Vol. Ll., No. 609;

# THE ANALYST

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

AN Ordinary Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, November 3rd, 1926, Mr. E. Richards Bolton, F.I.C., President, being in the chair.

A Certificate was read for the first time in favour of Mr. Stanley Grove Burgess, B.Sc., A.I.C.

Certificates were read for the second time in favour of:—Miss Gertrude Garland Andrew, B.Sc., Messrs. Charles Edward Barrs, F.I.C., Thomas Hedley Barry, Gordon Watson Douglas, B.Sc., Clarence Victor Ferriss, B.Sc., A.I.C., and Reginald Charles Pakes, B.Sc.

Mr. Stafford Aston, F.I.C., was elected a Member of the Society.

The following papers were read and discussed:—"Investigations into the Analytical Chemistry of Tantalum, Niobium, and their Mineral Associates: VI. The Precipitation of the Earth Acids by Sodium Compounds," by W. R. Schoeller, Ph.D., and C. Jahn; "A Simple Method of Testing for the Presence of Sulphites in Foods," by A. E. Parkes, F.I.C.; and "A Critical Review of the Methods of Analysing Waters, Sewages and Effluents, with Suggestions for their Improvement," by J. W. Haigh Johnson, M.Sc., F.I.C.

## NORTH OF ENGLAND SECTION.

A MEETING of the North of England Section of the Society was held in Manchester on Saturday, November 13th. Professor Roberts was in the chair, and sixteen members were present.

The following papers were read and discussed:—(1) The Determination of Mixtures of Palm Kernel and Coconut Oils; (2) A Short Method of Determining Small Quantities of Butter Fat in Margarine. By G. D. Elsdon, B.Sc., F.I.C., and Percy Smith.

The next meeting has been fixed for January 15th, 1927, at Leeds, when it is proposed to discuss the new Preservatives Regulations.

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# Notes on Variations in the Composition of Milk.

## By J. F. TOCHER, D.Sc., F.I.C.

(Read at the Meeting, April 7, 1926.)

In a recent monograph\* the results of a study of the nature and the extent of the variations in the percentages of the various constituents of milk were considered in detail. In order to make the results representative of the dairy cow population of Scotland, samples were taken at random all over Scotland, the randomness extending not only to the area, but also to the breed, age and stage of lactation period and other factors. The objects were to find the nature of the frequency distributions of the percentages of the various constituents of milk, and also to ascertain how far the various constituents were related to each other. The results are useful as showing how far milk from an individual cow varies in each of the constituents, including the water content of milk. From the scientific point of view minimum and maximum values of the constituents of milk from individual cows are important. From the legal point of view minimum values of the constituents of *bulked milk* from herds of various sizes are of great importance. By taking from the data random groups of cows from the total number of cows whose milk was sampled, one can determine minimum values for the various constituents of the bulked milk. Take, for example, solids-not-fat, being a combination of a number of the constituents—one can ascertain the nature of the distribution of the solids-not-fat content of the mixed milk of random groups of N cows. One can also determine, from the nature of the distribution and from the frequency. curve fitted to the data, the probability of milk containing, say, less than 8.5 per cent. solids-not-fat for these random herds. These and other problems have been discussed and definite results reached in the monograph above referred to.

The following notes summarise certain results reached since the publication of the monograph. These and other results will be fully discussed in a paper to be published at an early date.

RESULTS WITH DIFFERENT BREEDS OF Cows.—The table below (Table I) shows the mean values of the percentages of the various constituents, and also the number of pounds per milking for (1) Ayrshire cows, (2) Friesian cows, and (3) other cows, whose milk was sampled under the Inter-Departmental Committee's Scheme.

\* "Variations in the Composition of Milk." H.M. Stationery Office, Edinburgh, 1925. (ANALYST, 1926, 146.)

	breed of Low,						
	Ayrshires.		Frie	sians.	Others.		
Character.	Per Cent.	Lbs. per milking.	Per Cent.	Lbs. per milking.	Per Cent.	Lbs. per milking.	
Butter fat	4.085	0.547	3.625	0.560	3.819	0.556	
Solids-not-fat	8.753	1.173	8.617	1.331	8.856	1.289	
Albumin	0.753	0.101	0.766	0.118	0.725	0.105	
Casein	2.431	0.326	$2 \cdot 339$	0.361	2.417	0.352	
Protein	3.267	0.438	3.107	0.480	3.215	0.468	
Ash	0.691	0.093	0.707	0.109	0.709	0.103	
Lactose	4.568	0.612	4.616	0.713	4.703	0.684	
Water	87.162	_	87.758		87.325	,	
Yield	در	13.399		15.452		13.919	

TABLE I.



The results in the above table show that Ayrshires have the highest percentages of butter fat and protein, whilst "other cows" have the highest percentage of solids-not-fat. When, however, the total butter fat per milking is considered it is seen that Friesians have a slightly higher amount than Ayrshires and other cows. The differences, however, are not very great. Friesians have the highest amounts of total solids and yield per milking, due to the high correlations between yield per milking and solids generally per milking. A problem of practical importance is whether it is better to select cows with high average percentages of butter fat and solids-not-fat during a lactation period, or to select cows for their total butter fat and total solids-not-fat for a lactation period. Lactation periods

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vary in length, and there are large variations in yield for a lactation period. There is at present considerable selection for yield, on the one hand, and for high percentage of butter fat, on the other. The writer is engaged in a statistical study of these problems.

RELATIONSHIP BETWEEN YIELD AND (a) BUTTER FAT; (b) SOLIDS-NOT-FAT AND (c) TOTAL SOLIDS.—The correlation\* between yield per milking and total solids-not-fat per milking is very high (0.991). The correlation between yield per milking and total butter fat per milking is less, owing to the variable character of butter fat (0.842). The correlation between yield per milking and total solids per milking is intermediate between the foregoing (0.959). The diagram on p. 607 (Diagram I) shows the relationship between these factors. It is clear, therefore. that selection for yield during a whole lactation period means indirect selection of total butter fat and of total solids-not-fat for a lactation period. It has been shown; that there is a distinct relationship between the percentage of butter fat and the percentage of solids-not-fat. The degree of correlation is there shown to be 0.254. In other words, if the samples of milk, drawn on the same day from different cows or from different herds of cows are analysed, it is found that, on an average, a high percentage of butter fat is associated with a high percentage of solids-not-fat, and vice versa. This means that, in general, a good butter fat. producer is also a good solids-not-fat producer. On the other hand, if the daily samples of the bulked milk of the same herd are analysed day after day, it is found that the correlation between butter fat and solids-not-fat is negative. That is to

\* The correlation between two variables is measured by various methods. The chief of these are (1) by finding the correlation coefficient, and (2) by finding the correlation ratio. If the two variables are denoted by x and y, and their mean values by  $\bar{x}$  and  $\bar{y}$ , and their standard deviations by  $\sigma_x$  and  $\sigma_y$ , then the correlation coefficient is defined by  $r = \frac{S(x-\bar{x})(y-\bar{y})}{N\sigma_x\sigma_y}$ ,

where S means the summation of all products of x and y deviations from their respective means, and N represents the number of observations. The formula shows that the deviations are reduced to a common standard unit of variability, by dividing the relative deviations by their respective standard deviations. The value of r may range from -1 through zero to +1. When a straight line fits the observations, a negative value of r indicates that as the particular values of x increase, the mean values of the arrays of y decrease. A positive value of r then indicates that as the particular values of x increase, the mean values of the arrays of y also increase. An excellent method of seeing correlation graphically is to plot the mean values of the y arrays of x, that is  $\bar{y}_x$  for particular values of x. In many cases points so obtained lie approximately on a straight line, and if this line slopes upwards to the right, the correlation is positive, whilst if it slopes downwards to the right, the correlation is negative. If it happens that these mean values,  $\bar{y}_x$ , lie practically upon a straight line parallel to the x axis, then it can be said there is no correlation between x and y, that is, r is approximately zero.

The correlation ratio is defined by  $\eta = \sigma_M/\sigma_y$ , where  $\sigma_M$  is the standard deviation of the array means of y, *i.e.*,  $\sigma_M^2 = \frac{Sx_x(\overline{y}_x - \overline{y})^2}{N}$ , and  $\sigma_y$  is the standard deviation of the whole y distribution. This measure of correlation does not show whether the correlation is positive or negative, whilst the correlation coefficient does, as the value obtained by the formula has a + or - sign indicating positive and negative correlation, respectively.

It is seen from the following equation to the straight line  $\frac{y-\overline{y}}{\sigma_y} = r \times \frac{x-\overline{x}}{\sigma_x}$  that r is a.

constant of proportion. In many cases, however, the means of the arrays  $(\bar{y}_x)$  do not lie on a straight line, and regression equations have to be found to fit the data with regression curves. In these cases r loses its significance as a constant of proportion.

say, a high percentage of butter fat is associated, in general, with a lower percentage of solids-not-fat than the average. This arises from the fact that the percentage of total solids-not-fat of the bulked milk of one herd from daily samples, is much less variable (three times less) than the percentage of total solids-not-fat in the bulked milk of different herds. In a herd of 24 cows, for a period of 39 days, the value of r was found to be  $r_{fs} = -0.2271$ , where f = butter fat per cent. and s==solids-not-fat per cent. The values of  $r_{(f+s)f}$  and  $r_{(f+s)s}$  were found to be 0.3180 and 0.8514, respectively. If the percentages in total solids in these samples were always constant, the value of  $r_{fs}$  would, of course, be -1. The reason for the value of  $r_{fs}$  being -0.2271 from the daily samples in the same herd, when compared with the value  $r_{fs}$  + 0.2541, from milks of different herds, lies in the fact that the coefficient of variation for total solids from milks of different herds is 7.86, whilst for total solids from milks of the same herd it is 2.58, or three times less. Thus, with a more constant proportion of total solids in the milk of one herd, the positive correlation (from different herds) is converted into a negative correlation (from one herd) which would reach unity if the proportion of total solids was constant in every sample.

INDLE II.	1	ABLE	Ш.
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Butter Fat.	Percentage Casein.	Casein.	Percentage Butter Fat.
1.625	2.001	1.67	3.215
1.875	2.044	1.83	3.386
$2 \cdot 125$	2.088	1.99	3.556
2.375	$2 \cdot 132$	$2 \cdot 15$	3.726
$2 \cdot 625$	$2 \cdot 176$	$2 \cdot 31$	3.897
2.875	$2 \cdot 220$	$2 \cdot 47$	4.067
$3 \cdot 125$	$2 \cdot 264$	$2 \cdot 63$	4.238
3.375	2.308	2.79	4.408
3.625	2.352	$2 \cdot 95$	4.578
3.875	2.396	$3 \cdot 11$	4.749
$4 \cdot 125$	$2 \cdot 440$	3.27	4.919
4.375	2.483	3.43	5.099
4.625	$2 \cdot 527$	3.59	5.260
4.875	2.571	3.75	5.430
5.125	2.615		
5.375	2.659	—	
5.625	2.703		_
5.875	2.747		<u> </u>
6.125	2.791	,	
6.375	2.835		·
6.625	2.879		
6.875	2.923	<u> </u>	
7.125	2.966		
7.375	3.010	_	

RELATIONSHIP BETWEEN CASEIN AND BUTTER FAT.—It is important to consider whether any one constituent more than another in solids-not-fat is responsible for the correlation between butter fat and solids-not-fat percentages. The percentages of casein and the percentages of butter fat were tabulated, and the degree

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of correlation between them determined. The correlation coefficient, r, was found to be=0.46, whilst the correlation ratio,  $\eta$  was found to be 0.48. The relationship was found to be linear in character. Table II shows the average percentages of casein associated with particular values of butter fat percentages and the average percentage of butter fat associated with particular percentages of casein.

Diagrams II and III graphically illustrate the relationship. It is clear, therefore, that casein is mainly responsible for the relationship between solidsnot-fat and butter fat in the bulked milk of different cows or herds already established by the author.



RELATIONSHIP BETWEEN CASEIN AND PERIOD OF LACTATION.—It is important to know whether the proportion of casein increases or decreases according to the number of weeks a cow is being milked. The following Table (Table III) shows the average proportion of casein associated with particular numbers of weeks in milk.

## TABLE III.

Weeks Casein, per cent.	$2 \\ 2 \cdot 286$	$5 \\ 2 \cdot 300$	$\frac{8}{2\cdot 316}$	$\frac{11}{2\cdot 334}$	$\begin{array}{c} 14 \\ 2\cdot 354 \end{array}$	$17 \\ 2.376$	$\begin{array}{c} 20 \\ 2 \cdot 400 \end{array}$	$\begin{array}{c} 23 \\ 2 \cdot 425 \end{array}$
Weeks Casein, per cent.	$\begin{array}{c} 26 \\ 2 \cdot 453 \end{array}$	$\begin{array}{c} 29 \\ 2 \cdot 483 \end{array}$	$32 \\ 2 \cdot 514$	$35 \\ 2 \cdot 547$	$\frac{38}{2\cdot 583}$	$41 \cdot 7$ 2 \cdot 628	$52 \\ 8 2$	2·4 ·779



It is seen from Diagram IV that the proportion of case is lowest after calving (2.286 per cent.), and gradually rises during the lactation period, and is highest at the termination of the lactation period (2.779 per cent.). The proportion of case in is negatively correlated with yield of milk.

RELATIONSHIP OF NON-FATTY SOLIDS (OTHER THAN CASEIN) AND BUTTER FAT. —The relationship between non-casein-non-fatty solids and butter fat was next considered. The undernoted table (Table IV) shows the average percentage of butter fat associated with particular values of non-fatty solids other than casein (see also Diagram V).

### TABLE IV.

Non-casein solids-not-fat Butter fat, percentage	$5.05 \\ 4.273$	$5 \cdot 15 \\ 4 \cdot 254$	$5.25 \\ 4.236$	$5.35 \\ 4.217$	$5.45 \\ 4.198$	$5.55 \\ 4.179$	$5.65 \\ 4.160$	5·75 4·141
Non-casein solids-not-fat Butter fat, percentage	$5.85 \\ 4.122$	$5.95 \\ 4.103$	$6.05 \\ 4.084$	$6 \cdot 15 \\ 4 \cdot 066$	$6.25 \\ 4.047$	$6.35 \\ 4.028$	6∙45 4∙009	$6.55 \\ 3.990$
Non-casein solids-not-fat Butter fat, percentage	$6.65 \\ 3.971$	$6.75 \\ 3.952$	$6.85 \\ 3.933$	$6.95 \\ 3.914$	7∙05 3∙895	$7 \cdot 15 \\ 3 \cdot 877$	$7.25 \\ 3.858$	7∙35 3∙839

It is seen that a high average proportion of butter fat is associated with low values of non-fatty solids other than casein and that the average percentage of butter fat decreases with the increasing percentages of non-casein-non-fatty solids. In other words, the greater the amount of butter fat, the less on an average is that of the non-fatty solids other than casein—a result mainly due to lactose.\*

In order to see graphically the relationship between solids-not-fat and butter fat and casein and butter fat the Diagram VI has been prepared. The higher relationship between casein and butter fat, when compared with solids-not-fat and butter fat, can be seen at once from the diagram. The effect of the presence of lactose and other constituents in solids-not-fat is to reduce the correlation between solids-not-fat and butter fat, when compared with the correlation between casein and butter fat.

SUMMARY.—These notes show that the Friesians give a higher quantity of solids-not-fat in general, and of lactose in particular, per milking than Ayrshire or other cows. Ayrshires, however, show a higher *percentage* of butter fat and a higher *percentage* of solids-not-fat per milking.

Whilst the yield of milk is very variable from cow to cow, and whilst it has been shown that yield and percentage of butter fat are slightly correlated negatively, there is a high correlation between yield per milking and solids-not-fat per milking. The correlation between total butter fat per milking and yield per milking is also fairly high, but is significantly less than the correlation between total solids-notfat and yield. It is thus clear that yield can be used as a measure of the total amount of solid matter in milk with a fair degree of accuracy.

\* See pages 62 and 167, "Variations in the Composition of Milk."

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Whilst it has been shown that there is a positive correlation between solidsnot-fat and butter fat percentages in the bulked milk of different herds, this relationship does not hold for the daily samples of bulked milk from one herd. The relationship between solids-not-fat percentage and butter fat percentage in the latter case is negative, due to the small variation in the total solids from day to day in the bulked milk from one herd.

There is a positive correlation between the percentage of casein and the percentage of butter fat, and this high positive correlation is largely responsible for the positive correlation between butter fat and solids-not-fat percentages from the bulked milk from different herds of cows.

The percentage of casein present in milk is lowest after calving, and gradually rises during the lactation period until it reaches a maximum at the end of the period.

