THE ANALYST.

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

ORDINARY MEETING, NOVEMBER 5, 1919,

HELD at the Chemical Society's Rooms, Burlington House, Dr. Samuel Rideal, President, in the Chair.

Certificates were read for the first time in favour of Messrs. Percy Ewart Bowles, F.I.C., Ph.D. (Heidelberg), Charles Crowther, M.A. (Oxon.), Ph.D. (Leipsic), John William Hawley, B.Sc. (Lond.), A.I.C., Ernest Joseph Lush, B.A. (Cantab.), A.I.C., H. Percy Marks, B.Sc., A.I.C., and Robert Selby Morrell, M.À. (Cantab.), Ph.D., (Wurzburg), F.I.C.

Certificates were read for the second time in favour of Messrs. John Dalton and William Norman Leng.

The following were elected members of the Society: Messrs. John Haworth and Henry Turner Lea, M.Sc. (Birmingham), A.I.C.

The following papers were read: "Egyptian Bricks," by G. Rudd Thompson, F.I.C.; "The Analysis of Brazilian Zirconium Ore," by A. R. Powell and W. R. Schoeller, Ph.D.; and "The Halogen Absorption of Turpentine," by Ethel M. Taylor.

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THE ANALYSIS OF BRAZILIAN ZIRCONIUM ORE.

BY A. R. POWELL AND W. R. SCHOELLER, PH.D.

(Read at the Meeting, November 5, 1919.)

THE method for the analysis of Brazilian zirconium ore, described below, has been used by us for several years, during which time it has undergone various minor modifications. It contains nothing quite new, as the scheme for the estimation of zirconia embodies Dittrich and Freund's thiosulphate precipitation (Zeitsch. anorg. Chem., 1907, 56, 337), Weller's colorimetric titanium determination (Ber., 1882, 15, 2599), and Ferguson's separation of alumina from zirconia (Eng. and Min. J., 1918, 106, 356, 793); but we claim that the method used by us for opening up the ore (i.e., fusion with sodium carbonate followed by a bisulphate treatment) constitutes a great improvement on the usual bisulphate fusion. At the present time few chemists are familiar with the analysis of zirconium ores, hence the results obtained on the sam

sample by different operators sometimes show extraordinary disagreement. Our process is specially adapted for Brazilian ore low in titania and alumina, and containing only traces of thoria, if any. It may, however, also be used for zircon.

1. PREPARATION OF THE SAMPLE.—Proper grinding is the essential condition for the success of the analysis, and half an hour of careful work at this point saves a week's labour. The ore is first ground to 90-mesh in the usual manner, then carefully sampled down until 10 grms. are obtained. This final sample is halved; the first half, or part thereof, is treated with a horse-shoe magnet wrapped in paper until the iron introduced in crushing is extracted, and dried at 100° C. for an hour. One grm. of the dry ore is then ignited to constant weight in a tared platinum crucible to ascertain the loss on ignition. The other half is ground in agate under water or alcohol free from fixed residue, and the slime carefully decanted from time to time into a porcelain basin. When but little sandy material is left, a magnetised knife-blade is passed through the suspension to remove the iron. The sliming is finished on the remainder, the liquid evaporated, and the residue well dried. It is then scraped out of the basin, transferred to a clean sheet of glazed paper, and thoroughly mixed.

2. OPENING UP THE ORE.—Half a grm. of the slimed ore is weighed into a tared platinum crucible and heated to constant weight. This second determination of the loss on ignition is made to obtain the final summation of the constituents in the portion taken for analysis; if, owing to the sliming operation, the result should not agree with the loss as determined on the unslimed material the figure obtained under (1) is taken as correct, and the results of the analysis are calculated on the proportion of fixed residue in the unslimed ore.

The ignited mineral is carefully mixed with 5 grms. of pure sodium carbonate, the crucible covered with a platinum lid, and heated for an hour with the full flame of a Teclu or other blast burner; a clean open muffle may also be used. The melt is spread round the sides of the crucible and extracted with boiling water in a porcelain dish. The insoluble residue is transferred to an ashless filter (9 cm.) and washed with hot water, the filtrate being received in a porcelain dish. The residue is returned to the platinum crucible, ignited gently to destroy the organic matter of the filter, and transferred to a watch-glass. About 5 grms. of sodium or potassium bisulphate are then dehydrated in the crucible, left to solidify, the residue added, and fused gently until the lumps have disappeared and white semi-transparent silica is seen floating in the fluid mass. The cooled melt is leached with a minimum quantity of 1 per cent. sulphuric acid, the liquid filtered through ashless paper into an 800 c.c. beaker (Filtrate A), and the residue transferred to the filter and washed with cold water.

3. ESTIMATION OF SILICA.—While the bisulphate fusion is in progress, the filtrate from the leach of the sodium carbonate fusion is evaporated on the water-bath with an excess of hydrochloric acid; the residue is moistened with a few c.c. of the acid, taken up with water, and the silica filtered off on ashless paper and washed. The filtrate is once more taken to dryness to recover a further small quantity of silica; the residue is taken up with a little hydrochloric acid and water, filtered off, and washed as before (Filtrate B). The two silica precipitates, together with the

insoluble residue from the bisulphate fusion, are strongly ignited in a tared platinum crucible and weighed, then heated with 40 parts of hydrofluoric acid and 20 of sulphuric on a sand-bath ; the acids are driven off, the residue ignited with a fragment of ammonium carbonate and weighed ; the loss represents silica. The small residue is once more fused with bisulphate, when everything dissolves if the ore was properly slimed. The melt is dissolved in a little 1 per cent. sulphuric acid (Solution C).

4. TREATMENT OF THE SOLUTION.—The three solutions (A, B, C) obtained above contain all the soluble constituents of the ore ; B and C are added to the beaker containing A, and the liquid treated with pure sodium bicarbonate, or crystallised carbonate, until a white precipitate begins to form. This is dissolved in a slight excess of sulphuric acid, a little pulped filter fibre added, and the liquid saturated at 40° to 50° C. with hydrogen sulphide. The precipitate of platinum sulphide is left to settle overnight, filtered off, and washed with cold 1 per cent. sodium chloride solution. The filtrate is almost neutralised with freshly made sodium carbonate solution (but avoiding a permanent precipitate of ferrous sulphide) and boiled, whilst a fairly rapid current of carbon dioxide is passed to expel hydrogen sulphide.

5. ESTIMATION OF THE SUM OF ZIRCONIA, TITANIA, AND ALUMINA.—The liquid freed from hydrogen sulphide is treated with a concentrated solution of 10 grms. of pure thiosulphate and a thoroughly pulped ashless 9 cm. filter, and boiled for one hour, making good with boiling distilled water the loss by evaporation. The precipitate is allowed to settle, filtered off on ashless paper (11 cm.), and washed fifteen to twenty times with boiling water. Should a film of precipitate adhere tenaciously to the beaker, it is removed with a few drops of pure hydrogen peroxide and dilute sulphuric acid, and the liquid made alkaline with ammonia. The precipitate is best collected on a small filter and the filtrate rejected. Both precipitates are ignited wet in a tared platinum crucible until the organic matter has been burnt, transferred to a small beaker, and leached with hot water, again filtered off and washed, returned to the same platinum crucible, and ignited to constant weight. This is attained by half an hour's ignition over a Teclu burner. The product is weighed as $ZrO_2 + TiO_2 + Al_2O_3$.

6. SEPARATE ESTIMATION OF ALUMINA, TITANIA, AND ZIRCONIA.—The ignited precipitate is intimately mixed with thirty times its weight of pure sodium carbonate, and the platinum crucible heated for half an hour over a blast burner. The melt is boiled with distilled water and 1 to 2 grms. of sodium hydroxide in a platinum or nickel dish, and filtered into another metallic dish, using a rubber funnel and common paper, which resist the action of the alkali. The filtrate is returned to the first dish and once more filtered through the same paper ; this frees the filtrate from a small quantity of finely divided oxides, which frequently pass through in the first filtration. Filter and precipitate are then washed with 2 per cent. sodium carbonate solution.

(a) *Alumina*.—The alkaline filtrate is slowly poured into a 600 c.c. beaker containing excess of nitric acid (1 : 2 water). It is boiled to expel carbon dioxide, treated with a little filter pulp, and made faintly alkaline with ammonia. The precipitate is transferred to an ashless filter, washed with ammonium nitrate solution

ignited over a blast burner, and weighed as Al_2O_3. The purity of this should be ascertained by fusion with bisulphate, solution in 1 per cent. sulphuric acid, and addition of a drop of hydrogen peroxide ; a small fraction of a mgrm. of titania is sometimes found. This is deducted from the weight of the alumina and added to that of the titania. As an additional precaution we also test for zirconium by adding more hydrogen peroxide and a little sodium phosphate, but this test has always proved negative.

(b) *Titania.*—The residue from the last fusion is incinerated in the same platinum crucible, and transferred to a watch-glass. Five grms. of bisulphate are dehydrated in the crucible and the residue fused once more. The melt is extracted with 1 per cent. sulphuric acid, the solution made up to 200 c.c., and an aliquot volume (20 to 50 c.c.) transferred to a 100 c.c. Nessler cylinder The standard is made up by dissolving an equivalent amount of sodium bisulphate in another cylinder. Two drops of hydrogen peroxide are added to each cylinder, and the colour of the assay matched by adding a suitable amount of titanium solution (0·001 grm. TiO_2 per c.c.) to the standard. It is important not to exceed the prescribed amount of hydrogen peroxide, as otherwise a precipitate of zirconium peroxide may be produced in the assay.

(c) *Zirconia.*—The sum of the alumina and titania, found as above, subtracted from the weight of the ignited thiosulphate precipitate (5) gives the zirconia.

7. ESTIMATION OF FERRIC, MANGANOUS, CALCIUM, AND MAGNESIUM OXIDES.— The filtrate from the thiosulphate precipitate is boiled down with a slight excess of nitric acid and the precipitated sulphur filtered off. The filtrate is subjected to double precipitation with ammonia in presence of ammonium chloride, and the iron precipitate ignited and weighed. The filtrate is digested on a hot plate with bromine water, the small precipitate of manganic oxide filtered off, ignited, and weighed as Mn_3O_4. In the filtrate from the manganese, lime and magnesia are precipitated respectively as oxalate and phosphate. Whether the small quantity of lime and magnesia contained in 0·5 grm. of the ore can be estimated accurately in presence of much sodium salts is what we consider to be the only doubtful point about the method.

The whole analysis occupies about a week ; with a certain amount of practice this time can be reduced to five days. The estimation of the zirconia content involves all the operations described under paragraphs 1 to 6, except that the silica need not be weighed, but we always do so because the figure is important as a check, and its estimation involves very little extra trouble.

In a short discussion which followed the reading of the paper it was pointed out that the method described would not be applicable to phosphatic ores containing zirconia.

