DECEMBER, 1930. Vol. LV., No. 657.

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 5th, the President, Dr. J. T. Dunn, being in the chair.

Certificates were read for the first time in favour of:—Arthur Nicholls Ainsworth, B.Sc., Bertram Arthur Gough, William Henry Gough, M.Sc., A.I.C., William Henry Shilling, B.Sc., A.I.C.

Certificates were read for the second time in favour of Leonard Balmforth, B.Sc., F.I.C., Reginald Joseph Cole, B.Sc., Violet Dorothy Dudman, B.Sc., A.I.C., Frank George Edmed, O.B.E., B.Sc., A.R.C.Sc., F.I.C., Roy Gardner, D.Sc., F.I.C., William Victor Griffiths, B.Sc., A.I.C., Daoud Younis Haddad, B.Pharm., Percy George Terry Hand, F.I.C., Magnus Herd, B.Sc., A.R.T.C., F.I.C., Gilbert Underwood Houghton, B.Sc., A.I.C., Archibald Robert Jamieson, B.Sc., F.I.C., William Jefferys Lesley, M.Sc., Ph.D., A.I.C., Allison Reginald Murray MacLean, B.A., M.Sc., Ph.D., Frederick Henry Newington, F.I.C., Colin Paterson, B.Sc., A.I.C.

The following were elected Members of the Society:—John Herbert Bushill, M.Sc., A.I.C., Edward Quentin Laws, B.Sc., A.I.C., and Hubert Taylor, B.Sc., A.I.C.

The following papers were read and discussed:—"The Analysis and Composition of Vegetable Parchment used for Packing Dairy Products," by Paul Arup, M.Sc., F.I.C.; "The Determination of the Milk Proteins," by George M. Moir, M.Sc., Ph.D., A.I.C.; "The Lead Reduction Method for the Volumetric Determination of Tin, and the Interference of Copper and Antimony," by S. G. Clarke, B.Sc., Ph.D., A.I.C.; "A New Method for Determining Traces of Chromium in Steel," by W. J. Agnew, B.A.

NORTH OF ENGLAND SECTION.

A MEETING of the Section was held in Manchester on Saturday, October 25th, 1930. Nineteen members were present, and the Chairman (Mr. G. D. Elsdon) presided.

A paper was read by R. H. Kay on "The Examination and Commercial Analysis of Cotton Cloths," and was followed by a discussion.

A collection of notes, under the title of "Our Professional Forbears," was given by the Honorary Secretary, referring to the founders and early years of the Society. Afterwards several senior Public Analysts gave reminiscences and reflections which proved instructive and amusing.

Death.

WITH deep regret we record the death, on November 22nd, of Mr. E. W. Voelcker, a Past-President of the Society. An obituary notice will be published in a subsequent issue.

Obituary.

HARVEY WASHINGTON WILEY.

THE recent death of Dr. Harvey W. Wiley, Honorary Member of the Society of Public Analysts, at his home in Washington, June 30, 1930, at the age of 86, removes the most conspicuous figure in the history of pure food legislation in the United States. His diversified activities as chemist, teacher, publicist, lecturer, and author were all inspired by the one leading motive of his spectacular career—that of arousing public opinion against the debasement of the nation's food.

Dr. Wiley was born on October 18, 1844, in a log cabin upon a farm of the Indiana frontier. It was here that his interests were first awakened in agriculture and in farming technology, subjects which he later enriched by his scientific investigations. His inclinations were given a special direction towards chemistry by his studies at Hanover College, and his subsequent training in medicine at the Indiana Medical College first aroused his interest in the applications of chemistry to questions of the public health. Post-graduate studies were then undertaken at Harvard University and also at the University of Berlin. While at the latter institution, in 1878 and 1879, he worked with Doctor Sell of the German Imperial Health Office, and it was here that he received his first instruction in advanced methods of food analysis, more especially as a means of detecting adulteration. Upon his return to America the results of these years of study were amplified by his work as Professor of Chemistry at Purdue University and as State Chemist of Indiana, when he first began to publish contributions upon new types of apparatus and the chemical analysis of foods. His official report to the Indiana Board of

Health upon the adulteration of honey, syrups and molasses with commercial glucose, published in 1879, marks his entry into the field which was to demand so much of his attention during the next fifty years.

