmed with 2 to 4 c.c. of dinitrosoresorcinol solution and a few sodium acetate crystals, the presence of cobalt being shown by the formation of an orange-red precipitate or coloration. If the solution to be tested is decidedly green, the amount of the nickel present must be first reduced, best by treatment with dimethylglyoxime and filtration.

The dinitrosoresorcinol solution used should be freshly made, and is conveniently prepared by adding excess of the solid to boiling water and filtering; the solution is kept hot while making the tests. In neutral solution this reagent is capable of detecting 0·004 mgrm. of copper, 0·0035 mgrm. of iron, and 0·0033 mgrm. of cobalt in 1 c.c. of liquid. T. H. P.

Separation of Metals of the Ammonia and Ammonium Sulphide Group. K. K. Järvinen. (*Zeitsch. anal. Chem.*, 1925, **66**, 81–100.)—The separation of the sesquioxides and phosphoric acid from the monoxides is incomplete by the usual methods (ammonia, barium carbonate, calcium carbonate, basic acetate, ammonium carbonate), and the precipitation must be repeated several times. The author recommends precipitation of the iron as basic sulphate and completion of the sesquioxide precipitation by a nitrite; phosphoric acid also is precipitated completely if its amount does not exceed one-fifth of the sum of the sesquioxides. The solution, if free from sulphate, is treated with 2 grms. of ammonium sulphate, and 2*N* ammonium carbonate until distinctly turbid. On boiling, iron is precipitated as a dense basic sulphate, whilst much aluminium and chromium remain dissolved. To the hot liquid 3 to 5 grms. of nitrite are added gradually, with stirring. After cooling, the precipitate is collected and washed, and the filtrate boiled, any cloudiness being cleared by adding a minimum quantity of hydrochloric acid. After addition of 0·5 to 1 grm. of nitrite, the solution is again left to cool, and filtered as before. The combined precipitates contain the sesquioxides free from monoxides; the filtrate is free from sesquioxides, which is proved by addition of a few c.c. of ammonium carbonate solution. A detailed description is given of the subsequent separation of the monoxides. Zinc is precipitated first by hydrogen sulphide in faintly acid solution, the excess of nitrite having been previously destroyed by boiling with acid; the sulphide is converted into, and weighed as oxide or sulphate. Nickel and cobalt are precipitated next by hydrogen sulphide after conversion of the liquid into an acetate solution containing 1 to 2 per cent. of acetic acid. The precipitate is roasted, dissolved in hydrochloric acid, and converted into sulphates, which are weighed and separated by dimethylglyoxime. Manganese is precipitated by hydrogen sulphide from the filtrate, after concentration to 100 to 200 c.c. and addition of 2 c.c. excess of ammonia. It is weighed as sulphate. W. R. S.

Detection of Traces of Manganese in Iron Alloys. H. Bösche. (*Chem. Zeit.*, 1925, **49**, 378.)—The sensitiveness of the nitric acid and lead peroxide test is much diminished by large quantities of ferric salt, but phosphoric acid prevents this interference. The metal (0·1 grm.) is dissolved in 10 c.c. of nitric acid (1:1) in a test tube, and the solution boiled till the fumes have been expelled. Boiling is continued for 2 minutes after addition of 0·5 grm. of lead peroxide and a few c.c. of strong nitric acid. After being allowed to settle, the liquid is treated with 0·5 c.c. of strong phosphoric acid, when a pure purple coloration remains.

