THE ANALYST

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

AN Ordinary Meeting of the Society was held on Wednesday, April 4th, at the Chemical Society's Rooms, Burlington House. The President, Mr. P. A. Ellis Richards, F.I.C., was in the chair.

A Certificate was read for the first time in favour of Mr. Robert C. Grimwood, A.C.G.I., D.I.C., A.I.C.

Certificates were read for the second time in favour of:—Messrs. John Myers, F.I.C., and John Loudon Buchanan, F.I.C.

The following were elected Members of the Society:—Messrs. Joseph John Valentine Backes, A.R.C.Sc., A.I.C., D.I.C., Francis Kenelm Donovan, B.Sc. (Lond.), Alfred Edward Johnson, B.Sc. (Lond.), F.I.C., A.R.C.S.I., Ernest Victor Jones, F.I.C., S. Gordon Liversedge, F.I.C., Samuel Gordon Stevenson, A.I.C., Richard William Sutton, B.Sc.Tech. (Manc.), A.I.C., and Laurence Barnett Timmis, M.Sc. Tech. (Manc.), A.I.C.

The following papers were read:— "Physiological Standardisation," by Dr. Stanley White; "An Investigation into the Chemistry of the Reinsch Test for Arsenic and Antimony and its extension to Bismuth," by B. S. Evans, M.B.E., M.C., B.Sc., F.I.C.; and "The Estimation of Boric Acid in Liquid Eggs and other Foodstuffs," by G. W. Monier-Williams, M.A., Ph.D., F.I.C.

Obituary.

GEORGE EMBREY.

IT is with regret that we have to record the loss of another of the oldest of our Public Analysts, George Embrey, who passed away at the ripe age of 83 on March 10th, 1923.

He was born in Birmingham, May, 1840, and after his education at King Edward's School in that city went to the Birmingham and Midland Institute, and later to the Royal College of Chemistry.

His advent to Gloucester was in 1873, when he was appointed to the School of Science, remaining there as a master until 1895. He was appointed County

Analyst and City Analyst for Gloucester in 1885, and later official Agricultural Analyst to the same authorities, all of which offices, as well as those of Gas Examiner to Cheltenham and Gloucester, he held until his death.

He served for six years as a member of Council of the Society of Public Analysts, was elected as Vice-President in 1913, and became President for the years 1916 and 1917. He was an enthusiastic Freemason, Master of his Lodge (the Royal Lebanon) in 1903, and a Grand Officer of his Province. He celebrated his golden wedding in 1913, but had been a widower since 1915, and he now has left one daughter to mourn his loss, his son having died in early life.

To those of us who knew him intimately he was a charming man, somewhat dogmatic perhaps, but always exceedingly courteous. He had a highly artistic temperament, being a keen musician, vocalist and instrumentalist. He was also an expert in photography. He was a great student of Dickens, and never tired of recounting his various personal associations with the great novelist, and just a year ago he was President of the Dickens Fellowship in his city.

As a lecturer upon almost endless topics, he could always engage and maintain the interest of his audience, and as a chemist he knew his work, and was always a steady plodder with an infinitude of painstaking, a good reliable witness, sure of his facts, and never drawing upon his imagination, putting his whole soul into his work.

His Fellowship of the Institute of Chemistry dates back to 1901, and of the Chemical Society to 1885, and he was a very old member of our Society.

His first communication to the ANALYST was in 1891, when he read a paper on English and American cider, and later he contributed papers on "The Detection of Maize in Wheat Flour" (ANALYST, 1900, **25**, 315), and on "Uniformity in Soil Analysis" (ANALYST, 1900, **25**, 290). At the June meeting in 1917 he read an interesting paper on "Some Experiences in the use of Copper Sulphate in the Destruction of Algae," in which he gave an account of the nature and effect of the algae growing in water, and of his experience in destroying them by the application of copper sulphate (ANALYST, 1917, **42**, 264). This was his last contribution to our journal.

The county and city of Gloucester will deeply feel his loss after his long association with scientific, philanthropic, educational and religious work, for almost until the last he was one of those fortunate men who never seem to grow old, or, at any rate, he never showed it, his energy and mental activity never flagging, and the long list of distinguished men who attended his funeral on March 14th testified to the popularity of George Embrey.

GEO. RUDD THOMPSON.

The Examination of Firearms and Projectiles in Forensic Cases.

By A. LUCAS, O.B.E., F.I.C.

(Read at the Meeting, March 7, 1923.)

In cases of wounds or death from shooting, a considerable amount of valuable evidence may sometimes be obtained from the examination of a weapon found in the possession or on the premises of a suspected or accused person, and from an examination of the projectile taken from the body of the victim or found at the scene of the crime. Both these points have been discussed in my book *Forensic Chemistry*,* and I now propose to summarise briefly the results there described, and to add a considerable amount of new material.

FIREARMS.

Firearms comprise military and sporting rifles, shot guns, revolvers and automatic and other pistols.

From the point of view of criminal investigation, a very important aspect of firearms is the rifling of rifled arms, since this affects the bullet fired, imprinting on it distinctive and characteristic markings by means of which the nature of the weapon, and sometimes even a particular weapon, may be recognised. The usual number of grooves in a military rifle is four; in the British military rifle, however, it is five; in the Swiss military rifle it is three; and in the Danish military rifle it is six. The number of raised portions or "lands" between the grooves is of course the same as the number of grooves, and it is the lands which mark the bullet. In almost all military rifles of all nations the twist of the rifling is right hand, exceptions being the British Lee-Enfield rifle and the French Lebel rifle, in both of which the twist is left hand.

The following table gives particulars of the rifling of a few weapons:

Weapon		Description	No. of Lands	Direction of Twist
Rifle		Martini-Metford	7	Left
,,		Martini-Enfield	5	,,
,,		Lee-Metford	7	,,
,,		Lee-Enfield	5	,,
Automatic Pistol		Webley-Scott ·455	7	Right
,,	,,	Colt ·45	6	Left
,,	,,	,, ·38	6	,,
,,	,,	,, ·32	6	,,
,,	,,	Browning ·32	5	Right
,,	,,	,, ·25	6	,,
Revolver		Webley-Scott ·455	7	,,
,,		Smith & Wesson ·455	5	,,
,,		Webley-Scott ·380	7	,,
,,		,, ,, ·320	5	,,

* London, 1921 (C. Arnold & Co.).

Another important aspect of firearms when examined in connection with a crime, or suspected crime, is the nature of the fouling left in the barrel after the weapon has been fired. Apart from a small amount of metallic fouling caused by particles of the material of the projectile being scraped off and remaining in the barrel, the deposit left in a firearm after the weapon has been discharged consists of solid matter derived from the powder. The nature of the fouling therefore depends to some small extent upon the kind of projectile, whether, for instance, it is nickel-coated, or whether it is naked lead, but it depends more particularly upon the kind of powder used. Since metallic fouling has very little significance in criminal investigation, it will not be dealt with further.

POWDERS.—Powder for firearms is of two kinds, namely, black powder and smokeless or nitro powder.

Military rifle ammunition is loaded with nitro powder, sporting rifle ammunition may contain either nitro powder or black powder; automatic pistol ammunition always contains nitro powder; modern revolver ammunition generally contains nitro powder, but may contain black powder; shot-gun cartridges may be loaded either with nitro powder or with black powder.

Black powder consists of an intimate mixture of charcoal, potassium or sodium nitrate and sulphur. The solid residue left when black powder is consumed consists of potassium carbonate, potassium sulphate, potassium sulphide and potassium thiocyanate, a little of the original charcoal, and a little of the original sulphur, which last, however, combines with the potassium monosulphide to form polysulphide. Potassium thiosulphate is also found, but this is probably not an original product, but a secondary product, resulting from the oxidation of potassium sulphide. It is always stated too that a little potassium nitrate is left; but, although tests have frequently been made for nitrate, the results have always been negative. A trace of nitrite, however, is generally found after the sulphide has disappeared, which it soon does by oxidation.

Smokeless powder, as used in firearms, varies considerably in composition. The principal constituent is nitrocellulose, either alone or together with nitroglycerin, but other ingredients that may be present include potassium nitrate, barium nitrate and wood meal. With such varying composition, it follows that the products of combustion must also vary, and must necessarily differ from those derived from black powder. When smokeless powder is consumed there is no formation of sulphide and no carbonate, which differentiates the products at once from those of black powder; sometimes there is sulphate, but only a trace, and never so much as with black powder; there is always nitrite, and always more than from black powder, and usually nitrate, which varies from a trace to a considerable amount.

There is no difficulty whatever in ascertaining whether the powder used was black powder or nitro powder, and this fact alone may help materially in determining the guilt or innocence of a suspected person. But something more than this may be done. Thus the amount of nitrate found in the residue after discharge differs considerably with different powders, Ballistite, for example, giving less

nitrate than Bonax, Nobel's "Empire," Schultze or Walsrode powders. This might prove useful negative evidence, and it might be possible to show that a certain kind of powder which was suspected had not been used. The presence of such substances as potassium or barium would also provide useful indications of the nature of the powder.

METHODS OF EXAMINATION.—On the receipt of a firearm for examination, the first thing to do is to note the condition of the interior of the barrel, whether, for example, it is clean, whether it is oiled, whether there is any sign of fouling or of rust. In the case of a revolver, the condition of the chambers is also noted. It is also ascertained whether there is any smell at the muzzle end, and, if so, its nature and intensity. Except in the case of military rifles and automatic pistols, a test for sulphuretted hydrogen is then applied by means of lead test paper.

After this preliminary examination, the firearm should be held in a slanting position, and a small quantity of hot water poured down the barrel, a little at a time, from the breech end, an effort being made, by turning the weapon, to reach the whole area of the inside of the barrel. A receptacle to receive the water is placed at the muzzle end. The most convenient way of delivering the water is from a wash bottle or pipette, and a small beaker will be found suitable for receiving the washings. As a rule, about 20 c.c. of water will be found sufficient for a rifle or shot-gun, and about 10 c.c. for a revolver or pistol. The water which has been through the barrel a first time should be passed through once or twice again, the firearm being turned and held in a different position each time, so that all sides of the barrel are well washed. The colour, appearance and smell of the solution should all be noted, and the liquid should then be filtered. A small portion of the filtered solution should be tested for reaction to phenolphthalein, and another small portion with acetate of lead for sulphide. A further portion is acidified with hydrochloric acid and tested for sulphate with barium chloride, for ferrous iron with potassium ferricyanide, and for ferric iron with potassium thiocyanate. Another portion should be tested for nitrite with sulphanilic acid and naphthylamine acetate or chloride, and a further portion for nitrate with phenolsulphonic acid. The colour of the residue on the filter paper should be noted, and this residue should be treated with strong hydrochloric acid and the solution tested for both ferrous and ferric iron.

The results obtained will serve to distinguish between black powder and nitro powder, and sometimes may even indicate a particular kind of nitro powder. They will also show the presence or absence of rust. But the results may also show, within certain limits, the period that has elapsed since the firearm was last discharged. Thus, if gaseous hydrogen sulphide is present, the discharge has been very recent; if sulphide is present in the solution in more than a trace, the discharge has probably taken place within five hours; if rust is present, the discharge has probably been at least 24 hours previously.

Finally, the weapon as a whole should be examined for calibre, rifling, state of repair, working condition, manufacturer's marks, number, country of origin, proof marks, etc. For the examination of the interior of a barrel, a barrel-reflector will be found useful.

PROJECTILES.

The principal kinds of projectiles are bullets, slugs and small shot.   These, as also wads and cartridges, will now be dealt with.

BULLETS.—The points requiring attention in any examination of a bullet are (*a*) the composition, (*b*) the dimensions, (*c*) the weight, and (*d*) rifling and other marks.   These will be separately considered.

Composition.—Bullets are generally made either of lead or of lead enclosed in a thin envelope of some harder metal, usually cupro-nickel or copper plated with nickel, but may be of copper-zinc alloy, as in the case of the French military rifle bullet, or the envelope may be of copper, as in the Japanese military rifle bullet, or of steel as in the Austrian and Bulgarian military rifle bullets, or of steel plated with cupro-nickel as in the German, Greek, Dutch, and Turkish military rifle bullets. The British military rifle bullet, Mark VI., is of lead enclosed in a cupro-nickel envelope composed of 80 per cent. copper and 20 per cent. nickel. Mark VII. bullet consists of lead tipped with aluminium, or with an alloy of aluminium (90 per cent.) and zinc (10 per cent.) enclosed in a similar envelope to Mark VI. bullet. This envelope is not of a uniform thickness throughout, but varies from about 0·4 mm. at the base to about 0·7 mm. near the point. The Swiss military rifle bullet once had a body of hard lead with an envelope of nickel-plated steel on the point only, the remainder of the bullet being covered with paper lubricated with vaseline. The Snider rifle bullet was of soft lead, and the Martini-Henri bullet was of lead, containing 1 part of tin to 12 parts of lead (8·3 per cent. of tin).

Rifle bullets used for sporting purposes are frequently entirely of lead uncoated, while some kinds of bullets (soft nosed) are only partly coated, the front portion being left bare. Revolver bullets are generally uncoated. Automatic pistol bullets are practically always coated.

It may be mentioned in this connection that cases occasionally occur in which the envelope of a coated bullet is found, after firing, entirely stripped from the lead core.

The lead employed for bullets, which is ordinary trade lead, usually contains small proportions of various impurities, among which arsenic, antimony and tin may be specially mentioned. In addition to any antimony present as an accidental impurity, this metal is frequently added for hardening purposes in order to give greater penetration, the lead core of the British Army bullets, Marks VI. and VII., for example, being hardened by adding up to 5 per cent. of antimony.

For the determination of the exact composition of a bullet, a chemical analysis is necessary, and the methods employed will be described later. In the case of a coated bullet, both the core and the envelope should be analysed. These may be separated by melting the lead and pouring it out through the uncoated area generally left at the base.

Dimensions.—The diameter and length of a bullet should always be determined. The diameter of a bullet depends upon and varies with the calibre of the weapon.

used, naturally being slightly less.   The size of a bullet is usually given as that of the calibre of the weapon for which it is made, irrespective of its actual diameter. Thus, for example, a bullet for a ·455 automatic pistol or revolver is called a ·455 bullet, though it may not measure more than ·448 or ·450 inch.

Weight.—This is an important factor in the identification of a bullet, especially if the bullet is distorted.

Rifling and other Marks.—Bullets are sometimes marked by the manufacturer at the base with letters or figures.   All such marks should be noted, as they may be helpful for identification purposes.   In addition, however, to marks placed on a bullet by the manufacturer, all bullets which have been fired from rifled arms bear the marks of the rifling.   These marks take the form of longitudinal grooves, which are not parallel to the principal axis of the bullet, but which are slightly inclined to the right or left according to whether the twist of the rifling is in the direction of the hands of a clock or the reverse. The number of grooves, their width, direction and distance between them, should all be determined.   In most instances there will be longitudinal marking in the grooves themselves, and generally marking between the main grooves.   There may also be imperfections of the grooves.   These secondary markings are caused by rust, metallic fouling, scratches or other defects in the barrel, and they may be sufficiently characteristic to indicate the actual weapon employed.

