Analytical Chemistry.

Photochemical Reactions in Laboratory Work. KURT
GEBHARD (*Chem. Zeit.*, 1910, 34, 1269).—The author points out that
whilst a slightly acid solution of potassium iodide in starch mucilage
remains colourless in the dark, it rapidly becomes coloured in the light,
due to liberation of iodine, although in presence of a fourth substance
liberation of iodine may occur more rapidly in the dark than in the
light.   This and observations recorded by others (for example, Gibbs,
Abstr., 1909, i, 640, and Batik, 1910, i, 543) indicate that as the
result of unsuspected photochemical reactions, errors may often be
caused in ordinary laboratory work.   T. A. H.

Several Acids Suitable for Use as Standards in Acidimetry.
JOSEPH H. KASTLE (*Amer. Chem. J.*, 1910, 44, 487—493).—*p*-Nitro-
toluene-*o*-sulphonic acid (Kastle, this vol., i, 30), and *p*-amino-*o*-
sulphobenzoic acid and potassium *o*-nitro-*p*-sulphobenzoate (Hart,
Abstr., 1881, 1144) are recommended as standards for use in
acidimetry.   The last two are anhydrous, and can be thoroughly
dried without risk of decomposition.   *p*-Nitrotoluene-*o*-sulphonic acid,
although crystallising with $2H_2O$, is a definite and very stable com-
pound.   All these substances are stable in the air, are not hygroscopic
or deliquescent, and give very sharp end-reactions with phenol-
phthalein.   E. G.

**Use of Metallic Potassium in Estimating the Halogens in
Benzene Derivatives.**   C. H. MARYOTT (*Amer. J. Sci.*, 1910, [iv],
30,   378—380).—Stephanoff (Abstr.,   1907, ii, 50)   proposed to
estimate the halogens by treatment with alcohol and metallic sodium.
The author did not get satisfactory results, but on substituting
potassium for sodium, a complete reduction was effected.   About
0·4 gram of the substance is placed in an Erlenmeyer flask, and
10—15 c.c. of alcohol-benzene mixture are added (1 vol. of 98% alcohol
with 2 vols. of benzene free from sulphur compounds).   About ten
times the theoretical amount of potassium is now added gradually in
small pieces.   When the action slackens, two extra c.c. of alcohol are
added, and when the potassium has completely dissolved, the whole is
shaken with water.   The aqueous layer is then acidified with nitric
acid, and the halogen is precipitated with silver nitrate as usual.
                                                             L. DE K.

Estimation of Total Sulphur in Urine.   W. DENIS (*J. Biol.
Chem.*, 1910, 8, 401—403).—Benedict's method (Abstr., 1909, ii, 827),
in which the oxidising agent is a mixture of copper nitrate and sodium
or potassium chlorate, was compared with Folin's sodium peroxide
method.   The former method leads to spattering and loss, due to too
rapid evolution of nitrogen oxides.   It can be modified, and the
spattering avoided by mixing the copper salt solution with sodium

chloride and ammonium nitrate ; the results then are very near to those obtained by Folin's method. W. D. H.

Benedict's Method of Estimating the Total Sulphur in Urine. CARL L. A. SCHMIDT (*J. Biol. Chem.*, 1910, **8**, 423—425. Compare Abstr., 1910, ii, 827).—Benedict's and Folin's methods gave practically the same results. The presence of sugar makes no difference, but albumin causes the oxidation by Benedict's method to be violent ; it makes no difference in Folin's method. W. D. H.

Estimation of Sulphur in Organic Compounds. THEODOR ST. WARUNIS (*Chem. Zeit.*, 1910, **34**, 1285—1286).—$0\cdot2$—$0\cdot4$ Gram of the finely powdered substance is mixed in a spacious silver or nickel crucible with 10 grams of powdered pure potassium hydroxide and 5 grams of sodium peroxide by means of a silver wire. After covering the crucible with a lid, the mixture is heated for some fifteen minutes in an air-bath at 75—80°, and then heated over a gradually increased flame until it has melted completely. The mass is then allowed to cool, and dissolved in water, solution of bromine in hydrochloric acid is added, and the liquid filtered and boiled to expel the excess of bromine. In the filtrate, the sulphuric acid is estimated as usual.

A blank experiment should be made to allow for any sulphate present in the reagents or sulphuric acid absorbed from the gas employed. L. DE K.

Estimation of Sulphur and Phosphorus. CHARLES G. L. WOLF and EMIL ÖSTERBERG (*Biochem. Zeitsch.*, 1910, **29**, 429—438).—The authors have investigated various methods for the estimation of sulphur in tissues. They show that satisfactory results can be obtained by preliminary oxidation of the organic matter, and completion of this process by the reagent originally suggested by Benedict containing copper nitrate and potassium chlorate. They show also, that after destruction of the organic matter and precipitation of the sulphate in this way, the phosphorus can be estimated in the filtrate, so that only one sample is necessary for the estimation of both sulphur and phosphorus. S. B. S.

Remarks on the Iodometric Titration of Acids and Kjeldahl's Nitrogen Estimation. R. KOEFOED (*Zeitsch. physiol. Chem.*, 1910, **69**, 421—440).—Correct results are obtained by strictly adhering to the procedure given. The ammoniacal distillate is collected in a flask containing 15 c.c. of N-sulphuric acid until the total volume measures 100 c.c. When making the check, 15 c.c. of the acid are diluted up to 100 c.c. with water previously boiled. Ten c.c. of 5% potassium iodide, 2 c.c. of 2% starch solution (saturated with sodium chloride), and, finally, 4 c.c. of 4% potassium iodate are added. The iodine liberated, which represents the free acid, is then titrated with $N/10$-sodium thiosulphate, which operation should occupy just two minutes.