W. R. S.

Determination of Molybdenum in Steel. O. L. Maag and C. H. McCollam. (*Ind. Eng. Chem.*, 1925, **17**, 524.)—A rapid colorimetric method is described. A weighed portion of 0·5 grm. of the steel is dissolved in a mixture of nitric and sulphuric acids (nitric acid, sp. gr. 1·42, 350 c.c.; sulphuric acid, sp. gr. 1·84, 225 c.c.; and water, 750 c.c.), the solution is evaporated until sulphuric acid fumes are evolved, then cooled, treated with 30 c.c. of a mixture of hydrochloric and sulphuric acids (hydrochloric acid, sp. gr. 1·19, 100 c.c.; sulphuric acid, sp. gr. 1·84, 450 c.c.; and water, 1450 c.c.), the mixture is boiled until the salts dissolve, cooled, and treated with 5 c.c. of 5 per cent. potassium thiocyanate solution and 10 c.c. of stannous chloride solution (stannous chloride, 250 grms.; hydrochloric acid, sp. gr. 1·19, 200 c.c.; and water, 800 c.c.). The acid solution is then extracted with successive 10 c.c. portions of ether until all the colour has been removed, the ethereal solutions are placed in a colorimeter tube, and the colour compared with that obtained by treating a standard molybdenum steel in the same way.

W. P. S.

Gravimetric Determination of Aluminium and its Separation from Manganese. B. Solaja. (*Chem. Zeit.*, 1925, **49**, 337–338.)—Aluminium is precipitated quantitatively, like iron (ANALYST, 1923, **48**, 463) by a suspension of aminomercuric chloride ("infusible white precipitate"): $2AlCl_3+3ClHgNH_2+6H_2O=2Al(OH)_3+3HgCl_2+3NH_4Cl$. The cold chloride solution (200 c.c.) is neutralised with ammonia; 2 grms. of ammonium chloride are added, and 2 to 3 times the required quantity of precipitant. The precipitate is collected next day, washed 3 times by decantation with water, and ignited in platinum (gently at first). The precipitant should leave no fixed residue. The procedure separates manganese quantitatively; this can be recovered from the filtrate by precipitation with hydrogen peroxide and ammonia and ignition of the precipitate to Mn_3O_4.

W. R. S.

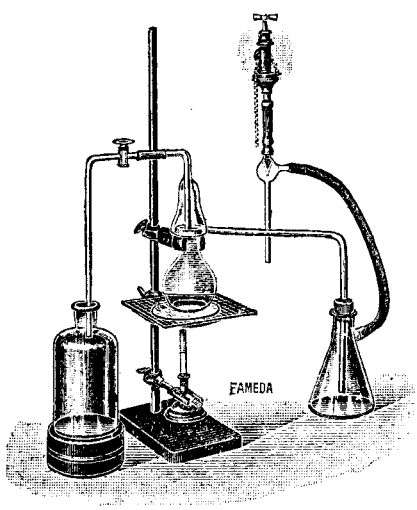
Potentiometric Determination of Cerium. O. Tomiček. (*Rec. Trav. Chim. Pays-Bas*, 1925, **44**, 410–415.)—The sulphate or chloride (not nitrate) solution (about 10 c.c.) is treated with about three-quarters of the necessary amount of 0·1 *N* ferricyanide solution, after which the air is completely expelled from the flask by a carbon dioxide current. A 50 per cent. solution of potassium carbonate is then added until the solution to be titrated contains about 30 per cent. of carbonate ion: $2K_3Fe(CN)_6+K_2CO_3.Ce_2(CO_3)_3=2K_4Fe(CN)_6+2Ce(CO_3)_2$. The titration is then completed as usual, the final volume being 50 to 100 c.c. The potential of the platinum electrode in the solution becomes constant very soon after each addition of ferricyanide. The potassium carbonate concentration at the end of the titration should be 20 to 25 per cent., if smaller, a precipitate is formed during the operation, and low results ensue.

W. R. S.

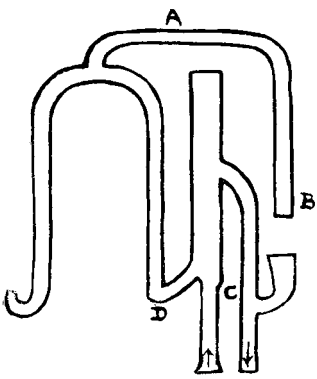
Physical Methods, Apparatus, etc.