When comparing different bullets of the same kind and size, it is manifest that if either the number of grooves or the direction of twist does not correspond, the bullets cannot have been fired from the same weapon; but when both these agree, the bullets have been fired from similar weapons, and possibly from the same weapon.   To determine whether this is so or not, the secondary markings must be examined; for if the bullets have been fired from the same weapon, the secondary markings will correspond in their main features, some of which may be very characteristic.   Absolute identity, however, between all the small details of the secondary markings is rarely if ever found.   This is probably due to very slight differences in the size and shape of bullets nominally alike, or to bullets not fitting tightly into the barrel; or, in some instances, to rust, scratches or other alteration in the condition of the barrel between the two firings.   It is always necessary to exercise great care before arriving at any decision regarding the identity of a weapon from the markings on a bullet, but it is particularly necessary when the bullet is a lead one, since the details of the markings on naked lead bullets are not so distinct as a rule as on coated bullets.

An excellent way of obtaining an impression of the main grooves on a bullet, in order to measure their width and the distance between them, is to roll the bullet along (*a*) a thin sheet of dentists' moulding composition, the surface of which has been carefully smoothed against a glass plate (this composition is used hot and sets hard when cold), (*b*) dentists' sheet wax, or (*c*) tin foil backed by a plastic material such as plasticine.   Another method which may be used to obtain an impression on paper is practically "offset" printing as employed by lithographers.   For this a strip of paper is inked on one side with printers' ink by

means of a roller in the usual way. On the inked side of this a clean piece of paper is laid, and the bullet rolled along the upper paper with considerable pressure. The impression obtained on the upper paper is then transferred to a thin sheet of india rubber by vigorous rolling with a dry roller, and from the rubber it is transferred in the same way to paper again. Enlarged photographs of the impressions obtained in one or other of the ways mentioned are useful for exhibition in court. The finer details of the secondary markings unfortunately do not reproduce well.

In order to examine and compare the markings on different bullets, a lens, or better, a stereoscopic microscope with a low power, should be used. Stereoscopic photographs are also helpful, and should be made whenever possible.

Before a bullet can be examined satisfactorily, it must be mounted in some kind of a holder so that it may be rotated easily. A simple and satisfactory method is to fasten the point of the bullet (which is free from markings and therefore is not needed for examination) into the end of a small glass tube by means of plasticine, the width of the tube being appropriate to the diameter of the bullet. Two bullets mounted in this manner, when placed side by side, can be compared readily. The bullets of course are upside down, but this does not affect the examination. If the bullets are mounted on any elaborate stand, microscopic examination is difficult. When mounting or handling bullets, it is of course essential that they should not be scratched or marked in any way, and therefore any adhesive used must be of such a nature that it can be washed or dissolved off, since scraping, which would injure the bullet, cannot be employed.

Reproduction of Markings.—To reproduce on a bullet for comparison purposes the rifling marks from a particular weapon, the bullet should be fired into soft earth or into a sack stuffed with cotton or rags or into water, since anything hard will distort the bullet and deface the markings. Even fine sand will so scratch a bullet that the rifling marks are partly obliterated. A very satisfactory method for revolvers and pistols is to fire the bullet into an iron pipe (length about 5 feet, diameter about 8 inches) filled with sifted earth.

Instead of firing a bullet to reproduce the markings, these may be obtained equally well if the bullet is forced through the barrel of the weapon by means of a brass rod aided by gentle tapping with a hammer. In all comparison tests, it is essential that the bullets should be identical in material, shape and size.

On a lead bullet which has passed through clothing, it is sometimes possible to see the imprint of the texture of the cloth first penetrated. In order to identify this, an enlarged photograph is taken of the imprint and of similar imprints made by firing bullets of the same kind and size into the suspected clothing.

In this connection it may be mentioned that the chemical examination of shot marks on clothing may occasionally prove of value. Thus an alkaline residue points to the use of black powder, and nitrate to nitro powder, while potassium might indicate either black powder or nitro powder.

SLUGS.—These should be examined like bullets for (a) composition, (b) dimensions, and (c) weight.

Composition.—Slugs are composed of soft lead which, being of commercial quality, generally contains small proportions of antimony and tin, and occasionally

traces of arsenic. Eleven slugs from different cartridges which were analysed all contained antimony, the amount ranging from 0·30 per cent. to 0·90 per cent. Five of these slugs did not contain tin, while six contained tin, which varied in amount from 0·17 per cent. to 0·55 per cent. Arsenic was present in several instances, but only as a trace.

Dimensions.—The slugs from any one cartridge are usually of a similar, though not absolutely identical, size, but there are exceptions to this, and occasionally different sized slugs occur in the same cartridge. Amateur-made slugs are of very irregular shape and size.

A square slug may become so distorted on impact against a hard body as to make an almost round hole, and when found may be round or oval with definite "mushrooming," and may show little or no evidence of having been square. The presence of straight lines or slight ridges, which are sometimes all that remains of the edges of the slug, should be searched for specially.

Weight.—Although the slugs from any one cartridge are generally nominally of the same weight, this is not always the case.

SMALL SHOT.—Originally English small shot was of two kinds, "soft" and "hard" or "chilled." Both these kinds of shot were made of ordinary commercial lead, to which a small proportion of arsenic was added for hardening purposes, and to enable the lead better to assume the spherical shape when the molten metal was dropped into water. Hard or chilled shot was specially cooled by means of a blast of cold air, and this caused the metal to harden more than was the case with shot not so treated. Shot is now hardened, however, by the addition of a small proportion of antimony, but when so made, it is still frequently termed "chilled."

The question of the size of shot is sometimes of considerable importance, as in the Monson case. The size of the pellets, however, may vary in the same cartridge, and great care is required before making any general deductions from the size of isolated pellets.

ANALYSIS OF PROJECTILE.—As a rule, in shooting cases, the amount of material available for analysis is very small, since not only is the total amount small, but of this as large a quantity as possible must be reserved for further analysis or for production in Court. In such cases, where a comparison between several different samples of lead only is required, the following method will be found useful:—Take 0·5 grm. of each sample in a finely divided condition, put into separate small conical flasks, and add to each 5 grms. of ammonium sulphate and 10 c.c. of strong sulphuric acid, and heat strongly until the metal is all dissolved and the precipitated lead sulphate is white or practically white. Although the bulk of liquid is small, there is no fear of the flask breaking. The lead is fully precipitated as sulphate, and any arsenic, antimony and tin present remain in solution. Cool and add to each flask 25 c.c. of a 10 per cent. solution of tartaric acid. This prevents the antimony and tin from being precipitated by the water, which, if it were not added with the tartaric acid, would have to be added to dilute the solution before filtration. Shake well and boil for five minutes in order to granulate the sulphate of lead. Filter into Nessler tubes, using ordinary filter paper and making no attempt to transfer all the lead sulphate to the paper, and

wash with dilute sulphuric acid (10 per cent. solution), using a minimum quantity and approximately the same amount in each case.   To the filtrates add a saturated solution of sulphuretted hydrogen.   Make up to the same volume (50 c.c.) with water in each case, stir well with a glass rod and compare.   The contents of the tubes may be transferred to glass-stoppered bottles and preserved for presentation in Court.   If tall narrow bottles of clear glass are used, the different amounts, colours and general appearance of the various precipitates are very marked, and demonstrate the results in a manner that no mere recital of percentages can possibly do.   If required, this method may be made quantitative, in which case, however, the filtration from the lead sulphate should be through asbestos, using a Gooch crucible.   The antimony and tin are best estimated volumetrically, after separating any arsenic present; the former by means of potassium permanganate, and the latter with iodine.

WADS.—Not only may the examination and analysis of the projectile prove of value, but the nature, diameter and thickness of any wads found may also provide valuable evidence.   As a rule, however, wads are only found in cases where a shot-gun has been used, since revolver and pistol wads, even when present, are very small and generally escape notice.

CARTRIDGES.—These should be examined and carefully measured, and the kind of case, the dimensions and all marks indicating the manufacturer, place of manufacture, date and size should be recorded.   One or more cartridges should then be opened and the bullet or pellets, the charge, the wads, and the case, should all be separately weighed, measured, and examined, and the charge analysed, a qualitative analysis generally being sufficient.

Cartridges belonging to revolvers and automatic pistols respectively may be distinguished in several ways: thus, revolver cartridges invariably have a rim at the base and the bullet is not coated, whilst automatic pistol cartridges have no rim, but there is a recess round the base for the extractor to engage in, and the lead of the bullet is nearly always coated with some harder metal such as cupro-nickel or copper plated with nickel.

Even from empty cartridge cases much may be learned, as, for instance, the nature and size of the shot and the nature of the weapon.   Sometimes, too, characteristic marks left on the case by some defect or peculiarity of the weapon, or by the manner in which the hammer has struck the cap, may serve to identify the particular weapon used.

It should not be forgotten that cartridges are not always employed with the particular kind of weapon for which they were intended, and an automatic pistol cartridge, for example, may be fired from a revolver.   A revolver cartridge cannot be fired from the magazine of an automatic pistol on account of the rim on the cartridge, but may be fired if placed in the barrel.   Rifle cartridges, too, are sometimes cut down to fit a revolver.   In the Dickman case, two ·250 automatic pistol cartridges were wrapped in paper and fired from a ·320 revolver.

Problems such as those concerning the spread of shot and the distance and direction from which a firearm has been fired, should be left to sportsmen or gunsmiths.

Sliding Scales for the Convenient Titration of Strong Liquids by Dilution and Use of Aliquot Parts.

By C. H. D. CLARK, B.Sc., D.I.C., A.I.C.

PART III. (CIRCULAR FORM).

IT has been suggested to me that the Sliding Scale which has been described might be more convenient for certain purposes if arranged in a circular form. I have therefore considered this question, and the result is shown in the diagram.
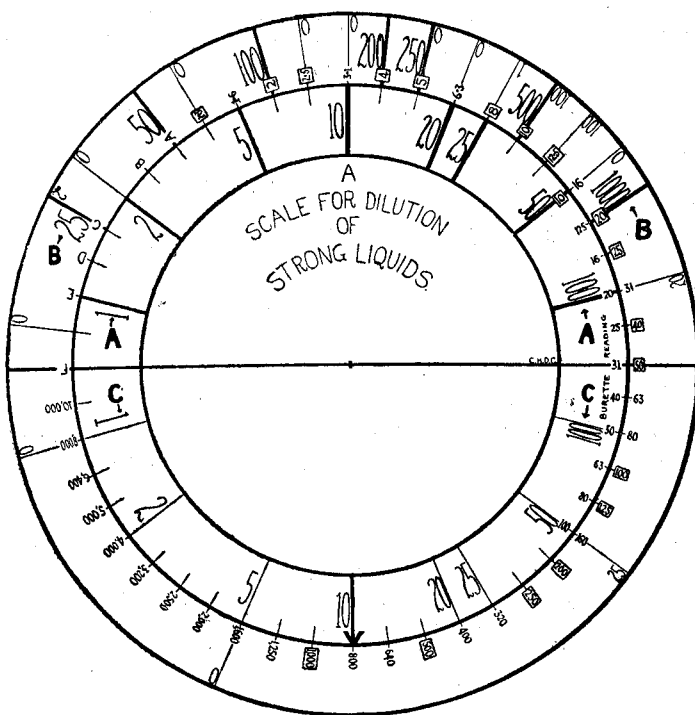
The Scale can be cut out as it stands and pasted on to cardboard, arranged so that the inner and outer scales can rotate independently about the centre, sliding on their common circle. Or, it can be constructed by copying the diagram carefully, after dividing a circle into 48 equal segments, and made to rotate as above. Scales A and C are here both on the inner circle, and scale B on the outer as shown. Scale F has been replaced by a new scale on the inner edge of the outer circle. Scale G, as formerly described, has been placed on the outer edge of

the inner circle, and is seen on the right hand side of the diagram. In the following description, these scales will, for convenience, be referred to as the "outer" and "inner" scales respectively. The arrow has been placed at the 10 division on scale C. Scale D is omitted.

For direct use, suppose 10 c.c. are to be made up to 250 c.c., and 10 c.c. are to be taken as aliquot part, the stronger solution being approximately 50 times as strong as the weaker standard. Then, placing the 10 on scale A opposite the 250 on scale B, we read off 20 on the inner scale opposite 50 on the outer. This means that about 20 c.c. of weaker standard will be needed. If the aliquot part be 20 c.c., then read off 640 opposite the 20 on scale C, and bring the arrow on scale C to this point, then the 50 on the outer scale reads 40 on the inner, so in this case 40 c.c. will be required, and similarly for all other cases. In this way, a cursor need not be attached.

For indirect use, suppose that the strong solution be about 10 times as strong as the standard, and that a burette reading of 25 c.c. is convenient. To find a suitable dilution, place the 10 on the outer scale opposite the 25 on the inner; then examining coincidences on scales A and B, we see that if we make up 25 c.c. to 100 c.c., or 50 c.c. to 200 c.c., and take 10 c.c., we shall achieve our purpose. Or, noting that 200 on the outer scale comes opposite to the arrow, we can bring another value, say, the 20 on scale C, to this point; then we see that, making 25 c.c. up to 200 c.c., and taking out 20 c.c. as aliquot part will attain the same result, and so on. The letters on the outer scale serve the same purpose as the numbers, as far as the use of the arrow is concerned. The principle involved is the same as that already explained in Parts I. and II., and reference should be made to those descriptions, if required. When using the scale indirectly, the ratios 1·3 and 1·6 on the outer scale must not be set with the 80 and 100 respectively on the inner scale.

The other marks on the scale are for convenience. The 10 on the inner scale has been placed in a frame, because the reading on the outer scale opposite to it gives the number of times the solution is being diluted, as previously given by a separate scale E. Only when the frame at the 10 comes opposite to a frame on the outer circle do any suitable numbers on scales A and B coincide, and noting this may save a little time. The numbers on the extreme outside edge are only to be read if that division comes opposite the arrow on scale C when using the instrument indirectly. In these cases, a zero mark indicates an impossible case, for no coincidences on A and B can be obtained with any values on scale C. If a number is placed there, then this indicates that only this one value on scale C will give any coincidence on scales A and B.

It is hoped that this further modification will have brought the scale into small and convenient compass. My best thanks are due to Mr. E. R. Bolton for the suggestion that I should attempt to construct the scales in a circular form.

Note.

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

AMYL ALCOHOL FOR MILK TESTING.

IN the "List of Reagents for Analytical Purposes," issued under the joint auspices of the Institute of Chemistry and the Society of Public Analysts and other Analytical Chemists, there appears under the heading "Amyl Alcohol" the following test:

Oily Impurity.—Two c.c. with 10 c.c. of water and 10 c.c. of sulphuric acid should not show any oily layer after centrifuging in a graduated Gerber milk tube for several minutes.

If the reagents are mixed together in the order mentioned it frequently happens that perfectly satisfactory samples of amyl alcohol possessing correct physical characteristics give anything from traces to very large amounts of an insoluble "oily layer" after centrifuging.

This is caused by reaction between the concentrated sulphuric acid and the amyl alcohol, and can be entirely avoided by mixing together the acid and the water, and, after cooling somewhat, adding the amyl alcohol. The latter then dissolves completely and forms a bright solution, which shows no oily globules whatever after centrifuging.

The interference or risk caused by following Gerber's original order of mixing was investigated by Richmond, who advised Gerber's "alternative" order of mixing when making milk tests (ANALYST, 1899, **24**, 146).

Literal adherence to the test as given in the "List of Reagents" gives misleading results.

THOMAS MORSON & SON, LTD.,   T. F. HARVEY.
    PONDER'S END.   CECIL O. HARVEY.

Notes from the Reports of Public Analysts.

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

KENT.

REPORT OF THE COUNTY ANALYST FOR THE QUARTER ENDED DECEMBER 31, 1922.

THE total number of samples of food and drugs examined during the quarter was 828, of which 10 were found to be adulterated. The decrease in the rate of adulteration is to be attributed to improvement in the milk. Of the 284 samples of milk examined seven were adulterated.