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THE HALOGEN ABSORPTION OF TURPENTINE.

By ETHEL M. TAYLOR.

(Read at the Meeting, November 5, 1919.)

IT is well known that the iodine absorption of turpentine varies with the time of action of the iodine solution (cf. Archbutt, J. Soc. Chem. Ind., 1902, **21**, 1439, and Worstall, ibid., 1904, **23**, 302), and, although the figure obtained approximates to the ratio 4 : $C_{10}H_{16}$, there is no indication that the absorption stops at this point ; indeed, all the evidence points to a main reaction in which 4 atoms of iodine are added, and a second slower action in which a further addition of iodine takes place with formation of a halogen acid, indicating substitution.

Repetition of Archbutt's experiments, extending, however, the time of action in both directions, gave results practically identical with those found by him, and, owing to the fact that finality of absorption was never obtained, did not admit of mathematical study. An attempt was made to measure the acidity produced in each case, but the variation of the result showed that a portion at least of the acidity was formed during the dilution with water during the titration.

The use of Wijs solution was therefore abandoned, and a $\frac{N}{1}$ solution of IBr_3, in chloroform adopted for the study of the action ; the first series consisted in submitting 0·1 grm. of turpentine to the action of 10 c.c. of $\frac{N}{1}$ IBr_3 for varying times, with results as under :

Time.	Halogen Absorbed.	
	Calculated as Iodine.	Calculated as Bromide.
15 minutes	384·5	242·0
30 ,,	388·5	244·6
1 hour	407·0	256·2
2 hours	418·6	263·4
3 ,,	420·1	264·5
4 ,,	425·3	267·6
6 ,,	441·7	275·7
18 ,,	445·0	280·0
24 ,,	469·5	293·1

On plotting out these results on squared paper they appeared to lie approximately on a logarithmic curve, and the value for time = one minute would be very close to the theoretical value for 4I. This indicated that the first action was extremely rapid with IBr_3 in chloroform, being practically instantaneous, and that the secondary reaction was sufficiently slow to be negligible under these conditions.

A second series was made, titrating the excess of iodine after about one minute's exposure and varying the amount of halogen added:

Halogen Added, Calculated as Bromine per Cent.	Halogen Absorbed, Calculated as Bromine per Cent.	Ratio Added to Absorbed.	Calculated.
120·2	97·6	1·231	97·5
129·5	102·6	1·262	103·5
138·8	109·4	1·269	109·5
148·0	116·7	1·268	115·5
156·2	121·4	1·278	121·5
166·5	127·7	1·305	127·5
185·0	139·4	1·327	139·5
370·0	212·1	1·745	—
925·0	237·0	3·90	—

It is seen that the absorption is nearly proportional to the amount added, with quantities of halogen insufficient to reach the ratio 4Br : $C_{10}H_{16}$, and when plotted they lie very close to a straight line of which the equation is

$$\text{Br. absorbed} = 19\cdot5 + 0\cdot6486 \text{ (Br added)},$$

while with a large excess the results are very close to the theoretical (235·3).

Incidentally this shows that the absorption does not take place in two stages, and there is no indication of a resting stage $Br_2 : C_{10}H_{16}$. The close approach of the ratio of Br added to Br absorbed to 1·333 (the ratio of IBr_3 to Br_3) during the straight line portion would appear to indicate that the halogen absorbed is bromine.

The conclusions drawn are:

1. The reaction between turpentine and halogen is

$$3C_{10}H_{16} + 4IBr_3 = 3C_{10}H_{16}Br_4 + 2I_2,$$
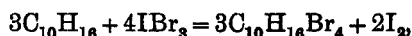
and there is no intermediate reaction.

2. This reaction can be realised with practically no secondary reaction by adding 10 c.c. $\frac{N}{1}$ IBr_3 in chloroform solution to 0·1 to 0·15 grm. turpentine dissolved in 10 c.c. chloroform, adding KI solution within one minute and titrating back immediately with thiosulphate solution.

The work was carried out in the Analytical Laboratory of Boots Pure Drug Co., Ltd., to whom my thanks are due.

[Mrs. Taylor is unfortunately prevented from continuing the work, owing to ill-health. She has sent me her notes, from which I have extracted the completed portion of her investigation.—H. D. RICHMOND.]

DISCUSSION.

Mr. ARCHBUTT remarked that the author's paper was of considerable interest, and fully confirmed his own observations. He had always regarded the test with Wijs solution as a purely empirical one, the value of which depended upon the strict observance of uniform conditions. The author's observations seemed to show that

the modified procedure which she recommended placed the halogen absorption test upon a more precise and scientific basis, and the method was well worth a trial, especially in view of the fact that some of the glacial acetic acid now offered by the dealers, even some sold as of A. R. quality, did not give a stable Wijs solution. He (Mr. Archbutt) had not yet discovered the cause of this, and it did not appear to seriously affect the determination of the iodine values of fixed oils, but it did very seriously interfere in the case of oil of turpentine.

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EGYPTIAN BRICKS.

By G. RUDD THOMPSON, F.I.C.

(Read at the Meeting, November 5, 1919.)

A WORD of apology is perhaps needed in bringing before the Society a paper which is largely of a historical character, but I venture to hope that it may prove of interest in some slight degree. To the average mind the aphorism of the impossibility of making bricks without straw leads one to believe that the bricks referred to (the Captivity period of the Jews) contained straw as an integral material, or some such material roughly so translated; but, though we have the authority of the late Professor Flinders Petrie in stating that it is not essential to mix straw with the native brickmaking material, although some of the ancient bricks may contain it, yet it can be shown that the actual bricks of the Captivity did not contain any.

It has been my lot to examine a number of interesting materials from the Land of Goschen (the actual site of the captivity), amongst them being bricks of undoubted Jewish origin. These came from one of the two fortified cities—Pithom, now Tel-el-Mashkuta. The Israelites being settled in Goschen, the Egyptians employed them to construct defences blocking the eastern road into Egypt in the Wady Tumilat running between the delta of the Nile and the canal. For the purpose of these defences large quantities of bricks were used made from the mud of the River Nile, and we read the order given by the Egyptian taskmasters in reply to the demand for straw: "There shall be no straw given you, yet shall ye deliver the tale of bricks." This of itself is almost evidence that straw did not enter into the composition of the bricks, but was rather used in the process of manufacture. The object of my investigation was to prove or disprove this theory.

I would first remind you that the probable date of manufacture of the bricks would lie between 1580 and 1220 B.C., these being the dates usually accepted for the commencement of the Oppression and the Exodus, so we have at any rate a fairly ancient material before us. The bricks as brought over are large, roughly moulded blocks about 14 × 10 × 6 in. of a dull grey colour, and, although fairly hard and compact, yet break easily when once roughened on the surface. My samples were selected from at least fifty such blocks collected over a considerable area of ground.

I am indebted to an American source for an analysis of River Nile mud taken

near Bubastis, close to the Land of Goschen, which agrees closely with my own results for the bricks. Although an interval of something like 3,300 years has elapsed there has been but little alteration, if any, in the actual materials brought down and deposited by the Nile.

In my investigations of the bricks close attention was paid to the microscopic examination for traces of vegetable matter, especially of the nature of straw or wood fibre; but in no single instance was there any trace of such, and, further, the determination of nitrogen and phosphorus led me to conclude positively that no straw entered into the composition of the bricks.

Now what really happened to give rise to the aphorism already quoted was that in the days of the Captivity the process of brickmaking was similar to that of the present day, and that in dealing with a sticky material such as Nile mud it was necessary to employ some non-adherent material like chopped straw for the workman to dip his hands into and so prevent the mud sticking to them during the moulding and rough surfacing; also to prevent the moulded bricks sticking to the ground during the sun-drying process. The lumps of mud or the sides of the mould were covered with chopped straw so as to enable the brick to be withdrawn, or to prevent the various bricks sticking together when in the plastic condition. I am convinced that the straw or stubble was not an integral part of the bricks, but was used in a purely mechanical manner to facilitate manufacture.

						Brick.	Nile Mud.
						Per Cent.	Per Cent.
SiO_2	...	....	...	...	...	70·40	71·02
Al_2O_3	...	...	...	...	...	9·42	9·15
Fe_2O_3	...	...	...	...	...	5·71	5·60
CaO	...	...	...	...	...	2·13	2·27
MgO	...	...	...	...	...	1·37	1·50
K_2O	...	...	...	...	...	2·32	}4·00
Na_2O	...	...	...	...	...	1·38	
TiO_2	...	...	...	...	...	trace	—
Loss on ignition trace of CO_2	...	...	...	7·20	6·50
Chlorine	...	...	...	...	...	trace only	
N ...	...	...	...	...	...	trace only	
P_2O_5	...	...	...	...	...	0·002	—
Total	...	...	...	...	...	99·932	100·04

Finally, I would mention that the authenticity of the samples is beyond doubt. The two cities or fortifications built by the Israelites were Pithom and the city of Rameses, now Tel-Rotab, the latter being about twelve miles along the narrow marshy valley and the former about ten miles east. The city of Rameses, or Tel-Rotab, is identified by the remains of a town and temple built by Rameses II. the Pharaoh of the captivity; for from this temple a large slab is now in Philadelphia

bearing a pictorial representation of Rameses with an inscription identifying it, and there is no other city except Pithom of this date along the valley, and the city itself is identified by the mention made in the above inscription of the temple of Tum— "Pa-Thum."

NOTE BY EDITOR.—Since this paper was read a communication has appeared (*J. Soc. Chem. Ind.*, 1919, **38**, 439R.) which has an interest bearing on the subject of Mr. Thompson's paper.

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NOTE.

The Editor desires 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.

THORPE'S ALCOHOLOMETRIC TABLES: CORRIGENDUM.

HAVING recently noticed an error in the above, I think many analysts would be glad to know of it with a view to making the necessary correction:

Sp. gr. 0·9670. Percentage of alcohol by volume, for 28·69 read 28·60.

Sir Edward Thorpe, in acknowledging my letter, tells me that he has instructed the publishers to alter the plate accordingly.

A. E. JOHNSON.

24, PARKDALE,
WOLVERHAMPTON

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Rapp's Method for the Estimation of Alkaloids. A. Heiduschka and L. Wolf. (*Apoth. Zeit.*, 1919, **34**, 134 ; through *Chem. Zeit. Rep.*, 1919, **43**, 193.)—The following modification of this method is recommended in order to insure better separation of the chloroform from the plaster of Paris magma. Three grms. of the sample (*e.g.*, cinchona bark) are heated on a water-bath for ten minutes with 15 c.c. of $\frac{N}{2}$ hydrochloric acid, cooled, and shaken for five minutes with the addition of 3 c.c. of 20 per cent. sodium hydroxide solution and 60 c.c. of chloroform. Thirty grms. of plaster of Paris are then added, and the shaking continued for a further minute. The chloroform layer separates readily, and is decanted through a filter ; 50 c.c. of the filtrate (equal to 2·5 grms. of bark) are shaken for two minutes in a separating funnel with 12·5 c.c. of $\frac{N}{10}$ hydrochloric acid, the acid solution diluted with 12·5 c.c. of water, and 20 c.c. (equal to 2 grms. of bark) are titrated with $\frac{N}{10}$ sodium hydroxide solution, using methyl-red as indicator. (See also ANALYST, 1919, 236.)