The relationship between non-fatty-solids (other than casein) and butter fat is negative. This is largely due to the fact that the correlation between butter fat and lactose is negative, as lactose forms a large proportion of the non-fattysolids other than casein.

### DISCUSSION.

The PRESIDENT remarked that so many figures were now available in answer to the question "What is milk?" that milk could no longer be adequately described as the fluid given by a cow. In future, he thought, it should be described as the fluid given by a well-fed and properly kept cow. He contended that the cowkeeper could readily ascertain the sort of milk that was being given. Milk from a C3 cow was not desirable.

Capt. JOHN GOLDING asked how far the composition of the milk depended on the age of the cow and the period of lactation. Had samples been taken at each milking? In his work on the analysis of the milk of individual cows he found a possible variation from 2 per cent. one morning to 7 per cent. the next evening. How was the keeper of the individual cow under such circumstances to maintain a "Presumptive standard" of 3 per cent? Legislation was called for.

Mr. F. E. DAY asked if the different states of lactation period were fairly evenly spread over the whole year, and if there were local variations in the composition of milk. He had found a sudden drop in the solids—not fat—at the beginning of the lactation period, followed by a rise.

Dr. TOCHER, in his reply, said that there was a widespread desire for a change in the Regulations governing the quality of milk sold to the public. The Scottish Inter-Departmental Committee which reported in 1922 recommended that there should be a legal minimum limit for both butter fat and solids-not-fat. If this recommendation were adopted by Parliament, it was clear that dairymen would have to exercise greater care in the selection of cows and would have to sell only the bulked milk from a fairly large herd; otherwise there was a probability of milk falling below the prescribed minimum limits for butter fat and solids-not-fat at various intervals. In answer to Captain Golding, Dr. Tocher said that in his monograph on "Variations in the Composition of Milk" he had shown the extent of the relationship existing between the composition of milk and age and the composition of milk and the number of weeks which the cows had been in milk. The milk analysed was the total milk given by the cow at one milking. He hoped to

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publish soon the results of the analysis of variations in the composition of milk from day to day. The percentage of butter fat and the yield of milk were clearly functions of age, particularly the yield per week. The precise relationship between butter fat and weeks in milk and of solids-not-fat and weeks in milk had both been worked out, and it was clear from the results that the proportions of both butter fat and solids-not-fat were largely a function of the duration of the lactation period. In answer to Mr. Day, Dr. Tocher said that an attempt had been made to ascertain whether there were local variations in the composition of milk. There was no direct evidence, from the Scottish data, of geographical differences, but this did not mean that geographical variations did not exist. It was extremely difficult to carry out an investigation on the geographical variations in the composition of milk, on account of the interference of other factors whose constancy could not be assured. The chief differences in the composition of milk were, however, more likely to be due to different breeds, to variations in age, to variations in the number of weeks milked, to variations in the feeding and to other factors.

# Investigations into the Analytical Chemistry of Tantalum, Niobium, and their Mineral Associates. VI. The Precipitation of the Earth Acids by Sodium Compounds.

BY W. R. SCHOELLER, Ph.D., AND C. JAHN.

(Read at the Meeting, November 3, 1926.)

THIS Section is mainly an account of the technique involved in the preparation of sodium tantalate and niobate, their solubility and composition, and the recovery of the oxides from the salt solutions in a weighable form. The analytical applications of the sodium compounds will, it is hoped, be published in due course, whilst the determination of their properties is preferably recorded in a separate paper.

An inquiry into the subject of earth acid analysis must lead to the conclusion that the chief cause of its difficulties and uncertainties is the want of easily accessible, non-colloidal tantalum and niobium compounds practically stable in aqueous solution. The earth acids as obtained by hydrolysis-by far the most common precipitate with which hitherto the analyst has had to deal-exhibit undesirable properties from the analytical point of view; they are obtained from an intermediate colloidal phase, a circumstance attended by unavoidable drawbacks such as strong adsorption and slow or incomplete flocculation; they have a tendency to form complexes with kindred elements and with each other ("loss of individuality"). It is due to this property that a satisfactory process for the separation of titania from the earth acids has not yet been discovered. On the other hand, Marignac's method for the separation of tantalum from niobium has been able, in spite of its weaknesses, to hold its own against rival processes because it utilises the crystallisable double fluorides; in Powell and Schoeller's tannin method for the same separation, niobium is converted into the comparatively stable, crystalloidal ammonium oxaloniobate (ANALYST, 1925, 50, 485).

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These few remarks may suffice to indicate the direction in which progress in earth-acid analysis will be achieved, namely, through the elaboration of methods in which precipitates of definite composition and crystalline habit or non-colloidal nature are obtained under conditions that preclude the formation of undesired complexes. These considerations induced us to investigate the analytical possibilities of the salts of tantalic and niobic acid, and in particular their sodium compounds.

TANTALATES AND NIOBATES OF THE ALKALIS.—These are obtained when the pentoxides are fused together with alkali carbonate or hydroxide; and, inasmuch as sodium tantalate and niobate are more or less insoluble in solutions of high sodium ion concentration, they are precipitated when solutions of the potassium salts interact with sodium compounds. The conditions of salt formation between the earth acids and the alkalies are unlike those obtaining with the stronger acids, and a number of salts of varying degrees of complexity, claimed to have been isolated and analysed, are described in the literature. The only ones we at present believe to be of analytical interest are the following:

$4K_2O.3Ta_2O_5.16H_2O$	$4K_{2}O.2Nb_{2}O_{5}.16H_{2}O.$
$4Na_2O.3Ta_2O_5.25H_2O$	$7N\bar{a}_2O.6N\bar{b}_2O_5.32\bar{H}_2O.$

The three salts in which the ratio of base to acid is 4:3 are known as hexatantalate and hexaniobate. In the modern, more precise nomenclature, which is here used, the ratio of base to acid is substituted for the prefix, thus: 4:3 tantalate. Precipitated sodium niobate has been variously described as the hexa (4:3) and meta (1:1) salt, but 4:3 sodium niobate has never been isolated: according to Bedford (*J. Amer. Chem. Soc.*, 1905, 27, 1216), the stable compound is the intermediate 7:6 salt. We may say at once that we confirm Bedford's conclusions, in that the sodium niobate we have obtained by precipitation is undoubtedly the 7:6 salt. Only one other salt, which has a certain bearing on this investigation, need here be mentioned. In the course of his researches on Niobium Compounds (*Ann. Chim. Phys.*, 1866, 8, 23), Marignac obtained a pulverulent precipitate by treating a solution of potassium niobate with one of potassium hydroxide. On analysis the precipitate proved to be 3:1:3 potassium sodium niobate,  $3K_2O$ . Na<sub>2</sub>O.3Nb<sub>2</sub>O<sub>5</sub>.9H<sub>2</sub>O, the potassium hydroxide used being found contaminated with sodium hydroxide. No analogous tantalate is described in the literature.

## PRECIPITATION OF THE EARTH ACIDS BY SODIUM SALTS.

A. PRECIPITATION IN CONCENTRATED SOLUTION.—Concentrated solutions of potassium tantalate and niobate are precipitated, not only by sodium hydroxide or carbonate, but by the chloride or nitrate. Obviously the use of neutral salts is to be preferred in analytical work, and we finally adopted sodium chloride as the most accessible and economical reagent.

Solubility Tests.—Twelve 20 c.c. portions of a solution, each containing 0.076 grm. Ta<sub>2</sub>O<sub>5</sub> and 0.046 grm. Nb<sub>2</sub>O<sub>5</sub> as potassium salts, were precipitated in the cold, with and without addition of potassium carbonate, with solid sodium chloride or

nitrate, as shewn below. After standing overnight, the solutions were filtered and the non-precipitated earth acids recovered from the filtrates by slight acidification and boiling with a little sodium acetate.

	Weight of earth acids in filtrates:				
Precipitant.	No K <sub>2</sub> CO <sub>3</sub> added.	3 grms. K <sub>2</sub> CO <sub>8</sub> added.			
NaNO <sub>3</sub> : saturated	0.0008 grm.	0.0026 grm.			
do. 🔒 ,,	0·0003 ,,	0·0016 ,,			
do. 1/2 ,,	0.0005 ,,	0.0142 ,,			
NaCl: saturated	0.0005 ,,	0.0014 ,,			
do. 🔒 ,,	0·000 <b>4</b> ,,	0.0019 ,,			
do. 1/2 ,,	0.0008 ,,	0.0426 ,,			

The figures show that there is little difference between the precipitating action of sodium nitrate or chloride, whether saturated or half-saturated, whereas the presence of potassium ion increases the solubility of the precipitate, especially in the half-saturated chloride solution.

Composition .- We ascertained that the precipitates are not Marignac's 3:1:3 potassium sodium niobate (loc. cit.), and its possible tantalum analogue. As will presently be shewn, the ratio of alkali to earth acid proved to be 4:3 for the tantalate, and 7:6 for the niobate, precipitate. A determination of the alkalis in the precipitate produced by sodium chloride in a solution of potassium tantalate and hydroxide gave a molecular K<sub>2</sub>O:Na<sub>2</sub>O ratio of 1:13.977; the same ratio determined in a niobate precipitate worked out at 1:9.720. Hence the potassium content of either precipitate is not only very subordinate, but does not bear a simple molecular relation to the earth acid. As the analytical importance of this small potassium (and, parenthetically, the water of crystallisation) content of the precipitates seems remote, we have not studied the question more exhaustively: till this is done, we feel inclined to regard them as sodium tantalate and niobate containing a residual, variable amount of potash, precipitation by the solid sodium chloride taking place before the complete exchange of potash for soda. If this view is correct, the composition may be formulated by 4(Na,K)<sub>2</sub>O.3Ta<sub>2</sub>O<sub>5</sub> and  $7(Na,K)_2O.6Nb_2O_5$ . The retention of potassium may also account for the greater solubility of the precipitates in a solution of higher potassium ion concentration; in this connection, we think that the mode of formation and composition of Marignac's 3:1:3 salt require re-investigation. As far as their analytical application is concerned, the precipitates will be referred to simply as sodium salts.

4:3 SODIUM TANTALATE.—We obtain this salt in the following manner:— Tantalic oxide (0.1 to 0.5 grm.) is fused with 3 grms. of potassium carbonate in a gold or platinum crucible over a blast burner until the fusion is quite clear; it is then allowed to solidify against the sides of the crucible. A fragment of potassium hydroxide (about 0.5 grm.) is added to the cold mass; the crucible is half filled with hot water and left on a hot plate until the contents form a clear or almost clear solution. This is transferred to a 250 c.c. beaker with a little hot water, and precipitated hot by the gradual addition of an excess of solid sodium chloride (about 17 grms. for 50 c.c. of solution). The sides of the beaker are rinsed down, if necessary, with half-saturated sodium chloride solution. After standing overnight, the pulverulent precipitate is collected on a 7 or 9 cm. filter, and washed by decantation with half-saturated sodium chloride solution till the solid sodium chloride is dissolved; it is then transferred to the filter and thoroughly washed with the same solution.

If the potassium hydroxide treatment just described is omitted, the solution of the fused mass in water may not be very clear, and will not react at once with the sodium chloride; the sodium tantalate is deposited gradually in fairly large, transparent crystals, and the precipitation is not so complete. This must be due to hydrolysis, during solution of the fused mass, with formation of a more complex salt than 4:3 potassium tantalate, such salt being gradually converted into the 4:3 sodium salt.

The precipitate is not suitable for direct weighing; it is therefore converted into tantalic acid by a simple process. Filter and precipitate are returned to the beaker and stirred up with 50 c.c. of hot distilled water until the paper is thoroughly disintegrated. One drop of methyl orange indicator and dilute hydrochloric acid are added, the latter cautiously until the solution is faintly acid. After some hours' digestion on the water bath, the precipitate is collected, washed with 2 per cent. ammonium nitrate solution, ignited strongly, and weighed as  $Ta_2O_5$ . It weighs a few mgrms. less than the  $Ta_2O_5$  taken, on account of the slight solubility of sodium tantalate.

We made the observation that the hydrochloric acid required for the decomposition of the sodium tantalate is a sufficiently accurate measure of the alkali in the precipitate to enable us to calculate the ratio of base to acid from the mean of a number of titrations. The operation is one of great simplicity, though the results are not extremely accurate, for reasons to be discussed later. Decinormal hydrochloric acid is run into the cloudy suspension, which is kept stirred, until a permanent colour change occurs. The reading must be taken at the neutral point; for the subsequent gravimetric determination of the tantalic acid, an excess of 0.5 c.c. of 0.1 N acid is added. It is the weight of the earth acid precipitated in the titration that was used for the calculation, not the weight taken.

Eight consecutive determinations of quantities unknown to the operator gave the following results:

				Ratio
No.	Ta <sub>2</sub> O <sub>5</sub> taken.	0.1 N HCl.	$Ta_2O_5$ recovered.	grm. Ta <sub>2</sub> O <sub>5</sub> /c.c.
	Grm.	c.c.	Grm.	
1	0.1558	$9 \cdot 15$	0.1530	0.01673
2	0.2034	$12 \cdot 20$	0.1996	0.01636
3	0.1540	9.15	0.1516	0.01657
4	0.1621	9.63	0.1200	0.01650
<b>5</b>	0.2050	12.38	0.2017	0.01629
6	0.3028	17.93	0.2987	0.01666
7	0.2024	11.83	0.1996	0.01687
8	0.3053	17.99	0.3003	0.01669
			Mean factor	·: 0.01658

Stoichiometric factor for  $4Na_2O.3Ta_2O_5$ : 0.01661

7:6 SODIUM NIOBATE.—In order to obtain this salt, we submit niobic oxide to precisely the same treatment as tantalic oxide for the preparation of the 4:3 sodium tantalate. From solutions of potassium niobate, the sodium salt is always precipitated as soon as the solid sodium chloride is added, whether the fused mass be dissolved in water, or in potassium hydroxide solution as prescribed for sodium tantalate; however, we always added potassium hydroxide when dissolving the fused mass, so as to secure uniformity in the procedure to be applied to the mixed oxides.

As in the case of tantalum, the volume of hydrochloric acid required to react with the sodium niobate is proportional to the quantity of alkali and niobic acid in the precipitate. The titration and subsequent gravimetric determination were carried out exactly as for sodium tantalate, the mean factor being in close agreement with that calculated for the 7:6 salt.

Results of eight consecutive determinations (quantities taken in Nos. 3 to 8 unknown to the operator):

No.	$Nb_2O_5$ taken.	c.c. 0·1 N H	Cl. $Nb_2O_5$ recovered.	Grm. Nb <sub>2</sub> O <sub>5</sub> /c.c.
1	0.3010	26.77	0.2973	0.01111
<b>2</b>	0.2482	21.59	0.2450	0.01135
3	0.2666	$23 \cdot 17$	0.2598	0.01121
4	0.3250	$28 \cdot 42$	0.3230	0.01137
<b>5</b>	0.2564	21.71	0.2508	0.01155
6	0.2528	21.47	0.2479	0.01155
7	0.2048	17.75	0.1976	0.01113
8	0.1814	15.06	0.1740	0.01155
			Mean fa	actor: 0.01135
	Stoichiome	tric factor	for 7Na <sub>2</sub> O.6Nb <sub>2</sub> O <sub>5</sub> :	0.01141
	,,	,,	,, 4Na <sub>2</sub> O.3Nb <sub>2</sub> O <sub>5</sub> :	0.00998
	,,	,,	,, $Na_2O.Nb_2O_5$ :	0.01331

B. PRECIPITATION IN DILUTE SOLUTION .- The precipitation of dilute solutions of potassium tantalate and niobate by neutral alkali salts is described in the following translated passages from Rose's Traîté Complet de Chimie Analytique (Paris, 1859, Vol. I, 301, 316, 328), though more recent advances in knowledge provide a better explanation for the observed facts than that given by Rose: "Solutions of neutral alkali salts produce a precipitate of acid tantalate in a solution of neutral sodium tantalate, since these salts deprive the neutral tantalate of some of its alkali, even at ordinary temperature, an action which, with the pure compound, takes place only on evaporation. When solutions of the neutral sulphates, nitrates, and chlorides of potassium and sodium are mixed with those of a neutral alkali tantalate, no immediate precipitate is formed, especially if the solutions are not too concentrated: at the most, the liquid becomes slightly opalescent. After some time a very copious precipitate is produced; frequently the tantalic acid is thus precipitated quantitatively, especially by sodium nitrate. . . . Solutions of alkali salts precipitate a solution of neutral sodium niobate in the same manner."

