DECEMBER, 1916. Vol. XLI., No. 489.

THE ANALYST.

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

AN ordinary meeting of the Society was held on Wednesday evening, November 1, in the Chemical Society's Rooms, Burlington House. The chair was occupied by the President, Mr. G. Embrey, F.I.C., and later by Mr. J. H. B. Jenkins, Vice-President.

The minutes of the previous ordinary meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. Gerrard W. Baker, 3, Haling Park Road, South Croydon, analytical chemist (now serving with the Royal Army Medical Corps), was read for the first time.

The following papers were read: "Quantitative Microscopy," by T. E. Wallis, B.Sc., F.I.C.; "Formula for converting Zeiss Butyro-Refractometer Readings into Refractive Indices," by C. C. Roberts, M.A., A.I.C.; and a "Criticism of Vaubel's Bromine Values," by Cecil Revis and H. R. Mitchell.

QUANTITATIVE MICROSCOPY.

BY T. E. WALLIS, B.SC. (LOND.), F.I.C.

(*Read at the Meeting, November* 1, 1916.)

INTRODUCTION.

THE use of the microscope for determining the proportions of substances present in mixtures is generally regarded as an unreliable method of procedure, and the results obtained have failed to carry weight as evidence in courts of law.

The difficulties experienced in such work arise partly from the methods of manipulation that must be followed for microscopical work, and partly from the nature of the substances examined. A common method of working has been to mix the powder with a fluid medium in a mortar, transfer a drop to a microscope slide, apply a cover-glass, examine with the microscope, and take a count of the number of certain characteristic structures seen in a particular number of fields. This result is then compared with that obtained by making similar counts of preparations made in an exactly similar way from powders of known composition.

Such a process is liable to error for various fairly obvious reasons, as, for instance, the variation in the size of the drop of liquid taken up by a glass rod and the dependence of the thickness of the film of liquid examined upon the weight of the cover-glass, the amount of pressure used in mounting, and the nature of the fluid. Moreover, no guidance is given as to the selection of the fields to be counted, and no indication is provided as to the range of error to which the resulting figures are subject.

Further difficulties arise from the smallness of the quantities actually examined during the process. For most ordinary work, I have found that a fluid containing 0·2 grm. of substance in 10 to 20 c.c. is suitable for examination with a one-sixth inch (4 mm.) objective, which it is necessary to use when one desires to count with precision. The depth of fluid between the cover-glass and slide must not exceed about 0·1 mm., and with a cover-glass of 19 mm. ($\frac{3}{4}$ inch) diameter the total volume of fluid is about one thirty-fifth of a cubic centimetre. Since 10 to 20 c.c. contain 0·2 grm. of the powder, the amount of material under the cover-glass is from two to four sevenths of a milligram. The diameter of the field given by the one-sixth inch objective that I use is 0·53 mm., and its area 0·22 sq. mm., and, the area of a circular cover-glass of 19 mm. diameter being 284 sq. mm., there are 1,291 fields under the cover-glass, and each field will represent approximately a quantity of material varying from about two to five ten-millionth parts of a gram. In twenty fields one is concerned with about one 250th to one 100th part of a mgrm. of material, and it is upon a correct analysis of this minute quantity of the substance that the final result depends. When one considers this fact, in addition to the uncertainty and lack of precision arising from the methods commonly adopted, one cannot wonder that figures based upon microscopical methods are regarded with a certain scepticism, and it is easy to understand the failure of such methods, in the absence of any exhaustive investigation of their worth, to carry weight as evidence of extent of adulteration.

It has also been recommended in the case of mixtures of starches that photographs of preparations from mixtures containing known percentages of the mixed starches should be made, and that the proportion present in an adulterated starch should be estimated by comparison of a photograph of a preparation from the sample with the photographs of the standard mixtures. This method must inevitably be unsatisfactory, since the ratio of the two kinds of starch grains to one another varies considerably from field to field of the same preparation, and a true proportion can only be obtained by counting a sufficiently large number of fields. The accompanying figures, representing counts of potato and maize starch granules in a 50 per cent. mixture of the two, illustrate this point. The mixed starches were rubbed down in a mortar with dilute glycerol, and the counts of ten fields, selected according to the plan suggested below, were as follows:

Potato starch	28	18	28	21	25	19	18	18	34	22
Maize starch	185	140	171	186	195	183	158	188	174	173

If the number of potato starch grains in each field is represented by 100, the numbers representing the maize starch are 661, 778, 611, 886, 780, 963, 878, 1,044, 512, and

786 respectively, figures which show a difference of 100 per cent. between the extreme values.

The types of substance to be examined may be classed as follows:

1. Material consisting of portions all of the same kind and all varying in size within certain definite limits, such as starches, pollen grains and spores.

2. Material that is all of the same kind—*e.g.*, wholly cellulose or wood—but composed of particles of sizes varying between arbitrary limits, depending upon the brittleness or toughness of the substance and upon the method of sifting or grading. Fibres, pine-wood sawdust, powdered coco-nut shells, and olive stones, are examples.

3. Material presenting a variety of structures, among which there are some, such as starch grains, crystals, stone cells, etc., which are of diagnostic value. The majority of food-stuffs and drugs fall under this head.

The difficulty of dealing with these types increases in the order in which they have been enumerated, and although one may be able, with a small amount of preliminary research, to obtain results of sufficient accuracy with the more simply constituted materials, it will be only after extended investigation that one can hope to decide upon the line of approach that will lead to the successful application of a reliable method of counting in the case of a substance exhibiting structures of many different types. The work recorded in this communication relates to experiments whose aim has been to devise a general method of procedure that will give precision to counts made under the microscope, and so to enable one to obtain, by the use of this instrument, quantitative results which will carry conviction in much the same way as do figures based upon ordinary chemical processes.

Admixture of a Substance as a Standard for Comparison.

The plan adopted by bacteriologists for the counting of bacteria in a vaccine by mixing it with blood and determining the ratio of bacteria to red blood-corpuscles suggested the idea that, if one could add to powders a substance consisting of uniform grains corresponding to the corpuscles, it would be possible to use a similar device in the case of powders. This procedure eliminates the errors due to variations in the size of the drops falling from the glass rod used in making the mount, and renders it unnecessary to bring the volume of liquid to any exact measure. The difficulty arising from variation in the thickness of the film of liquid between the cover-glass and the glass slide is also removed. A substance suitable for admixture in this way must exhibit certain important characteristics:

1. It must consist of grains of uniform size and having dimensions comparable with those of starch grains.

2. The grains should be fairly resistant to pressure, and have strongly marked characters clearly differentiating them from all ordinary vegetable structures.

3. It should be unaffected by clearing reagents and stains used in microscopical work—that is, it must not be rendered invisible or be destroyed by such things as caustic soda, chloral hydrate, clove oil, and strong hydrochloric acid.

4. It must be fairly easy to obtain the substance commercially.

It is somewhat difficult to find a suitable material for admixture; but after a few trials I concluded that something which occurs naturally in uniform grains could be the only possibility. I finally decided to try lycopodium spores, which are highly resistant to strong clearing reagents and have a diameter of from 20 μ to 28 μ, which is not larger than that of many starch grains. Their characteristically sculptured surface and tetrahedral shape makes them easy to identify, and distinguishes them clearly from all structures commonly found in vegetable powders. A few experiments made it evident that they fulfilled the requirements exactly.

Use of a Suspending Medium.

In my earlier experiments I tried working with the dry powders without any suspending fluid. The lycopodium and the powder under investigation were thoroughly mixed, and then a small portion was taken with the point of a knife and placed upon a glass slide, moistened with alcohol or, in the case of a woody adulterant, with a 1 per cent. solution of phloroglucinol in alcohol, dilute glycerol or other mountant added, the whole stirred with a glass rod, and a cover-glass applied. The results were surprisingly exact; but every now and then a count which was obviously very far from correct would be obtained, and this was due to the difficulty experienced in producing a perfect distribution of the constituents of the mixed powder. Such variations introduce an element of uncertainty, and make it impossible to determine the proportions of the materials in the mixture so satisfactorily that one would feel justified in reporting the figures found.

To obtain a more complete mixing and to prevent the separation of heavier from lighter particles by the liquid, a number of suspending agents were tried. Such a substance should possess the following characters:

1. Its density should be less than that of lycopodium spores—*i.e.*, less than 1·086—so that all the material will tend to sink, and not be separated into a floating layer and a sediment consisting of entirely different materials.

2. The viscosity should be moderately high, so that the suspended powders may sink slowly.

3. The medium should wet the powders readily and penetrate the substance of the walls of cellular structures, so that air is excluded and the particles appear transparent.

4. It must wet oily, as well as dry, and moist powders.

5. It should prevent the deposition of the suspended matter, or should allow the sediment to be easily shaken up to form an evenly distributed suspension.

6. It must mix readily with such reagents as alcohol and strong hydrochloric acid.

There is no substance which satisfies all these conditions; but there are a few which show a near approach, and by varying the details of working to meet special requirements one may hope to find a medium suited to every case.

Of the two common mucilages which immediately suggest themselves as suspending agents, mucilage of acacia closely approaches the condition of a true solution, and for this reason behaves as a viscous fluid of considerable density. It is

not very suitable for microscopical work, because it completely separates a mixture containing lycopodium spores, which after an interval of about twenty-four hours form a floating layer, while starch and other matters collect as a sediment. The whole shakes up again fairly well, but it is undesirable to use any medium which effects so complete a separation of the substances to be counted. Its stickiness and property of drying rapidly to form a hard, brittle film when spread in a thin layer are also features which militate against the use of mucilage of acacia.

Mucilage of tragacanth has an entirely different structure; it depends for its efficacy upon the presence of a fine network of cellulosic material distributed through an aqueous fluid. For this reason the mucilage tends to keep suspended particles in a more or less fixed position with respect to one another. That this is the case I have shown by making counts after suspensions have stood undisturbed during an interval of from three to five days, when the proportions of lycopodium spores and of other particles have been found to remain constant. The suspending properties of the mucilage are unaffected by alcohol and by strong hydrochloric acid. Mucilage of tragacanth is therefore a very efficient suspending agent, and is most useful for quantitative work with the microscope.

There are, however, certain drawbacks to the use of mucilage of tragacanth, such as the occurrence of a few granules resembling starch grains, its lack of complete transparency, and its unsuitability for use with oily powders. In the majority of cases the starch grains introduce no difficulty, as they are only few in number and are readily distinguishable from most other starches. The medium is rendered more transparent by using a mixture of 1 volume of glycerol and about 4 volumes of mucilage of tragacanth. If used for oily powders, the fatty matter must be removed by a preliminary extraction with a suitable solvent.

Oils, such as olive oil and castor oil, have proved extremely useful, not only in the case of oily powders like mustard and pepper, but also for general use with powdered vegetable substances. Their densities are slightly less than that of water, and hence all the suspended matters are deposited on standing, and settling takes place slowly, owing to their considerable viscosity. In the case of olive oil, the sediment is easily shaken up again to form a uniform mixture; but with castor oil this is not effected so readily, and consequently, owing to its very high viscosity, castor oil is not so useful as a suspending agent. The great penetrating power of the oils causes them to mix readily with dry powders, to displace air from cavities, and to render all the particles transparent. Oils are useful for starches, especially when one wishes to use the polariscope to aid in distinguishing one kind of grain from another.

The possibility of using soft paraffin suggested itself as the result of experience gained while examining an ointment composed of mucilage of starch and vaseline. A few experiments demonstrated its utility as a suspending medium, and an example of its application is given under the heading of mustard among the experiments cited below. Soft paraffin is less generally useful than olive oil, because more time and trouble are needed to obtain a satisfactory mixture of the powders and medium than is the case with the oil. Also, when polarised light is used, the crystalline structure of the paraffin produces a confused network of bright lines which tends to

obscure the objects to be counted. It is quite possible, however, that the occasion may arise when the peculiar properties of soft paraffin will indicate its employment in preference to any other suspending agent.

Glycerol can be used successfully, but its high density results in a complete separation of the lycopodium as a floating layer after the preparation has stood for some hours, and its great viscosity increases the difficulty of redistributing the materials evenly when the suspension is shaken up again. A mixture of alcohol and glycerol in equal volumes has been recommended for suspending such materials as mixed starches ("Admixture of Oatmeal with Barley Meal," by E. L. Cleaver, ANALYST, 1877, I., 189); but alcohol decreases the viscosity to such an extent that one finds considerable difficulty in producing a uniform mixture of the powders with the fluid and in breaking up small groups of spores or starch grains. Mixing the powders first with glycerol and thinning with alcohol afterwards is also unsatisfactory.

GENERAL STATEMENT OF THE METHOD OF WORKING.

Expressed in general terms, one proceeds in the following way:

1. Make a mixture of the pure substance with an equal weight of the adulterant whose amount it is desired to determine. The two substances may either be dried at 100° C. or, preferably, used air-dry; estimate the moisture present, and apply the necessary corrections in the calculations. Mix 0·2 grm. or other convenient weighed quantity of this standard mixture with 0·1 grm., or other suitable amount, of lycopodium and sufficient of the suspending fluid to produce a liquid of which 1 drop, when mounted and examined with a one-sixth inch objective, shall show from 10 to 20 lycopodium spores in each field. In most cases this result will be obtained when the total volume is about 20 c.c. A drop of the suspension is transferred to a slide by means of a glass rod and a cover-glass applied. Count the number of particles of adulterant and of lycopodium spores in ten fields selected according to the scheme detailed below. Mount a second drop on another slide, and again record ten counts. Find, for each set of ten counts, the ratio of the number of lycopodium spores to the number of characteristic elements of the adulterant, and express the results as the number of characteristic elements counted for every 100 lycopodium spores. The numbers found for the two sets of counts should not differ by an amount greater than 10 per cent.; should they do so, fresh counts must be made.

