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The Absorption of Copper during the Digestion of Vegetables Artificially Coloured with Copper Salts.

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(*Preliminary Note. Read at the Meeting, April* 2, 1924.)

In spite of a large literature on this subject, it is still uncertain whether the use of a small amount of copper sulphate for keeping the colour of green vegetables that are "canned" or preserved in glass receptacles is harmful to man. A careful review of this literature reveals many conflicting statements, and shows that no small proportion of the researches that have been reported are open to criticism for one reason or another.

In the course of an examination of certain preserved vegetables that had been subjected to "greening" with copper sulphate it was desired to determine whether the metal is liberated in a condition capable of being absorbed by the blood-stream during digestion.

Forms of Combination of the Copper.—It seems fairly clear from the researches of Tschirch (*Das Kupfer*, Stuttgart, 1893) that the preservation of the green colour of vegetables on the addition of copper salts, particularly the sulphate, is largely due to the formation of a complex with chlorophyll, termed by him phyllocyanate of copper.

The amount of copper required to effect "reverdissage" is believed to depend on the amount of chlorophyll present. Tunnicliffe (*Appendix* 5–7, *Report of Departmental Committee on Preservatives*, 1901) showed that, on an average, 50 mgrms. of copper per kilo. are sufficient for most green vegetables, a figure that is of the same order as that of 55 mgrms. per kilo. suggested by the German Committee as the maximum to be tolerated.

Actually, it is common to encounter samples of "greened" vegetables with amounts of copper much in excess of these figures, and values as high as 150 to

180 mgrms. per kilo. have been recorded. In such cases it is obvious that more copper has been added than is necessary to preserve the green colour.

It can be shown that "greened" vegetables, especially those with a proportion of copper considerably higher than 50 mgrms. per kilo., contain the metal in combination not only with chlorophyll but also with proteins. The formation of copper compounds of proteins has been studied by Born (*Biochem. Bull.*, 1915, **2**, 293) and by Osborne and Leavenworth (*J. Biol. Chem.*, 1916, **28**, 109), and it has been stated by Wiley (*Adulteration of Foods*, 1911) that compounds of this type liberate soluble copper compounds on digestion with pepsin and trypsin. On the other hand, it has been claimed that the copper compound of chlorophyll, the so-called phyllocyanate of copper, is unattacked by the ordinary digestive enzymes of the mammalian alimentary tract, so that the metal is excreted without absorption.

ARTIFICIAL DIGESTION EXPERIMENTS.—A few experiments were made, therefore, to determine whether, during the digestion of "greened" vegetables containing a normal amount of added copper, there is liberation of appreciable amounts of the metal in a form likely to be absorbed by the walls of the gut, and, if so, whether the absorption of such copper is likely to be harmful to animals, particularly when occurring over long periods.

To determine the facts about absorption it was decided to employ, in the first place, artificial digestion experiments *in vitro*, and to check the results by experiments on animals bearing the well-known Pavlov fistulae.

A number of experiments in which enzyme preparations *in vitro* have been used have been described from time to time in the literature, but in practically all cases no attempt has been made to go further than showing that soluble copper compounds are liberated during the digestion. The question whether these soluble compounds are dialysable has usually been neglected.

Dialysable Products.—A considerable number of digestion trials were started in an apparatus designed to permit of an examination of the dialysable products of the digest. The mixture was placed inside a collodion thimble surrounded by a slowly circulating current of water, and the whole maintained at 37° C. Parallel experiments were always made with samples of "greened" peas and with control groups in which unadulterated peas were used. Determinations were made at intervals, by means of the potassium ethyl xanthate or the ammonia colorimetric methods, of the copper passing out into the dialysate. Usually the digestion was effected by pepsin in acid solution for 4 hours, followed by trypsin in alkaline medium for a further 12 or 24 hours.

The amount of copper in the dialysate from the digestion of samples of "greened" peas was found to be very small. Thus in one set of experiments the digestion of 100 grms. of tinned peas containing 5·6 mgrms. of copper resulted in the passage of 0·015 mgrm. of copper into the outer fluid in the four hours of peptic digestion, and a total of 0·05 mgrm. in the 24 hours' digestion with both enzymes. Similar figures were given by a large number of trials on different samples. The normal peas used as a control contained traces of copper, but the amount passing into the dialysate was too small to determine. From the experiments *in vitro*, therefore, it seems certain that small amounts of copper are liberated during the action of the ordinary digestive enzymes in a form that is likely to be absorbed, and this conclusion is confirmed by the results of the experiments on the dogs with Pavlov gastric fistulae; in these subjects it is possible to withdraw a sample of the stomach contents when desired for examination. Meals consisting of cooked meat, bread, peas, either normal or "greened," were given to the dogs, and, at definite intervals after feeding, samples of the stomach contents were withdrawn and subjected to dialysis in collodion thimbles. Determinations of copper in the dialysates gave figures of the same order as those obtained in the experiments *in vitro*, and indicated that considerably under 0·5 per cent. of the metal present in the coloured vegetables is absorbed during the time the food is in the stomach, usually about 5 to 6 hours. I have to thank Dr. G. V. Anrep for kindly allowing me to use his experimental animals for this work, and for his assistance and advice on many occasions.

EVIDENCE AS TO EFFECTS OF ABSORPTION OF COPPER.—It having been ascertained with certainty that copper is liberated in an absorbable form during digestion of "greened" vegetables, it was necessary to determine whether the absorption of such small amounts of heavy metal is detrimental, especially when prolonged over considerable periods.

It will be remembered that the existing information on this question is very unreliable and confusing. On the one hand, we have a number of authorities who hold that administration of copper over long periods leads to disease, particularly nephritis, and quote cases showing a high incidence of kidney diseases among workers in copper mines or smelting works. Others challenge these results and point to the workers who are employed in copper sulphate works, in the "greening" of vegetables, or in the extensive use of copper-containing spraying mixtures, and who apparently show a normal standard of health. Tunnicliffe (*loc. cit.*) found no ill effects in a study of children, but his experiments were of somewhat short duration. The most elaborate experiments are undoubtedly those made by Long and Chittenden, and published by the American Department of Public Health. This long series of metabolism experiments on men failed to establish that the consumption of "greened" vegetables is deleterious, although the Committee who drew up the Report gave the public the benefit of the doubt and suggested that, in view of the negative results, it might be well to discourage the use of copper in "greening" vegetables. More recently Mallory, Parker and Nye (*J. Med. Res.*, 1921, **42**, 461) claim to have obtained evidence that the prolonged administration of copper salts to certain animals will induce the pathological condition of haemachromatosis, and on these grounds issue a warning against the use of "coppered vegetables." Their experiments lose value in that they gave much larger doses of copper salts than are ever likely to be taken in the form of coloured vegetables, *e.g.* 0·1 grm. of copper acetate daily to rabbits, and also from the fact that haemachromatosis is not associated with workers exposed to copper poisoning. Their argument that the disease is more common than usual in districts where wine is largely drunk, and that it is due to the presence of copper in the wines, is valueless in face of their admission that the abnormal condition can also be produced experimentally by administration of alcohol.

EXPERIMENTS ON RATS.—To gain more information on this point, experiments were started in which groups of rats were fed over long periods on diets containing approximately one-third of their weight in the form of peas, both normal and "greened." The growth, general health, and metabolism of these rats were closely studied, and at the end of the experiment the animals were killed, and their organs examined histologically.

The rats were ingesting about 12·5 grms. of the food mixture daily and were, in the case of the group on "greened" peas, ingesting approximately 1·5 mgrms. of copper daily per 100 grms. weight of rat.

General health.—Throughout the experiment, which lasted some three months, the animals showed good health and quite normal behaviour. They ate well, their coats were in good condition, and at the end of the experiment they looked fine, healthy rats.

Growth.—Records of the animals' weights were kept, and it was found that the groups fed on coloured peas showed an identical rate of growth with those fed on the ordinary peas. In all cases the rate of growth was slightly subnormal, but this may be attributed to the inclusion of so large a proportion of peas in the diet, for McCollum has shown that a diet rich in peas tends to cause some retardation of growth, owing to digestive disturbances.

Metabolism.—Careful records of food intake were kept in certain cases, and, by analysis of the collected excreta, a balance-sheet could be constructed for copper. In confirmation of the tests on digestion, and of the metabolism experiments recorded by the American investigators, Long and Chittenden, it was found that some copper is undoubtedly absorbed and excreted in the urine. Generally speaking, the proportion of the ingested copper appearing in the urine was of the order of 0·5 to 0·7 per cent. There was marked retention, it having been frequently observed that absorbed copper tends to be held by the liver (Ryan, *Biochem. Centralbl.*, 1908, **6**, 791; Yagi, *Arch. Intern. Pharm.*, 1910, **20**, 51; Van Italie and Van Eck, *Pharm. Weekblad*, 1913, **49**, 1157).

Condition of Organs.—Contrary to expectation, it was found, on careful *post-mortem* examination of the animals at the close of the tests, that no abnormality could be detected in the group fed on "greened" peas which could be ascribed to the copper. Especial care was devoted to the examination of the kidneys and livers, but no lesion could be seen with the naked eye or by microscopic examination which could be ascribed to the copper. Indeed, more abnormalities were found in the control groups than in the group fed on the "coppered" peas.

GENERAL DISCUSSION.—It would seem apparent that, in the digestion of vegetables which have been subjected to "reverdissage" with copper sulphate, a certain amount of the copper is liberated in a form capable of passing a dialysing membrane, and therefore likely to be absorbed in the animal body. There is also definite evidence that animals can absorb traces of copper, and that part of this absorbed metal is held for a time by the liver until it is excreted either by way of the urine or the faeces. The experiments on rats recorded in this paper make it questionable whether it has yet been proved that the absorption of small traces of copper over long periods of time is likely to lead to harmful consequences.

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Investigations into the Analytical Chemistry of Tantalum, Niobium, and their Mineral Associates.*

IV. A New Method for the Separation of Tantalum from Niobium.

V. The Detection and Determination of Tantalum in Niobium Compounds.

BY ALAN RICHARD POWELL AND
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(*Read at the Meeting, October* 7, 1925).

IV. A NEW METHOD FOR THE SEPARATION OF TANTALUM FROM NIOBIUM.

THE classical method of Marignac for the separation of tantalum from niobium by the fractional crystallisation of potassium fluotantalate (*Ann. Chim. Phys.*, 1865, **8**, 49) is still regarded as the standard process, though attempts have been made from time to time to provide a more satisfactory solution for this perennial problem of mineral analysis. Since none of the other methods proposed is an improvement upon Marignac's, we may, for the sake of brevity, dispense with giving an account of them here. In the absence of a more reliable method for the separation of the two elements, methods have been worked out for the volumetric determination of niobium in mixtures of the two oxides, tantalum pentoxide being computed by difference; but the unreliability of such methods has recently been demonstrated by Schoeller and Waterhouse (ANALYST, 1924, **49**, 215).

The degree of accuracy of Marignac's method is said to be about 1 per cent., or even 0·5 per cent., if a large quantity (20 to 25 grms.) of mineral is used (Hauser and Lewite, *Zeitsch. angew. Chem.*, 1912, **25**, 100). It is, however, quite certain

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* Sections I. and II., ANALYST, 1922, **47**, 93; Section III., *ibid.*, 1924, **49**, 215.

that such favourable results are obtainable only by a skilled analyst thoroughly versed in the procedure; in the hands of less qualified operators, there is almost no limit to the magnitude of the error. The practical disadvantage of the process is that the acid fluoride solution necessitates the use of several platinum or gold basins.

In the method here described the separation is based on the differential hydrolytic dissociation between oxalotantalic and oxaloniobic acids in presence of tannin in slightly acid solution, a reaction discovered by us in 1920; the experimental work required to make it available for quantitative separations occupied four years. No fluoride or alkaline solutions are utilised, hence ordinary glassware can be used; and, as the preliminary pyrosulphate fusion is carried out in a silica crucible, we are able to dispense entirely with costly vessels of platinum or other precious metals. The greatest advantage of our separation method over all those previously proposed is that coloured precipitates are produced, the colour of the tantalum precipitate showing at a glance whether it is free from, or contaminated with, niobium.

CHEMISTRY OF THE PROCESS.—Oxalotantalic acid is stable only in presence of a certain amount of free oxalic acid, partial or complete neutralisation causing precipitation of tantalic acid. Oxaloniobic acid is much more stable: Russ (*Zeitsch. anorg. Chem.*, 1902, **31**, 42) observed that its dilute solution was not precipitated by ammonia, and attempted a separation of tantalum from niobium in this manner. He found, however, that niobium was precipitated completely in presence of oxalotantalic acid.

Now we have observed that tannin favours the dissociation of oxalotantalic acid in the boiling, slightly acid solution; the precipitate that falls under these conditions is not white tantalic acid, but the sulphur-yellow tannin adsorption complex. Oxaloniobic acid is dissociated in presence of tannin only at a much higher concentration and lower acidity, with precipitation of the bright vermilion adsorption complex. The dissociation is a reversible reaction: addition of oxalic acid to the boiling solution causes the precipitates to redissolve, the niobium precipitate easily, to form a colourless solution, whereas the tantalum precipitate is only redissolved at a much higher hydrogen ion concentration, to form a yellow solution; addition of dilute sulphuric acid to the boiling oxalate solution has the same effect. We assume that our procedure converts the niobic oxide into the comparatively stable oxaloniobate, $Nb_2O_5.3(NH_4)_20{\cdot}6\,C_2O_3.3H_2O$, prepared and described by Russ (*loc. cit.*), tantalic oxide being apparently unable to form an analogous compound.

When a solution containing both tantalum and niobium in the form of their oxalo-complexes is boiled with tannin, tantalum is precipitated either alone or mixed with more or less niobium, according to the tantalum, niobium, and hydrogen ion concentration. Now the colour of the tantalum precipitate is utterly different from, and much paler than, that of the niobium compound, and the vermilion colour of the latter is sufficiently intense to betray the presence of a small admixture of niobium in a precipitate by a more or less pronounced orange tint easily distinguishable from the decided sulphur-yellow of the pure tantalum compound. The coloration of the precipitate from a mixed solution therefore acts as a sensitive indicator of the purity or composition of the precipitate.

SOURCES OF ERROR.—The method is subject to two causes of error: (1) Contamination by adsorption, unavoidable in all cases in which a precipitate is formed by the flocculation of an intermediate colloidal phase; and (2) the narrow interval between complete precipitation of tantalum and incipient precipitation of niobium, which was to be expected from the close similarity in the chemical deportment of the two elements, and the most difficult part of our investigation proved to be the adjustment of the acidity necessary for a quantitative separation. When the tantalum concentration is high and the niobium concentration low, the interval is sufficiently wide for a satisfactory separation, but at low tantalum and high niobium concentrations the two stages generally overlap, and the whole of the tantalum cannot be precipitated without considerable contamination with niobium. Such precipitates, the colour of which is orange to red, are to be regarded as concentration products which yield to a repetition of the treatment.

FRACTIONAL PRECIPITATION.—In order to counteract these causes of error, we operate by fractional precipitation in two or three stages. We propose using the following notation for designating all the possible fractions; it may be pointed out that in actual practice it is never necessary to obtain all these fractions in the course of one and the same analysis. Thus, for a very small amount of tantalum requiring fractionation at the P^3 stage, the first precipitation can be done in a single fraction P^1, and the first re-treatment in one or two fractions, P^2 and P^{2a}; whilst a mixture rich in tantalum pentoxide is fractionated without re-treatment, to ΣP^1.

	Tantalum Fractions.			
	Main.	Second.	Third.	Sum of Fractions.
First precipitation	P^1	P^{1a}	P^{1b}	ΣP^1
Re-treatment of ΣP^1	P^2	P^{2a}	P^{2b}	ΣP^2
Re-treatment of ΣP^2	P^3	P^{3a}	P^{3b}	ΣP^3

For the clearer understanding of the procedure, two cases will be considered:

(1) *The tannin precipitate P^1 is yellow*, that is, free from niobium (indicating high tantalum, low niobium, and/or high hydrogen ion concentration). In this case the precipitation of the tantalum is not quite complete, but we always endeavour to eliminate the greater part as a pure yellow precipitate, P^1, free from niobium before completing the separation. The reason for this procedure is that the operator is guided in the subsequent adjustment of the acidity, required for complete separation, by a delicate change of colour—like the end point of a titration—from light yellow to a faint orange tinge; this colour change is masked or obscured if the yellow precipitate is at all substantial, and attempts at completing the separation in one operation generally lead to high tantalum results, the first change of colour of the precipitate indicating incipient precipitation of the niobium.

The filtrate from the yellow precipitate generally shows a more or less decidedly yellow colour, indicative of the tantalum remaining in solution. Neutralisation is effected at boiling heat with dilute ammonia, added drop by drop, with constant stirring; each drop produces an orange coloration, which disappears on stirring, as long as the liquid is sufficiently acid. In our earlier test separations we continued the addition of ammonia until a barely perceptible colour change occurred; but this mode of working proved somewhat uncertain, especially as our work had to be carried out by artificial light. After much investigation it occurred to us to depress the hydrogen ion concentration by increasing that of the oxalic ions, and this procedure was found to be more reliable. The boiling yellow filtrate, after approximate neutralisation with ammonia as above, is treated gradually with saturated ammonium oxalate solution until the precipitate, P^{1a}, flocculates and the supernatant liquid is practically colourless. It is advisable to test for complete precipitation by re-treating the filtrate in the same manner with ammonium oxalate (for P^{1b}).

(2) *The tannin precipitate* P^1 *is orange to red,* that is, contains niobium (indicating low tantalum, high niobium, and/or low hydrogen ion concentration). At high niobium concentrations, contamination with niobium of the tannin precipitate does not necessarily imply complete precipitation of the tantalum (see Exps. 7, 19, and 20, in which P^1, although orange-coloured, weighed less than the Ta_2O_5 taken); it is therefore safest to produce a secondary precipitate, P^{1a}, by further neutralisation. As the combined precipitates require re-treatment, careful adjustment of the acidity by ammonium oxalate is not necessary, but it is always advisable to add the ammonia very gradually to the boiling solution and observe the depth of colour of the precipitate at the time of its formation; this may give an indication of the presence of noticeable quantities of tantalum.

It must be borne in mind that the filtrate from the precipitate P^{1a} should be colourless, though sometimes it may be of a straw tint (whether due to an alteration product of the tannin or to traces of the niobium-tannin complex we have so far been unable to decide), different from the pure yellow tantalum colour; nor should the filtrate be orange. This indicates excess of tannin, which causes part of the oxaloniobic acid to dissociate and form the colloidal adsorption complex which does not flocculate at the prevailing degree of acidity and, in the deflocculated condition, appears to act as a protective colloid in preventing complete flocculation of the tantalum complex. This occurred in Exp. 18, which gave a relatively large negative error. We have observed that the colour of the tannin precipitate after flocculation is always the same as the colour produced in the solution by the addition of tannin and neutralisation: this proves that the precipitates are formed from an intermediate colloidal phase. Addition of ammonium chloride favours flocculation.