The author has tried the various chemicals from time to time proposed as standards, including glycine and hippuric acid after conversion into ammonium sulphate by Kjeldahl's process. The results

which are tabulated, do not differ much, except those with glycine
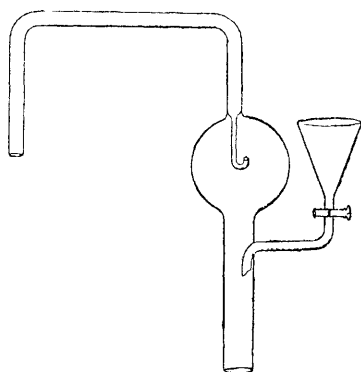and hippuric acid, which give somewhat higher figures.

In most cases, Kjeldahl's original process (use of potassium perman-
ganate) is recommended; in such cases where there is danger of forma-
tion of piperidine rings, the Gunning modification (use of potassium
sulphate) is more appropriate.   L. DE K.

Error in Estimating Nitrogen in Soil. EILHARD A.
MITSCHERLICH and ERNST MERRES (*Chem. Zentr.*, 1910, ii, 495; from
Landw. Jahrb., 1910, **39**, 345—367).—In soil sampling, a sample should
be taken by means of a borer from each square metre; according to the
soil, the error will vary from ± 4 to 6%. The error due to changes
during the drying of soils in the air can be avoided by adding tartaric
acid. Soil extracts should be kept under carbon dioxide.

                                                        N. H. J. M.

**Distillation Arrangement for Ammonia and Nitrogen
Estimations.** CARL MÜLLER (*Chem. Zeit.*, 1910, **34**, 1308).—The
apparatus (see figure) is placed on
the distilling flask, and the reducing
agent and the sodium hydroxide
solution are admitted through the
funnel. In this manner there is
no loss of ammonia to be feared
owing to gases given off, as these
have to pass through the acid in
the receiver.   L. DE K.

**Folin's Method for the Esti-
mation of Urinary Ammonia
Nitrogen.** MATTHEW STEEL (*J.
Biol. Chem.*, 1910, **8**, 365—379.
Compare Abstr., 1908, ii, 776).—In
urines containing large quantities
of ammonio-magnesium phosphate, there is in Folin's method an
incomplete decomposition of this substance, and so the yield of
ammonia is too low; the error may exceed 50%. Some alkali must be
added which will liberate all the ammonia from the phosphate but
not from other substances, such as urea. The plan adopted is to
substitute sodium hydroxide for sodium carbonate. If plenty of
sodium chloride is added also, urea and other substances are not
decomposed. For the 3 grams of sodium carbonate employed by
Folin, 0·5 to 1 gram of sodium hydroxide plus 15 grams of sodium
chloride are substituted.   W. D. H.

Nitrometer Appendage Flasks. FRANZ MICHEL (*Chem. Zeit.*,
1910, **34**, 1237).—The H-vessel (see figure), which dispenses with the
use of the pipettes and funnels, is used as follows: Tube *a*, which for
nitrogen estimations carries two marks at 25 and 50 c.c. respectively,
contains the alkaline hypobromite solution. Tube *b* also carries two
marks of 5 and 10 c.c. respectively, and is intended for the substance

to be tested. The diameters of the tubes are so chosen that their length is about equal. If a solid substance, such as an ammonium salt, has to be tested, a weighed quantity is placed in tube b, and water is added up to the desired mark. Liquids are introduced up to the desired mark, or may be delivered from a pipette. After closing the tubes (a with a rubber cork, b with a stopcock tube), the pressure in both tubes is equalised as usual. To start the reaction, the apparatus is inclined in such a manner that the liquid from b gradually enters a, and when the effervescence has ceased, the liquid is allowed to again enter b, and this operation is repeated a few times. When all the liquid is once more in tube a, the apparatus is placed in a horizontal position, a downwards, and well shaken. In this manner it is impossible for any liquid to enter the stopcock tube. L. DE K.
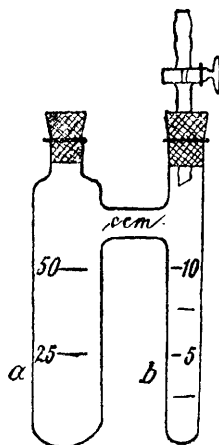
Application of Busch's "Nitron" Method to the Analysis of Chili Saltpetre. LEOPOLD RADLBERGER (*Chem. Zentr.*, 1910, ii, 685, 686 ; from *Osterr.·ung. Zeitsch. Zucker-Ind. Landw.*, 1910, **39**, 433—436).—Busch's method (Abstr., 1905, ii, 282) may be employed for estimating the quantity of nitrate in Chili saltpetre provided that this contains not more than the normal amount of perchlorate. If the salt contains a large amount of perchlorate, the results obtained by the process are untrustworthy.

W. P. S.

Analysis of Nitrates by Grandval and Lajoux's Method. Estimation of Nitrates in Water by a Sulphosalicylic Reagent. HUBERT CARON and DÉSIRÉ RAQUET (*Bull. Soc. chim.*, 1910, [iv], **7**, 1021—1025, 1025—1027).—In the first paper the modified form of Grandval and Lajoux's process introduced by Perrier and Farcy (Abstr., 1909, ii, 344) is criticised, and in the second the use of a solution of salicylic acid in sulphuric acid is suggested in place of phenol dissolved in sulphuric acid as a colorimetric reagent for nitrates.