Application of Wood's Light to the Examination of Olive Oils and Turpentine Oil. Frehse. (*Ann. Falsificat.*, 1925, **18**, 204–206.)—Under the influence of the light of a quartz mercury vapour lamp furnished with a nickel oxide screen (Wood light), olive oils of different origins showed the following characteristics: (1) Refined olive oil, bluish-green fluorescence; (2) refined "sulphur" oil, more marked and more green fluorescence; (3) French, Algerian, Tunisian, Italian, Spanish, and St. Denis du Sig oils, orange fluorescence; (4) St. Cézaire oil, yellow fluorescence with green tint. The presence of 5 per cent. of oil (2) caused replacement of the orange by the green colour, which appears also in presence of 10 per cent. of oil (1) except for the native (French) oil, with which 15 per cent. was necessary. Other observations made with this light are as follows: It imparts a violet fluorescence to sesamé and arachis oils; the addition of white spirit to oil of turpentine changes the green to a violet fluorescence; non-fluorescent silks give, with certain loadings, ash showing faint blue fluorescence, and with other loading, ash devoid of fluorescence; silks dyed to the same tint with naphthol yellow S and auramine O show respectively a very deep and a very bright colour. T. H. P.

New Apparatus for Distilling Mercury. J. Wetzel. (*Chem. Zeit.*, 1925, **49**, 373).—The apparatus consists of a distillation-flask of about 80 mm. diam., with a neck about 85 mm. high, providing an air-condenser with a "gutter," 6 mm. deep, which collects the condensed mercury and prevents its re-entering the distillation-flask. The air-condenser is efficient with a laboratory temperature of 25° C. The "gutter" is connected with the receiver by a side-tube, and an inlet-tube, connected with a vessel containing the mercury previously purified with dilute nitric acid, is provided with a tap and passes into the flask through the air condenser. The apparatus is insulated with asbestos up to the air-condenser. The flask is three parts filled and the distillation carried out in a vacuum of 12 mm. over a wire gauze and a small flame. The distillation can be made continuous, and will produce about 6 kilos. of mercury in 8 hours. The apparatus, which can also be used for small amounts of mercury, can be obtained from the firm Fameda A–G. Ringbahnstrasse, 21 Tempelhof, Berlin. R. F. I.



Apparatus for Determining the Specific Gravity of Aggregates. F. H. Tucker. (*Ind. Eng. Chem.*, 1925, **17**, 517–520.)—The apparatus is intended for use in determining the specific gravity of aggregates separated from bituminous mixtures where the particles may vary in size from those passing a 200-mesh sieve to those retained on a 4·4 cm. mesh. It consists of a cylindrical glass jar, 15 cm. in depth and 12·5 cm. in diameter and of about 1·5 litres capacity, for holding the liquid (*e.g.* kerosene). An overflow tube passes through a tubulure near the top of the jar; the short inner end of this tube is bent at an angle of 90° to form a miniature siphon, and the outer end is bent downwards, a sharp start and break in the overflow of liquid being thus effected. A weighed quantity (260 to 1100 grms.) of the material under examination is added gradually to the jar through a funnel having an oblique wide stem, and the displaced liquid passing through the overflow tube is collected and weighed. W. P. S.



Constant-Level Regulating Device. R. L. Stehle. (*Ind. Eng. Chem.*, 1925, **17**, 466.)—The apparatus shown has been devised with the object of preventing the collection of air bubbles and consequent interruption of the action of the siphon. The second siphon, *A*, is provided, and this removes the air bubbles as they are liberated from the water. The rate of siphoning is adjusted by lengthening or shortening the tube *B* by means of a length of rubber tubing, or by constricting the end of *B*. Free escape of occasional large air bubbles in the tap water is insured by the enlargement of the inlet tube at *C* and by the downward bend of the main siphon at *D*. W. P. S.