Our own interpretation of the precipitation of Rose's "acid tantalate" is as follows: the action takes place, as he points out, in dilute, neutral solution. In other words, hydrolysis takes place, with the formation of free alkali and tantalic (niobic) acid as a colloidal suspension. Here the addition of the alkali salt causes flocculation of the colloidal earth acid, and the precipitate adsorbs alkali, hence the old designation "acid tantalate." The precipitate forms gradually, with the increase in hydrolytic dissociation.

Now we have found that the above reaction takes place also in solutions containing alkali in the form of bicarbonate, and that it is fairly rapid at waterbath temperatures. It may be put to practical use in the recovery of the small quantity of earth acid remaining in solution after sodium chloride precipitation, without the necessity of rendering the filtrate acid or introducing a salt of a heavier metal. We treat the filtrate from the sodium chloride precipitate with a drop of phenolphthalein and dilute hydrochloric acid until the red colour is nearly discharged. The liquid is then digested on the waterbath, and the occasional cautious addition of dilute acid continued until the pink colour remains very faint. Flocculation usually sets in after 15 to 20 minutes. After several hours' digestion the precipitate is collected, washed with sodium chloride solution, and returned to the beaker together with the filter. The latter is disintegrated with a little water, and the liquid digested for an hour on the waterbath with a faint excess of dilute acid: this treatment removes the adsorbed alkali. The precipitate is filtered off, washed with ammonium nitrate solution, and ignited to pentoxide. Results:

No.	Oxide taken.	Recovered.	Error.
1	0.0088 grm. Ta <sub>2</sub> O <sub>5</sub>	0.0093	+0.0005
2	0.0034 ,, ,,	0.0041	+0.0002
3	$0.0095$ , $Nb_2O_5$	0.0058	-0.0032
4	0.0036 ,, ,,	$\cdot 0.0022$	-0.0014
<b>5</b>	0.0070 ,, (Ta,Nb) <sub>2</sub>	O <sub>5</sub> 0.0064	-0.0006

The recovery of the tantalic acid shows a positive error, as the acid has a greater tendency to adsorb impurities than niobic acid; the latter is not completely precipitated by itself, whilst the recovery of the mixed oxides is more satisfactory.

ANALYTICAL APPLICATION.—In our opinion, the precipitation of the earth acids as sodium salts will prove a valuable adjunct in analysis. Under the conditions given, sodium chloride is almost a specific reagent for tantalum and niobium; for, whilst antimonic acid also is precipitated when its solution, after potassium carbonate fusion, is treated with sodium chloride, it can be separated from the earth acids in the form of sulphide (*cf.* Section I, ANALYST, 1922, 47, 93).

The sodium chloride precipitates are dense, micro-crystalline powders which settle readily and are easily filtered off and washed. With precipitates of this nature, adsorption is reduced to a minimum. In short, they are the most tractable tantalum and niobium precipitates yet produced. These valuable properties undoubtedly outweigh their one disadvantage, namely, their slight solubility, which necessitates an additional operation for the recovery of the non-precipitated

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fraction of earth acid; we have already indicated one such method of recovery from a bicarbonate solution, and are experimenting further in this direction.

The conversion of the earth acids into their sodium salts will facilitate their purification and permit of their separation from certain elements the sodium salts of which are easily soluble. We are at present investigating the separation of tungsten from the earth acids, which will form the subject of our next communication. The separation of silica also is being studied.

SIMULTANEOUS VOLUMETRIC DETERMINATION OF TANTALUM AND NIOBIUM.— Having applied the acidimetric method to the determination of the alkali in sodium tantalate and niobate, we tested the possibility of using it for the simultaneous determination of tantalum and niobium in a mixed sodium chloride precipitate. The process, if workable, would supply a surprisingly simple solution to such a notoriously difficult problem as the separate determination of the earth acids.

The procedure used was exactly the same as that described in this Section under Sodium Tantalate, but the results obtained were too wide to justify us in recommending the method in that form. It is easy to account for the discrepancies: the differential factor for the indirect titration is a high one (1 grm. Nb<sub>2</sub>O<sub>5</sub> requires 87.66 c.c., 1 grm. Ta<sub>2</sub>O<sub>5</sub> 60.20 c.c. 0.1 N acid; 87.66-60.20 =27.46 c.c.; each 1 c.c. over 60.20 represents 0.0364 grm. Nb<sub>2</sub>O<sub>5</sub>); therefore, the end-point must be ascertained to a single drop. Now methyl orange is comparatively indifferent to decinormal acid, whilst the end-point is made more uncertain by the amorphous earth acid precipitate which, moreover, adsorbs alkali or acid; hence the uncertain interval between alkalinity and acidity extends over about 0.5 c.c. If a stronger acid is used, the factor will be correspondingly higher. Methyl red, cochineal, phenacetoline and phenolphthalein are decidedly inferior to methyl orange: we observed a more or less marked disturbance in the normal behaviour of the indicators, connected with adsorption phenomena by the amphoteric gel. This was especially the case with cochineal, which formed a purple lake. The iodate-iodide mixture for the measurement of excess acidity by thiosulphate in presence of starch proved erratic as repeated after-blueing took place. Though we have no doubt that the reaction proceeds stoichiometrically, the results, in practice, are vitiated by the use of colour indicators. The potentiometric determination of the neutralisation curve should circumvent this obstacle, and arrangements are being made for verifying the correctness of our inference.

SUMMARY.—When tantalic oxide is fused with potassium carbonate and the solution of the mass treated with sodium chloride, 4:3 sodium tantalate is precipitated. Niobic oxide treated in the same manner yields 7:6 sodium niobate. The precipitates are dense, micro-crystalline powders. The filtrates contain a few mgrms. of non-precipitated earth acid, substantial recovery of which is effected by partial neutralisation to the bicarbonate stage and digestion on the water-bath: hydrolytic dissociation in alkaline solution takes place, and the earth acid is precipitated. Sodium tantalate and niobate are decomposed by dilute acid; the quantity of the latter is proportional to that of the alkali in the precipitates.

This reaction was applied to the indirect volumetric determination of the earth acids in the mixed sodium salts, but the results were rather wide as the end-point could not be ascertained with sufficient sharpness by the use of colour indicators. The analytical applications of the precipitates are under investigation.

The Sir John Cass Technical Institute, Aldgate, London, E.C.3.

# A Simple Method of Testing for the Presence of Sulphites in Food-stuffs.

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BY ALBERT E. PARKES, F.I.C.

(Read at the Meeting, November 3, 1926.)

A METHOD suitable for use where only small quantities of materials are available was embodied by the author in a note in the ANALYST, 1921, p. 402.

Extended use of the method has resulted in the improvement of the apparatus and increased sensitiveness of the process. It is simple to operate and can be speedily carried out.

In view of the approach of the new regulations governing the use of preservatives in food-stuffs, it may be of general interest.

Originally the liberated sulphur dioxide gas was made to act on a piece of starch paper placed over the neck of a small conical flask, a drop of weak iodine solution being placed on the centre of the paper. This process, though satisfactory in its way, had several disadvantages which need not now be detailed.

The apparatus, as improved, consists of a conical flask of 50 to 100 c.c. capacity, narrow necked and closed with a rubber cork carrying a small thistle funnel, bent twice, and having a bulb in each limb capable of holding about 2 c.c. The tube of the funnel has an internal diameter of 3 to 4 mm. In this apparatus the issuing

gases are concentrated into a small space without loss and made to act on a small volume of reagent. In consequence, it is possible to detect very small quantities of sulphur dioxide.

The details of the new process are as follows:—Ten grms. of solid material, such as meat or fish paste, jams, dried fruits, etc., are mixed with about 10 c.c. of water and transferred to the conical flask. In the case of liquids, such as beer, cider, white wines, vinegar, cordials, etc., 10 c.c. are taken. Ten c.c. of dilute hydrochloric acid (about 2N strength) and 2 or 3 small pieces of marble, about the



size of peas, are now added, and the flask immediately closed with the rubber cork carrying the thistle funnel. In the funnel are placed 2 to 3 drops of 0.01 N iodine solution and 1 drop of barium chloride solution. This liquid forms a seal through which all gases liberated must pass. When the action of the acid on the marble has moderated the flask is placed over a small flame and heated gently to boiling. Immediately the first drop of condensed liquid passes over into the funnel, if sulphur dioxide be present the colour of the iodine is discharged, and a white opalescence due to barium sulphate is formed.

Hydrochloric acid is now recommended in place of other acids, as it gives a brisker evolution of carbon dioxide gas and is not so liable to contain traces of sulphur dioxide.

Various reagents have been tried in the funnel, such as iodine solution and starch bromine solution, sodium iodate solution, and hydrogen peroxide (Chapman's reagent, ANALYST, 1922, 47, 204). All of these are satisfactory in a way, but for general work the iodine solution and barium chloride solution are preferred. It is important that all reagents be tested to see that they are free from traces of sulphur dioxide.

Some fruit products (jams, wines, etc.) may contain volatile substances which discharge the colour of iodine solutions, but do not give any reaction with barium chloride solution. The immediate formation of any barium sulphate is an indication of the presence of sulphur dioxide.

A larger apparatus on the same principle could be used, if desired, but it is doubtful if it would have any advantages over the usual distillation method, which can always be used where larger amounts of materials are available.

Some materials will be found to froth badly when boiled, and in these cases it will be necessary to watch carefully or to add a reagent such as amyl alcohol to prevent frothing.

The process may be made partly quantitative by continuing the boiling for 1 to 2 minutes, when most of the sulphur dioxide will be driven over into the funnel. If necessary, 0.1 N iodine solution is dropped into the funnel with a small pipette until there is an excess. The reagent and precipitate are now washed out of the funnel into a small beaker and can be matched against a known amount of sulphuric acid and barium chloride in a nephelometer, or, if a permanent record is desired, the barium sulphate can be filtered on to a black filter paper and dried, being afterwards compared with the barium sulphate from a known amount of sulphuric acid also filtered on black paper.

Although this is not claimed to be a quantitative method, results so obtained have been found to agree fairly well with those obtained by distillation.

### DISCUSSION.

The PRESIDENT opened the discussion, and was followed by Messrs. Hope Cox, Arnaud, and Evans.

Mr. G. N. HUNTLY stated that hydrogen sulphide would decolorise the iodine, but would not give rise to the barium sulphate precipitate: hence sulphites were only present if the double effect was obtained. Dr: MONIER WILLIAMS supported the test as being useful for sorting-out purposes. His criticism was that it was a test for all volatile sulphur compounds, and he recommended the use of hydrogen peroxide. If hydrogen peroxide were treated with barium chloride and filtered, a clear liquor would be obtained which would react exclusively to sulphur dioxide at ordinary temperatures, giving an immediate precipitate. Hydrogen sulphide reacted only if the liquid was heated.

Mr. PARKES, in his reply, explained that by the completion of the reaction he meant the action of the acid on the marble, the main object of which was to drive oxygen out of the apparatus. Very little sulphur dioxide came off in the cold. If a little copper sulphate solution were put in the flask with the substance to be examined there was not so much liability of getting volatile sulphur compounds other than sulphur dioxide. Hydrogen peroxide had been tried, but was not particularly advantageous, especially when copper was used. Starch iodate solution could be used in the funnel.

# A New and Simple Method for the Determination of Carbon Dioxide in Carbonates.

By J. R. I. HEPBURN, M.Sc.

THE determination of carbon dioxide in carbonates has usually been made by the gravimetric methods of Schrötter and of Fresenius and Classen, or by the gasvolumetric methods of Pettersson and of Lunge and Marchlewski (see Treadwell,



Analytical Chemistry, 1919, Vol. II.). All these methods necessitate the use of expensive and complicated apparatus. In the author's experience, no simple and accurate method has yet been devised. The method described below is applicable to all carbonates, is simple in manipulation, and is yet capable of yielding results which are within 0.5 per cent. of the theoretical value for the carbon dioxide 'content. Moreover, only small amounts of carbonates are required for the determination.

APPARATUS.—As shown in the diagram, this consists of the filtration flask (capacity 750 c.c.) fitted with the 50 c.c. separating funnel B. A test-tube C is contained in the flask, and the apparatus is completed by the vacuum rubber tubing D, provided with the spring or screw clip E.

METHOD.—The method depends on the liberation of carbon dioxide from the carbonate by means of an acid, absorption of the carbon dioxide in baryta solution, and back titration of the excess of baryta against standard oxalic acid.

From 0.15 to 0.3 grm. of the carbonate (the actual amount depending on the carbon dioxide content) are weighed out into the test-tube, and covered with water

free from carbon dioxide. Fifty c.c. of 0.1 N baryta solution are put into the flask, the test-tube placed in position, and the separating funnel (the stop-cock of which is well lubricated), fitted to the apparatus. Dilute (3 N) hydrochloric acid (free from carbon dioxide) is put into the funnel. The filtration flask is connected with a good water-pump, or air-pump, and evacuated to a pressure of 2 cm. of mercury. The clip E is closed. Such an arrangement is capable of maintaining the vacuum for 24 hours.

The acid is added cautiously, drop by drop, to the carbonate in the test-tube. Violent effervescence occurs, the carbon dioxide evolved filling the vacuous space and being rapidly absorbed by the baryta solution. Under normal conditions the carbon dioxide is completely evolved from the liquid at the ordinary temperature, the evolution and absorption being materially assisted by the presence of the vacuum.

When no further effervescence occurs on addition of acid, the flask is set aside for 12-24 hours to complete the absorption. At the end of this period, the remainder of the baryta is back-titrated against 0.1 N oxalic acid solution, phenolphthalein being used as indicator.\* The oxalic acid solution is made up as a normal solution and diluted to 0.1 N strength with water free from carbon dioxide, when required for use.

RESULTS.—Among the numerous results obtained the following may be quoted:—

Sodium Carbonate.—Pure sodium bicarbonate was heated to 300° C. for 1 hour in a platinum dish, cooled, repowdered, re-heated for 20 minutes, and finally cooled in the desiccator.

Carbon dioxide (theory)=41.52; found: 41.58, 41.76, 41.02 per cent.

Calcium Carbonate.—Iceland spar was finely powdered in an agate mortar, dried at 100° C. for 1 hour, and cooled in the desiccator.

Carbon dioxide (theory)=44.00; found: 43.71, 43.51, 44.37 per cent.

III. Malachite  $(2CuO.CO_2.H_2O)$ .—This was prepared by the method described by the author (J. Chem. Soc., 1925, 127, 1007).

Carbon dioxide (theory)=19.90; found: 19.81, 20.02, 19.66, 20.00 per cent.

In a series of comparative experiments on the use of standard hydrochloric acid for the back titration, instead of oxalic acid, the results obtained were uniformly high, independently of the strength of the acid, as, for example:

Sodium Carbonate.—Strength of acid employed=0.25 N.

Carbon dioxide (theory)=41.52; found: 43.03, 42.83, 43.22 per cent.

Strength of acid employed = 0.025 N; carbon dioxide found: 43.39 per cent.

Calcium Carbonate.--Strength of acid employed=0.25 N.

Carbon dioxide (theory)=44.00; found: 44.61, 44.68 per cent.

\* Note.—The end-point in this titration is taken as the point where no further pink colour is observed. This precaution is necessary, since the original bright pink colour changes suddenly to a faint pink before the titration is completed.

NOTES

It is possible that the hydrochloric acid attacks the barium carbonate before all the free baryta has been neutralised, and this may provide an explanation of these divergencies (*cf.* Treadwell, Vol. II, 1919, p. 564). The end-point, however, with phenol-phthalein as indicator, was quite well defined.

If a shorter period than 12 to 24 hours is allowed for the absorption of carbon dioxide, the last steps are incomplete, and the results obtained are low, as, for example:

Time allowed for absorption = 3 hours.

- Sodium Carbonate. Carbon dioxide (theory)=41.52; found: 39.36, 40.50, 40.00 per cent.
- Calcium Carbonate. Carbon dioxide (theory)=44.00; found: 43.02, 42.98 per cent.

The author is indebted to Mr. F. Mackley for a suggestion which led to the development of this method, and to Dr. T. J. Drakeley for the interest he has shown in the work.

NORTHERN POLYTECHNIC,

(Department of Chemistry and Rubber Technology), LONDON, N.7.

# Notes.

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

## SOURCES OF ALBUMIN IN ANCIENT EGYPT.

I HAVE read with the greatest interest the able paper on "Problems in Connection with Ancient Egyptian Materials," read by my *confrère* of Egypt, Mr. A. Lucas, and published in the September issue of THE ANALYST, with the discussion thereon.

Discussing the paper, Miss M. A. Murray is reported to have said that "the possibility of the use of egg albumin for the fixation of pigments had been suggested, but there were no fowls in ancient Egypt, nor did they arrive in the West until the days of Alexander the Great, who introduced them from India."