It is stated that the coefficients of decolorisation quoted by Farcy (Abstr., 1909, ii, 616) do not agree with those calculated from the results given in his previous paper (Abstr., 1909, ii, 344). Further, the dry residue from the water is liable to absorb moisture, and unless this is guarded against, different results are obtained by different workers for the same proportion of nitrates. The results obtained vary also with the method used in preparing the solution of phenol in sulphuric acid, and the authors recommend the use of a reagent freshly prepared by dissolving 1 c.c. of the liquefied phenol, specified in the Codex Français, in 10 c.c. of sulphuric acid.

In the second paper, it is pointed out that several phenolic substances dissolved in sulphuric acid give colours with nitrates, and a solution of from 1 to 5% of salicylic acid in sulphuric acid is suitable

for use in estimating nitrates in water.   The process suggested is as follows : Evaporate 10 c.c. of the water to dryness with 1 c.c. of a 1% solution of sodium salicylate ; add to the residue 1 c.c. of sulphuric acid, and, after mixing thoroughly for some minutes, add 10 c.c of water and 10 c.c. of ammonia, and compare the colour given with that furnished by a known nitrate solution similarly treated.   The quantity, X, of nitrate in the water is given by the formula :

$$X = Ph/h' \times V'/V,$$

where h and h' are the respective heights of the two solutions giving the same tint in the colorimeter, V and V' the original volumes of the two solutions, and P the amount of nitrate in the standard solution.
                                                                                                        T. A. H.

Estimation of Free Carbon Dioxide in Water.   J. TILLMANS and O. HEUBLEIN (*Zeitsch. Nahr. Genussm.*, 1910, **20**, 617—630).— Titration with calcium hydroxide solution yields trustworthy results, provided that phenolphthalein is used as the indicator.   Rosolic acid is quite useless for the purpose, as the hydrogen carbonates of the alkalis and alkaline earths exhibit a strongly alkaline reaction towards this indicator.   W. P. S.

Acid Content of Moor Water.   H. STREMME (*J. pr. Chem.*, 1910, [ii], **82**, 519—520).—It is pointed out that Endell in his paper on the acidity of moor waters (Abstr., 1910, ii, 1005) has worked according to the method first given by the author two years ago (*Zeitsch. prakt. Geol.*, 1908, 126).   T. S. P.

Separation of Alumina and Ferric Oxide.   PHILIPPE BARBIER (*Bull. Soc. chim.*, 1910, [iv], **7**, 1027—1028).—The material is dissolved in the ordinary way, excess of sodium acetate added, and the acids neutralised by sodium hydroxide.   A 10% aqueous solution of sodium hyposulphite is then added until the red coloration of the liquid just disappears.   The mixture is boiled, when alumina is precipitated as a dense powder, which can be collected and weighed as usual.   Iron can be estimated in the filtrate by any of the ordinary methods.   Glucina is precipitated with the alumina if present, and can be separated by digesting the precipitate with ammonium carbonate solution during thirty-six hours, when the alumina remains undissolved, and the glucina can be recovered quantitatively by prolonged ebullition of the solution.   T. A. H.

Estimation of Manganese in Steel by the Volhard-Wolff Method.   AUGUST KAYSSER (*Chem. Zeit.*, 1910, **34**, 1225—1226).— —One gram of steel is boiled with 25 c.c. of hydrochloric acid, D 1·12.   When solution is complete, a 1 gram tablet of compressed potassium chlorate is added, and the boiling continued until the odour of chlorine has passed off.   The liquid is transferred to a flask, and, after precipitating the iron with zinc oxide, avoiding an excess, the hot solution is titrated with potassium permanganate solution (1·9 gram per litre ; 1 c.c. = 0·1% of manganese in the sample).   When dealing with samples rich in carbon, more potassium chlorate should be used.   L. DE K.

Method of Dissolving Tinstone. ADOLPH GILBERT (*Zeitsch. offentl. Chem.*, 1910, 16, 441—442).—Natural tinstone is much more refractory than artificial stannic oxide towards fusion with alkali. If 0·5—1 gram tinstone is fused with 10—15ᵉ grams of sodium hydroxide in a silver crucible, a little finely-powdered wood charcoal being added (50 mg.), a reaction takes place with considerable development of heat, and solution is complete in three to five minutes. The heating must be continued in order to burn off the excess of carbon. All the tin passes into solution in water, iron oxide remaining insoluble. It is desirable to remove copper and sulphur by a preliminary roasting and extraction with hydrochloric or nitric acid. The quantity of carbon used is only a small fraction of that required to reduce the tin oxide.

The reaction has been described by Burghardt (Abstr., 1890, 1027), but has remained unnoticed in the literature. C. H. D.

Precipitation of Vanadic Acid as Silver Vanadate and Estimation of Phosphoric and Vanadic Acids in the Presence of One Another. GRAHAM EDGAR (*Amer. Chem. J.*, 1910, 44, 467—472).—The solution of the alkali vanadate which may contain excess of alkali carbonate, but no other matters precipitable by silver nitrate, is acidified with nitric acid, and boiled to expel carbon dioxide. Dilute sodium hydroxide is then added to the boiling solution until the yellow liquid turns colourless. An excess of $N/10$-silver nitrate is added, and then ammonia to dissolve the precipitate formed, and the excess of ammonia is boiled off ; a few fragments of porous tile should be added to prevent bumping. The silver vanadate is collected on a filter, and well washed, and then the excess of silver is estimated in the usual manner by acidifying the filtrate with nitric acid and titrating with $N/10$-ammonium thiocyanate with ferric nitrate as indicator. One c.c. of silver consumed $= 0·00304$ gram of vanadic acid.