The combined orange-coloured precipitates are ignited and again treated as the original mixed oxides, yielding a yellow precipitate, P^2, containing the bulk of the tantalum free from niobium, and the remainder of the tantalum as a small secondary precipitate P^{2a} after treatment of the filtrate with ammonia and ammonium oxalate as before. The filtrate from P^{2a} is again neutralised and treated with ammonium oxalate: should any tantalum still remain in solution, it will be obtained as P^{2b}. In most cases the precipitates, ΣP^2, are free from niobium; occasionally (where the niobium is exceptionally high) a third treatment (giving ΣP^3) is necessary. The necessity for such is shown by the colour of ΣP^2, which will then be orange instead of yellow.

We have ascertained that gallic acid (present as an impurity in commercial gallotannin) does not precipitate the oxalo-complexes of the earth acids.

The above observations led to the formulation of the following directions for carrying out the separation:—

The Separation.—About 0·25 to 0·5 grm. of the mixed oxides is accurately weighed, and fused with 3 to 6 grms. of previously dehydrated potassium pyrosulphate in a silica crucible. The quantity of mixed oxides taken for the separation is increased with the niobium content, and is adjusted to a maximum of 0·25 grm. of tantalic oxide; if this quantity is much exceeded, the voluminous precipitate becomes inconveniently large. In case any difficulty is experienced in obtaining a clear melt, the fused mass is allowed to cool, about one c.c. of strong sulphuric acid is added, and the mass again fused. If the oxide mixture becomes yellow while hot and is easily attacked by the flux, preponderance of niobium is indicated. The reverse is the case if the mixed oxides remain white on being heated and prove more refractory.

The cold melt is leached by boiling with a saturated solution of 2 to 4 grms. of ammonium oxalate, any lumps of earth acids being subdivided by means of a flattened glass rod. If solution is difficult (with high tantalum contents) this may be due to excessive expulsion of sulphur trioxide in the fusion; solution is facilitated by addition of a few drops of sulphuric acid.

This treatment generally results in a clear solution free from residue; if not, the hot solution is filtered into a 500 c.c. beaker, and the residue collected and washed with hot water. Filter and residue are returned to the silica crucible, ignited, and the residue again fused with a little pyrosulphate. The mass is leached with a suitable proportion of ammonium oxalate solution, and the solution thus obtained added to the main filtrate. It may be advisable to filter this second extraction liquor through a small filter, as a visible residue of silica (derived from the crucible or the flux), and possibly calcium oxalate, is sometimes left.

The solution now contains the whole of the two elements as oxalo-acids and a certain amount of free oxalic acid (or acid oxalate) resulting from the action of the acid flux on the ammonium oxalate. The degree of acidity of the solution therefore varies with that of the melt at the conclusion of the fusion, whilst the most suitable acidity for the separation depends on the relative proportions of the two elements in the solution.

The first step in the separation consists in the adjustment of the acidity. The solution containing 0·1 grm. of oxides in 100 to 150 c.c., is heated to boiling and treated with 10 c.c. of freshly-made 2 per cent. tannin solution. This may produce a permanent turbidity or precipitate after short boiling; if not, the boiling solution is titrated, while being vigorously stirred, with 0·5 *N* ammonia until a permanent turbidity is produced. The tannin here acts as an indicator both for the acidity and the relative proportions of the two metals; the appearance of a turbidity indicates that neutralisation has proceeded to the desired point. Further, an orange or reddish coloration proves niobium to be present in excess, whilst a sulphur-yellow turbidity or precipitate shows tantalum pentoxide to form one-third (roughly speaking) or more of the oxide mixture.

The acidity having been adjusted, more tannin solution is added, if necessary, to the boiling liquid. The quantity may be said to vary from ten times the amount of Ta_2O_5 present, if small (0·2 grm. of tannin being a minimum in all cases), to about five times the amount if large; in practice, therefore, the total amount of tannin required would be judged as follows:—

For less than 0·03 grm. Ta_2O_5: 0·2 grm. tannin.
For 0·03 to 0.06 grm. Ta_2O_5: 0·3 to 0·4 ,, ,,
,, 0·06 ,, 0·12 ,, ,, : 0·5 ,, 0·6 ,, ,,
,, 0·12 ,, 0·18 ,, ,, : 0·7 ,, 0·8 ,, ,,
,, 0·18 ,, 0·25 ,, ,, : 0·9 ,, 1·0 ,, ,,

A saturated solution of 5 grms. of ammonium chloride is next added to the solution, which is kept boiling for 10 to 15 minutes after flocculation has set in. One of two cases will now present itself:

A. The precipitate, P^1, is orange to red (see Procedure A).
B. The precipitate, P^1, is sulphur-yellow (see Procedure B).

PROCEDURE A.—The precipitate, as the result of the above manipulations, should be completely flocculated, and the solution, though sometimes of a pale straw tint, should be free from any orange colour or cloudiness. If not, boiling is continued, with the gradual addition of 0·5 N ammonia drop by drop, until the above conditions are realised. The precipitate is collected on loose paper and washed with 2 per cent. ammonium chloride solution. The filtrate should be clear and colourless (or of a faint straw tint) if the preceding directions have been properly carried out; it is again heated to boiling and treated with 5 to 10 c.c. of the tannin solution; this will produce an orange to red colloidal solution, which is decolorised, with separation of a flocculent precipitate, P^{1a}, of the same colour, by addition of 0·5 N ammonia drop by drop. This time the precipitate is digested on the waterbath for some time, set aside overnight, then collected and washed as before. The two wet precipitates, which contain the whole of the tantalum, are ignited together in a silica crucible. The ignited oxide mixture is weighed as a guide, after which it is fused with pyrosulphate and again submitted to the whole treatment as far as "Procedure A"; the precipitate will now be yellow, and the separation is continued as described below (Procedure B).

PROCEDURE B.—The beaker is set aside for a short time until the precipitate has settled; the hot liquid is decanted through a loose filter, on which the precipitate is collected and washed with 2 per cent. ammonium chloride solution. As soon as the precipitation vessel is washed substantially free of precipitate, the filtrate is returned to it and boiled down to about three-quarters its original bulk. The washings and a few c.c. of the tannin solution are added; boiling is continued, and 0·5 N ammonia added, drop by drop, until the orange coloration produced around the drops disappears only slowly; a saturated solution of ammonium oxalate is then added from time to time in portions of 25 to 10 c.c. until the yellow precipitate (P^{1a} or P^{2a}) has coagulated and the solution is clear and colourless. The liquid is preferably left for some time on the water-bath, then set aside overnight to allow the last of the tantalum precipitate to flocculate. The precipitate is collected and washed as before, the combined wet precipitates ignited gently at first, then over a powerful burner, and weighed as Ta_2O_5.

The filtrate from the second precipitate, which should not be yellow (it may have the faintest yellow tinge) is tested for complete precipitation by a repetition of the ammonium oxalate stage of Procedure B, after previous addition of a drop or two of 0·5 N ammonia, if necessary. If a further small yellow precipitate is obtained, its weight, after ignition, is added to that of the Ta_2O_5; if the precipitate is orange-coloured, the separation may be considered complete.

Care should be taken to avoid the presence of lime as an impurity in the water and reagents used, as it would contaminate the tantalum precipitate. The ignited precipitate may be leached with hot dilute hydrochloric acid and again ignited and weighed, as a test for the absence of lime or alkali.

PROCEDURE C.—This is a combination of the preceding, and presents advantages in cases in which both oxides are present in substantial amounts; it can be applied as an alternative to Procedure A, and may also be used at discretion in doubtful cases. It consists in obtaining the greater part of the tantalum, free from niobium, as the sulphur-yellow precipitate, P^1, which is ignited and weighed. The balance of the tantalum is then precipitated, together with more or less niobium, as the orange precipitate, P^{1a}, which is ignited and re-treated by Procedure B; the remaining tantalum is thus obtained pure as ΣP^2.

In the application of Procedure C, if the addition of 0·2 grm. of tannin to the boiling solution produces an orange-coloured liquid, N sulphuric acid is cautiously added, drop by drop, until the orange coloration has given place to a pure yellow one. The remainder of the tannin is then added and followed by the ammonium chloride. The yellow precipitate is ignited, weighed, and eliminated from the analysis. The filtrate is boiled and titrated with 0·5 N ammonia for the production of the orange precipitate, P^{1a}, which is ignited and purified by Procedure B.

The advantage of Procedure C is that the whole of the tantalum does not have to be re-treated; the small quantity is more easily fused, and the neutralisation in a smaller bulk of solution decreases the chances of error either from incomplete precipitation of the tantalum or co-precipitation of the niobium.

DETERMINATION OF THE NIOBIUM.—The direct determination of the niobium presents no advantages over the determination by difference between the weight of mixed oxides taken and the tantalic oxide found, especially where the niobium content is high. For want of time we have carried out but few direct niobium determinations. For this purpose, the combined filtrates from the tantalum precipitates are made feebly ammoniacal, and heated to boiling. The red precipitate, which is discoloured by the oxidation products of tannin in the alkaline medium, is collected, washed, and ignited, like the tantalum fraction. In Exp. 4, a quantity of niobic oxide unknown to the operator was fused with 4 grms. of pyrosulphate, dissolved in a solution of 2 grms. of ammonium oxalate, and precipitated with 0·5 grm. of tannin, 5 grms. of ammonium chloride, and a small excess of ammonia; the ignited precipitate weighed 0·1114 grm., against 0.1108 grm. taken.

The ignited niobic oxide should be yellow while hot and white after cooling. A buff coloration of the cold oxide points to slight contamination with iron, always liable to be present or introduced as an accidental impurity, and precipitated by the ammonia. In such a case, the ignited precipitate must be purified by fusion with pyrosulphate and hydrolysis of the leach liquor with thiosulphate. The ferric oxide in the precipitate may also be determined by fusion with pyrosulphate, leaching with tartaric acid, and precipitation of ferrous sulphide from ammoniacal solution.

RESULTS OF TEST ANALYSES.—All the test analyses shewn below are those of unknown mixtures, that is to say, oxide mixtures the composition of which was kept secret from the operator until he had completed the separation. Further, the list of these tests is a complete one; in other words, it does not represent a selection of the most favourable results. In order not to complicate the table, the weights of the P^a and P^b fractions (where obtained, and weighed separately) are not included, but the weights of the main fractions, where ascertained, are given.

In Exp. 6, no niobic oxide had been added. The filtrate from P^1 was boiled with a slight excess of ammonia, yielding 0·0012 grm. of a ferruginous precipitate. This was submitted to micro-analysis in a cup, weighing 0·18 grm., made from platinum foil. Found, 0·0003 SiO_2, 0·0006 Fe_2O_3, and 0·0003 grm. Ta_2O_5. The yellow tannin precipitate was obtained with the utmost sharpness in 3 c.c. of solution.

		Taken							
Exp.	Procedure	Nb_2O_5 grm.	Ta_2O_5 grm.	P^1 grm.	ΣP^1 grm.	P^2 grm.	ΣP^2 grm.	ΣP^3 grm.	Error grm.
1	A	0·1968	0·0557	—	—[1]	0·0539	0·0586	—	+0·0029
2	B	0·1025	0·1941	0·1854	0·1909	—	—	—	-0·0032
3	B	0·1549	0·1172	0·1119	0·1159	—	—	—	-0·0013
5	A	0·0956	0·1257	0·1412	0·1677	—[1]	0·1260	—	+0·0003
6	B	0·0000	0·1049	0·1049	0·1052	—	—	—	+0·0003
7	A	0·1041	0·1583	0·1518	0·1674	0·1532	0·1620	—	+0·0037
8	A	0·1950	0·0609	—	—[1]	0·0502	0·0597	—	-0·0012
9	A	0·2140	0·1058	0·1129	0·1177	0·0944	0·1051	—	-0·0007
10	B	0·0251	0·2049	0·1618	0·2056	—	—	—	+0·0007
11	A	0·1207	0·0469	—	—[1]	0·0383	0·0463	—	-0·0006
12	C	0·0763	0·2101	0·2013	—	0·0112[2]	0·1081	0·2094[3]	-0·0007
13	B	0·0982	0·1099	0·1053	0·1091	—	—	—	-0·0008
14	B	0·0720	0·2197	0·1920	0·2193	—	—	—	-0·0004
15	A	0·1966	0·0128	—	0·0136	—	0·0120	—	-0·0008
16	B	0·1010	0·0953	0·0857	0·0953	—	—	—	0·0000
17	A	0·4568	0·0024	—	0·0053	—	0·0025	—	+0·0001
18	A	0·3012	0·0066	0·0131	0·0131	—	0·0075	0·0053	-0·0013
19	A	0·2048	0·0535	0·0447	0·0633	0·0525	0·0545	—	+0·0010
20	A	0·2138	0·0563	0·0544	0·0687	0·0463	0·0558	—	-0·0005
21	B	0·0206	0·1286	—[1]	0·1293	—	—	—	+0·0007
22	A	0·1070	0·0368	0·0423	0·0488	—[1]	0·0394	—	+0·0026
23	A	0·1576	0·0433	0·0673	0·0880	0·0413	0·0458	—	+0·0025
24	A	0·1709	0·0284	0·0354	0·0397	—[1]	0·0289	—	+0·0005
25	C	0·2092	0·1010	0·0949	—	0·0105[2]	0·0081	0·1030[3]	+0·0020

[1] Not weighed. [2] This is P^{1a}, which was re-treated alone, P^1 being pure. [3] This is $P^1 + \Sigma P^2$.

Accuracy. The magnitude of the error in the 23 test separations tabulated above is as follows:—

Error not exceeding 0·0010 grm. : 14 cases.
„ „ „ 0·0020 „ : 4 „
„ „ „ 0·0030 „ : 3 „
„ „ „ 0·0040 „ : 2 „

The two tests in which the error exceeds 0·0030 grm. are Exp. 2 and 7, closely followed by Exp. 1 with an error of +0·0029 grm.; they are early tests in which the separations were carried out by neutralisation with ammonia only, and the errors are fairly typical of those usually obtained while the method was still undergoing improvement. Since these relatively large errors do not recur in the later tests, we submit that the three results in question may be disregarded while the accuracy of the method is under consideration. If this is granted, an accuracy equal to that reached by standard analytical methods (that is, an error not exceeding 0·001 grm.) was obtained in 70 per cent. of the number of test separations. The error is liable to be greater where the precipitates ΣP^1 undergo re-treatment, and the next highest errors occur accordingly in Procedures A (Exp. 22 and 23) and C (Exp. 25). Our conclusion is that with high niobium and low tantalum percentages the maximum error may reach, but need not exceed 0·0025 grm., and this can be halved if duplicate or triplicate determinations are made; with low niobium and high tantalum percentages, on the other hand, the degree of accuracy obtained compares quite favourably with that of the standard methods of analytical chemistry.

CHECK TESTS CARRIED OUT IN THE U.S. BUREAU OF STANDARDS.—During the past year the authors were fortunate in reaching a co-operative agreement with the late Dr. W. F. Hillebrand, Chief Chemist of the Bureau of Standards, Washington, for the purpose of accelerating and extending the analytical research work on tantalum, niobum, and their mineral associates. After Dr. Hillebrand's death, the work was continued by his associate, Dr. G. E. F. Lundell. Upon the conclusion of their test analyses, the authors forwarded a description of the present method to Dr. Lundell, who was good enough to test it in collaboration with Mr. H. B. Knowles. We desire here to acknowledge our indebtedness to Dr. Lundell and Mr. Knowles for the very valuable assistance they have rendered us in so doing. In Dr. Lundell's words, the process is "most excellent and a decided improvement over the older methods." The results obtained by the American chemists are given below; they completely confirm our conclusions, recorded above, as to the accuracy of the method.

	Taken						
Exp.	Nb_2O_5	Ta_2O_5	P^1	ΣP^1	P^2+P^{2a}	ΣP^2	Error
1	0·1022	0·1017	0·0785	0·1053	0·0966	0·1015	−0·0002
2	0·1011	0·0119	0·0149[1]	0·0167	0·0132	0·0150	+0·0031
3	0·0112	0·1012	0·0917	0·1037	0·0921	0·1003	−0·0009
4	0·1519	0·1516	0·1399[1]	0·1546	—	—	+0·0030
5	0·1547	0·0117	0·0140	0·0157	—	—	+0·0040
6	0·0103	0·1532	0·1430[1]	0·1514	—	—	−0·0018

[1] This is P^1+P^{1a}.

Behaviour of other elements.—In a mineral analysis the oxides of tantalum and niobium must be separated quantitatively from other elements prior to their separation from each other by the above method. We have tested the behaviour towards tannin of a few other elements when in solution as oxalo-acids, with the following results. Zirconium and tungsten are not precipitated in slightly acid solution. Antimony (both in the antimonious and antimonic condition) gives a heavy, white precipitate. Titanium in acid solution gives a red precipitate resembling the niobium compound; it interferes, therefore, in this as in other processes, and the separation of tantalum from niobium must of necessity be preceded by their separation from titanium. This difficult problem is at present under investigation, in co-operation with Dr. Lundell and Mr. Knowles. We also intend studying the applicability of tannin to certain other separations.

Summary.—A new method for the separation of tantalum from niobium is described. It is based upon differential hydrolytic dissociation between oxalotantalic and oxaloniobic acids in presence of tannin in slightly acid solution. The method possesses the following advantages. The colour of the tantalum precipitate indicates at a glance whether it is free from, or contaminated with, niobium; no platinum vessels are required; and the accuracy approximates to that reached in the separation of most of the common elements.

V.—THE DETECTION AND DETERMINATION OF TANTALUM IN NIOBIUM COMPOUNDS.

The reducibility of niobium pentoxide to a strongly-coloured lower oxide (Giles, *Chem. News*, 1907, **95**, 1; Meimberg, *Zeitsch. angew. Chem.*, 1913, **26**, 83) is a convenient means for the detection of a small admixture of niobium in tantalum compounds. On the other hand, the detection of little tantalum in presence of much niobium is very uncertain, if undertaken by methods other than spectrography. The late Dr. Hillebrand informed us privately that by the spectrographic method "it is quite possible to detect very little of either columbium or tantalum in the presence of much of the other, certainly as little as 0.01 per cent., but that it is difficult to ascribe a quantitative figure, *i.e.* whether 0·1 or 0·001 per cent. is present."

As regards wet tests, the production of the crystalline precipitate of potassium fluotantalate is somewhat uncertain, owing to the possibility of the separation of the isomorphous fluoniobate, K_2NbF_7; further, if the $Nb_2O_5 : Ta_2O_5$ ratio of the mixture under investigation is higher than 25, the solution, upon evaporation, will deposit crystals of potassium fluoxyniobate before any separation of fluotantalate occurs. In such a case it is necessary to examine the dry crystals microscopically and sort out the needles, if possible, for a confirmatory test.

A reaction that has been advanced as the most sensitive for small quantities of tantalum is the precipitation of the fluoxytantalate, $K_4Ta_4O_5F_{14}$, a white insoluble powder produced by boiling a solution of potassium fluotantalate. Meyer and Hauser (*Die Analyse der seltenen Erden und der Erdsäuren*, p. 185) consider it to be the most reliable tantalum reaction; yet, on another page (188) they state that there is no rapid specific reaction for that metal. Krüss and Nilson (*Ber.*, 1887, **20**, 1676) proved that potassium fluoxyniobate free from tantalum gives a similar insoluble compound when its solution is boiled. In our own experience, if an insoluble white powder is obtained when the mixed complex fluorides are boiled with water, some niobium is found in the precipitate and a good deal of tantalum in the filtrate, even after the salt mixture has been kept at 170°C. for several hours; further, the tantalum is not completely removed from the niobium fraction after several repetitions of the procedure, contrary to Hall's statement (*J. Amer. Chem. Soc.*, 1904, **26**, 1239). The reaction must be rejected as unreliable.