W. P. S.

Chemical Test to distinguish between Caffeine and Theobromine. F. P. Stroup. (*Amer. J. Pharm.*, 1919, **91**, 598-599.)—In caffeine a methyl group replaces a hydrogen atom in theobromine, but, in spite of this close similarity in

composition, a marked distinction attends their behaviour towards a mixture of potassium dichromate and sulphuric acid (1 part by weight of salt in 20 parts by volume of concentrated acid).

Method A.—A trace of the alkaloid is flattened out on a white tile to cover a space of about a square centimetre, and 2 drops of the reagent are put in the centre. In the case of caffeine the yellow colour of the reagent is almost at once changed to a bright bluish-green ; while with theobromine the colour changes from yellow, through dark purplish, to purplish-green, olive-green, and finally reaches the same bluish-green tint given by caffeine.

Method B.—Five or 6 drops of the reagent are placed in separate spots on a tile, and into the centre of it is put a speck of the alkaloid under examination. Caffeine dissolves almost at once, and in a few seconds the whole drop is coloured bright bluish-green, while, in the case of theobromine, solution takes place much more slowly. A zone of colours forms successively of the same tones as in Method *A* above.

<div style="text-align:right">H. F. E. H.</div>

Detection of Nicotine. O. Tunmann. (*Apoth. Zeit.*, 1918, **33**, 485 ; through *Chem. Zeit. Rep.*, 1919, **43**, 193.)—A drop of the alkaloid solution is brought into contact with a small quantity of *p*-dimethylaminobenzaldehyde dissolved in a drop of concentrated hydrochloric acid. Nicotine yields a red coloration which soon changes to violet-red ; the reaction may be obtained with as little as 0·2 mgrm. of nicotine. Pyridine, coniine, and the common alkaloids do not interfere with the test. With a cold saturated picric acid solution containing 10 per cent. of concentrated hydrochloric acid, 0·01 mgrm. of nicotine yields a distinct precipitate, whilst 0·003 to 0·005 mgrm. gives microscopic crystals (these appear in about two minutes) ; pure pyridine also gives crystals with this test, but no amorphous precipitate.

<div style="text-align:right">W. P. S.</div>

Analyses of Barleys and of the Malts made from them. A. R. Ling. (*J. Inst. Brew.*, 1919, **25**, 288-296.)—The analyses of barleys and malts were carried out in 1912-13 ; the Californian and Damascus barleys not being of the first grade. The values found for starch are probably high, having been determined by the polarimetric method of Ewers (ANALYST, 1906, **31**, 25 ; 1908, **33**, 10ĭ, 481), which yields optically active substances derived from the pentosans present.* The apparent losses of dry substance occurring during the conversion of barley into malt are, on the average, as follows :

Matter extracted during steeping	...	...	1·8 to 2·0 per cent.	
Loss due to removal of rootlets	...	...	3·0 ,, 3·5	,,
,, ,, respiration (as CO_2)	...	...	3·5 ,, 4·5	,,

This last is the chief and most variable source of the loss of solid matters in the malting process, and is minimised by the maltster as far as possible in order to

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* The author points out that the difference may be judged approximately from the fact that a sample of wheat which showed 62·5 per cent. of starch by Ewers' method yielded 56·9 per cent. by the Taka Diastase method employed by Davis and Daish (ANALYST, 1914, **39**, 312).

obtain the best results combined with economy, the loss in " forced " malts some-
times running as high as 7 to 8 per cent.

	Californian.			Damascus.			Norfolk.			Polish.		
	Barley.	Malt.	Malt *x*.	Barley.	Malt.	Malt *x*.	Barley.	Malt.	Malt *x*.	Barley.	Malt.	Malt *x*.
Starch	57·05	50·65	43·56	54·64	50·28	47·42	61·56	56·18	49·90	58·19	51·34	47·98
Albumenoids (insoluble)	6·96	5·75	4·52	7·21	5·86	5·53	7·63	5·85	5·19	7·53	6·61	6·18
Albumenoids (soluble)	2·14	3·47	2·98	2·52	3·47	3·27	1·65	3·59	3·19	2·93	4·37	4·08
Ash (insoluble)	1·86	1·80	1·55	2·07	1·86	1·75	1·93	1·65	1·47	1·79	1·54	1·44
Ash (soluble) ...	0·90	0·66	0·57	1·00	0·61	0·58	0·72	0·80	0·71	0·73	0·80	0·75
Fat	1·99	1·94	1·67	2·02	1·93	1·82	2·36	1·78	1·58	2·47	1·87	1·75
Sugar (invert)...	0·67	3·84	3·30	0·56	3·14	2·96	1·27	4·63	4·11	1·43	6·04	5·65
Sugar (cane) ...	2·31	7·18	6·17	1·90	5·53	5·22	1·01	5·36	4·76	1·27	5·12	4·79
Gums, etc. ...	1·52	2·59	2·23	1·72	2·83	2·67	0·95	3·35	2·97	1·84	4·06	3·80
Fibre	7·86	8·41	7·23	6·02	6·78	6·39	5·63	5·69	5·05	5·86	5·41	5·06
1,000 corn weight (dry)	41·23	35·46	—	41·46	39·11	—	35·46	31·51	—	37·62	35·17	—
Extract (lb. per 336 lbs.)	—	92·5	—	—	86·5	—	—	97·9	—	—	93·9	—
Diastatic power (deg. Lintner)	9	20	—	16	30	—	19	27	—	—	23	—
Tint (10 per cent. solution 1-inch cell) (Lovibond)	—	6·0	—	—	3·5	—	—	3·5	—	—	4·5	—

The results in the first two columns of each class of barley are calculated
as percentages on dry substance. The analyses of the malts in the third column—
Malt *x*—are expressed " on that weight of the corresponding dry barleys yielding
100 parts by weight of dry malt." This is effected by using the formula : $x = \dfrac{100\,M}{B}$,
in which *x* is the composition of the malt expressed on the standard just mentioned,
M = the 1,000 corn weight of the malt, and B the 1,000 corn weight of the barley.

H. F. E. H.

Detection of Hydrogen Chloride in Chloroform. D. Vorlander. (*Ber. Deut.*

Pharm. Ges., 1918, **28**, 337; through *Chem. Zeit. Rep.*, 1919, **43**, 193.)—Ten c.c. of
the chloroform are treated with a very small quantity (about 0·01 mgrm.) of powdered
p-dimethylaminoazobenzene ; if hydrogen chloride is present a violet-red coloration
is obtained. In the absence of hydrogen chloride the chloroform is coloured yellow.
An excess of the reagent may mask the coloration given by very small traces of
hydrogen chloride. The violet-red coloration is destroyed by free chlorine.

W. P. S.

Colour Standards for Cottonseed Oil. H. V. Arny, C. Kish, and F.

Newmark. (*J. Ind. Eng. Chem.*, 1919, **11**, 950-953.)—The authors have developed
a series of chemical colour standards for the classification of cottonseed oils, possessing

certain advantages over the tintometer glasses as regards constancy. These standards, termed the Co-Fe-Cu standard are composed as follows : red, $\frac{N}{2}$ solution of cobalt chloride in 15 per cent. hydrochloric acid ; yellow, a $\frac{N}{2}$ solution of ferric chloride ; and blue, a $\frac{N}{2}$ solution of copper sulphate, all in 15 per cent. hydrochloric acid. With these solutions, blended in various proportions and diluted with various quantities of water the colour of any commercial samples of cottonseed oil may be accurately matched. Another set of standards, termed the Co-Cr-Cu standards, is made up in ammoniacal solution, but these are not adapted for cottonseed oils. Experiments have shown that the Co-Fe-Cu standard solutions are perfectly stable and give identical results after keeping for four years ; moreover, they are readily controlled by analysis. The comparisons are conveniently made in 1 oz. round bottles with sufficient accuracy, and perfectly accurate results are obtained by comparison side by side in $\frac{1}{2}$ in. cells in the Lovibond instrument. The following limits for the specification of commercial grades of cottonseed oil have been defined in terms of the above Co-Fe-Cu acid standards.

	From—				To—			
	Fe.	Co.	Cu.	Water to—	Fe.	Co.	Cu.	Water to—
	c.c.	c.c.	c.c.	c.c.	c.c.	c.c.	c.c.	c.c.
Prime white	6	0·4	—	50	16·0	1·4	—	60
Choice summer yellow ...	22	3·4	—	50	33·3	3·3	—	50
Off. summer yellow	42	6·2	—	60	39·0	7·2	—	50

J. F. B.

Estimation of Lactose in Milk. E. Hildt. (*Ann. Chim. anal.*, 1919, [ii.]1, 309-311.)—The estimation of lactose in milk which has undergone partial decomposition, especially in milk preserved with potassium dichromate, is rendered untrustworthy owing to the fact that the copper-reducing power of the resulting mixture of sugars (lactose, galactose, and dextrose) is not known. The author recommends, therefore, that the lactose be hydrolysed completely before the estimation is made. For the hydrolysis the barium or sodium salt of a sulphonic acid of the benzene series may be used, an equivalent quantity of sulphuric acid being also added. A 0·5 per cent. lactose solution is hydrolysed completely in four hours at 100° C. in the presence of 1 per cent. of sulphonic acid. Ten c.c. of Fehling's solution (titration at boiling-point) are equivalent to 0·0708 grm. of crystallised lactose, 0·0506 grm. of hydrolysed lactose, or 0·0495 grm. of invert sugar. (*Cf.* ANALYST, 1919, 237.)

W. P. S.

Analysis of Saccharin. O. Beyer. (*Chem. Zeit.*, 1919, **43**, 537-538.)—As the result of an examination of methods which have been proposed for the estimation of saccharin, the author finds that only two methods are useful—namely, Richmond and Hill's method (*cf.* ANALYST, 1918, **43**, 353), and the *Reichsgesundheitsamt* method.

According to the latter method 0·5 to 0·7 grm. of the sample is boiled for two hours under a reflux condenser with 20 c.c. of 20 per cent. sulphuric acid, the solution then cooled, diluted, rendered alkaline with sodium hydroxide, and distilled, the ammonia being collected and titrated in the usual way. When the melting-point of a sample of commercial saccharin falls below 220° C., it is advisable to estimate the amount of p-sulphaminobenzoic acid present. For this purpose, 1 grm. of the sample is heated for two hours under a reflux condenser with 10 c.c. of hydrochloric acid (sp. gr. 1·124) and 10 c.c. of water; the solution (filtered if necessary) is evaporated to dryness on a steam-bath, the residue dissolved in 10 c.c. of hot water and the solution kept for at least twelve hours at a temperature below 10° C. The para acid separates out as crystals, which are collected, washed with a small quantity of cold water, dried at 100° C., and weighed. W. P. S.