BUTTER.—During the quarter 84 samples of butter were examined, and all found to be genuine. Of these samples 30 per cent. contained a boric acid preservative, the amount varying from 0·14 to 0·31 per cent.

LIQUID EGGS.—Three samples contained 0·49, 0·66 and 0·69 per cent. of boric acid respectively. It is suggested that 0·5 per cent. should be the limit permitted.

CAKES.—Seven samples of sponge cake were all free from boric acid. Five ordinary cakes, however, contained the following amounts of that preservative: Plain cake, 0·02, 0·08, 0·015, 0·02, and 0·015 per cent.

COCOA.—For the last six months most of the samples taken for the county have been specially tested for arsenic, but in no case has any material quantity been found.

In some countries a limit is fixed for the amount of potassium carbonate which may be present in a prepared cocoa, but there is no standard enforced in England.

F. W. F. ARNAUD.

Legal Notes.

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

POLLUTION OF RIVER WITH GAS WORKS EFFLUENT.

Marquess of Granby v. Bakewell Urban District Council.

IN this case, heard in the Chancery Division, March 23, 1923, the plaintiff claimed an injunction to restrain the defendants from discharging into the River Wye any gas tar or noxious product so as to kill or injure the fish in the river. He also claimed £500 damages for the injury done.

The defendants by their defence admitted that on May 17, 1922, refuse matter from their gas works had found its way into the Wye, but they denied that the amount was sufficient to kill fish, or that it did kill fish. They stated that the drain pipe had carried the surface water from the gas works into the river for 20 years, and that no complaint had ever before been made of injury to the fish.

Mr. Justice P. O. Lawrence, in his judgment, stated that after the incident on May 17, 1922, the defendants constructed a concrete tank through which the effluent passed before being discharged into the river. Although this filter tank would probably intercept any solids which might be contained in the effluent, it would have little or no effect on organic matter passing through it, and it was useless to prevent ammoniacal liquor from escaping from the gas works into the river. In his judgment it had been proved beyond reasonable doubt that the fish killed in May were killed by the effluent; and that on October 25, 1922, a much stronger dose of ammoniacal liquor than usual was discharged into the river, and that this caused the death of the fish on that occasion. He warned the defendants that so long as they continued to discharge into the river an effluent which was highly poisonous to fish they were adopting a dangerous line of action, and they must not expect any leniency if the effluent should again cause death or injury to the fish in the river.

There would be judgment for the plaintiff for an injunction and for £150 damages and costs.

FAULTY DISPENSING.

On March 28, John McIntosh was summoned at the instance of the Bermondsey Borough Council for selling a compounded drug not of the nature, quality and substance demanded. Evidence was given that he had made up for the Inspector a panel patient's prescription, and that the analyst's certificate showed that, instead of 180 grains of potas. iodide to the 12 fl. ounces, there were only 42 grains, or $3\frac{1}{2}$ grains to the dose instead of 15 grains.

Dr. King Brown, M.O.H., said that in all there were five ingredients in the prescription. Presumably the others were in correct proportion as the analyst made no reference to them. Medicine deficient of one drug, like this, would not have the effect the doctor intended.

Mr. Kirby, in defence, urged that the certificate in its present form was not sufficient evidence that the medicine was not in accordance with the prescription. The certificate should have shown the proportion of each drug. There were three courses open to a Public Analyst. He could simply certify the sample as genuine, give the actual components, or the percentage of foreign ingredients. In this case he had adopted the second course with a variation. In the certificate itself he had struck out the words referring to the other two courses. But instead of giving the actual components, he says: "The sample is deficient in pot. iod." He also might have gone on to compare, in percentages, what was in the sample with what was in the prescription, showing that only one ingredient in five was inaccurate. He had not done so, but had adjudicated upon the sample instead of giving evidence. The medicine was made up by an assistant, whose only explanation is that he must have taken a stock solution of pot. iod. (1 in 4) instead of the solid drug, which would about account for the deficiency.

The magistrate (Mr. Fry) said that the point was a good one; the ingredients must be stated in the analyst's certificate. He thought he could assume that the deficiency was due to an unintentional mistake, and should dismiss the summons on payment of two guineas costs.

LEAD AND ARSENIC FROM GLASS.

On April 5th Boots Cash Chemists (Western), Ltd., were summoned by the Birmingham Corporation for the sale of potassium carbonate containing about 10 parts of arsenic and about 80 parts of lead per million.

Mr. Liverseege, the City Analyst, said that the substance sold was impure commercial "salt of tartar" instead of the pure B.P. product. The limits for lead and arsenic incorporated in the B.P. had not been challenged as too stringent. The price of the pure drug was charged for the impure article, and therefore the sale was to the prejudice of the purchaser. It was improbable that the arsenic and lead would be absorbed from the glass, as the potassium carbonate was dry.

Mr. Bramall, solicitor for the defence, said that the potassium carbonate was perfectly pure when it left Nottingham. The only conclusion was that the lead and arsenic were derived from the bottle—the ordinary kind of glass container used in pharmacists' shops, and experiments had been made to determine the point. The bottle in question had been broken up, and lead and arsenic found in the parts. As a consequence of that discovery, every similar bottle had been withdrawn from the firm's 600 shops, and new bottles of clouded glass, which contained neither lead nor arsenic, substituted. It would have to be proved, before there could be a conviction, that the sale of the potassium carbonate was effected to the prejudice of the purchaser. In this case there was no such prejudice.

Mr. H. Droop Richmond said that he had believed that an apparently dry substance could not absorb lead or arsenic from glass, but, as a result of finding that Mr. Liverseege's certificate was correct, he had investigated the matter, and found that potassium carbonate, which was a hygroscopic substance, had derived lead and arsenic from the glass. On putting potassium carbonate in a bottle similar to the one in question he had found at the end of a fortnight that the lead had increased from 5 to 15 parts per million.

The Chairman of the Magistrates dismissed the case.

Food Preservatives.

IN reply to a questionnaire sent by Dr. R. Dudfield, Medical Officer of Health for Paddington, to Medical Officers of Health in different parts of the country, replies from 109 areas have been received. The information has been summarised in a report to the Paddington Public Health Committee, and the following table shows: (1) The widespread use of these preservatives; (2) their varied character; and (3) the extraordinary ranges in the quantities added by different manufacturers:

Commodity	Preservative	Quantities of Preservatives	
		Max. Gr. per lb.	Min. Gr. per lb.
Bacon	Boric acid	35·0	2·0
Dried fruit	Sulphur dioxide	15·2	1·4
Jam	Salicylic acid	10·0	0·07
Margarine	Boric acid	175·0	trace
Fish pastes	Boric acid	105·0	trace
Vegetables	Copper sulphate	10·2	trace
Lemon squash	Salicylic acid	13·1	0·06

"Chemist" and "Pharmacist."

THE President of the Institute of Chemistry, Mr. A. Chaston Chapman, F.R.S., has sent the following letter to the Press :

17th March, 1923.

DEAR SIR,

THE TITLE "CHEMIST."

The Retail Pharmacists' Union, in their recent announcement with regard to the subject of the accurate dispensing of medicines, have, I think it will be generally agreed, made a good statement on behalf of the profession of pharmacy.

I would, however, venture to place before your readers a matter, arising from the above, which is one of no small concern to those chemists who are engaged in the practice of CHEMISTRY as distinct from PHARMACY—that is to say—who are concerned with the science of chemistry and its applications to the arts and manufactures, and have nothing to do with the dispensing of medicines.

The Retail Pharmacists' Union have headed their announcement with the words "THE CHEMIST," and I would like to make, in the public press the suggestion which has so often been made in the scientific press, that the time has come for the pharmacist to relinquish the use of the term "chemist" in favour of those who definitely practise chemistry.

The war was instrumental in directing the attention of the public more closely to the existence of the profession of chemistry, the profession on which the country for nd it necessary to rely for the production of explosives and materials of war generally, as well as for the means of protecting our troops against the methods of chemical warfare initiated by the enemy.

During the past half century, the science of chemistry has developed by leaps and bounds, and its practice as a profession has become more and more firmly established. In this country the science is now taught in 1000 or more public and secondary schools, and in nearly 300 technical schools, while our University Departments of Chemistry are renowned throughout the world.

The Institute of Chemistry, as the representative Chartered professional body of chemists, numbers upwards of 4000 Fellows and Associates, whose qualification demands a four years University course, or the equivalent, and the majority of whom are engaged in the many branches of industry on which the science has a bearing.

In other countries, the strict equivalent of the word "chemist" signifies, as it should, one who professes chemistry, and not in any case the pharmacist, druggist, or dispenser of medicines. How the misapplication of the word has arisen in this country is a matter which I need not here pursue. The enormous importance of chemistry is becoming daily more widely recognised, and it is highly desirable, in order to avoid confusion, that the word "chemist" should be confined to those who practise chemistry in the full sense of the word. It is bad enough when mental confusion is the unfortunate consequence of the poverty of a language, but in this instance the correct and distinctive words are ready to our hand.

I would like to express the hope which I have already expressed on a good many occasions, that our friends the Pharmacists—notwithstanding the provision of the Pharmacy Act—will lose no opportunity of referring to their ancient and important calling by the word which more accurately defines and describes it. In addition, I would appeal to the press, which is so important a factor in the enlightenment of the general public, to assist so far as they can by employing the terms "Chemist" and "Pharmacist" respectively in their obviously correct significations, so that those practising chemistry may be described as Chemists, and those practising pharmacy as Pharmacists.—Yours faithfully,

A. CHASTON CHAPMAN.

With reference to this letter, the President of the Society of Chemica Industry, Dr. E. F. Armstrong, F.R.S., has written as follows :

SIR,

THE TITLE "CHEMIST."

It would appear desirable to support the plea recently so admirably stated by Mr. Chaston Chapman, the President of the Institute of Chemistry, in your columns for the restriction of the word " chemist " to those who practise chemistry in the full sense of the word. Surely the time has arrived when we can come to an amicable agreement with our friends the pharmacists on this point, and I

venture to suggest that much would be gained if it were possible to hold a round table conference officially representative on the one side of the Pharmaceutical Society, the Retail Pharmacists' Union, and any other bodies concerned, and on the other side, of the leading chemical societies.

The object of such a conference would be to discuss the possibility of agreeing as a point of professional etiquette to the limitation of the terms "pharmacist" and "chemist," in the sense defined by Mr. Chapman.

If this could be achieved, the rest would follow, and, with the support of the Press, the public would soon learn the proper meaning of the terms.

Yours etc.,

E. FRANKLAND ARMSTRONG.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Relation of Flour Strength to the Diastatic Enzymes Present. L. A. **Rumsey.** (*Bull. Amer. Inst. Baking*, 1922, No. 8.)—The author has investigated the effects of concentration, time, temperature, acidity and diastatic power upon the action of diastase in panary fermentation with 14 varieties of wheat flour. Emphasis is placed upon the necessity of conducting experiments under the conditions which exist in the usual panary fermentation, and not in aqueous extracts or with gelatinised starch, since results obtained under the latter conditions are abnormal. The general procedure adopted was to allow diastatic action to proceed in a 10 per cent. suspension of the flour at 27° C. for 1 hour. After dilution the liquid was neutralised to thymol blue, and the proteins were precipitated by the addition of 3 c.c. of 15 per cent. sodium tungstate solution and 0·4 c.c. of concentrated sulphuric acid, this addition serving to stop diastatic action and prevent further change in the sugars present. The liquid was then diluted to 200 c.c. and centrifuged for 5 minutes, after which the sugars present in the clear liquid were estimated by the Munson-Walker official method. The use of sodium hydroxide for inhibiting diastatic action is unsatisfactory, since the concentration necessary is liable to decompose the reducing sugars present. The maximum activity of diastase in dough is not exerted immediately on mixing owing to insufficient water being present, but this is compensated for by the retarded loss of activity of the enzyme, and the rate of formation of maltose is constant for any definite temperature. The optimum P_H value for diastase is 4·7, but the usual value for dough lies between 5 and 6, and to this is attributed the increasing formation of maltose towards the end of fermentation. The chief factor in the control of diastatic action in dough is temperature, and increase in this factor indirectly induces completion of aeration during proofing of the loaves. The optimum temperature for the action of diastase is 63·5° C., but this is maintained for a few minutes only during baking. Flour

showing the higher diastatic power should show the greater strength and baking value, providing that the quality and amount of gluten present is the same. Comparative experiments made on the estimation of diastatic power of flour indicate the necessity for making use of a standard starch substrate. The paper is provided with numerous curves and tables showing the results obtained, and a brief review is given of previous work on the subject, together with an extensive bibliography.   T. J. W.

Action of Malt and Malt Extract upon the Strength of Flour. F. A. Collatz. (*Bull. Amer. Inst. Baking,* 1922, No. 9.)—Seven varieties of flour derived from wheat grown under different climatic conditions, and the influence of malt upon the various factors affecting the quality of bread, were investigated. The diastatic power of flours, malt and doughs was estimated by the method described in the previous abstract. Proteolytic activity was shown to be proportional to the viscosity of the solution obtained by digesting flour, equivalent to 18 grms. of the dry material, in 100 c.c. of water at 40° C. for 4 hours, cooling the mixture and determining the viscosity after the addition of 5 c.c. of N lactic acid. To estimate the buffer value 80 grms. of flour were shaken with 400 c.c. of water, digested at 25° C. for 1 hour and centrifuged. To 25 c.c. portions of the clear liquid varying proportions of 0·02 N hydrochloric acid or sodium hydroxide were added, and the P_H value of the solutions was determined electrometrically. The optimum conditions found for the diastatic activity of malt flour were P_H 4·26 and a temperature of 65° C., the activity being constant during a period of 8 hours at 27° C. The maximum increase of diastatic activity is produced by addition of the first 10 per cent. of malt flour to dough; further additions yield higher values, but in diminished proportion. The starch of strong flours appears to be more readily hydrolysed by malt diastase than that of weaker flours. The viscosity of suspensions of strong flours is higher than that of corresponding suspensions of weak flours, either with or without the addition of malt flour. Gas formation is unaffected by the addition of malt extract to strong flours, but a similar addition to weak flours greatly augments the volume of carbon dioxide produced. During fermentation of dough the P_H value steadily increases, and in nearly all the experiments this value was found to be approximately 5·0 when the dough was ready for baking. The volume of bread is in all cases increased by the addition of malt flour or malt extract to the dough, and a loaf superior in grain and texture, indicating increased baking strength, is obtained by this addition. A summary of the results obtained by previous workers and an extensive bibliography are included in the original paper.   T. J. W.

Zamia Starch. T. E. Wallis. (*Pharm. J.,* 1923, **110**, 235–238.)—The author has confirmed the conclusions of Clevenger (ANALYST, 1922, **47**, 170), but finds that some grains measure 75μ in length, thus exceeding the maximum of 40μ given in the above paper. The grains are very variable in form, and commercial samples often show fissures. One feature of this starch not previously described is the presence of characteristic spherical cluster crystals of calcium

oxalate mostly about 35μ in diameter. These may be readily separated by shaking the starch with chloroform, when the crystals, together with a little sand, fall to the bottom of the vessel. Starches from other cycads also contain such crystals, but those derived from *Cycas neocalidonica* Lind. are mostly prismatic and twins mixed with a few clusters. It appears unlikely that *Zamia floridana* A.D.C. is identical with *Z. integrifolia* Ait.   T. J. W.