Our tannin reaction, described in the preceding section, may be used, not only for the detection, but also for the quantitative determination in niobium compounds, of small quantities of tantalum. The test has not the rapidity of a test tube reaction, as one or more re-precipitations may be required for small quantities; but, unlike any other tantalum test known to us, we claim it to be reliable if carefully made. It has the great advantage of being based on a colour reaction, and is also sensitive, as we have detected one part of tantalum pentoxide in several thousand parts of niobium pentoxide; there appears to be no exact limit to the sensitiveness, which increases with the quantity of sample taken for the test. The tannin reaction is included among the tests for tantalum and niobium in most textbooks on qualitative analysis, but the directions given apply merely to the identification of either metal in the state of a pure compound. The reaction as carried out by the procedure here described will undoubtedly provide the most reliable means for the characterisation of the two metals in admixture.

IDENTIFICATION OF TANTALUM AND NIOBIUM.—The oxide mixture to be tested must be free from titanium; a little zirconium does not interfere. About 0·05 to 0·2 grm. is fused with potassium pyrosulphate in a silica crucible. The cold mass is dissolved by boiling in 50 c.c. of ammonium oxalate solution; a few c.c. are tested for freedom from titanium with hydrogen peroxide. The remainder of the solution is treated, while boiling, with 0·2 to 0·3 grm. of tannin followed by 0·5 N ammonia, drop by drop, if necessary, till a flocculent precipitate is formed. If the precipitate is pale yellow to bright yellow, the presence of tantalum is indicated. If, however, the precipitate is orange to red, niobium is present, whilst tantalum is either absent or present in a more or less subordinate amount. In such a case the precipitate, after being filtered off and washed, is rinsed back, boiled with 25 c.c. of oxalate solution, and dissolved by gradual addition of N sulphuric acid; when the solution has cleared, 0·1 to 0·2 grm. of tannin is added, boiling is continued, and 0·5 N ammonia added, drop by drop, until a flocculent precipitate is again obtained. This will now be yellow if the amount of tantalic oxide present is not too small; if again orange, the precipitate should be filtered off, and the treatment repeated once more. For the detection of traces of tantalum in niobium compounds, it is necessary to proceed as for the quantitative determination described below, under "Procedure for Traces of Tantalum."

In presence of much tantalum, niobium is readily detected in the filtrate from the yellow tannin precipitate; the liquid is boiled, treated with a little more tannin, and a slight excess of sodium or ammonium acetate. The oxalic acid is thus converted into oxalate, the oxaloniobic acid undergoing dissociation, with formation of the vermilion tannin adsorption complex. For qualitative work this procedure is preferable to addition of excess of ammonia, which leads to discoloration of the precipitate. On the other hand, quantitative precipitation of the niobium must be effected with ammonia (see Section IV.).

QUANTITATIVE DETERMINATION OF SMALL AMOUNTS OF TANTALUM IN NIOBIUM COMPOUNDS.—The most important niobium compound is the pentoxide, the tantalum determination in which is carried out according to the directions given in Section III. for the analysis of mixed oxides; with this difference, that much more material may have to be taken if the percentage of tantalum is very low, and that two to three re-precipitations may be required for the complete elimination of the niobium.

Procedure for Traces of Tantalum.—Several one-grm. portions are weighed into silica crucibles, each of which contains 5 grms. of previously dehydrated potassium pyrosulphate. (10 grms. of material should be taken if the Ta_2O_5 is of the order of about 0·02 per cent.). Each fused mass is dissolved in 100 c.c. of saturated ammonium oxalate solution, diluted to 400 c.c., and boiled with 0·2 grm. of tannin and 5 grms. of ammonium chloride; neutralisation is unnecessary unless the precipitate does not flocculate. After standing overnight the precipitates are collected, washed with 2 per cent. ammonium chloride solution, ignited, combined, and weighed. This concentrate (in two portions if over 1 grm.) is again submitted to the same process, yielding the precipitate P^2; the latter is once more fused with a smaller quantity of pyrosulphate, etc., and the resulting solution precipitated with 0·1 grm. of tannin, or somewhat less. The product of the last treatment, ΣP^3, will generally be more or less yellow, but should be purified by a fourth treatment, in which the oxalate solution of the fusion is treated with a few drops of dilute sulphuric acid, boiled, and filtered through a very small paper to eliminate calcium oxalate, silica, and any insoluble impurities accumulated from the large quantity of reagents used in the process. The clear filtrate (50 c.c. or less) is precipitated with a suitable quantity of tannin (5 times ΣP^3) and ammonia, giving ΣP^4 which, if yellow, is ignited and weighed as Ta_2O_5. We have not found it necessary to proceed beyond the P^4 stage.

In the above process it would be a mistake to work on portions of more than 1 grm. and add less than 0·2 grm. of tannin for the production of P^1, in an endeavour to expedite the work. We have proved that, if a deficiency of tannin is added to a solution of high niobium concentration, the precipitation of the tantalum is generally incomplete, as the large excess of niobic acid adsorbs the tannin required for the formation of the tantalum complex.

The subjoined Table contains the results of analyses of three preparations of niobic oxide. In the case of sample A, 1 grm. was treated with 5 grms. of pyrosulphate and 4 of oxalate, and P^1 precipitated with 0·25 grm. of tannin; P^1 was red, P^2 a faint orange, and ΣP^3 yellow. The analysis of "mixed salt," D, is that of a preparation obtained by the crystallisation of a solution containing potassium hexatantalate and hexaniobate. A weighed quantity of the salt was dissolved in water, and an aliquot part of the solution treated with filter pulp and boiled with a faint excess of hydrochloric acid, followed by ammonia till just alkaline. The precipitate was ignited, weighed, and treated according to Procedure B, Section IV.

For the determination of tantalum in potassium fluoxyniobate, one or more 2 grm. portions are evaporated with excess of concentrated sulphuric acid in platinum or gold basins until copious fumes are evolved; after the complete expulsion of the hydrogen fluoride, 3 grms. of potassium sulphate are added, and the excess of sulphuric acid is expelled by heating. The fused mass is then treated as above.

Material	No.	Taken grm.	P^1 grm.	ΣP^2 grm.	ΣP^3 grm.	ΣP^4 grm.	Ta_2O_5 per cent.
A. Nb_2O_5	1	1·0000	—[3]	0·0608	0·0472	—	4·72[1]
	2	1·0046	0·113	0·0490	0·0465	—	4·63[1]
	3	1·0088	0·110	0·0481	0·0467	—	4·63[1]
	4	1·0000	0·152	0·0534	0·0474	—	4·74[2]
	5	1·0000	0·130	0·0522	0·0466	—	4·66[2]
B. Nb_2O_5	1	1·0012	0·225	0·0142	0·0034	0·0014	0·14[1]
	2	0·5010	0·092	0·014	0·0030	0·0008	0·16[1]
	3	1·0000 1·0000 1·0000	0·0860 0·0567 0·0843	0·0145	0·0104	0·0047	0·157[2]
C. Nb_2O_5	1	1·000[4]	0·0296	0·0020	—[3]	0·0003	0·03[2]
	2	1·000[4]	0·052	0·0030	0·0020	0·0005	0·05[2]
D. Mixed Salt.[5]	1	0·5000	0·1568[6]	—	—	—	31·36[1]
	2	0·5000	0·1565[6]	—	—	—	31·30[1]

Analyst.—[1] A.R.P. [2] W.R.S. [3] Not weighed. [4] Not enough material available for larger tests. [5] Nb_2O_5 content by difference: 21·40 and 21·26 per cent. [6] This is ΣP^1.

SUMMARY.—The inadequacy of the usual qualitative tests for tantalum is discussed. The tannin method for the separation of tantalum from niobium is shown to be applicable to the detection as well as the determination of small quantities of tantalum in niobium compounds, and to the certain identification of the two elements in admixture by the production of precipitates of characteristic colour.

THE SIR JOHN CASS TECHNICAL INSTITUTE,
ALDGATE.

DISCUSSION.

Mr. C. A. MITCHELL asked the authors what reasons they had for concluding that their precipitates were adsorption complexes and not definite compounds. By using a tannin free from glucose it might be possible to prepare definite tannates of tantalum and niobium.

Mr. A. Chaston Chapman congratulated the authors on their paper. He asked for information as to the initial separation of the oxides of these metals.

Mr. Powell, replying, said that they would be glad to make experiments with a purer tannin to see whether precipitates with approximately uniform proportions of tannin and earth acids were obtained. But, so far, their experience led them to view these precipitates as adsorption complexes. The fact was that tantalic and niobic acids, after they had been precipitated from a solution of their potassium salts by an acid, adsorbed tannin just as did hide powder, and in the authors' process the tantalum precipitate appeared to be formed by the mutual precipitation of two colloidal phases. Whilst the tannates of strongly electropositive metals (*e.g.* potassium, barium) were definite salts, the authors could not conceive how substances of weakly acidic character, such as the earth acids, could form analogous compounds. Even with fairly electropositive metals, such as nickel, Prof. Donnan and Dr. Rideal (discussing Singh's paper on nickel tannates, *J. Soc. Chem. Ind.*, 1914, **33**, 172) considered the tannin precipitates to be mixtures or adsorption complexes. The difficulty was that one was dealing with amorphous precipitates of variable or indefinite composition, which could not be purified by preparative methods.

In reply to Mr. Chapman, it was explicitly stated in the paper that the separation method was to be applied only to the pure mixed oxides of tantalum and niobium after they had been separated quantitatively from other elements. In the case of complex minerals, the separation from other elements was the larger problem, the solution of which formed the object of this series of papers. For the present, the separation of tantalum from niobium described above could be applied to pure tantalites and columbites (which contained only a few tenths per cent. of titania), the mixed oxides being obtained as directed in Section I. of this series (*loc. cit.*); the final earth acid precipitates, however, should be tested colorimetrically for titania.

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 ARACHIS OIL IN OLIVE OIL.

In the last paragraph of Mr. A. D. Powell's Note in the August number of The Analyst (p. 396), he mentions the probability of stearic acid being present in commercial olive oils of to-day. If this is so, it may be a contributory cause of a precipitate being formed in Lüer's modification of Bellier's test at a temperature above 16 or 17° C.

Some time ago I carried out the test on three samples of oil, one (No. 1) without substitution, one (No. 2) with the substitution of 0·05 grm. of commercial stearic acid (m.pt. 53·6° C.) for the olive oil prescribed, and one (No. 3) with the substitution of 0·1 grm. of the stearic acid, and I obtained the following results:—No. 1, Perfectly bright at 15·5° C.; No. 2, Separation took place at 16° C.; No. 3, Separation at 19° C.

F. F. Shelley.

THE COMPOSITION OF LEMON CHEESE.

I AM afraid thas Mr. T. R. Hodgson in his note which has recently appeared (ANALYST, 1925, 396) has fallen into error in saying that I have attempted to set up my own standard for commercial "Lemon Cheese." The paper printed in THE ANALYST was really not intended for publication in its then form, and the points are probably made clearer in the fuller report which has since been issued; but the formula which I gave is quite distinctly described as "The composition of the article domestically known as lemon cheese," and later on I say: "It is quite obvious that such products (*i.e.* those which Mr. Hodgson admits to be unsatisfactory) as these have little in common with the article known to the housewife as lemon cheese. Even if it is admitted that there is no rigid standard for lemon cheese, there must, of necessity, come a time when a product of any nature can no longer claim that title; the question to be decided is in regard to the point when this time arrives." In all this, it should be quite obvious that I am not fixing a standard, but merely making a statement of fact as to what "lemon cheese" has been. Mr. Hodgson evidently considers that manufactured articles need not be as good as articles made in the home and described in the same manner—it was with the idea of getting the Society of Public Analysts to move one way or another in this matter that the subject was approached; the paper was intended as evidence, not as a judgment.

It is curious that Mr. Hodgson quotes *Sandys* v. *Rhodes* and *Anderson* v. *Britcher* (with both of which cases, I am, of course, quite familiar) against *me,* for I consider that they are entirely against *him.* "Lemon cheese" is not considered by the public to be an ordinary cheese flavoured with lemons, but a conserve prepared from sugar, butter, eggs, and lemons, and therefore, according to these decisions, the latter and not a flavoured cheese should be supplied in answer to a demand for "lemon cheese." Mr. Hodgson will agree that "known commercially" does not refer to some inner private knowledge of some particular trade, but what is usually accepted as the basis of contract between retailer and public. At least one stipendiary magistrate had laid this down very definitely, and Mr. Hodgson will doubtless recall the case in which the High Court has stated the principle that no trader or body of traders may fix its own standards in matters of this kind.

Mr. Hodgson states that "lemon cheese" is a generic name for a substance known to the public as consisting of a mixture of sugar, edible fat, and egg substance, flavoured with lemon. If "egg substance" here means eggs, and "edible fat" butter, then I agree, but if this means that margarine may be used without disclosure, then, in my opinion, it is quite improper that the same name should be used for an article prepared, on the one hand, from butter, and, on the other hand, from a substitute. In any case, it will be noticed that Mr. Hodgson does not bring one scrap of evidence to support this definition, whereas in my own report some six foolscap pages of typescript are taken up with this point.

Sub-section (1) of Section 6, has not been overlooked—it was actually quoted in a report to my Committee—but the whole Sub-section has been considered, and not only the first portion. Moreover, contrary to the opinion of Mr. Hodgson, the presence of starch is not necessary, as an examination of commercial samples will quite easily prove, and he will also remember that the burden of proof is on the defendant (Sec. 24), so that he need not, as a Public Analyst, be unduly worried on this point. Sentences from my previously issued reports on these subjects read as follows:—"It may be agreed later that a small quantity" (of glucose syrup) "is not particularly objectionable." "It has been suggested that a trace of farinaceous material, such as cornflour, may be an improvement in certain cases."

Mr. Hodgson apparently assumes that, because he has actually seen the manufacture of "lemon cheese" on the commercial scale, he is therefore the final authority on the subject (page 397, para. 2). I have in my possession many commercial samples of "lemon cheese" which contain neither starch nor glucose, and before me as I write is such a sample made by a manufacturer six years ago and which, apart from a little crystallisation on the surface, is still of quite good consistence. Surely Mr. Hodgson will not argue that because certain individuals cannot do certain things, therefore these are impossible! The history of the Sale of Food and Drugs Acts affords a complete refutation of any such idea.

Mr. Hodgson states that lemon oil is generally used in place of lemon. It is for the public to judge whether they are satisfied with artificially prepared mixtures in place of natural articles. Can Mr. Hodgson give his authority for assuming that lemon oil and citric acid are the dietetic equivalents of lemon juice? And is this authority unchallenged?

I regret that Mr. Hodgson has charged me with "total ignorance" on a most important matter (page 397, last paragraph), but I can safely leave this side of the question to the trade themselves. I should like to point out, however, that this "total ignorance" is certainly not due to lack of experience, as for the last eighteen months I have been almost continuously in touch with many of the largest makers of "lemon cheese" in the country, and also with retailers of real experience and understanding.

It is possibly necessary to add that I have taken the subject of lemon cheese merely as a type of the many compounded articles on the market for which there is no definite standard. Standards for such articles are extremely desirable, and, in the absence of official help, the Society as a whole should undertake the work of making suggestions for the guidance of its members.

G. D. Elsdon.

POINTS ARISING FROM THE ANALYTICAL STANDARDISATION OF BRITISH CHEMICAL STANDARDS.

As time did not allow of our replying to the criticisms on our paper (Analyst, 1925, 437) at the meeting we should like to make the following remarks:

Wherever work of the same kind has to be done repeatedly, experience has shown that it is carried out best by standardising the procedure—that is, by thinking out a pre-arranged plan and then adhering to it.

Our observations over many years have convinced us that this does not produce mechanical chemists*; in fact, obedience to fixed rules is a necessary and legitimate step which must be taken in order to lead up to more advanced work in which judgment, based on experience, may be put into practice. Moreover, dealing with the routine work by mechanicalised methods not only trains young workers to recognise the necessity of exactitude in detail, but also leaves them more time to study and practice the large number of analyses which cannot be standardised. This is a logical mode of procedure which, we should imagine, Dr. B. S. Evans is following, in the main, in his laboratory.

We have dealt more fully with "The case for Standardisation" in a pamphlet issued by us to users of mechanicalised methods; and we also send to users a sheet of "Chemical Principles" explaining the reactions, and showing the equations, expressly to impress on them the importance of understanding what they are doing.

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* Nearly 200 chemists have endorsed this conclusion by using such methods. See also 4th par., p. 432.

With regard to the disagreement as to the arsenic content of the white metal, the question of homogeneity has been dealt with on page 427; and the subsequent agreement in the final results is probably the best independent demonstration of the homogeneity.

The total in the specimen tabulation of results was expressed to three decimal places, because it seemed desirable to show an actual total of the figures above it. It might possibly be a better mode of expression to put the figures in the second and third decimal places in brackets, thus: 99·9[06], and, when convenient, make the bracketed figures smaller (*e.g.* 99·9[06]).

Regarding Mr. J. Myers' remarks, whilst we admit that, from some points of view, there is a good deal in what he said, we do not consider that this disposes of our contention.

In practice, one chemist often tests successive cargoes over long periods, and we have known of instances where substantial differences from another chemist systematically occurred, almost invariably in one direction, and such differences have actually involved large sums.

Then, as regards lime, etc., if analyses are incorrect to anything like 0·5 per cent. (not merely 0·10 per cent., as Mr. Myers mentioned), whether the blast furnace manager expected greater accuracy or not, does not alter the effect on the blast furnace working, and economy.

It may not have occurred to him that it would matter, and yet it may have accounted for some of the unexplained differences in behaviour. The blast furnace is a very sensitive implement, especially on certain classes of iron (we speak from practical experience); and in these days when the tendency is towards finer working, furnace managers cannot afford to neglect anything which may influence it.

We quite agree that the other variations mentioned introduce further difficulties, but that is no reason for ignoring those that can be lessened.

C. H. Ridsdale.
N. D. Ridsdale.

Errata.—On page 425 delete "grms." and insert "dead fine—chiefly graphite," before the word "sieved." The footnote † only applies to carbon total and carbon graphitic.

On page 436, bottom of 1st column of analysis of iron ore, for "1·45" read "1·35,"

Also on p. 428, "$Mg_2P_7O_7$" should read "$Mg_2P_2O_7$."

Notes from the Reports of Public Analysts.

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

COUNTY BOROUGH OF BOLTON.

Annual Report of the Public Analyst for 1924.

During the year 462 samples under the Food and Drugs Acts and 702 samples of water were analysed. 1004 bacteriological examinations were made for the Medical Officer of Health, 288 examinations for the Gas Engineer, and 40 miscellaneous investigations for various departments of the Corporation.

The food and drugs samples included 222 of milk (7 of condensed milk), 34 of butter, 21 margarine, 13 tea, 21 coffee, and mixtures, 12 cocoa, 12 jams, 24 cakes, 17 meat products, and 75 miscellaneous. With the exception of 10 milks, all were genuine.