2. Mix 0·2 grm., or other suitable amount, of the sample in which the percentage of adulterant is to be determined with 0·1 grm., or other convenient amount, of lycopodium and about 20 c.c. of suspending fluid. Mount a drop on each of two slides, and count ten fields on each. Calculate the ratio of the number of spores of lycopodium to the number of characteristic elements of the adulterant, and express the result in the same form as for the standard mixture.

3. The numbers obtained for the foreign substance in the two sections of work are directly proportional to the amounts present, and a simple calculation gives the quantity sought. A correction must be applied for moisture. Further manipulative details are given below in describing the experiments which were planned and carried out in order to illustrate and test this method of making quantitative determinations by means of the microscope.

Discussion of Details.

The quantities of mixture and of lycopodium recommended for preparing the suspensions are such as have proved generally useful. In cases where the amount of material to be counted is very small, as in the determination of small percentages of powdered olive stones, the proportion of mixture should be considerably increased; and where the number of particles is very large, as when one is dealing with a small-grained starch, a greater proportion of lycopodium must be used. Examples of these two cases will be found among the experiments.

The powders used in preparing the 50 per cent. standard mixtures must be mixed very thoroughly by trituration in a mortar, turning out on a tile or sheet of glass and mixing with a spatula, returning to the mortar and repeating the process a few times until an intimate mixture is obtained. When dealing with powders in which woody structures are to be counted, the mixing should not be done on paper, since most paper contains woody tissue, and a certain small amount of the fibres from the surface of the paper works up into the powder and causes errors in making counts of woody elements. A 50 per cent. mixture is recommended for use in most cases instead of the pure adulterant, because difficulties that will arise in dealing with the sample to be tested will also arise with the standard mixture, and the operation of counting the sample will be facilitated and rendered more precise. There are occasions when counts made with the pure substance are very useful, as in the case of a mixture of two powders both containing woody elements or other diagnostic structures of which a particular kind is to be estimated. Some confusion may tend to arise between those proper to the powder and those belonging to the adulterant, and a useful check may be obtained by counting the pure adulterant against lycopodium as well as counting a 50 per cent. standard mixture. Close agreement between these two results will greatly strengthen one's reliance upon the accuracy of one's judgment in distinguishing the woody elements, starch grains, or other structures similar in type, but different in origin.

Before adding the liquids to the weighed quantities of lycopodium and other substances, these powders should be thoroughly mixed in a dry state by rubbing them together upon a sheet of glass with a spatula. This thorough trituration distributes the constituents much more evenly, and breaks down any little collections of adhering spores or starch grains. These mixings may be carried out in a mortar, provided that the trituration is gentle; otherwise the lycopodium spores will be largely broken. Mixing in a mortar presents no advantage, and, on the whole, the use of a plate and spatula as described is to be preferred, since it is almost impossible to break the spores by this means, and the mixing is quite satisfactory.

Choice of the Fields to be Counted.

In selecting the fields to be counted, care must be taken to avoid the possibility of counting the same field twice, and the positions of the fields chosen should be evenly distributed over the whole preparation. Their positions must also be fixed arbitrarily by some previously accepted arrangement, so as to avoid the errors that unintentionally arise in selecting fields which one thinks suitable for counting.

This selection can only be made by the use of a mechanical stage having graduations on the two movements at right angles to one another. For an instrument having a plain stage one can use one of the very satisfactory forms of attachable mechanical stage. The position of the fields to be counted is best fixed by choosing positions at certain distances from the middle point; if the slide is then placed on the stage so that the lens is over the centre of the cover-glass, one can note the reading for the position of the right-hand near corner of the slide on the two scales of the mechanical stage, and then move it precisely into the positions required for making the counts. For my own experiments I have counted ten fields on each slide, and the positions selected are shown in the accompanying diagram, all distances being

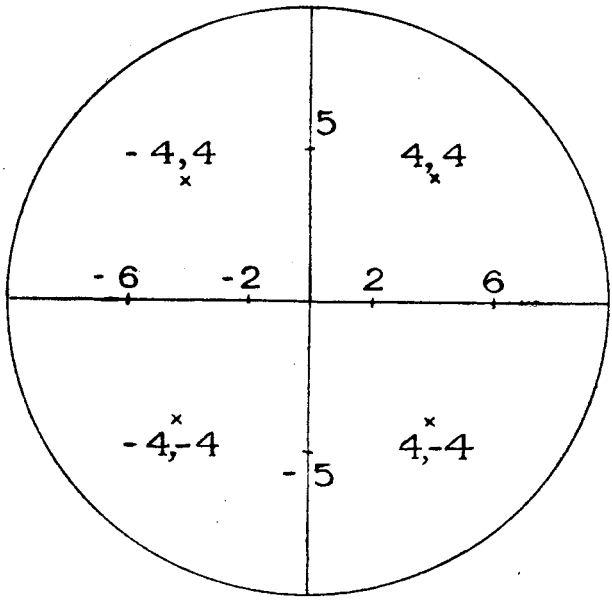


DIAGRAM SHOWING THE POSITIONS OF THE FIELDS COUNTED.

The numbers indicate distances in millimetres from the centre of the cover-glass.

given in millimetres from the centre of the cover-glass. To make the actual counts it is necessary to use an eye-piece micrometer ruled in squares, and to count regularly along each line of squares in succession until the whole field has been covered.

The number of fields to be counted will depend upon the intimacy of the mixing. If perfect mixing could be secured, a very few counts—say three—would be sufficient; but since each field contains only about one four-millionth of a grm. of substance and half that amount of lycopodium, it is very difficult and, in my experience, impossible to obtain by ordinary means so complete a mixing. One must therefore continue until so many fields have been counted that the counting of an additional one will not appreciably alter the ratio obtained. In actual practice I have found 20 fields give a reliable result; and if these are counted in two sets of 10 upon different slides, one set acts as a check upon the other, and so adds to the precision of the operation.

Record of Experiments.

The following examples will serve to illustrate the method employed, the kind of difficulties that arise in the course of such work, and the way in which modifications may be introduced in order to meet special conditions. The mixtures chosen for experiment are such as have been reported as occurring in actual practice, and consist of materials which cannot be determined quantitatively by other than microscopical methods.

Mixtures of Wheat Flour and Cornflour.—The mucilage of tragacanth used as a suspending agent was prepared from the dry powder at the time of mixing; 0·2 grm. of the mixed flours, 0·1 grm. of lycopodium, and about 0·12 grm. of powdered gum tragacanth, were carefully mixed together and put into a cylindrical weighing bottle of about 40 c.c. capacity. About 1·0 c.c. of alcohol was added and shaken with the powders; 20 c.c. of water were poured in rapidly, the stopper replaced, and the whole shaken vigorously for two or three minutes. The counts made were as follows:

Mixture of Wheat Flour 50 *per Cent. and Cornflour* 50 *per Cent.*—First set of ten fields:

Lycopodium ..	14	15	7	11	11	5	4	13	12	10	= 102
Maize starch ..	43	74	53	33	61	39	25	38	57	59	= 482

$$\text{Ratio } \frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{100}{473}.$$

Second set of ten fields:

Lycopodium ..	11	11	11	7	12	7	4	9	9	12	= 92
Maize starch ..	51	45	57	52	63	28	23	37	47	36	= 439

$$\text{Ratio } \frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{100}{477}.$$

$$\text{Average ratio } \frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{100}{475}.$$

Mixture of Wheat Flour 62·2 *per Cent. and Cornflour* 37·8 *per Cent.*—First set of ten fields:

Lycopodium ..	13	10	14	20	21	11	4	14	16	12	= 135
Maize starch ..	48	38	42	87	64	25	24	43	77	49	= 497

$$\text{Ratio } \frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{100}{368}.$$

Second set of ten fields:

Lycopodium ..	11	6	5	8	12	5	8	8	14	6	= 83
Maize starch ..	30	28	34	46	26	17	18	11	44	40	= 294

$$\text{Ratio } \frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{100}{354}.$$

$$\text{Average ratio } \frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{100}{361}.$$

$$\text{Amount of cornflour found} = \frac{361 \times 50}{475} = 38{\cdot}0 \text{ per cent.}$$

Mixture of Wheat Flour 90 *per Cent. and Cornflour* 10 *per Cent.*—Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{188}{187} = \frac{100}{99{\cdot}5} \text{ and } \frac{109}{101} = \frac{100}{92{\cdot}7}.$$

$$\text{With an average of } \frac{100}{96{\cdot}1}.$$

$$\text{Amount of cornflour found} = \frac{96{\cdot}1 \times 50}{475} = 10{\cdot}1 \text{ per cent.}$$

N.B.—Where the counts of the individual fields are not given, each ratio from a set of ten counts is expressed in two forms; the first fraction has for numerator and denominator the sum of the ten actual counts of lycopodium spores and starch grains or other characteristic elements respectively, while the second fraction has in every case 100 for the numerator, representing the lycopodium spores, and for the denominator the corresponding number, representing the starch grains or other structures. By dividing the numbers in the first fraction by 10, one has immediately the average number of grains or particles counted in each field.

MIXTURES OF POTATO STARCH AND MAIZE STARCH.—These mixtures were treated in the same way as the mixtures of wheat flour and cornflour.

Mixture of Potato Starch 50 *per Cent. and Maize Starch* 50 *per Cent.*—Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{58}{338} = \frac{100}{583} \text{ and } \frac{40}{218} = \frac{100}{545}.$$

$$\text{With an average of } \frac{100}{564}.$$

Mixture of Potato Starch 90 *per Cent. and Maize Starch* 10 *per Cent.*—Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{108}{117} = \frac{100}{108} \text{ and } \frac{72}{84} = \frac{100}{117}.$$

$$\text{With an average of } \frac{100}{113}.$$

$$\text{Amount of maize starch found} = \frac{113 \times 50}{564} = 10{\cdot}0 \text{ per cent.}$$

Mixture of Potato Starch 95 *per Cent. and Maize Starch* 5·0 *per Cent.*—Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{111}{63} = \frac{100}{56{\cdot}8} \text{ and } \frac{144}{82} = \frac{100}{56{\cdot}9}.$$

$$\text{With an average of } \frac{100}{56{\cdot}9}.$$

$$\text{Amount of maize starch found} = \frac{56{\cdot}9 \times 50}{564} = 5{\cdot}1 \text{ per cent.}$$

The method of making the mucilage of tragacanth at the moment of mixing has its advantages in aiding the even distribution of the constituent powders; but

if the drops of suspension are mounted and examined immediately, currents are produced, owing to the continued swelling of the gum. If a few hours are allowed to elapse until the mucilage has properly formed, this difficulty does not arise and the counts are easily made.

It may be suggested that, where a mixture of two starches is being dealt with, the addition of lycopodium is unnecessary, because the ratio of the two kinds of granule to one another can be obtained. Although this is true, it is preferable to add lycopodium, because by doing so a greater precision is secured. It is impossible to mistake lycopodium spores for starch grains, whereas if a few grains of one kind of starch are mistaken for those of a second sort, a double error results, since what is added to one side is subtracted from the other. So large an error cannot possibly occur when lycopodium is used; time also is saved, because it is much easier to count the spores than to count starch grains.

Up to this point no account was taken of the moisture present in the starches, and since the powders used in the various mixtures were identical with those used in the standard mixtures, no error was introduced. In ordinary practice it would be necessary to make corrections for moisture; hence in the remaining experiments moisture was always estimated and the corrections applied.

MIXTURES OF MUSTARD FLOUR AND CORNFLOUR.—The mixtures were made with cornflour dried at 100° C. For the standard mixture the mustard was also dried, but for the 25 per cent. mixture the ordinary air-dry powder was used. In place of mucilage of tragacanth, the 0·2 grm. of mustard and 0·1 grm. of lycopodium were mixed with olive oil by working them with a spatula upon a piece of glass until a thick paste was formed; this was further gradually diluted to the consistence of thin cream. The fluid was then transferred to a weighing bottle and more olive oil added until the volume was about 20 c.c. The whole was well shaken and a drop mounted for examination.

Mixture of Dried Mustard 50 *per Cent. and Dried Cornflour* 50 *per Cent.*—Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{194}{1289} = \frac{100}{664} \text{ and } \frac{100}{645}.$$

$$\text{With an average of } \frac{100}{655}.$$

Mixture of Air-Dry Mustard 75 *per Cent. and Dried Cornflour* 25 *per Cent.*—Moisture = 6·67 per cent. Hence dry substance = 93·33 per cent.

The mixture was dried before weighing out and mixing with the lycopodium and oil.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{66}{261} = \frac{100}{395} \text{ and } \frac{87}{307} = \frac{100}{353}$$

$$\text{With an average of } \frac{100}{374}$$

$$\text{Amount of cornflour found in the dry substance} = \frac{374 \times 50}{655} = 28{\cdot}5 \text{ per cent.}$$

$$\text{Or in air-dry substance} = 26{\cdot}6 \text{ per cent.}$$

In order to test the possibility of replacing olive oil by soft paraffin, fresh counts were made for the mixture containing 25 per cent. of cornflour. The weighed quantities of the powders were thoroughly incorporated with about 5 grms. of soft paraffin by means of a spatula and glass plate. A small amount of this strong mixture was diluted with more paraffin until examination under the microscope showed that a mixture containing a suitable proportion of the powder had been produced.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Maize starch grains}} = \frac{82}{303} = \frac{100}{370} \text{ and } \frac{95}{371} = \frac{100}{390}.$$

With an average of $\frac{100}{380}$.