Estimation of Small Amounts of Starch in Plant Tissues.   F. E. Denny. (*J. Assoc. Off. Agric. Chem.*, 1922, **6**, 175–191.)—The following methods are developments of those published by Von Fellenberg (ANALYST, 1917, **42**, 279) and Long (*T. Kan. Acad. Sci.*, 1916–17, **28**, 172). The plant tissue is ground to pass a 30-mesh sieve and is then boiled with concentrated calcium chloride solution. After filtration, the starch in solution is precipitated by the addition of iodine solution under standard conditions, and the estimation is carried out by (*a*) titration with standard sodium thiosulphate solution of the iodine contained in the starch iodide; (*b*) a similar titration of the iodine remaining in the solution; or (*c*) hydrolysis of the starch iodide by hydrochloric acid and subsequent estimation of the sugars formed by Fehling solution or by Scales' iodine method (ANALYST, 1919, **44**, 349). The method adopted was (*a*), and comparisons were made with known amounts of starch under the same conditions. Duplicate estimations showed results varying within 10 per cent. of the amount of starch present. Detailed descriptions of the methods used are given, and the suggestion is made that these are suitable for tissues containing small amounts of starch only.

T. J. W.

Precipitation of Proteins from Cereal Extracts by means of Sodium Tungstate.   L. A. Rumsey. (*J. Ind. Eng. Chem.*, 1923, **15**, 270–272.)—Sodium tungstate solution is recommended for the clarification of cereal extracts previous to the estimation of reducing sugars therein. The rapid and complete flocculation of the precipitate formed depends on the final hydrogen ion concentration of the liquid; this may be adjusted in the following way. The flour suspension or extract, containing 10 grms. of material per 100 c.c., is diluted to about 175 c.c., a few drops of thymol-blue solution are added, and 3 c.c. of 15 per cent. sodium tungstate solution introduced. The mixture is shaken thoroughly and concentrated sulphuric acid is added drop by drop from a micro-pipette until the colour of the indicator changes to pink. Three further drops of the acid are then added, and the mixture diluted to 200 c.c., shaken, and submitted to centrifugal action. Excess of sodium tungstate remaining in the solution does not interfere with the estimation of the sugars by Fehling's method.   W. P. S.

Estimation of Reducing Sugars by means of Fehling Solution with Methylene Blue as Internal Indicator.   J. H. Lane and L. Eynon. (*J. Soc. Chem. Ind.*, 1923, **42**, 32–37T.)—The following volumetric method yields results equally as accurate as those given by the usual gravimetric Fehling estimation, and is unaffected by the presence of starch, proteins, suspended matter and substances retaining cuprous oxide in solution. The solutions employed are the

usual Soxhlet's modification of Fehling solution and a 1 per cent. solution of methylene blue as indicator. Maximum accuracy is attained by adhering to a consistent procedure, when the end-point may be determined to within 1 drop of sugar solution. It is advisable in all cases to add the indicator solution only toward the end of the titration, since the approach to the end-point is masked if the indicator is added at an earlier stage. A preliminary titration is carried out by adding 15 c.c. of sugar solution to 10 or 25 c.c. of mixed Fehling solution and boiling the mixture for 15 seconds, when further 10 c.c. portions or less of the sugar solution are added, the liquid being boiled for the above period between successive additions. When most of the copper is reduced a few drops of the indicator are run in and the sugar solution is added in small increments, while the liquid is maintained in constant ebullition until the methylene blue is decolorised. This estimation is followed by another, carried out according to the standard method adopted by the authors: Ten or 25 c.c. of Fehling solution are put into a flask, and the sugar solution is added to (if possible) within 1 c.c. of the amount required to complete the titration, and the mixture is boiled for 2 minutes. Three to 5 drops of the indicator are then added, and the titration is completed in one minute, the liquid being maintained in continuous ebullition for the whole three minutes. Burettes fitted with glass taps are unsuitable for use with this method, as the heat of the steam is liable to cause jamming. The following abridged tables were constructed for use with this method by employing pure samples of various sugars.

TABLE I.

Invert Sugar Table for 10 c.c. of Fehling Solution.

Solutions containing besides invert sugar

C.c. of sugar solution required	No sucrose		1 per cent. sucrose		5 per cent. sucrose		25 per cent. sucrose	
	Invert sugar factor	Mg. Invert per 100 c.c.	Invert sugar factor	Mg. Invert per 100 c.c.	Invert sugar factor	Mg. Invert per 100 c.c.	Invert sugar factor	Mg. invert per 100 c.c.
15	50·5	336	49·9	333	47·6	317	43·4	289
20	50·9	254·5	50·2	251	47·6	238	43·2	216
30	51·5	171·7	50·5	168·3	47·7	159	42·5	142
40	52·0	130·1	50·8	127	47·7	119·2	41·8	104
50	52·5	105·1	51·0	102	47·7	95·4	41·0	82

TABLE II.

Invert Sugar Table for 25 c.c. of Fehling Solution.

Solutions containing besides invert sugar

C.c. of sugar solution required	No sucrose		1 per cent. sucrose	
	Invert sugar factor	Mg. Invert per 100 c.c.	Invert sugar factor	Mg. Invert per 100 c.c.
15	123·6	824	122·6	817
20	123·8	619	122·8	614
30	124·3	414·3	123·1	410·4
40	124·8	311·9	123·4	308·6
50	125·3	250·6	123·8	247·6

TABLE III.

Dextrose (anhydrous) Table.

C.c. of sugar solution required	For 10 c.c. of Fehling solution		For 25 c.c. of Fehling solution	
	Dextrose factor	Mg. dextrose per 100 c.c.	Dextrose factor	Mg. dextrose per 100 c.c.
15	49·1	327	120·2	801
20	49·5	247·4	120·3	601·5
30	50·1	167	120·8	402·7
40	50·6	126·5	121·2	303·1
50	51·1	102·2	121·8	243·6

TABLE IV.

Lævulose (anhydrous) Table.

C.c. of sugar solution required	For 10 c.c. of Fehling solution		For 25 c.c. of Fehling solution	
	Lævulose factor	Mg. lævulose per 100 c.c.	Lævulose factor	Mg. lævulose per 100 c.c.
15	52·2	348	127·4	849
20	52·5	262·5	127·6	638
30	53·2	177·2	128·1	427
40	53·6	134	128·6	321·5
50	54·0	108	129	258

TABLE V.

Maltose (anhydrous) Table.

C.c. of sugar solution required	For 10 c.c. of Fehling solution		For 25 c.c. of Fehling solution	
	Factor	Mg. per 100 c.c.	Factor	Mg. per 100 c.c.
15	77·2	515	197·8	1319
20	76·8	383·8	196·2	980·7
30	76	253·3	193	643·4
40	75·4	188·6	190·5	476·2
50	75	150·1	188·7	377·3

TABLE VI.

Lactose (anhydrous) Table.

C.c. of sugar solution required	For 10 c.c. of Fehling solution		For 25 c.c. of Fehling solution	
	Factor	Mg. per 100 c.c.	Factor	Mg. per 100 c.c.
15	64·9	432	163·9	1093
20	64·6	323	162·3	811·8
30	64·4	214·7	160·6	535·1
40	64·5	161·2	159·4	398·5
50	64·9	129·8	158·7	317·5

The factor given in the above tables is the number of mgrms. of the sugar corresponding to the volume of Fehling solution used. By employing the preliminary method of titration only, results may be obtained with an error not exceeding 1 per cent. of the amount of sugar estimated, but, in order to eliminate the personal factor, which is of importance in such incremental methods, it is advisable for the operator to compile his own tables to suit his own method of working. This modification is unnecessary when the standard method is adopted. Full details are given of sources of error and the precautions necessary to avoid them, and the tables furnished are worked out to the nearest c.c. of sugar solution used, ranging from 15 to 50 c.c.   T. J. W.

Estimation of Caramel in Sugar Products. A Criticism of the Ehrlich Method. G. P. Meade. (*J. Ind. Eng. Chem.*, 1923, **15**, 275.)—The Ehrlich method is based on the non-precipitation of saccharan, one of the constituents of caramel, by basic lead acetate, and on the fact that the colouring matter remaining in any lead-clarified filtrate is due to saccharan which can be estimated colorimetrically by comparison with standard saccharan solution. The author finds that when a sugar solution contains any material which forms a precipitate with lead acetate, the precipitate removes part or all of the caramel (saccharan) from solution. The method is, therefore, useless in the case of molasses, etc., which contain impurities yielding voluminous precipitates with either lead acetate or basic lead acetate.   W. P. S.

Hydrolysis of Pectin. F. Tutin. (*Biochem. J.*, 1923, **17**, 83.)—In a former paper (ANALYST, 1921, **46**, 505) the author showed that pectin when treated with cold aqueous alkalis or with pectose yielded, among other products, methyl alcohol and acetone, and deduced that this compound is the dimethyl *iso*-propenyl ester of pectic acid. This conclusion has been criticised on the ground that whether the methyl alcohol yielded by pectin is estimated from the amount of alkali required for hydrolysis or by Zeisel's method, identical results are obtained, and that, therefore, there is hardly room for an *iso*-propenyl group in the molecule. Attention is drawn to the facts that the amount of alkali required to eliminate an *iso*-propenyl group is the same as that required for a methyl group, and that by Zeisel's method acetone (or an *iso*-propenyl group) yields the same weight of silver iodide as would a methyl group.   T. J. W.

Composition of Mayonnaise. A. Behre, A. During, H. Ehrecke, and K. Thimme. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1922, **44**, 333–336.)—Results of the analyses of 10 samples of mayonnaise (as sold in Germany) are recorded; in each case, the meat constituents were removed as far as possible before the analysis was made. The samples contained: Water, 14 to 65 per cent.; acidity (as acetic acid), 0·16 to 0·37 per cent.; proteins, 1·19 to 2·80 per cent.; oil, 26 to 83 per cent., wheat flour, none to 9·4 per cent. As a rule the sample containing most flour also contained most water; the oil was generally rape oil, linseed oil or poppy oil, olive oil being used in a few of the products.   W. P. S.

Reaction of Crude Vegetable Oils with Copper Acetate.   W. H. Dickhart. (*Amer. J. Pharm.*, 1923, **95**, 108–109.)—On shaking a petroleum spirit solution of the crude vegetable oils containing 10 grms. of the oil in 150 c.c. of solvent with 40 c.c. of a freshly prepared 5 per cent. aqueous solution of copper acetate, a green colour is produced in the oil solution in the case of the majority of oils. Crude cottonseed oil does not show this reaction, and refined oils also give no coloration.   The author has applied this reaction to the detection of foreign oils in crude cottonseed oil, a distinct coloration being produced by the presence of 0·5 per cent. of the adulterant.   The green colour appears to be colloidal in character, since it readily passes through an ordinary filter paper, but is retained by parchment.   T. J. W.

Methods for the Examination of Cacao Butter.   R. E. Doolittle. (*J. Assoc. Off. Agric. Chem.*, 1923, **6**, 278–279.)—The following tests recommended by the Committee were approved as tentative methods at the Annual Convention of the Association, November, 1922:

I. CRITICAL TEMPERATURE OF SOLUTION IN ACETIC ACID.—*Reagents.*— (*a*) Glacial acetic acid, as free as possible from water; (*b*) 0·1 *N* potassium hydroxide solution.

Apparatus.—Insert a thermometer reading to 0·1° C. into a cork that fits a $6 \times \frac{3}{4}$ inch test tube.   The thermometer should extend so far into the tube that the bulb will be covered by 10 c.c. of liquid.   Place the test tube in a larger tube ($4 \times 1\frac{1}{4}$ inch), containing glycerin, and hold firmly in place with a cork having a groove cut in the side to equalise the pressure when heat is applied.

Determination.—Filter a portion of the sample to be examined through a dry filter paper in an oven where a temperature of about 110° C. is maintained, to remove traces of moisture.   Allow the filtered sample to cool until barely warm, and weigh 5 grms. of the sample and 5 grms., of the acetic acid reagent (*a*) into the test tube.   Insert the cork holding the thermometer and place the test tube in the glycerin bath.   Heat and shake the apparatus frequently until a clear solution of the fat and acetic acid is obtained.   Allow the solution to cool, with constant shaking, without removing from the glycerin bath.   Note the temperature at which the first sign of turbidity appears.   Make a similar test with the same acetic acid on a sample of pure cacao butter.   Free fatty acids lower the turbidity temperature.   A correction must therefore be made for the acid value of the sample.

Correction Factor.—If the strength of the acetic acid reagent is such that the turbidity temperature of the pure cacao butter is, approximately, 90° C. one unit of acid value will cause a reduction of 1·4 degrees in the critical temperature of solution.   If the turbidity temperature is approximately 100° C., one unit of acid value will cause a reduction of 1·2 degrees.   For intermediate temperatures the reduction is proportional.

Determine the acid value (mgrms. of potassium hydroxide required to neutralise the free fatty acids in 1 grm. of the sample) of both the sample and the pure cacao

butter. Multiply the acid value by the correction factor and add the result to the observed turbidity temperature. The figure' obtained is the true critical temperature of solution. If the true critical temperature of dissolution of the sample is lower by more than 2 degrees than that of the pure cacao butter, adulteration with coconut, palm kernel, cottonseed oils or stearines, maize oil, arachis oil or other vegetable oils is indicated.

II. ACETONE AND CARBON TETRACHLORIDE TEST.—*Reagent.*—A mixture of equal parts of acetone and carbon tetrachloride.

Determination.—Dissolve 5 c.c. of the warm fat, which has been previously filtered through dry filter paper in an oven at about 110° C. to remove traces of moisture, in 5 c.c. of the acetone and carbon tetrachloride reagent in a test tube. Allow the solution to stand in ice water for 20–30 minutes. Run a blank on a sample of pure cacao butter at the same time. If hydrogenated oil, tallow, oleostearine or paraffin wax is present, a white flocculent precipitate will soon appear. If the water is cold enough, cacao butter may solidify. If a precipitate is formed, remove the sample from the ice water and allow it to remain at room temperature for a time. Solidified cacao butter will soon melt and go into solution, but, if the precipitate is due to any of the above-mentioned possible adulterants, a much longer time will be required for it to go into solution.

Limeseed Oil and Meal. (*Bull. Imp. Inst.*, 1922, **4,** 465–468.)—Limeseeds from the Leeward Islands contained 39·8 per cent. of a light brown liquid oil, which, after filtration, gave the following figures:—Specific gravity at 15/15° C., 0·9236; n_D^{40}, 1·4635; solidifying point of fatty acids, 34·9° C.; acid value, 13·6; saponification value, 197·7; iodine value (Hubl 17 hours), 109·5; unsaponifiable matter, 0·4 per cent.; c.c. of potassium hydroxide required to neutralise the volatile soluble acids from 5 grms. of oil, 0·27; and to neutralise the insoluble acids, 0·48. The bitter taste of the oil was removed on refining with soda, but the oil was not found very suitable for refining for edible purposes. It would, however, serve for soap making. The residue left after expression of the oil had high manurial and feeding values, and cattle soon get accustomed to the slightly bitter taste. The whole crushed seed contained: Nitrogen, 3·43; potash (K_2O), 0·48; and phosphoric anhydride, 0·74 per cent. The figures obtained for the pressed residue were:— Moisture, 15·08; crude protein, 30·5 (true protein, 20·7); fat, 14·2; carbohydrates, 17·0; crude fibre, 20·05; and ash, 3·17 per cent.   D. G. H.

Effect of Babassu Fodder on Butter and Lard. N. Bengtsson. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1922, **44,** 336–339.)—When cows were fed for some weeks on babassu cake, prepared from the seeds of the S. American palm, *Attalea funifera (Orbignia speciosa)* in quantity up to 1 kilo. per animal per day, the Polenske value of the butter fat obtained from the milk of the animals rose to **3·3,** and there was a slight increase in the iodine value and a decrease in the melting point of the fat. In the case of pigs fed with babassu cake, the iodine value of the lard decreased by several units, the melting point was raised by 1° or 2° C., and the refractometer value lowered slightly.   W. P. S.