In 1883 Dr. Wiley was persuaded by Commissioner G. B. Loring to accept the appointment of Chemist of the Department of Agriculture. His services in this position were along three principal lines of work, in each one of which he won the highest distinction.

The first of these was his chemical and technological investigation of the sugar-producing crops of the United States—the maple, the sorghum, the sugar cane and the sugar beet. His experiments upon improving the methods of extracting sugar from the sugar cane caused manufacturers to make a complete departure from the archaic type of cane mill that had remained unchanged for many generations. Even more important was his work in determining the climatic boundaries within which the sugar beet could be grown successfully in the United States. This work ranks as the best example of the climatic survey of a crop in the annals of American agricultural chemistry.

A second important contribution of Dr. Wiley was his work in standardising and improving the methods of agricultural chemical analysis. He was a great student of this subject, devising new pieces of apparatus and originating new methods of procedure. His work in this field is best exemplified by his well-known three-volume treatise, *Principles and Practice of Agricultural Analysis*, and by his work as a founder and member of the Association of Official Agricultural Chemists.

But chemical analysis with Dr. Wiley was only a means to the one important end of protecting the public against the debasement of its food—the third and crowning achievement of his career. The analyses of American food products which he initiated immediately after his appointment as Chemist of the Department of Agriculture and the results of which were published in that important series of ten brochures upon "Foods and Food Adulterants," known as Bulletin 13 of the Bureau of Chemistry (1887–1902), revealed an almost incredible state of sophistication. It was to the correction of this evil that he consecrated the remainder of his life. By his writings and addresses he aroused public opinion to such a point that he finally secured, in 1906, the passage by Congress of the Food and Drugs Act, after more than twenty years of determined opposition by selfish commercial interests. Confronted with an even more determined resistance he then began the administration of this Act under difficulties which would have discouraged a less resolute reformer. The obstacles, the treachery, the abuse, which he incurred in the discharge of his public duties at this time are matters of common knowledge.

In his twenty-nine years as Chemist of the Department of Agriculture, Dr. Wiley built up an organisation from six to more than six hundred employees. He originated many lines of chemical research in such fields as soils, milk products, road construction and standardisation of apparatus which afterwards became the nuclei of separate bureaus.

Dr. Wiley retired in 1912 from his position as Chief of the Bureau of Chemistry in order to become Director of the Bureau of Foods, Sanitation and Health, of the *Good Housekeeping Magazine*, in which office by his monthly editorials and by his numerous books he wielded a great influence in keeping public opinion aroused against the perils of food adulteration. Because of the infirmities of age Dr. Wiley retired from the active duties of this office at the commencement of the present year, but he continued to manifest an interest in pure food legislation until the very day of his death.

The cause of pure food in America was most fortunate in having as its first great protagonist a man with the courage, crusading spirit and prodigious energy of Dr. Wiley. His unfailing good humour, wit and oratorical ability were additional qualities that helped him to hold the public interest. "Whole souled, helpful, full of enthusiasm and a magnificent man among men" was the characterisation of him by a prominent agricultural chemist of Holland. He was known in many foreign countries, to some of which he rendered services as an adviser. He assisted the French Government in 1907 in revising its food laws in accordance with the principles of pure food legislation which he had initiated in the United States, a service for which he was made *Chevalier de la Légion d'Honneur.*

Dr. Wiley's fortitude, his unfailing optimism when confronted with almost insuperable difficulties, and his sacrifice of private financial opportunities in order to serve the welfare of the people, will always remain as shining examples for future generations of chemists.