In the presence of phosphoric acid, this is co-precipitated as tri-silver phosphate with the vanadic acid. The latter is then estimated separately by dissolving the precipitate in sulphuric acid and reducing the vanadic acid by boiling with sulphur dioxide. The vanadium is titrated hot with $N/20$-permanganate and the phosphoric acid is found by an easy calculation. L. DE K.

An Indirect Method for Estimating Columbium and Tantalum. HARRY W. FOOTE and RALPH W. LANGLEY (*Amer. J. Sci.*, 1910, [iv], 30, 393—400).—The process is based on the great difference between the densities of the oxides of columbium (4·552) and tantalum (8·716).

When applied to the mineral stibiotantalite, a sufficiency of the sample is treated with hydrofluoric acid, the solution is largely diluted with water, and the antimony and bismuth are removed by means of hydrogen sulphide ; the precipitate is then treated further by the usual process. The filtrate is evaporated to dryness, and the residue heated with sulphuric acid to expel the fluorine. After diluting with water, the solution is made alkaline with ammonia, and the precipitate is collected, washed, ignited, and weighed After heating over a blast

lamp for an hour, the density of the mixed oxides is determined and their composition ascertained by referring to a table.   L. DE K.

A Recent Method for Separating Tantalum and Columbium. HARRY W. FOOTE and RALPH W. LANGLEY (*Amer. J. Sci.*, 1910, [iv], 30, 401—402).—Weiss and Landecker (Abstr., 1909, ii, 942) have stated that tantalic acid may be precipitated from its solution in sodium carbonate by means of a current of carbon dioxide ; any columbic acid remains in solution, and may be separated by boiling with sulphur dioxide.   The authors, however, agree with Rose and with Ostwald that the columbium is almost completely co-precipitated with the tantalum, and that, therefore, this process cannot be employed for their separation.   L. DE K.

Estimation of Both Phenol and *p*-Cresol in Urine. MAX SIEGFRIED and R. ZIMMERMANN (*Biochem. Zeitsch.*, 1910, 29, 368—388). —The method is based on two determinations : (1) the total amount of bromine required to convert the phenol and *p*-cresol in the liquid into tribromophenol and tribromo-*p*-cresol, and (2) the quantity required to convert the phenol into tribromophenol and the cresol into dibromo-*p*-cresol.

Since 6 atoms of bromine are necessary for the formation of either of the tribromo-compounds, whilst that of dibromo-*p*-cresol only requires 4 atoms, the quantities of phenol and *p*-cresol present are readily calculated from the difference between the two determinations.   For the first estimation, a modification of Koppeschaar's method is employed.   Twenty to 30 c.c. of sulphuric acid $(1:1)$ are added to a measured volume of the liquid contained in a stoppered flask, and a standard solution of potassium bromide and bromate $(0\cdot834$ gram $KBrO_3$ and $2\cdot97$ grams KBr per 1000 c.c.) is run in from a burette, the liquid being continually agitated, until the precipitate collects together into a mass, and the liquid becomes coloured distinctly yellow.   At this point, the volume that has been added is noted, and an eighth part of this volume is further run in.   The mixture is then shaken at frequent intervals for one hour.   It is then filtered through glass-wool into 25 to 30 c.c. of 5% potassium iodide solution, and the iodine liberated titrated with $N/10$ thiosulphate.

The second reaction is carried out with the same quantity of the original liquid, 30 c.c. of 25% hydrochloric acid are added, and the whole diluted to 500 c.c.   The volume of the solution of potassium bromate and bromide required to produce a yellow coloration, as previously determined, is then run in, the liquid being gently rotated throughout, and the mixture left without shaking for fifteen minutes. Twenty-five to 30 c.c. of 5% potassium iodide are added, and the flask left for one hour in front of a light.   The iodine liberated is titrated with thiosulphate.   The method gives good results.

A new *tribromo*-p-*cresol* was obtained by the action of potassium bromide and bromate on *p*-cresol in the presence of hydrochloric acid, and the subsequent addition of potassium iodide ; it crystallises from acetic acid in colourless needles, m. p. 139°.   W. J. Y.

An Improvement in Scherer's Reaction for Inositol. ERNST SALKOWSKI (*Zeitsch. physiol. Chem.*, 1910, 69, 478—481).—A trace of inositol is dissolved in a few drops of nitric acid, a drop of 10% calcium chloride solution added, and then a drop of 1% platinum chloride solution; the whole is carefully evaporated in a porcelain capsule. A red colour develops; on the addition of water it becomes orange; the red returns on heating once more, with a blue tinge; on stronger heating, it becomes a dirty green. Performed in this way, the reaction is more delicate, and the colours more intense. W. D. H.

A Simple Method for the Estimation of Sugar in Blood. LEONOR MICHAELIS and PETER RONA (*Zeitsch. physiol. Chem.*, 1910, 69, 498).—Polemical against K. Moeckel and E. Frank (Abstr., 1910, ii, 1116). W. D. H.