Amount of cornflour found in the dry substance $\frac{380 \times 50}{655} = 29{\cdot}0$ per cent.

Or in air-dry substance = 27·1 per cent.

The remaining experiments were made with air-dry powders; this was done because dry vegetable powders are very hygroscopic, and may take up appreciable quantities of moisture during manipulation. The use of air-dry powders also results in a reduction of the time required for the whole operation.

Mixtures of Wheat Flour and Potato Starch.—It is generally easy to distinguish grains of wheat starch from those of potato starch; but there is a number of grains, forming about 10 per cent., of the potato starch which it is difficult to distinguish with certainty. With the polariscope wheat starch polarises feebly, whereas potato starch grains show a strongly marked cross. These differences are very clearly shown when the starches are mounted in oil, and for this reason olive oil was used as the mountant for the mixtures, and the counts were made with the polariscope, using crossed Nicols. Although the majority of wheat starch grains polarise feebly, one finds here and there one which shows a brilliant effect, owing to the fact that the grain is on its edge. These grains are, however, distinguishable, because the potato starch grains always show a cross formed by the intersection of two lines, whereas in the case of grains of wheat starch turned upon their edges the "cross" is composed of five lines, of which four are arranged in pairs bifurcating from the ends of the fifth line, thus >—<. Some few potato starch grains show a circular outline and a cross formed by the intersection of two diameters, much as one finds in wheat starch. Such grains may be distinguished from brightly polarising grains of wheat starch by the fact that in potato starch the lines forming the cross are usually thicker towards the circumference and taper off to the point of intersection, which is clearly marked, while the lines on wheat granules become wider as they approach the centre, which is itself marked by a darker circular area. Keeping these points in mind, it is possible to make an accurate count of potato starch mixed with wheat flour.

In each case 0·2 grm. of the substance was mixed with 0·1 grm. of lycopodium, and the powders worked up with olive oil in the manner described above for the mustard mixtures.

Mixture of Wheat Flour 50 *per Cent. and Potato Starch* 50 *per Cent.*—Moisture in the potato starch = 20·76 per cent.

Hence the mixture contains 39·62 per cent. of potato starch dry at 100° C. Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Potato starch grains}} = \frac{86}{68} = \frac{100}{79{\cdot}1} \text{ and } \frac{108}{85} = \frac{100}{78{\cdot}7}.$$

$$\text{With an average of } \frac{100}{78{\cdot}9}.$$

Mixture of Wheat Flour 80 *per Cent. and Potato Starch* 20 *per Cent.*—Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Potato starch grains}} = \frac{151}{51} = \frac{100}{33{\cdot}8} \text{ and } \frac{154}{56} = \frac{100}{36{\cdot}4}.$$

$$\text{With an average of } \frac{100}{35{\cdot}1}.$$

Hence percentage of dry potato starch in the mixture $= \frac{39{\cdot}62 \times 35{\cdot}1}{78{\cdot}9} = 17{\cdot}6$, or 22·2 parts of air-dry starch.

Mixtures of White Pepper and Ginger.—In this case the powders were mixed with castor oil, using a spatula and a sheet of glass; the oil was added until a suitable dilution was obtained. In each experiment 0·2 grm. of the mixture and 0·1 grm. of lycopodium were used.

Mixture of White Pepper 50 *per Cent. and Ginger* 50 *per Cent.*—Moisture in the ginger = 13·91 per cent.

Hence the mixture contains 43·05 per cent. of ginger dry at 100° C.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Ginger starch grains}} = \frac{81}{247} = \frac{100}{305} \text{ and } \frac{68}{206} = \frac{100}{303}.$$

$$\text{With an average of } \frac{100}{304}.$$

Mixture of White Pepper 90 *per Cent. and Ginger* 10 *per Cent.*—Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Ginger starch grains}} = \frac{343}{234} = \frac{100}{68{\cdot}2} \text{ and } \frac{135}{87} = \frac{100}{64{\cdot}4}.$$

$$\text{With an average of } \frac{100}{66{\cdot}3}.$$

Hence percentage of dry ginger in the mixture $= \frac{66{\cdot}3 \times 43{\cdot}05}{304} = 9{\cdot}4$, or 10·9 per cent. of air-dry ginger.

Mixtures of White Pepper and Rice Starch.—Rice starch consists of very small grains, and, judging from the relative size of maize starch grains, it seemed probable that, for a preparation containing starch and lycopodium in the proportion of 0·2 of the former to 0·1 of the latter, the number of grains to be counted in each field would approach 2,000, which is too large a number for comfortable working. The proportions were accordingly altered, and for this set of experiments

0·2 grm. of lycopodium was mixed with 0·05 grm. of starch or pepper. Olive oil was used as the fluid medium, and the volume was made up to about 20 c.c. as before.

In addition to counting a standard mixture of pepper and rice starch, pure rice starch mixed with lycopodium was also counted. This was done to accustom the eye to the size and appearance of the rice starch, and also to give a ratio that would act as a check upon the remainder of the work.

To count rice starch in the presence of pepper is not so difficult as might be anticipated, and that for two reasons. In the first place, by far the greater part of the pepper starch occurs in angular masses, so that the number of loose grains is small; and, secondly, pepper starch grains vary in size from 0·5μ to 5·0μ, while rice starch grains vary from 5μ to 8μ in diameter, so that, if one omits all starch grains having a diameter less than half that of the larger rice starch grains, the count obtained will represent the rice starch.

Pure Rice Starch.—Moisture in the rice starch = 15·96 per cent.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Rice starch grains}} = \frac{218}{5855} = \frac{100}{2686} \text{ and } \frac{181}{5135} = \frac{100}{2836}.$$

$$\text{With an average of } \frac{100}{2761}.$$

Mixture of White Pepper 50 per Cent. and Rice Starch 50 per Cent.—This mixture contained 42·02 per cent. of rice starch dry at 100° C.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Rice starch grains}} = \frac{211}{2670} = \frac{100}{1266} \text{ and } \frac{141}{1703} = \frac{100}{1208}.$$

$$\text{With an average of } \frac{100}{1237}.$$

Mixture of White Pepper 80 per Cent. and Rice Starch 20 per Cent.—Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Rice starch grains}} = \frac{200}{921} = \frac{100}{460{\cdot}5} \text{ and } \frac{265}{1232} = \frac{100}{465}.$$

$$\text{With an average of } \frac{100}{463}.$$

Hence percentage of dry rice starch in the mixture $= \frac{463 \times 42{\cdot}02}{1237} = 15{\cdot}72$, or 18·71 per cent. of air-dry rice starch.

The figure 1237 for the rice starch in the 50 per cent. mixture is about 10 per cent. smaller than the figure 1380 obtainable by calculation from the pure starch. This is due to one's anxiety to avoid the inclusion of pepper starch in the counts, and the consequent omission of some of the smaller rice starch grains. This fact also emphasises the desirability of using a mixture for the standard rather than to base the calculations upon a figure obtained from counts of the pure adulterant.

MIXTURES OF GENTIAN ROOT AND COCONUT SHELL.—The powdered gentian root was an ordinary commercial sample, and the coconut shell was a No. 80 powder prepared in the laboratory. In the case of the pure coconut shell, the 50 per cent. and the 5 per cent. mixtures, the mixed powder and lycopodium were rubbed together on a glass plate with about 1·0 c.c. of a 1 per cent. solution of phloroglucinol in alcohol until nearly dry; 1·0 c.c. of strong hydrochloric acid was next added, and the mixing continued; 3 c.c. of glycerol were then incorporated, and the volume adjusted by gradually adding mucilage of tragacanth. The whole was transferred to a weighing bottle and well shaken. For the 29·3 per cent. and the 8·5 per cent. mixtures, about 0·12 grm. of powdered gum tragacanth was added, and all the powders were thoroughly mixed in the dry state. They were then treated with phloroglucinol, hydrochloric acid, and glycerol as described above. The thin paste thus produced was transferred to a weighing bottle, water was added, and the whole shaken vigorously until a uniform mixture resulted; the suspension so formed was very satisfactory.

No attempt was made to count the individual stone cells present; any portion or group of stone cells was counted as one unit. The bright rose-pink colour assumed by the woody elements of gentian helped in distinguishing them from the coconut shell, which nearly always takes a yellowish-red colour.

Pure Powdered Coconut Shell.—Moisture in the coconut shell = 12·42 per cent.

The quantities used were: Coconut shell 0·2 grm. and lycopodium 0·1 grm.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Stone cells}} = \frac{235}{197} = \frac{100}{83{\cdot}8} \text{ and } \frac{230}{191} = \frac{100}{83{\cdot}0}.$$

$$\text{With an average of } \frac{100}{83{\cdot}4}.$$

Mixture of Gentian Root 50 per Cent. and Coconut Shell 50 per Cent.—This mixture contained 43·79 per cent. of coconut shell dry at 100° C.

The quantities used were: Mixture 0·2 grm. and lycopodium 0·1 grm.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Stone cells}} = \frac{164}{71} = \frac{100}{43{\cdot}3} \text{ and } \frac{160}{62} = \frac{100}{38{\cdot}8}.$$

$$\text{With an average of } \frac{100}{41{\cdot}05}.$$

Mixture of Gentian Root 70·7 per Cent. and Coconut Shell 29·3 per Cent.—The quantities used were: Mixture 0·4 grm. and lycopodium 0·05 grm.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Stone cells}} = \frac{65}{55} = \frac{100}{84{\cdot}6} \text{ and } \frac{76}{67} = \frac{100}{88{\cdot}2}.$$

$$\text{With an average of } \frac{100}{86{\cdot}4}.$$

If the quantities used had been the same as for the 50 per cent. mixture, the value 86·4 would become $86{\cdot}4 \div 4 = 21{\cdot}6$.

Hence percentage of dry coconut shell in the mixture $= \frac{21{\cdot}6 \times 43{\cdot}79}{41{\cdot}05} = 23{\cdot}04$, or 26·3 per cent. of air-dry coconut shell.

Mixture of Gentian Root 91·5 *per Cent. and Coconut Shell* 8·5 *per Cent.*—The quantities used were: Mixture 0·4 grm. and lycopodium 0·05 grm.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Stone cells}} = \frac{109}{33} = \frac{100}{30{\cdot}3} \text{ and } \frac{135}{41} = \frac{100}{30{\cdot}4}$$

$$\text{With an average of } \frac{100}{30{\cdot}35}.$$

If the quantities used had been the same as for the 50 per cent. mixture, the value 30·35 would become $30{\cdot}35 \div 4 = 7{\cdot}6$.

Hence percentage of dry coconut shell in the mixture $= \frac{7{\cdot}6 \times 43{\cdot}79}{41{\cdot}05} = 8{\cdot}1$, or 9·25 per cent. of air-dry coconut shell.

Mixture of Gentian Root 95 *per Cent. and Coconut Shell* 5 *per Cent.*—The quantities used were: Mixture 0·8 grm. and lycopodium 0·05 grm.

Counts of ten fields on each of two slides gave the ratios:

$$\frac{\text{Lycopodium spores}}{\text{Stone cells}} = \frac{118}{38} = \frac{100}{32{\cdot}2} \text{ and } \frac{136}{41} = \frac{100}{30{\cdot}15}.$$

$$\text{With an average of } \frac{100}{31{\cdot}2}.$$

If the quantities used had been the same as for the 50 per cent. mixture, the value 31·2 would become $31{\cdot}2 \div 8 = 3{\cdot}9$.

Hence percentage of dry coconut shell in the mixture $= \frac{3{\cdot}9 \times 43{\cdot}79}{41{\cdot}05} = 4{\cdot}2$, or 4·8 per cent. of air-dry coconut shell.

When examining mixtures such as this of coconut shell and gentian root, it would ordinarily be necessary to determine, by measurement and by comparison with standard powders, the degree of disintegration to which the powdered substance to be counted had been reduced, and to make the standard mixtures with a similar powder. Also, where the amount to be determined is very small, the dried crude fibre could be worked upon instead of using the original substance.

Conclusion.

For the purpose of reviewing as a whole the results obtained by the method described, I have arranged them in tabular form:

	Substance.	Admixture.			Suspending Agent.
		Name.	Amount Present per Cent.	Amount Found per Cent.	
1	Wheat flour	Cornflour	37·8	38·0	Powdered gum tragacanth
2	,, ,,	,,	10·0	10·1	Glycerol
3	Potato starch	,,	10·0	10·0	Powdered gum tragacanth
4	,, ,,	,,	5·0	5·1	Powdered gum tragacanth
5	Mustard	,,	25·0	26·6	Olive oil
6	,,	,,	25·0	27·1	Soft paraffin
7	Wheat flour	Potato starch	20·0	22·2	Olive oil
8	White pepper	Ginger	10·0	10·9	Castor oil
9	,, ,,	Rice starch	20·0	18·7	Olive oil
10	Gentian root	Coconut shell	29·3	26·3	Powdered gum tragacanth and glycerol
11	,, ,,	,, ,,	8·5	9·3	Powdered gum tragacanth and glycerol
12	,, ,,	,, ,,	5·0	4·8	Mucilage of tragacanth and glycerol

A study of the figures in the table shows that a high degree of accuracy is attainable. The errors may be taken to represent such as one may expect to find in everyday practice. It is difficult to draw any general conclusion as to the magnitude of error, because the conditions of working vary so largely in the different experiments, and a degree of precision attainable with one type of mixture is not always possible with another. The table shows, however, that one may generally anticipate that the error of working will not exceed 10 per cent. on the amount present, while in many cases it should be much smaller. Even when the larger errors occur, one could reduce them by increasing the number of counts made or by repeating the work. In each instance one can gain a very good estimate of the probable error in the working by preparing a mixture containing the materials in the proportions found, and then determining its composition microscopically; this additional work is a wise precaution to take in many cases, as, for instance, where the problem includes a determination of the degree of disintegration of a powdered substance.