There were, however, innumerable birds, and also reptiles, in ancient Egypt, and hence a rich source of supply for egg albumin. As the crocodile was a sacred animal, is it not barely possible that a fictitious value might have been placed on albumin prepared from its eggs?

I should like to think that it was possible for an analytical chemist to distinguish between albumin derived from the eggs of a small species of lizard and that from the egg of a species of ostrich, but have some doubts as to whether this could be done, though, given a large amount of material, the determination of the kind and quantity of amino acids might solve the problem; and on quite

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small quantities a qualitative differentiation could certainly be shown by means of the precipitin reaction, possibly even in the case of very ancient material, if this had been preserved in a very dry condition in the dark at a moderately low temperature.

With respect to the quantities available for analysis of Egyptian antiquities, I imagine the problem to be quite insoluble. But my main point is, that a large amount of egg albumin must have been available in ancient Egypt, independent of the presence therein of the domestic fowl.

### HERBERT S. SHREWSBURY.

Government Analyst, Trinidad and Tobago, B.W.I.

## THE ANALYSIS OF COMMERCIAL LIME.

In my experience sufficient attention is not paid to the rapid absorption of carbon dioxide by the caustic lime in commercial samples of lime; hence inaccurate results are frequently returned. A method of determining the caustic lime which has proved satisfactory in use in my laboratory is to grind 0.4 to 0.5 grm. of the sample with successive portions, up to 400 c.c. in all, of distilled water, pouring the extracts into a flask, and finally adding the residue of the sample. The flask is closed with a stopper and shaken for half-an-hour, its contents then rapidly filtered into a flask containing, say, 10 c.c. of N sulphuric acid, the filter washed once, and the unneutralised acid titrated (phenolphthalein as indicator).

T. P. BLUNT.

THE WYLE COP, SHREWSBURY.

# Notes from the Reports of Public Analysts.

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

### CITY AND COUNTY OF KINGSTON-UPON-HULL.

ANNUAL REPORT OF THE PUBLIC ANALYST FOR 1925.

DURING the year 1925 the number of samples examined was 5737, which included 959 foods and drugs, 33 milks for bacteriological examination, 4529 bacteriological specimens (pathological), and 4 fertilisers and feeding stuffs. Of the food and drug samples, 495 were formal (47 adulterated), 376 informal (7 adulterated), and 88 miscellaneous (1 adulterated). In addition to those returned as adulterated, 91 of the samples were of suspicious character.

MILK.—The increase in the number of adulterated samples was mainly due to the larger number of dirty milks (about 25 per cent. of the total adulterated samples). The following table gives the results of the special examinations for dirt in milk from *suspected sources*, as compared with the two previous years:—

	No of	Milks containing	Percentage	P	arts per 100,	ment, 000.
Year.	samples.	dirt per 100,000.	dirty milks.	Max.	Min.	Average.
1925	60	11	18.3	<b>9</b> .3	0.0	1.7
1924	33	1	$3 \cdot 0$	$2 \cdot 5$	0.4	1.0
1923	44	7	15.9	5.0	0.4	1.45

During the year 13 samples of milk were returned as adulterated on account of their containing excessive amounts of dirt (mainly dung). In these samples the proportion of moist extraneous matter varied from 1.8 to 9.3 parts by vol. per 100,000 parts of milk.

FLOUR.—The 30 samples of flour examined during the year included 16 samples which had either been treated with bleaching agents (2), had been mixed with chemical "strengtheners" or "improvers" (persulphates, 7), or had been subjected to both forms of treatment (7). As the report of the Departmental Committee on the chemical treatment of flour has not yet been published, all these 30 samples of flour were passed as genuine. Recent examinations in the City Laboratories have shown evidence of the use of chlorine gas in the bleaching of flour.

BAKING POWDER.—Of 27 samples examined, 21 were prepared with an acid tartrate basis only, and 6 contained acid calcium phosphate. One phosphate powder (informal) was condemned by reason of its containing 3.6 per cent. of calcium sulphate as an impurity (=42 per cent. in the acid phosphate used in the manufacture of the powder). In my opinion, this impurity should not exceed 5 per cent. in the phosphate, although a more lenient limit (10 per cent.) was suggested in Dr. Hamill's Report to the Local Government Board, 1911.

ARSENICAL APPLES.—Eight samples of American "Jonathan" apples were tested for arsenic. Seven contained from 1/3500th to 1/170th grain of arsenious oxide per lb. of the whole fruit, and one 1/55th grain. Practically the whole of the arsenic was contained in the outer parts (skin) of the fruit.

GLASS CONTAMINATION OF FOODS.—Since the beginning of the investigation in 1924 (cf. ANALYST, 1925, 50, 393) 95 samples have been specially examined, and 29 found to contain glass fragments in notable quantity. The particles ranged in size from 1/4 and 1/6 inch to 1/25th inch in length, and many were of an average area of 1/4000th square inch. The following table gives the results of the 67 samples examined during the year.

	Number examined.	Free from glass.	Containing glass fragments.
Jam and marmalade	 12	7	5
Pickles and sauces	 12	4	8
Lemonade crystals	 6	2	4
Coffee and chicory essences	 6	3	3
Aerated waters	 12	9	3
Meat pastes	 6	5	1
Other foods	 13	13	0
		<u> </u>	
	67	43	<b>24</b>

It is not necessary to use the language of exaggeration in referring to these discoveries, as has unfortunately been done elsewhere by some writers; but it will be agreed that such contaminated food ought to be eliminated. The British glass

### LEGAL NOTES

manufacturers should therefore place on the market, at a reasonable price, glass containers of suitable quality for food-packing purposes, and the use of lowquality glass containers should be prohibited, especially those known as the "burst-off" type. A regulation requiring a maker's identification mark on all glass containers would be useful in tracing the origin of unsuitable bottles and jars.

ARNOLD R. TANKARD.

# Legal Notes.

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

## SALE OF MEDICATED WINE WITHOUT A LICENCE.

## SHARP v. SPARKES.

ON October 20th an appeal from a decision of the Brighton justices was heard in the King's Bench Division, before the Lord Chief Justice, Mr. Justice Salter and Mr. Justice Talbot. The appellant, a druggist, had been convicted of selling an intoxicating liquor, to wit, wine known as "Wincarnis," without being the holder of a justices' licence, and had been fined  $\pounds 1$  and ordered to pay the analyst's fee (cf. ANALYST, 1926, 186). He contended that "Wincarnis with Quinine" was not an intoxicating liquor within the meaning of the Licensing Acts. The respondent contended that the labels on the bottle and wrapper were *prima facie* evidence that the liquid was made with wine, that no evidence was called to rebut it, and that it was intoxicating liquor within Sec. 65 of the Licensing (Consolidation) Act, 1910.

Mr. Roland Oliver, K.C., for the appellant, argued that by Sec. 110 of the 1910 Act, "the expression 'intoxicating liquor' means spirits, wine, beer, porter, cider, perry, and sweets, and any fermented, distilled, or spirituous liquor which cannot, according to any law for the time being in force, be legally sold without an Excise licence." By Sec. 111 (2) "nothing in this Act shall apply to . . . (b) the sale of medicated or methylated spirits or spirit made up in medicine and sold by medical practitioners or chemists and druggists."

If, therefore, said counsel, he could show that what was sold was not "wine," it could only come under the definition of "spirituous liquor," and would be exempt, as medicine. "Wine" was not defined in the Licensing Acts, but no one would desire to drink, for the gratification of his reasonable appetite, a liquid strongly impregnated with anything so unpleasant as quinine. Regard must be had to the two objects of the licensing law; first, the due preservation of public order; and, secondly, the safe-guarding of the revenue. He submitted that the passage in Paterson's "Licensing Acts," in a note to Sec. 111 (2), to the effect that a wine licence was required for the sale of medicated wine, was wholly wrong. No authority was cited for it. He submitted that the question was one of palatability, not of chemical composition, and relied on the finding that the article tasted of quinine. He cited as authority the case of *Attorney-General* v. *Bailey* (17 L.J., Ex. 9).

The Lord Chief Justice enquired whether, if palatability was the test, it would be a defence to selling wine without a licence that the wine was so badly corked that no reasonable person would drink it. To this Mr. Oliver replied that this was an extremely bitter liquid.

Counsel then continued that, in his contention, "Wincarnis with Quinine" was a spirituous liquor according to Sec. 3 of the Spirits Act, 1880, and, being medicated, was exempt.

Mr. Rimmer, for the respondent, said that the question of palatability depended on the individual's palate, and there were recognised beverages of which quinine formed an ingredient. The fact that the Excise authorities did not require druggists to take out licences was purely a departmental matter, not in any way binding on the prosecution. It was immaterial that quinine was mentioned in the British Pharmacopoeia; so was sherry, and he thought that Mr. Oliver would hardly argue that sherry was not wine. The distinction between wine and spirits contemplated by the Act was that "spirits" implied distillation and "wine" fermentation without distillation. If a liquid before an admixture was a fermented liquor in the sense of being wine, it remained after the admixture a "fermented" and not a "spirituous" liquor.

The Lord Chief Justice, in giving judgment, said that the only question before the Court was whether there were materials for the justices to come to the conclusion to which in fact they came-namely, that this liquid was wine. Counsel had urged that the liquid was so unpalatable that it ceased to be wine. But there seemed to be a commercial question involved: the question how to command a large sale of rather inferior port, with the aid of persons who do not ordinarily sell wine, to persons who would not ordinarily buy it if it were called wine. The answer seemed to be: "Put a little medicine into it and sell it in chemists' shops." The question was one of degree. At one end of the scale, if a man put a very small quantity of a pleasant medicine into a very large quantity of very good wine, it would not be argued that it was not wine. At the other end, if a very large quantity of a nasty medicine were put into a small quantity of wine, it might be that no one could argue that it was wine. Between the extremes were many degrees, and the commercial problem was how to put enough medicine into the wine to make it cease to be wine-at least for the purpose of argument-and at the same time not enough to make it unpalatable to the men, women and children who would drink it—and especially the women who thought they suffered from nervous disorders and wanted an excuse for drinking port at 11 o'clock in the morning. There was abundant evidence to justify the finding of the magistrates, and the appeal would be dismissed.

The other Justices concurred.

## JAM: AVAILABILITY OF WARRANTY.

### FORSTER v. STAR TEA CO.

ON October 28th an appeal was heard in the Divisional Court from a decision of the Gloucester justices, who dismissed a summons for the sale of strawberry jam which contained apple pulp, on the ground that the respondents had a sufficient warranty from the firm from whom they had bought the jam.

Mr. Du Parcq, K.C., for the appellant, an inspector under the Food and Drugs Acts, said that it was not enough to produce evidence showing an oral agreement or one made after the purchase of the goods, for the warranty, to be of use, must have been part of the contract at the time of the sale. Upon the invoices received by the respondents from the jam manufacturers there was a guarantee that the goods complied with the conditions of the Food and Drugs Act, and the labels on

### LEGAL NOTES

the jars contained a similar warranty. The justices refused to convict, having come to the conclusion that the respondents had purchased the jam by warranty and had no reason to believe that it was other than that which was demanded. Counsel submitted, however, that the respondents had not discharged the onus upon them, because they did not show the written contract of purchase and warranty.

Mr. Cecil Whiteley, K.C., for the respondents, said that in this case it was found that there was in existence a contract for a supply yearly of the jam at specified intervals, and the justices certainly found as a fact that all the jam sold under that contract was sold with written warranties which were plainly indicated on the invoices sent with the jam. In addition, each jar had a label on it on which were the words "guaranteed pure." Counsel asked that the case should be remitted to the justices, for decision on the question whether the jam really was sold to the prejudice of the purchaser, seeing that only a very small amount of apple pulp was found in it by the Government Analyst.

The Lord Chief Justice, giving judgment, said that the question was whether the justices could, on the materials before them, hold that the defendants did prove to their satisfaction that they purchased the article in question as the same in nature, substance and quality as that demanded of them by the purchaser, and that they had a written warranty to that effect. The meaning of Section 25 of the Act was not really open to any dispute, for there had been a long series of cases decided upon it, and emphasis had been laid upon the fact that the warranty, if it were to be of avail, must be an integral part of the contract of purchase and sale, and not something that the parties entered into as an after-thought. Afterthoughts were of no avail, for reasons it was not difficult to imagine. The law was that there should be a warranty when the article was delivered that must be part of the contract of sale. In his opinion, there were no materials on which the justices could come to the conclusion that there was a written warranty, and the case must go back to them with the direction that Section 25 of the Act had not been complied with. As the respondents still wished to develop the contention that the analysis revealed no evidence that the jam was not of the nature, substance and quality demanded, that course could be adopted.

The appeal was allowed with costs.

Mr. Justice Avory and Mr. Justice Salter concurred.

# EXCESS WATER IN BUTTER: CASE DISMISSED ON LEGAL GROUNDS.

A SHOPKEEPER was summoned at Derrylin, Co. Fermanagh, for having sold butter, which, according to the certificate of the Public Analyst, contained 17.23 per cent. of moisture.

The solicitor for the defence contended that the water was within the statutory limit, and he read Sec. 4 (2) of the Butter and Margarine Act, 1907, to show that in this case 24 per cent. of water was the limit. There were, he pointed out, numerous regulations, applicable to factories, which allowed only 16 per cent. of water, but in this instance the vendor had not been guilty of an offence. The Act of 1907 had superseded the previous standard upon which the County Analyst had relied.

The Bench dismissed the case.

### FORMALDEHYDE IN SAUSAGES.

ON October 29 a pork butcher was summoned at Salford for selling sausage not of the nature, substance and quality demanded. It was stated for the prosecution that the sausage, when analysed, was found to contain 0.002 per cent. of formal-dehyde. The defendant, who pleaded guilty, said that he had used a preservative during the summer months, and was not aware that it contained anything harmful or that its use had been prohibited. A fine of 20s. was imposed.

## ADULTERATED FOOD IN RESTAURANTS.

WHAT were described as the first cases of the kind were heard at Wellington, New Zealand, on July 16, when three restaurant keepers were charged with selling milk not complying with the New Zealand Sale of Food and Drugs Act. All pleaded guilty.

The inspector stated that he took samples from the tables, and the analyses showed in one case 32 per cent. and in the second 30 per cent. of added water, and added sugar in the third. The prosecutions were brought under the Amendment of 1924.\*

Counsel for the defence said that the practice was general, and that the law would be served by the payment of costs. Restaurants obtained milk from farms and usually added water to it.

Each defendant was convicted and ordered to pay costs.

\*Note.—The Amendment of 1924 reads:—"For the purposes of the principal Act any article of food being part of or supplied with any meal or food for which payment is made or required to be made, and which is supplied for consumption in any shop, hotel, restaurant, or eating house, or at any stall or other place, shall be deemed to have been sold or offered or exposed for sale."

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# New Zealand.

## FIFTY-EIGHTH ANNUAL REPORT OF THE DOMINION ANALYST.

IN his Laboratory Report for the year 1924 (published 1926), the Dominion Analyst, Dr. J. S. Maclaurin, states that, during the year, 4548 samples were examined at Wellington. Of these, 2545 were Public Health samples, 259 for the Customs, 836 for the Mines Department, 223 for Post and Telegraph, 56 for the Police, and the remainder for other Government Departments. At the new Auckland Branch 1061 samples were examined, of which 1024 were for the Health Department.

POLICE.—In two exhibits in cases of suspected poisoning strychnine was found, and in another case caustic soda. One sample, sold as a non-alcoholic beverage, was found to contain 14 per cent. of proof spirit.

A horse-hide was examined at the Auckland Branch for evidence of dyeing in a case of alleged "ringing in." The hair was of a fine black colour, but showed light bay underneath. Some portions were found to contain copper to the extent of 0.5 per cent. In places two layers of hair could be separated, one light-coloured and the other dark. The light-coloured hair contained practically no copper. It was concluded that the hair had been treated with a dye containing copper.

### NEW ZEALAND: ANNUAL REPORT OF THE DOMINION CHEMIST

MINES.—Full details are given of the analyses of coals and carbonaceous minerals, limestones, marls and clays, rock minerals and ores. Attention is drawn to the fact that any person finding a mineral specimen may have it analysed free of charge provided that the exact locality is given, thus enabling a correct mineral record to be kept.

HEALTH DEPARTMENT.—The tabulated list shows that a great variety of goods was forwarded by inspectors under the Sale of Food and Drugs Act.

The examination of beer for the presence of arsenic, mentioned in the previous year's report, was continued, but only one sample contained traces of the metal, and the amount was considerably less than the limit imposed by the regulations.

Butter was again frequently examined, and in three samples only was there a large excess of water. In three others the amount found was slightly over the standard of 16 per cent.