Reischauer's Titration Process for the Estimation of Diabetic Sugar. GUSTAV OTTO GOEBEL (*Apoth. Zeit.*, 1910, 25, 614—615).—Into six separate test-tubes are introduced the usual quantity of Fehling's solution and water, and to each is added an increasing amount of the urine to be tested. After being heated in the boiling water-bath for fifteen minutes, they are cooled rapidly, shaking being avoided. They are now all tested for excess of copper in the following manner: 0.5 gram of potassium iodide dissolved in a little water is added, and, after gentle shaking, 5 c.c. of dilute sulphuric acid ($1:5$). Excess of copper will be noticed by the formation of cuprous iodide, and also free iodine, which may be recognised by adding starch solution; no notice should be taken of a blue colour appearing gradually.

The contents of the tube which show no excess of copper are those where about the right amount of urine has been added, and serve as a guide for a second trial, when a little less urine should be used.

L. DE K.

Estimation of Sugar by Safranine. K. A. HASSELBALCH and J. LINDHARD (*Biochem. Zeitsch.*, 1910, 29, 416).—The authors, in reply to Wender's criticism (Abstr., 1910, ii, 1116), maintain that their safranine method for the estimation of sugar (Abstr., 1910, ii, 905) is new, in that they so elaborated the details that the reaction can be used quantitatively, and not merely qualitatively. S. B. S.

Polarimetric Estimation of Lactose. H. DROOP RICHMOND (*Analyst*, 1910, 35, 516—517).—Of the many substances which have been proposed for the removal of proteins previous to the polarimetric estimation of lactose in milk, that most commonly used is mercuric nitrate, but the author finds that this reagent does not effect complete precipitation of all the proteins present. If the filtrate obtained from milk which has been treated with mercuric nitrate is further treated with phosphotungstic acid, a somewhat voluminous precipitate is obtained, and the optical activity of the solution is increased. In the case of milk itself, the difference in the polarisation, before and after treatment with phosphotungstic acid, is small, but with dried milks and other milk products the difference may amount to several units

per cent. As, however, the use of mercuric nitrate is so convenient and involves such a small dilution, the author recommends that its use be continued in conjunction with the addition of phosphotungstic acid. The milk should be treated with mercuric nitrate in the usual manner ; after the addition of 5% of phosphotungstic acid and 5% of sulphuric acid (1 : 1), the mixture is filtered, and the filtrate is examined in the polariscope. The readings observed are multiplied by 1·1.   W. P. S.

Estimation of Lactose in the Presence of the Commonly-occurring Sugars. JULIAN L. BAKER and H. F. E. HULTON (*Analyst*, 1910, 35, 512—514).—The method proposed depends on the fact that brewers' yeast ferments such sugars as dextrose, invert sugar, maltose, and sucrose, whilst lactose is not affected. The estimation is carried out by adding about 0·5 gram of washed brewers' yeast to 100 c.c. of a 2—3% solution of the mixed sugars, allowing fermentation to proceed for seventy hours at a temperature of 27°, then filtering and boiling the solution, and determining its copper-reducing power. If the sugar solution is non-nitrogenous, a small quantity of sterile yeast water or asparagine may be added. Lactose alone appears to be slightly attacked by the yeast, results of experiments showing that from 90—95% of the quantity present is found at the end of the fermentation process, but in the presence of other sugars the loss of lactose is much less. The fermentation must not be prolonged for more than seventy-two hours, as after this period of time bacteria develop, the solution becomes acid in reaction, and the lactose diminishes rapidly. The process is particularly suitable for the estimation of lactose in the presence of flour, for instance, in infants' and invalids' foods ; when sucrose is also present, it is advisable to invert this sugar with invertase or citric acid, before proceeding with the estimation of the lactose. The method is not, however, applicable in the case of foods, etc., containing commercial sugars, such as dextrin-maltose, glucose, and the like, as these, even after fermentation in the presence of diastase, leave a residue which reduces Fehling's solution.
                                                                    W. P. S.

Estimation of Lactose in Milk. VITOUX (*Ann. Falsif.*, 1910, 3, 471—472).—It is shown that the process described by Denigès (treatment of the milk with sodium metaphosphate and hydrochloric acid, filtering, and determining the cupric reducing power of the filtrate) yields results which agree closely with those obtained by the official (French) method of estimating lactose in milk. The latter method is, however, to be preferred for the analysis of milks which have been preserved by the addition of dichromate, as the end-point of the titration with Fehling's solution is not obscured.   W. P. S.

New Method for the Quantitative Estimation of Sucrose in the Presence of other Sugars. ADOLF JOLLES (*Zeitsch. Nahr. Genussm.*, 1910, 20, 631—638).—The method is based on the fact that sugars, such as arabinose, rhamnose, dextrose, lævulose, galactose, mannose, invert sugar, maltose, and lactose, are rendered optically

inactive when heated in dilute alkaline solution, whilst sucrose remains
unaffected. The reaction to some extent depends on the concentration
of the sugar and alkali; for instance, a 1% dextrose solution containing
sufficient sodium hydroxide to render the alkalinity of the whole
approximately $N/100$ becomes optically inactive after being heated at
a temperature of 37° for twenty-four hours. The solution, however,
then becomes acid in reaction, and to avoid this, it is recommended
that the solution have an alkalinity approximately $N/10$ and contain
not more than 2% of the above-mentioned sugars; the quantity of
sucrose present does not matter. Instead of heating at 37° for twenty-
four hours, the mixture may be boiled in a reflux apparatus or
heated in a closed flask in a boiling-water bath for thirty minutes, but
the lower temperature is to be preferred, as the solution becomes less
darkly coloured. As applied to the estimation of sucrose in wine, con-
densed milk, etc., the details of the process are as follows: A definite
quantity of the sample is neutralised, clarified by the addition of lead
acetate, filtered, and the excess of lead removed from the filtrate by
means of sodium phosphate. After removing the lead phosphate by
filtration, an aliquot portion of the filtrate is treated with a sufficient
quantity of sodium hydroxide solution to render the alkalinity of the
solution equivalent to $N/10$, and the mixture is then heated as
described. After cooling, the solution is examined in the polariscope;
any rotation observed is due solely to sucrose, and the quantity of the
latter in the sample is then calculated. The results obtained by the
process agree closely with those yielded by the ordinary polarimetric
method of estimating sucrose. W. P. S.