The time required to make one set of ten counts varies from about fifteen minutes to as much as an hour and a half in exceptional cases; the average time s about half an hour.

This method of working constitutes a process of general applicability, and makes it possible to obtain precise quantitative results by means of the microscope. The use of lycopodium enables the observer to attack successfully many problems that it has been hitherto impossible to solve with any approach to certainty, and in the case of such mixtures as that of two starches, where the counting method has already been applied with only an approximate accuracy, the error of working is reduced to such an extent as to give the results a real value subject to only a small error.

Discussion.

The Chairman (Mr. J. H. B. Jenkins), in inviting discussion, remarked that the lycopodium, presumably, simply served as a unit for calculation, and one did not know to what extent the weighing of a definite quantity of it was essential. So long as it simply functioned as unity, it did not seem to him to make much difference what quantity of it was present.

Professor H. G. Greenish said that a good deal of work had been done in this direction, but he had never seen the problem attacked by the method which Mr. Wallis had used. All who had tried to determine the proportion of a substance like coconut shell or almond shell when mixed with gentian root, liquorice root, etc., must have felt that the results of their examination were very uncertain, amounting really to little more than an approximate guess. In cases like the first four in Mr. Wallis's table, one could probably attain a fair degree of precision without the use of lycopodium; but with mixtures of mustard and wheaten flour, pepper and rice, or gentian root and coconut shell, the problem was very much more difficult. In the case of, say, gentian root and coconut shell, it became a question of making the standard mixture with coconut shell reduced to about the same degree of fineness as the powder under examination, by measuring the particles of coconut shell and endeavouring, by some suitable method of grinding, to produce particles of the same size for use in making the standard mixture. To succeed, as Mr. Wallis had, in determining the proportion of coconut shell in such a mixture within 10 per cent. either way, was more than he should have thought possible.

Mr. W. Partridge said that in counting the bacteria in a vaccine, the bacterial emulsion was mixed with normal human blood in a known proportion, the slide was dried and stained, and the bacteria and blood corpuscles counted against one another. This method, by which the different elements were fixed *in situ* and stained, where it could be adopted, seemed preferable to counting the particles in a liquid medium. With a liquid medium the Thoma-Zeiss counting chamber, designed for counting the red and white cells in blood, might be used with advantage. The cells were counted in a standard volume of liquid, the height being that mentioned by Mr. Wallis, namely, one-tenth of a millimetre. The fields counted were, moreover, squares, which made the counting easier than with circles, as there was no difficulty with regard to particles occurring on the edges; those touching the top and right edges could be included and those on the bottom and left ignored, or *vice versa*.

Mr. C. Revis said that in counting milk cells diluted with red blood corpuscles he had experienced very great difficulty with circular fields, but this was obviated by the use of a square diaphragm. He had had no experience with larger particles, but when red blood corpuscles were used as a "diluent" of larger cells of the size of white

corpuscles, he had found it very difficult to get a properly proportioned mixture of the two. With larger particles it would probably be less difficult to get a satisfactory mixture.

Mr. A. CHASTON CHAPMAN remarked that sodium nucleate might be useful in some cases as a suspending medium. Solutions containing about 1 to 2 per cent. of sodium nucleate were quite limpid, whilst solutions containing about 5 per cent. (of the gelatinising variety) were almost solid, so that varying degrees of viscosity could be obtained. One objection was that the solution was very apt to froth, and the sodium nucleate would, of course, be decomposed by some of the reagents used in making the microscopical preparations.

Mr. C. C. ROBERTS suggested that it might be worth while to photograph the fields counted, in order to preserve a record of them for use as evidence.

Mr. E. T. BREWIS asked whether Mr. Wallis had considered what was generally meant by, say, a No. 80 powder. It meant a powder which had passed through a sieve having meshes of the size indicated, but such a powder might consist of particles of any degree of fineness from No. 80 to still finer, and the question was what effect any such variations in fineness might have upon the counting. In the case of the first four items of Mr. Wallis's table, all the particles would be of approximately the same size; but in the case of the white pepper or the gentian root the ultimate particles of pepper or of gentian root would be different in size and also possibly in shape, and would have a tendency to sift apart. Again, before the actual examination was begun, the question would arise as to whether the sample worked upon was a fair average portion of the few ounces constituting the sample submitted, which again would perhaps be taken from a bulk weighing some hundredweights or tons.

Mr. WALLIS, in reply, said that, although the mixtures referred to in the table were actual binary mixtures, it did not matter how many substances were present. The counting in each case had reference to one constituent only, so that all mixtures were binary mixtures from that point of view. He had found that weighing was necessary in order to get accurate results. At first he thought that weighing might be dispensed with, and had made a number of experiments in which everything was measured, with, in some cases—mixtures of ground olive stones with pepper, for instance—very good results; but that method was bad in principle. The only cases to which it could be satisfactorily applied would be cases in which the different constituents were of almost the same density. The question of the counting of particles at the edge of a field was a little difficult. He used a squared micrometer, and the particles at the edges were averaged as fairly as possible. The particles could not very well be fixed as in bacteriological work. He was not quite sure whether sodium nucleate would be a satisfactory suspending medium, because of the possible action of reagents upon it. Alcohol, for instance, might create some difficulty; but it would be well worth trying. The No. 80 powder was prepared by sifting through a No. 80 sieve, and of course contained particles of varying sizes. He had not made any attempt to investigate the difficulties that might be caused by such variations, but he thought that with patience it would be possible to obtain correct results. As to how far the 0·2 grm. worked upon was representative of the bulk, this would probably depend upon the care taken in mixing before weighing out. If the samples he had worked upon had not been representative, none of his experiments would have succeeded.

ZEISS BUTYRO-REFRACTOMETER: THE CONVERSION OF SCALE-READINGS TO REFRACTIVE INDICES.

By C. C. ROBERTS, M.A., A.I.C.

(*Read at the Meeting, November* 1, 1916.)

In the book of directions sent out with the Zeiss butyro-refractometer there is a table showing the refractive index for sodium light corresponding to every tenth degree of the scale of the instrument.

For 0° of the scale the refractive index is 1·4220, and for 100° it is 1·4895.

In a paper by Leach and Lythgoe (Analyst, 1905, **30**, 176) it is pointed out that the change in the refractive index corresponding to a rise of 10° in the scale-reading is less between 90° and 100° than between 0° and 10°.

If, however, the increase of the refractive index per 1° of scale-reading decreases at a uniform rate, it will be possible to express the relation between the refractive index and scale-reading by the formula

$$1000[n]_D = 1422 + ax - bx^2,$$

where $[n]_D$ is the refractive index for sodium light and x is the scale-reading; a and b are constants.

Using the values of $[n]_D$ for 0°, 50°, and 100° on the scale, I find that

$$1000[n]_D = 1422 + 0{\cdot}817x - 0{\cdot}00142x^2.$$

Using these values for a and b, I find that the results calculated by the formula agree perfectly with those in the table supplied with the instrument at 10, 30, 40, 60, 80, and 90, and that at 20 and 70 there is a difference of one unit in the fourth place of decimals; from this I think that the formula may be safely used for converting intermediate scale-readings into the corresponding refractive indices.

The following will perhaps be found more convenient for calculation:

$$[n]_D = 1{\cdot}4220 + 0{\cdot}00142x\left(\cdot 5753 - \frac{x}{1000}\right).$$

Discussion.

Mr. E. R. Bolton said that by the use of this formula a chart could be constructed from which the figures could be read off without calculation. It could be constructed to read to the fourth decimal place, which probably would not be given very accurately by the slide-rule, while the butyro-refractometer almost gave the fifth place. While referring to this subject he should like to put in a plea for uniformity of temperature in the recording of refractive indices. He had made it a practice not to record a refractive index at any other temperature than 40° C.; but one found figures obtained at 15°, 20°, 25°, 40°, or 45°.

Mr. Roberts said that his object in working out the formula in the first instance had been to provide a ready means of converting readings when a chart had been mislaid—as had happened in his own case.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Progressive Oxidation of Cold-Storage Butter. D. C. Dyer. (*J. Agric. Research,* 1916, **6,** 927-951.)—Since approximately 10 per cent. of the volume of butter is air, it was considered that an examination of the air in packages of butter and of butter-fat might furnish data as to whether the undesirable chemical changes (development of unpleasant flavour) occurring in stored butter are caused by a progressive oxidation of the fat itself or in some one or other of the non-fatty ingredients. The air contained in various cold-storage butters was, therefore, separated and analysed. The composition of the air confined within a package of pasteurised sweet-cream butter known to contain bacteria and made from cream having an acidity of 0·11 per cent. (calculated as lactic acid) showed little or no variation from its original composition after storing for six months at 0° F.; when stored under like conditions, the butter-milk from the same cream did not affect the composition of the air in the vessel containing the milk. When the butter was kept at 32° F., the composition of the enclosed air underwent a decided change, and this change was increased when the butter remained for a short time at ordinary temperature, the oxygen content decreasing and the carbon dioxide content increasing. The butter itself possessed no unpleasant flavour after six months' storage at 0° F. The composition of the air enclosed in a butter made from sweet cream and churned immediately after the addition of 15 per cent. of a commercial starter showed but little change after storage for six months at 0° F., and the butter kept well; the air from butter made from cream to which lactic acid had been added underwent considerable change during storage, the oxygen and carbon dioxide decreasing in quantity. The flavour of this butter became unpleasant after three months. A small quantity of butter-milk from butter prepared with the addition of lactic acid was exposed to the action of a very large and confined surface of air at 0° F.; after one month the whole of the oxygen had disappeared. The carbon dioxide content, originally 2·37 per cent., had increased to 34 per cent.; it then decreased. Further experiments with pure butter-fat and the same mixed with varying quantities of protein, lactose, etc., showed that after storage at 0° F. the quantity of carbon dioxide in the enclosed air was directly proportional to the amount of non-fatty ingredient present, and that the oxygen content showed a relative decrease. The pure butter-fat was not oxidised to any appreciable extent, but when the fat was spread on pumice and exposed to the action of a large volume of air at 32° F., a slight oxidation was noticed. The results of the investigation may be summed up as follows: The unpleasant flavours which develop in butter during cold storage are not produced by the oxidation of the fat itself, but by some chemical change which takes place in one or more of the non-fatty ingredients. The extent of this chemical change is proportional to the quantity of acid present in the cream from

which the butter was prepared. The quantity of carbon dioxide present in cold-storage butter probably depends on the amount of butter-milk in the butter, and may increase to a maximum, followed by a progressive decrease. W. P. S.

Estimation of Citric Acid in Milk. R. Kunz. (*Archiv. Chem. Microsk.*, 1915, **8**, 120-133; through *Int. Rev. Sci. and Prac. of Agriculture*, 1916, **7**, 739.)—Stahre's method for the estimation of citric acid in wine (ANALYST, 1915, **40**, 464) may be used for the estimation of this acid in milk. Fifty c.c. of the milk are treated with 20 c.c. of 50 per cent. sulphuric acid, 2 c.c. of 40 per cent. potassium bromide solution, and 20 c.c. of phosphotungstic acid solution; the mixture is diluted with water to 200 c.c., shaken and filtered. To 150 c.c. of the filtrate are added 25 c.c. of freshly prepared saturated hydrobromic acid solution; the mixture is heated at 50° C. for five minutes, and then treated with 10 c.c. of 50 per cent. potassium permanganate solution, the latter solution being added gradually while the mixture is stirred. The remaining part of the process is then carried out as described in the case of wine. Fresh milk contains about 0·19 grm. of citric acid per 100 c.c.; the quantity is slightly larger in the first milk drawn than in that obtained at the end of the milking. In ordinary milk the citric acid content diminishes progressively as the milk sours; curdled milk is free from citric acid, but "Yoghurt" contains about 0·16 grm. per 100 c.c., and this quantity does not decrease when the preparation is kept. W. P. S.

Estimation of the Fat Content of Dried Whole Milk. K. Mohs. (*Zeitsch. ges. Getreidew.*, 1916, **8**, 37-41; through *J. Soc. Chem. Ind.*, 1916, **35**, 1127.)—Samples of dried whole milk which have been kept for some time show an apparent reduction of fat content as determined by extraction with ether. This appears to be due to adsorption of the fat by the coagulated protein of the milk. The adsorbed fat is not removed by simple extraction with ether. The following procedure, based on a method described by Neumann (*Zeitsch. ges. Getreidew.*, 1912, **4**, 8) is recommended for the determination of the fat: 1·5 grms. of the finely divided milk powder are heated with 50 c.c. of water and 6 c.c. of hydrochloric acid of sp. gr. 1·125 for one and a half hours in a boiling water bath. After cooling, the solution is made neutral to methyl orange by addition of concentrated sodium hydroxide solution, then acidified with dilute hydrochloric acid, and filtered. The filter with its contents is dried for two hours at 105° C., and then extracted with ether for six hours in a Soxhlet apparatus.