Modified Benedict Method for Estimation of Sulphur in Foods and Fæces. J. O. Halverson. (*J. Amer. Chem. Soc.*, 1919, **41**, 1494-1503.)—A weighed quantity of the material (2 to 4 grms.) is rolled up in a filter-paper and placed in a 300 c.c. Kjeldahl flask. Hot water (10 to 15 c.c.) is added and then 20 c.c. of a mixture of fuming and concentrated nitric acid (1 : 4). The flask is stoppered with a small funnel and set aside for half-a-day. It is then heated on the steam bath for four days. A further 10 c.c. of the acid mixture is then added and digestion continued until the solution is clear or nearly so. The solution is then transferred to a dish and treated with 20 c.c. of the Benedict reagent during evaporation to thorough dryness on the steam bath. The reagent consists of 200 grms. copper nitrate, 50 grms. potassium chlorate and distilled water to make 1,000 c.c. Blanks should be run on the reagents, of which copper nitrate is the most difficult to obtain free from sulphur. To prevent spattering in heating the blanks, 5 c.c. of 10 per cent. cane sugar solution should be used. The partly oxidised material in the dish is baked on a sand bath and the blackened residue finally heated until the mass fuses. When cool again, 20 c.c. of dilute (1 : 4) hydrochloric acid are added and the solution filtered, made up to at least 100 c.c., heated to boiling, and sulphur precipitated by addition of 10 c.c. of boiling 5 per cent. barium chloride added drop by drop. After digestion on the steam bath for two hours, the barium sulphate is filtered off. Silica does not interfere as it does in the peroxide method. · The method is slow, but requires less attention than those from which it has been developed. G. C. J.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Yeast Nutrient Media. E. Friedmann. (*Mürch. Med. Woch.*, 1918, **65**, 76. Through *J. Inst. Brew.*, 1919, **25**, 315.)—A clear extract of yeast may be obtained by allowing 10 grms. of dried yeast and 5 grms. of sodium chloride to stand with a litre of water, for three-quarters of an hour, at room temperature followed by boiling for one hour in a steam chamber. Five c.c. of colloidal iron solution (*liq. ferri oxychlorati*) are then stirred into the hot turbid liquid, and the whole is again heated for twenty minutes, after which the solution is filtered, till clear, through double pleated

papers. After sterilisation, the yeast solution thus made serves as a substitute for meat extract for the preparation of nutrient agar, fuchsine agar, litmus milk-sugar agar, or Löffler's blood-serum.   H. F. E. H.

ORGANIC ANALYSIS.

German Aviation Motor Spirits. G. Chavanne, L. P. Clerc, and L. J. Simon. (*Comptes rend.*, 1919, **169**, 693-695.)—By fractionation of twenty-two samples of German aviation motor spirit, and determining the sp. gr. and critical temperature of solution of the fractions in aniline, before and after nitration, the following average results were obtained: Sp. gr. before nitration, 0·72265; after nitration, 0·7140; critical temperature of solution before nitration, 50·0° C.; after nitration, 57·4° C. These figures gave the data for calculating the composition, as described in a previous communication (ANALYST, 1919, 348)—viz., aromatic hydrocarbons, 8·76 per cent.; saturated cyclic hydrocarbons, 40·4 per cent.; and acyclic hydrocarbons, 50·6 per cent.   C. A. M.

Distillation of Aqueous Solutions of Related Organic Substances. J. Reilly and W. J. Hickinbottom. (*Chem. News*, 1919, **119**, 185.)—The regularities or constants obtained when dilute solutions of organic fatty acids are distilled may be expressed in terms of Nernst's law of distribution; for large changes of concentration this law does not, however, hold good, and a method of steam distillation is recommended in which only small changes of concentration occur. By plotting the constants for the fatty acids (formic to caprylic) against the number of carbon atoms present in the acid, an approximately straight line results, showing an interdependence between molecular constitution and rate. of steam distillation. Acids containing a side carbon chain (as isovaleric acid) have larger constants than have normal acids of the same molecular weight. In the case of isomeric substances, for example, normal and isobutyric acids, the distillation constants are distinct enough to differentiate between the substances. There is also a relation between the molecular complexity and the distillation constants of the lower saturated monohydric alcohols, phenols, etc., and when the constitution of a substance which is volatile in steam is uncertain a comparison of the distillation constants with those of allied substances may assist in deciding its molecular constitution. Within certain limits, the distillation constants for formic and acetic acids and the lower saturated aliphatic alcohols are subject to variations depending on the concentration and on the presence of non-volatile acids or salts. The distillation constants of the saturated fatty acids and alcohols increase with the molecular weights, the reverse of what might be expected from a knowledge of the vapour tensions of the pure substances. It is considered likely that the tendency for the acids or. alcohols to form molecular complexes may account for this behaviour. The steam distillation method is of use in the analysis of fermentation products, technical preparations containing volatile fatty acids, etc., and a method has been worked out for the detection of butter and fats of the coconut-oil type in margarine; by estimating the distillation constants of the water-soluble fatty acids under definite conditions

constants for the fats are obtained which differ considerably in the case of butter fat compared with those of the other fats (coconut, palm kernel, and babassu).

W. P. S.

Analysis of Aromatic Sulphochlorides. F. Neitzel. (*Chem. Zeit.*, 1919, **43**, 500.)—Three grms. of the powered sulphochloride are mixed with 10 c.c. of water, phenolphthaleïn solution is added, and free sulphonic and mineral acids which may be present are neutralised with $\frac{N}{1}$ sodium hydroxide solution. A measured excess of the latter solution is then added (45 c.c. are usually sufficient), together with 20 c.c. of alcohol, and the mixture is boiled for fifteen minutes under a reflux condenser. After cooling, the remaining excess of sodium hydroxide is titrated with $\frac{N}{1}$ hydrochloric acid.

W. P. S.

Improved Method for Estimation of Carbon by Wet Combustion, using Barium Hydroxide as Absorbent. P. L. Hibbard. (*J. Ind. and Eng. Chem.*, 1919, **11**, 941-943.)—The method, which is a modification and combination of previously published methods, and is described in detail and illustrated in the original, is briefly as follows: The substance is heated in a Kjeldahl flask with chromic anhydride and sulphuric acid, whereby carbon is oxidised to carbon dioxide, which is carried into a solution of barium hydroxide by a current of purified air. After the reaction is completed, the excess of barium hydroxide is estimated by titration with standard hydrochloric acid. The amount of barium hydroxide neutralised by carbon dioxide measures the amount of carbon in the substance taken. The apparatus described is somewhat simpler than that required for the gravimetric method, and there is no difficulty due to moisture which must be guarded against in the gravimetric estimation. The error is probably less by the volumetric method, which has the further advantage that a single worker could probably operate a larger number of units. On the other hand, in case but few estimations have to be made, it would be simpler to use the gravimetric method, as in this case fewer solutions and reagents are required.

G. C. J.

Estimation of Methyl Chloride in Gas Mixtures. V. C. Allison and M. H. Meighan. (*J. Ind. and Eng. Chem.*, 1919, **11**, 943-946.)—In the chlorination of natural gas, some method of quickly and approximately estimating the methyl chloride produced was necessary as a control over the operation. In the chlorination apparatus, the chloroform and carbon tetrachloride were removed by water scrubbers, the dichloromethane and most of the water vapour by an ice-salt bath, and the excess chlorine by a soda lime tower. The effluent gas, therefore, consisted of methyl chloride, natural gas, and a little water vapour. For the rapid and approximate estimation of methyl chloride in such a mixture, the most convenient method is the absorption of the methyl chloride in glacial acetic acid contained in a Hempel pipette. The mixture is passed in and out of the pipette six times, which suffices for the complete absorption of the methyl chloride. Longer contact with the reagent is undesirable, as glacial acetic acid dissolves four times its own volume of natural gas. The method is less accurate when much water vapour is present.

A less convenient method is the method of partial pressures, in which an unmeasured sample of the gas is introduced into an evacuated bulb connected to a manometer, the bulb is cooled to $-135°$ C. in a liquid air-pentane bath, natural gas pumped off, the system sealed, allowed to come to room temperature, and the vapour pressure read. In this method water vapour is removed by having the bulb two-thirds filled with a mixture of glass wool and phosphoric anhydride. The combustion method is not suitable for the analysis of mixtures of methyl chloride with other combustible gases, and in pure methyl chloride the temperature required for complete combustion is too high for the platinum spiral to last long.   G. C. J.

Laboratory Tests on Mineral Oils. A. Philip. (*Chem. Trade J.*, 1919, **65**, 445-447.)—SAMPLING.—I. *Liquids with a density less than that of water :* The bulk sample is allowed to stand for at least seventeen hours, the vessel tilted, and a 100 c.c. pipette introduced so as to draw up a sample (*A*) from the bottom. If no water is visible, the sample is returned to the vessel and the bulk sample is thoroughly shaken. If any material quantity of water is visible, the whole sample is allowed to stand for another seventeen hours, after which three-quarters are decanted (*B*), and the remaining quarter (*C*) is poured into a separating funnel and left for another seventeen hours. The water and about a quarter of the oil are run off from the funnel, mixed with an equal volume of wet kerosene, and allowed to stand over night. The volume of separated water is measured, and calculated into the percentage of *visible separated water*. II. *Liquids with a density equal to or above that of water :* For creosotes and low-temperature tars a representative sample may be obtained as in the case of Sample *A*, whilst for dense tars a modification of the method may be used in which the water is separated from the top instead of the bottom. DISTILLATION TESTS ON CRUDE OILS.—The methods to be used will differ according to whether the products belong to the petroleum-oil group, the shale-oil group, or the tar-oil group. As far as possible any cracking process must be avoided, and the results should approximate those which will be obtained when the oil is distilled on a large scale in the refinery. The method recommended is to carry out the distillations in two operations, each in a special apparatus. The oil (1,000 c.c.) is first submitted to a topping distillation up to 260° C., by which, in the case of a crude petroleum oil, the petrol, water, and kerosene are separated together. During the distillation a current of 2·4 cubic feet per hour of air is drawn through the liquid by means of an electric motor-driven air-pump, and thence through a condenser in which condensible vapours are separated, whilst the air is again heated and made to circulate through the oil until the distillation is finished. In the second stage the residue (700 to 800 c.c.) in the flask is fractionated. CALORIFIC VALUE OF MOTOR SPIRIT.—The usual method of determining the initial distillation-point of motor spirit by recording the temperature at which the first drop falls from the thermometer or other part of the apparatus gives discordant results in the hands of different observers. At best it can only be regarded as a rough measure of the pressure which the spirit may produce when stored in hermetically closed containers. It is, therefore, preferable to determine the temperature at which the vapour-pressure of the sample is equal to that of the atmosphere, which may be done by the U-tube