The Sulphuric Acid Test for Liver Oils. A. Harden and R. Robison. (*Biochem. J.*, 1923, **17**, 115–116.)—The general resemblance of this test to the Mylius-Pettenkofer reaction for bile acids suggested to the authors that the former might be due to condensation between two substances under the influence of sulphuric acid. A purple colour resembling that given by this reaction may be obtained by the addition of sulphuric acid to a petroleum spirit or chloroform solution of cholesterol containing furfuraldehyde or ω-hydroxy-methylfurfuralde- hyde. Also, the addition of furfuraldehyde to oils yielding only a faint coloration gives rise to an intense purple colour on the addition of sulphuric acid. Attempts were made to obtain furfuraldehyde or a substance capable of replacing it in this reaction from coal fish oil, but without success. The reaction is a delicate test for cholesterol, a faint purple colour being produced by 0·1 mgrm. in 5 c.c. of petroleum spirit (*cf.* following abstract). T. J. W.

New Reactions of Sterols. G. S. Whitby. (*Biochem. J.*, 1923, **17**, 5–12.)— (A) From 1 to 2 mgrm. of the sterol is dissolved in 2 c.c. of chloroform, and the solution is shaken with a similar volume of a mixture containing 50 volumes of concentrated sulphuric acid and 1 volume of formalin. After separation of the liquids, on standing, the lower sulphuric acid layer is brownish-red in colour, with an intense green fluorescence, whilst the upper chloroform layer shows a cherry-red coloration. When this layer is poured into a dry test tube, and treated with two drops of acetic anhydride, a bright blue colour is developed, which persists for a considerable time, eventually turning green. Amyrin yields no colour in the chloroform layer with this reagent, and abietic acid gives nothing characteristic, whilst the sensitiveness of the reaction for sterols exceeds ten times that of the Salkowski reaction. (B) Twenty-five drops of the above sulphuric acid and formalin reagent are shaken with a solution of 0·2 to 0·5 mgrm. of the sterol in 2 c.c. of glacial acetic acid; a rose-coloured fluorescent solution is produced which slowly changes to yellowish-brown. Although this reaction is twice as sensitive as the Liebermann-Burchard reaction, it is unsuitable for quantitative estimation. Amyrin yields a result similar to that given by sterols with this reagent, but abietic acid produces a greenish-brown colour.

(C) A few mgrms. of the sterol are added to 1 drop of acetic anhydride, the mixture is gently heated until melted, and the excess of the solvent is evaporated. After cooling, the residue is moistened with concentrated nitric acid, which produces a blue or bluish-green colour. This reaction is much more readily given by choles- terol than by phytosterols, but the distinction between the two results is insufficient to discriminate between these compounds. Sterolins give most of the colour reactions of sterols, but may be distinguished by dissolving a few particles of the compound in 1 to 2 c.c. of warm concentrated sulphuric acid. After cooling, a saturated aqueous solution of thymol is poured on to the surface of the acid, and, in the presence of a sterolin, an orange colour with green fluorescence is developed in the lower layer, and a violet ring between the two layers. Sterols yield a similar colour and fluorescence, but no violet ring. The mechanism of these and

other reactions for sterols has been experimentally investigated by the author, the conclusions arrived at being that the essential features in most instances are similar, and consist in the dehydration of the sterol molecule, with formation of a colourless substance, the coupling of this with a second substance derived from the sterol or added as a reagent, and finally the use of a reagent to render the medium anhydrous.   T. J. W.

The Sterol Content of Cow's Milk. F. W. Fox and J. A. Gardner. (*Biochem. J.*, 1923, **17**, 94–102.)—An examination of numerous samples of fresh and condensed milk and butter was carried out by the following method, and the free and combined cholesterol were estimated. The fat was separated by the Rose-Gottlieb method, and, for the free cholesterol estimation, was dissolved in alcohol, and treated with an excess of 1 per cent. alcoholic digitonin solution. After standing overnight the mixture was evaporated to dryness, washed with ether and warm water, transferred to a Gooch filter, dried and weighed. Estimation of the total cholesterol was carried out by dissolving the fat in ether and boiling the solution with a large excess of sodium ethoxide in alcohol. After hydrolysis the liquid was diluted with water and extracted with ether, the ethereal solution being washed with water and evaporated to dryness, after which the residue was dissolved in alcohol and precipitated with digitonin as detailed above. The ratio of cholesterol to total fat was found to be variable—a result in agreement with the observations of previous workers. The proportion of free cholesterol to combined cholesterol was on an average 1 to 0·54, but the variations extended over a fairly wide range. Most of the free and combined cholesterol exists in milk dissolved in the fat globules, but a certain proportion is contained in aqueous solution, probably in the colloidal form or in combination with proteins. The unsaponifiable matter in milk fat contains from 33 to 50 per cent. of sterols, the remainder consisting of sticky yellow oils. No sterols other than cholesterol were detected in the fat, and no pure substances were isolated from the unsaponifiable oils not precipitated by digitonin.   T. J. W.

Feder's Value for Minced Meats. Pannwitz and Harder. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1922, **44**, 344–348.)—Analyses of 102 samples of fresh minced meats show that the ratio of water content to the sum of the organic non-fatty constituents is always less than 4 (usually 3·2 to 3·7) for unadulterated products. This ratio was first pointed out by Feder. There is but little difference between Feder's ratio and the ratio of water to proteins.   W. P. S.

Spectrophotometric Estimation of Aromatic Amino Compounds. W. E. Mathewson. (*J. Assoc. Off. Agric. Chem.*, 1922, **6**, 16–28.)—The transmissive indices (extinction coefficients) of azo dyes produced by diazotising various aromatic amines and subsequently coupling with 1-2-naphtholsulphonic acid, have been determined for wave lengths of 436, 546 and 579$\mu\mu$, a quartz mercury lamp being used as illuminant. The results obtained have been applied to the estimation of amines by the following method:—A solution containing 0·5 to

1·0 mgrm. of the base in 100 c.c. of 0·25 N hydrochloric acid at the ordinary temperature is treated with 1 c.c. of 0·5 N sodium nitrite solution, and allowed to stand 2 minutes. It is then rapidly poured into a mixture of 15 c.c. of 5 N sodium carbonate and 5 c.c. of 5 per cent. sodium α-naphtholsulphonate, the solution then being diluted to a known volume and the transmittance at 546$\mu\mu$ determined. In accurate work it is advisable to carry out a parallel estimation with known amounts of the pure amine to eliminate errors due to temperature, alkalinity and salt content of the solutions. A modification of this method which yields more accurate results consists in the addition of 4 c.c. of 3 per cent. hydrazine sulphate and stirring for 20 seconds before the sodium carbonate and α-naphtholsulphonate mixture is added. The observed transmissive indices range between 0·55 for anthranilic acid and 2·68 for naphthionic acid when a wave-length of 546$\mu\mu$ is used. A colorimetric method for the estimation of β-naphthylamine in commercial oil-soluble food dyes is based on this principle, and shows an accuracy within 10 per cent.   T. J. W.

Estimation of Sodium Cresylate. E. Insard. (*J. Pharm. Chim.*, 1923, **6**, 217.)—An exact weight of sodium cresylate (100 grms.) is placed in a 150 c.c. graduated flask and diluted with water. Two grms. of calcium chloride are added, and the solution made up to the mark. After being shaken and allowed to stand, the solution is filtered, 100 c.c. of the filtrate transferred to a decanting flask, excess of hydrochloric acid added, and the whole extracted twice with 20 c.c. of ether. To effect complete extraction of the cresol the extracted solution is treated with 10 grms. of pure sodium chloride, and after this has dissolved the liquid is again extracted twice with ether. The united ethereal extracts are washed with water, then treated with dry anhydrous sodium sulphate to remove moisture, filtered and distilled on an oil bath from a weighed flask provided with a thermometer. Almost all the ether distils at 36° C.; the temperature is then raised to 80° C. and remains at that point for some time, until the temperature suddenly rises, after which the flask is removed from the flame, cooled and weighed.
                                                                        R. F. I.

Microchemical Identification of Certain Medicinal Phenolic Esters. M. G. Deniges. (*J. Pharm. Chim.*, 1923, **27**, 196–197.)—The reactions made use of are (1) the type of crystal formed in contact with one, or sometimes two, solvents, used one after the other; (2) the colour resulting from mixing the crystals with a drop of sulphuric acid and adding a trace of formaldehyde; (3) the melting point of the crystal residues, obtained by floating a cover glass on a mercury bath and observing the temperature when the crystals on the cover glass become clear.

Benzonaphthol.—On treating a few drops with chloroform on the end of a glass rod, concentric crystalline groupings remain after evaporation of the solvent. Microscopic examination of these crystals treated with acetic acid shows an arborescent structure, often accompanied by isolated or crossed prisms. On treatment with sulphuric acid and formaldehyde the preparation becomes brown, blackening on slight heating.

Salol.—On treating as above with chloroform fine droplets remain, which, after taking up with alcohol, and evaporating the alcohol, remain as rhombic plates still mixed with oily droplets. On adding a trace of crystalline salol, micro-crystals at once form. Treatment with sulphuric acid and formaldehyde produces a red coloration, deepening on heating.

Betol behaves with chloroform like salol. Re-dissolved in acetic acid isolated rhombic plates form after a short time, nearly always mixed with acicular masses with rhombic bases. With sulphuric acid and formaldehyde the action is the same as in the case of benzonaphthol.

Cresalol. Since the melting point of this substance is low (36° C.), crystallisation can only be induced by adding a trace of the solid product. Sulphuric acid and formaldehyde give a red coloration.

Salophene. This compound has a high melting point, and crystals are formed directly. Acetone is the best solvent, and rhombic plates resembling cholesterol are obtained. A rose tint results on treatment with sulphuric acid and formaldehyde in the cold, deepening, on heating, to a brown colour.　　　D. G. H.

Influence of Papaverine on the Optical Activity of Narcotine in Acid Solution. H. E. Annett. (*J. Chem. Soc.*, 1923, **123**, **124**, 376–379.)—Since narcotine in acid solution is dextrorotatory, whilst the free base dissolved in chloroform, toluene or alcohol, is lævorotatory, and papaverine is optically inactive, a polarimetric method for estimating narcotine in admixture with papaverine would appear possible, and preliminary work with this end in view established the following results:—(1) Papaverine is optically inactive both in acid solution and in an inactive solvent. (2) The specific rotation of narcotine in various solvents for the sodium flame, and for the yellow and green lines of the mercury arc, were determined and the figures are recorded. (3) On adding papaverine to a solution of narcotine in 1 per cent. sulphuric acid, the optical activity of the solution was found to decrease until the ratio of papaverine to narcotine was 3:2, whilst an increase in ratio to 4:2 had no appreciable effect. On diluting a 2 per cent. solution of narcotine to 0·4 per cent. the effect was much less noticeable. (4) Although in a solution of hydrochloric acid the optical activity of narcotine is less than in sulphuric acid, the effect of papaverine is greater. In acid solution it would appear that papaverine and narcotine form associated molecules which become dissociated on greater dilution. (5) Since no effect is observed when the free bases are present together in an inactive solution, it would seem that acid is necessary to cause alteration in the structure of the molecule. (6) A solution of narcotine in an acid alcohol is much more dextrorotatory than a solution in acid only, and a solution in ethyl alcohol is lævorotatory.

　　　D. G. H.

Biochemical, Bacteriological, etc.

Presence of a New Globulin, Conphaseolin, in the Navy Bean (*Phaseolus vulgaris*). H. C. Waterman, C. O. Johns, D. B. Jones, and S. Phillips. (*J. Biol. Chem.*, 1923, **55**, 93–104.)—The work of Osborne (*J. Amer. Chem. Soc.*, 1894,

16, 633, 703, 757) upon the coagulation of saline extracts of *P. vulgaris* seeds indicated the existence of another protein in addition to phaseolin and phaselin, and fractional precipitation of similar extracts by means of ammonium sulphate carried out by the authors confirm this conclusion. Conphaseolin was isolated by extracting the finely-ground beans with 5 volumes of aqueous 2 per cent. sodium chloride solution, with continuous agitation, for 2 hours. Torn filter paper was added to the suspension, and the whole pressed in cloth bags until about 85 per cent. of the original volume of solvent was recovered. The turbid liquid was then filtered through paper pulp moistened with 2 per cent. sodium chloride solution, and finely powdered ammonium sulphate was added to the clear filtrate in small portions until a concentration of 30 per cent. saturation was reached. Further procedure was identical with that employed in the preparation of α-globulin from mung bean (*J. Biol. Chem.*, 1920, **44**, 303). Conphaseolin precipitated by dialysis consists of minute spheroids, insoluble in neutral solvents and soluble with difficulty in 0·1 N acids and alkalis, and amounting to approximately 0·4 per cent. of the dry weight of the beans. It has the general properties of the α-globulins derived from other species of *Phaseolus*, and the specimen examined had the following composition:—Carbon, 53·81; hydrogen, 6·86; oxygen, 22·24; nitrogen, 15·73; and sulphur, 1·36 per cent. Analysis of the protein by the van Slyke method yielded the following percentages of amino-acids:—Cystine, 1·18; arginine, 6·88; histidine, 0·79; and lysine, 10·68. The relatively high percentages of sulphur and lysine sharply differentiate conphaseolin from other proteins with which it is associated in the navy bean.   T. J. W.

Estimation of Sugar in Blood. E. G. B. Calvert. (*Biochem. J.*, 1923, **17**, 117–129.)—The blood is collected direct from a finger-prick in a small platinum capsule, which is immediately weighed on a torsion balance and dropped into a test tube containing 7·3 c.c. of distilled water, and, after mixing, the capsule is removed. The proteins are then precipitated by the Folin-Wu method (ANALYST, 1920, **45**, 227), 0·3 c.c. of 10 per cent. sodium tungstate solution and 0·3 c.c. of $\frac{2}{3}$ N sulphuric acid being used, and the liquid is filtered through a starch-free paper. Five c.c. of the filtrate are mixed with 2 c.c. of a modified Fehling solution, and heated in a boiling water bath for 6 minutes, after which 2 c.c. of a phosphomolybdic acid solution are added, and the solution diluted to 12·5 c.c. and allowed to stand 5 minutes. The blue colour produced is compared in a colorimeter with a standard consisting of a blue and a yellow glass previously calibrated by means of estimations carried out with blood containing known amounts of dextrose. The modified Fehling solution is prepared by dissolving 40 grms. of anhydrous sodium carbonate in 500 c.c. of water, adding 7·5 grms. of tartaric acid and, after complete solution is effected, 4·5 grms. of crystallised copper sulphate are added and dissolved without heating. The phosphomolybdic acid solution is made by dissolving 35 grms. of molybdic acid in 200 c.c. of 10 per cent. sodium hydroxide solution, adding 200 c.c. of water, and boiling the liquid until free from ammonia. After cooling, the solution is diluted to 350 c.c., and 125 c.c. of 85 per cent. phosphoric

acid are run in, the whole being diluted to 500 c.c. A table and curves are provided for estimating the real dextrose values from the "apparent" values obtained, and full details of manipulation and calculation are given. The method is expeditious and simple. T. J. W.