C. A. BROWNE.

The Volumetric Determination of Reducing Sugars. Part IV. Invert Sugar.

BY ARTHUR R. LING, M.Sc., F.I.C., AND WILLIAM A. CARTER.

IT will be agreed that the volumetric determination of reducing sugars by titration with Fehling's solution, using ferrous thiocyanate as indicator, as described by Ling and Rendle (ANALYST, 1905, **30**, 182; 1908, **33**, 167),* leaves little to be desired as regards accuracy. It has indeed been shown by Ling and Jones (*ibid.*, 1908, **33**, 160) that the average error is about 1 in 300. Evidence has also been brought forward (*loc. cit.*) that the volumetric method is not only more convenient, but, with commercial products, more accurate than the gravimetric method.

Whilst the statements in the last paragraph in regard to the accuracy of the method of Ling and Rendle have been amply confirmed by subsequent work, there is one disadvantage which attaches to it, namely, the fact that the indicator employed is an external one.

* There is an unfortunate error in the title of Part III of these communications (Ling and Rendle, *ibid.*, 1908, **33**, 167). It should read:—"The Determination of Invert Sugar in Presence of varying Quantities of Sucrose."

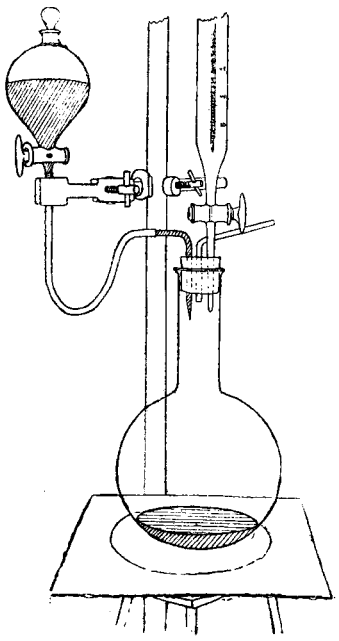
Fortunately, during the last few years, Lane and Eynon (*J. Soc. Chem. Ind.*, **1923**, **42**, 32T.), have suggested the use of methylene blue as an indicator which can be used internally. This suggestion has proved to be a most valuable one; and that when the volumetric method is carried out on the correct lines, with the use of methylene blue as an internal indicator, it is far more expeditious than when the external indicator, ferrous thiocyanate, is employed. The purpose of this communication is to indicate the precise procedure necessary to ensure the greatest accuracy. In this paper we deal with invert sugar, but the reducing power of other sugars will be dealt with in a subsequent paper. We find that, in order to secure accuracy in this method, three conditions must be complied with: (1) The liquid must be kept boiling throughout the titration; (2) the titration must occupy at least ten minutes; (3) air must be excluded from the surface of the assay liquid. The instructions given by Ling and Rendle go far to secure these conditions. They say, "Freshly mixed Fehling's solution (10 c.c.) is accurately measured into a 200 c.c. boiling flask and raised to boiling. The sugar solution . . . is run into the boiling liquid in small amounts, commencing with 5 c.c. After each addition of sugar solution the mixture is boiled, the liquid being kept rotated."

It should be noted that, throughout their work, Ling and Rendle and Ling and Jones employ 10 c.c. of Fehling's solution as a standard method, the concentration of the sugar solution being so adjusted that from 20 to 30 c.c. are required to complete the reduction. With larger volumes of Fehling's solution the method becomes cumbersome, but the same results are obtained, as was shown by Ling and Jones (*loc. cit.*, **164**).