CAKES.—No boric acid was found in sponge cakes and sponge fingers, but it was almost invariably present in other cakes, the quantities varying from 1·4 to 13·0 grains per lb.

Two samples of liquid eggs contained 53 and 63 grains of boric acid per lb.

SHREDDED SUET.—The four samples examined were free from preservatives, and contained from 79 to 89·08 per cent. of beef fat, and from 10·92 to 21·0 per cent. of cereal. Prior to the Food Control Recommendations (since revoked) 10 per cent. of cereal was considered adequate to keep the fat granules isolated, and this amount should still be regarded as the maximum, any excess over this limit being probably added solely for the sake of additional profit.

HARRY HURST.

CITY OF BIRMINGHAM.

REPORT OF THE CITY ANALYST FOR THE SECOND QUARTER, 1925.

THE number of samples submitted was 1403, of which 1247 were under the Food and Drugs Acts, 1215 being bought informally (44 adulterated), and 32 formally (6 adulterated).

MILK.—Of 689 samples examined, 41 were adulterated. Thirty-five of 463 samples taken from farmers, chiefly at the railway stations, were adulterated. Samples of milk from 9 farmers who sent 2 or more churns showed an irregular distribution of the fat. For example, two churns from one farmer showed 2·6 and 3·7 per cent. of fat respectively.

SYRUP.—A sample sold as "Amber Syrup" was, as usual, largely glucose syrup.

BORIC ACID OINTMENT.—A sample labelled "Prepared according to B.P." contained 12·3 per cent. of boric acid, instead of 10 per cent. The vendor was cautioned.

GREGORY'S POWDER.—The B.P. requires this drug to be composed of 22 parts of rhubarb, 12 of ginger, and 66 of light magnesia. During the 11 years ending February 27th, 25 samples yielded 11·4 to 14·1 per cent. of soluble extract, and 2 other samples gave 10·4 and 10·7 per cent. Five standard samples have been prepared with various specimens of magnesia, rhubarb and ginger, and in these the amount soluble in water ranged from 11·5 to 12·5 per cent.

Three informal samples bought in May showed 9·4, 7·3 and 11·8 per cent. respectively, of soluble matter, but further experiments have indicated that, under certain conditions not previously met with, it is possible for properly prepared Gregory's powder to give 8 per cent. or less of matter soluble in water, instead of the 12 per cent. previously found. The question is still under investigation, but it has been found out that there is a considerable difference in the action of different specimens of calcined magnesia in preventing the solution of the soluble matter in rhubarb.

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.

MILK OF SULPHUR.

On August 10th a Brixton firm was summoned for having, in their drugs department, sold milk of sulphur which was not of the nature, substance and quality demanded. The article was certified by the Public Analyst to contain 46·5 per cent. of calcium sulphate, whereas the solids in milk of sulphur should consist entirely of precipitated sulphur.

For the defence it was stated that the firm had bought the sulphur from a first-class wholesale house, and the invoice described it as pure. Two kinds of milk of sulphur were manufactured—commercial and medicinal—and the wholesale house must by mistake have supplied the defendants with the former.

A fine of 40s. with 1 guinea costs was imposed.

PECTIN IN FRUIT JELLY.

The following is typical of numerous cases that have been decided in U.S.A. District Courts.*

On August 13, 1923, the U.S. Attorney for Western Wisconsin, acting upon a report by the Secretary of Agriculture, asked for the seizure and condemnation of 5 cases of jelly in unbroken packages, alleging that the article had been shipped from the State of Illinois into the State of Wisconsin, and charging adulteration and misbranding in violation of the Food and Drugs Act.

The article was labelled: "Tip Top Brand Imitation Fruit Jelly," etc., and adulteration was alleged for the reason that pectin had been mixed and packed therewith, so as to reduce, lower and injuriously affect its quality and strength, and in that acidified, fruit-coloured pectin had been substituted wholly or in part for the said article.

Misbranding was alleged for the reason that the statement "Apple Jelly," appearing on the pails containing the article, and the statement "Imitation Fruit Jelly," appearing on the shipping containers, were false and misleading and misled purchasers.

Misbranding was alleged for the further reason that the article was offered for sale and sold under the distinctive name of another article.

On June 25, 1924, no claimant having appeared for the property, judgment of the Court was entered,and the product was ordered to be destroyed by the U.S. Marshal.

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* U.S. Dept. Agriculture, Bureau of Chemistry. Notices of Judgment under the Food and Drugs Act, Supplement, March 5, 1925, No. 12734. (For other judgments to the same effect see Nos. 12612, 12622, 12703, 12735, 13312.)

MISBRANDING OF KIDNEY PILLS.

On May 20, 1925, judgment of condemnation and forfeiture was entered in the District Court of Ohio, U.S.A., of 6 dozen bottles of Foley's kidney pills which had been shipped into the State from Chicago.

The article was labelled "Kidney Pills for Irritation of Kidneys and Bladder, for Backache and Rheumatism due to Kidney Disorders . . . Kidneys . . . weakened by disease . . . inflamed and congested."

Analysis of a sample of the article by the Bureau of Chemistry, U.S. Dept. of Agriculture, showed that the pills contained potassium nitrate, methylene blue, hexamethylene tetramine, and material derived from plant sources, including resin and volatile oil similar to juniper oil; they were coated with sugar and calcium carbonate.

Misbranding of the article was alleged for the reason that the above-quoted statements regarding the curative and therapeutic effects of the said article were false and fraudulent, since it contained no ingredient or combination of ingredients capable of producing the effects claimed.*

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* U.S. Dept. Agriculture. S.R.A. Chem. Suppl. 197. July, 1925. Judgment No. 13346.

DRUNKENNESS FROM METHYLATED SPIRIT.

The Licensing Statistics,* 1924, issued on August 25th, afford an instructive commentary on the effectiveness of the methods of denaturing methyl alcohol (*cf. Report of the Government Chemist, infra*, p. 506). The convictions for drunkenness from methylated spirit increased from 683 (334 males, 349 females) in 1923 to 693 (346 males, 347 females) in 1924. In Sunderland they increased from nil to 15; in London County from 100 to 138; in Leeds from 31 to 47. They decreased in Newport (Mon.) from 32 to 11; in Middlesbrough from 7 to 2; in Cardiff, from 57 to 45, and in Swansea from 37 to 9. The figures were practically the same as in the previous year in Liverpool, Birkenhead, Manchester, Salford, and Newcastle. Only where the total numbers are small do the methylated spirit cases stand in a high proportion to the total number of convictions.

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* H.M. Stationery Office. Price 3s. 6d.

Report of the Government Chemist upon the Work of the Government Laboratory.

For the Year ending March 31st 1925.*

The chemical work of the following Departments is now carried out wholly or in part by the Government Laboratories—Admiralty, Ministry of Agriculture and Fisheries, Air Ministry, Colonial Office, Crown Agents for the Colonies, Board of Customs and Excise, Geological Survey, Ministry of Health, Board of Inland Revenue, Ministry of Pensions, Post Office, Department of Scientific and Industrial Research, Home Office, Board of Trade, War Office, Office of Woods and Forests, Privy Council, and Office of Works. The total number of samples

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* Obtainable at Adastral House, Kingsway, W.C.2. Price 1s. 6d. net.

examined in the year, including those dealt with at the Chemical Stations, was 448,106, an increase of 57,685 over the previous year, the increase being chiefly in connection with wines, sugar, tea, exported and imported beer, cocoa products and spirituous preparations, whilst the number of samples of beer for the detection of dilution fell by nearly 1000, of exported tobacco and snuff by over 4000, due to cessation of large exports to the Irish Free State. Work in connection with the Safeguarding of Industries Act involved nearly 10,000 samples.

MINISTRY OF AGRICULTURE AND FISHERIES.—*Butter.*—Four out of 860 samples of butter contained over 16 per cent. of water, whilst in two cases importers were warned where the description of "hard butter" was applied to palm-kernel preparations. Two samples of "Indian Ghee" consisted of vegetable fat.

Margarines.—Seven out of 390 samples exceeded the water limit.

Cheese.—The examinations were made chiefly to determine the nature of the fat (which varied in proportion from 19·9 to 60 per cent. on the dry matter).

Condensed Milk.—Out of 150 samples taken under the new regulations a large number (48) did not satisfy the requirements, chiefly owing to foreign manufacturers not understanding the regulations. Only 8 samples were deficient in fat and 2, in addition, in total solids.

Sheep Dips.—Ten out of 71 samples were reported against.

Fertilisers and Feeding Stuffs Act.—Reference samples from Local Authorities comprised 9 fertilisers and 16 feeding stuffs. Two samples of basic slag contained mineral phosphate, one was deficient in total phosphate, another in citric-soluble phosphate, as also was one sample of slag phosphate. Ground oat samples were adulterated (from 35 to 50 per cent.) with oat husks, wheat, tapioca and barley. A sample of meat meal contained 25 per cent. cereal matter and one of barley, 50 per cent. of tapioca, and another 55 per cent. of oat husks and wheat flour. A cod-liver oil food, a fattening meal and a pig meal were deficient in oil or albuminoids, whilst two samples of "decorticated" cotton meal were deficient in both, and in addition contained husk corresponding to two-thirds of the seed being "undecorticated." There were only two cases of disagreement with the results of agricultural analysts.

Merchandise Marks Act.—Ground oats taken under these Acts were also found adulterated. Of 5 samples of barley meals, 2 were barley offals and 2 contained, respectively, 7 and 20 per cent. of oat husks.

Miscellaneous Articles.—Altogether 245 such samples were examined. For an investigation into the proportion of fat in cream and cheese produced or sold in this country 176 samples of cheese and 32 of cream were taken.

AIR MINISTRY.—An increase, due mainly to examination and sorting of stocks, resulted in 804 samples being examined, chiefly of metals and alloys.

CROWN AGENTS FOR THE COLONIES.—A large scale experiment was made to ascertain conditions under which recovery of potassium chloride could best be made from the Dead Sea by the use of solar heat, and a separate Report was issued (*Production of Minerals from the Waters of the Dead Sea,* published by the Crown Agents for the Colonies). Ninety-three medicines and drugs were examined for conformity to specification.

CUSTOMS AND EXCISE.—*Beer.*—The total number of samples examined in connection with duty was 54,398 (increase of 6665 over 1924); of materials used in brewing, 385; of unfinished wort or beer, 7911 (original gravity of the wort was declared by the brewers at a figure between one and five degrees low in 406 cases, and in one case more than 5 degrees low); of sour beer, 1853; of beer as retailed, 3890, of which 207 showed evidence of dilution. Arsenic was sought in 1257 cases in beer and brewing materials, and slightly exceeded the limit in 90 cases (1/100th of a grain per gal. of arsenious exide).

Cocoa and Chocolate.—Of prepared cocoas, 12,046 samples were examined for composition for assessment of relative duties.

Coffee and Chicory.—Seven out of 1047 samples were found inaccurately declared.

Dangerous Drugs Act.—Out of 113 samples 22 were found to contravene the provisions of the Act.

Dyestuffs.—Five hundred and thirty-five samples of imported colours, lakes, etc., were examined.

Matches.—None of the 211 samples submitted was found to contain white phosphorus.

Safeguarding of Industries Act.—Examination for ingredients liable to tax was made of 9433 samples.

Spirits.—Samples of wood and mineral naptha (731) and pyridine (135) for use in preparation of methylated spirits were examined, and 1082 samples of denaturants, denatured alcohol, etc. Pyridine has been adopted as an additional denaturant. Spirit strength for drawback was tested on 1541 samples of spirits for export, and sugar on 1181 for checking of exporters' claims, which were found overstated in 323 cases. Of exported spirituous preparations, 16,823 samples were examined for spirit strength and character, whilst 11,346 samples of imported spirits and spirituous preparations were tested mostly for "obscuration."

Sugar, Glucose and Saccharin.—Sugar is assessed for duty according to its polariscope reading by a scale from 76 degrees upwards, all foreign raw sugar below 76° being rated at 5s. 7d. per cwt., foreign refined sugar above 98° at the full rate of 11s. 8d. per cwt., intermediate qualities according to scale, and sugar of Empire origin at lower rates. The number of articles examined was 58,310; of glucose, 141 samples were taken from charging vessels, 993 for assessment of drawback on exportation and 749 of imported glucose and glucose used in syrups. Saccharin was looked for in 313 samples. In connection with the subsidy on home-grown beet sugar 450 samples were examined.

Tea.—A total of 58,022 samples was examined and 535 reported against. Rejected tea denatured with lime or assafœtida may be used for manufacture of caffeine.

Tobacco.—Moisture was tested for in 9915 and oil in 1784 samples of manufactured tobacco for home use; moisture in manufactured tobacco for export in 12,349 samples; 813 samples of snuff were examined. For offal tobacco, moisture, inorganic matter and sand have usually to be determined and often extraneous matter, and 25,474 samples of stalks for drawback, 7983 for uniformity of condition, and 6274 of offal snuff shorts and smalls were examined. Denaturants, nicotine preparations, essential oils, etc., were also dealt with.

Wines.—A total of 116,546 samples was examined.

GEOLOGICAL SURVEY.—Complete analyses were made of 59 rocks and there were 32 partial analyses.

HOME OFFICE.—A total of 367 samples were analysed for the Department, including drugs from smuggled goods, lead glazes, inks for a forgery case, etc.

INDIA OFFICE.—Thirty-two samples of anaesthetics were tested for conformation to specification.

Pigments for fiscal stamps were examined for the INLAND REVENUE; paper for affidavits, blood stains, etc., for the SUPREME COURT OF JUDICATURE; 237 foods for the MINISTRY OF PENSIONS; 17 samples of waters and disinfectants for the GOVERNMENT OF NORTHERN IRELAND; paper, pigments and gum used for stamps, and miscellaneous stores for the POST OFFICE; an old seal of the PUBLIC RECORD OFFICE was found to have disintegrated owing to mould growth; 103 samples of fortified wine and lemon juices (4 unsuitable) for the BOARD OF TRADE; 4286 foods for the WAR OFFICE, etc.

OFFICE OF WORKS.—Examination of materials for purchase for public service revealed many well known materials put up as specially valuable for particular uses. A case of blistering of scagliola was traced to the use of tin filings with the plaster; corrosion of iron pipes laid in magnesite flooring was due to the chloride present in the magnesium oxychloride cement used.

SALE OF FOOD AND DRUGS ACT.—Analysis of the reserved portion of samples analysed under the Act was made in 51 cases of foods and drugs, and in 11 of these there was disagreement with the analyses put forward by the prosecution. The following were the cases in which there was disagreement:—Five samples of milk alleged to be deficient in non-fatty solids were found to contain 8·58, 8·66, 8·61, 8·50, and 8·61 per cent. respectively. A sample of lard alleged to consist of another fat was found to contain no fat other than lard. A sample of cream of tartar alleged to contain water and sand contained not more than traces of both of these substances. In this case, however, the sample was not enclosed in an air-tight receptacle, and moisture may have been lost between the times of sampling and analysis. A sample of sponge fingers alleged to contain boric acid was found to be free from boric acid, and three samples of butter alleged to contain foreign fat did not show any evidence of the presence of foreign fat.

D. G. H.

Ministry of Health.

SIXTH ANNUAL REPORT, 1924—1925.*

Inspection and Supervision of Food.—The number of licences in operation on the 31st of March under the Milk (Special Designations) Order of 1923 was as follows:—Producers of Certified Milk, 73; of Grade A Tuberculin Tested Milk, 71; issued by Local Authorities, Certified Milk, distributors, 715; Grade A Tuberculin Tested Milk, bottling establishments, 46; other distributing premises, 334; Grade A Milk, producers, 89; bottling establishments, 47; other distributing premises, 207; pasteurised milk, pasteurising establishments, 62; other distributing premises, 367.

The number of licences issued in Wales was:—Producer's licences to use the designation "Grade A," 14; dealers licenses for "Grade A," 11; dealers' licences for "Certified," 5; for "Grade A" (Tuberculin Tested), 2; and for "Pasteurised," 5. Four Welsh County Councils still continue not to exercise the powers conferred on them by Article 4 of the licences to produce "Grade A" milk. One Rural District Council has been authorised to grant licences for its area in place of one of the four County Councils in question.

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* Obtainable at Adastral House, Kingsway, W.C.2. Price 3s. 6d. net.

Lard, dripping, edible tallow and similar rendered fats have been added to the class of meat admitted to this country only if accompanied by a recognised official certificate.

SHELL FISH.—An investigation into a complaint as to the mussel beds off the coast of Heysham is being made, and a public enquiry into an order made for prohibition of distribution or relaying of cockles from Silverdale. Draft regulations have been prepared to ensure that cockles taken from the Pegwell Bay beds should be sterilised.

SALE OF FOOD AND DRUGS ACT.—Forty-one appointments of Public Analysts in England have been approved during the year. The total number of samples analysed was 118,000 (an increase of 3154 over the previous year), 5·9 per cent. of which were reported against as adulterated or not up to standard against 6·1 per cent. for 1923, and 6·2 per cent. for 1922. Of the 62,133 samples of milk examined, 7·7 per cent. were reported against (7·8 for 1923 and 7·7 for 1922). In Southampton a confirmatory test applied for added water was the detection of small traces of impurities usually present in tap water (nitrates) by the diphenylamine test. Of the 10,516 samples of butter examined, 151 were reported adulterated, 74 containing water in excess of 16 per cent., and 68 foreign fats, and 9 excessive proportions of boric acid. Of the 33 adulterated samples of margarine (out of 3456 examined) 4 contained over 10 per cent. butter fat and one a large percentage of free fatty acids. In spite of the increase of samples examined of condensed and dried milks (1574) the percentage reported against has remained much the same. Three samples were found contaminated with lead, copper or tin, and 57 were deficient either in fat or total solids. The sale of articles of food bearing exaggerated statements or suggestions of the properties of the contents is attracting attention, particularly "custard powders," "custard" and "cream custard." Sixteen such samples were reported against either for containing a coal dye or because they consisted wholly of dyed and flavoured maize flour. In one case of tomato purée the contents of the tin had dissolved 14 grains of tin per lb.

Drugs.—The principal drugs or medicines reported against were borax, sweet spirits of nitre and medicines prepared on prescriptions.

In Wales, sanction has been given to the appointment of a deputy analyst to two County Councils. In Wales, during the year, 5794 formal samples and 424 informal samples were submitted for analysis. Of the 4217 samples of milk, 337 were returned as non-genuine. There were five cases of annatto in milk, and proceedings were taken in three of them. No preservatives were found in the 3715 samples of milk tested for them. Of 45 samples of cream, 25 contained preservative.

METROPOLITAN WATER SUPPLY.—The average daily quantity of water taken in 1924 by the Metropolitan Water Board was from the Thames, 153·5 million gals.; from the Lee, 64·2 gals., the latter an increase of 10·3 per cent. over 1923; from other sources, 42 million gals., a decrease of nearly 10 per cent. over 1923. The quantity supplied to customers increased 2·06 per cent., and was 252,781,800 gals. to 1,163,814 separate premises. The daily consumption averaged 36·5 gals. per head.

POLLUTION OF RIVERS.—During the year an Order was issued under which legal proceedings may now be taken under the Rivers Pollution Acts in connection with alleged pollution by dairying operations. Increasing use is being made of the activated sludge process for the purification of such waste waters.