Artificial Daylight Spectacles for the Laboratory. H. Weiss. (*Chem. Zeit.*, 1925, **49**, 197.)—The "Lumina Spectacles" have been devised to obtain an artificial daylight with the use of any half-Watt electric lamp. They enable shades of colour to be accurately matched and titrations (*e.g.* with methyl orange as indicator) to be done which are uncertain with ordinary artificial light.

Reviews.

THE STORY OF EARLY CHEMISTRY. By JOHN MAXSON STILLMAN, late Professor Emeritus of Chemistry, Stanford University. Pp. xiii.+566. New York and London: D. Appleton & Co. Price 18s.

This scholarly book will be regarded by many as a memorial to its late author, who died only a few days before the first proof sheets were delivered by the

publishers. Professor Stillman's interest in the chemical world had a considerable bias towards its history, as was well shown by his contributions on the chemistry of the fifteenth and seventeenth centuries to American periodical literature, as well as by his book on "Paracelsus," which was reviewed in these pages. In the foreword, written by his colleague, Prof. Stewart W. Young, it is stated that this volume grew out of the author's lectures, and we learn from the preface that the opportunity for the writing of it came when he retired as emeritus professor in 1917. Some of us occasionally deplore the steady stream of old books and manuscripts from these shores to those across the Atlantic. When we realise that such large private libraries as that of Mr. Herbert C. Hoover, as well as the Stanford University library, with their fine store of treasures relating to early chemistry, metallurgy and mining, are turned to such good account for the benefit of chemical students in general, our regrets should be much sweetened with gratitude for an acquisitiveness which is largely immune from selfishness.

Prof. Stillman has written a compendious treatise in which he "planned to develope in parallel from the earliest known beginnings the history, on the one hand of the chemical arts, on the other hand of chemical thought and theory, concluding the work with the downfall of the phlogiston theory. He aimed at a book that should be found readable by those whose knowledge of the science was not profound, as well as by those professional chemists who find little time to delve into such matters for themselves." This aim he has quite successfully achieved, and has made good use of the results of his own historical researches, as well as of all the chief authorities on the history of alchemy and chemistry, such as M. Berthelot, von Meyer, Sudhoff, Ferguson, Kopp, and von Lippmann, in addition to the older historians, such as Mangetus, Boerhaave, Bergman and Thomson. The bibliography which the author has supplied comprises fourteen pages, and it does not contain by any means the whole of the works referred to in the text and in the footnotes. There are one or two books, however, for which one would like to have found a place because of their general interest in relation to the subject. Professor Stillman cast the net of his reading most widely, but he does not furnish any evidence of having read the late Dr. Hastings Rashdall's *Universities of Europe in the Middle Ages*. It may be that for this reason there is little, if any, consideration given to the incorporated magisterial teaching of what we now call science. Another book which should have found a place in the bibliography is Sir Wm. Ramsay's *Essays—Biographical and Chemical*.

The earlier chapters are arranged in a manner which is representative of the successive stages through which the development of the arts of life must have passed, and as presented to the curiosity of enquiring minds seeking a reasoned knowledge of the constitution of materials. They are "The Practical Chemistry of the Ancients," "Earliest Chemical Manuscripts," "Theories of the Ancients," "The Early Alchemists," and "The Chemical Knowledge of the Middle Ages." The second half of the book deals with the growth of chemistry in an almost strictly chronological manner. An attractive feature is the large number of excerpts from the works of the many old writers named, which confer a variety of diction

as well as a spice of antiquity to its pages. The learning of the ancients is presented in an agreeable form, and much of it may have a flavour of novelty. It was hardly to be expected that Professor Stillman should have been so fortunate as to introduce some entirely unknown name in the history of chemistry; yet it may well be that there are not a few chemists who have not hitherto known that the Russian poet Lomonosoff (Mikhail Vasilievich Lomonósov) was by eighteen years a forerunner of Lavoisier in the rejection of the phlogiston theory.