**A Modification of the Furfuraldehyde Method of Estimating
Pentosans.** K. H. Böddener and Bernhard Tollens (*J. Landw.*,
1910, **58**, 232—237).—It is usual to estimate pentosans by distilling
with hydrochloric acid, collecting the furfuraldehyde formed, and then
converting this into a weighable substance by precipitating with phloro-
glucinol in the cold, leaving the mixture for sixteen to twenty hours.
The authors find that precipitation is practically complete in a much
shorter time if the mixture is heated to 80—85°, and then allowed to
cool for one and a-half to two hours; bluish-green flocks are thus
formed, which are readily filtered and washed. The phloroglucide is
not the same as is obtained in the cold, the reaction being $C_5H_4O_2 +
C_6H_6O_3 = C_{11}H_4O_2 + 3H_2O$, whilst in the cold the products are
$C_{11}H_6O_3 + 2H_2O$. The necessary tables are given for the conversion.
The modification is not suitable if it is desired to estimate the methyl-
pentosans separately; further (this vol., ii, 63), it does not give identical
results with the older method, but the differences are only small. Its
great value is the considerable saving of time it effects. E. J. R.

Estimation of Starch. Friedrich Schubert (*Chem. Zentr.*,
1910, ii, 688—689; from *Österr.-ung Zeitsch. Zucker-Ind. Landw.*,
1910, **39**, 411—422).—The author discusses the more important
processes which have been proposed for the estimation of starch, and
describes the following modification of Lintner's process for the
estimation of starch in barley (Abstr., 1907, ii, 823): A weighed

quantity of the barley is rubbed down with 25 c.c. of a 1% phospho-tungstic acid solution, 75 c.c. of hydrochloric acid are then added, and, after the lapse of thirty minutes, the solution is poured through a filter.   As exactly 100 c.c. of reagents have been added to the barley, there is no need to dilute the mixture to any definite volume, but the optical rotation of the filtrate must be corrected for the quantity of water present in the barley and for the volume of the dissolved starch.   One gram of pure starch occupies a volume of 0·49 c.c.

W. P. S.

Estimation of Acetaldehyde by means of Pyrrole and Application of this Method to the Estimation of Lactic Acid. W. SOBOLEWA and J. ZALEWSKI (*Zeitsch. physiol. Chem.*, 1910, 69, 441—451).—Into a number of test-tubes is placed the reagent, consisting of 5 c.c. of aqueous pyrrole and 10 c.c. of 2·2% hydrochloric acid.   Into each of the tubes is introduced an increasing volume of acetaldehyde solution of known strength (about 0·25 gram per litre), and after half an hour the minimum quantity of acetaldehyde required to produce an opalescence is ascertained.   This then serves as a measure for the estimation of the concentration of acetaldehyde solutions.

On boiling a lactate with dilute sulphuric acid in a current of air and cautiously adding potassium permanganate, 80—92% of the lactic acid is obtained as acetaldehyde, which may then be estimated by the pyrrole method.   L. DE K.

Titanium Chloride in Volumetric Analysis. IV. Estimation of Quinones.   EDMUND KNECHT and EVA HIBBERT (*Ber.*, 1910, 43, 3455—3457).—Quinones are reduced by titanium chloride in the cold.   To the solution of the quinone in cold water is added an excess of the titanium trichloride, and the excess of the latter determined by titration with iron alum, using potassium thiocyanate as indicator.   The titration of the quinone may also be carried out directly with the titanium chloride, using methylene-blue as an indicator ; the quinone is quantitatively reduced before the methylene-blue enters into reaction and is decolorised.

Analytical results are given with p-benzoquinone, toluquinone, and β-naphthaquinone.

In a footnote it is pointed out that methylene-blue may be used as an indicator instead of potassium thiocyanate in the titration of ferric salts.   T. S. P.

Acids in Tan Liquors.   HENRY R. PROCTER and A. SEYMOUR-JONES (*J. Soc. Chem. Ind.*, 1910, 29, 1354—1362).—The estimation of the acids present in tan liquors is of technical importance, since these substances act on hide in such a way as to facilitate the absorption of tannin.   Such action is confined to the relatively strong acids, and is not exhibited by weak acids, such as the phenols, and for that reason the method of estimation must discriminate between the two classes.   Many methods have been devised for this purpose, and in the present investigation the best known of them have been examined, and the results obtained are discussed and criticised.

Among the methods available, the discrimination referred to above is effected in one of two ways, (a) the tannins and other weak acids are removed by precipitation with gelatin, hide powder, or some similar agent, and the acids in the filtrate estimated by titration with standard alkali, using a suitable indicator, or (b) liquors are titrated directly with standard alkali in presence of an indicator, which will react with the stronger acids only. A special case of class (a) is the " lime-water" method, which depends on the fact that lime forms soluble salts with the stronger acids, and insoluble salts with the tannins, phenolic matters, etc., the formation of a definite faint turbidity being taken as the end-point. The general objections to methods of class (a) are (1) co-precipitation of stronger acids, and (2) undue dilution of the liquors. Methods of class (b) present as chief difficulty the selection of a suitable indicator. The authors think that the best results are obtainable with methods of group (b), and they are now engaged in the investigation of suitable indicators. The most promising results were obtained with Congo-red (10^{-4}), methyl-orange (10^{-4}), hæmatein $(10^{-1}$ to $10^{-15})$, and fluorescein $(10^{-5}$ to $10^{-7})$. The figures given in brackets after the name of each indicator represent the concentration of hydrogen ions at which colour change occurs.