method of determining boiling-points. *Distillation tests of motor spirit :* The most suitable method of determining the calorific value of motor spirit is by means of a bomb calorimeter. The liquid (about 0·5 grm.) is weighed into a sealed glass bulb or stoppered tube which is then placed on the platinum tray of the bomb in contact with a small weighed quantity (about 0·25 grm.) of pure naphthalene which has been fused previously. On igniting the naphthalene by means of the ignition wire the heat bursts the glass bulb and the spirit is ignited without possibility of loss. In the case of heavier oils 1 grm. of the sample is taken. For the simultaneous determination of the calorific value and the water value the weighed oil and naphthalene are placed in the bomb, which is then closed, and about 1 litre of oxygen is passed through it in a slow current to remove nitrogen, after which the exit tube is closed, oxygen admitted until the pressure rises to 30 atmospheres, and the calorific value determined in the usual way. The exit tube of the bomb is then connected with an absorption train composed of: (1) A small U-tube immersed in broken ice, in which most of the water is deposited; (2) a 5-inch U-tube containing pumice, saturated with sulphuric acid, to absorb the remainder of the water; (3) a series of three potash bulbs to absorb carbon dioxide; and (4) a calcium chloride tube. After the gas ceases to flow, the bomb is heated in an air-bath to about 250° F., while purified air is drawn through the apparatus to remove the residual carbon dioxide. Possible errors to guard against are the formation of nitric acid during the explosion in the bomb, and the retention of water by the sulphuric acid formed in the combustion of any sulphur in the oil. WATER IN OILS, TARS, ETC.— In removing water by bubbling a stream of dry air through the oil at 100° C., a large amount of frothing may occur. This may be prevented by mixing the 200 grms. of tar, creosote, etc., with an equal weight of sand or dried pumice, and the determination may conveniently be carried out in a conical copper flask. FREE ACIDITY OF OIL FUELS.—(1) *Acidity to Alcohol :* About 50 grms. of the oil are mixed with 100 c.c. of alcohol and the mixture titrated with $\frac{N}{10}$ sodium hydroxide solution, with phenol-phthaleïn as indicator. In the case of thick or dark oils the sample is heated on the water-bath nearly to the boiling-point of the alcohol. The alcohol, which separates in ten to fifteen minutes, will be clear enough for titration. This process is continually repeated until a permanent coloration is obtained (six to seven hours). (2) *Acidity to Water :* Fifty grms. of the sample are shaken in a separating funnel with 100 c.c. of water, the mixture left to settle, and the water drawn off. This is twice repeated with two portions of 50 c.c. of water, and the united washings are titrated with $\frac{N}{10}$ sodium hydroxide solution, with methyl orange as indicator. In the case of very thick oils it may be necessary to use hot water, and to immerse the separating funnel in a hot-water bath. EFFECT OF MOISTURE ON THE FLASH-POINTS OF OILS.—The presence of moisture in an oil causes the flash-point results obtained by different observers to show considerable differences, and, in general, the higher the flash-point of the oil the more pronounced are the variations due to this cause.

C. A. M.

Essential Oil from *Juniperus procera* grown at Nairobi. A. F. **Macculloch.** (*J. Soc. Chem. Ind.*, 1919, **38** 364T.)—The oil obtained by steam-

distillation of fine shavings of the wood of *Juniperus procera* grown at Nairobi, British East Africa, had the following characters : Sp. gr. 0·987 at 15·5° C., $[a]_{D20} = -16°$, $[n]_{D25} = 1·480$. The oil was miscible in all proportions with alcohol of 90 per cent. and over, soluble (1 : 60) in 70 per cent. alcohol. The amount of cedrol formed depended on the interval between the disintegration and distillation of the wood. After exposure of the wood for some weeks to a hot sun the distilled oil solidified to a mass of crystals. The oil obtained by distilling wood which had not been exposed to the sun after disintegration contained 38 per cent. of cedrol (m. pt., 75·5° C.) C. A. M.

Oil from Sumac (*Rhus Glabra*). H. W. Brubaker. (*J. Ind. and Eng. Chem.*, 1919, **11**, 950.)—The seeds separated from sumac berries yielded on extraction with ether 11·71 per cent. of fatty oil, the average characters of which were as follow : sp. gr. at 15° C., 0·92577 ; $[n]_{D 20°}$ C., 1·4710; acid value, 0·9 ; acetyl value, 9·235 ; saponification value, 192·6 ; iodine value, 126·76 ; soluble fatty acids, 0·766 ; insoluble fatty acids, 93·54 per cent. The characters of the insoluble fatty acids were : m. pt., 17° C. ; solidifying pt., 6° C. ; $[n]_D$, 1·470 ; iodine value, 121·8 The oil is quite viscid at ordinary temperature, and has a mild odour ; it possesses moderate drying properties, and compares favourably with other vegetable oils, such as cottonseed oil. It is applicable as an edible oil or as a material for soap making, or as a semi-drying oil for paints. It is estimated that 6 million lbs. of oil could be obtained annually from sumac seed in the State of Kansas. J. F. B.

Estimation of Fibres in Paper. R. C. Griffin. (*J. Ind. and Eng. Chem.*, 1919, **11**, 968-970.)—The fibre analysis of paper by microscopic examination is made either by the " count " system or the system of estimation by inspection. The author describes the procedure in the latter case, which is controlled by frequent examination of carefully prepared mixtures of known compositions. Two slides of the fibres from each of the papers to be analysed, as well as from the standards, are stained with the zinc chloride-iodine reagent and examined independently by several different observers, and the results are tabulated and averaged. The results obtained are accurate to within 5 per cent. in most cases and within 10 per cent. in the case of mechanical wood-pulp mixtures. The method of estimation by inspection is considered to be the more satisfactory of the two. J. F. B.

Estimation of Rosin in Rosin-Pitch Mixtures. A. Wogrinz and P. Vari. (*Chem. Zeit.*, 1919, **43**, 506-507.)—Mixtures of petroleum or coal-tar pitch with rosin or " lime-rosin " (prepared by heating colophony with dry lime) are now used for various technical purposes. For the estimation of rosin in such mixtures 250 grms. of the material are dissolved in about 75 to 80 c.c. of benzene and boiled for one to one and a half hours with 125 c.c. of alcoholic $\frac{N}{1}$-potassium hydroxide solution. The alcohol and benzene are then distilled off, and the residue extracted with three successive portions of 500 c.c. each of boiling water for about one hour each time, the liquid being allowed to cool before each decantation. The united extracts are evaporated to about 200 c.c., neutralised with dilute hydrochloric acid (methyl-orange as indicator),

and evaporated to dryness. The dry residue is freed from calcium chloride by treatment with cold water, and the aqueous extracts passed through a filter. Finally the basin is cleaned out with moist gypsum, which is also collected on the filter, and the latter is then dried and extracted for one to one and a half days with alcohol in a Soxhlet apparatus. The cold alcoholic extract (about 200 c.c.) is decanted from separated particles of pitch, which are subsequently washed with alcohol. The alcoholic extract and washings, which will contain the whole of the colophony in the original mixture, are used for the determination of the saponification and iodine values of the dissolved substance. In determining the Hübl value, an aliquot portion of the alcoholic solution is evaporated to dryness, and the residue dissolved in about 25 c.c. of chloroform. The following results were obtained with various samples of these substances:

	Saponification Value.		Iodine Value (Hübl).	
	Minimum.	Maximum.	Minimum.	Maximum.
Colophony	156·0	189·0	132·0	151·0
1 per cent. lime-rosin (1·17 per cent. CaO) ...	180·9	182·2	146·7	149·5
5 per cent. lime-rosin (4·96 per cent. CaO) ...	177·5	179·6	146·9	150·5
Petroleum pitch (softening point 39·5° C.) ...	1·8	1·8	—	—
Coal-tar pitch (softening point 43° C.) ...	6·09	6·58	2·28	2·42

For petroleum pitch preparations the saponification value enables an estimation of colophony to be made within about 2 or 3 per cent. of the amount present, whilst in the case of coal-tar pitch preparations the iodine value gives more trustworthy information.   C. A. M.

Conservation of Solutions of Starch and Organic Acids by means of Mercury. A. Junk. (*Chem. Zeit.*, 1919, **43**, 258, through *J. Inst. Brew.*, 1919, **25**, 313.)—Solutions of oxalic, tartaric, and citric acids may be preserved from the attacks of moulds by means of mercury even when frequently exposed to air. Starch used as an indicator in iodine titrations may also be preserved in a similar manner, by boiling 1 litre of the 0·2 per cent. solution with about 2 c.c. of metallic mercury for half an hour. After standing over night the liquid is decanted off, and the well-washed mercury again added to the opalescent liquid, the whole being well shaken in order to break up the metal into fine particles.   H. F. E. H.

INORGANIC ANALYSIS.

Modifications of Pearce's Method for Arsenic. J. Waddell. (*J. Ind. and Eng. Chem.*, 1919, **11**, 939-941.)—Neither Pearce's original method nor the modifications of Canby (*Zeitsch. anal. Chem.*, 1890, **29**, 187) or Bennett (*J. Amer. Chem. Soc.*, 1899, **21**, 431) are satisfactory. Bennett's modification is likely to give high results,

whilst those obtained by Canby's modification may be so low as to be useless. Both methods may be modified to give practically concordant results, and if duplicates, determined one by the modified Bennett method and the other by that of Canby, agree, the results may be considered correct.

The suggested modification of Bennett's method is as follows : The material (for example a cobalt ore) is fused with 10 to 12 times its weight of sodium peroxide in a nickel or iron crucible. The fusion after extraction with water is made up to 1,000 c.c., and a sufficient aliquot taken to require at least 14 c.c. of the thiocyanate in the final titration. This aliquot is acidified with nitric acid, made slightly alkaline with sodium hydroxide, and then very slightly acid with acetic acid. The arsenic is precipitated as silver arsenate by addition of silver nitrate, the silver arsenate is washed, dissolved in 20 c.c. of hot, dilute (1 : 1) nitric acid, the solution diluted to about 200 c.c., 5 c.c. of a saturated solution of ferric alum added, and the silver titrated with thiocyanate. The results may be 1 per cent. high calculated on the arsenic, but are never low.

The suggested modification of Canby's method is similar to the above up to the addition of sodium hydroxide to the acidified solution of the product of fusion. Nitric acid (instead of acetic acid) is then added in very slight excess, and after addition of silver nitrate, this slight excess is neutralised by addition of zinc oxide. The precipitate of silver arsenate is then treated as above described.   G. C. J.