ANALYSIS OF SEWAGE AND SEWAGE EFFLUENTS.—The Association of Managers of Sewage Disposal Works has, as an outcome of a suggestion made by the

Department, formulated a series of proposed standard methods, and a Committee composed of chemists concerned with sewage and river purification problems, representatives of River Boards, and medical officers of County Councils, active in pollution prevention work, has been called by the Department to consider these proposals. The Committee has held two meetings, and standard methods are being formulated.

D. G. H.

ADVISORY COMMITTEE ON WATER.

REPORT ON MEASURES FOR THE PROTECTION OF UNDERGROUND WATER.*

WITH increased consumption of water per head of population and the demand for higher standards of purity, further protection of public supplies becomes essential. The two main problems are those of supply and prevention of pollution. In the main the proposals of the Committee are as follows:—

(1) "For the safeguarding of supplies, that power be given to the Minister of Health to schedule areas where the demands on underground water are such that special measures of protection should be adopted, and that, in any such area, no fresh wells (except for domestic or agricultural purposes) or mines be sunk until a permit in that behalf has been obtained, the intention being, as regards mines, that the Minister shall act in consultation with the Minister of Mines and that measures be taken, when new mines are sunk, to avoid waste of needed water supplies, and

(2) For preventing pollution, that Statutory Water Undertakers be given powers, already possessed by some, for protecting their supplies from pollution by the acquisition of land, by restrictions on its use or by requiring particular measures of drainage, proper compensation being made in respect of any interests injuriously affected."

REPORT OF THE SUB-COMMITTEE APPOINTED BY THE ADVISORY COMMITTEE.

I. *Abstraction and waste of underground water.*—Under existing law every landowner has unfettered rights to all water under his land which does not flow in defined channels, and cannot be required to give compensation for any injury to others by way of deprivation or diminution of their water supplies. Water Authorities, on the hand, have only limited power as to use of underground water. There is therefore need for alteration of the law, particularly with regard to the safety of the underground supplies being endangered by the operations of the colliery companies, which may compel Water Authorities to go long distances for their supplies. Such operations may involve the running to waste of large quantities of water, the draining of water in overlying strata into the mine workings; sea water may be drawn into the underground supply by heavy pumping (App. A, case C); existing supplies may become contaminated by water pumped by a colliery company (App. A, case D); water-bearing strata may be disturbed and the natural flow diverted.

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* Obtainable at Adastral House, Kingsway, W.C.2. Price 2d. net.

Suggested remedial measures.—Some form of State control (see Proposal I. above). When new wells are to be made or old ones extended in the area to which the regulations have been applied by order, notice in a local newspaper should be given not less than 4 weeks before commencing work. Any local authority, water undertaker or person affected should be entitled to object, and the issuing of the licence should be in the hands of the Minister of Health, and should not be refused unless it is shown that public needs will be prejudicially affected. *Compensation for damage* is bound up with the question of compensation for damage due to mining subsidence generally, and has been deferred pending the report of the Royal Commission on Mining Subsidence. Unnecessary waste from overflowing boreholes should be prevented.

II. *Contamination of underground water.*—Common law gives little or no security in the case of the great majority of underground water supplies where it is difficult or impossible to establish that the supply is endangered, or, if pollution has been shown to have occurred, to determine its precise source, and in fact the express provisions of the general law for prevention of pollution are mainly directed towards the protection of surface water. The Sub-Committee recommend that certain sections of the Waterworks Clauses Act be altered to apply to underground supplies, and that the maximum penalties prescribed in section 61 be altered from £5 to £20. They also recommend that certain provisions of special Water Acts be made general (see Proposal II. above). Thus Water Authorities should have power (1) to acquire compulsorily or by agreement land around the site of the well or above the adits; (2) to enter into agreement with owners of property to effect alterations in drainage arrangements which many constitute a possible source of pollution of the undergound supply; (3) to make and enforce byelaws for securing the purity of the water which they are authorised to appropriate, and (4) should make provision or requirements as to lining the well or boring to a specified depth, or limiting the quantity of water to be raised. Further, sections 182–186 of the Public Health Act, 1875 (which require confirmation by the Minister of Health and make provision as to procedure) should apply as far as practicable to the bye-laws made for the purpose of protecting the purity of the water, and it should not be necessary to obtain the consent of the Attorney-General to the taking of proceedings for the recovery of any penalty imposed by the bye-laws. A general provision should make it unlawful for any person to cause or suffer the deposit of any solid in the sub-soil, or the discharge of any liquid into the sub-soil in the vicinity of any well or adit used or likely to be used for domestic purposes. In exceptional cases it is recommended that the Minister of Health should have power to make regulations for preserving the purity of underground water and to enforce them in the area specified by order. He should also be empowered to obtain returns of the quantities of water abstracted from underground. Finally the amendment and consolidation of legislation relating to water supply, which is scattered over a large number of Acts of Parliament, is recommended.

Appendix A. comprises specific examples of failure of supply and contamination owing to mining operations, examples of special Parliamentary concessions, covenants, etc., whilst Appendix B contains sections 61–64 (penalties) of the Water Works Clauses Act of 1847, with suggested alterations.

D. G. H.

Minnesota State Dairy and Food Commissioner.

TWENTIETH BIENNIAL REPORT, 1924.

SEVERAL of the questions dealt with in this Report by the Chief Chemist are of more than local interest. In all, 7661 samples were examined, and of these, 2119 were declared illegal.

BAKING POWDER.—The definition and standard adopted for baking powder includes, among other requirements, that it shall yield not less than 12 per cent. of available carbon dioxide, and shall be labelled wth a declaration of ingredients in terms commonly understood by the purchaser. One sample examined yielded only 10·3 per cent. of available carbon dioxide, and was accordingly condemned.

BUTTER.—Four samples showed unmistakable evidence of adulteration with lard. They gave the following results:—

Water Per Cent.	Fat Per Cent.	Reichert-Meissl value.	Valenta test (Acetic acid 99·5 Per Cent.) °C.	Crystals from ethereal solution.
16·03	78·6	21·1	41·0	showed lard
16·36	79·2	22·0	36·5	,, ,,
16·95	79·6	25·0	32·5	,, ,,
—	—	22·2	40·0	,, ,,

The lard crystals were very characteristic after the dissolved fat had been left in a refrigerator for 48 hours, and photomicrographs of them were produced in Court. At the trial the defence contended that one of the samples gave a Reichert-Meissl value of 24·85, and therefore complied in an important particular with the U.S.A. Government standard. The State, however, convinced the Court that the Reichert-Meissl value was below the minimum for pure butter made in the locality, and, in addition, showed positive microscopic evidence of adulteration.

The Valenta test of Fryer and Weston (ANALYST, 1918, **43**, 3) has been constantly used in the laboratory for two years, and has proved a valuable confirmatory test, as well as a rapid sorting test for butters. The Valenta figures obtained for Minnesota Creamery butters were as follows:—Maximum, 49·5; minimum, 46·5; average, 47·7.

CACAO PRODUCTS.—A sample of chocolate yielded 52·2 per cent. of fat, which was found to be cacao butter.

CEREAL PRODUCTS.—The use of artificial colouring matters, chiefly coal tar dyes, for colouring cereal pastes, is still quite prevalent, notwithstanding the action of the manufacturers against the practice some years ago. The standard requiring 5 per cent. of egg solids in egg "noodles" is operative in this as in several other States, and is now effective under the national food law.

CHEESE.—A sample sold as "Farmer Cheese" had the following composition:—Moisture, 52·8; fat, 28·5; fat in total solids, 60·3 per cent.; n_D^{40} of fat, 1·4488; Reichert-Meissl value, 7·39; and Valenta number (Fryer and Weston), 36·2. It was found to consist essentially of milk casein combined with coconut oil.

A common variety of American Cheddar cheese is sold extensively under the name of "Full Cream Cheese." The term is legal in some of the States, but it is suggested that the use of the word "cream" in any form to describe normal milk is a relic of the inaccurate terminology of former generations, and should be abandoned in the interest of clearness and precision.

VANILLA EXTRACTS.—Eight of 13 samples were condemned, as they consisted, for the most part, of mixtures of vanillin and coumarin coloured with caramel.

JELLIES.—The use of glucose (corn) syrup in imitation jellies has been largely superseded by pectin preparations. The use of pectin should only be tolerated when its presence is declared on the label in unmistakable terms.

MILK.—The systematic application of cryoscopic tests has had a pronounced effect in eliminating the addition of small quantities of water. The custom has been to report as adulterated all samples which, as a result of cryoscopic tests, showed added water in amounts exceeding 3 per cent. By reliance on the well-known methods of testing for fat and total solids the milkman has succeeded in deluding himself into the belief that milk which complies with the standards for those ingredients is legal, although it may have been "standardised" by the addition of water or skimmed milk.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Discoloration of Canned Food. A. E. Stevenson. (*Canning Age*, Dec. 1924 ; *Chem. Abstr.*, 1925, **19**, 1739.)—The most common type of discoloration in canned food is due to the formation of ferrous sulphide, due to the action of hydrogen sulphide produced by thermal decomposition of sulphur-containing proteins at the " processing " temperature. The oxygen of the air is frequently an indirect factor in that it increases solution or oxidation of the iron. The discoloration of canned sweet potatoes is brought about by the enclosure of excessive air during sealing or by leakage of the can. Discoloration of canned beets is sometimes due to tyrosin, which is, however, destroyed at a fairly low temperature. In canned fruits pink discoloration is the most common type ; it may occur in canned apples, peaches and pears, and is generally associated with overcooking of the products. The pink colour is due to the reaction of some constituent of the fruit with the tin from the container. Copper in peas or vegetables is sometimes a cause of discoloration, owing to its reacting with sulphur compounds in the material to form copper sulphide. Only a few parts per million of copper are required to cause discoloration.

Nickel and Cobalt in the Animal Kingdom. G. Bertrand and M. Macheboeuf. (*Bull. Soc. Chim.*, 1925, **37**, 934–940.)—Nickel was determined by ashing at low temperature, lixiviating with hydrochloric acid, dissolving the ash in hydrochloric acid, adding calcium carbonate and ammonium hydroxide in the presence of hydrogen peroxide, and eliminating copper and zinc successively from the solution. The nickel and cobalt were dissolved in hydrochloric acid and separated by means of dimethylglyoxime. In order to weigh the very small quantities involved, the glyoxime compound may be dissolved in chloroform, which is then allowed to evaporate spontaneously, and the residue compared with that from known quantities of nickel. Nickel was found in all the material examined apart from smooth and striated muscle fibre and fat. The proportions found were small except in the case of marine molluscs. Maximum amounts in thousandths of a mgrm. per kilo. of dried material were :—In human organs : liver 330, and pancreas 230 ; in bulls : liver 360 and pancreas 410 ; in calves' liver, 510 ; in turkey feathers, 330 ; in fishes, 68 ; in crayfish, 435 ; and in mussels, 2350. Cobalt is present in still smaller quantities. (*Cf.* ANALYST, 1925, **84,** 348.)

D. G. H.

Clerget Method (Inversion Coefficients). E. Saillard. (*Comptes Rend.*, 1925, **181,** 139–140.)—The values of the Clerget inversion coefficient, for solutions containing per 100 c.c. 4, 6, 8, 10, 12, 14, 16 grms. and the French normal weight of sucrose, are respectively 142·86, 143·04, 143·22, 143·41, 143·60, 143·79, 143·98, and 144.

T. H. P.

Lecithin in Fats. E. Boedtker. (*J. Pharm. Chim.*, 1925, **117,** 107–113.)—About 1 grm. of dry and filtered oil is treated, drop by drop, with concentrated nitric acid, the mixture heated on a boiling water-bath for half-an-hour, the nitric acid boiled off, and 6 c.c. of 10 per cent. nitric acid added to the residue. After cooling and stirring, the liquid is filtered, 15 c.c. of molybdic reagent added, and the tube heated to 70° C., and left until next day for any yellow precipitate to settle. By this means lecithin was found in all the fats examined, vegetable and animal, except in the case of castor oil. The reaction can readily be made quantitative, but obviously requires the absence of phosphate in other form in the oil. Lecithin might be detected by means of its nitrogen, but experiments so far leave the matter in doubt.

D. G. H.

Murumuru, an American Palm-kernel Fat. E. André and F. Guichard. (*Comptes Rend.*, 1925, **181,** 228–230 ; *cf.* ANALYST, 1917, **42,** 38.)—The fat extracted by petroleum spirit from two samples of murumuru kernels reached 40–42 per cent., and gave the following analytical constants :—M.pt., 34–35° C. ; sp. gr. at 15° C., 0·918 ; n_D^{31}, 1·4535 ; saponification value, 240–241·5 ; iodine value (Hanus), 11·2–11·5 ; Reichert-Meissl value, 2·8–3·14 ; Polenske value, 6·9 ; and Hehner value, 92·7. By fractional distillation caprylic and capric acids, but not caproic acid, were isolated, and among the solid acids one of higher m.pt. than stearic acid was found, possibly arachidic acid.

D. G. H.

Determination of Sulphur Dioxide in Foods. K. K. Järvinen. (*Zeitsch. Unters. Nahr. Genussm.*, 1925, **49,** 283–286.)—Sulphur dioxide in dried fruits may be detected by means of potassium iodate and starch paper, but for reliable indications dilute acid must be used, not simply water. Ten grms. of the fruit are gently warmed on the water bath with 10 c.c. of 2*N* hydrochloric acid in a flask with a piece of iodate starch paper under the cork. In the presence of sulphur dioxide a blue colour appears within 5–10 minutes. For the quantitative determination the ordinary distillation method is used, the air being displaced by carbon dioxide either produced by the addition of marble and hydrochloric acid, or introduced from outside. The sulphur dioxide in the distillate is determined as barium sulphate after oxidation by means of iodine ; the direct titration of the distillate with thiosulphate was found to give high results. H. E. C.

Benzoic and Salicylic Acids in Wines. J. L. Chelle. (*Bull. Soc. Pharm. Bordeaux*, 1925, No. **1** ; *Ann. Chim. anal.*, 1925, **7**, 225–230.)—This is a critical study of the recent methods proposed for the determination of these acids in wines. For salicylic acid the author prefers a colorimetric determination on benzene extracts, using standards containing 1, 2, 4, 6, 8, and 10 mgrms. of acid per litre. For benzoic acid the method proposed by Blarez is preferred. The wine is extracted with ether and the arborescent form of the crystals in the residue left on spontaneous evaporation, is noted, and the usual tests of identification are made.

D. G. H.

Determination of Benzoic Acid in Wines. (*Ann. Chim. anal.*, 1925, **7**, 230–231.)—The following method is Filandeau and Bonis' modification of von der Herde and Jacob's method. Sodium hydroxide is used to neutralise 100 c.c. of wine, and a few drops in excess added, the volume reduced to half, the liquid filtered, acidified with 5–10 c.c. of 10 per cent. sulphuric acid, extracted twice with 15–20 c.c. of ether, and the ether decanted, washed twice with 3–4 c.c. of water, and extracted with 5 c.c. of 1 per cent. sodium hydroxide solution. To 10 c.c. of the alkaline solution 2 per cent. permanganate solution is added until the rose colour persists for some time. The colour is discharged with sulphurous acid or dilute sodium bisulphite, 2 c.c. of 10 per cent. sulphuric acid added to dissolve the manganese dioxide, and the solution extracted twice with 10 c.c. of ether. The 20 c.c. of ethereal solution are washed twice with 3 c.c. of water, extracted with 5 c.c. of 5 per cent. sodium hydroxide solution, evaporated to dryness on a water bath and the residue taken up with 20 drops of 1·84 sulphuric acid, and treated with 20–25 mgrms. of finely crystalline potassium nitrate. After warming, the clear liquid is washed into a tube with 10 drops of warm sulphuric acid, and the tube either heated to 120–130° C. for 10 minutes or on a water bath for 20 minutes, and, after cooling, 1 c.c. of water and enough ammonium hydroxide solution to render alkaline are added. A yellow coloration, due to *m*-dinitrobenzoic acid, is formed. Nitrites are destroyed by boiling, the solution cooled and a few drops of ammonium hydroxide solution added, and then one drop of freshly prepared ammonium sulphide solution, a characteristic red-orange ring indicating the presence of benzoic acid. The limit of sensitiveness appears to be 1 mgrm. of benzoic acid in the extract. D. G. H.

Reactions of Glycyrrhizin. P. Bertolo. (*Giorn. Chim. Ind. Appl.*, 1925, **7**, 404–405.)—Commercial glycyrrhizin may be readily purified by treating its solution with cadmium chloride solution, heating the liquid and then cooling it, and repeatedly washing the friable mass thus obtained with boiling water. The cadmium compound, suspended in alcohol, is decomposed by means of hydrogen sulphide, the alcoholic glycyrrhizin solution evaporated, and the residue crystallised repeatedly from acetic acid. The colour reactions given by glycyrrhizin with aromatic hydroxy-aldehydes resemble those obtained with atractylin, contained in the extract of *Atractylis gummifera* often used for adulterating liquorice extract (see ANALYST, 1923, **48,** 30 ; 1924, **49,** 534 ; 1925, 193). Atractylin is, therefore, best detected by means of its valeric and sulphonic groups, which are absent from glycyrrhizin. T. H. P.

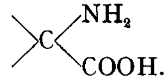
Colour Reaction of Ergot. W. P. H. van den Driessen Mareeuw. (*Pharm. Weekblad,* 1925, **62,** 789–793.)—A colour test for ergotinine is based upon the fact that it forms a bright blue ring at the zone of contact when its ethereal solution is poured on to dilute (50 per cent.) nitric acid.

Biochemical, Bacteriological, etc.

A Theory of Taste and its Experimental Verification. P. Lazarev. (*J. Russ. Phys. Chem. Soc.*, 1922, **54,** 106–109 ; *Chem. Abstr.*, 1925, **19,** 1867.)—The author's experiments have indicated that taste sensitiveness depends upon a physico-chemical process. It is assumed that the tongue contains different classes of papillae, which separately are sensitive to the sensations of sweetness, sourness, bitterness, and saltness. Each of these papillae contains a taste-sensitive substance which is decomposed by the exciting substance to which it is sensitive. The product of this decomposition is ionised and excites the nerve terminals in the papilla. Some sweet substances may affect to some extent sour-sensitive, salt-sensitive or bitter-sensitive papillae ; this gives differentiation or " shade " to the taste. If C is the concentration of the sensitive substance in the papilla, B a critical concentration of the ionised products at which taste is first noticeable, and C the concentration of the substance to be tasted, it is shown that when taste is first observed $E=1/C_2=\gamma_1 C/\gamma_2 B$, where E represents the " sensitiveness."

If one considers C as variable, having at the beginning the value C_0', it is shown that $$[(\gamma_1 C_0/\gamma_2 B)-E]/[\gamma_1(C_0-C_0')/\gamma_2 B]=e^{-\gamma_3 l},$$ where γ_1, γ_2, and γ_3 are constants. This equation has been checked by observing the time required to taste the salt in a 0·1, 0·7, 0·05, and 0·03 *N* sodium chloride solution respectively. These taste-sensitive substances appear to be of protein character.