Flour was obtained from the principal mills in the Dominion and tested for artificial bleaching agents, but in no case were any detected. Some attention was also given to so-called flour-improvers.

Examination of samples of hop-beer and ginger-beer from all parts of New Zealand showed that it was a common practice, amounting almost to a trade custom, to add saccharin to such drinks without declaration on the label. The practice has received a salutary check.

A considerable amount of work was done on genuine and artificial extracts of vanilla. The analytical differences were quite marked, and manufacturers of the genuine extract need not fear unfair competition from artificial preparations (cf. ANALYST, 1925, 50, 548).

The use of artificial colours in foodstuffs appears to be under better control than formerly, and labelling regulations are complied with to a much greater extent.

MILK.—The number of samples taken under the Sale of Food and Drugs Act was 2230. Of these 1740 came from Wellington City and suburbs, and almost all the remainder from the chief towns of Taranaki, Hawke's Bay, Wellington, and Nelson.

Wellington City.—The City Inspector was responsible for over 80 per cent. of the samples taken in and around Wellington. The Laboratory results show the supply to be almost free from adulteration in the ordinary sense. Of the 1740 samples analysed, only eight were seriously deficient in fat, and eight watered. In these respects the high standard of the previous year was maintained. The conditions as regards dirt, and staleness arising from insufficient cooling after milking, or prolonged keeping prior to sale, were again not so satisfactory. There were six prosecutions for such defects, and a large proportion of the thirty-eight warnings issued were due also to one or other of these causes.

To facilitate testing of milk for staleness (cf. ANALYST, 1926, 458) the inspector has now been provided with an ice-chest in which to place the samples immediately after collection.

The use of preservatives in milk, which was very prevalent some years ago, is seldom practised now. Only one sample during the whole year contained preservative, in this case boric acid.

The milk samples taken in the country districts also indicated that, while deliberate adulteration is not general, more cleanly conditions are desirable in many supplies.

# Medical Research Council.

## FURTHER STUDIES OF THE SALMONELLA GROUP.\*

THIS is the outcome of the detailed re-investigation of the group of Salmonella strains usually spoken of, when of animal origin as *B. suipestifer*, and when of human origin as *B. paratyphosus* C, to which reference was made in the previous report (ANALYST, 1925, 50, 239).

After a brief revision of the recent literature, details are given of the work from which the following conclusions have been drawn:--(1) The groups or grades of B. suipestifer described by Andrewes and Neave have been confirmed. (2) The "Arkansas" type of Schütze has been identified with the Group I Suipestifer of Andrewes and Neave. (3) The "G" type of Schütze has been identified with the Group II Suipestifer of Andrewes and Neave. Further, it has been found that whilst the Group I strains normally show both the "specific" and "non-specific" races described by Andrewes, the Group II forms represent a totally and permanently "non-specific" condition. The ready absorption of the Group II sera by Group I forms depends upon the presence of the non-specific variant of those The view of Andrewes and Neave, that the Hirschfeld Paratyphosus C forms. strains are serologically identical with B. Suipestifer strains, has not been confirmed, and the investigation has shown that there is no homogeneity in the strains from man which are termed Paratyphosus C.

With the exception of the Glässer strains (2) and the Witts' Blood and Urine strains (derived from the same patient and probably Eastern in origin), all the strains of Suipestifer and Paratyphosus C collected from Britain and Western Europe (27 in all) yielded only the non-specific phase. Two strains from Malta, one American Suipestifer, and one American Paratyphoid C culture were also non-specific. All the remaining 16 strains from America, and the 8 Paratyphoid C strains from India, Africa, Baghdad, and the Balkans, readily yielded the specific race, and in all cases, except one, also the non-specific form. The exceptional purely specific culture was the rough strain "Arkansas." (Full details of the work are given.)

NEW SALMONELLA TYPE.—Typical colonies resembling those of the Salmonella group were isolated from the faeces of two patients suffering from food poisoning in the Reading district. Further cultures showed that the organism was a typical Salmonella, having sharply defined specific and non-specific phases, rapidly fermenting dulcite and inosite, and therefore biochemically identical with the following types—Derby, Paratyphosus B, Aertrycke and Reading. Salmonella types are usually named after the place of origin, but in this instance the Reading type of Schütze interferes with this practice, and the name "L" type is suggested.

SEROLOGICAL VARIATION IN THE SALMONELLA GROUP.—The so-called "rough variation," due to Arkwright, is possibly to be regarded as pathological. Weil and Felix noted that variations in the growth characteristics of Proteus X are associated with striking serological differences. One form, termed the "O" (*Ohne Hauch*) form, forms discrete circular colonies and gives with its own antiserum only finely granular agglutination in compact clumps. The other variant, the "H" (*Hauch*) form, produces a spreading filmy growth, and reacts to its antiserum in large loose flocculi. In 1920 Weil and Felix demonstrated an essentially

\* Special Report Series, No. 103. By P. Bruce White, B.Sc. Pp. 160. H.M. Stationery Office, 1926. Price 5s. net.

similar condition for the "Typhoid-Paratyphoid Group," and this has since been confirmed by several observers. It was also found that there are two groups of receptors or antigens, stable and labile, each matched in the corresponding antiserum by its own agglutinative anti-bodies. In the typical Salmonella the growth forms do not show the distinctive characteristics of the Proteus, except that there is a tendency for the form containing the stable antigen to produce a whiter and more opaque culture than the other. The terms "O" and "H," as applied to the Salmonella Group, indicate only a serological differentiation.

The author's experiments confirm the statement of Weil and Felix that treatment of bacilli with absolute alcohol destroys the heat-labile complex, leaving the heat-stable complex unchanged. No evidence of the existence of forms showing only the "H" labile component has been obtained. The diphasic phenomenon depends solely upon the labile "H" components, the "O" stable factors being the same for both phases, and in many cases set a limit to the degree of specificity of the "specific phase."

ROUGH VARIATION IN THE SALMONELLA GROUP.—In the former Report it was maintained that the sole *positive* agglutinative feature which differentiates the rough variant from the smooth parent organism is the appearance of new antigen or antigens belonging to the "O," heat-stable granulating series. The study of this question has now been extended to other types than the Aertrycke type, with the aid of many strains and sera. (Full particulars of the technique are given.)

Great variation in the amount and quality of the normal heat-stable antigen present in rough variants of the same type, or even the same strain, has been observed. Some exhaust the smooth serum of "O" agglutinins quite readily, whilst others do so with difficulty, or partially, or not at all.

ANTIGENIC STRUCTURE.—Agglutination experiments with different sera showed that eight typical Salmonella types could be classified into four "O" agglutination groups, *viz.*:

Group I. Composed of B. enteritidis.

- (Shows weak or moderate serological relationship with Group II; none or practically none with Groups III and IV.)
- Group II. Comprises: Aertrycke, Paratyphosus B, Stanley, Reading and Derby.
  - (Shows weak or moderate agglutinative relationship with Groups I and III; practically none with Group IV.)

Group III. Composed of the Newport type.

- (Shows weak interaction with Group II; considerable interaction with Group IV, but none with Group I.)
- Group IV. Composed of B. suipestifer.
  - (Shows definite serological relationship with Group III, but none, or practically none, with Groups I and II.)

The general serological relationships of B. *abortus equi* and the new "L" type, and the inter-relationships of B. *enteritidis*, B. *typhosus* and the fowl typhoid bacilli, are considered individually.

The phylogeny and classification of the group are then discussed at length, and a new scheme of classification is suggested.

The Report concludes with an alphabetical bibliography containing 54 references.

# Report of the Committee on Nomenclature in Inorganic Chemistry.

## WASHINGTON MEETING.\*

THE Committee, at their meeting in Washington, on September, 1926, considered the Report of the October 17, 1925, meeting of the Comité de Travail de Réforme de la Nomenclature de Chimie Minerale (ANALYST, 1926, **51**, 194), and unanimously agreed to its recommendations, except that:—

(1) In addition to valency names "formula" names, such as diantimony trisulphide (or 2-antimony 3 sulphide), are suggested as worthy of consideration, especially for indexing purposes.

(2) The prefix di- is preferred to bi- in such names as lead disulphate, owing to a possible misunderstanding with so-called acid salts.

(3) Formulae based on degree of polarity for non-metallic elements are approved, and the alphabetical order of symbols is suggested for the formulae of compounds formed between metals.

(4) The suggested use of "hydro" for so-called acid salts is recommended for reference to the inorganic nomenclature committees of the English-speaking countries.

(5) In compounds in which oxygen is replaced by sulphur the prefix "thio," not "thiol," should be used where all the oxygen has been replaced.

D. G. H.

### \* Chem. Weekblad, 1926, 486-487.

## ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

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# Food and Drugs Analysis.

Detection of Incipient Putrefaction of Meat. R. Herzner and O. Mann. (Z. Unters. Lebensm., 1926, 52, 215-242.)—Experiments with the flesh of the horse, ox, and pig show that electrometric measurement of the hydrogen ion concentration, either of the meat itself or of its aqueous extract, serves as a means of detecting incipient decomposition of ripened flesh of warm blooded animals. Measured with the quinhydrone electrode the value of  $P_{\rm H}$  is 6.0 to 6.2 for the sound meat, and above 6.2 for meat which has undergone deterioration. This method is inapplicable to meat which has been treated with chemical preservatives, as these exert an appreciable influence on the hydrogen ion concentration.

T. H. P.

Plantain (Banana) Flour. E. A. Ullán. (Quim. e Ind., 1926, 3, 261–263.)—Banana flour contains starch granules, together with certain cellular residues, which serve for its identification. The starch granules, observed under 150–200

magnifications, vary in size, and are cigar-shaped, elliptical, straight or bent, bean-shaped or pear-shaped. Clearly marked are the zones of hydration and the hilum, which may be near either the narrow or the broad end. Particularly characteristic are the rounded granules of more or less irregular contour, with a sharp point to one side of the hilum. The author has never observed in green Canary bananas the pairs of granules joined at the ends to a sickle-shaped form to which Tschirch and Oesterle attach importance, but of common occurrence are aggregates of a number of small or medium granules pressed within a cellular pellicle. The greatest dimension of the small (large) granules is 6 to  $15\mu$  (15 to  $57\mu$ ). If a microscope preparation is treated with a drop of 10 per cent. ferrous chloride solution, the hydration zones of the starch granules are rendered more distinct and the brown cellular residues turn violet-blue. The cellular residues are best observed in a preparation made by boiling for 15 minutes with 3 c.c. of hydrochloric acid, a paste of 3 grms. of the starch diluted to 300 c.c., and examining the red powder which subsequently settles. This contains : Giant cells, 57 to  $280\mu$  in diameter, which occur in chains in the fibro-vascular bundles; secretory canals, 28 to  $114\mu$ ; fine tracheae, 9 to  $33\mu$ ; libriform vessels and parenchymatous cells. T. H. P.

Occurrence of Gentiobiose in the Products of the Commercial Hydrolysis of Corn Starch. H. Berlin. (J. Amer. Chem. Soc., 1926, 48, 2627–2630.)— The mother liquor (hydrol) obtained in the commercial manufacture of crystalline *d*-glucose by the hydrolysis of maize starch consists of approximately 70 per cent. (on dry basis) of fermentable and 30 per cent. unfermentable material. The latter closely resembles iso-maltose in physical and chemical properties, but the name is not regarded as applicable to it, as only comparatively little gentiobiose is present, and the product is apparently a mixture of carbohydrates. The gentiobiose was identified from the acetylated unfermentable part of the hydrol by isolation of  $\beta$ -octa-acetyl gentiobiose with a m.pt. 195–196° C. and  $[\alpha]_D^{26}$  (in chloroform) of -5.61. The yield corresponded to about 5.7 per cent of free gentiobiose. D. G. H.

Deterioration of Fats and Oils and its Detection. J. Pritzker and R. Jungkunz. (Z. Unters. Lebensm., 1926, 52, 195–215.)—Owing to the ready alterability of sodium sulphite, the reagent suggested by von Fellenberg (ANALYST, 1925, 50, 245) may be replaced with advantage by one prepared by dissolving 5 grms. of magenta in 800 grms. of warm water, adding a concentrated solution of  $5\cdot4$  grms. of potassium metabisulphite and 100 c.c. of N hydrochloric acid, and making up to 1 litre. The red colour is removed by filtering the liquid with animal charcoal. According to Kreis (Schweizerisches Lebensmittelbuch, 47) deterioration of a fat or oil may be detected by shaking 1 c.c. of the oil or melted fat with 1 c.c. of hydrochloric acid (d, 1·19) for 1 minute, then adding 1 c.c. of a cold, saturated solution of resorcinol in benzene, and again shaking vigorously; bleached oils or fats, or those which have become tallowy, colour the acid intense reddish-violet.

If the resorcinol solution is replaced by a 0.1 per cent. ethereal solution of phloroglucinol, a bright red coloration is obtained. In 41 of 54 instances von Fellenberg's and Kreis's tests gave concordant results; in no way is the former preferable to the latter test. No relationship is discernible between these tests and the colour reactions given by the oils.

With reference to the occasional failure of Kreis's test with obviously deteriorated fats and oils, it is shown that the reaction is not interfered with by either free fatty acids or peroxides of the fats. Of a number of fatty acids and derivatives of these which were examined, the only one which gave the above tests was ethyl oleate, and this only after exposure to light for a time subsequent to preparation; after such exposure the ester gives an intense peroxide reaction. This ester or methyl oleate is recommended for use in the study of the mechanism of the deterioration of fats and oils. T. H. P.

Reagent for Oil Extracted by Carbon Disulphide, and Identification of Olive Oil extracted by Solvents. S. Fachini. (Giorn. Chim. Ind. Appl., 1926, 8, 428.)—For the recognition of olive oil which has been extracted by means of carbon disulphide, saponification of the oil by excess of alcoholic sodium or potassium hydroxide, followed by incineration and examination of the ash for sulphur, is recommended (cf. Canzoneri, *ibid.*, 1926, 8, 217). The value of the reaction previously given (ANALYST, 1926, 416) for identifying olive oil extracted from the pressed residue is upheld. T. H. P.

Sensitive Reaction for Carbon Disulphide. P. Saccardi. (Giorn. Chim. Ind. Appl., 1926, 8, 315-316.)—When boiled with a liquid containing as little as 1 part of carbon disulphide in 1,160,000, a reagent composed of a benzene solution of lead plaster mixed with alcoholic potassium hydroxide solution gives a black coloration. This reaction serves for the detection in olive oil of oil which has been extracted by means of carbon disulphide. T. H. P.

Solubility of Corrosive Sublimate in Ether. F. Richard. (J. Pharm. Chim., 1926, 118, 306-307.)—The French Codex requires that corrosive sublimate be soluble in 4·1 parts of ether, and this figure appears to have been repeated by various authors. An investigation showed that 1 grm. of corrosive sublimate requires at least  $14\cdot4$  grms. of ether of  $d \ 0.720$ , whether solution is effected at ordinary temperature, or by prolonged boiling and subsequent cooling.

D. G. H.

## Biochemical, Bacteriological, etc.

Gas Analysis Apparatus Modified for Determination of Methane in Metabolism Experiments. T. M. Carpenter and E. L. Fox. (J. Biol. Chem., 1926, 70, 115–121.)—The metabolism of certain types of animals, particularly ruminants, is accompanied by the alimentary production of methane. The

amount of this material given off into the air of a respiration chamber may be determined either by the passage of a given volume of air, which has been freed from carbon dioxide and water, through a combustion furnace, or gasometrically, by means of a gas analysis apparatus. A gas analysis apparatus designed by Carpenter (J. Metabol. Research, 1923, 4, 1) for the determination of carbon dioxide and oxygen in the outgoing air from the chamber has now been modified in such a way that the added determination of methane may be carried out with approximately the same degree of accuracy as is obtained in the other analysis of chamber air and outdoor air. A sketch of the modified apparatus is given, together with a detailed description of it and of the procedure for an analysis. The apparatus is much as originally described. It is constructed on the Haldane principle, with the burette so modified that carbon dioxide may be determined up to 1.7 per cent. and oxygen values may be obtained corresponding to a deficit of 2 per cent. Before use it must be tested to see that there is no leak in the apparatus. The analysis of gas containing methane is similar to that of other combustible gases. The gas is freed from carbon dioxide, passed into the combustion pipette where the combustible gas is slowly burned and again brought back for the absorption of carbon dioxide resulting from the combustion. This combustion of gas and absorption of carbon dioxide is repeated until the readings of the burette show that there is no more gas to be burned and the absorption of the carbon dioxide produced comes to an end. A table shows the results of determinations made on different mixtures of methane and outdoor air. One diffculty encountered in the work was that of making sure that the Douglas bag used was quite empty before the introduction of the gas. P. H. P.