T. A. H.

Separation of Saturated Fatty Acids (Palmitic and Stearic) from Oleic Acid. PIETRO FALCIOLA (*Gazzetta*, 1910, 40, ii, 217—229). —See this vol., i, 5.

Estimation of Total Tartaric Acid by the Goldenberg Process. CHARLES ORDONNEAU (*Bull. Soc. chim.*, 1910, [iv], 7, 1034—1041).—In order to ascertain the effects on the analytical results obtained by the Goldenberg method for the examination of factory and commercial tartaric products of impurities likely to occur in such materials, the author has applied the 1907 modification of that process (Abstr., 1908, ii, 237) to potassium hydrogen tartrate containing known amounts of various impurities.

With calcium tartrate alone the results are 3·53% too low when phenolphthalein is used as indicator, and 3·60% for litmus, whilst with potassium hydrogen tartrate containing 5 and 10% of calcium tartrate, the corresponding losses are 2·24 and 2·39% for litmus and 1·25 and 1·34% respectively for phenolphthalein, but these losses are somewhat reduced by the presence of alumina, although the loss due to the presence of 5% of the latter alone is 19·35% for litmus and 8·80% for phenolphthalein. Similar errors are caused by ferrous, ferric, and aluminium phosphates. The errors caused by these and other substances are tabulated in the original. In general, the losses are about twice as great for litmus as for phenolphthalein, and an approximation to the correct figure may be got as a rule by adding to the result obtained with phenolphthalein the difference between this result and that furnished by the use of litmus as an indicator, although a further correction is needed in some cases. Phenolphthalein may be used in all cases in place of litmus if the titration is carried out at 50°, after boiling and cooling the liquid. Certain of the changes suggested

by the author have been made in the most recent form of the Golden-berg process, adopted by the London Congress of Applied Chemistry (Abstr., 1910, ii, 758).   T. A. H.

Detection of Hydrocyanic Acid in an Exhumed Corpse and the Stability of Hydrocyanic Acid in Presence of Putrefying Matter. WILHELM AUTENRIETH (*Ber. pharm. Ges.*, 1910, 20, 432—446).—It is generally assumed that hydrocyanic acid disappears rapidly in animal organisms by (1) combination with dextrose in the blood ; (2) interaction with proteins ; (3) hydrolysis to formic acid, or (4), according to Ganassini (Abstr., 1904, ii, 758), conversion into xanthine derivatives. The detection of the acid in a child's corpse which had been exhumed after forty-three days lead the author to investigate the stability of hydrocyanic acid in presence of decomposing matter of various kinds, when it was found that, although the acid disappears, it does so comparatively slowly.

In a mixture of pig's stomach, intestine, and pancreas (50 grams) with bitter almond water (50 grams) and water (200 c.c.), hydrocyanic acid could still be detected by the Prussian blue, guaiacum, or thiocyanate tests after forty-five days, and this was also the case when dilute potassium cyanide solution was substituted for bitter almond water. When the quantity of acid initially present was reduced to 1 in 15,000 it could still be detected after thirty-six days. One part of the acid in 10,000 of cow's blood could still be detected by the Prussian blue reaction after forty-eight days.

In a series of quantitative experiments, an aliquot part of the mixture was distilled after twenty and also after sixty days, and the acid estimated by Liebig's method. The percentages of the acid originally present, found after twenty days, varied from 47·7 to 87·5, and after sixty days from 41·4 to 63·3. No evidence of more rapid disappearance of the acid in presence of dextrose or proteins could be obtained, and in this respect the behaviour of the acid seems to be different in the dead and living organism, probably because active enzymes are present in the latter case.   T. A. H.

Influence of the Alkalinity of the Wash-Water on the Percentage of Water in Butter. W. MEYERINGH (*Chem. Weeklad*, 1910, 7, 951—953).—Butter washed with acidified water appears to have a lower percentage of fat than that washed with alkaline water.   A. J. W.

Reactions of Cyclic Amines. F. LAVILLA LLORENS (*Anal. Fis. Quim.*, 1910, 8, 127—132).—The following bases give characteristic colorations when oxidising agents, such as potassium permanganate, potassium ferricyanide, potassium or ammonium persulphate, are added to their solutions in dilute acid. Potassium permanganate is the best reagent to use, a few drops of a 0·2% solution developing a coloration rapidly.

Aniline : greyish colour, initially, then more or less reddish-violet tinge, which changes to a characteristic blue ; this, after a long time, takes on a greenish hue.

Methylaniline gives the same indications as aniline.

o-*Toluidine*: in absence of much free acid, a characteristic blue coloration, distinct from that of aniline ; in presence of an increasing proportion of hydrochloric acid, the colour changes to bluish-green or yellowish-grey.   With acetic acid, even in concentrated solution, only the blue shade appears.   The coloration given by aniline is nearly independent of the amount of free acid, but with the toluidines and the following bases it varies greatly according to the concentration of the acid.

m-*Toluidine* : initially reddish coloration, changing to blue.

p-*Toluidine* : final coloration, red.

o-4-*Xylidine* : red colour, slightly violet in shade.

m-4-*Xylidine* : red colour, more violet than with *o* 4-xylidine.

p-*Xylidine* : red colour.

a-*Naphthylamine* : blue coloration ; *β-naphthylamine* : greenish-grey coloration, only faint.   W. A. D.