The method adopted by Lane and Eynon is, using their own words, as follows:—"Ten or 25 c.c. of Fehling's solution is measured into a flask of 300 to 400 c.c. capacity and treated cold with almost the whole of the sugar solution required to effect the reduction of all the copper, so that, if possible, not more than 1 c.c. is required later to complete the titration. The approximate volume of the sugar solution required is ascertained by a preliminary incremental titration. . . . The flask containing the cold mixture is heated over a wire gauze; after the liquid has begun to boil, it is kept in moderate ebullition for 2 minutes, and then, without removal of the flame, 3 to 5 drops of the methylene blue indicator are added, and the titration is completed in 1 minute further, so that the reaction liquid boils altogether for three minutes without interruption." The preliminary titration —increment method, as it is called by the authors, is carried out as follows, again quoting their own words. "Ten or 25 c.c. of Fehling's solution in a 300–400 c.c. flask is treated cold with 15 c.c. of the sugar solution, and without further dilution heated to boiling over a wire gauze. After the liquid has been boiling for about 15 seconds, it will be possible to judge if the copper is almost all reduced. . . . A few drops of methylene blue indicator are added, boiling is continued for 1–2 minutes from the commencement of ebullition, and then the sugar solution is added in small quantities, say 1 c.c. or less at a time, the liquid being allowed to boil

for about 10 seconds between successive additions, until the colour of the indicator is completely discharged."

EXPERIMENTAL.—The titration is carried out in a 200 c.c. boiling flask fitted with a trebly bored cork or rubber stopper, previously boiled with distilled water. The jet of the burette passes through one hole, a tube connected with a reservoir of 1 per cent. methylene blue solution through a second, and an open tube bent at a right angle through the third. The boiling is carried out on wire gauze covered with asbestos.



The apparatus employed, which was devised by one of us (W. A. C.), is shown in the accompanying sketch.

The invert sugar solutions were prepared by the method described by Ling and Rendle (*loc. cit.*). The concentration was 0·2 grm. per 100 c.c. The volume of Fehling's solution used was in all cases 10 c.c. It is important that the same volume of 1 per cent. methylene blue solution be used in standardising the Fehling's solution and in the actual assay, since the indicator acts in virtue of the fact that it is reduced to the leuco base by the sugar solution, and therefore a larger volume of assay liquid is required to complete the reduction when a larger quantity of methylene blue solution is employed. In all the experiments herein recorded 5 drops of methylene blue solution were added just before reduction was complete. These conditions having been fixed, it remains to be shown how the results are influenced by the total time occupied by the reduction, the exposure of the reduced or partly reduced Fehling's solution, when not in ebullition, to the atmosphere, and the use of a flask closed as above described, as compared with an open flask.

FIRST SERIES (Fehling's Solution "A").—*Influence of Time.*—The liquid was maintained in ebullition during the whole of the experiment, and the assay liquid added in small quantities at a time.

FEHLING'S SOLUTION "A."

Duration of titration. Minutes.	Volume of assay liquid required. c.c.
5	25·0
8	24·7
10	24·3
10	24·3
15	24·2
15	24·2
15	24·2

It is evident from these results that the titration should occupy about 15 minutes. This can be secured by adding to the boiling Fehling's solution successive quantities of 0·5 c.c. of assay liquid every 15 seconds. In this way the liquid may be maintained in gentle ebullition throughout the experiment.

SECOND SERIES.—0·5 c.c. of assay liquid run in every 15 seconds.

FEHLING'S SOLUTION "A."

Duration of titration. Minutes.	Volume of assay liquid required. c.c.
13	24·25
14	24·25
13	24·2
13	24·2
13	24·25

THIRD SERIES.—Fehling's Solution "B." Closed flask. 0·5 c.c. of assay liquid run in every 15 seconds.

FEHLING'S SOLUTION "B."

Duration of titration. Minutes.	Volume of assay liquid required. c.c.
6	24·4
8	24·3
10	24·0
11	24·0, 23·9
15	24·0

FOURTH SERIES.—Lane and Eynon's "incremental" method. The titration period was 5 minutes.

	Volume of assay liquid required. c.c.
200 c.c. flask (closed)	25·2, 25·5
300 c.c. ,, (open)	25·0
300 c.c. Florence flask (open)	25·2

FIFTH SERIES.—Lane and Eynon's "Final" titration. A volume of 24 c.c. of assay liquid was run in before boiling. The titration period was 3 minutes.