Recrystallisation of Urease. J. B. Sumner. (J. Biol. Chem., 1926, 70, 97-98.)—In a preceding paper by Sumner (ANALYST, 1926, 587) it was stated that attempts to recrystallise urease were not entirely successful. The conditions which might have caused the crystallisation from the original extracts have now been considered. The  $P_{\mu}$  of the original extracts was determined by the indicator method and found to be  $6 \cdot 1$ . A phosphate solution of  $P_{\mu} 6 \cdot 1$  was prepared by the addition of 14.3 c.c. of 0.1 M sodium hydroxide solution to 50 c.c. of 0.2 M potassium dihydrogen phosphate solution and then enough acetone to make the acetone concentration 32 per cent. The crystals from 100 grms. of jack bean meal were dissolved in 3.5 c.c. of water, centrifuged from insoluble matter, mixed with 1.5 c.c. of acetone and cooled in the ice chest. It was found that the addition of the buffer solution, a few drops at a time and only 25 drops in all, to the enzyme solution caused a good crop of octahedral crystals to be obtained. A buffer solution of  $P_{H}$  6.3 could also be used. Analyses on the basis of nitrogen content have shown that recrystallised urease has the same activity as once crystallised urease. Recrystallisation twice showed a total loss of about 80 per cent. of the enzyme. One drop of concentrated urease solution instead of 1 c.c. of dilute solution avoids inactivation when testing solutions of crystallised urease. " Arlco " jack bean meal has a urease content of 0.15 per cent., *i.e.*, a urease activity of 150 units per grm., whereas 2 other samples showed activities of 108 and 140 units per grm. respectively. The "Arlco" meal may be richer in urease because it has been defatted, dried and perhaps freed from the inactive hulls. For the preparation of urease crystals from jack bean meal it is best to use the diluted acetone at about  $28^{\circ}$  C. P. H. P.

Serological Differentiation of Yeasts. F. Ottensooser. (Chem. Ztg., 1926, 50, 790.)-The crossed precipitin reaction may be applied to the differentiation of yeast species. To prepare the immunising antigen the cultures are completely freed from beer-wort protein, dried and extracted for 4 hours at 35° C. with physiological salt solution. The test antigens are brought to equal protein contents according to Uhlenhuth's method, and the nitrogen determined by Kjeldahl's method. In agreement with earlier results, Aspergillus oryzae almost always reacted negatively. Endomyces capsularis is found to occupy an intermediate position to E. Magnusii and E. fibuliger. The results obtained with the Saccharomycetes confirm Klöcker's arrangement, according to which the Saccharomyces and the Zygosaccharomyces are united in one genus; the two subgenera are, however, readily distinguished by this method. Further, the two families, Saccharomycetes and Schizosaccharomycetes are united by Endomyces capsularis, which reacts with Zygosaccharomyces more strongly than with E. fibuliger. T. H. P.

Use of Commercial Dextrin in Artificial Diets. L. Randoin and R. Lecocq. (J. Pharm. Chim., 1926, 118, 289–294.)—Dextrin is sold either as a white or as a pale yellow powder, and in comparative tests on pigeons and rats fed on Randoin and Schimmel's diet the white product was found to cause loss of weight in pigeons (remedied on changing to the yellow product), and still greater loss of weight and even death in the case of rats. Probably the white dextrin is rather nearer in its composition to starch, whilst the yellow dextrin represents a more advanced stage of dextrination with an augmentation in the digestibility of the polysaccharide. D. G. H.

Anti-Rachitic Value of Irradiated Cholesterol. II. Separation into an Active and Inactive Fraction. A. F. Hess, M. Weinstock and E. Sherman. (J. Biol. Chem., 1926, 70, 123-127.)—The various reasons and experiences are given which led the authors to believe that irradiated cholesterol, which protects young rats from rickets, is a mixture of active and inactive material. Beumer (Münch. Med. Woch., 1925, 72, 1585) reported that activated cholesterol could be separated into a digitonin-precipitable and non-precipitable substance. The authors (J. Biol. Chem., 1926, 67, 413) attempted, but without success, to obtain an active fraction of irradiated cholesterol by the method of precipitation with an alcoholic solution of digitonin. The technique of fractionation has now been modified in several particulars. Thus the separation is carried out in an atmosphere of nitrogen, the oily menstruum in which the fractions are suspended is mixed immediately with the fractions, and then curative tests, instead of prophylactic tests,

are employed. The animals which received the digitonin-precipitable fraction showed no subsequent healing of the rachitic process, but those which received the non-precipitable fraction showed marked healing within the experimental period of 9 days. This active fraction constituted only about 4 to 5 per cent. of the total irradiated cholesterol. Thus only about 5 per cent. of irradiated cholesterol possesses anti-rachitic properties. There is evidence that a close chemical similarity exists between the active principles of irradiated cholesterol and cod liver oil. Liquid ammonia separates the active fraction from cod liver oil, and an experiment was carried out with liquid ammonia and irradiated cholesterol. An ammonia-soluble fraction of nearly 4 per cent. was obtained, and it was found to be active. In spite of various chemical points of similarity, the female sex or ovarian hormone, from follicular fluid and other tissues, and activated cholesterol are quite different in their essential properties. P. H. P.

Colorimetric Determination of Cholesterol and Lecithin in Blood in Connection with Folin and Wu's System of Blood Analysis. G. M. De Toni. (J. Biol. Chem., 1926, 70, 207-210.)—A method is described by which blood cholesterol and lecithin are determined by analysis of the protein precipitate of Folin and Wu. The protein precipitate is washed, dried and extracted with hot chloroform in a similar manner to that used by Myers and Wardell (J. Biol. Chem., 1918, 36, 147) for total blood. In this extract cholesterol is determined as usual by means of Liebermann's reaction, and lecithin is determined as lipoid phosphorus by Whitehorn's recent method (J. Biol. Chem., 1924–5, 62, 123; ANALYST, 1925, 50, 82). Tests which were made show that de-proteinisation according to Folin and Wu's method in no way prevents the determination of cholesterol and lecithin in the protein precipitate which, instead of being thrown away as at present, can be used for this purpose. P. H. P.

**Colorimetric Method for the Determination of Acetone Bodies in Blood** and Urine. J. A. Behre and S. R. Benedict. (J. Biol. Chem., 1926, 70, 487-494.)—A colorimetric method is described for the determination of such small amounts of acetone as are found in normal urine and blood, based on the reaction between acetone and salicylic aldehyde in alkaline solution, with the formation of the coloured product, dihydroxydibenzene acetone. It provides for the determination of 0.005 mgrm. of acetone in 5 c.c. of distillate, so that with the dilution necessary for complete distillation a determination can be made with 2 c.c. of blood, if the blood contains not less than 0.001 per cent. of acetone. Greater accuracy is attained if larger amounts are used.  $\beta$ -hydroxybutyric acid is oxidised during a second distillation with potassium dichromate and sulphuric acid, as in the method of Shaffer (J. Biol. Chem., 1908-9, 5, 211) modified by Hubbard (J. Biol. Chem., 1921, 49, 351). The substances in urine which increase the colour given by this second distillation in the final determination, are removed with copper sulphate and calcium hydroxide previous to the distillation, as suggested by Van Slyke (J. Biol. Chem., 1917, 32, 455). This is not necessary in blood filtrates. In the procedure of Csonka (J. Biol. Chem., 1916, 27, 209) the salicylaldehyde is added in alcoholic solution in the presence of sodium hydroxide, and the tubes are heated for 20 minutes in water kept at  $45-50^{\circ}$  C. In the present method the salicylaldehyde is added undissolved, and the tubes are heated in boiling water for 3 to 5 minutes. Acetone cannot be determined directly in urine or in blood filtrate with accuracy by this method. Full experimental details are given and tables of the results. A formula is given for all the calculations.

P. H. P.

Identification of Acetaldehyde in Normal Blood and its Quantitative Study in Blood of Normal and Diabetic Dogs. A. H. Bee and I. L. Chaikoff. (1. Biol. Chem., 1926, 70, 151-165.)—This work was carried out (1) to verify the statement that acetaldehyde is actually present in blood and (2) to examine the methods on which Fabre (Bull. Soc. Chim. biol., 1926, 7, 429) and Supniewski (J. Biol. Chem., 1926, 70, 13) base their claim that the aldehyde content of blood rises to several times its initial value after the removal of the pancreas. The reagent used for the isolation of acetaldehyde was 5, 5-dimethylcyclo-hexandion-1,3(dimethyl-hydroresorcinol), also known as dimedon. The product of the condensation of 2 molecules of dimedon with 1 molecule of acetaldehyde is aldomedon, which is approximately 7 times the weight of the combining acetaldehyde, and thus permits the manipulation of small quantities. The colours of the fused reagent and derivatives are different. Dimedon, aldomedon and the anhydride of aldomedon are white crystals; after melting dimedon turns red and the anhydride turns brown, but aldomedon remains white, on solidification. The procedure throughout is described in detail. The presence of acetaldehyde in ox blood was verified and demonstrated qualitatively and the aldehyde content of dog blood quantitatively. Normal dog's blood contains 2 to 6 mgrms. of acetaldehyde per litre, which amount is 5 to 10 times as great as the total acetaldehyde of Fabre and Supniewski. Three large dogs were de-pancreatised, and acetaldehyde analyses of their blood were made, precisely as for normal dogs. A table shows the results. In the diabetic blood obtained from these dogs following pancreatectomy there was no significant increase of acetaldehyde from the level of that found for normal dogs. P. H. P.

Optical Rotation of Glucose and Insulin Solutions in Contact with Muscle Tissue in Vitro. H. H. Beard and V. Jersey. (J. Biol. Chem., 1926, 70, 167–171.)—Lundsgaard and Holbøll (J. Biol. Chem., 1924–5, 62, 453) have reported the effects upon the specific rotatory power of glucose-insulin solutions in contact with muscle tissue in vitro. They advance the theory that under the conditions of their experiments there is a fall in the specific rotation of the glucose solution from the usual value,  $+52.5^{\circ}$ , for  $\alpha$ ,  $\beta$ -glucose to a new value which varies from +22 to 40°. This form they call new-glucose. The authors describe experiments which show that the specific rotatory power of glucose-insulin solutions in contact with fresh muscle tissue is only slightly lower than the usual value,  $+52.5^{\circ}$ . These variations are due to experimental error. With the use of larger concentrations of glucose, the reducing and rotatory values and also the specific rotatory powers agree closely. They have been unable to confirm the results of Lundsgaard and Holbøll as to the production of new-glucose *in vitro* from the glucose-insulinmuscle solutions. The muscle tissue used was obtained from rats or rabbits. The results obtained are in close agreement with those of Barbour (*J. Biol. Chem.*, 1926, **67**, 53) and Paul (*J. Biol. Chem.*, 1926, **68**, 425). Barbour used muscle tissue from various animals and Paul studied both the dialysates and ultrafiltrates from the glucose solutions. P. H. P.

Action of the Typhoid Bacillus on Milk. C. Gorini. (Compt. rend., 1926, 183, 681–682.)—The action of B. typhosus on milk takes place in two phases. The first lasts 30 to 40 days during which the casein becomes soluble, and leaves the milk light yellow and alkaline in reaction. In the second stage, which lasts the same period, the milk becomes white, the casein regains its colloidal state and then coagulates, while the reaction changes to acid. These changes are accelerated by the addition of yeast-water or of blood. They are used to distinguish organisms which coagulate milk by virtue of their strong acid-producing powers from those which produce less acid. B. typhosus belongs to the second class (mixed ferments or acidoproteolytics), while types of B. coli and of streptococci of each class are recognised. (Cf. ANALYST, 1926, 530.) J. G.

## Toxicological and Forensic.

A Case of Aniline Poisoning. J. Inkster. (Lancet, 1926, 211, 752–753.)— Details are given of a case in which a boy of aged 12 inadvertently swallowed less than 2 c.c. of a mixture of aniline and water. For three hours nothing abnormal was noticed, and the boy made no complaint. He then suddenly collapsed and became unconscious, the finger tips were blue and the lips almost black. After repeated administration of oxygen and stimulants, there was frequent vomiting, and the patient gradually improved, the dilatation of the pupils decreased, and the colour of the skin had improved. After 3 days no ill effects were felt, except headache, and the blueness had disappeared.

Glaister (*Medical Jurisprudence and Toxicology*, 1915, p. 783) cites an instance of aniline poisoning from the application to the ear of cocaine dissolved in equal parts of aniline and rectified spirits, of babies being poisoned by napkins which had been marked with aniline marking ink, and of the poisoning of children through wearing yellow boots which had been stained with an aniline preparation.

Fatal Poisoning by Sodium Nitrite. L. Musso. (*J. Pharm. Chim.*, 1926, 118, 345–360.)—Four fatal cases resulted from taking purgative lemonade which contained sodium nitrite instead of sodium tartrate. Alkaline nitrites are not included in the French Codex and are not on the dangerous drugs list. Even

if the presence of nitrites were established, it might be difficult to prove that they had been taken, since nitrites are readily formed from nitrates in the presence of organic matter. The observed symptoms of poisoning were, nausea, vomiting, collapse, and death in a few minutes. In one case the body was reported to show a black coloration. Nitrites transform oxyhaemoglobin into methaemoglobin, and have a depressing influence on the nervous system. In the cases under consideration each bottle of purgative lemonade contained 23 to 26 grms. of sodium nitrite. Nitrite was detected by various reactions including formation of an intense blue coloration in the presence of potassium iodide and starch; a green coloration with an acetic acid solution of antipyrin; an orange coloration with the Denigès aniline reagent in acetic acid; and by a red-violet colour with Denigès' resorcinol reagent in sulphuric acid. The quantitative determination was made by means of potassium permanganate, and, after dilution, by means of the reaction of Kalmann-NaNO<sub>2</sub>+2HI = NaI+I+NO+H<sub>2</sub>O. Nitrites were found in the stomachs of the victims in proportions varying from 8.138 to 0.301 grms. of N<sub>2</sub>O<sub>3</sub> per kilo. of viscera, but some, at least, of this quantity might have been due to organic putrefaction. D. G. H.

Detection of Blood in Urine. G. Poirot and A. Lambert. (J. Pharm. Chim., 1926, 118, 337-345.)-In the case of a urine free from albumin, 5 c.c. of ammonium hydroxide, 8 c.c. of acetic acid and 25 c.c. of ethyl acetate are added successively, shaking between each addition, to 100 c.c. of urine. After 10 minutes the lower clear layer is withdrawn, and the emulsion is separated into two clear layers by causing it to be absorbed by a loose plug of absorbent cotton, which is subsequently pressed out. The upper layer is then used for testing. In the presence of albumin in the urine coagulation is brought about by heat, and the blood pigment (largely held up in the coagulate) extracted with ethyl acetate. Five c.c. of ammonium hydroxide and 5 c.c. of acetic acid are added to 100 c.c. of urine, boiled, filtered boiling, and the coagulum washed with 30-50 c.c. of boiling distilled water. The filter is then placed in a wide-mouthed 250 c.c. flask, 5 c.c. of water and 5 c.c. of acetic acid added, and the whole well shaken to bring the paper to a pulp, after which 20 c.c. of ethyl acetate are added, and the contents of the flask run on to absorbent cotton, as before. Blood may then be detected :--(1) By adding 1 or 2 drops of a solution of 10 grms. of guaiacum in 100 c.c. of pyridine, 5 c.c. of a solution of 1-2 drops of 100 volume hydrogen peroxide in 100 c.c. of 95 per cent. alcohol, and 2 c.c. of the solution to be tested, and shaking, when an intense blue colour, stable for at least 1 hour, results in the presence of blood; and (2) by adding in succession 0.5 grm. of pyramidone, 5 c.c. of a solution containing 3 c.c. of pyridine in 100 c.c. of 95 per cent. alcohol, and 1 c.c. of 100 vol. hydrogen peroxide, and, after solution of the pyramidone, 2 c.c. of the solution: A blue-violet colour forms if blood is present, intense but very fugitive for 1-2 drops of blood in 1 litre of urine, and clearly positive for 1 drop in 10 litres. D. G. H.

Physiological Action of Hashish. Wiechowski. (Chem. Ztg., 1926, 50, 790-791.)—The action of hashish may be determined with sufficient accuracy by ascertaining the minimum dose which proves fatal when administered to white mice by intraperitoneal injection. Of the preparations examined, the original drug as demanded by the drug manuals is the most active, African hashish being less effective, and that of German culture still less so. The whole of the active material is extractable with petroleum spirit. The action is not merely on the higher functions of the brain, but includes also marked vegetative effects. Of the central nervous actions, the most important is the maintenance of the reflexes and also of the upright position of the body until death occurs. Persistence of consciousness likewise contrasts sharply with the effects of alcoholic narcotics. As regards vegetative functions the breathing is retarded considerably, and the body temperature, blood pressure, and pulse register fall. The effects of hashish are slow in appearing, but prolonged.