Quantitative Estimation of Choline in Blood. J. S. Sharpe. (*Biochem. J.*, 1923, **17**, 41–42.)—The following method is a development of one devised by Staněk, and overcomes the difficulties met with in the application of his procedure. From 20 to 50 c.c. of blood are added to four times the volume of absolute alcohol, and the mixture is well shaken and allowed to stand 12 hours, after which it is filtered. The residue is rubbed and well washed with absolute alcohol, the washings being added to the filtrate, and the whole evaporated to a syrup, which is dissolved in distilled water and again evaporated until free from alcohol. The resulting syrup is dialysed into 200 c.c. of distilled water for 24 hours, this being repeated with a second 200 c.c. of water, after which the dialysates are mixed, evaporated to about 5 c.c., and treated with 20 to 30 volumes of saturated iodine solution in potassium iodide, the mixture being left to stand overnight for the choline periodide to crystallise. The crystals are collected on a Gooch filter, washed with ice-cold water, and treated with a mixture of equal volumes of water, nitric acid and chloroform. The chloroform layer is washed with water to remove free acid, and the iodine in solution is titrated with 0·05 N sodium thiosulphate solution until the pink colour just disappears. One c.c. of the sodium thiosulphate solution is equivalent to 0·7 mgrm. of choline. Estimations carried out with blood to which known amounts of choline were added showed a recovery varying from 90·6 to 94 per cent. T. J. W.

Estimation of Allantoin. J. More. (*J. Pharm. Chim.*, 1923, **6**, 209.)—If Nessler solution be added to a solution of allantoin, a yellowish colour is produced, which becomes darker, and eventually black, through the formation of reduced mercury if the ureide is in excess. This reaction is still given by 10 c.c. of a solution containing 0·01 grm. per litre. Other ureides and ammonium salts do not interfere, since the precipitate formed is redissolved on adding acid or potassium cyanide. The method of estimation is to place 0·01 grm. of allantoin in a 250 c.c. flask with 20 c.c. of water and 2 to 3 drops of strong sodium hydroxide solution. When dissolved, 30 c.c. of a solution containing 27·1 grms. of mercuric chloride and 72 grms. of potassium iodide per litre, are added, together with 10 c.c. of 30 per cent. sodium hydroxide solution. When the reduction has reached a maximum (12 hours) the solution is acidified with hydrochloric acid (1:4), and 10 c.c. of 0·1 N iodine solution are added, which readily dissolves the products of reduction. The excess of iodine is titrated back with sodium thiosulphate solution (10 grms. per litre). Two atoms of iodine correspond to one molecule of allantoin (158). The result must be corrected by the factor 1·08, since numerous experiments have shown that only 92 per cent. of the allantoin is hydrolysed into glyoxylic acid and urea under these conditions. Imino-allantoin, obtained by oxidising uric acid in ammoniacal solution, reacts in a similar way to free allantoin, since it is hydrolysed

in presence of acid into ammonia and allantoin. In attempting to estimate allantoin in pathological urines, difficulties arise owing to the possible presence of dextrose and creatinine, both of which reduce Nessler's solution, and there is no satisfactory method at present for isolating the allantoin. R. F. I.

Digestibility of Fats. C. F. Langworthy. (*J. Ind. Eng. Chem.*, 1923, 15, 276–278.)—In the feeding experiments (on men) described, twenty-three animal fats, thirty-four vegetable fats and oils, and six hydrogenated oils were utilised; they were administered in the form of a cornflour pudding or blanc-mange. The results obtained show that there is very little difference in the digestibility of the fats, but those having melting points above body temperature appear to be not quite so thoroughly assimilated. W. P. S.

Protection of Vitamin C in Foods. E. F. Kohman. (*J. Ind. Eng. Chem.*, 1923, 15, 273–275.)—In previous work on the stability of vitamin C towards heat, the effect of oxygen does not appear to have been taken into account; it has, however, been proved that this effect is considerable, but it has not been proved to what extent the destruction of the vitamin is due to heat and to what extent to oxidation. Methods for the elimination of oxygen are suggested; liquids may be subjected to reduced pressure at ordinary temperature with thorough agitation, whilst solid foods may be treated similarly with or without the addition of water. Nitrogen is subsequently admitted to the vessel in which the operation has been carried out. In the case of fruits and vegetables, their natural respiration may be utilised to deprive them of oxygen. W. P. S.

Efficiency of Common Antiseptics. E. H. Harvey. (*Amer. J. Pharm.*, 1923, 95, 105–108.)—The method previously described (ANALYST, 1923, 131), has been used to estimate the relative efficiency of further antiseptics, and the various substances are again arranged in the order of decreasing efficiency as indicated by the results obtained, viz.:—Boric acid, abrastol, sodium hydrosulphite, saccharin, clove oil, sassafras oil, sodium fluoride, and β-naphthol. Similar experiments carried out with sodium chloride in concentrations varying between 5 and 25 per cent. showed that the action of invertase in yeast is but slightly inhibited by the more dilute solutions of this salt and progresses fairly rapidly with the higher concentrations. For comparison with the action of invertase on sucrose, 10 grms. of this sugar were dissolved in 100 c.c. of 1/12 N hydrochloric acid and maintained at a temperature of 23° C., polarimetric readings being taken at 10-hour intervals. The curve obtained by plotting the results bears a close resemblance to those given by the enzyme inversion of sucrose, thus indicating a similarity of action between these two processes, probably of a catalytic nature. T. J. W.

Actinomyces in Milk. C. R. Fellers. (*J. Dairy Sci.*, 1922, 5, 485–501; *Chem. Abstr.*, 1923, 17, 155.)—Actinomyces are frequently present in market milk, sometimes to the extent of 2·5 per cent. of the total organisms present. The

main sources of infection of the milk are hay, straw, grain, soil and dust from these substances. It may lead to the development of a characteristic unpleasant mouldy odour and a bitter taste, for which the term "actinomyces odour and taste" is suggested. The two most active species of 11 identified are *A. griseus* and *A. albus*, which grow readily in milk, and are able to bring about pronounced changes in casein and whey. The presence of actinomyces may also cause stale and musty odours in other foods, and they are liable to occur in association with moulds in foods, especially those in dried condition. Incubation at low temperatures on synthetic media should be used for their isolation.

Influence of Certain Factors on the Methylene Blue Reduction Test for Milk. E. G. Hastings, A. Davenport and W. H. Wright. (*J. Dairy Sci.*, 1922, **5**, 438–454.)—Impurities which may be present in different grades of the dyes used for the methylene blue reduction test have no practical influence on the results, nor has the age of the solution of the dyestuff. There is no reason for deviating from the usual temperature (38° C.) in making the test. Whilst every organism growing in the milk appears to assist in the reduction of the dye, they vary in their activity in this respect. The reaction of any sample of milk to which the test would be applied in practice would not be such as to interfere with the results; agitation of the milk has practically no effect upon the reduction time. The results indicate that the test only shows in a general way the keeping quality of the milk, for the diversity of bacterial flora in milk prevents the use of any one absolute test or probably any group of tests. The test should be made as follows: —One part of the stock solution of medicinal methylene blue (1:2000) is diluted to 10 times its volume, and 1 c.c. of the dilute solution added to 10 c.c. samples of the milk in test tubes of the same diameter and of such size that 11 c.c. do not more than half fill them. The test tubes should be steamed, but need not be sterilised, and the pipette should be rinsed three times with boiling water between the taking of each sample. After addition of the dye solution the tubes are closed with the thumb, inverted once or twice, and then placed in a water bath at 38° C. Observation beyond a period of six hours is seldom necessary.

Toxicological and Forensic.

Toxic Properties of Robinia Pods. E. M. Holmes. (*Pharm. J.*, 1923, **110**, 263.)—*Robinia pseud-acacia* is the botanical name of the tree popularly termed "acacia" and "cassia." It somewhat resembles a laburnum, but has white flowers, and produces flatter and longer pods. The poisonous action is due to a toxalbumin termed robine. It resembles cytisine in causing violent vomiting and purging in human beings. A recent instance of three sheep being poisoned through eating the pods is recorded, all the animals dying within three days. In cases of Robinia poisoning the kidneys should be examined for the characteristic agglutination of the red corpuscles.

Agricultural Analysis.

Quantitative Estimation of Amino Acids of Linseed Meal, Wheat Meal, Soya Beans, and Red Clover Hay. T. S. Hamilton, N. Uyei, J. B. Baker, and H. S. Grindley. (*J. Amer. Chem. Soc.*, 1923, **45**, 815–819.)—By means of the modified van Slyke method described by Hamilton Nevens and Grindley (ANALYST, 1922, **47**, 33), the amino acids of the above-mentioned feeding stuffs have been estimated with the mean results set out in the following table:

Expressed as % of Total N. in the Feed	Soya Beans	Wheat Bran	Linseed Meal	Red Clover Hay
Total Basic N.	28·94	24·02	26·81	15·40
Ammonia N.	9·38	9·17	10·86	7·35
Humin N.	2·87	6·86	5·09	13·09
Arginine N.	15·70	11·99	15·92	6·86
Cystine N.	1·46	0·81	1·07	0·87
Hystidine N.	5·60	7·32	6·14	5·05
Lysine N.	6·18	3·90	3·68	2·62
Mono-amino acid N., amino N. in filtrate from bases	42·28	38·07	44·55	39·82
Proline, oxyproline, tryptophane, etc., non-amino N in filtrate from bases	2·43	3·59	3·26	3·28
Ether soluble N.	0·16	0·04	0·12	0·62
Alcohol soluble N.	0·58	1·23	0·37	1·57
Non-protein N. sol. in cold 1 per cent. trichloracetic acid in filtrate from colloidal iron	5·55	15·26	7·88	11·29
N. lost in method of analysis	2·23	2·27	2·80	6·87
Total	100·53	100·51	101·74	99·29

H. E. C.

Organic Analysis.

Preparation of Absolute Alcohol by means of Calcium Chloride and Lime. W. A. Noyes. (*J. Amer. Chem. Soc.*, 1923, **45**, 857–862.)—A pure alcohol of 99·0 to 99·5 per cent. may be prepared from strong alcohol of about 92 per cent. by distillation in a specially designed apparatus containing anhydrous calcium chloride. For laboratory purposes, where such special apparatus is not available, distillation from lime affords the best results. Ten litres of alcohol of about 92 per cent. purity and 2000 grms. of good lime are heated under a reflux condenser for 24 hours in a 12-litre flask. The alcohol is then distilled with the use of a trap and a condenser so arranged that contact with air is avoided, and the distillate, which is usually turbid owing to the presence of calcium ethylate, is mixed with 50 per cent. more than the theoretical amount of calcium oxide and 10 per cent. of that amount of anhydrous calcium chloride, heated under a reflux condenser for 24 hours and distilled. The product is a pure alcohol of at least 99·9 per cent. concentration. Metallic sodium is quite unsuitable for the dehydration of alcohol. H. E. C.

Estimation of Ethyl Ether in Air, Blood and other Fluids. H. W. Haggard. (*J. Biol. Chem.*, 1923, **55**, 131–143.)—The method described is based upon the liberation of iodine from heated iodine pentoxide by the vapour of ether, 1 molecule of which sets free 4·8 atoms of iodine. The apparatus consists of a tube containing calcium chloride connected indirectly with a similar one containing solid potassium hydroxide for purifying the air passing through the apparatus. Between these tubes is fitted a small wash bottle with two three-way stopcocks and a bye-pass, in such a manner that air may bubble through the liquid or may pass direct to the potassium hydroxide tube. The outlet of this tube is connected with a U-tube which contains alternate layers of glass wool and iodine pentoxide (40 grms.), and is immersed in an oil bath, the outlet of which is connected with an efficient absorption tube containing potassium iodide solution. The iodine pentoxide is prepared for use by heating the oil bath to about 250° C. for several hours, while a stream of air is drawn through the apparatus until the evolution of iodine is small and is constant in amount. For the actual estimation the temperature of the oil bath is raised to 200° C., during the passage of a current of air, and when this temperature is reached, 10 c.c. of 10 per cent. potassium iodide solution are run into the absorption tube, and the air current is continued for 30 minutes. The liberated iodine is titrated with sodium thiosulphate solution, 1 c.c. of which is equivalent to 0·1 mgrm. of ether, and the result obtained is taken as the blank for the actual estimation. A few c.c. of the liquid under examination are then transferred to the small wash bottle, or the vessel of air containing the ether vapour is connected with one of the three-way stopcocks, and, after the potassium iodide solution has been replaced, a current of air is drawn through the apparatus at the rate of 200 c.c. per minute for 30 minutes, after which the iodine is titrated as before. The liquid or gas analysed should not contain more than 5 mgrms. of ether, and the experimental error of the method will then not exceed 3 per cent. Acetone may also be estimated by this method, and should be absent from the fluids used for the estimation of ether. Experiments were carried out to determine the distribution of ether between water and air and blood and air between temperatures of 24° and 40° C., and the results are tabulated and plotted as a curve in the original paper. T. J. W.

Effect of Fuller's Earth on Pinene and Other Terpenes. C. S. Venable. (*J. Amer. Chem. Soc.*, 1923, **45**, 728–734.)—When α-pinene obtained from the fractionation of turpentine is left in contact with fuller's earth (Florida earth, burned at 400° C., and containing 6 per cent. of water), it undergoes intra-molecular rearrangement due to surface catalysis, with the formation of dipentene and terpinene, but not of camphene. The products polymerise slowly at ordinary temperatures to dipinene and polyterpenes in the presence of the fuller's earth. At high temperatures the dipinene decomposes into paracymene and hydrocarbons of low boiling point. It is shown also that similar changes are undergone by nopinene, dipentene, α-limonene, terpinene, camphene, sabinene, terpineol, and borneol, but not by cineol or paracymene. Oxygenated substances, such as.

acetone or alcohol, used as diluents partially inhibit the reaction, but diluents such as benzene or carbon tetrachloride have no such action. The changes are not brought about by silica-gel, iron-gel or activated charcoal. H. E. C.

Separation of Xylenes. H. T. Clarke and E. R. Taylor. (*J. Amer. Chem. Soc.*, 1923, **45**, 830–833.)—Contrary to statements in much of the literature on the subject, the three xylenes cannot be obtained in a state of purity from a mixture by distillation only. It is necessary to carry out four operations, fractional distillation, selective sulphonation, fractional crystallisation of the sulphonic acids and hydrolysis of the acids. *m*-xylene can be obtained in a crude state by simple distillation; to purify it, it may be treated with dilute nitric acid, which only oxidises the *o*- and *p*-xylenes. Technical *m*-xylene may be purified by sulphonating it at 95° C. for four hours, neutralising the product with 40 per cent. sodium hydroxide solution, separating the crystals, and recrystallising the sodium salt of the *o*-xylene sulphonic acid; the salt is then hydrolysed by steam-distillation with dilute sulphuric acid. For the purification of the *p*-xylene, the technical product is sulphonated with sulphuric acid in the cold, the undissolved portion being retreated in the same way; the undissolved residue contains the *p*-xylene, and is separated and sulphonated at 95° C., and the product slowly mixed with concentrated hydrochloric acid; the *p*-xylene sulphonic acid, which crystallises out, is separated and hydrolysed by steam-distillation with dilute sulphuric acid. The first runnings contain some *m*-xylene, but the fraction distilling above 145° contains pure *p*-xylene, which is again fractionated, and then yields a product boiling between 136·5° and 137° C. H. E. C.