Analysis of Basic Slag. G. Hughes. (*Chem. Trade J.*, 1919, **65**, 220.)— In order to effect a complete separation of silica when analysing samples of basic slag it is necessary that the original hydrochloric acid solution should be evaporated to complete dryness in a water-bath kept at boiling temperature, otherwise the amorphous silica is not rendered completely insoluble. Such evaporation to hard dryness makes the elimination of the phosphoric acid from the residue a difficult matter even with strong hydrochloric acid, and it is therefore necessary again to exhaust the siliceous matter with more acid. If this is not done, from 1 to 2 per cent. of phosphate of iron may remain unaccounted for in the residue. The author recommends that 1 grm. of sodium chloride be added to the 2 grms. of basic slag taken for analysis; if the hydrochloric acid solution be then evaporated to dryness in the usual way, the elimination of the phosphoric acid from the siliceous matter is much more readily and completely effected.   H. F. E. H.

Estimation of Yield in Processes for the Oxidation of Ammonia. J. Baumann. (*Chem. Zeit.*, 1919, **43**, 466.)—The entering mixture is air and ammonia gas. The mixture leaving the plant, apart from abnormal cases when a fog of ammonium nitrate may be present, consists of oxides of nitrogen mixed with air poor in oxygen, which is used up in the process. If the process proceeded theoretically the ratio of elementary nitrogen to combined nitrogen would be the same in the outgoing as in the ingoing gases. In practice, a proportion of the ammonia is oxidised to nitrogen and water, and it is important to be able to judge the extent of this undesired reaction. To do so, a 5-litre aspirator is connected to the inlet main through an absorption tower containing 25 c.c. of normal acid, and a similar 5-litre

aspirator to the outlet main through a train of three towers, the first containing 10 c.c. permanganate solution (1 c.c. = 0·001 grm. N) and the others 200 c.c. of sulphuric acid or potash solution. Five litres of gas are collected in each aspirator, the ammonia in the entering gas is estimated by titrating the unneutralised acid in the tower, and the fixed nitrogen in the outgoing gas by analysis of the contents of the towers in the outlet train. The permanganate solution is titrated with ferrous ammonium sulphate, and the contents of the other towers are mixed, made up to 400 c.c., and 20 c.c. analysed in the nitrometer. The outgoing gas, freed from nitrogen compounds, is analysed by absorbing the oxygen in a phosphorus pipette, the rest being nitrogen. The ratio of elementary to fixed nitrogen in the ingoing and outgoing gases can be calculated. In practice, the ratio in the case of the ingoing gases appears to be 16 or 17 to 1. In the outgoing gases it is always somewhat greater than in the ingoing gases, and may be much greater when the plant is functioning badly. In the single example given, the ratio was 16·55 for the ingoing and 20·5 for the outgoing gases, showing a yield of about 82 per cent.

G. C. J.

Determination of Bromine in Mineral Waters and Brines. W. F. Baughman and W. W. Skinner.

(*J. Ind. and Eng. Chem.*, 1919, **11**, 954-959.)—The estimation of bromides in the presence of an overwhelming proportion of chlorides requires certain special precautions in order to avoid the liberation of a small amount of chlorine with the bromine. The authors have investigated and developed a method proposed by Wyss, according to which the solid residue of chlorides and bromides, from which the iodides have previously been removed (*Med. Klinik*, 1910, **24**, 288), is treated with a concentrated solution of chromic acid at the ordinary temperature. Bromine is liberated with only a trace of chlorine, the bulk of the latter remaining in solution in the form of chromyl chloride. At the same time a little chlorobromide is formed, which is removed with the bromine by aspirating. Three tall, 250 c.c., glass-stoppered Drechsel washing cylinders are joined together in series, the connection between the first two being united by fusion and that between the second and third closely coupled by rubber. The bromine is liberated in the first cylinder, and its removal by aspiration is facilitated by filling half the cylinder with glass beads. The intake tube, which is drawn out to a point, extends through the beads nearly to the bottom, and a small funnel is attached to its upper end. The lower end of the intake tubes of the two other cylinders is expanded to a small ball pierced horizontally round the circumference with small holes in order to break up the bubbles. The first cylinder is charged by introducing glass beads to the depth of 1 inch, and then as much of the evaporated saline residue as can be scraped in, lastly filling up half the cylinder with glass beads. A solution is made containing 1 grm. of sodium sulphite and 0·2 grm. of sodium carbonate in 25 c.c. Twenty c.c. of this solution is placed in the second cylinder and 5 c.c. in the third, and each is made up with water to about 200 c.c. The three cylinders are connected together, and a slow current of air is drawn through; then 15 grms. of chromic anhydride, dissolved in 10-12 c.c. of water, is added to the reaction vessel, and the residues of salt left in the evaporating basin in which the sample had been dried are washed in, using water sufficient

to bring the total quantity of liquid in the first cylinder to about 25 c.c. The air is aspirated until the contents of the first cylinder are thoroughly dissolved and mixed, and is then discontinued; the inlet tube is closed with a piece of rubber tubing and a clamp, and a slightly reduced pressure is established in the apparatus to prevent the escape of bromine through the joints of the stoppers. The whole is allowed to stand over night, and a stronger current of air (about 0·5-0·75 litre per minute) is aspirated through for three hours with the addition of four portions of 2 c.c. each of 3 per cent. hydrogen peroxide at intervals of thirty minutes. The aspiration is then stopped, and the contents of the two absorption cylinders containing the sulphite solution are evaporated nearly to dryness. The reaction cylinder is cleaned out, and freshly charged with glass beads and 15 grms. of chromic anhydride. The first absorption cylinder is charged with 10 grms., and the second with 3-4 grms., of potassium iodide each dissolved in 200 c.c. of water. The apparatus is again connected, air is drawn slowly through, and the contents of the evaporating basin are introduced into the reaction vessel using 25 c.c. of liquid. A stronger current of air is passed through for one hour, and the bromine absorbed in the potassium iodide is estimated by titration with thiosulphate solution. Thus the first operation so concentrates the bromine relatively to the chlorine that the estimation in the second operation can be performed with satisfactory accuracy.   J. F. B.

Sulphite Method for Separation and Estimation of Gallium when Associated with Zinc. L. E. Porter and P. E. Browning. (*J. Amer. Chem. Soc.*, 1919, **41**, 1491-1494.)—It is assumed that any indium originally present has been previously separated by sodium hydroxide and that the greater part of the zinc has been removed by precipitating the gallium hydroxide in the presence of ammonium chloride. The reagents used are sodium hydrogen sulphite, and ammonium hydrogen sulphite, formed by saturating, respectively, with sulphur dioxide a 10 per cent. solution of sodium sulphite and dilute (1 : 4) ammonia. To detect gallium, about 5 c.c. of the neutral or slightly acid solution is boiled for two minutes with a drop or two of either of these reagents. A precipitate will appear if 0·2 mgrm. or even less gallium is present. To estimate it, 200 c.c. of the solution are boiled with 5 c.c. of ammonium hydrogen sulphite for five minutes. The supernatant liquor is decanted through a filter and the granular precipitate dissolved by addition of a few drops of hydrochloric acid. The solution is diluted to 200 c.c., gallium precipitated as before, the precipitate filtered off, ignited and weighed as Ga_2O_3. Quantities up to 50 mgrm. can be estimated with an error never exceeding 0·3 mg.   G. C. J.

Estimation of Oxygen and Nitrogen in Electric Weld Metal. J. H. Paterson and H. Blair. (*J. Soc. Chem. Ind.*, 1919, **38**, 328-330T.)—*Nitrogen.*—The sample (2·5 grms.) is dissolved in cold hydrochloric acid and then digested on a hot-plate until any black residue has disappeared. The solution is neutralised with potassium hydroxide and a measured excess (about 3 grms.) added. One or two pieces of zinc which have been coated with copper and washed are then added and the mixture distilled into $\frac{N}{20}$ sulphuric acid, the excess of which is finally titrated with $\frac{N}{50}$ potassium hydroxide, using methyl-orange as indicator. A blank is run on pure

iron turnings which have been heated in a current of hydrogen. This meets Tschischewski's objection (*J. Iron and Steel Inst.*, 1915, Vol. II.) to the use of a copper-zinc couple, which he suggested might reduce nitrogen compounds in the acid used for dissolving the metal. Dispensing with the couple leads to results which may be 40 per cent. below the truth. *Oxygen.*—In steels containing nitrogen as well as oxygen the latter can be estimated by passing purified hydrogen over the heated drillings in the usual manner, provided a correction is made for ammonia formed by reduction of a portion of the nitrogen. This is readily carried out by placing, between the combustion tube and the usual absorption tube, a small tube containing about 3 grms. of phosphorus pentoxide. This is weighed with the absorption tube and the contents are then distilled with alkali hydroxide to estimate the ammonia. This quantity deducted from the increase in weight of the tubes gives the true weight of water formed from the reduction of the drillings. The main difficulty in estimating oxygen in metal arises in the sampling. Coarse drillings may show 0·07 per cent., fine drillings 0·19 per cent, dust 0·68 per cent., complete sample 0·34 per cent., to quote one set of figures given by the authors. G. C. J.

Modification of the Reddrop and Ramage Method for Determination of Manganese in Steel. P. L. Robinson. (*Chem. News*, 1919, **119**, 187-188.)—The steel (1·1 grm.) is heated with 30 to 40 c.c. nitric acid (sp. gr. 1·2) until red fumes cease to be evolved. Then, without cooling, 5 c.c. of ammonium persulphate solution (120 grms. per litre) are added. This immediately oxidises the carbonaceous matter. Five to ten minutes' boiling is allowed to decompose the excess of persulphate. The assay is allowed to become quite cold, 1 to 1·5 grms. sodium bismuthate are added, and the manganese completely oxidised to the permanganic state as in the original method. Excess of bismuthate is removed by filtration through asbestos, and the filtrate is titrated with $\frac{N}{10}$ ferrous ammonium sulphate and $\frac{N}{10}$ permanganate.

It will be seen that this method differs from that of Reddrop and Ramage only in the means adopted for preliminary oxidation of carbonaceous matter and reduction. This is reduced to one operation, time is saved (as no cooling is required), the use of sulphurous acid is avoided, the consumption of bismuthate (an expensive reagent) is reduced by one half, through the use of persulphate, and filtration is made easier. The quantity of persulphate given above suffices for steels with carbon up to 1·5 per cent. It may be increased for high carbon steels. In the case of steel containing chromium reduction is naturally necessary and the final oxidation and titration must be carried out rapidly. G. C. J.