# Organic Analysis.

Determination of Selenium in Organic Compounds. W. E. Bradt and R. E. Lyons. (J. Amer. Chem. Soc., 1926, 48, 2642-2646.)-The weighed halogenfree seleno-organic compound is subjected to the Carius treatment until decomposition is complete, the contents of the tube washed with the minimum amount of water into a 100 c.c. graduated cylinder, and the solution made alkaline to litmus by addition of halogen-free sodium or potassium hydroxide solution. Two drops of methyl orange solution are added, the solution acidified with nitric acid, halogen-free zinc oxide added in slight excess, with stirring, until alkaline, and the solution then titrated with 0.1 N silver nitrate solution and a halogen-free soluble chromate as outside indicator. The final volume of solution is observed, the temperature of the solution noted, and a blank determination made under the same conditions. Diseleno-acetone, diphenyl-diselenide and solutions of selenious acid in fuming nitric acid were analysed by this method with satisfactory results. The error for 10 analyses varied between +0.14 and -0.33per cent. of selenium. D. G. H.

Anthraquinone- $\beta$ -Carboxylic Acid Chloride as a Reagent for Alcohols, etc. T. Reichstein. (*Helv. Chim. Acta.*, 1926, 9, 803–806.)—Anthraquinone- $\beta$ carboxylic acid chloride forms characteristic well crystallised derivatives with small quantities of alcohols. It is prepared (D.R.P., 80, 407) by interaction of toluene and phthalic anhydride in the presence of aluminium chloride, the resulting p-toluyl-benzoic acid being oxidised by alkaline potassium permanganate to benzophenone carbonic acid. The action of strong sulphuric acid for 2 hours at 150° C. is to produce anthraquinone- $\beta$ -carboxylic acid, which is crystallised from strong nitric acid, washed, dried, and its acid chloride formed by the action of phosphorus oxychloride. When purified by distillation (b.pt. 210° C. at 0.2 mm.), it crystallises in yellow needles (m.pt. 147° C.). One drop of the dry alcohol is mixed with excess of the reagent in a solution of pure benzene and ether. An excess of pyridine is added, the whole allowed to stand, filtered, the filter washed with dilute hydrochloric acid, and the filtrate mechanically mixed with 2 c.c. of 50 per cent. potassium hydroxide solution. The solvent is removed, after filtration, by distillation, and the bright yellow ester dried in a vacuum and crystallised from benzene or petroleum spirit. The m.pts. of the ether-soluble esters of a number of alcohols are given. That of farnesol (from musk kernel oil) ranges from  $37^{\circ}$  to  $43^{\circ}$  C., which indicates that it is a mixture of cis- and trans-isomers. No residue should be left by the reagent when a blank test is made. J. G.

Saturated Aliphatic Alcohols from Sperm Oil and Spermaceti. E. André and T. François. (Compt. rend., 1926, 183, 663-665.)—The authors have fractionated under low pressure the acetic ethers of the alcohols from the head of the sperm whale, and have obtained, on saponification, small quantities of tetra- and octadecylic alcohols (m.pts. 38° and 61° C., respectively) and a large quantity of hexadecylic alcohol. Tetradecylic alcohol has also been found in spermaceti, but in neither case has dodecylic alcohol been found, though its existence is admitted as possible. Tetradecylic alcohol has hitherto been produced only in the laboratory by the reduction of myristic acid, and the nature of the product obtained from the sperm whale was confirmed by establishing the identity of the phenylurethanes of the natural and synthetic alcohols. Whereas the m.pts. of these alcohols are higher for the higher members of the homologous series, those of the phenylurethanes of tetra- and hexadecylic alcohols (70° and 73° C. respectively) are lower than those of do- and octadecylic alcohols (78° and 79.5° C., respectively). (Cf. ANALYST, 1926, 200.) I. G.

The Coniferyl Reaction of Resins. F. Reinitzer. (Z. anal. Chem., 1926, 69, 114-121.)—The intense red colour produced by the addition of a drop of solution of phloroglucinol and a drop of hydrochloric acid to coniferyl alcohol may be used as a general test for resins. Strong reactions are given by Siam and Sumatrabenzoin, tolu balsam, Peru balsam, storax, asafoetida, galbanum, pine oil, black pine, and larch resins, yellow and red acaroid, dammar, Manila elemi, copavia. balsam, and olive resin; and weak reactions by olibanum, myrrh and mastic. The test is very sensitive if it is carried out in a white porcelain dish or on a slide under the microscope, but often depends on the physical state of the resin. Thus, in the cases of myrrh and dammar the residue from an evaporated alcoholic solution must be used, and in some cases (e.g. galbanum and asafoetida) the test is best carried out on a portion of the freshly broken surface of the resin. The reaction has been variously ascribed to the presence of an allyl or cinnamic acid grouping. The evidence is inconclusive and contradictory, but the active substance may be produced during the reaction and not be present in the original resin. Caffeic acid gives only a doubtful reaction, and ferulic and paracumic acids, gamboge, galipot and dragon's blood give none at all. The reaction is due to the presence of elemicin and eugenol in the cases of Manila-elemi and myrrh respectively, and is suggested as a guide to the constitution of resins. I. G.

Determination of Citric Acid and Malic Acid in Tobacco. H. Rundshagen. (Chem. Ztg., 1926, 50, 764.)-The original method of Schlösing and Kissling, in which the acids are determined in an extract of the tobacco by precipitation with lead acetate or baryta, is long and inaccurate. It is better to liberate the acids by grinding the tobacco with sulphuric acid (10 to 40 per cent. according to the sample). The dried mixture is then further ground to a fine powder mixed with pumice, and completely extracted with ether in a Soxhlet apparatus. The solution is evaporated, the dirty brown residue, which contains resins, fats, and the acids, is thoroughly dried and esterified by the action of strong sulphuric acid in a solution of absolute alcohol. The solution is neutralised, the alcohol removed, and the solution of the esters in ether and water is salted out with the aid of the centrifuge. The esters are finally dried over sodium sulphate, fractionated by distillation at low pressure and identified from their b.pts. and refractive indices. Their hydrazides, and thence the benzylidene derivatives, may be prepared and identified with the help of the true and mixed m.pts. The method is not quantitative for oxalic acid, which should be determined as calcium oxalate. J. G.

Determination of Deliquescent Substances in Sized Cotton Materials. S. M. Neale. (J. Text. Inst., 1926, 17, T511.)—Tests are made for zinc and magnesium chlorides and glycerin. Five grms. of the cloth or yarn are extracted by boiling with dilute nitric acid and decanting, followed by extraction with water, and the two extracts are mixed (Extract A). In the case of size about 1 grm. (wet) is heated overnight with 10 c.c. of 2 N nitric acid at 90° C. (Extract B). Chlorine. An aliquot part of Extract A, or the whole of extract B, is boiled with a known excess of 0.1 N silver nitrate and filtered. The filtrate is titrated with 0.1 N potassium thiocyanate as usual. Zinc and magnesium.---A further aliquot part of Extract A is taken, and an extract of the size prepared as follows :—About 1 grm. is repeatedly evaporated to dryness with fuming nitric acid in a 250 c.c. narrownecked hard-glass flask provided with a funnel, the white residue is dissolved in hot dilute hydrochloric acid, and the solution diluted to about 100 c.c. (Extract C). These extracts are treated with 20 c.c. of 2N ammonium chloride and ammonia till alkaline and boiled, and any iron filtered off. The filtrate is made just acid with hydrochloric acid and boiled with 20 c.c. of 10 per cent. diammonium hydrogen phosphate and 15 c.c. of concentrated ammonia. The mixture is shaken in a stoppered bottle for half an hour, allowed to stand, filtered from the magnesium ammonium phosphate, and the precipitate washed. The filtrate is boiled to remove ammonia, and acetic acid is added till the solution is neutral to bromcresol-purple; after half an hour the zinc is all precipitated as zinc ammonium phosphate. The washed precipitates of both zinc and magnesium ammonium phosphates are dissolved off their filters and from the sides of the precipitating vessels with dilute nitric acid, boiled with ammonium nitrate and 50 c.c. of 3 per cent. ammonium molybdate, added from a tap funnel, with constant stirring. After 15 minutes the ammonium phosphomolybdate in each case is filtered off and washed, the filter papers, with the precipitates, are treated with a known

excess of N sodium hydroxide solution, and the solution back-titrated with N hydrochloric acid, phenolphthalein being used as indicator (1 grm. -equivalent of alkali=3 grms. of  $P_2O_5$ ), from which results the zinc and magnesium can be calculated.

Glycerin in Yarn or Cloth.—Ten grms. of the fabric are extracted in a hot Soxhlet apparatus with acetone for 4 hours, and the filtered extract evaporated to dryness, the residue being extracted 3 times with 25 c.c. of alcohol-ether mixture (2:1) (Extract D). Size.—Two grms. are ground up with 40 c.c. of alcohol (added gradually) and 20 c.c. of ether; the clear liquid being decanted. The extraction is repeated, and the solutions mixed (Extract E). A little boric acid is added to Extracts D and E to reduce the loss of glycerin, and the solutions evaporated to dryness, the last traces of alcohol being removed by a current of air. Fat is then removed with petroleum spirit, and the residue entirely freed from the solvent. It is now oxidised with chromic acid at 100° C. in a special jacketed burette, and the volume of carbon dioxide measured as usual. When the amount of deliquescent substances is small, certain blank corrections have to be made. R. F. L.

# Inorganic Analysis.

New Metallic Pyrocatechol Compounds and their Use in Microchemical Analysis. A. Martini. (Anal. Assoc. Quim. Argentina, 1926, 14, 177-184.)-Molybdenum, tungsten, and vanadium cations may be detected by means of the complex compounds which they form with pyrocatechol acetate in conjunction with either aniline or piperazine. With a drop of 1 per cent. ammonium molybdate solution, on a microscope slide, a drop of pyrocatechol acetate, added by means of a gold pen, produces an orange coloration. If then a small quantity either of pure aniline or of saturated piperazine solution is stirred into the drop by means of the same pen, a red precipitate appears; this consists of isolated and grouped crystals showing, under the polarising microscope, marked dichroism, strong refraction, and oblique extinction with angle varying from  $30^{\circ}$  to  $40^{\circ}$ . The reaction is given also by a drop of 0.1 per cent. ammonium molybdate solution, and similar results are obtained with ammonium tungstate or vanadate. Photographs are given of the microscopic appearances of the different crystalline T. H. P. compounds.

Detection of Cadmium in Presence of Copper. G. M. Karns. (J. Amer. Chem. Soc., 1926, 48, 2626-2627.)—In the usual scheme of qualitative analysis, the ammoniacal filtrate from the bismuth hydroxide is carefully neutralised; it should contain about 0.4 grm. of ammonium chloride. An equal volume of saturated sodium bicarbonate solution is added, which produces a white precipitate of cadmium carbonate, easily visible in the blue ammoniacal copper solution. The reaction detects 0.1 mgrm. of cadmium per c.c. in presence of 5 times that quantity of copper. W. R. S.

Qualitative Separation of the Platinum Metals. S. C. Ogburn. (J. Amer. Chem. Soc., 1926, 48, 2493–2507; 2507–2512.)—A scheme is presented in which the metals are precipitated from the mixed chloride solution in the following order: Palladium, by dimethylglyoxime; platinum, by  $\alpha$ -furildioxime; rhodium, by potassium nitrite; ruthenium, by sodium hydroxide; iridium and osmium, by zinc dust. The last two are digested with hypochlorite solution, osmium dissolving. For actual details the second paper should be consulted. W. R. S.

Attempted Separation of Hafnium and Zirconium by Ionic Migration. J. Kendall and W. West. (J. Amer. Chem. Soc., 1926, 48, 2619–2626.)—The method of ionic migration by electrolysis of an agar gel containing the two elements as complex anions gave only a very slight concentration of hafnium in the rear section of the gel. The degree of separation was much smaller than that attained in the case of rare-earth metals. Ionic migration is not a practical method of resolving a zirconium-hafnium mixture into its constituents. W. R. S.

Determination of Iodine by means of Hydrazine Sulphate. E. Cattelain (Bull. Soc. Chim., 1926, 39-40, 1279-1280.)—In the presence of crystalline sodium acetate and a large excess of iodine, hydrazine sulphate is quantitatively oxidised— $H_2SO_4, NH_2.NH_2+2I_2=4HI+N_2+H_2SO_4$ . Two grms. of crystalline sodium acetate and 0.065 grms. of dry and powdered hydrazine sulphate are dissolved in 50 c.c. of water and 40 c.c. of about 0.1 N iodine solution added. After 10 mins. approximately 0.1 N sodium thiosulphate solution is run in to discolouration point, and the quantity of iodine required to effect the oxidation calculated.

D. G. H.

Volumetric Determination of Selenium and Tellurium. W. T. Schrenk and B. L. Browning. (J. Amer. Chem. Soc., 1926, 48, 2550-2553.)-Selenium or tellurium dioxide (about 0.2 grm.) is dissolved in 25 c.c. of 40 per cent. sulphuric acid. In the case of tellurium, heating is necessary. The solution is diluted to 150 c.c., and treated with disodium phosphate (12 grms.), which prevents the precipitation of manganese dioxide; chlorides must be absent. A measured amount of standard permanganate (about 10 c.c. excess) is added, and the solution allowed to stand for 10 to 30 minutes. The excess permanganate is titrated electrometrically with ferrous sulphate. Two methods may be used for the simultaneous determination of selenium and tellurium : (a) Tellurium is determined by oxidation with excess of dichromate, and electrometric titration of that excess with ferrous sulphate, followed by the above permanganate method, giving selenium; (b) determination of selenium and tellurium by permanganate as above, determination of tellurium in a separate portion by dichromate, and selenium by difference. Very good results were obtained. W. R. S.

Micro-analytical Determination of Sulphur and Halogens. A. Récsei. (*Chem. Ztg.*, 1926, 50, 785.)—Asboth's method (*Chem. Ztg.*, 1895, 2040) may be applied to the micro-determination of sulphur in organic compounds in the following

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manner. With relatively non-volatile substances, 2 to 8 mgrms. are mixed in a nickel crucible of 2 to 2.5 cm. diameter with 100 times its weight of a mixture of 2 parts of sodium potassium carbonate and 1 part of sodium peroxide. The mass is heated with a 1 cm. flame, 4 to 5 cm. below the crucible, for 5 minutes, the flame being then gradually increased until the mixture melts. The cooled mass is heated with a few c.c. of water until it dissolves, the solution being transferred to a beaker, neutralised with hydrochloric acid, faintly acidified with the same acid containing bromine, and boiled until the bromine is expelled. The liquid is kept gently boiling, and 1 to 2 c.c. of 0.5 N barium chloride solution added from a capillary tube in drops at intervals of 10 seconds. The barium sulphate, which settles in granular form to the bottom of the liquid, is collected on a platinum Neubauer or filter crucible, washed with 50 per cent. alcohol, dried in an air oven, washed with a few drops of hot water, ignited and weighed.

To determine halogens, 2 to 10 mgrms. of the substance are treated as above, and the solution obtained boiled with a little bisulphite, acidified with nitric acid and, when cold, precipitated with 1 c.c. of 0.5 N silver nitrate solution. The liquid is heated on a water-bath or in an oven until the precipitate coagulates and then filtered through a filter tube fitted with a siphon or through a filter crucible, the precipitate being washed with water containing nitric acid and with alcohol and dried at 125° C. This method is applicable to nitrogen compounds other than cyano-derivatives. T. H. P.

On the Presence of Amines in the Distillate from Kjeldahl-Gunning Nitrogen Determinations. R. A. Cortner and W. F. Hoffman. (J. Biol. Chem., 1926, 70, 457-459.)-During the course of a study of the effect of various inorganic salts on the extraction of proteins from wheat flours in which study the amount of protein extracted was followed by Kjeldahl nitrogen determinations on aliquot portions of the extract, when certain salts were used a pronounced odour of (presumably) methylamines was noticed in the distillates when they were made This suggested that a considerable part of the nitrogen was being alkaline. distilled in the form of amines rather than as ammonia; an attempt was therefore made to determine whether aliphatic amines are present in the distillates from normal Kjeldahl-Gunning digests, and to find the effect of added salts upon their presence. The method of Weber and Wilson (J. Biol. Chem., 1918, 35, 385; J. Ind. and Eng. Chem., 1919, 11, 121) was used to determine the amines in the presence of ammonia. The results are tabulated. Amines are present in the distillate from a normal Kjeldahl-Gunning digest to the extent of approximately 7 per cent. of the nitrogen. Salts of the dibasic metals, magnesium, calcium, strontium, and barium have a marked influence on the amount of amines which are present in such a distillate. Nearly one-fifth of the total nitrogen may be in the form of amines if the digest is made in the presence of 5 grms. of MgSO<sub>4</sub>.7H<sub>2</sub>O. This is a preliminary paper. P. H. P.