Valuation of Nitrogenous Compounds in Feeding-Stuffs. N. Passerini. (*Annali Chim. Applic.*, 1916, **6**, 162-164.)—The method is based on the fact that the proteins of ordinary feeding-stuffs are rapidly hydrolysed by 25 per cent. sulphuric acid into soluble dialysable substances (peptones, etc.). After three hours' boiling the amount of ammoniacal nitrogen in the hydrolysed liquid, derived largely from the amines and amino-acids, remains constant. For example, 5 grms. of the sample hydrolysed with the acid yielded the following amounts of ammoniacal nitrogen: After one hour, 0·347 grm.; after three hours, 0·397 grm.; and after four hours,

0 397 grm. For the analysis 1 grm. of the finely divided material is taken for the estimation of the total nitrogen (*a*). The amines are then separated in 2 grms. of the sample by Kellner's method of precipitating the proteins with copper oxide, and the protein nitrogen (*b*) is estimated in the copper precipitate. Kellner's method is repeated on 5 grms. of the sample, and the copper precipitate is boiled for four hours on a sand-bath beneath a reflux condenser with 100 c.c. of 25 per cent. sulphuric acid. The liquid is treated with hydrogen sulphide to eliminate the copper, made up to 500 c.c. and filtered. The excess of hydrogen sulphide is expelled by boiling from an aliquot part (200 c.c.) of the filtrate, and the ammoniacal nitrogen (*c*) is distilled. In the residue left on the filter the non-hydrolysable nitrogen (*d*) is estimated, and is to be regarded as *nucleinic* nitrogen. The amounts thus obtained may be assigned as follows: (1) Nitrogen of free amines (asparagin, etc.) $=a-b$; (2) nitrogen of amino-acids $=b-(c+d)$. The following results obtained with a vetch flour illustrate the method. Total nitrogen, 3·66; nucleinic nitrogen, 0·14; nitrogen of amino-acids, 1·79; ammoniacal nitrogen in hydrolysed liquid, 0·42; and preformed amino-nitrogen, 1·30 per cent. C. A. M.

Presence of Copper in Tomatoes and Tomato Preserves. G. Liberi, A. Cusmano, T. Marsiglia, and C. Zay. (*Ann. Staz. chim.-agrar. sperim. Roma*, 1916, **8**, 163-303; through *Int. Rev. Sci. and Prac. of Agriculture*, 1916, **7**, 662-664.)—Numerous samples of tomatoes grown at the experimental farm belonging to the Station of Agricultural Chemistry, Rome, and other samples obtained from various districts in Italy, were all found to contain copper in quantities varying from 0·14 mgrm. to 2·1 mgrms. per kilo of juice and pulp, or from 3·88 mgrms. to 19·45 mgrms. per kilo of dry substance. All the soils on which the tomatoes had been grown contained copper, the maximum quantity found being 110·74 mgrms. per kilo of dry soil. The amount of copper in the fruits was not affected when the plants had been sprayed with Bordeaux mixture. Preserved tomatoes contained corresponding quantities of copper. W. P. S.

Analysis of Rhamnus Barks. O. Tunmann. (*Apoth.-Zeit.*, 1915, **30**, 642; through *J. Chem. Soc.*, 1916, **110**, ii., 504.)—The red-coloured foam which is obtained when drugs containing anthraquinones are shaken with sodium hydroxide solution is probably due to the presence of chrysophanol; the latter is present, therefore, in *Rhamnus carniolicus* as well as in *R. catharticus*, and the reaction simply serves to distinguish these from the American rhamnus bark, *R. purshiana*. Tschirch's colorimetric method indicates that the quantities of anthraquinone derivatives in *R. frangulus* and in *R. purshiana* have a ratio of 4 : 1, whilst according to the author's gravimetric method the ratio between the two respective quantities is 3 or 2·5 : 1.

Use of the Iodic Acid-Starch Reaction in the Examination of Wine and Vinegar. J. Jaenprêtre. (*Chem. Zeit.*, 1916, **40**, 833.)—A mixture of sulphurous acid, iodic acid, and starch develops a blue colour after some length of time—a reaction which was first pointed out by Landolt (*Ber.*, 1886, **19**, 1317). Investigation

of this reaction showed that the development of the blue coloration depends on the kind and concentration of the acid, the temperature, etc. The reagent used consists of a mixture of 10 c.c. of 0·2 per cent. sodium iodate solution, 10 c.c. of 0·2 per cent. sodium sulphite solution, 5 c.c. of 0·5 per cent. starch solution, and 75 c.c. of water. When 10 c.c. of this reagent are mixed with 10 c.c. of $\frac{N}{10}$ acid solutions, the time which elapses before the coloration appears is as follows: Acetic acid, 438 secs.; succinic acid, 320 secs.; malic acid, 52 secs.; citric acid, 45 secs.; tartaric acid, 26 secs.; oxalic acid and mineral acids, immediately. As the time required for the development of the coloration is considerably diminished by the presence of a trace of free mineral acid, the reaction may be used for the detection of mineral acids in wine and vinegar. If, when mixed with the reagent, 10 c.c. of the sample give a blue coloration in less time than do 10 c.c. of citric acid of equal strength, an abnormal amount of free mineral acid is present. As a rule, a non-sulphured, non-plastered wine reacts as slowly as does a succinic acid solution. W. P. S.

Detection of Artificial Colours in Wines. H. Kreis. (*Chem. Zeit.*, 1916, **40**, 832.)—Certain dark red wines, even when diluted, contain colouring matter which fixes on wool and cannot be removed by washing the wool with boiling water; consequently, this test may indicate the presence of aniline dye although this may be absent. If the wine dyes wool, the latter should be heated on a water-bath with a small quantity of 1 per cent. ammonia solution which destroys the natural colouring matter derived from the wine, whilst aniline dyes, for the most part, go into solution. The ammoniacal solution is then acidified with sulphuric acid and heated with a fresh thread of wool. The latter remains white if aniline dyes are absent. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Detection and Estimation of Hydrocyanic Acid in Beans. L. Guignard. (*Ann. Falsific.*, 1916, **9**, 301-305.)—Burmah beans may be imported into France provided that the consignment, on analysis, does not show a higher hydrocyanic acid content than 0·02 per cent. The beans, which belong to the *Phaseolus lunatus* species, are of two kinds, red and white, and the white beans are frequently used for food. The author has found 0·025 per cent. of hydrocyanic acid in certain samples of the dried beans. The method employed for detecting the presence of hydrocyanic acid consisted in mixing the powdered bean with five times its weight of water in a flask and suspending in the upper part of the flask a strip of paper which had been dipped in solution containing picric acid and an excess of sodium carbonate. An orange-red colour developed on the paper within twelve hours if the bean contained a hydrocyanic glucoside. The quantity of hydrocyanic acid was estimated by macerating 20 grms. of the powdered bean with water for twelve hours, then submitting the mixture to steam distillation, and collecting the distillate (125 c.c.) in a receiver containing dilute ammonia. The distillate was then titrated with $\frac{N}{10}$ silver nitrate solution, using potassium iodide solution as the indicator. W. P. S.

Salicylic Acid Reaction of Soya Beans. H. C. Brill. (*Philipp. J. Sci.*, 1916, **11**, 81-89.)—All the samples of Japanese soya beans tested gave the ferric chloride colour test for salicylic acid. American, Chinese, and native beans gave either negative or faintly positive tests with the same reagent. All samples of soya beans gave a negative result for salicylic acid with the Millon reagent and with the Jorissen reagent (see ANALYST, 1910, **35**, 252 and 253). The reacting compound in the beans has all the ordinary test properties of salicylic acid, but is undoubtedly similar to the maltol of Brand (*Ber.*, 1894, **27**, 806). Jorissen's reagent should therefore be employed in testing beans for salicylic acid. H. F. E. H.

Resistance of Non-Sporing Bacteria in Milk to the Action of Heat. C. Gorini. (*Rend. R. Institut. Lombardo Sci. Lettere*, 1915, **48**, 956-961; through *Int. Rev. Sci. and Prac. of Agriculture*, 1916, **7**, 740.)—Experiments showed that the presence of non-sporing bacteria in milk which had been pasteurised was due to the formation of a protective covering of casein round the bacteria, this covering probably being caused by the action of the bacteria themselves, before or during the sterilising process. Thus the explanation of the apparent resistance of non-sporing bacteria to the action of heat is rendered more comprehensible. In none of the experiments was any case found of resistance to heat above 85° C., and no bacterium, even when artificially covered with casein, ever survived a temperature of 90° C., whilst under normal conditions the bacteria resisted sterilisation at 100° C. As the surviving bacteria, however, were localised in small clots of casein and had acid-coagulating properties, the difference is attributed by the author to the unavoidable difference between natural and artificial conditions, which do not affect the theory that the thermo-resistance is due to the protective layer of casein. W. P. S.

Yeast Preparation for Use in the Estimation of Crystallisable Sugar by Inversion. H. Pellet. (*Procès-Verbaux de l'Ass. d. Chim. de Sucr. et d. Dist.*, 1915, **33**, Bull. 1-3, 12-13; through *Int. Rev. Sci. and Prac. of Agriculture*, 1916, **4**, 592.)—A preparation of yeast which is very active and retains its inverting capacity for a prolonged period may be made by the addition of sodium salicylate at the rate of 0·2 grm. of salicylate per 3 grms. of yeast, which is thereby liquefied almost instantaneously. Yeast may be thus treated in quantity and when required for use diluted in the proportion of 30 grms. in 100 c.c.; 10 c.c. equivalent to 3 grms. of yeast being used per 50 c.c. of sugar solution (neutral and free from lead). Inversion is complete in half an hour at 55° C., the ordinary Clerget formula being employed to calculate the cane-sugar, using the constant $141{\cdot}8 - \frac{1}{2}t$ in place of the 144 (Clerget) or 142·7 (German formula and method). H. F. E. H.

ORGANIC ANALYSIS.

Detection of Arachidic Acid. R. H. Kerr. (*J. Ind. and Eng. Chem.*, 1916, **8**, 904.)—The following method is simpler than Renard's official method, and obviates the use of ether: Twenty grms. of the oil are heated with 200 c.c. of 95 per cent.

alcohol in a 300 c.c. Erlenmeyer flask, and 10 c.c. of 10 per cent. potassium hydroxide are added to the boiling liquid. After saponification of the oil the excess of alkali is neutralised with a solution of 50 c.c. of glacial acetic acid in 150 c.c. of 95 per cent. alcohol, and 5 c.c. of a 5 per cent. solution of magnesium acetate in a mixture of equal volumes of water and 95 per cent. alcohol are added. The flask is cooled, with occasional shaking, and then left in a refrigerator at 10° to 12° C. until the next day. The precipitate is filtered off, washed twice with 50 per cent. alcohol and three times with water, and returned to the flask. It is then treated with 100 c.c. of hot water and sufficient dilute sulphuric acid (50 : 150 c.c.) to decompose the magnesium salts, and heated until the fatty acids form a clear layer. After solidification, these are washed with hot water, again left to solidify, drained from water, and dissolved in 100 c.c. of 90 per cent. (by volume) alcohol. The arachidic acid which crystallises is separated by the method of the Ass. Off. Agric. Chemists (Bull. 170, Revised, Bureau of Chemistry, p. 146). The method gives results as good as those obtained by Renard's method, and is capable of detecting 5 per cent of arachis oil in olive oil, cottonseed oil, soya-bean oil and maize oil. C. A. M.

Estimation of Carbohydrates—V.: The Supposed Precipitation of Reducing Sugars by Basic Lead Acetate. W. A. Davis. (*J. Agric. Sci.*, 1916, **8**, 7.)—Since Gill (*J. Chem. Soc.*, 1871, **24**, 91) observed that the addition of an excess of basic lead acetate to a solution of invert sugar reduced the negative rotation of the lævulose owing to the formation of a soluble compound, many workers have assumed that this sugar is precipitated by the defecating agent to a greater or less degree. The author shows that, at least in dilute solutions, lævulose is never precipitated by basic lead acetate even in presence of salts such as chlorides, sulphides, or carbonates. No loss of lævulose occurs unless the excess of lead is allowed to act for some length of time upon the sugar before the lead is precipitated. Thus, if basic lead acetate is left with a solution of pure lævulose for periods of time such as fifteen minutes, one hour, or twenty-four hours, and the lead is then precipitated by sodium carbonate or sulphate, amounts of lævulose are found to have disappeared, depending solely on the time of action, the solution becoming more and more yellow, but no visible separation of lævulose occurs. It is probable under these conditions that a substance resembling or identical with the so-called glutose of Lobry de Bruyn and van Ekenstein (*Rec. Trav. Chim.*, 1897, **16**, 262) is formed, having little or no optical activity and a reducing power about one-half that of dextrose. It is shown that, provided basic lead acetate solution is added in small quantities at a time until precipitation is just complete, and that the excess is not allowed to reach more than about 5 c.c. in 300 to 500 of sugar solution, there is no loss whatever of reducing sugar. The excess of lead should be removed with sodium carbonate or sulphate as soon as possible after filtering off the precipitate. Dextrose and maltose remain practically unchanged in presence of a considerable excess of basic lead acetate, although eventually even with these sugars soluble lead compounds having a different optical activity may be formed (*cf.* Watts and Tempany, ANALYST, 1908, **33**, 130; Eynon, *ibid.*, 1909, **34**, 349). H. F. E. H.