Cause of and Remedy for Certain Inaccuracies in Hausmann's Nitrogen Distribution Methods. S. L. Jodidi and S. C. Moulton. (*J. Amer. Chem. Soc.*, 1919, **41**, 1526-1531.)—Hausmann's methods (*Z. Physiol. Chem.*, 1899, **27**, 95 ; 1900, **29**, 47, 136) estimate acid amide nitrogen correctly, but they underestimate monoamino and diamino nitrogen. The error is the greater, the larger the quantity of magnesium oxide used in the distillation process. Sufficient must be added to make the mixture alkaline, but the least possible excess should be used. Monoamino nitrogen is better estimated by the difference between 100 and the sum of the acid amide nitrogen,

the "humin" nitrogen and the diamino nitrogen than by Hausmann's direct method which leads to low results owing to the interference of phosphotungstic acid, the use of which in large quantity is incidental to his method.   G. C. J.

Separation of Vanadium from Aluminium. P. Wenger and H. Vogelson.

(*Helv. Chim. Acta*, 1919, **2**, 550-553.)—The oxides are mixed with six times their weight of sodium carbonate in a platinum crucible. It is important that this proportion be closely followed. The mixture is heated cautiously until gas is no longer evolved and is then maintained in quiet fusion for three hours. The cooled mass is taken up in water, which yields a clear solution of vanadate and aluminate of sodium. This is diluted to 500 c.c., heated to boiling, and crystals of ammonium nitrate (ten times the weight of the mixed oxides) are added in small portions at a time. The precipitated alumina is filtered off, washed with a very dilute solution of ammonium nitrate, ignited, and weighed. The filtrate containing the vanadate is acidified with hydrochloric acid, neutralised with ammonia, and the vanadium precipitated as barium vanadate by the addition of barium chloride to the boiling solution. The precipitate is filtered off after twenty-four hours, washed with cold water, dried, ignited, and weighed.

When iron is present as well as vanadium and aluminium, all three are precipitated in the cold by means of ammonia, the precipitate washed with very dilute ammonium nitrate solution, and ignited in a platinum crucible. It is then fused with sodium carbonate as described. On treatment with water, the ferric oxide remains undissolved and is filtered off. Vanadium and aluminium are then estimated in the filtrate as described above.   G. C. J.

Analysis of Commercial Zinc. S. Rothschild. (*Chem. Zeit.*, 1919, **43**,

529-530.)—In high grade zinc the impurities likely to exist are cadmium, lead, and iron, and their estimation is easy. For the estimation of cadmium and lead 20 grms. of the metal are treated with 14 c.c. sulphuric acid (insufficient to effect complete solution) and 150 c.c. water. The solution is decanted off and reserved for the cadmium estimation, whilst the undissolved residue is dissolved in nitric acid, and then heated to fuming with sulphuric acid. The filtrate from the lead sulphate is added to the main solution of the zinc and the cadmium in the mixture is precipitated as sulphide. Iron is estimated by dissolving 20 grms. of the metal in excess (20 c.c.) of sulphuric acid in a flask fitted with a Bunsen valve, and then titrating with permanganate.

In less highly refined zinc, which may contain copper, tin, and aluminium, the analysis is much simplified by the use of "cupferron," which separates copper from cadmium and iron from aluminium. The zinc (20 grms.) is treated with 14 c.c. sulphuric acid and 150 c.c. water as above described. The insoluble residue is treated with nitric acid and the insoluble stannic acid separated. Lead is next separated and the filtrate from the lead sulphate added to the main zinc solution in which copper and iron are next precipitated by means of "cupferron." In the filtrate cadmium is precipitated as sulphide, and, finally, aluminium as hydroxide.
                                        G. C. J.

APPARATUS, ETC.

Electrolytic Hydrogen Generator for Laboratory. L. D. Williams. (*J. Soc. Chem. Ind.*, 1919, **38**, 355-356T.)—The apparatus figured in the paper consists of a 250 c.c. pump flask fitted with a cork carrying a straight tube 30 cm. long, and as wide as the neck of the flask will allow, about 15 mm. The anode, which hangs down in this tube, is a strip of sheet lead, 25 cm. long, the lower end as wide as possible, while the remainder is about 10 mm. wide. The upper end is soldered to a copper lead, which is bent over the top of the tube to support the electrode, so that its lower end is 1 cm. above the bottom of the tube. The cathode is a strip of lead foil, thin enough to pass between the cork and the neck of the flask without causing a leakage of gas. It should not be lower than the bottom of the anode tube, and may be conveniently wrapped round this. The electrolyte consists of 20 per cent. sulphuric acid, and should fill the flask nearly to the level of the side tube. The normal load for a cell is 2 ampères, but twice this may be used if external cooling be resorted to. At the normal load, a potential difference of 6 volts is required, corresponding to an efficiency of less than 50 per cent., but this in a small installation is not very important. A convenient arrangement consists of ten such cells, run normally at 1 ampère, a variable resistance to handle up to 3 ampères, an ammeter, and a purifying train. The probable impurities are hydrogen sulphide, oxygen, water vapour, and spray. Oxygen containing ozone may be obtained by reversing the current, or by closing the mouth of the anode tube and providing a delivery tube.
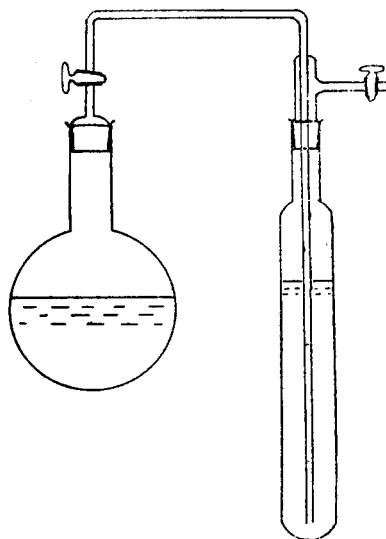
G. C. J.

Method for Determining the Permeability of Balloon Fabrics. J. D. **Edwards and S. F. Pickering.** (*J. Ind. and Eng. Chem.*, 1919, **11**, 966-968.)—The method generally used consists in clamping the fabric between two halves of a circular metal cell which is thereby divided into two equal chambers. Through one chamber is passed a current of pure, dry hydrogen maintained under a pressure of 30 mm. of water as compared with the other side. Generally a current of dry air is passed through the other chamber, and the amount of hydrogen which passes through is estimated either by means of a gas interferometer or by an ordinary combustion method gravimetrically. According to the new method, carbon dioxide is employed instead of air for carrying away the hydrogen which diffuses through the fabric into the second chamber. It is very important to make the permeability tests at a standard temperature, and the cell is suspended in a bath maintained at 25° C. The stream of hydrogen passes out through a water seal which provides the requisite excess pressure in the cell. The carbon dioxide containing the diffused hydrogen from the other chamber passes into the base of an absorption vessel through a small mercury seal. The height of the levelling bulb is adjusted so that the gas enters in a steady stream at the proper pressure, the depth of the mercury seal being carefully adjusted. After the time allowed for the test has elapsed the current of carbon dioxide is diverted and the hydrogen with a little air which has collected in the absorption bulb is transferred to a graduated gas burette, where it is exploded

after mixing with a sufficient volume of air. The volume of hydrogen is then calculated from the contraction observed.

An error common to all permeability methods is introduced by the diffusion of the hydrogen laterally along the textile passing through the rubber where it is clamped between the edges of the cell and 'back through the textile on the other side. This error is best corrected by arranging the fabric with a margin not exceeding 4 mm. in depth in a cell having an area of 250 sq. cm. The error can then not exceed 9 per cent. and may be allowed for if desired.   J. F. B:

Molecular Weight Determination by Direct Measurement of the Lowering of the Vapour Pressure of Solutions. R. Wright. (*J. Chem. Soc.*, 1919, **115-116**, 1165.)—The barometric method as employed by Raoult is too cumbersome for general use, and, at the ordinary temperature, the lowering of the vapour pressure obtained with most solvents is too small to admit of accurate measurement. The author's method for use in the case of a volatile liquid, such as ether (*Proc. Chem. Soc.*, 1912, **28**, 96), is unsuitable for ordinary solvents. Menzies (ANALYST, 1910, **35**, 449, 450) has described an apparatus in which the lowering of the vapour pressure produced by a solute is measured at the boiling-point of the solution, the manometric liquid used being the solution itself, thus enabling very dilute solutions to be employed. The present apparatus resembles that of Menzies in employing the solution itself as the manometric liquid, but the author carries out all measurements at the ordinary temperature. The flask is about 150 c.c. in capacity, and the test-tube 20 cm. long and 2 to 3 cm. wide, all ground connections being carefully made air-tight with rubber grease, which precludes the use of benzene as a solvent, and renders carbon tetrachloride somewhat unsatisfactory. An unsuccessful attempt to replace the rubber grease with syrupy phosphoric acid was made, the acid being incapable of holding up the vacuum.

A weighed quantity of the solute is placed in the test-tube, and, after both flask and tube have been half filled with solvent, the apparatus is connected together and exhausted by means of a vacuum pump, so that all air is boiled out of solvent and solution by gentle warming. When exhaustion is complete, in about half an hour's time, both taps are closed, and the whole is left for a few hours to attain the room temperature. The tap between flask and test-tube should be closed during this period to prevent the forcing back of the solution into the pure solvent as a result of accidental leakage of air into the test-tube. The tap above the flask is gently opened, and, as the vapour pressure of the solvent exceeds that of the solution, the liquid in the delivery tube is depressed below the ordinary level of the liquid in the test-tube.

The difference in height is measured either by means of a cathetometer or by having the delivery tube graduated in mm.; in either case the observed height must be corrected for capillarity. The room temperature is noted, and air is then admitted to the apparatus by gently raising the tap above the flask out of its seat, care being taken to prevent a rapid inrush of air. The effect of capillarity is now noted, after which the test-tube is removed and weighed, thus determining the mass of the solution, which should have its specific gravity determined in addition if the solution is a concentrated one. The molecular weight of the solute is calculated from the usual formula for the lowering of the vapour pressure:

$$M = \frac{P' Sm}{(P - P')s}$$

where M and m are the molecular weights of solute and solvent, S and s their weights in grms., P the vapour pressure of the pure solvent, and P′ that of the solution. P, at the temperature of the experiment, is obtained from tables, interpolating if necessary on the assumption that the curve connecting the *log.* of the vapour ressure and the temperature is a straight line. The following results were obtained with alcohol:

Solute.	S	s	T	P	H	P − P′	M	M (true).
Benzoic acid ...	5·698	78	19·5	42·6	17·8	1·084	128	122
Carbamide ...	4·043	108	19·0	41·4	21·0	1·237	56	60
Acetamide ..	3·781	102	17·0	36·6	17·5	1·039	59	59

Column H gives the manometric readings in mm. of the solution (corrected for capillarity). Water as a solvent is unsuitable, owing to its low vapour pressure and molecular weight. H. F. E. H.

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ERRATA.

(*P. 343 October issue.*)

Fourth line from top, for "hot alcohol" read "hot water."
Sixth ,, ,, ,, for "20 c.c." read "10 c.c."

REVIEWS.