The sixteenth century is dealt with in a manner which shows once more what a careful study the author had made of that period. In discussing the alchemists of the two previous centuries not a few of the adepts named receive rather more scant treatment than is due to students. It is a difficult period to understand because of the mythical ascriptions of many of the manuscripts then in the hands of the *cognoscenti*. One instance of the confusion which surrounds the historian is the question of the origin of the *Tabula Smaragdina*. It is not at all clear whether Prof. Stillman was prepared to make a definite statement as to his own opinion beyond leading his readers to understand that it might have been compiled by its alleged translator Hortulanus in the eleventh or twelfth century, or even in the fourteenth century, if Berthelot's view were accepted. The likelihood of a much earlier origin for it has been suggested many times, and the legend connected with it carries its professed beginnings into very early times. The author seems to have accepted the idea of a mediaeval source too readily, and, strangely enough, was apparently not acquainted with Mr. Holmyard's article on "The Emerald Table" (*Nature*, 1923). In this it is explained how it has come to pass that now we may assume with some confidence that it may be antedated by at least four hundred years, and that it is most probably one of the earliest fragments of the alchemical writings.

The seventeenth and eighteenth centuries gave chemistry a number of students whose names are not very familiar to-day, but who played quite prominent parts in the development of the science. Among them were Robert Hooke, Le Febure, N. Lémery, Glaser, Hoffmann, and Hermann Boerhaave. The last of these did much to extend the knowledge of this fascinating science. His *Elementa Chemiae* passed into many editions and was translated into the chief European languages. Thomas Thomson said of it that "it was the most luminous treatise on chemistry that the world had yet seen." The most interesting edition for the English student is the second edition (1741) of Peter Shaw and E. Chamber's translation, in two quarto volumes, to which the translators have added numerous historical footnotes.

Prof. Stillman's work is an essential handbook for all who desire to be informed on the history of chemistry. It is scholarly without being dull; it is authoritative, but it stimulates extended enquiries, and it is suffused with the spirit of the humanities. It is a book to be well recommended. There is a good index and an excellent bibliography. It is well printed, well-bound and comfortable to handle. W. KIRKBY.

THE SCIENTIFIC PRESERVATION OF FOOD. By THOMAS M. RECTOR. Pp. xl. + 213. New York: John Wiley & Sons, Inc.; London: Chapman & Hall, Ltd. 1925. Price 10s. net.

The author in his preface states that he has endeavoured to eliminate technical language as much as possible in the hope that the book will be of interest to the non-technical man, as well as to the technologists of the food industries. In so far as the non-technical man is concerned, he has certainly achieved his aim, and most men engaged in these industries would benefit considerably by a careful study of the subject as discussed in this book. Whether the technologist, if by this term is meant the experienced chemist or other expert, will be equally interested is rather more doubtful, as the author deals mainly in generalisations which are, or should be, already well known to those chemists and bacteriologists who have to deal with the preservation of foodstuffs.

For the less experienced technologist who is just taking up this branch of work, the author's review of the various forms of spoilage and methods of combating these will no doubt be helpful and will save him from many pitfalls. At the same time, the complete absence of technical details for the various methods of preservation discussed is a serious omission. Many examples of this could be quoted, but the following are particularly noticeable. In discussing methods of preservation by cold storage the most suitable temperatures for different types of foodstuffs are not given. Again, regarding sterilisation no details are forthcoming beyond the general statement that the "neutral" class of foods is usually sterilised in closed steam retorts at temperatures of from 230° to 250° F., for from 20 minutes to 3 hours. This statement, if taken literally by an inexperienced worker, might easily lead to disastrous results.

With reference to chemical preservatives the author is still more vague, merely indicating the type of foodstuff in which each preservative has been used. No attempt is made to give relative values of the preservatives, nor the dosage necessary in any particular case.