Analysis of Crude Calcium Cyanamide. E. Truninger. (*Schweiz. Ver. anal. Chem.*, May 26 and 27, 1916.)—A slight alteration is to be made in the method previously described: the cyanamide should be precipitated with neutral silver nitrate and the ammonia added afterwards. For the present, the dicyandiamide may be determined indirectly by the simultaneous precipitation of cyanamide and dicyandiamide with silver nitrate and 2 per cent. potassium hydroxide solution, and subsequent determination of the nitrogen in the precipitate by Kjeldahl's method. The determination of urea has not been deeply studied because it is not yet certain whether that substance is present. Caro's method (ANALYST, 1916, **36**, 76) could not give accurate results, because the prolonged heating with alkali to drive off the ammonia would cause losses not only of dicyandiamide, but also of urea if present. Determinations of the insoluble nitrogen in the residue from the extraction of the crude material with water, and dilute nitric acid showed an average of 1 per cent.; in a few cases 2 per cent. was found. Agricultural experiments with calcium cyanamide on oats confirmed its favourable effects; with the exception of a single sample rich in dicyandiamide, an increased yield was recorded. The injurious influence of considerable quantities of this substance was shown at an early stage, but could be largely inhibited by the simultaneous application of a readily assimilable nitrogen compound. In considering the unfavourable influence of the dicyandiamide, its great stability in the soil must be taken into account; nitrification had not taken place after a period of two months. Secondary ill-effects were observed in the case of oats even in the second year; on the other hand, calcium cyanamide which had lain for a long time on the moist ground had lost much of its valuable properties. Vegetation experiments with urea and urea nitrate showed excellent results with oats. With winter wheat a top dressing of calcium cyanamide and urea showed the great value of urea used in this form.

Casein and its Technical Applications. D. Marotta. (*Annali Chim. Applic.*, 1916, **6**, 165-176.)—Pure casein has a sp. gr. of 1·259. When dried in the air at 70° to 80° C. for five hours it loses 5 to 8 per cent. in weight, while when dried *in vacuo* it retains 2 per cent. of moisture. It is readily soluble in 1 per cent. solutions of sodium fluoride, ammonium oxalate, and potassium oxalate, and in 5 per cent. solutions of ammonium chloride or sulphate. It ought to be free from soluble salts, lactose, or peptones, and should yield less than 1 per cent. of ash on ignition. Technical casein is yellowish, and yields up to 6 per cent. of ash. It contains 12 to 13 per cent. of water, 12 to 13 per cent. of nitrogen, and about 0·5 per cent. of fat. When prepared by the action of acids, it is soluble in solutions of alkalis and sodium salts of the following strengths: Sodium hydroxide, 2·0; carbonate, 2·5; bicarbonate, 3; silicate, 10; arsenate, 20; sulphite, 9; tungstate, 12·5; borate, 9·5; and ammonia 2·5 per cent. The casein obtained by the action of rennet is insoluble in solutions of sodium carbonate and bicarbonate, partially soluble in borax and ammonia solutions, and soluble in sodium phosphate solution. Of the pharmaceutical preparations of casein, *periodo-casein* contains 17·89; *iodo-casein*, 15·7; and *caseo-iodine*, 8·7 per cent. of iodine. A casein still richer in iodine (21·6 per cent.) is prepared by adding iodine to milk and precipitating the compound with acetic

acid. The *lacto-iodine-periodine* of commerce contains 5·7 per cent. of iodine. Preparations of *bromo-casein* contain from 4·5 to 11 per cent. of bromine. There is also a chlorine preparation with 2·8 per cent. of chlorine, and a *fluoro-casein* containing 1·6 to 1·8 per cent. of fluorine. *Silver caseinate* contains 8·76 to 9·66 per cent. of silver, and there are also preparations containing 4, 10, and 15 per cent. The compound "*argonine*," which is prepared by treating sodium caseinate with silver nitrate and precipitating the compound with alcohol, contains 4·2 per cent. of silver. Another commercial product, "*argonine L*," contains 10 per cent. of silver. *Iron caseinate* contains 3·6 per cent. of iron. Compounds of casein with alkaloids are prepared by suspending the casein in alcohol and adding an alcoholic solution of the alkaloid. C. A. M.

Comparison of Barbituric Acid, Thiobarbituric Acid, and Malonylguanidine as Quantitative Precipitants for Furfural. A. W. Dox and G. P. Plaisance. (*J. Amer. Chem. Soc.*, 1916, **38**, 2156-2166.)—Unger and Jüger (*Ber.*, 1902, **35**, 4440, and 1903, **36**, 1222) applied the reaction between barbituric acid and furfural to the quantitative estimation of the latter, but a large excess of the acid appears to be necessary for complete precipitation although the reagent has the advantage of not precipitating hydroxymethyl furfural. The authors found that for various reasons thiobarbituric acid—$CH_2(CO)_2(NH)_2CS$—is much the better reagent to use, and this was prepared according to the method of Fisher and Dilthrey (*Ann.*, 1904, **335**, 350), in which twice the theoretical amount of sodium dissolved in a little alcohol is mixed with 16 grms. of malonic ester and 7·6 grms. dry thiourea previously dissolved in absolute alcohol. The mixture is heated for fifteen hours in a closed tube at 105° C., and the product, after acidifying with hydrochloric acid, separates as a slightly yellowish crystalline powder containing 19·6 per cent. of nitrogen. The yield is 45 per cent. of theory. The precipitation of the furfural is carried out in 12 per cent. hydrochloric acid solution at room temperature in a total volume of 400 c.c., using slight excess of the thiobarbituric acid exactly as in the case of phloroglucinol, and after being allowed to stand overnight the precipitate is filtered off and dried till constant at 100° C. In the case of malonylguanidine, the condensation of furfural is not quantitative, the best results being 50 per cent. in error, while with barbituric acid only 95 per cent. of the furfural taken is recovered when using about 60 mgrms. of furfural, while with small amounts of furfural (12 to 35 mgrms.), only from 25 to 80 per cent. is recovered. With thiobarbituric acid, however, the theoretical weight of precipitate is obtained working on quantities of furfural varying from 11 to 60 mgrms. Variations in the amount of precipitant were of little influence. The substance formed—furfuralmalonylthiourea ($C_9H_6O_3N_2S$) is a bright yellow precipitate, very flocculent and voluminous, practically insoluble in cold dilute acids, alcohol, ether, petroleum ether, methyl alcohol, acetic acid, benzene, carbon disulphide, and turpentine. In ammonia, pyridine, and caustic alkalis it dissolves readily, giving a greenish-blue solution which gradually loses its colour. It is essential that the thiobarbituric acid employed should be strictly pure and free from dicyandiacetylthiourea, or error will be introduced, and it is recommended that the malonic ester used for its prepara-

tions should be subjected to a repetition of the simultaneous saponification and esterification before condensation with thiourea, and that the thiobarbituric acid itself be purified by one or two crystallisations of its sodium salt. The authors do not find that any reliance can be placed upon the separation recommended by Ishida and Tollens (*J. Landw.*, 1911, 60) for the separation of the phloroglucides of furfural and methyl furfural based on the solubility of the latter in alcohol. Since methyl furfural is also precipitated by thiobarbituric acid, evidence can only be obtained of its presence by analysis of the condensation product, which in the care of the methyl salt contains nitrogen 11·86 per cent. and sulphur 13·56 per cent., as against the 12·6 and 14·41 in the furfural compound. The author considers that such an analysis would show the presence of methyl furfural if present to the extent of 1 in 3 of furfural; but where the ratio is less, the lowering of the nitrogen would be within the limits of analytical error. H. F. E. H.

Benzoyldihydromethylketol Hydrazine. New Reagent for Galactose. J. von Braun. (*Ber.*, 1916, **49**, 1266-1268; through *J. Soc. Chem. Ind.*, 1916.)—Benzoyldihydromethylketol hydrazine,

$$CH_3.CH<\begin{matrix}CH_2\\N(COC_6H_5)\end{matrix}>C_6H_3.NH.NH_2,$$

is a specific reagent for galactose, with solutions of which it gives a colourless crystalline precipitate in from half to two hours according to the concentration. With dextrose, lævulose, mannose, arabinose, and xylose no precipitate is produced. The base is prepared from benzoyldihydromethylketol, which is nitrated and reduced to *m*-amino-N-benzoyldihydromethylketol. This is diazotised and reduced with stannous chloride, and the hydrazine isolated in the usual way. It crystallises from alcohol in colourless needles (m.-pt. 150-151° C.), and is apparently quite stable when dry. The hydrochloride melts at 197° C., and the semicarbazide derivative at 213° C.

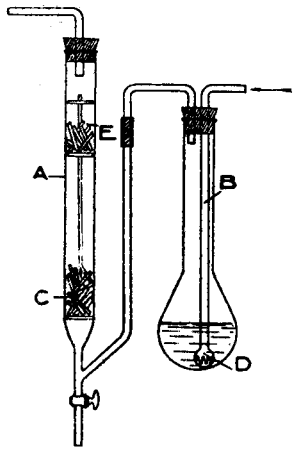
Highly Unsaturated Hydrocarbon in Shark Liver Oil. M. Tsujimoto. (*J. Ind. and Eng. Chem.*, 1916, **8**, 889-896.)—*Ai-zamé oil*, extracted from the liver of the Japanese squaloid shark, *Squalus mitsukurii*, gave the following analytical values: Sp. gr. at 15°/4° C., 0·8644; solidification point, below −20° C.; acid value, 0; saponification value, 22·98; iodine value (Wijs), 344·63; $[n]_{D\ 20°\ C.}$, 4930. *Fatty acids* (10·62 per cent.): Neutralisation value, 168·52; iodine value, 119·25. Glycerol, 0·52 per cent.; and unsaponifiable matter, 90·17 per cent. *Heratsuno-zamé oil* from the liver of another squaloid shark, *Deania eglantina*, gave the following values: Sp. gr. at 15°/4° C., 0·8721; acid value, 0·49; saponification value, 52·46; iodine value (Wijs), 261·72; $[n]_{D\ 20°\ C.}$, 1·4850. *Fatty acids* (26·59 per cent.): Neutralisation value, 168·39; iodine value, 73·35. Glycerol, 0·39 per cent.; and unsaponifiable matter, 72·88 per cent. In each case the unsaponifiable matter, after deducting the cholesterol (0·55 and 1·24 per cent. respectively), consisted mainly of a hydrocarbon or hydrocarbons, which could be separated by shaking the saponified oils with petroleum spirit. It was a colourless oily liquid and had the composition $C_{30}H_{50}$, and showed

the following characters: Boiling-point, 262° to 264° C. (10 mm. pressure); solidification point, −75° C.; sp. gr. at 15°/4° C., 0·8587; iodine value, 388·12 (Wijs); $[n]_{D\ 20^{\circ}C.}$, 1·4965. It dried rapidly, yielding a colourless film superior to that formed by vegetable drying oils. It formed an addition compound with bromine, $C_{30}H_{50}Br_{12}$. The hydrogenated product, $C_{30}H_{62}$, resembled the so-called liquid paraffins, but was more stable in the cold. It could be used for lubricating machinery.

C. A. M.

INORGANIC ANALYSIS.

Aeration Method for Ammonia. B. S. Davisson, E. R. Allen, and B. M. Stuttlefield. (*J. Ind. and Eng. Chem.*, 1916, **8**, 896-899.)—The authors have investigated the conditions under which ammonia can be quantitatively distilled in a current of cold air, with minimum risk of decomposing nitrogenous organic matter and with complete absorption of the ammonia in standard acid. Magnesia is preferable to stronger alkalis for liberating the ammonia, as it has less effect on organic matter and is less liable to be carried forward mechanically into the standard acid by strong air currents. Magnesia, of which 0·5 grm. is usually sufficient, works admirably, but rapid air currents are requisite when using it, if the determination is to be complete in two and a half hours. For these rapid air currents (about 1,000 litres an hour), Folin tubes are useless, and the apparatus illustrated is used by the authors. The main part of the absorption tube is 1½ inches in diameter and 15 inches long. The side arm has a bore of 8 mm. *C* and *E* are glass rods, about 1½ inches long, held in place by baffles made from rubber gasket. The purpose of the baffle at *E* is to stop any acid which spatters from the lower part of the tower. The aeration bulb *D* is well perforated, so that complete stirring of the solution will be obtained, and should extend well to the bottom of the flask. The tubes connecting the flask to the absorption apparatus on one side as shown, and to the next unit in the series, which may conveniently be as many as ten sets, are of 5 mm. bore. The entering air should be scrubbed successively with sodium hydroxide and 25 per cent. sulphuric acid. If the scrubbing with sodium hydroxide be omitted, the part set in the series may yield low results, owing to the small quantity of magnesia used becoming largely carbonated, and it is desirable not to use large (10 grm.) quantities, as is sometimes done.



G. C. J.

NOTE BY EDITOR.—The use of magnesia is undesirable in ammonia distillations in presence of phosphates, as some ammonia may be retained as ammonium magnesium phosphate.

Volumetric Estimation of Cobalt. W. D. Engle and R. G. Gustavson. (*J. Ind. and Eng. Chem.*, 1916, **8**, 901-902.)—The method depends on the fact that sodium perborate, in presence of alkali hydroxide, oxidises cobalt to cobaltic hydroxide, but

does not oxidise nickel. The excess of the reagent is readily decomposed by boiling and the cobalt can then be estimated iodometrically. The ore or other material is dissolved by means of acids, and the metals of the copper and iron groups, and also manganese, are removed by standard methods. The solution so obtained may contain cobalt, nickel, and zinc, but must be free from substances capable of liberating iodine from an acid solution of potassium iodide. The solution (100 c.c.) is acidified with dilute sulphuric acid, of which an excess of about 5 c.c. is added. Sodium perborate (1 to 2 grms.) is added, and, after it has dissolved, sodium hydroxide is added to strong alkaline reaction and the mixture boiled for ten minutes to decompose the excess of perborate. When cool, 1 grm. potassium iodide is added, the solution acidified with dilute sulphuric acid, and, after solution of the precipitate, the liberated iodine is titrated against standard thiosulphate. The latter may be standardised against pure, anhydrous cobalt sulphate, treated as described, or more conveniently against potassium bichromate, $K_2Cr_2O_7 = 6$ Co. The extreme error of the method, even in presence of ten times as much nickel as cobalt, appears to be no more than corresponds to 0·1 c.c. of the standard thiosulphate used.