TEXTBOOKS OF PHYSICAL CHEMISTRY.   STEREOCHEMISTRY.   By ALFRED W. STEWART,
    D.Sc., Professor of Chemistry in the Queen's University of Belfast.   Second
    Edition, with fifty-eight illustrations.   1919.   London : Longmans, Green
    and Co., 39, Paternoster Row.   Price 12s. 6d. net.

In the second edition of Professor Stewart's " Stereochemistry " most of the
faults of the first edition have disappeared and a large amount of new matter has
been added.   The result of this revision is a book of a much higher standard of
excellence, which contains a very satisfactory account of all the essential phenomena
covered by the word stereochemistry.   Full references are given to the original
papers and there are very few points in which the author's treatment of the subject
is open to criticism.   It may be suggested that Pickard's work on Homologous
Series should now find a place in the text, where at present it appears to have been
omitted, even from the references and footnotes.   It is also incorrect to state (on
page 91) that ethyl tartrate shows mutarotation ; if this were so, there would be no
need for further demonstration of the view that the changes of rotatory power which
result from alterations of solvent or of temperature are due to chemical changes
instead of being purely physical phenomena.   It is particularly refreshing to find in
Part III. of the book a critical account of Werner's hypotheses, which have sometimes
been allowed to escape criticism on account of the close study which is required for
their interpretation.

As a general point it may be suggested that the stereochemistry of carbon
compounds would be more intelligible if, in making use of the tetrahedron formula for
the carbon atom, more attention were directed to the lines joining the central carbon
to the four corners of the tetrahedron and less prominence given to the edges of the
tetrahedron ; the latter do not appear to have any chemical significance, in view of
the absence of important forces acting between the hydrogen atoms of the marsh gas
molecule.   This criticism becomes still more pointed when, as on page 128, two of
the edges of the tetrahedron are knocked away in an attempt to convert the carbon
into a nitrogen atom.   The elastic rods which join the atoms together in Engler's
models give a far more graphic representation of the facts of valency and of the
theories of stereochemistry than the relatively clumsy tetrahedra, and it would be a
real advantage if some diagrammatic representation based upon these models were
made use of in printed discussions of stereochemistry.

T. M. LOWRY.

THE CHEMISTRY OF COLLOIDS.   Part I., Kolloidchemie; by RICHARD ZSIGMONDY,
    translated by ELLWOOD B. SPEAR.   Part II., Industrial Colloidal Chemistry ; by
    ELLWOOD B. SPEAR.   1917.   London : Chapman and Hall, Ltd.   Price 13s. 6d. net.

The greater portion of this book (238 pages out of 274 of text) is an American
translation of Zsigmondy's "Lehrbuch der Kolloidchemie," first published in 1912.
A work by one of the inventors of the ultra-microscope, and the author of classical
papers on gold sols, protection, etc., naturally calls for attention, while the deliberate
description "Lehrbuch"—i.e., textbook—challenges criticism from the pædagogic
point of view.   In this respect the book can hardly be pronounced entirely successful.
The first four chapters are devoted to General Considerations, Classification,
Properties of Colloids, and Theory.   They inevitably refer to individual examples of
which the student, supposed to be unfamiliar with the subject, has no knowledge, so
that his comprehension will be necessarily incomplete until he has read the
descriptive part of the book.   This consists of eight chapters devoted to the descrip-
tion of such individual sols and gels, which are simply divided (for the first time, as
the author states) into Inorganic and Organic Colloids.   Now the problem of classi-
fication is of course a very old one, faced already by the Greeks, who fully recognised
that any *differentia* could be taken as a basis : men can be divided into rich and poor
as well as into (say) brachy- and dolicho-cephalic.   The choice must be settled by the
purpose of the classification ; in the present instance hardly anyone will agree that
the deliberate selection of a division which does not coincide with differences in any
one of the fundamental properties of colloids, *qua* colloids, can be considered a happy
one.   Some organic dyes—e.g., Kongo Rubin—behave in many respects almost exactly
like gold sols, while silicic or stannic acid approximates much more to some typical
organic sols than to metal or sulphide sols.   The reviewer—in common, as he believes,
with most recent workers in this field—unhesitatingly prefers Wo. Ostwald's
classification, being convinced of both its reality and its fruitfulness.

The treatment in the descriptive chapters is clear and adequate, with the
exception of many of the descriptions of experimental procedure, which are quite
insufficient.   Even intelligent students will not be able to make good dialysers, or to
prepare silicic or stannic acid, by the aid of the book alone.   A certain disproportion
in the space allotted to various subjects is noticeable and must be put down to the
author's bias in favour of those to which he has made important contributions.
forty pages devoted to metal sols, a pure laboratory product, and thirty to all the
proteins, including a typical substance like gelatin, can hardly be explained on any
other assumption.   The reader familiar with the subject can correct this lack of
balance, but it is apt to distort the views of the beginner.   Notwithstanding these
defects, the book is one to be read by anyone seriously interested in the subject.

The translation is on the whole correct, though it shows in many places the
subtly demoralising effect which translating from the German seems somehow to
exert on the style of writers presumably familiar with their own language.   Nobody
writing a book himself would say " Under soaps we will understand the salts, etc.,"
or " Noteworthy is the iron content"   In the hopeless search after an equivalent

for *Quellung* the translator has hit on "distention" : even correctly spelled the term is less felicitous than the usual "swelling." On the other hand, words like *lipoid-unloeslich*, *Schwellenwert*, and *anaetzen*, for which there are quite obvious English terms, are left untranslated in inverted commas.

The translator has added a second part, entitled "Industrial Colloidal Chemistry," which, in about thirty pages, attempts to deal with subjects "of no inconsiderable magnitude" such as Smoke Prevention, Rubber, Tanning, Milk (its inclusion under the heading of industry is not very obviously justified), Clays and Soils, etc. All that these sketches do amounts simply to pointing out that colloid chemistry has, or may at some future time have, a bearing on these subjects. Since many of the industries employing typically colloidal material are as old as history, or older, this is probably all that can be done at present; whether it is worth doing is another matter. The reviewer cannot but feel a decided lack of sympathy with all such attempts, too frequent of late, to justify science in the eyes of those who neither know nor care about it, by claims or promises of material results.

                                                                    EMIL HATSCHEK.

PRACTICAL PHYSIOLOGICAL CHEMISTRY. Fifth Edition. By SIDNEY W. COLE, M.A.
    1919. Cambridge: W. Heffer and Sons, Ltd. Price 15s. net.

During recent years great advances have been made in the analytical technique of biochemistry, as a result of which considerable additions have been made to our knowledge in physiology and clinical medicine.

A complete compendium of methods would require a handbook of much larger proportions than that under review. Considerable discretion must, therefore, be used by any author writing for students, who has to supply a textbook of reasonable dimensions. As great latitude is possible for admissions or exclusions of methods, correspondingly great differences of opinion can exist as to the adequacy or otherwise of a book like that of Mr. Cole's. The writer of this review may, however, unhesitatingly express the opinion that the fifth edition of Mr. Cole's book is a valuable addition to biochemical literature. He would like to point out, in the first instance, the parts of the book which he considers of especial value. As might be expected from a worker in the Cambridge laboratories, the parts dealing with the degradation products of the proteins and the preparation of the amino-acids are admirable. These will be found useful not only to students as exercises in preparation, but also to research workers, who so often find it necessary to prepare amino-acids for their investigations.

Equally useful and adequate are the descriptions of the new methods for estimation of sugars, and the newer "micro"-methods for determining the constituents of the blood, due for the most part to the labours of Folin in America and Ivar Bang in Sweden.

The methods of urine analysis are also well described. There are, however, parts of this book which might, with advantage, be amplified. The space accorded

to the chemistry of the fats seems quite insufficient. A more detailed account should have been given of such important analytical operations such as the determination of saponification and iodine values. The earlier methods for estimation of sugars, such as the ordinary Fehling method, the gravimetric method, etc., which have formed the basis of so many classical researches, should have been described in greater detail. The reviewer would also like to suggest that the methods of preparing a few more proteins might have been added; he has himself found, for example, that the preparation of the wheat proteins forms an admirable class exercise.

As a complete compendium of analytical operations was not aimed at by the author, it is very easy for a reviewer to suggest additions. · If the book is to be kept within reasonable limits, certain deletions should also be proposed. An account of the principles of stereoisomerism might very well be left to an ordinary elementary textbook on organic chemistry. Less space might also with advantage be given to the description of the large number of colour reactions with which biological chemistry abounds. This question of elisions will require to be carefully considered by the author when he prepares the next edition of his book, as even since this went to the press important papers have been published, such as that of Dakin on the amino-acids, which will require a further extension of parts of this book.

S. B. SCHRYVER.

THE SIMPLE CARBOHYDRATES AND THE GLUCOSIDES. By E. FRANKLAND ARMSTRONG, D.SC., PDH., (MONOGRAPHS ON BIOCHEMISTRY. Edited by Dr. R. H. A. PLIMMER and Dr. F. GOWLAND HOPKINS.) Third Edition. Pp. x + 239. 1919. London: Longmans, Green and Co. Price 12s. net.

The first edition of this monograph was published in 1910 and contained 112 pages, whilst the second, with 170 pages, appeared in 1912. Extended by 69 pages, the present edition consists of an introduction, nine chapters, a bibliography, and an index. The general plan of the work has been retained, and, as this is so well known, nothing need be said concerning it. Although of the many sugars that have been prepared in the laboratory by synthesis few have up to the present been found in nature, we may in the future expect to come across many of these. Moreover, since the ordinary monohexoses have been shown by Lobry de Bruyn to undergo changes into isomerides by the action of alkalis and alkali earths, it is not surprising that glutose has been found in cane molasses to the extent of 1 to 5 per cent. Its formation is probably to be ascribed to the action of the lime used in sugar manufacture on the natural reducing sugars of the cane juice. These facts are in themselves sufficient to commend a work such as Dr. Armstrong's, dealing essentially with modern researches on the sugars, to the attention of analytical chemists.

A clear and concise account is given of the work of Emil Fischer and of those who have followed him, among which must be mentioned that of Dr. Armstrong himself. The chapters on the natural and synthetic glucosides, and of the significance of the glucosides in metabolism, are particularly interesting and suggestive. The view is

put forward that the products of the hydrolysis of glucosides other than sugars by specific enzymes stimulate down-grade changes which occur at night.   Respiration in plants and the ripening of fleshy fruits is also dealt with, and an explanation is given of the formation of fats and other compounds in plants from carbohydrates. Not the least important feature of the monograph is the excellent bibliography.

<div align="right">ARTHUR R. LING.</div>

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NOTICE.

THE Presidents of the Institute of Chemistry and this Society propose to send a joint letter to all Local Authorities relating to the question of the payment of higher fees to Public Analysts.   If any Public Analyst takes exception to this course being adopted in his particular case, he is requested to communicate with the Registrar of the Institute of Chemistry.