Relation between Constitution and Taste of α-Amino Acids. A. Heiduschka and E. Komm. (*Zeitsch. angew. Chem.*, 1925, **38,** 291–294 ; *Chem. Abstr.*, 1925, **19,** 2092.)—The method of Pauli (ANALYST, 1920, **45,** 452) was used to determine the relation between the constitution and the taste of α-amino acids, and the results are expressed in terms of Paul's " constant method " (ANALYST, 1922, **47,** 261). Taking beet sugar as unity, the following degrees of sweetness and molecular degrees of sweetness were found :—For glycine, 0·64 and 0·14 ;

sarcosine, 0·62 and 0·16; *d*-*l*-alanine, 0·90 and 0·24; and *d*-alanine, 0·73 and 0·19. In this series methylation does not decrease the sweetness, nor does increase of the C chain from 2 to 3. Leucine, however, has only a faintly sweet taste. The substances all contain the "dulcigen" group  $>C<^{NH_2}_{COOH}$. (*Cf.* ANALYST, 1923, **48,** 334; 1924, **49,** 345.)

Presence of Argon in Living Cells. A. Pictet, W. Scherrer and L. Helfer. (*Comptes Rend.*, 1925, **181,** 236–238.)—After being dried at 25° C. in a vacuum over sulphuric acid, yeast is found to contain from 0·28 to 0·31 c.c. of argon per grm. This gas is also present in animal tissues, 1 grm. of dried sheep's brain containing 0·86 c.c. The argon appears to be included in the gaseous form in the animal and vegetable cells, from which it escapes when the cell-walls are ruptured. T. H. P.

Ageing of Natural Mineral Waters. O. Baudisch and L. A. Welo. (*J. Biol. Chem.*, 1925, **64,** 771–779.)—Certain mineral springs have curative properties which they lose in the course of a short time. These curative qualities are not always due to radioactivity, since non-radioactive springs are sometimes therapeutic; nor are they due to the mineral contents alone. The authors have studied the Glauberquelle III. at Franzensbad, in which all the iron is present in the form of ferrous bicarbonate, and have obtained an insight into the mechanism of the therapeutic action, by simple analogies, and an understanding as to the way in which the curative properties vanish when the water is allowed to stand. The salts dissolved in the water as they come out of the deep interior of the ground are in a particularly labile configuration. The ageing process is connected with the slow rearrangement in space of the atoms, of the molecules, and of groups of molecules. Light accelerates the ageing. The relation between chemical properties and crystal structure can be applied to *dissolved* iron salts. The iron is called "active" in the curative water and "inactive" when the healing properties have disappeared. In the active ferrous carbonate, dissolved in mineral water, the specific catalytic action may be assumed to be connected with the spatial arrangement of the HCO_3 and the OH_2 groups around the central iron atom. With the re-arrangement of the molecules in the inner sphere, brought about either by heat, light, or simply by ageing, the central iron atom of these complex salts loses its special active property, and ferrous carbonate or hydroxide is precipitated. If oxygen enters the reaction, red ferric hydroxide is precipitated. Possibly, in a similar way that an active iron salt, such as haemoglobin, aids the growth of certain bacteria, the active iron salts in fresh mineral waters aid the life processes of body cells as soon as the salts enter the stomach and intestines and begin to take part in the metabolism. It is probable that all minerals in the healing springs play a similar part to that of iron. P H. P.

The Jendrassik Reaction for Water-Soluble Vitamin *B*. N. Bezssonoff. (*J. Biol. Chem.*, 1925, **64,** 589–590.)—The essential part of the first claim of Levine (*J. Biol. Chem.*, 1924, **62,** 157) that the ferric ferricyanide reaction proposed by Jendrassik (ANALYST, 1923, **48,** 562) is not a specific test for water-soluble vitamin B, but is given by phenol, was contained in a study published several months earlier by Bezssonoff (*Bull. Soc. Chim. Biol.*, 1924, **6,** 35). The main difference between the observations of Levine and Bezssonoff consists in the latter's indication of the special and great sensitiveness of the ferric ferricyanide reaction for the ortho- and para-polyphenols. In dealing with typical reactions for a vitamin, only substances which possess a very great sensitiveness for this reaction are of value. P H. P.

The Jendrassik Reaction for Vitamin *B*. V. E. Levine. (*J. Biol. Chem.*, 1925, **64,** 591–593.)—The work of Bezssonoff and of Levine is discussed. Both authors have concluded that the inability of active vitamin *B* preparations to give a positive Millon or Liebermann reaction is no proof that vitamin *B* is not a phenol. Levine does not claim, as Bezssonoff asserts, that the Jendrassik reaction is a general test for phenols. He tested 57 phenols, whereas Bezssonoff investigated only 4. Some phenols are negative, and some non-phenolic compounds are positive. The Jendrassik reagent gives a positive reaction with certain compounds of biological occurrence, and this renders the reaction inapplicable as a definite test for vitamin *B*. Jendrassik believed in the specificity of the reagent. Contrary to the findings of Levine, uric acid is reported by Bezssonoff to react negatively. Bezssonoff drew conclusions as to the sensitiveness of the reaction based upon tests with one monophenol, one orthodiphenol and one paradiphenol. Perhaps the most important finding recorded by Levine is the effect of alkali. After treatment with it, 57 compounds, which ordinarily gave positive reactions, gave negative ones. Phenols resemble vitamin *B* with respect to the destructive effect of alkali, and the use of alkali cannot serve to differentiate vitamin *B* from all other compounds which give a positive Jendrassik reaction.
P. H. P.

Fatty Acids in Human Subcutaneous Fat. H. C. Eckstein. (*J. Biol. Chem.*, 1925, **64,** 797–806.)—The ratio of liquid to solid fatty acids of the subcutaneous fat from the abdomen of man is similar to that reported by others for the fat of the blood and faeces. The method of extraction of the lipoids in the fatty tissue is described, and the results of experiments with it are given. The presence of fatty acids with two, three and four double bonds has been demonstrated for this fat. Of these, linolic acid predominates, as 0·5 per cent. has been isolated. The author is of the opinion that more of this acid than this figure would indicate is present, since, by the method employed, all of the acid is not precipitated as the crystalline tetrabromide. Only 0·33 per cent. of the acid containing four double bonds, and but 0·03 per cent. of the triply unsaturated acid were found. To some extent, at least, the distribution of the unsaturated acid of human abdominal fat is similar to that reported by others for the blood lipoids.

It is probable that about 1 per cent. of myristic acid is present. Traces of lauric acid appeared to be present, and 0·24 per cent. of cholesterol, as determined by the digitonin method, was found (*cf.* ANALYST, 1896, **21**, 171). P. H. P.

Effect of Chlorinated Milk on the Albino Rat. J. W. Read. (*J. Agric. Res.*, 1925, **30**, 889–892.)—Hypochlorites are extensively used in the U.S.A. for the disinfection of milk cans. With the object of discovering whether milk containing free chlorine or hypochlorite is injurious to health, fresh milk impregnated with chlorine and hypochlorite in various proportions from 1:3000 to 1:15000 was given to albino rats over a period of 10 months. No deleterious effects were observed, and the growth curves were normal. Chlorine to the extent of 1:5000 imparts a distinct flavour to the milk, but this did not deter the rats from consuming large quantities thereof. The treatment of milk with chlorine, as has been advocated by some in the States, is, however, not recommended. H. E. C.

Bactericidal Power and Chemical Structure. F. Ishiwara. (*Zeitsch. Immunitäts*, 1924, **40**, 429–452 ; *Chem. Abstr.*, 1925, **19**, 999.)—The bactericidal action of numerous chemicals upon bacteria was determined, the micro-organisms tested including typhoid, diphtheria, anthrax and dysentery bacilli, and staphylococci, meningococci, pneumococci, and gonococci. Amino and alkyl derivatives of benzene are practically without action when the substituting group is single. When, however, both groups are present and attached to different C atoms of the nucleus, as in toluidine, there is pronounced bactericidal action. Azo- and azoxybenzene are actively bactericidal. The nitro group behaves like the alkyl groups. Although alkyl derivatives are almost without action, substitution of phenol for the alkyl group enhances the action 3 to 10 times. The effectiveness increases with the number of C atoms, the ethyl radicle conferring twice as much bactericidal power as the methyl group, etc. Phenol with two substituted alkyl groups is five times as active as phenol. In the fatty acid series the relationship is reversed, increase in the number of carbon atoms reducing the bactericidal power. Normal fatty acids and alcohols are twice as active as the iso-compounds. Although benzene and methane have no action, their hydroxy and carboxyl derivatives are active. Of the OH- substituted phenols, hydroquinone is the most active. Aldehyde groups enhance the action even more than hydroxyl groups. The presence of the COOH and SO_3H groups increases bactericidal action by 10 to 100 times in both the methane and the benzene series. Condensed compounds of the aliphatic and aromatic series are, for the most part, inactive, except when one or both of the condensed constituents are active alone. Of the free halogens, chlorine is the most, and iodine the least active. In combination, however, this relation is reversed, iodine compounds being the most bactericidal. Although substitution of two alkyl, halogen, hydroxyl, or carboxyl groups enhances bactericidal action, the effectiveness is not further increased by the addition of third or fourth groups, either in the aliphatic or aromatic series. Of the metallic compounds, lead, bismuth, and calcium are without effect ; aluminium, iron, zinc, silver, and particularly mercury, are very active. The inorganic and organic mercury compounds are practically equal in bactericidal power, but the organic compounds are less organotropic.

It was not possible to make a definite grouping of bacteria as regards sensitiveness, although gonococci appeared most sensitive, particularly towards mercury compounds, then cholera vibriones, and then dysentery bacilli.

Fermentation Products of Certain Mannitol-Forming Bacteria. H. R. Stiles, W. H. Peterson and E. B. Fred. (*J. Biol. Chem.*, 1925, **64**, 643–654.)—The formation of mannitol in fermenting mixtures of plant origin is known to be due to a group of bacteria which act upon fructose and reduce this sugar to its corresponding alcohol. Mannitol-forming bacteria have been found in wine, sauerkraut, silage, canned goods, yeast and cereal infusions. Four strains of the bacteria have been isolated from cereals, and their fermentation characteristics and products determined. Culture 26 fermented both xylose and arabinose, Culture 19 destroyed only arabinose, Culture 36 attacked only xylose, and Culture 23 fermented neither of these sugars. The pentose molecule was decomposed, with the production of lactic and acetic acids. Tables shew the results and the organisms are described. Fructose was completely fermented, with the formation of lactic and acetic acids, carbon dioxide and mannitol. All the cultures fermented sucrose, and three of them produced small quantities of mannitol from it. Raffinose was partly destroyed by Cultures 26 and 36, with the production of lactic and acetic acids, alcohol, carbon dioxide and mannitol. Glucose was fermented, with the production of ethyl alcohol, carbon dioxide and lactic acid. Galactose and lactose were attacked to a less degree, but if fermented, formed similar products. The lactic acid produced was mainly inactive, irrespective of the sugar fermented. Calcium lactate was slowly fermented by Culture 36, with the production of volatile acid. The same organism slowly destroyed mannitol, with the formation of lactic and acetic acids and probably of carbon dioxide.

P. H. P.

Turnip Flavour of Milk. Orla-Jensen. (*Lait*, 1925, **5**, 30–34; *Chem. Abstr.*, 1925, **19**, 1460.)—A turnip or mustard flavour in milk is due to essential oil of mustard liberated from the glucoside, which passes into the milk from turnips given as food to the cow. The bacteria generally responsible for the decomposition of the glucoside liquefy gelatin and do not form spores, and they may be introduced into the milk from manure or from water. The trouble is more common in pasteurised than in fresh milk. Directions for the control and prevention of this flavour are given.

Toxicological and Forensic.

Toxicology of Hydrogen Sulphide. H. W. Haggard. (*J. Ind. Hyg.*, 1925, **7**, 113–121; *Chem. Abstr.*, 1925, **19**, 2095.)—Hydrogen sulphide is nearly as toxic as cyanogen or hydrocyanic acid, and its action may be as rapid. Symptoms of local irritation appear after many hours' exposure to 0·01 and 0·015 per cent.

of the gas ; local irritation in 1 hour, and slight general symptoms in longer time after inhalation of 0·02 and 0·03 per cent., local irritation and slight general symptoms in 1 hour, and possible death in several hours by inhaling 0·05 to 0·07 per cent. ; general symptoms in less than 30 minutes, and possible death in less than 1 hour by 0·09 per cent. ; death in 15 to 30 minutes by 0·15 per cent. ; and instant death upon inhalation of 0·18 per cent. Hydrogen sulphide is a non-cumulative poison. It is rapidly oxidised in the blood, and the oxidation products are non-toxic ; it forms no combination with, nor does it in any way alter, the haemoglobin of the blood until the blood is completely reduced. Possible chronic effects produced by long exposure to low concentrations (0·01 per cent.) are probably due to local irritation and depression of the nervous system.

Post-mortem presence of Nitric Oxide Haemoglobin. Its Medico-Legal Significance. H. A. L. Banham, J. S. Haldane and T. Savage. (*Brit. Med. J.*, Aug. 1, 1925, 187–189.)—A case is described in which, although the *post mortem* appearances suggested carbon monoxide poisoning, there was reason to believe that they were due to the formation of nitric oxide haemoglobin after death. The stoker of the boiler furnaces of a colliery became ill on his way home after work. The symptoms suggested carbon monoxide poisoning, and he was kept in bed. After nine days he was removed to the hospital and died almost immediately. The *post-mortem* appearances agreed with those in cases in which death has occurred while carbon monoxide was being breathed or directly afterwards. The whole of the blood from whatever part of the body had a red or pink colour, on dilution with water gave a pink precipitate with tannin, and the pink colour remained after the addition of sodium hydroxide to the diluted blood. In view of the difficulty in assuming that carbon monoxide could have been retained for nine days in the living body, it was suggested at the inquest that the poisoning had occurred through exhaust gas from the engine of the motor car conveying the patient to the hospital. This possibility was excluded by the evidence, and the coroner therefore found that carbon monoxide absorbed at the colliery nine days before death was the cause of death. In view of the facts that another man working beside the deceased was not affected, that the *post-mortem* appearances were only consistent with cases in which the blood is highly saturated with carbon monoxide, and that this gas is expelled so rapidly when pure air is breathed that its presence cannot be detected in the blood after a few hours, a claim made against the colliery company was dismissed.

The only cause known to be capable of producing the red colour of the blood and the reactions observed in this case is nitric oxide haemoglobin, which is formed when nitric oxide is brought into contact with reduced blood. A double-banded spectrum closely resembling that of oxyhaemoglobin or CO-haemoglobin is also produced, but the bands are much less sharply defined than those of oxyhaemoglobin, and somewhat less sharply than those of CO-haemoglobin. The NO-haemoglobin band in the yellow extends, also, to a slight distance on the red side of the D line. On great dilution with water, oxyhaemoglobin solution appears yellow in daylight, whilst CO-haemoglobin appears pink. NO-haemoglobin gives a tint which is pinker than that of haemoglobin, but not nearly so pink as that of CO-haemoglobin. The solution of NO-haemoglobin can be distinguished at once by boiling, since it gives a pink coagulum, whilst oxyhaemoglobin and CO-haemoglobin give a dull gray coagulum. This difference is due to NO-haemochromogen being stable in boiling water, and of a red colour. In other respects NO-haemoglobin gives similar reactions, including those shown by the spectroscope, to those given by CO-haemoglobin.

It has been shown by Haldane (*J. Hygiene*, 1901, **1**, 115) that the red colour of raw salted meat is due to NO-haemoglobin, whilst the corresponding colour of cooked salt meat is due to NO-haemochromogen. The nitre used in salting the meat is reduced to nitrite, and this, in the presence of the reduced haemoglobin, becomes further reduced, and the resulting nitric oxide combines with the reduced haemoglobin. In animals killed after receiving a dose of nitrite the blood becomes bright red after death owing to the formation of NO-haemoglobin, and the possibility of this being mistaken for CO-haemoglobin has been pointed out (Haldane, Makgill and Mavrogordato, *J. Physiol.*, 1897, **21**, 160). Nitrite poisons an animal by depriving the haemoglobin of its oxygen-carrying power, just as carbon monoxide does ; but the animal has a cyanosed appearance, contrasting with the red colour in carbon monoxide poisoning. The cyanosis is not due to the presence of reduced haemoglobin, but to that of the methaemoglobin. On the addition of further nitric oxide the methaemoglobin is reduced and then converted into NO-haemoglobin, but there is no sufficient evidence that NO-haemoglobin is a compound of nitric oxide with methaemoglobin, as suggested by Anson and Mirsky (*J. Physiol.*, 1925, **60**, 100).

From these data it follows that the production of nitrite within the body by an infective micro-organism would account for the presence of NO-haemoglobin *post-mortem*. That the occurrence of such nitrifying infective organisms is not exceptional is shown by the *post-mortem* appearances of the bodies and blood in cases of influenzal pneumonia, in which carbon monoxide poisoning could not have occurred. The conclusion is drawn that in this colliery case (in which there was marked cyanosis) the cause of death was broncho-pneumonia produced by a nitrifying organism, the red colour of the blood being produced after death. In all probability the cyanosis was largely due to methaemoglobin formed in the arterial blood, as in poisoning by nitrite.

Methaemoglobin and other colour compounds of decomposition of haemoglobin can be detected most easily by diluting a drop of the suspected blood until the colour becomes yellow, diluting to an equal degree of colour a drop of normal blood, and then saturating both solutions with coal gas or carbon monoxide. The presence of abnormal pigment is indicated by the fact that the corresponding solution is less pink.

Elimination of Cocaine from the System. M. Walrand. (*J. Pharm. Belgique*, 1925, **6**, 701–705, 717–725, 741–744, 757–766, 781–783 ; *J. Pharm. Chim.*, 1925, **117**, 119–120.)—Cocaine administered by mouth should be looked for in the digestive tract, urinary system and liver, whilst, if injected subcutaneously it will be found in most of the organs, but should in every case be sought with the least possible delay, owing to its rapid decomposition. Cocaine was not found in the milk of a goat poisoned by the drug by either channel. It is present in the bile of animals which have received subcutaneous injections. Positive methyl alcohol and benzoic acid reactions are insufficient to justify the assumption of cocaine poisoning, but the recognition of ecgonine is conclusive. D. G. H.

Identification of Egg Yolk Stains. Barthe. (*Bull. Soc. Pharm. Bordeaux*, 1924, No. **4**; *Ann. Chim. anal.*, 1925, **7**, 231.)—The deposits are detached, and phosphorus, which is present in both lecithin and cerebrin (constituents of egg yolk) is determined. Also, the ash of the stains, to which a little potassium carbonate is added, give with ammonium nitromolybdate and with ammonium strychnomolybdate the yellow coloration characteristic of phosphoric acid. D. G. H.

Agricultural Analysis.