G. C. J.

Use of Solutions of Borax and Boric Acid in the Colorimetric Estimation of the Concentration of Hydrogen Ions in Sea-Water. S. Palitzsch. (*Compt. Rend. Trav. Lab. Carlsberg*, 1916, **11**, 199-211.)—In Soerensen and Palitzsch's method for the estimation of the concentration of hydrogen ions in sea-water by colorimetric comparison with standard solutions with the addition of suitable indicators (phenolphthalëin, naphtholphthalëin, methyl red), the standard solutions of sodium borate and hydrochloric acid indicated (see ANALYST, 1910, **35**, 216; 1913, **38**, 394) give satisfactory results. In the case of a Polar expedition, however, practical difficulties arose which made it desirable to prepare the standards beforehand in the form of accurately weighed quantities of solid ingredients which could be dissolved in distilled water when required for use. The author has therefore worked out a combination of borax and boric acid, the utility of which is not confined to the special purpose for which it was designed, and which shows many advantages over the older standards for general use.

Borax specially purified for analysis and suitable for the purpose is readily obtainable; its purity should be checked by titration of the base with $\frac{N}{10}$ hydrochloric acid in presence of methyl red, which gives a much sharper end-point than methyl orange. The moisture is determined by heating carefully but fairly strongly to redness in a platinum crucible. The boric acid is titrated in presence of glycerol and a moderately large quantity of phenolphthalëin, using for comparison a blank solution of glycerol; results sufficiently accurate are obtained with 75 c.c. of glycerol for 0·5 grm. of borax, although 150 c.c. are required for very high degrees of accuracy. If carbonates are present the carbonic acid must first be expelled by boiling with sulphuric acid under a reflux condenser for five minutes. The concentrations of the solutions most suitable for estimations in sea-water are: $\frac{M}{20}$ of borax, equivalent to $\frac{N}{10}$ sodium hydroxide—*i.e.*, 19·108 grms. per litre—and $\frac{M}{5}$ of boric acid—*i.e.*, 12·404 grms. per litre—to which are added 2·925 grms. of sodium

chloride to compensate the "salt error," the error due to the modification of the colour of the indicator by the salts apart from their influence on the ionic concentration. The author has standardised mixtures of borax and boric acid solutions by the electrical method, obtaining values set forth in the table below:

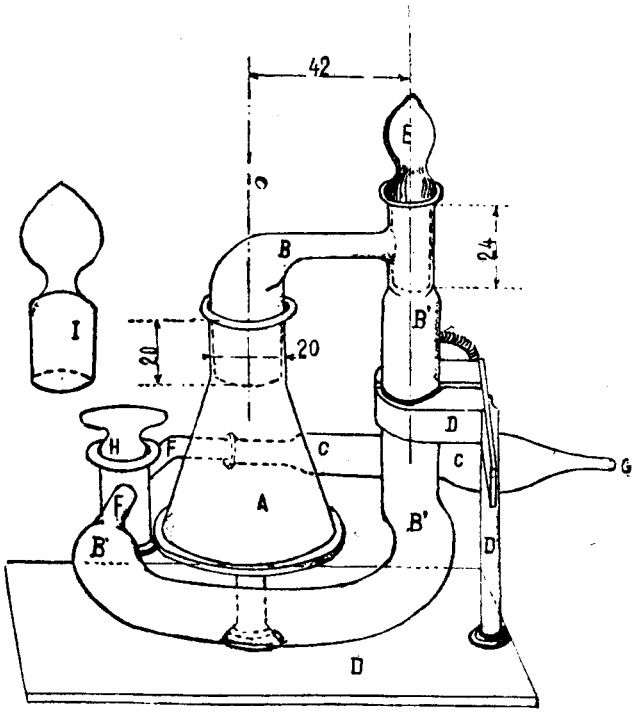
CONCENTRATIONS OF HYDROGEN IONS IN MIXTURES OF BORAX AND BORIC ACID.

Borax $\frac{M}{10}$.	Boric Acid $\frac{M}{5}$.	P_H^+.	$C_H^+ \times 10^9$.
c.c.	c.c.		
10·0	0·0	9·24	0·58
9·0	1·0	9·11	0·78
8·0	2·0	8·98	1·05
7·0	3·0	8·84	1·45
6·0	4·0	8·69	2·04
5·5	4·5	8·60	2·51
5·0	5·0	8·51	3·09
4·5	5·5	8·41	3·89
4·0	6·0	8·31	4·90
3·5	6·5	8·20	6·31
3·0	7·0	8·08	8·32
2·5	7·5	7·94	11·5
2·3	7·7	7·88	13·2
2·0	8·0	7·78	16·6
1·5	8·5	7·60	25·1
1·0	9·0	7·36	43·7
0·6	9·4	7·09	64·6
0·3	9·7	6·77	170

The errors due to the salts in sea-water have not been re-determined in the case of the borate solutions now proposed, but the corrections to be applied are the same as were prescribed for the older standards (ANALYST, *loc. cit.*). J. F. B.

Loss of Phosphoric Acid during Fusion with Ammonium Fluoride. W. A. Davis and J. A. Prescott. (*J. Agric. Sci.*, 1916, **8**, 136-138.)—In the analysis of salts or minerals containing phosphoric acid, treatment with hydrofluoric acid or ammonium fluoride was found to result in a considerable loss of phosphoric acid, which appears to be volatilised as phosphorus fluoride. The loss is least with phosphates of the alkali metals, while the loss from phosphates of the alkaline earths is considerably greater and may rise to over 50 per cent. in the case of minerals such as apatite. The loss from disodium hydrogen phosphate is less than that from potassium dihydrogen phosphate. Experiments showed that the loss which occurs during ignition with sulphuric acid only takes place when there has been previous treatment with ammonium fluoride. H. F. E. H.

Estimation of Moisture in Resinous Woods. E. Azzarello. (*Annali Chim. Applic.*, 1916, **6**, 154-157.)—From 1 to 3 grms. of fine shavings of the wood are weighed in the tared flask *A* provided with a stopper I. The stopper is then replaced by the tube *B-B*, which has previously been charged with fragments of calcium oxide placed in alternate layers with tufts of glass wool and then weighed. To the end of the small tube *F* is attached the tube *C*, filled with calcium chloride, to prevent the lime in *B* absorbing moisture from the air. The stopper *E* is turned so as to put *B* in communication with *A*, the tap *H* is opened, and the apparatus is placed in an oven maintained at 125° to 130° C. After thirty minutes it is allowed to cool with the tap *H* closed, and then replaced in the oven with the tap open, this process being repeated until water is no longer condensed on the walls of the connection between *A* and *B*. The flask *A* may then be detached and weighed from time to time until the weight diminishes less than 2 to 3 mgrms. The loss gives the amount of volatile oil and water. The tube *B-B* is then heated with the tap open in an oven at 200° C. until the weight becomes constant within 2 to 3 mgrms. This gives the moisture absorbed by the lime. Experiments with mixtures of water, volatile oil, and dry mineral matter show that the results are invariably too high, but are accurate within 0·5 per cent. C. A. M.



Detection of Small Quantities of Selenium and its Distinction from Arsenic. J. Meunier. (*Comptes rend.*, 1916, **165**, 332-334.)—Selenious acid and selenites, when reduced with zinc and sulphuric acid, yield hydrogen selenide, a gas which is readily decomposed by heat with the deposition of selenium. The deposit of selenium obtained in a Marsh tube, however, has a red colour, particularly at its densest part, and is quite unlike the deposit yielded by arsenic under similar conditions. When oxidised, the deposit is converted into white selenious acid. Larger quantities of selenious acid may be distinguished from arsenic by treating their hot solution with hydrogen sulphide; a turbidity due to precipitated sulphur is obtained, and, by heating the mixture for some time on a water-bath, this sulphur settles and

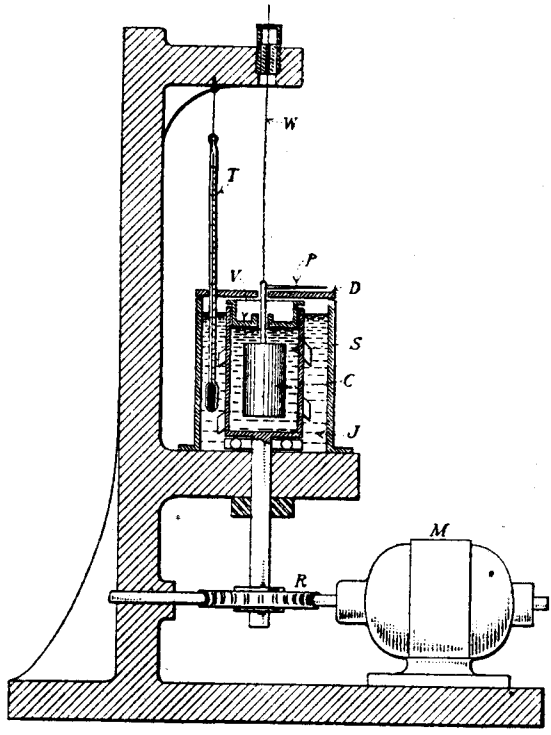
entrains the selenium and arsenic sulphides which are formed. If selenium sulphide is present, the precipitated sulphur has a brown appearance, whilst the yellow colour of the sulphur is not altered by the presence of arsenic sulphide. If the precipitate is collected, dried, and cautiously heated in a tube, the free sulphur volatilises and a black residue of selenium sulphide remains. W. P. S.

APPARATUS, ETC.

Washing Precipitates by Mechanical Means. E. Sinkinson. (*Chem. News*, 1916, **114**, 170-172.) — The paper describes and illustrates an elaborate device which is intended to supersede the ordinary wash-bottle in washing precipitates, once they have been transferred to the filter. The funnel containing the filter is supported in a ring at the end of one arm of a balance, the other arm carrying a movable counterpoise and also an electrical commutator. Over the beam is fixed a table carrying two electric motors, one to control through a specially constructed mercury valve the flow of water to a jet, which is rotated by the other motor. The two motors are connected to the current supply through the commutator in such a way that, when the funnel end of the beam is up, the water supplied by gravity from a flask or tin-lined copper tank, passes freely to the jet, which sprays the water round the edge of the paper containing the precipitate. The counterpoise at the other end of the beam is moved into such a position that, when sufficient water has flowed into the funnel, the increased weight causes the beam to drop, thus shutting off the water supply to the jet, so that no current is wasted during the period it is not required. As the water drops from the funnel, the arm supporting it becomes lighter; the beam rises and water again enters the filter from the rotating jet. G. C. J.

Respiration Calorimeter, partly Automatic, for the Study of Metabolic Activity of Small Magnitude. Langworthy and Milner. (*J. Agric. Res.*, 1916, **6**, 703-720.)—The apparatus is similar in principle to, but modified in various ways from and much smaller than, that described by the same authors for the purpose of human experiments (ANALYST, 1916, 49), and can be used for investigations on the ripening of fruits and the wintering of bees. Chambers of varying sizes can be employed; that generally used has a capacity of 185 litres, being 45 cm. square and 91 cm. deep. The respiration chamber is part of a closed air circuit through which a stream of air is constantly moving; the air which leaves the chamber is passed through purifying devices and returned again to the chamber. The amount of heat resulting from the activity of the material employed is ascertained from determinations of (1) the quantity of latent heat in the water-vapour of the outgoing water, (2) the quantity of sensible heat absorbed and carried away by water flowing in a coil of pipe in the chamber, and (3) the quantity of heat involved in changes in the temperature of the active material and of other objects in the chamber and also of the walls of the chamber. No gain or loss of heat occurs through the walls of the chamber. Photographs and very full details of the apparatus are given in the original paper. H. F. E. H.

New Form of Viscosimeter. H. C. Hayes and G. W. Lewis. (*Chem. Eng.*, 1916, **24**, 103-104.)—The apparatus is based on the principle that a solid body having a surface of revolution, when suspended in a rotating liquid, is subjected to a torque which is proportional to the viscosity of the liquid. In the illustration of the instrument (see figure), the sample *S* is contained in a cylindrical chamber which is rotated uniformly by a motor, *M*, through a worm-drive, *R*. A cylinder, *C*, is suspended in the liquid by a thin steel wire, *W*, so that the axis of the cylinder coincides with the axis of the rotating liquid. The rotating container is provided with a cap, *B*, so shaped that the excess of liquid can overflow when the cap is seated and thus give constant conditions within the chamber. The specimen chamber is surrounded by an oil jacket, *J*, with a thermometer, *T*, and the outside of the rotating chamber carries small mixing wings which cause a circulation of the jacket-oil; the jacket may be heated to any desired temperature by means of a coil or other device. The cover of the jacket chamber, *D*, is graduated in the form of a scale marked in degrees or calibrated in terms of a standard liquid, and the torque imparted to the suspended cylinder is indicated by the pointer *P*. The suspended cylinder and rotary container are made of copper to ensure rapid uniformity of temperature. If the temperature of the jacket be slowly raised, it is possible to secure direct readings for the temperature-viscosity curve in a continuous experiment. The accuracy of this instrument has been verified by comparison with observations made on gas-engine oils by a standard capillary tube instrument; on the other hand, determinations made with the same oils in a short capillary tube instrument and an orifice instrument showed far lower results. With the new rotary viscosimeter the sensitiveness can be varied by varying the speed of rotation or using suspension wires of various diameters. The density or change in density of the liquid is not a factor in the results, so that the indications are correct at all temperatures. The error of measuring short time intervals is excluded and the presence of suspended particles in the oil is without effect. J. F. B.