While exposure to atmospheric humidity is rightly stated to be a prolific cause of spoilage, no figures are given to show at what relative humidity damage might be likely to occur. Indeed, all details of this description are conspicuous by their absence. This fact, together with the plan on which the author has treated the subject, will make it difficult for a manufacturer, who is interested only in one particular class of foodstuffs, to get a clear idea of the possible causes of spoilage he is likely to meet with, and still less will he be able to find cut-and-dried remedies. For example, anyone interested in the preservation of eggs would require to refer to fourteen different sections of the book, and would then have to develop for himself all details of any method of preservation selected.

There is no bibliography, and no references are given to any published work on the subject. The excellent fundamental work on preservation by cold storage carried out by the Food Investigation Board in this country does not appear to be known to the author. Sterilisation methods have been investigated by various

research organisations in America, and much useful information has been published, references to which would have been most useful.

Few people outside the foodstuffs industries realise the numerous difficulties with which manufacturers have to contend in marketing their products, and a more widespread knowledge of the causes of food spoilage can only result in benefit to manufacturer and consumer alike. In not a few cases manufacturers have been forced to use preservatives, not so much to preserve their products prior to sale, but rather to protect them from the consequences of careless and ignorant handling on the part of both the retailers and consumers, whom they have found it very difficult to educate in these matters. It is to be hoped, therefore, that this book may be widely read by the general public and the trades concerned in the handling of foodstuffs for whom it is obviously best suited. T. MACARA.

THE CONFIGURATION OF THE SACCHARIDES. By J. BOESEKEN, Ph.D. Translated from the Dutch by SAMUEL COFFEY, M.Sc., Ph.D. In two volumes. Vol. I., pp. iv.+71; Vol. II., pp. iii.+58. Leyden: A. W. Sijthoff's Publishing Co. Paper covers. Price 3fl. each.

The last few years have witnessed a remarkable activity in the field of research dealing with the constitution of the carbohydrates, and this activity has been so fruitful both in facts and in theories that a book embodying the latest advances should be welcome. In the present work Dr. Boeseken, who has himself made valuable contributions to our knowledge of the subject, gives a fairly complete summary of recent research in carbohydrate chemistry.

The butylene oxide ring structure generally assigned to glucose, and frequently referred to in the text as a "five ring," is discussed at length, and due consideration is given to the other ring structures which are or may be present in the monosaccharides and their derivatives. The author makes an unfortunate slip in stating (Vol. II., p. 10) that the reducing power of the reducing bisaccharides towards Fehling's solution is about half that of the monosaccharides. Such a statement would hold for the iodine absorptive power of aldose sugars in dilute alkaline solution, where the reaction is practically confined to the conversion of the aldehyde into the corresponding carboxylic acid. The reaction between reducing sugars and Fehling's solution is much more complicated, and it is well known that the copper-reducing power of the reducing bisaccharides is considerably more than half that of the monosaccharides.

None of the numerous views as to the constitution of starch appears to find favour with the author. The pioneering work of H. T. Brown and his collaborators on the mechanism of the diastatic hydrolysis of starch is not mentioned, an omission which, in the reviewer's opinion, is not justifiable.

The work of translation has not been well done. Many sentences retain the Teutonic structure of the original, some passages require a second or third reading

to render their meaning clear, and a few passages are even more refractory; the grammar and punctuation are sometimes faulty, and misspelt words are frequent. These faults are especially objectionable in a book dealing with so intricate a subject as the constitution of the saccharides. LEWIS EYNON.

BOILER FEED WATER. By N. SIMPKINS, M.Sc., A.I.C., and A. DAWE, B.Sc., A.I.C. Pp. 44. London: H. F. & G. Witherby. Price 2s. 6d. net.

This little book is issued under the auspices of the Lancashire and Cheshire Coal Research Association, and gives a general account of the waters occurring in the coal districts and the various chemical problems arising from their use for steam raising.

It does not pretend to be a scientific treatise. Its aim is essentially practical, but no one can carry out the treatment of boiler waters intelligently without some knowledge of the theoretical considerations on which it is based. This is briefly but very clearly set forth.