Methods Employed in Silage Analysis. H. E. Woodman. (*J. Agric. Sci.*, 1925, **16**, 343–357.)—In a summary of the usual methods employed in the analysis of silage, it is shown that, especially in the case of sour silage, there is loss of amide nitrogen and organic acids in the drying process; this may even amount to as much as 83 per cent. of the total amides originally present. It is of no avail to add organic acids with the view to trapping the ammonia, as the loss is really due to the hydrolytic decomposition of the ammonium salts. For the same reason attempts to estimate the free volatile and non-volatile acidity by distillation with steam are abortive. Foreman's volatile base method (*Biochem. J.*, 1920, **14**, 451; ANALYST, 1920, **45**, 380) constitutes the only safe method for the estimation of the amino acids in a silage extract. No method has been devised for the accurate determination of the free volatile and non-volatile acids when more than traces of ammonium compounds are present. It is recommended to determine total volatile and non-volatile acid by a modification of Foreman's method in which no titrations are carried out in the presence of ammonium salts, and the analysis is carried through in one operation. The first stage consists in the titration with 0·1 *N* sulphuric acid of the amino acids and volatile bases on 50 c.c. of the alcoholic solution, to determine the alkalinity of the residue after the steam-distillation. The second stage is to add sufficient 0·1 *N* sulphuric acid to make, with the volume for the amino acid estimation, an equivalent of the titration value of the 50 c.c. of alcoholic solution. The organic acids so liberated are distilled with steam and titrated in the usual way. H. E. C.

Non-volatile Organic Acids of Alfalfa. W. A. Turner and M. Hartman. (*J. Amer. Chem. Soc.*, 1925, **47**, 2044.)—Citric, malic and malonic acids have been identified in alfalfa by the following method:—Chopped and ground green plants (36 kilos.) were treated with excess of dilute hydrochloric acid and pressed in a hand press. The expressed liquor was concentrated in a steam-jacketed kettle, the proteins precipitated with alcohol, and the liquor filtered. Lead acetate was added to the filtrate, and the mixed lead salts (containing also sulphate, chloride and phosphate) filtered off. These were converted into the ethyl esters, dried over sodium sulphate and fractionally distilled at 10 mm. pressure. Six fractions were obtained, two (Nos. 4 and 6) being greater in amount than the others.

Fraction	1	2	3	4	5	6
Boiling pt.	85–90°	90–100°	100–116°	123–135°	135–165°	165–168°
Weight, grms.	1·84	0·74	1·61	8·94	4·37	13·92

Fractions 4 and 6 were tested for optical activity, and the m.p. of their hydrazides was determined. These tests showed them to be mainly ethyl *l*-malate and triethyl citrate. The lower fractions were found to consist mostly of ethyl malonate. A discrepancy between the m.p. of the citric trihydrazide of Nelson (145° C.) and that of Curtius (107° C.) was investigated. The former was found to be anhydrous, and the latter to contain 1 molecule of water of crystallisation. Citric acid was found in largest proportion, the amount of malic acid about two-thirds, and that of the malonic acid about one-quarter of the amount of citric acid. R. F. I.

Organic Analysis.

Qualitative Colour Test for the Grignard Reagent. H. Gilman and F. Schulze. (*J. Amer. Chem. Soc.*, 1925, **47**, 2002.)—One-half to one c.c. of the solution to be tested for the Grignard reagent is treated at room temperature with an equal volume of a 1 per cent. solution of Michler's ketone (tetramethyldiaminobenzophenone) in dry benzene. One c.c. of water is slowly added with gentle shaking, and then several drops of a 0·2 per cent. solution of iodine in glacial acetic acid, a characteristic greenish-blue colour developing if Grignard reagent is present. Twenty-five Grignard reagents were tested, all giving positive results, that with phenylacetetenylmagnesium bromide ($C_6H_5C{\equiv}CMgBr$) being comparatively weak, yet pronounced. Benzene, dimethylaniline, pyridine, or quinoline may be used instead of the usual solvent ether. A variety of $-OMgX$, $-NMgX$, $-SMgX$, and $AsMgX$ compounds was studied. These gave negative results, showing that no colour is obtained in the case of compounds which have the $-MgX$ group attached to an element other than carbon. Negative results were also obtained with such bodies as ethyl zinc iodide, zinc diethyl, magnesium chloride, etc. The test is sensitive for ethyl magnesium bromide in a solution of $0{\cdot}037M$. R. F. I.

Rapid Determination of Iodine Values. L. W. Winkler. (*Zeitsch. Unters. Nahr. Genussm.*, 1925, **49**, 277–280.)—The iodine value of fats can be determined in less than ten minutes by using as reagent a solution of 3 grms. of bromine in 1 litre of pure glacial acetic acid, in the presence of mercuric chloride as catalyst, and with sodium acetate as buffer. The results of a number of experiments with various kinds of oil are in close agreement with the Wijs values.

To such quantity of the oil as will require 5 to 10 c.c. of the reagent are added 2 to 3 c.c. of carbon tetrachloride, then 0·1 grm. of finely powdered mercuric chloride and 0·1 grm. of crystallised sodium acetate. The mixture is titrated directly with the bromine solution, 1 c.c. quantities being added at first until the yellow colour persists for several seconds, after which the solution is warmed so that the salts are entirely dissolved and a clear solution is obtained; if this does not readily happen a few drops of water may be added, but the liquid must be quite clear before completing the titration. To the clear solution more bromine is added, drop by drop, until a faint yellow colour is obtained which persists for 2 or 3 minutes; the end point is determinable within ±0·05 c.c. The bromine solution is standardised against 0·1 *N* arsenite solution. A special form of burette is described which is convenient in use. The following quantities of oil should be taken:—

Iodine value	200	100	50	30	10
Weight of oil	0·05	0·10	0·20	0·30	1·00 grm.

H. E. C.

Identification of Glyoxylic Acid by means of Hydrazine and Xanthydrol. R. Fosse and A. Hieulle. (*Comptes Rend.*, 1925, **181**, 286–288.)—Treatment of xanthydrol with aqueous alcoholic hydrazine hydrate solution in presence of acetic acid yields hydrated trixanthylhydrazine, which melts at 159° to 162° C., resolidifies, and melts again at 167° to 175° C. Glyoxylic acid may be identified as follows: 1 grm. of xanthydrol and 200 c.c. of acetic acid are added, gradually and with shaking, to a mixture of 0·2 grm. of glyoxylic acid, 200 c.c. of water, and 5 drops of 50 per cent. hydrazine hydrate solution. After addition of water as long as further precipitation occurs the liquid is shaken, and the flocculent precipitate separated with the aid of the pump, washed, pressed between filter-paper, mixed in a centrifuge tube with 96 per cent. alcohol, and rendered feebly alkaline by means of *N*-alcoholic sodium hydroxide. Addition of acetic acid to the centrifuged liquid causes the formation of a crystalline precipitate which, when dissolved in chloroform and precipitated by petroleum spirit, and dried below 100° C., retains 1 mol. of water, this being lost at 105° C. or in a vacuum at a lower temperature; the anhydrous compound, dixanthyl-hydrazone-glyoxylic acid, has the formula $C_{28}H_{20}O_4N_2$. The silver salt was also analysed. T. H. P.

Detection of Methylhexaline in Textile Oils. J. Marcusson. (*Chem. Zeit.*, 1925, **49**, 656.)—Hexaline (hydrogenated phenol) and methyl-hexaline (hydrogenated cresol) are now incorporated, together with hydrocarbons, in some textile soaps. For the detection of hexaline or methyl-hexaline in the presence of tetraline, trichlorethylene and alcohol in the soap or an oil, about 150 grms. are acidified, then steam-distilled until the hydrocarbons and phenols have all passed over; then the distillate is shaken out with alkali to remove any fatty acids which have come over, and the residual oil is separated. This residue is benzoylated by heating with benzoyl chloride under a reflux condenser for about a quarter of an hour. Hydrocarbons are separated from the benzoyl ester by distillation with steam, and the residue, after all hydrocarbons have been removed, is extracted with ether, washed, and dried. The benzoyl esters are recognised in the ordinary way; they are both denser than water and have refractive indices 1·5103 and 1·5108 for hexaline and methyl-hexaline respectively (temperature not stated). One per cent. of hexaline can be detected in this way. H. E. C.

The Höchst Test for the Determination of Anthracene. F. H. Rhodes, M. L. Nichols and C. W. Morse. (*Ind. Eng. Chem.*,1925, **17**, 839–842.)—The following modification is recommended with the object of rendering the Höchst method more rapid and decreasing losses due to oxidation of the anthraquinone by the chromic acid. One grm. of the sample is boiled with 45 c.c. of glacial acetic acid in a flask attached to a reflux condenser, and a solution of 300 grms. of chromic acid in 400 c.c. of 50 per cent. acetic acid is added, drop by drop, until the liquid in the flask shows a permanent light brown colour; the mixture is then boiled for a further two hours. After cooling, the mixture is diluted with 400 c.c. of water, the anthraquinone is collected on a filter after thirty minutes, washed with hot water, then with 500 c.c. of boiling 1 per cent. sodium hydroxide solution, and again with 500 c.c. of hot water. The anthraquinone is transferred to a flat-bottomed porcelain basin, dried on a water-bath, 10 c.c. of fuming sulphuric acid (10 per cent. oleum) are added, the mixture is heated on a water-bath for ten minutes and then placed over water under a bell-jar for twelve hours. After this time the contents of the basin are diluted with 200 c.c. of water, the anthraquinone is collected on an asbestos filter, washed with water, sodium hydroxide solution, and water as described above, dried at 110° C., and weighed. The anthraquinone is then sublimed at 350° C., and the basin re-weighed. To the weight of anthraquinone thus found is added 0·0004 grm., and the result is multiplied by 0·9558 to obtain the weight of the anthracene present. W. P. S.

Inorganic Analysis.

Determination of Oxygen and Nitrogen in Commercial Electrolytic Hydrogen. W. Steuer. (*Chem. Zeit.*, 1925, **49**, 713.)—The errors attending the determination of oxygen in hydrogen by absorption with alkaline pyrogallol solution may be greatly diminished as follows: Use is made of two Hempel or Winkler burettes, connected by means of a transparent quartz capillary, 8 to 10 cm. long and 1 mm. in bore, containing a platinum wire 3 cm. in length and 0·8 mm. thick. The connection between the capillary and the second burette is broken, and the first burette filled through the capillary with the hydrogen to be analysed. Connection is then made with the second burette and, while the capillary is heated by a very small Bunsen flame, the gas is passed slowly into the second burette and back again. After repetition of this operation, the contraction (K_1) in volume is measured. The second burette is again disconnected, charged with about 60 c.c. of oxygen, and once more connected, the mixed gases being passed as before through the heated capillary. If the contraction now occurring is K_2, the percentage of oxygen in the gas is $K_1/3$, that of hydrogen $2(K_1+K_2)/3$, the rest being nitrogen. Hydrogen in electrolytic oxygen may be determined similarly. T. H. P.

Electrolytic Determination of Antimony. A. Schleicher and L. Toussaint. (*Chem. Zeit.*, 1925, **49**, 645–646.)—The following mode of working was found to give serviceable results in the analysis of antimonious chloride: Solution of 0·3 grm. in 15 to 20 c.c. of hydrochloric acid, dilution to 100 to 120 c.c., and addition of one grm. each of tartaric acid and hydrazine sulphate. The liquid is heated to 60° C. and electrolysed at 0·6 V. with a rapidly rotating anode. After 10 to 15 minutes, another 0·5 grm. of hydrazine sulphate is gradually added, alternating with 0·2 grm. of ammonium persulphate, also added in portions (*cf.* ANALYST, 1923, **48**, 291). The initial current density is 0·35 amp. The deposition is complete after about 30 minutes; the addition of the tartaric acid may be dispensed with, but as it does not interfere and prevents possible hydrolysis, it may sometimes be of advantage. W. R. S.

Determination of Ferric Oxide in Insoluble Silicates. O. Hackl. (*Zeitsch. anal. Chem.*, 1925, **66**, 401–430.)—The method consists in the decomposition of the silicate by hydrofluoric and sulphuric acids and titration with 0·1 *N* titanous chloride solution, with thiocyanate as internal indicator (*cf.* ANALYST, 1924, **49**, 352). The interference of hydrofluoric acid (which prevents the formation of ferric thiocyanate) is counteracted by the addition of boric acid (150 c.c. of 4 per cent solution for 10 c.c. of hydrofluoric acid). The paper is a lengthy description of tests carried out with pure iron salts to test the accuracy of the volumetric process, but it contains no particulars of work done on silicates. The tests prove that oxidation during the manipulation will cause an error of about 0·002 grm. Fe_2O_3; this is of the same order as the error in the usual processes of determination of ferrous oxide in insoluble silicates. W. R. S.

Physical Methods, Apparatus, etc.

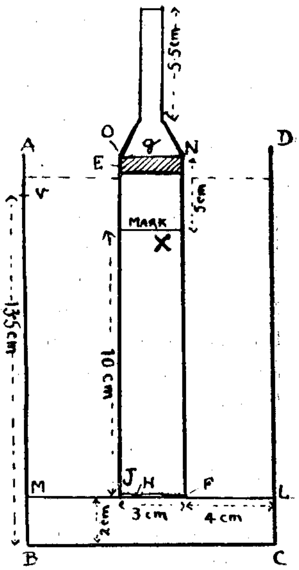
Simple Low-Temperature Thermometer. W. A. Noyes. (*J. Amer. Chem. Soc.*, 1925, **47**, 1942.)—This has been devised for the approximate determination of temperatures between the boiling-point of liquid air and zero. It consists of a bulb of capacity 2·4–5 c.c. blown at the end of a capillary, 50 cm. long and 0·8 mm. bore. A small globule of mercury, enough to occupy 5 mm. of the capillary, is introduced and brought about 10 cm. into the capillary, which is then sealed. Taking the length of the column of air above the mercury as 1 at 0°, the length at any other temperature may be calculated approximately by the formula 273 × 1/T, in which T is the absolute temperature. This gives the following values:

Temp. °C.	0	−20	−40	−60	−78·5	−100	−120
273 × 1/T	1	1·079	1·172	1·282	1·404	1·578	1·784

Temp. °C.	−140	−160	−180	−190	−191·6
273 × 1/T	2·052	2·415	2·936	3·286	3·351

The instrument is calibrated in melting ice, a mixture of carbonic acid snow and alcohol (−78·5° C. at 760 mm.), and in fresh liquid air (−191·62° C.). It can be seen from the table that the thermometer is most sensitive at low temperatures. R. F. I.

Viscometry of Highly Viscous Fluids (Rubber Solutions). R. Ditmar. (*Chem. Zeit.*, 1925, **49**, 676–677.)—A simple and easily-cleaned apparatus has been designed for use with highly viscous fluids, such as 15 per cent. rubber solution. It consists of a beaker, A B C D, of dimensions shown in the figure, having a wide mesh wire gauze, M L, 2 cm. from the bottom. It is filled with the liquid as far as the mark V. O J F N is a tube of 3 cm. diameter, having a fine wire gauze of about 1 mm. mesh fastened across the lower end. A highly viscous fluid will take longer to rise through the gauze up to the mark X than will a fluid of lower viscosity; the time of rise is proportional to the viscosity. It is, therefore, only necessary to push the tube as quickly as possible down to M L, and note the time taken for the fluid to rise to the mark. The results are reduced to proper units by standardising against a liquid of known high viscosity. The apparatus is suitable also for the examination of solutions of glue or celluloid, and its range may be extended by the use of different sizes of wire mesh.



H. E. C.

Porous Electrode for Oxidations or Reductions. M. Knobel. (*Ind. Eng. Chem.*, 1925, **17**, 826.)—A hole, 0·25 inch in diameter, is drilled axially nearly the whole length of a graphite rod, 0·5 inch in diameter and 3 inches in length, and a brass tube is tapped into the open end. The usual plating baths (lead, copper, zinc, nickel, iron, or silver) are used at a current density which would normally produce a smooth plating. While the electrode is plating, a current of air at a pressure of 0·25 to 0·5 atmosphere is maintained through it; care must be taken to keep the air passing through the sides of the electrode during the whole of the operation, or the pores will soon be closed; a fair thickness of metal (1 to 2 mm.) should be deposited to give the requisite strength. W. P. S.

Elimination of Surface Devitrification on Laboratory Silica Ware. F. C. Vilbrandt. (*Ind. Eng. Chem.*, 1925, **17**, 835–837.)—The devitrification of silica is a surface effect; it results frequently from the deposition of slag particles on the surface, and the silica becomes very brittle. Devitrified silica may be cleaned by immersing it for a few moments in hydrofluoric acid containing a small quantity of sulphuric acid; such treatment greatly increases the life of silica ware.

W. P. S.

Reviews.

ALLEN'S COMMERCIAL ORGANIC ANALYSES. Vol. III. Fifth Edition. Pp. ix. + 732. London: Churchill. 1925. Price 30s. net.

This volume of Allen's Organic Analysis has been thoroughly revised, and may fairly claim to be a full record of the properties and methods of examination of hydrocarbons, bitumens, naphthalene and anthracene, phenols, aromatic acids, and explosives. The individual articles leave an impression of thoroughness in compilation, and, in general, the methods described may be followed with confidence. There are, however, so many irritating misprints as to call for strong comment. One or two examples will suffice. The table of thermometric degrees on p. 721 has obviously gone astray. In the description of the method of testing for β-naphthol on p. 223, third line from bottom, there are two misprints, the substitution of "nitrate" for "nitrite" being serious.

The preface states that the late Dr. Sadtler's article on "Bitumens" was so nearly complete before his death as to need the addition of but few minor details. This is not the impression one gets from reading the contribution. The article is full, but should have been carefully revised, and we have no doubt the author would have done this. A method for the analysis of gas by the Orsat-Burrell apparatus is described on p. 69, where the lettering of the diagram of the apparatus is described, although the illustration itself is not reproduced. While one agrees cordially with the general remarks on lubricants on pp. 119 and 120, the paragraph dealing with the coefficient of friction on p. 121 is quite obscure. The description of the process for determining paraffin scale on p. 165 is either badly worded or is incomplete. Presumably the Pensky-Martens flash-point apparatus is meant on p. 136, where the "Pensky-Martin" closed cup tester is mentioned. The method described for sulphur in oil on p. 86 will usually give low results with oil low in sulphur, if the stated times of settling of the precipitate are adhered to. Directions on p. 178, implying that the carbon disulphide filtrate of bitumen should be burnt off, appear to be dangerous for any ordinary laboratory. The last paragraph on p. 158 seems meaningless.

The methods given by Dr. Sadtler for the testing of oils and lubricants are American methods, and while these are, on the whole, accepted here, there are some omissions which cannot be allowed to pass unchallenged, seeing that the work is presumably written for use in this country as well as in America. The "Tag" flash-point apparatus is fully described, and a similar full description should have been accorded to the standard Abel flash-point apparatus; its omission is unwarranted. In describing motor spirit or gasoline no reference is made either to the "toluene number" or to the work done on aromatics in this country. The section on viscosimetry likewise fails to refer to work done here as shown in the I.P.T. and B.E.S.A. publications.

The sections dealing with hydrocarbons, naphthalene and anthracene are full and satisfactory, little, if anything, of importance being omitted. A protest must be made against the use of such laboratory slang terms as "Oxidise 25 c.c. aliquots with vanadic acid" (page 221).