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## Physical Methods, Apparatus, etc.

Sliding-gauge Colorimeter, and the Determination of Small Amounts of Ammonia, Nitrites, Lead, and Iron. A. L. Bernoulli. (Helv. Chim. Acta, 1926, 9, 827-840.)—A strongly constructed precision colorimeter, especially suitable for water analysis, is described. Rays of light from a common source pass through two diaphragms. One ray continues through a small glass cell containing the standard solution, to a Lummer-Brodhun photometer-cube, and thence through a focussing lens to the evepiece, whilst the other is carried through the solution to be tested by means of prisms, and is finally directed by the photometer-cube and lens so as to form a small circle inside the illuminated evepiece. The distance between the prisms is adjusted till the fields are of equal intensities, and is read on a vernier-scale. For every colour the "optical layer thickness" must be determined from the reading  $(I_{a})$  obtained with the same solution in each vessel. The solution of unknown concentration (C') is then matched against a standard of concentration C. If the reading obtained is I', it is shown that  $C'/C = I_o/I'$ . The methods of Snell (Colorimetric Analysis) are adapted for use in the instrument. Lead (0.0005 to 0.000005 grm. per litre) may be determined as lead sulphide, and 0.00002 mgrm. has been determined with an accuracy of 1 per cent. Dichromates are determined directly, and trivalent iron by the pyrocatechol method with an accuracy of 0.5 per cent. The phenol and hypochlorite reagent is preferable to that of Nessler for ammonia, of which 0.005 mgrm. in 1 c.c. may be determined to within 0.1 per cent. For nitrites the Griess reagent enables an accuracy of 1 per cent. to be obtained for  $5 \cdot 0.10^{-8}$  grm. of nitrite-nitrogen per c.c. If the positions of the solutions are interchanged, the instrument may be used for microdeterminations; for example, 0.002 mgrm. of iron in 0.1 c.c. has been determined with an accuracy of 0.4 per cent. J. G.

Standardisation of Methods for Testing the Fastness of Dyes. A. Crummett. (J. Soc. Dvers and Col., 1926, 42, 301.)—The author discusses the desirability for standardising methods of testing dyes in general and also describes details of tests that he has carried out on the fastness of dyes on silk. Fastness to Light.—Sunlight is used in preference to that from any artificial source. The dyed samples are exposed, night and day, in a frame in the open air facing south, but protected from weather by a sheet of glass, although allowing free circulation of air over the samples. One portion is covered, to begin with, and other portions after one, three and six weeks, thus showing the progress of fading. Fastness to Water.—The dyed silk is plaited with a white silk and a white cotton and immersed overnight in 40 times its weight of cold water. It is then squeezed, dried and examined after unplaiting. Records are made as to loss of colour, and staining of. the white silk and cotton. Fastness to Washing.-A similar plait is washed and scoured in a 0.5 per cent. "flake-soap" solution for 1 hour at 40° C., and another plait at 80° C. Fastness to Degumming.-The dyed silk with another forms the warp, and a hard twisted thread in the gum forms the weft. Degumming is

carried out by boiling in 1 per cent. soap solution for two hours. Little or no colour should be lost, and the white ground-work should not be stained. *Fastness to Perspiration.*—This consists in treating the plait with a salt and acetic acid solution at  $40^{\circ}$  C. for 24 hours..

Results obtained by the American Association of Textile Chemists are also discussed. R. F. I.

## **Reviews.**

A TEXTBOOK OF ORGANIC CHEMISTRY. By A. F. HOLLEMAN. Edited by A. JAMIESON WALKER and O. E. MOTT. Sixth English Edition. Pp. xx. +581. London: Chapman & Hall. Price 17s. 6d.

The value of this textbook, previous editions of which have already been reviewed in the pages of this journal, may be gauged from the number of editions already issued amounting to a total of 44 in English and other European languages.

The present issue is in no way inferior to former editions, but considerable revision has taken place and reference is now made to Irvine's researches upon the carbohydrates, the relation of colour to constitution of the dyestuffs, and the physical chemistry of organic compounds, prominence being given to the theories upon which the subject is based, and the theoretical proofs of constitutional formulae. The physico-chemical sections are an admirable feature serving to retain the interest of the student in those borderlands between two sciences in which so many advances have been made during the last few decades.

The author evidently possesses a wide knowledge of the requirements of the student and the subject matter is expressed in a clear and instructive manner, emphasis being laid upon the relationships between different compounds in which the interest of organic chemistry, apart from practical work, so largely rests. Since frequent references are made to the author's *Laboratory Manual of Organic Chemistry* the 14 pages devoted to laboratory methods and the theoretical principles upon which these are based seem somewhat redundant.

The onerous duty of proof-reading such a textbook has been carried out with great care, this being reflected in the almost complete absence of errors in the text, the numerous numerical references and the combined subject and name index containing over 3,000 headings. One or two examples of quaint expression are to be met with, such as "apparatuses" on p. 15 and the verb "yeasting" in connection with bacterial inoculation on p. 57, and the omission of the second "n" from amino-naphthalene is noticeable on p. 453. No doubt many chemists will disagree with the statements that acetone and ethyl benzoate possess peppermint-like odours.

The volume is well produced, the typescript being legible and the illustrations clear, and the book has the additional merit of lying practically flat at whatever page it is opened.

The author, translators and publishers have combined to produce a thoroughly reliable work which will still further enhance the reputation achieved by earlier editions and will be viewed with satisfaction by teachers and students alike.

T. J. WARD.

A ТЕХТВООК OF INORGANIC CHEMISTRY. By Dr. FRITZ EPHRAIM. English Edition by P. C. L. THORNE, M.A., M.Sc. Pp. xii. + 805. London: Gurney & Jackson. 1926. Price 28s. net.

English-speaking chemists with an inadequate command of German must be grateful to Mr. Thorne for the production of the handsome volume translating the third edition of Ephraim's Inorganic Chemistry. As the original German work has not been reviewed in these pages, no excuse is needed for a sketch, in a few lines, of its scope, substantially as given in the Author's Preface. If the general perspective is not to be lost in a mass of unco-ordinated detail, an account of the advance in our knowledge of inorganic chemistry during the last half century must be given according to a more rational method of presentation similar to that used in organic chemistry. This consists in treating related compounds together, thus emphasising comparisons and connections, and their causes and effects, instead of arranging the compounds in the manner of a dictionary. Generalisation effects a simplification in the process of acquiring and classifying knowledge; whilst the customary textbook treatment fails to place related facts together, and the connection between the isolated statements is never sufficiently emphasised.

There can be no doubt that, on the whole, the author's plan has succeeded admirably; the lucid presentation and discussion of relevant facts, which had to be selected from the mass of available material, including the most recent discoveries, combined with the principle of systematic generalisation adopted, have produced a most instructive as well as fascinating treatise of chemical philosophy. The reviewer cannot help contrasting this work with one he recently criticised (ANALYST, 1924, 49, 255); even after making due allowance for the fact that the present book is intended for more advanced reading, one can hardly conceive of a more fundamental difference in mental attitude on the part of the respective authors, or believe that the two books are contemporaneous.

An outstanding novel feature and merit of Ephraim's work is, that it introduces Werner's co-ordination theory almost at once and utilises it fully in the elucidation of the constitution of all the less simple compounds. No other textbook extant appears to approach the work under review in this respect.

The text-matter is subdivided into seven Sections, namely, (1) The Elements, (2) Halogen Compounds, (3) Oxides of Hydrogen and the Metals, (4) Compounds of Sulphur, Selenium, and Tellurium, (5) The Nitrogen, Phosphorus, Arsenic Group, (6) Elements of the Fourth Group and Boron, and (7) The Rare Earths, Alloys, and Radioactivity. Here the writer feels inclined to join issue on a

debatable question, *i.e.* the mode of grouping adopted for the elements of the Fifth Group. Section 5 is devoted to nitrogen, phosphorus, arsenic, antimony, and bismuth, whilst the discussion of vanadium, niobium, and tantalum is scattered mainly over Sections 2 and 3. It would seem more natural, in view of numerous analogies, to have discussed the oxides and acids of vanadium, niobium, and tantalum along with those of the other elements of the group; or else, antimony, and especially bismuth, might have been transferred to Sections 2 and 3, as being more metallic than the three elements excluded from Section 5.

A careful study of this treatise, and of Section 6 in particular, may serve as a reminder to the generation of chemists now in middle life, many of whom have specialised too narrowly in organic chemistry, that the vast edifice of the carbon compounds is, after all, if the most imposing, yet only one of a number of structures, and that inorganic chemistry, after a period of stagnation, has re-conquered its position of rightful pre-eminence.

The reader is occasionally reminded that our English inorganic nomenclature is still awaiting standardisation, even for binary compounds. Thus we say "nitrogen trioxide" for  $N_2O_3$  (p. 564), but, if prefixes are to continue in use, the name should be "nitrogen sesquioxide," as against "tungsten trioxide,"  $WO_3$ ; "nitrous anhydride" may still be used, though really too vague for precise terminology. The use of "pentoxide" for  $N_2O_5$  might be defended on the ground that true pentoxides  $MO_5$  are unknown; but here again, "trioxide" for  $MoO_3$  and "pentoxide" for  $Mo_2O_5$  is not logical. A numerical system of nomenclature seems free from these objections: "2:3 nitrogen oxide" for  $N_2O_3$ ; "2:5 nitrogen oxide" for  $N_2O_5$ ; "1:3 tungsten oxide" for  $WO_3$ ; "1:4 osmium oxide" for  $OsO_4$  (cf. ANALYST, 1925, 50, 192).

The following terms referring to complex fluorides were noticed: "hydro-fluosilicic" (p. 666); "silicofluorides" (668); "hydrofluoboric" (666; indexed as such and as "fluoboric"); and "fluorochromic" (392). The "fluo"-acids mentioned in p. 405 are indexed as "fluoro-" acids. In the double fluorides, the old prefix "fluo" should give way to "fluoro." The most correct terms would seem to be "hexafluorosilicic," "hexafluorosilicate," "tetrafluoroboric" (cf. ANALYST, 1924, 49, 552), though the prefix of number is generally omitted for the sake of brevity.

There are only a few minor details that may require comment or criticism. According to the table on p. 38, the existence of osmium octafluoride is doubtful, though Ruff and Tschirch (*Ber.*, 1913, 46, 929) claim to have isolated this compound. The statement that "the diamond surpasses all other bodies in hardness and refractive index" (p. 91) is true as regards hardness, but cinnabar, pyrargyrite, and rutile have higher refractive indices. Among sulphato-acids (p. 482) the interesting zirconylsulphuric acids have received no mention. The same applies to the oxysulphides of antimony, which are better known than those of phosphorus (p. 638); the most familiar, Sb<sub>2</sub>S<sub>2</sub>O, occurs native as kermesite, and has also been prepared from the thioiodide SbSI; the other, Sb<sub>4</sub>OS<sub>5</sub>, is obtained by a fusion method

(Quercigh, J. Soc. Chem. Ind., 1912, 31, 384). It may surprise many readers to find that the discussion on the constitution of the natural silicates and aluminosilicates is condensed into two pages, but the author's view is that "this cannot be entered into further in the compass of this book" (p. 708). A brief synopsis of the latest discoveries and theories in magnetochemistry and isomorphism would have been most welcome.

A comparatively small number of misprints occurs, chiefly in the formulae; most of them are quite obvious. The following should be pointed out: "SnI<sub>3</sub>" (p. 192, line 15; read SnI<sub>2</sub>); "Cl<sub>2</sub>O" (p. 310, line 4 from bottom, and p. 311, line 5; read ClO<sub>2</sub>); "H<sub>5</sub>TeO<sub>5</sub>" (p. 446, line 14; read H<sub>4</sub>TeO<sub>5</sub>, and "tetra" for "penta"); atmospheric "temperature" (p. 432, line 11; read pressure); and "H<sub>2</sub>SiF<sub>4</sub>" (p. 666, last line; read H<sub>2</sub>SiF<sub>6</sub>). "Moh's" scale of hardness (pp. 30, 329) is mis-spelt in quite a number of books; the mineralogist who gave his name to the scale was Friedrich Mohs.

### W. R. SCHOELLER.

ORGANIC SYNTHESES. Vol. VI. HENRY GILMAN, Editor-in-Chief. Pp. 120. New York: John Wiley & Sons; London: Chapman & Hall, Ltd. 1926. Price 7s. 6d. net.

One or two new features of a minor character are to be found in No. VI of this series; an index to authors has been added; there are a couple of pages of additions and corrections to the preceding volume, and some references are given to later work on preparations previously described. There is an increase in the number of contributors of whom one-third are now from countries other than the United States. Of twenty-nine syntheses mention may be made of the following: Acrolein, benzil, ethylacetoacetate, racemic acid, and phenylisothiocyanate. It is evident that the editors spare no pains to make this series of volumes convenient to use and thoroughly up-to-date.

### A. F. KITCHING.

SYNTHETIC RUBBER. By S. P. SCHOTZ, D.Sc., F.I.C. Pp. 129. London: Ernest Benn, Ltd. 1926. Price 21s. net.

"The present volume," according to the author, "forms not merely an account of the synthesis of rubber from simple bodies, but particular stress is laid on those lines of production of artificial rubber which are likely to prove easiest. The relative importance of various methods for industrial purposes has constantly been borne in mind, and the purely scientific considerations had to take second place in comparison with processes capable of industrial application." The reviewer, nevertheless, considers that the merit of Dr. Schotz' volume lies in his collation and exposition of the scientific data underlying the attempts to produce synthetic rubber, and that if it is to be looked at from a technical or commercial point of view a process of sternly destructive criticism would be essential. To judge by the foregoing quotation and those such as: (a) "... the author has

attempted to give a fair account of the way in which the converging efforts of chemists of all nations have resulted in a stupendous achievement"; (b) "The attempts to synthesise rubber are of very recent date. . . . But once started, these attempts followed close on one another with astounding results"; (c) "Here we have a body unsurpassed in its complicated structure and yet prepared from the simplest possible compounds in a very short number of operations," etc., the uninitiated reader might well believe that synthetic rubber had (commercially or at least technically) "arrived." Yet the fact is, as the reviewer believes, that up to the present not a single pound of synthetic rubber even remotely comparable in quality and cost of production with the natural article, has been produced. This statement, however, in no way detracts from the merit of the extremely fine scientific work that some of the most brilliant organic chemists in various parts of the world have devoted to the synthetic rubber problem, and, as has already been indicated. Dr. Schotz has done well to collate this work, and, moreover, has well collated it. The matter in the volume under review is sub-divided as follows: Chapters I to III deal with the production and properties of "raw materials," namely, isoprene, butadiene and dimethylbutadiene. Chapter IV covers methods of polymerisation or condensation, catalysts, etc. In Chapters V and VI vulcanisation and stabilisation respectively are described, and the last two chapters are headed "Properties and Constitution of Natural Caoutchouc and Synthetic Rubber," and "History of Synthetic Rubber," respectively. The book contains a number of interesting illustrations of apparatus and plant, and the printing, paper and general "get up" are admirable.

P. SCHIDROWITZ.

CHEMICAL SYNONYMS AND TRADE NAMES. By WILLIAM GARDNER. Pp. 355. London: Crosby, Lockwood & Son. 1926. Price 30s. net.

In this, the third edition of Mr. Gardner's comprehensive dictionary of chemical synonyms and trade names, many of the definitions have been re-written, as the result of later and more exact information, and many new definitions have been added. The book now contains nearly 20,000 definitions and cross references. The author has compiled a series of definitions of substances under their trade names, covering alloys, dyestuffs, commercial chemical products, explosives, minerals, pigments, and pharmaceutical preparations, which must be well-nigh exhaustive.

A careful testing of the definitions, over a wide range of substances, shows that the greatest care has been taken in the compilation. The work provides information which is rarely furnished by the largest treatises on the branches of chemical knowledge dealt with, and consequently it becomes at once a valuable and exceedingly useful work of reference.

That a third edition has become necessary is itself the highest testimony of the usefulness of the book. It can be unreservedly recommended to all engaged with chemical substances in any form or for any purpose.

J. F. Spencer.