Estimation of Urea.   STANLEY R. BENEDICT (*J. Biol. Chem.*, 1910, 8, 405—421).—The Folin method yields traces of ammonia nitrogen from creatinine, rather more from uric acid, and allantoin yields all its nitrogen.   The method recommended is to add to the urine, potassium hydrogen sulphate, zinc sulphate, a piece of paraffin, and a little powdered pumice.   The mixture is boiled to dryness, and then placed in a sulphuric acid bath at 162—165° for an hour.   The contents are then washed with water into a distilling flask, sodium hydroxide added, and distillation continued for forty minutes with standard acid.   The residual acid is then titrated, and the urea nitrogen calculated.   W. D. H.

New Reactions of Morphine.   GEORGES DENIGÈS (*Compt. rend.*, 1910, 151, 1062—1063).—Ten c.c. of a solution of morphine or one of its salts (minimum concentration 0·03 gram per litre) are treated with 1 c.c. of hydrogen peroxide (5—12 vols.) and 1 c.c. of ammonia. After shaking, one drop of a solution of copper sulphate containing 1—4% of the crystallised salt is added.   On shaking again, the liquid develops a colour, varying from rose-pink to intense red, according to the amount of morphine present.

Sparingly soluble morphine salts should be dissolved in a few drops of hydrochloric acid before dilution.   The reaction gives positive results with certain morphine derivatives, such as *apo*morphine or heroine, but not with codeine, thebaine, papaverine, narceine, or narcotine.   It has the advantage of being applicable to solutions containing organic matter, such as sugar, and to plant extracts.

W. O. W.

Volumetric Estimation of Quinine in Drugs, etc.   JULIUS KATZ (*Ber. Deut. pharm. Ges.*, 1910, 20, 316—329).—In the method described, the quinine (or cinchona alkaloids) is converted into its hydrogen salt by evaporating its alcoholic solution with an excess of hydrochloric acid ; sodium chloride is added to aid the evaporation of the excess of the acid, and the quinine hydrochloride is then titrated with alcoholic potassium hydroxide solution, using Poirier's Blue as

indicator.   The details of the process as applied to the estimation of quinine alkaloids in cinchona bark are as follows : 6 grams of the dry and powdered bark are shaken for thirty minutes with 15 grams of chloroform and 5 grams of 5% sodium hydroxide solution ; 1 gram of magnesia and 45 grams of ether are then added, and the mixture is poured on a filter.   Forty grams of the filtrate are evaporated to a volume of about 1 c.c., and the residue is rinsed into a porcelain basin with alcohol ; after the addition of 10 drops of hydrochloric acid and 0·25 gram of sodium chloride, the mixture is evaporated to dryness.   The residue, after being dried for fifteen minutes in the water-oven, is dissolved in alcohol, 5 drops of a 0·2% Poirrier's blue solution are added, and the solution is titrated with $N/10$-alcoholic potassium hydroxide solution.˙   Each c.c. of the latter solution is equivalent to 0·0162 gram of quinine.   W. P. S.

Caution as to Testing for " Saccharin " in Sweetened Foods and Beverages.   EZIO COMANDUCCI (*Boll. chim. farm.*, 1910, 49, 791).—When solutions of dextrose and sucrose were extracted with a mixture of equal volumes of light petroleum and ether, it was found that the residue after distilling off the ethereal liquid yielded, on heating with resorcinol and sulphuric acid and subsequently treating with alkali and water, a fluorescent solution, the production of which has been regarded as a test for " saccharin."   If the ethereal extract is washed with water three or four times, however, the residue obtained no longer shows this reaction, whilst " saccharin " would not be removed by such treatment.   R. V. S.

A Sensitive Reaction for Scatole.   TAKAOKI SASAKI (*Biochem. Zeitsch.*, 1910, 29, 395).—In the reaction already described (Abstr., 1910, ii, 166) the iron salt concentration should not be too great. A convenient reagent is obtained by adding one drop of a 1% ferric sulphate solution to 100 c.c. of iron-free concentrated sulphuric acid. The methyl alcohol employed must be perfectly free from aldehydes.
                                S. B. S.

Iodometric Estimation of Antipyrine in Migrainine.   C. SLEESWYK (*Pharm. Weekblad*, 1910, 47, 1282).—" Migrainine " is prepared by dissolving 90 parts of antipyrine, 9 parts of caffeine, and 1 part of citric acid in water, and evaporating the solution to dryness on the water-bath.   In order to test the commercial product, the author uses the iodometric method, originally proposed by Bougault, and since modified by Zernik, with a further modification.

1·351 Gram of iodine, 2·5 grams of mercuric chloride, and 1·1 gram of the sample are each dissolved in 200 c.c. of 95% alcohol ; 20 c.c. of the migrainine are then mixed with 20 c.c. of the mercuric chloride, and the iodine is run in until the liquid turns yellow ; 1 c.c. of iodine = 0·005 gram of antipyrine.   It is as well to check the iodine by means of pure antipyrine.   The migrainine may also be dissolved in water instead of alcohol.   The titration should be repeated, the bulk of the iodine being added at once, and the last drops more carefully ; the end-point is then very distinct.   L. DE K.