It deals with the characters of the waters likely to be employed and their suitability or otherwise for the purpose as regards the prevention of corrosion and the formation of scale. The various methods of treatment are rapidly reviewed, and the chemical reactions involved between the various salts under the action of temperature and pressure are adequately explained, the methods for the removal of oil and the control of salinity receiving due consideration.

The more typical softening plants are described, together with the mechanical devices which enable the control of the process to become more or less automatic.

Practically every chemical and physical problem connected with the treatment of waters, with some small exceptions, is referred to, and there are special paragraphs on oil removal, on the cause of priming, and on the use of boiler compositions.

To deal with such a wide range of complex phenomena, in what is little more than a pamphlet, in a scientific manner, without being too abstruse and speculative, is a difficult task which the authors have achieved with ability and success.

Only about four pages are devoted to corrosion, but these, by avoiding obscure and conflicting theories, cover nearly every essential point.

Reference might perhaps have been made to the comparatively recently introduced "de-gassing" of waters, and also to what is not yet fully recognised, the possibility of pitting with softened waters containing a moderate excess of caustic alkali, and, to make the story still more complete, the electrolytic methods of preventing corrosion, including the Cumberland process, might also have received notice.

This little brochure is an excellent production, creditable to the authors and the publishers alike. CECIL H. CRIBB.

FORENSIC MEDICINE. By SYDNEY SMITH, M.D. (Edin.), D.P.H., Principal Medico-Legal Expert, Egyptian Government Service, Professor of Forensic Medicine in the Royal Schools of Medicine and Law, Cairo. With Introduction by Prof. HARVEY LITTLEJOHN, F.R.C.S. (Edin.), F.R.S.E., Professor of Forensic Medicine, University of Edinburgh. Pp. xiv. +498. London: J. & A. Churchill. 1925. Price 21s. net.

This book, though written mainly for medical students and practitioners, is one which will make a valuable addition to the library of others to whom problems relating to forensic medicine and toxicology may present themselves.

Of the 28 chapters into which the book is divided, 21 are confined to forensic medicine and allied subjects, and 7 deal with toxicology.

In addition, there are 5 appendices, of which the first, (dealing with the systematic examination of viscera for poisons), will be of special interest to analysts. Appendix 2 deals with the examination of firearms, etc.; Appendix 3 is a short note on the Kaiserling method of preserving specimens; Appendix 4 is a most useful note relating to the approximate weights of organs, etc.; and Appendix 5 gives an interesting survey of medico-legal work in the East, with special reference to Egypt.

In the chapter dealing with finger prints (Chap. V.) (which in a book of this character, dealing with so many branches, can obviously not be of great length) it is to be regretted that no mention is made of chemical vapour methods of examination, and no reference given to Dr. Fauld's pioneering work on finger prints.

Chapter XI. (though unavoidably somewhat short) deals fairly fully and very concisely with the examination of bloodstains.

With regard to Appendix 1, the subject of chemical examination of organs is dealt with in the main as thoroughly as possible in a book of the capacity of the present volume.

A few omissions will be noted and regretted, but it is impossible to deal with the subject as fully as the author might wish, in an appendix of about 30 pages; more references, however, would help to counteract this, and would add considerably to the value of the book.

In the article on arsenic, it is a pity that Dr. Smith has not laid rather more stress on the *electrolytic* "Marsh" test, and surely he is somewhat overcautious in stating that "the test is extremely delicate, and one-fiftieth of a milligramme can be detected with *comparative ease*," when 1/500 milligramme can be quite easily detected by the use of platinum electrodes, and a still less quantity when lead electrodes are used.

There is an interesting introduction to the book by Prof. Littlejohn, in which he advocates the formation of a medico-legal institute in London, under the auspices of the Home Office, of a character similar to those of certain institutes in other European countries.

The book is well illustrated, the index fairly extensive (a few more subsidiary references would enhance its value), making this volume a welcome addition to our books on forensic medicine and toxicology. JOHN WEBSTER.