Analysis of TNT and Tetryl Mixtures. C. A. Taylor and W. H. Rinkenbach. (*J. Ind. Eng. Chem.*, 1923, **15**, 280–281.)—A method proposed for the analysis of these mixtures depends on the different solubilities of the two substances in carbon tetrachloride. At 0·3° C., 100 grms. of carbon tetrachloride dissolve 0·215 grm. of TNT, and at 0·5° C., 0·0073 grm. of tetryl. In order to use a volume of about 100 c.c. of the solvent (sp. gr. 1·629) the weight of sample taken is fixed at 0·35 grm., so that, if it is pure TNT, it will all be dissolved. This weight of the dry, well ground sample is placed in a weighed 100 c.c. flask provided with a glass stopper, about 100 c.c. of carbon tetrachloride are added, the mixture is heated until the solvent boils, then cooled to ordinary temperature, and the flask and its contents are weighed. The flask is now immersed in an ice-bath for four hours, and shaken every fifteen minutes, and at the end of this time as large a volume as possible of the clear supernatant liquid is drawn off, transferred to a weighed flask, the carbon tetrachloride is evaporated by means of a current of dry air, the residue is dried at 65° C., and weighed. The residual solution in the 100 c.c. flask is brought to ordinary temperature and weighed in order to obtain the weight of the aliquot portion withdrawn. Knowing the solubility of tetryl in carbon tetrachloride at 0·3° to 0·5° C. (the temperature at which the aliquot portion is taken), the amount dissolved is calculated and deducted from the weight of the total dissolved solids to give the amount of TNT in them. (See also ANALYST, 1923, 137.) W. P. S.

Examination of Coloured Wax Compositions. H. Smelkus. (*Chem. Zeit.*, 1923, **47**, 168–169.)—A portion of 50 to 100 grms. is distilled in a current of steam, turpentine oil, resin oil, benzine, etc., then passing quantitatively into the distillate. If the presence of water in marked proportion is indicated, as by spurting on heating, another portion is distilled with xylene on an oil bath, the free water distilling being measured in a cylinder. If the iodoform test shows alcohol in the distillate, the composition, mixed, if necessary, with potassium hydrogen sulphate to prevent frothing, is distilled from an Engler flask, the alcohol being estimated by determining the specific gravity of the fraction distilling below 100° C. The presence of turpentine oil, or resin oil, in the solvent used in preparing the composition is shown by the odour, bromine value, and Herzfeld's test for resin oil. Benzine is indicated by absence or depression of the bromine value and by indifference towards concentrated nitric acid, and benzene hydrocarbons by the dracorubin test.

The residue from the steam distillation is freed from water mechanically, dried at 105° C., and weighed. Separation of the wax from the colouring matter is now desirable. This may be effected by means of benzine or ether, if the wax dissolves well in these solvents; the latter dissolve only traces of nigrosine, which is commonly present in boot-polish. In some cases it is preferable to remove the colouring matter by means of dilute hydrochloric acid, which serves also to eliminate traces of colouring matter (nigrosine) from the ethereal or petroleum spirit solution of the wax. In the case of azo dyestuffs (yellow, red, orange), such residual traces may be expelled by reduction with tin and dilute hydrochloric acid.

If the material contains soaps, these may be separated from the non-saponified substances by means of water. Should the colouring matters be soluble in water (*e.g.* soluble nigrosine), the aqueous filtrate containing the soap and colouring matter is evaporated and acidified with dilute hydrochloric acid, the acids of the fat or wax thus separated being then extracted by ether, benzine, etc.

When wax, soap, colouring matter, and water are present together, it is simpler to melt the material on a water-bath or in an oven at 100° C., and allow it to cool, the solid surface of the cake of wax, which still contains a considerable amount of undissolved soap, being then broken and the water or coloured aqueous emulsion poured into a porcelain basin. The residue is again fused with a little water, and the resultant emulsion poured off. The residue and the emulsions are separately freed from water and volatile solvents, and are then united, mixed with sand, and extracted in a Gräfe apparatus with benzene, ether or petroleum spirit, to remove unsaponified wax constituents and hydrocarbons. Soap and water-soluble colouring matter are then dissolved from the sand by means of water or alcohol, the solvent being afterwards evaporated. If these constituents behave similarly towards the ordinary solvents, the mixture is acidified and the acids of the fat and wax then extracted by means of benzine, ether or benzene.

When the non-saponified and saponified wax or fat have been obtained by one of these methods in a pure state, free from colouring matter, the acid, saponification, and ratio values are determined. T. H. P.

Inorganic Analysis.

Absorbent Mixtures for Carbon Monoxide. **A. Piutti.** (*Giorn. Chim. Ind. Appl.*, 1923, **5**, 70–73.)—Of a number of absorbents for carbon monoxide, a mixture of cuprous chloride, fir charcoal, granules of pumice and powdered soda lime gave the best results. This mixture is stable in air. T. H. P.

Detection of Minute Quantities of Arsenic. **O. Billiter.** (*Helv. Chim. Acta*, 1923, **6**, 258–259.)—The process already described (*cf.* ANALYST, 1919, **44**, 51) depends upon the evolution of the arsenic as trichloride upon distillation with sulphuric acid and sodium chloride, followed by evaporation with hypochlorous acid and then "Marshing." It is now found that fuming nitric acid may with advantage be substituted for the hypochlorous acid. The gaseous distillate from the distillation as described in the original paper is passed into 4 or 5 c.c. of fuming nitric acid; the hydrochloric acid escapes as nitrosyl chloride, leaving a pure solution of arsenic acid, which is evaporated, and the residue is introduced into a Marsh apparatus, which should be of the smallest possible dimensions to secure a maximum sensibility. H. E. C.

Estimation of Bismuth. **Koster.** (*Chem. Zeit.*, 1923, **47**, 22.)—The fine red colour produced by dilute hydriodic acid when dropped upon the cupelled lead affords a simple and accurate method for the estimation of small quantities of bismuth in lead. The lead or ore containing the bismuth is cupelled in the usual way, cooled and a drop of dilute hydrogen iodide is placed upon the metal; the amount of bismuth is estimated by comparison of the intensity and extent of the red patch with a standard or scale prepared from lead samples made up to contain 0·002, 0·006, 0·010, 0·030, and 0·050 per cent. of bismuth. The red colour gradually disappears, but may be partially restored by the addition of a further drop of hydriodic acid. Small amounts of copper do not interfere with the test, which may also be applied to the detection of bismuth in ores by cupelling them with a lead regulus and completing the test as before. The dilute hydrogen iodide solution may conveniently be prepared by passing hydrogen sulphide through iodine water and filtering. H. E. C.

Estimation of Bismuth as Phosphate. Separation from Lead, Copper and Cadmium. **G. Luff.** (*Chem. Zeit.*, 1923, **47**, 133–134.)—Moser has shown (*Zeitsch. anal. Chem.*, 1906, 19) that bismuth may be estimated as phosphate, but the procedure described by him is not satisfactory for its separation from lead, copper and cadmium. Acid ammonium phosphate is preferable to the alkaline diammonium phosphate for the precipitation. The reaction

$$Bi(NO_3)_3 + NH_4H_2PO_4 \longrightarrow BiPO_4 + NH_4NO_3 + 2HNO_3,$$
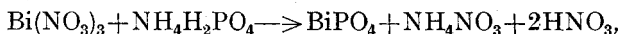
being reversible, it is necessary to adhere strictly to the conditions described as to concentration of acid and phosphate; a complete separation is then obtained. The nitric acid solution of the metals is carefully neutralised with ammonia,

methyl orange being used as indicator; the precipitate which forms is redissolved by the addition of exactly 4 c.c. of nitric acid (sp. gr. 1·40), and the volume of liquid is made up to 80 c.c. The mixture is now heated just to boiling, 1 grm. of ammonium phosphate dissolved in 40 c.c. of dilute nitric acid (5 c.c. of nitric acid, sp. gr. 1·40 in 100 c.c. of water) is slowly added from a dropping funnel, and the mixture is kept just at the boiling point under an air reflux condenser for 6 to 10 hours. The precipitate, which is white and crystalline, is filtered off, washed with water, ignited with the paper until quite white, and weighed as $BiPO_4$. Lead, copper or cadmium in the filtrate may be separated in the usual way as sulphate, thiocyanate, or sulphide respectively. H. E. C.

Detection of Peroxides and Per-salts. A. Blankart. (*Helv. Chim. Acta*, 1923, **6**, 233–238.)—Although the identification of pure peroxides and per-salts is simple, it is not always easy to identify them in mixtures with other salts, such as commonly occur in washing powders. The following table summarises the colour reactions of these compounds with different reagents; in each case an excess of the solid substance is shaken with the solution of the cold reagent, except in the case of the cochineal test in which the mixture is warmed to 70° C. For the cobalt nitrate test a layer of benzene is run on to the solution before adding the solid substance. The reactions with aniline water and with cochineal are less sensitive than the others. The presence of alkaline earth metals interferes with some of the reactions, so that in the presence of these elements it is sometimes impossible to decide with which acids the active oxygen is combined. In the case of mixed per-oxides or per-salts the colour effects given in the table may be modified, but identification is possible by noting the combined effects of the two compounds.

Reagent.	Na_2O_2.	$K_2C_2O_6$.	Na_2CO_4.	$Na_2CO_3.H_2O_2$.
Alcoholic p-amidophenol	black	colourless	colourless	colourless
30 per cent. potassium iodide solution	colourless	brown	brown	colourless
Silver nitrate, 0·05N	brown	yellowish	yellowish	black
Aniline-water	colourless	—	brown	colourless
Alcoholic solution of cochineal	decolourised	—	decolourised	red
Cobalt nitrate, 0·25 per cent. solution	brown	—	green	greyish-brown
Cobalt nitrate and soda	green	—	green	brown

Reagent.	$NaBO_2$.	$Na_2HPO_4.H_2O_2$.	$Na_4P_2O_7.H_2O_2$.	$Na_2S_2O_8$.
Alcoholic p-amidophenol	colourless	colourless	colourless	colourless
30 per cent. potassium iodide solution	yellow or colourless	turns brown slowly	colourless	turns brown slowly
Silver nitrate, 0·05N	black[1]	yellow	white	violet
Aniline water	light-brown	yellowish brown	—	brown
Alcoholic solution of cochineal	partly[2] decolourised	red	red	red[3]
Cobalt nitrate, 0·25 per cent. solution	light-brown	blue	pink	pink
Cobalt nitrate and soda	greyish-brown	greyish-brown	brown	violet

[1] Sometimes white.   [2] Decolorised only in presence of alkali.
[3] Decolorised if alkali is present.

H. E. C.

Separation of Radium from other Elements. M. Cialdea. (*Gazz. Chim. Ital.*, 1923, **53**, I., 42–48.)—Radium bromide, accidentally dropped, was recovered by evaporating the sweepings and washings of the pavement on a water bath and fusing the residue thus obtained with a mixture of 2 parts of sodium carbonate, 2 parts of potassium carbonate, and 1 part of sodium nitrate. The radium is thus brought into solution and, if barium sulphate is precipitated from the liquid, is carried down completely by the precipitate.   T. H. P.

Electrometric Titration of the Halides in the Presence of One Another. H. H. Willard and F. Fenwick. (*J. Amer. Chem. Soc.*, 1923, **45**, 623–633.)—Chlorides can be accurately titrated with silver nitrate, or *vice-versa*, in acid solution, by using the bimetallic electrode system already described by the authors (*cf. J. Amer. Chem. Soc.*, 1922, **44**, 2516, Abst.; *J. Chem. Soc.*, 1923, **124**, ii., 33), in which the end-point is indicated by a rise, followed by a fall, in potential difference. Bromine may be estimated by titration in hydrocyanic acid solution with potassium permanganate to bromine cyanide, but the end-points on the bimetallic system are unsatisfactory; the titration may, however, be effected by plotting a curve and using the usual mono-metallic system. Bromide may be similarly estimated in the presence of chloride, as selective oxidation takes place; the first end-point, which is indicated by a slight fall, is not always quite sharp, and is sometimes absent; the titration is therefore continued to a maximum, and a correction of –0·10 c.c. of 0·1 N solution is applied. Iodide, if present, is oxidised to iodine cyanide, and must be estimated with the bromide and then separately. The separate estimation may be based upon the selective oxidation with permanganate with the use of the bimetallic system; there is a sharp break in the curve at the first end-point (oxidation of iodine), even in the presence of chloride and moderate concentration of bromide. A more accurate method for the iodide, however, is by oxidation to iodate by means of excess of alkaline hypobromite, the excess of the hypobromite being titrated electrometrically with sodium arsenite. Bromides or chlorides do not affect the accuracy of this method, and either the mono-metallic or the bimetallic system may be used.   H. E. C.

Electrometric Estimation of Sulphur in Soluble Sulphides. H. H. Willard and F. Fenwick. (*J. Amer. Chem. Soc.*, 1923, **45**, 645–649.)—By using the bimetallic system (*cf.* preceding abstract) sodium sulphide may be titrated with ammoniacal silver solution; the end-point is indicated by a sudden drop in the potential, which is so sharp that there is no need to plot a curve. The titrating liquid is 0·05 N silver nitrate solution containing about 30 c.c. per litre of a 28 per cent. solution of ammonium hydroxide. Varying concentrations of sulphites, sulphates, thiosulphates, polysulphides, and chlorides are without effect on the titration, so that the method may be used in the presence of all these salts. Sulphur in steel may be estimated by dissolving the steel in hydrochloric acid and absorbing the sulphide in a solution of sodium hydroxide, the last traces of the gas being washed out by means of a stream of hydrogen, and the sulphide titrated electrometrically as above.   H. E. C.

Volumetric Estimation of Small Amounts of Phosphorus by means of a Standard Solution of Methylene Blue. W. M. Thornton, jun., and H. L. Elderdice, jun. (*J. Amer. Chem. Soc.*, 1923, **45**, 668–673.)—The process depends upon the precipitation of the phosphorus as ammonium phosphomolybdate in the usual way, the subsequent solution of the precipitate in ammonia solution, and titration with a standard solution of methylene blue. The methylene blue solution contains 4 grms. per litre, and is standardised with titanous sulphate in an atmosphere of carbon dioxide. The amount of phosphorus taken for a titration should not exceed 0·4 mgrm. The phosphomolybdate precipitate is dissolved in excess of dilute ammonia solution in a wide-mouthed flask fitted with a four-hole bung in which are inserted a dropping funnel, a burette, and an inlet and an outlet tube connected with a carbon dioxide generator and an air pump respectively. Five grms. of granulated zinc are put in the flask, and the air displaced by means of a stream of carbon dioxide; then 50 c.c. of hydrochloric acid are added gradually from the dropping funnel until the reduction of the molybdenum is complete; the remaining acid is added, the solution boiled to dissolve the zinc, more carbon dioxide is passed through the flask, and the solution is titrated with the methylene blue until the colour changes to a dark green. The method is suitable for the estimation of phosphorus in steel. H. E. C.

Physical Methods, Apparatus, etc.

Method of Deciphering Charred Records. R. Davis. (*U.S. Bureau of Standards, Sci. Papers*, 1922, **18** (454), 445–450.)—A method of deciphering written or printed characters on paper which has been charred consists in attaching the paper, with the characters face downwards, to the emulsion side of a photographic plate, which is then left in the dark for a week or two, after which it is developed in the usual way. The ink in the written or printed characters appears to act as a screen to protect the plate from the gaseous products in the charred paper, which fog the plate. The result of the long exposure, therefore, is that the characters are shown in white on a black background on the developed plate. A plate of fast or medium speed must be used, since slow plates are not sufficiently sensitive for the purpose, and a longer time of contact between the charred paper and plate is required if the paper has been exposed to the air. If films are used instead of plates, an exposure of two or three months is required, and a positive result is obtained, the inked characters appearing black on a white background. By washing the films with water and drying them in the dark, however, before exposing them to the action of the charred paper, a negative result is obtained, as in the case of plates.