The section on "Phenols" is an able and well-informed contribution to a rather difficult subject. The methods described on p. 293 for determining the composition of tar acids are excellent when carried out carefully, and are to be recommended in spite of their tediousness. The Ditz method for estimating *m*-cresol on p. 287 does not give true results, because the assumption that *o*- and *p*-cresols stop at the dibromide stage when *m*-cresol has formed the tribromide is not quite correct. Cresote oil analysis is confined, in this section, to methods accepted in the United States, but we prefer the simpler methods used here, as exemplified by B.E.S.A. specification No. 144, for they give nearly all the information wanted for cresote oil for timber. Full details of the Rideal-Walker method of testing disinfectants, as well as the U.S. Hygienic Laboratory method, are supplied. No reference, however, is made to the important contribution to this subject by Chick and Martin as long ago as 1908. Lowe's method of assaying carbolic acid is not described. No doubt the method is somewhat faulty, but so long as it remains a purchasing method here, it ought to be included in any work on commercial analysis dealing with phenols.

Aromatic acids, gallic acid and phthalic acid are dealt with in separate sections in a satisfactory manner. Full and accurate accounts of the properties, methods of analysis, and detection of these substances are supplied.

The most recent information is incorporated in an admirable article on "Modern Explosives." It is noted that on p. 613 the reference to Silberrad, Phillips and Merriman is again given erroneously. On p. 638 bad misprints for the setting points of trotyl occur. It is not necessary to heat tetryl above its melting point before determining that constant, nor has the pure substance an acid reaction, as stated on p. 641.

Taking the volume as a whole, it would be an advantage to all concerned if correction slips could be issued to enable readers to correct misprints and errors.

J. J. Fox.

OFFICIAL AND TENTATIVE METHODS OF ANALYSIS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS. 2nd Edition, revised to July 1, 1924. Published by the A.O.A.C., Washington, U.S.A. 1925. Price $5·50.

Those who know the second edition of this work (revised to 1919) are aware that the field covered is much larger than that with which official agricultural chemists in this country are, as such, concerned. The methods of analysis described deal, indeed, with the major number of articles of food for man, and with many drugs. The sections dealing with fertilisers, soils, agricultural liming materials, plants, insecticides and fungicides, and feeding stuffs, number but six, in a total of thirty-two.

It may be explained to those unfamiliar with the work that it is a growth resulting from discussion and criticism at the annual meetings of the A.O.A.C. The methods are either "official" or "tentative": they are adopted on the recommendation of a referee in the first instance as tentative, and at subsequent meetings, after further critical study by referees and others, they may become official. The matters are thus subject to annual revision, and the work as now presented is the outcome of some forty years of collaborative effort on the part of many hundreds of American chemists.

This volume is no dry chronicle of regulation methods: it is an interesting book, and the treatment goes deep. For instance, water analysis goes as far as the determination of barium, strontium, lithium, manganese, iodine and bromine; in the section on meat and meat products the amino nitrogen method of Van

Slyke is found as a tentative method: methods for water- and acid-soluble boric acid are official for fertilisers, and so on.

It is difficult to see the reason for the inclusion of some of the alternative methods given. Where methods give absolute results, alternatives are an advantage, but in processes which are more of an empirical character it would seem to be preferable for one method only to be official. Here, however, for iodine values, both the Hanus and the Wijs methods are official; similarly both the Reichert-Meissl (alcohol saponification) and the Leffmann-Beam methods for the determination of soluble volatile acids are official; in both these cases, however, the caution is given that, in reporting, the method used should be specified.

There are no less than seven official methods for determination of reduced copper (after the cuprous oxide has been obtained) in the Munson and Walker general method for sugar, two being used only in solutions of reducing sugar of high purity.

The aim of the Committee charged with revision "to state the procedure with such lucidity and in such detail as to make it possible for any trained chemist to operate without being in doubt at any time," has been well carried out; occasionally directions might be more precise as, for instance, in the case of the determination of citric acid in fruit juices, &c., where the direct pentabromacetone method is allowed "when the quantity of sugar or other permanganate reducing substances is not excessive"; there may legitimately be a doubt when the excessive stage is reached.

The book is conveniently arranged and the system of cross references excellently devised; the editing has clearly been extremely careful.

The methods of analysis are naturally broadly familiar to analysts in this country, but much interest attaches to the particular methods chosen and to the precise modes of operation. There are points of detail to which English chemists will probably not give approval, and it is suggested that some further revision might have been given to the determination of boric acid in foods, and the details of the Polenske process for insoluble volatile acids; those who look forward with some misgiving to the coming into force of the Preservatives Regulations here would be glad to feel sure that all volatile sulphur oxidised by bromine is in truth sulphur dioxide.

The A.O.A.C. "Methods," indispensable in the United States, has been most useful to analysts in this country, and this revision will be much appreciated; the labours of the American chemists responsible for its production have earned a grateful tribute. E. HINKS.

MODERN INORGANIC CHEMISTRY. New Edition. By J. W. MELLOR, D.Sc. Pp. xv.+1103. London: Longmans, Green & Co. 1925. Price 12s. 6d. net.

The outstanding feature of the new edition is that it contains 193 pages more than did the previous edition, and yet its price has not been advanced. In the opinion of the reviewer, these additional pages have not added proportionately to the educational value of the book as a text-book of chemistry. Comparatively few of the additions appear to be of fundamental importance so far as the study of chemistry is concerned, although it should be said that they make very interesting reading. They might perhaps stimulate the imagination, but they render the student's task of searching for the chemistry more difficult.

The new matter, scattered throughout the volume, which has necessitated these extra pages, comprises chiefly an increase in the number of quotations given at the headings of those paragraphs which previously had been without them, and in a few instances, paragraphs which had possessed them have now been allotted either additional or different ones. The first chapter on "The Evolution of Chemistry" has undergone the most change, having been largely re-written on the lines of Chapter I. on the "Logic and Methodology of Science," in Dr. Mellor's "Comprehensive Treatise." The subject matter of the other chapters remains to a great extent unchanged, except for the additional matter, which usually deals with the historical and sometimes anecdotal side of chemistry. Thus, opening the book at random, it happens that on page 400 some new matter is found relating to a possible explanation why Moses was able to reduce Aaron's golden calf to ashes.

Some small additions have been made which deal with chemistry itself, though the following appear to be not beyond question. It is stated on page 537 that $BeSO_4, 7H_2O$ is isomorphous with the heptahydrate of magnesium sulphate, an error which has brought this text-book into line with some other modern elementary text-books. The existence of a heptahydrated sulphate of beryllium was claimed by one worker, Klatzo, but his contention has long been disproved by many other workers, and in fact, Dr. Mellor, in Vol. IV. of his Treatise states that the "analogies between beryllium and magnesium salts probably misled G. Klatzo into believing that possibly an imperfectly dried (beryllium) sulphate was analogous in composition with heptahydrated magnesium sulphate." The matter dealing with the so-called "chromosulphuric acids" is of a dubious character and would be better omitted from an elementary text book (p. 542). They are extremely improbable, and the formulae which have been assigned to them are based on little more than conjecture, especially the "chromopyrosulphuric acid," $Cr(OH)(HS_2O_7)_2$, with the radicle "HS_2O_7."

In writing this book in the first place Dr. Mellor scored a success in attempting to correlate physical with inorganic chemistry, and it is in this direction that further improvements in the presentation of the subject might have been effected. Many of the facts of inorganic chemistry could have been made more intelligible if the importance of the physico-chemical principles and methods had been made more manifest. Take the case of boric acid. Nothing is given of its strength, its dissociation constant and its mode of ionisation, although earlier in the book sufficient has been given of the dissociation theory to make such information intelligible. The result is that the student obtains a confused and inaccurate idea of the reactivity of that acid, for the neutralisation equation represents the formation of sodium metaborate, and yet on the same page it is stated that neither meta- nor pyro-boric acids exist in solution, but pass into orthoboric acid. The phase rule, although adequately treated in the volume, has not yet been made use of in the study of double and complex salts. Typical phase rule diagrams would have made clear the conditions under which they exist and may be prepared, and would thus have obviated the need of giving empirical methods of preparation and of attempting to explain their existence on purely valency grounds. This might have been done in the case of the polychromates, the acid sulphates, and the acid carbonates of the alkali metals.

It is a pity that the author has not taken this opportunity to eliminate a few points which were given in the previous editions, through probably a too enthusiastic zeal to systematise. Thus it is still suggested that tetravalent cerium may possibly form cerates, and thorium, thorates, and so be in conformity with the other members of the particular periodic sub-group. There is some reason for discussing cerium with thorium, but it is difficult to understand why praseodymium and neodymium should be considered with the niobium family, or lanthanum with scandium and yttrium.

Two typographical errors have been noticed. On page 774 in Serpek's process aluminium *hydride* should be *nitride*, and the word *langugage* appears on page 725.

As the book in general is written on the right lines and in the fascinating style of its author, there is no reason to doubt that it will maintain the wide popularity which it has already acquired. HUBERT T. S. BRITTON.

ORGANIC MEDICAMENTS AND THEIR PREPARATION. By ERNEST FOURNEAU. Authorised Translation by W. A. SILVESTER. Pp. x. + 262. London: J. and A. Churchill. 1925. Price 15s.

M. Fourneau's work is so well known to English chemists that a book by him is sure of a warm welcome. We all appreciate him as the Anglo-French punster in stovaine, as the chemical Gaboriau who tracked "Bayer 205" to its lair, as the author of those critical and sometimes pungent abstracts of current chemotherapeutical literature, which appear each month in *Chimie et Industrie*, and most of all as a chemist, who has made notable additions to the list of synthetic drugs.

In 1917 M. Fourneau was invited to give a course of lectures and laboratory instruction in the synthesis of drugs to a group of students in Spain and this book is one of the results.

In arranging this course M. Fourneau seems to have avoided the temptation to say something about everything in chemo-therapy and to have adopted the plan of talking about the things in which he was personally interested. Much is left out that a merely conscientious compiler would have added, but what is included is written by a master of his subject, and there can be no two opinions as to the wisdom of his method.

In his preface to the French edition Dr. Roux pointed out that no instruction in drug synthesis is given in either technical schools or universities in France. That is also true of Great Britain. In spite of this, both countries have made useful contributions to this subject, and both have sometimes left to other nations the commercial exploitation of their discoveries, possibly because neither of them is distinguished for the plodding patience necessary to turn a laboratory discovery into a commercial enterprise. It would be an interesting experiment to found at one of the universities a chemico-pharmacological institute in which selected chemical students would work through M. Fourneau's course of practical instruction, and the pharmacological students gain an insight into the experimental side of their science by testing on animals the products made by their chemical colleagues, and both would, after a time, proceed to joint research. Joint work on such lines is being developed in the United States, and it is to be hoped that the publication of the English edition of M. Fourneau's book will do something towards stimulating interest in these matters in this country.

M. Fourneau has been fortunate in his translator. Mr. Silvester has not only done his work well, but has added to the English edition a useful bibliography and a number of notes on recent developments, all of which are relevant and in keeping with the spirit of the original. T. A. Henry.

The Action and Uses in Medicine of Digitalis and its Allies. By A. R. Cushny. Pp. xi + 303. London: Longmans, Green & Co. 1925. Price 18s.

Any chemist who reads M. Fourneau's book, reviewed above, must feel a certain sense of satisfaction at the indebtedness of therapeutics to chemistry. His professional pride will experience the proverbial fall when he turns to Prof. Cushny's volume, for there is probably no field in which chemists have interested themselves to less effect than that of natural drugs, and in that field one of the least satisfactory corners is occupied by digitalis and its allies, strophanthus, squill and the other heart poisons. With regard to most drugs it is safe to say that the chemist knows more about their constitution than the pharmacologist does about their action, but in the digitalis group, chemistry limps painfully and uncertainly in the wake of pharmacology.

Professor Cushny is able to tell us the little that is known about their chemistry in twenty pages, but he needs nearly 250 to summarise what he knows about their action in the human body.

It must not be supposed, however, that the book is without interest to chemists, for anyone plucky enough to embark on the difficult task of isolating the active principles of these drugs and the determination of their constitution will find his work facilitated if he checks it at every stage by the pharmacological and clinical evidence already accumulated, and Prof. Cushny's admirable summary of this evidence will prove of great value to such workers. The author is one of the few people in this country who takes a research worker's interest in the correlation of chemical constitution with pharmacological action, and no one will be better pleased than he, if in the next edition of this book he has to divide his available space more evenly between chemistry, pharmacology and therapeutics.

T. A. Henry.

The Manufacture of Sulphuric (Contact Process). By F. D. Miles. Vol. IV. of the Manufacture of Acids and Alkalis. By G. Lunge, Ph.D. Completely revised and rewritten under the editorship of A. C. Cumming, O.B.E., D.Sc. Pp. xv. +427. London: Gurney & Jackson. 1925. Price 36s.

The book, which is divided into ten chapters, commences with a short historical sketch of the manufacture of fuming sulphuric acid. The theoretical considerations involved in the contact process are then fully treated in the ensuing chapters.

It is notable that in the consideration of the theoretical and scientific aspects of the subject, their inter-relation with the practical point of view is most carefully borne in mind. The properties and analysis of oleum are fully described in Chapter II., and among many valuable data is included an alignment chart for oleum mixing. These alignment charts are of great value in saving time and labour when calculations of this nature have frequently to be carried out. Chapter III. describes the gas equilibrium and velocity of re-action; and, starting from the quantitative form of the "Law of Mass Action," the author shows the relative effect of temperature and gas concentration on the conversion of sulphur dioxide. The equilibrium constant Kp is usefully replaced by $k_p = \frac{1}{\sqrt{K_p}}$ as the values of the latter are easier to remember, and also increase as the equilibrium favours trioxide formation.

The question of catalysis, catalyst poisoning, and catalyst carriers, is followed by a description of the preparation of contact mass, and the recovery of platinum after "poisoning." Various forms of converters are described in detail with excellent illustrations, followed by an interesting account of heat exchangers, which includes a comparative table giving data as to the surface, temperatures, and amount of heat exchanged on different types of heat exchangers; this table would have been of much greater value if the over-all heat transmission coefficient had been given instead of the quantity of heat transferred per sq. ft. surface per hour. Furthermore, a column showing the velocities of the gas in each case would be useful, seeing that the heat transmission coefficient is almost directly proportional to the velocity. With these two columns added, one would have been able to obtain a comparison of the different types of heaters at a glance, in order to determine which construction is better from the point of view of heat transmission. Useful alignment charts are given for estimating the percentage conversion of dioxide from the Reich and other tests.

Chapter VI. is devoted to catalyst poisoning, acid mist and gas purification; another useful alignment chart is given for finding the weight of drying acid required for gases containing various amounts of water vapour. In the next chapter follows a very full description of the absorption of the trioxide leaving the converters, showing the influence of concentration of the absorbing acid, temperature, etc., on the absorption. Data are given as to the heat effects of acid mixing and trioxide absorption, with several examples worked out for different typical cases; the different construction of absorbers is discussed, and also the various systems of arranging these for working together.

The application of the vapour pressure data recently given by M'David (1924) enables calculations to be made as to the percentage of trioxide in the gas and in the acid entering the various absorbers in a system; in an example cited these calculated figures are seen to agree very closely with those actually found in the working of a plant. A short description is given of pumps that are used for circulating the acid, although no mention is made of the glandless plunger pumps that have come to the fore since the war.

The remaining chapters, VIII., IX. and X., are respectively devoted to a very detailed description of the Grillo, Tentelew and Mannheim plants. The details of the Grillo plant are, in the main, taken from the records of the D.E.S. plants erected during the war.

The question of heat exchange would seem to require more attention from a technical point of view, seeing that the fundamental principles involved are now more clearly defined. It is to be hoped that the use of alignment charts will be more generally adopted in other technical works.

Altogether, this is a most admirable volume presenting in most readable form a mass of sound workable data in connection with the principles on which the contact process is based. ERIC A. REAVELL.

RULES FOR COMPOSITORS AND READERS AT THE UNIVERSITY PRESS, OXFORD. By H. HART, M.A. Twenty-seventh edition. Pp. 135. London: Humphrey Milford. 1925. 2s. net.

In view of the large amount of discussion upon the advisability of standardising analytical methods, it is interesting to note the extending success of an attempt to standardise the words of the language in which the results of an analysis are expressed.

Originally this little book was compiled for the guidance of those who produce the books at the University Press, but it has now become known far outside its birthplace, as is shown by the fact that this is the thirteenth edition that has been available to the general public.

In the later issues the spelling of the words has been revised by the Editors of *The Oxford Dictionary*, who were in a position to make use of the immense quantity of literary material that had been accumulated for the purposes of that publication.

Although, where there is a permissible choice, most persons prefer to use the forms of words and the spelling to which they are accustomed, yet there are numerous points on which it is advisable to know the selected version of the majority of authorities, and here these amended rules will be found a useful guide.

The ground covered includes lists of words ending in "ise" or "ize" (in which, by the way, the choice seems somewhat arbitrary), the spelling of medical terms, and of chemical compounds, abbreviations, French technical press terms, names and examples of English founts of type, proof correcting, and so on.

Moreover, the book is not only a specialised dictionary for constant reference, but is also entertaining reading. EDITOR.

S.P.E. TRACTS. NO. IX. THE LANGUAGE OF ANATOMY. By W. C. MORTON, C.B.E., M.A., M.D. NO. XV. THE SPLIT INFINITIVE. By H. W. FOWLER. Oxford University. Price 2s. 6d. each.

The Society of Pure English has undertaken the difficult task of improving the standard of the language, and one of the means it uses for this purpose is the publication of a series of "tracts" dealing with some special subject or usage of words. These aim at indicating right principles and giving information upon which correct decisions may be based.

The first of these brochures under review, which deals with the terminology of anatomy, is edited, and has an introduction, by Dr. Bridges. Its contention is that all scientific nomenclature should have its basis in every national language, and that whatever classical or foreign words are introduced, these should be, as far as possible, adapted to the national speech. It directs attention to the wrong usage of many medical terms, and suggests a scheme of nomenclature in which the principles put forward are carried into practice. Incidentally the need of consistency is illustrated by a favourable comparison with chemistry, in which the terminology has been standardised by the use of special suffixes to denote various types of compounds.

Evidently from what is said here, chemistry is far ahead of medicine in the standardisation of its terminology.

The other tract, on the "Split Infinitive," is of more general application, since the question it discusses is of perennial recurrence, and not infrequently requires consideration even in a chemical report. Its author, Mr. W. H. Fowler, who is one of the editors of *The Oxford Dictionary*, pithily classifies the English-speaking world into the following groups according to their attitude towards this rule of construction:—(1) Those who neither know nor care what a split infinitive is; (2) those who do not know, but care very much; (3) those who know and condemn; (4) those who know and approve; and (5) those who know and distinguish.

The text makes it quite clear that the author belongs to the fifth group. He makes one feel that, while it is often, or even usually, wrong to split an infinitive, there are cases in which splitting is less of an evil than a too obvious attempt to avoid it. "It is of no avail," he says, "merely to fling oneself out of temptation, one must so do it that no traces of the struggle remain; that is, sentences must be thoroughly remodelled, instead of having a word lifted from its original place and dumped elsewhere." And again: "It does not add to a writer's readableness if readers are suddenly pulled up now and again to wonder—why this distortion? Ah, to be sure, a non-split die-hard!"

In short, the moral, which is enforced by numerous examples, is that it is better to split an infinitive occasionally than to be ambiguous or artificial, and that even reconstruction is not always a good alternative. EDITOR.