✣ ✣ ✣ ✣ ✣

Erratum.—Page 296, line 4, *for* $\frac{N}{2}$ *read* 2 N.

REVIEWS.

A Method for the Identification of Pure Organic Compounds. Vol. II. By Samuel Parsons Mulliken, Ph.D. John Wiley and Sons, New York; Chapman and Hall, Ltd., London. Price 21s. net.

By the author's system of classification, described in Vol. I., organic compounds are divided into order, genus, division, section, and species, which, when it is not logical, is at least biological. Having dealt with Order I., Dr. Mulliken now brings under review Order II., comprising the substances which contain the elements (*a*) carbon and nitrogen, (*b*) carbon, nitrogen, and hydrogen, (*c*) carbon, nitrogen, and oxygen, (*d*) carbon, nitrogen, hydrogen, and oxygen, an imposing array of close upon 4,000.

Preceding the tables in which these materials, with their physical and chemical properties, are arranged according to the above system, there are described the tests necessary to supplement those already given in Vol. I., and among them are included scales of bitterness, sweetness, and pungency, based upon quinine sulphate, cane-sugar, and ammonium hydroxide, respectively. Then follow the tables:—Suborder I., Genus I. (Acidic), Division A (Solid), Division B (Liquid). Suborder I., Genus II. (Basic), Division A (Solid), Division B (Liquid). Suborder I., Genus III. (Neutral), Division A (Solid), Division B (Liquid).

All these are colourless, or have colours "less saturated than Tint 3 of the color standard accompanying Vol. I.," and it is stated in a footnote to page 3 that "this color standard, consisting of the two cards A and B and a perforated screen, will be mailed to any person owning this work upon receipt of a postal money order for one dollar." Suborder II. comprises the solid "species" of this order, which are more saturated in colour than Tint 3 of the standard, but, for a reason which is obscure, acids, bases, and neutrals in this suborder are not distinguished.

The compounds in each division are tabulated in ascending order of melting-point or boiling-point, as the case may be. The work is thus directed towards paving a royal road to identification, and at first sight would seem to be a godsend to the harassed examinee; it looks almost as simple as furnishing at Drage's. Nobody has yet invented a thornless rose, however, and one disadvantage of the book from the standpoint of the sanguine student lies in the enormous number of materials involved, and of which hardly more than 3 per cent. are likely to be presented by any humane examiner. Consequently, to be useful, melting-points must be taken with meticulous accuracy, since a popular temperature, slightly confused by an unfaithful thermometer, may confront the victim with twenty or thirty alternatives. Moreover, there is always Tint 3 lurking in the background, tempting the ingenious examiner to crystallise his picric acid from petroleum.

Dr. Mulliken and his associates deserve the greatest possible credit for the monumental industry which they have exercised, but it remains questionable whether the tortoise method of identification is not, after all, the best education for the chemical sleuth.

M. O. Forster.

CHANGES IN THE FOOD-SUPPLY AND THEIR RELATION TO NUTRITION. By LAFAYETTE B. MENDEL. Oxford University Press, 1916. Price 2s. 6d. net.

In this suggestive essay, written for the meetings of the second Pan-American Scientific Congress at Washington, December, 1915, the author touches, somewhat lightly, on many interesting aspects of the question of national food-supply, more especially as affecting America, the object being rather to indicate the importance of many of the factors concerned than to make any definite suggestions. Short sections deal in turn with food production, preservation, and transportation, custom in diet, and changing social and hygienic conditions, and many interesting and curious facts are brought forward.

The author lays stress on the circumstance that the most diverse diets in various parts of the world are known to produce adequate nutrition, so that no one food material can be regarded as essential. He further points out, basing his observation on the recent work on nutrition carried out both in America and at home, that a profound modification of the food problem may be effected by the addition to unsatisfactory food-stuffs, such as maize, of a small proportion of some other material rich in the constituents which are missing from the former.

The importance of " accessory food factors " is also duly and rightly insisted on, and the danger accruing from the unrelieved employment of " artificial products " is indicated.

It is comforting to find that as regards the future food-supply of the world the author takes an optimistic view, considering that the progress of knowledge will for a very long period be more than able to cope with the increase of population and all its attendant difficulties. A. HARDEN.

MICROSCOPY OF VEGETABLE FOODS. By A. L. WINTON, PH.D. Second Edition. New York, J. Wiley and Sons, Ltd.; London, Chapman and Hall, Ltd., 1916. Price 27s. 6d. net.

The first edition (1906) of this excellent work—which, however, is but a slightly improved issue, in English, of the second edition (1905) of Moeller's " Mikroskopie der Nahrungs-und Genussmittel "—was reviewed in the ANALYST (1907, **32**, 138).

This second edition (1915) is little more than a reprint of the first edition, and those who possess Moeller's work or Winton's first edition will hardly need to provide themselves with this.

There are exactly the same number of pages in the two editions—viz., 701. The Table of Contents, Glossary, and Index are exact reprints of those in the First Edition, and the General Bibliography so closely follows the earlier one that no additions to it have been made, though there is mention of some in the text of the work, and all that has been done is to substitute the date of a later for an earlier edition. That there are only five references in the General Bibliography to any work issued since 1906 would seem to indicate that finality had been reached in this subject.

As one puts the two editions side by side, page after page is ruled off without coming to any alteration, and when such does occur, by substitution of one diagram for another, or by the interpolation of new matter, there is seen the anxiety to catch up and get the old similarity of page-numbering and page-material restored.

The failure to have observed, or unwillingness to recognise, any work done subsequently to 1906, and to bring the present edition up to date, is shown by the occurrence in identical terms in the two editions of the footnote on p. 62: "Recently Brahms and Buckwald have found that the name aleurone cells is . . . erroneous." What was recent in 1906 would hardly be so in 1916.

Beyond the possible call for a re-issue of the work, and which one can well understand and appreciate—for the book is a really good and valuable one—the main idea of the second edition has been to give prominence to the work of the author's collaborateuse, Kate Barber Winton, and of Miss Kate G. Barber, whom, from the similarity of the diagrams in the two editions, one takes to be the lady who assisted in the preparation of the first edition.

Thus, there are now 635 illustrations as against 589 in 1906, 68 new ones—all but one being by one or other of the above-named ladies—being introduced, while 22 (mostly Moeller's) have been dropped, and sometimes not to advantage. The new matter is stated in the preface to consist of "additions to the sections on wheat and flour; a complete revision of such parts of the chapter on oil seeds as treat on mustards, rapes, cruciferous weed seeds, and linseed; a description of the histology of alfalfa, with distinctions from red and Alsike clover; a revision of the sections on pomes and drupes, with practical hints on the examination of almond pastes, jams, preserves, and other products; and rewritten descriptions of the cucurbitaceous fruits used as food and adulterants." An examination of these new features will lead to the conclusion that the revisions are not important nor extensive, nor are the additions material. In short, little or nothing has been done to supplement the information given in the first edition or to remedy defects in it.

When it is noticed that the subjects of mustards, rapes, etc., and of alfalfa, as well as of fruits generally, are those with which the additional illustrations supplied by K. B. Winton are associated, and that K. G. Barber has made all the illustrations of cucurbitaceous fruits, it will be seen that the new matter of this edition is concerned practically with the work of these ladies.

To take the new features in detail. Under Wheat and Flour come—on p. 50 an expansion of the description of the Pekar colour test, and on p. 55 a similar one of Banichl's test; two new cuts on p. 66, a slight alteration of the descriptive text on p. 67, the replacing of one of Moeller's illustrations by a K. B. W., and the dropping out of another illustration (p. 68 in first edition) by Moeller, which, to the reviewer's mind, gives to the analyst accustomed to deal practically with these matters a better idea than all the other illustrations, of what he is likely to come across in the actual examination of samples he has to identify. These are all the changes in this section.

Under Oil Seeds there are—a slight change in the analytical key to cruciferous seeds; three additions to the Bibliography (none of them noted in the Index); 10 illustrations by K. B. Winton of white, black, and brown mustard, and rape, in place of 6 of Moeller's, all of which have been dropped. Moeller suffers similar replacement in the case of false flax, but there are 4 new illustrations by K. B. Winton of shepherd's purse and pepper grass. Under Linseed there are 5 new

K. B. W. illustrations, Moeller's 2 being now omitted, and an addition of 10 lines is made to the histological description of the plant.

Under Legumes, alfalfa now has 3 pages devoted to it in place of 20 lines, the extra space being occupied mainly by 8 new illustrations, all by K. B. Winton.

Coming next to Fruits, there are 12 new illustrations by K. B. Winton, and the additions to the text do not occupy over a dozen lines.

In the section devoted to Vegetables, Miss Kate G. Barber has her turn, coming out strongly with 14 new illustrations of pumpkin, squash, cucumber, and melon, 2 of Moeller's having to make room for them. After the description of water-melon —which ends at p. 410 in each edition—the pages to the very end (p. 670) are exact duplicates throughout.

It is a pity that the opportunity was not taken to amplify in some cases the information given concerning the occurrence and use of the materials described, and to revise the somewhat loose statements met with here and there. If more is dealt with than the actual description and the microscopical characteristics, such additional information should be fairly complete and also correct. This is not always the case here—*e.g.*, it is merely said of linseed cake that it is " often contaminated with cruciferous and other seeds " (p. 206), and that it is " the mucilaginous substance found in linseed that gives the seed its value in medicine " (p. 204); alfalfa (p. 266) is spoken of as being grown especially in the arid and semi-arid regions of the United States, but there is no mention of the Argentine or other parts where it is extensively cultivated; to Carob bean (p. 277) the alternative and better-known name " locust bean " is not given, nor to pea-nut (p. 269) those of " earth-nut " and " ground-nut "; to Soya bean (pp. 248-9), because of its now largely increased use, more space might have well been given, and there is no mention of its being made into a feeding cake or of its other uses in the manufactures.

Here and there, and especially in the section relating to condimental foods, paragraphs bearing on " chemical examination " have been inserted, but these are of such a general nature as to be practically useless. Either this should be set out adequately or it should be omitted altogether, and reference be made to special books where it can be found.

It is to be regretted that the author, while giving such prominence to vegetables such as cucumber, melon, etc., has not seen his way to deal more fully with the distinctions that occur between seeds, etc., that may occur together and be readily mistaken one for the other—*e.g.*, cotton seed and kapok (silk-cotton) seed. The work would be made much more useful to the practitioner if it contained more information of this kind. It is comparatively seldom that the analyst has single materials only to recognise, but he has more frequently to identify different ones in the presence one of another, and a setting-out of the main characteristics as they present themselves in actual practice would be a great help to him. Thus, while it may be comparatively easy, by the aid of such illustrations as this book supplies, to recognise single substances, the occurrence of several together requires special experience to enable one to identify them, and it is to be feared that this work, valuable though it is, will give only modified help. Materials as they occur in actual practice, as, for example, in the case of an adulterated linseed cake, a compound feeding cake, or a food containing castor bean or other poisonous seed, do not present the clear

appearances set out in the illustrations given, and the analyst looks in vain for a setting-out of those characteristic features presented by each material or seed on which his experience tells him he must mainly rely. There is still a field open for a work which will supply this practical guidance. J. A. VOELCKER.

TECHNICAL CHEMIST'S HANDBOOK: TABLES AND METHODS OF ANALYSIS FOR MANUFACTURERS OF INORGANIC CHEMICAL PRODUCTS. By GEORGE LUNGE, PH.D. Second edition, revised. London: Gurney and Jackson, 1916. Price 10s. 6d. net.

The author's intention in writing this text-book has been to assist as far as possible in securing uniformity not only amongst practical chemists and analysts, but also amongst buyers and sellers, in regard to numerical data and analytical methods employed in checking processes and testing the resulting products.

As in the previous edition, one good analytical method only is given in each case, one process for the preparation of standard solutions, and also one for the examination of each of the materials used in the particular industry concerned. This, as Dr. Lunge points out, tends to avoid discrepancies such as might arise should two or more methods be described.

In the present edition numerous small changes occur in the numerical data given, due to the recalculation of the latter on the basis of the atomic weights published by the International Committee for 1916. The first few tables include percentage compositions of the majority of chemical compounds in general use, factors for calculating gravimetric analyses, densities of gases and vapours, solubilities of salts and gases in water, specific gravities of solids and liquids, melting and boiling points. These are followed by gas analysis tables, properties of liquefied gases of commercial importance, mathematical tables, and also factors for the conversion of weights, measures, and coinage of different countries.

A very useful chapter devoted to fuel and furnaces gives also concise methods for testing the gases evolved, and tables to facilitate the work of the analyst. The manufacture of sulphuric acid, the examination of the materials employed and by-products obtained, are briefly described, the necessary calculation tables being appended. Some fifty pages in all are allotted to this important industry.

Bleaching powder, chlorate of potash, soda ash, and nitric acid, their manufacture and commercial examination, are all dealt with in turn, and the descriptions, though brief, are clearly given, and should suffice for the trained chemist, for whom the book is primarily intended.

In the final sections the cement industry and the production of coal-gas are discussed in a similar way, and some useful notes provided on the preparation of standard solutions and on methods of sampling.

The little book is compiled with skill and discretion, and in addition possesses the advantage of being handy in form, going conveniently into the pocket. Great care has evidently been taken in revising the proofs, a matter that in a work of this nature, filled as it is with numerical data, is all-important. There is little doubt that the need for tables of this kind is experienced by every analyst and technical chemist, whatever branch of work he may be engaged in, and hence Dr. Lunge's Handbook should find a welcome place in every laboratory.

P. A. ELLIS RICHARDS.