Collection and Examination of Explosive Dusts in Air. L. J. Trostel and H. W. Frevert. (*J. Ind. Eng. Chem.*, 1923, **15**, 232, 236.)—The paper-filter method is described and recommended; it consists essentially in drawing a measured volume of dusty air through a paper thimble by means of a calibrated hand pump or an electrically-driven blower fitted with a Venturi meter and manometer.

The filter thimbles used measure 33 by 94 mm., and are fitted into the top of a small brass chamber a side tube on which connects with the pump; a quantity of loose cottonwool is placed in the thimble to act as a support for the dust and prevent it clogging the filter, and the thimble is dried thoroughly and weighed before and after use. About 2 cubic feet of air per minute should be drawn through the filter during the test; the absorption of moisture from the air, even when the relative humidity is as high as 90 per cent., does not affect the rate of flow of air through the filter. The collected dust, after it has been weighed, may be examined microscopically and a portion of it submitted to an explosibility test in which 75 mgrms. of the dust are exploded in a spherical glass bomb under standard conditions, and the resulting pressure is noted.   W. P. S.

Celluloid for Microscopic Work. O. Nuzzi. *Riforma Med.*, 1922, **38,** 818; *Pharm. J.*, 1923, **110,** 134.)—Thin sheets of celluloid can be used with advantage as cover slips. The thinnest sheets made average 0·11 mm. in thickness, or somewhat less than the thickness of glass cover slips (0·14 to 0·17 mm.). In addition, the refractive index of celluloid (1·5002) is very near to that of cedar-wood oil (1·5100), so that celluloid is suitable for mounting specimens which are to be examined under oil-immersion lenses.

Reviews.

THE CHEMISTRY TANGLE UNRAVELLED. By FRANCIS W. GRAY, M.A., Sc.D. Pp. viii.+148. London: Longmans, Green & Co. 1923. Price 6s. net.

This book has been written to serve as a guide to the student of inorganic chemistry. There is a distinct tendency for textbooks to give an undue share of attention to details concerning the various compounds as compared with that given to the principles involved. This is especially the case to-day, now that the chemical knowledge available has become so extensive. Hence the need for a book of the nature of the one under review.

In it the author has aimed at giving a clear exposition of the principles, substantiated by examples, and, moreover, has made an attempt to show how these conceptions may all be linked together in the form of a new periodic system in which the rare-earth elements and the believed missing elements all find places. Thus the relationship between atomic structure, valency and reactivity has been traced by applying some of the best ideas of Abegg on valency and the recent theories of Kossel and Langmuir. The views held by Sir W. H. Bragg and his son on crystal structure have led the author to generalise that there is complete dissociation into ions, in either the fused salt, in the concentrated aqueous solution and in the aqueous solution at infinite dilution; the main factor which determines conductivity being the extent to which inter-ionic attractions impede

the movement of the ions. The main aspects of chemical reactions are discussed. Union between atoms, being due to inter-atomic affinity, is regarded as taking place in two stages: (1) The charging of the neutral atoms, due to their electro-affinity, and (2) the union of the charged atoms to give the neutral molecule, due to interionic affinity. Very lucid treatment is given to each of these types of affinity. It is shown how they are dependent on both the atomic numbers and the atomic volumes, and, therefore, how a knowledge of these factors may be gathered from a consideration of the relative positions of the elements in the modified periodic system. Then follows an application of these conceptions to the nature of hydroxides and hydrides, hydration, hydrolysis and their ammonia analogues; complex and multiplex ions. Stress is laid on the part played in analytical work by complex ions, ammines and the substituted ammonia compounds. The book ends with a discussion of double decomposition and oxidation and reduction. An excellent bibliography is appended.

From cover to cover the book is stimulating to thought, and, indeed, one is led to see how fundamentally important these conceptions of atomic structure are to the chemist. It remains to be seen whether the author has forced his conclusions too far. One thing is certain, that he has given a good working picture of the mechanism of chemical combination, and has done a good deal towards removing the discrepancies of the periodic system.

A few points, however, demand attention. The classification of beryllium and magnesium in Group IIA. is passed over without comment (p. 6), whereas, it is pointed out that the first two members of both Groups III. and IV. may be placed equally well in either the A or B Sub-group. On page 92, magnesium is wrongly stated to be the only element in Group II.A capable of forming a double chloride. There is some evidence regarding the formation of double chlorides with the alkaline earth elements; in fact, the formation of the double chloride of calcium and cæsium has been suggested recently as a microchemical test for calcium. The well-known action in analysis of an excess of ammonium chloride in preventing the precipitation of the hydroxides of magnesium, ferrous iron, manganese and cobalt, on the addition of ammonium hydroxide, is attributed to the formation of a complex chloride of the type $(NH_4)_2MCl_4$. This is certainly not the modern view.

The book is well produced, and will be of appreciable service to students.

HUBERT T. S. BRITTON.

ORGANIC SYNTHESES. Vol. II. J. B. CONANT, Editor-in-chief. Pp. 100. New York: John Wiley & Sons; London: Chapman & Hall. 1922. Price 7s. 6d. net.

The first volume of this series was reviewed in Vol. **47** of the ANALYST, April, 1922. Vol. II. is in all respects similar to its predecessor, and therefore requires little additional comment. Of the twenty-five syntheses described, nine or ten are likely to prove useful in the instruction of students who have successfully carried out the more elementary preparations usual in a course of organic chemistry. The yields claimed vary from 15 per cent. of theoretical in the case of mesitylene, to

99 per cent. in the case of nitroso-β-naphthol, and are over 80 per cent. in the majority of the syntheses.

The careful attention to detail, the clear diagrams of apparatus, and the excellent printing, which were notable features of the former volume, are to be found also in the present one. It is greatly to be hoped that the example set by the editors of this series will be followed by others, so that a more satisfactory literature of the subject may gradually be built up.

A. F. KITCHING.

FUNDAMENTALS OF BIO-CHEMISTRY IN RELATION TO HUMAN PHYSIOLOGY. By T. R. PARSONS. Pp. x.+281. Cambridge: W. Heffer & Sons, Ltd. 1923. Price 10s. 6d. net.

The reading of this book has been a real pleasure to the reviewer, to whom it at first appeared that the author had underestimated his task in writing what he describes "the most elementary of introductions to the subject." But these doubts vanished as the study of this very interesting book progressed, with the result that the reviewer does not hesitate for a moment to congratulate the author on the successful manner with which he has handled the subject. The material is well chosen, the important points are well illustrated and emphasised, and, above all, the different sections follow each other in logical sequence. All these points go far to show Mr. Parsons' abilities as a teacher; this is particularly evident from the choice of the bibliography for further study which is attached to each chapter. These bibliographies contain a great deal of first-rate material, and it remains only to be hoped that the average medical student has the energy and diligence to follow Mr. Parsons' prescription.

The reviewer's enthusiasm was, however, marred by the many errors which one notices in the book. Some of these errors are certainly only of a minor character, but, since the book is written for beginners, they ought to have been strictly avoided. It is not the reviewer's intention to summarise these errors, but special reference may be made to the very remarkable formula for lactic acid on page 83. Unfortunately this formula is followed by the following sentence: "It will perhaps be objected that we have arranged these molecules in such a way as to suit our immediate purpose," and the reviewer has to confess that it took him some time before he realised that Mr. Parsons' formula was a printer's error only, and not a special manipulation. However, there is no doubt that there will be a demand for a second edition, when it is sincerely hoped that these errors will be carefully corrected, which would greatly add to the value of the book.

In conclusion the reviewer would like to make a plea for the inclusion of Werner's formula for urea in the next edition. This formula has not only the advantage of giving the correct chemical expression to the properties of urea, but it is also a great help to the teacher, since it explains, for example, the reversibility of the conversion of urea into biuret, which is otherwise hard to explain and to understand.

M. NIERENSTEIN.

SYNTHETIC COLOURING MATTERS: DYESTUFFS DERIVED FROM PYRIDINE, QUIN-
OLINE, ACRIDINE, AND XANTHENE. By J. T. HEWITT, D.Sc., F.R.S.
Pp. 401. London: Longmans, Green & Co. 1922. Price 14s. net.

This is one of the volumes on *Synthetic Colouring Matters* included in the series of Monographs on Industrial Chemistry, edited by Sir Edward Thorpe.

It might appear difficult at first sight to put together a good-sized volume on the subjects indicated in the title alone, but a glance at the table of contents fully justifies the work having been made self-contained. The author, whose researches in several branches of the subject are well known, has elaborated a treatise of absorbing interest in which the purely theoretical part is clearly stated, while for the practical side of the question he has relied for much of the information on the patent literature, although he is careful to point out in the preface that a specification does not always contain the whole truth and nothing but the truth.

With the exception of quinoline yellow and the quinoline dyes (cyanines and isocyanines), which latter have proved so useful as sensitisers in photography, there is little of intrinsic practical value in the section coming directly under the first two headings of the title. The main interest from this point of view attaches to the following 14 chapters in which such important dyestuffs as phosphine acridine yellow and orange, benzoflavine and other acridine derivatives are fully discussed. The most important section of the work centres, however, in the very full account, under the heading xanthene, of the phthaleins, which include the whole range of the eosins, the rhodamines, anisolines and gallein. The question of the structure of fluorescein, in the light of the researches of Richard Meyer and others, is discussed at considerable length, and the view that all such dyestuffs are true fluoran derivatives appears fully justified. The stability of the complex towards drastic reagents is sometimes remarkable. Thus it has been observed that rhodamine B, after boiling for two hours with 100 per cent. sulphuric acid, still showed its characteristic intense colour, and it was only after prolonged boiling that the colour finally disappeared.

In this, as in several other recent monographs on colouring matters, the real practical issue, *i.e.* the use or uses of the dyestuffs, which is, after all, the thing that matters, is not stated fully enough. Thus there is no mention of the use of the eosins for the production of vermilionettes and other lakes, which purpose takes almost the whole output at the present time. As dyestuffs for textiles they have long since ceased to be of any importance. It might also have been of some interest to mention the titration of eosin and rhodamine by means of titanous chloride. Such titrations can easily be carried out, and afford means for the rapid and exact valuation of these colours, while at the same time adducing additional proof of the correctness of the views expressed regarding their constitution.

Great credit is due to Professor Hewitt for the able manner in which he has handled a somewhat difficult subject. The work, which is produced in the same useful style as other volumes of the series, will rank as a valuable addition to our scientific and technical literature on dyestuffs. E. KNECHT.

CEMENTS AND ARTIFICIAL STONE. By JOHN WATSON, M.A. Edited by R. H.
    RASTALL, Sc.D. Cambridge: W. Heffer & Sons, Ltd. 1922. Price 6s. net.

This little book is primarily a descriptive catalogue of specimens of cement
and artificial stone in the Sedgwick Museum at Cambridge. It is, however, much
more than a mere catalogue of the 136 specimens which are carefully described,
for it is, in addition, a very readable introduction to the technology of the subject,
written for the guidance of students of economic geology, who, in order to be in a
position to apply their specialised geological knowledge to industrial requirements,
must acquire a clear insight into the technical problems involved.

It is regrettable that the author died before the completion of the volume,
but the manuscript he left has been carefully edited by Dr. Rastall.

More than half the book is devoted to the history, properties, manufacture
and application of Portland cement, and covers the ground in a satisfactory
manner and interesting style. The remainder deals with Blue Lias lime, Selenitic
cement, plaster of Paris, etc. The book is well printed and free from errors—one
of the few misprints being "Bloxham" instead of "Bloxam," on pages 41 and 122.

The book calls for very little criticism. With regard to the objection that the
iron and steel employed in reinforced concrete may corrode, no mention seems to
be made of the method of "painting" girders, etc., with a "wash" of Portland
cement to prevent rusting, an example of which may be seen to-day in the skeleton
of the proposed Park Lane Hotel in Piccadilly. No allusion is made to artificial
stone produced by cements other than Portland, such as magnesian, hydro-
silicate, etc., but, in view of the ease with which Portland cement can be used for
this purpose, they are perhaps not of sufficient economic importance. It is a little
surprising that there is no reference to the recovery of potash in the manufacture
of Portland cement which was carried out during the war.

The book will be found useful to many besides students of economic geology,
and in some ways recalls the handbook to pottery and porcelain issued by the
authorities of the Museum of Practical Geology in Jermyn Street, with its excellent
introduction to the study of clays and ceramics. It is a type of book that could
be advantageously copied, for it emphasises the interdependence of science and
industry, and shows that even the older Universities are energetically engaged in
training men for the practical application of scientific methods and research.
The author did much for the promotion of the study of economic geology in
Cambridge, and the diffidence he felt in dealing with the technological aspects of
the subject is unnecessary in view of the excellent way in which he discharged the
task.   WALTER C. HANCOCK.

STARCH AND STARCH PRODUCTS. By H. A. AUDEN, D.Sc. Pp. 121. (Common
    Commodities and Industries Series.) London: Pitman & Sons. 1922.
    Price 3s. net.

This book, in common with others included in this well-known series, is
written chiefly for the general reader, and fully maintains the high standard
attained by earlier volumes. Chapter I. is devoted to an interesting account of

the uses of starch in olden times, and is followed by a chapter in which the general properties of this substance are described. The succeeding six chapters give admirable and complete accounts of the preparation of starch from many sources, such as potatoes, cereals, sago, cassava, and other plants less known in this country, but found in profusion in tropical parts of the world. The last three chapters deal with the bleaching and colouring of starch and its commercial conversion into dextrin, sugars and alcohol. The subject matter includes the history, folklore, chemistry, botany, technology, and other aspects of the subject woven into a compact and pleasing whole, and an extraordinary wealth of accurate information is compressed into a small volume.

The majority of the illustrations provided are good, but Figs. 5, 6 and 8 are badly reproduced, and thereby lose much of their value. It is doubtful whether the reproductions of manufacturing plant shown in Figs. 14 to 19 serve any useful purpose, since the general reader will gain nothing from them owing to the absence of any explanation in the text. Reference is made, on page 11, to the microscopical appearance of starch grains, but additional interest would have been given by the inclusion of photomicrographs or drawings of a few of the commoner starches under a magnification of approximately × 200.

Among the few doubtful statements deserving comment are the following: On page 12 the enzyme which converts maltose to glucose is referred to under the old name of "glucase," but, according to present nomenclature, this should be "maltase." In the preparation of malt from barley, described on page 43, it is stated that "diastase is usually at its maximum when the *rootlets* are about two-thirds of the length of the grain." Obviously acrospire or plumule should be substituted for rootlets. On page 91 the colorations produced by the action of iodine upon amylodextrin, erythrodextrin and achroodextrin are given as violet, red and yellow respectively, but page 97 gives the antagonistic information "that dextrins are no longer present, *i.e.* no blue colour results after the addition of iodine solution." This latter statement requires revision. Glucose certainly occurs in fruits, but it is not generally known as "fruit sugar," although this synonym is incorrectly given on page 96. Fruit sugar is applicable only to lævulose, but is an unsatisfactory term, the use of which should be avoided. Only two examples of mis-spelt words have been noticed throughout the volume, these occurring on page 70, where "weevil" is given as "weavil," and on page 121, where the second letter is missing from "alcohol."

It is particularly unfortunate that the general excellence of this volume should be marred by the unsatisfactory nature of the index, in which some four or five per cent. of the references are incorrectly paged. Considering the importance of this portion of the book, and the annoyance caused to the reader by such inaccuracies, it is essential that a drastic revision should be made in future editions.

This work contains much accurate and reliable information expressed in an interesting manner, and should be of value, not only to the general reader, but also to all interested in starch from an industrial or professional standpoint.

T. J. WARD.

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