		Volume of assay liquid required.
Fehling's solution "A."	200 c.c. flask (open)	25·7
	,, ,, ,, ,,	25·7
	300 ,, ,, ,,	25·55
	,, ,, ,, ,,	25·6
Fehling's solution "B."	200 c.c. flask (closed)	25·5, 25,5
	,, ,, ,, ,,	25·5, 25·3
	300 ,, ,, (open)	25·6, 25·5
	,, ,, Florence flask (open)	25·6, 25·5

DISCUSSION.—A consideration of the results set forth in this paper indicates plainly that our method of titrating solutions of invert sugar with Fehling's solution yields more concordant results and gives a higher value for the reducing power of invert sugar than any other volumetric method, employing Fehling's solution, previously described. This applies to the method described by Lane and Eynon, although they made a great advance in suggesting the use of methylene blue as indicator. Not only are the results obtained by our method more concordant than when Lane and Eynon's method is employed, but, in addition, there is the distinct advantage that no preliminary titration is necessary, the first titration being as accurate as the succeeding ones. Thus the method is more expeditious than any other known to us.

The necessary conditions are: The use of a closed flask, in which an atmosphere of steam is maintained during the titration; the addition of successive small volumes, *e.g.* 0·5 c.c., at a short interval of time, of assay solution to the boiling Fehling's solution, so that boiling is continuous throughout the titration; and, lastly, the length of time occupied by the titration, which must be more than 10 minutes.

As regards concordance of results, the agreement between separate titrations is not so close with Lane and Eynon's method as with our own. It is interesting to note, however, that small differences only are obtained when Lane and Eynon's method is carried out in our closed flask and in an open flask. The difference between the results obtained by our method and by Lane and Eynon's final method is due principally to the fact that in the latter the bulk of the assay solution is added to the Fehling's solution in the cold, and that the time of boiling is too short. The actual titration volumes obtained, when Lane and Eynon's conditions and our own are adhered to, differ very greatly. Whilst, therefore, on the evidence submitted, we must naturally claim greater accuracy for our method than for that of Lane and Eynon, at the same time we freely admit that the accuracy of the two methods does not differ seriously. Thus, when Fehling's solution has been standardised by the method described by Lane and Eynon, and the assay carried out in the same manner, the results would generally be close to the truth.

In conclusion, we have to express our thanks to Mr. J. W. Green for carrying out some of the experimental work herein recorded.

THE UNIVERSITY,
BIRMINGHAM.

Investigation of Rye Oil.

By J. W. CROXFORD, A.I.C.

(Work done under the Analytical Investigation Scheme.)

(Read at the Meeting, October 1, 1930.)

It is curious, and rather surprising, that there is no record of any detailed investigation of this oil, notwithstanding the importance of rye as a food, especially now that rye biscuit is being widely used. The only analysis on record appears to be that of R. Meyer in 1903, quoted by Lewkowitsch, Vol. II. The chief drawback to a full examination is, possibly, the small amount of oil present in rye (usually about 2 per cent.). It is necessary to extract somewhat large quantities of oil for experimental purposes. There has, therefore, been a gap in our knowledge of the nature of oils in common food products.

The oil used in this work was obtained from two samples of rye, one sample of "Ryvita crispbread," light (which I presume to mean light in colour), and one sample of rye flour. In each case the oil was extracted with petroleum spirit (b.pt. 40 to 60° C.), which had been redistilled before use; the solvent was distilled off, and the residual oil dried in an atmosphere of carbon dioxide at 70° C. The rye flour was obtained in order to avoid the necessity of grinding previous to extraction.

The oils obtained from these different sources varied in colour from dark green to yellowish brown, and were of a semi-solid consistence at 15° C., with a faint characteristic odour.

The physical and chemical characteristics of these oils and their respective fatty acids are shown in the following tables, which also include, for comparison, the results obtained by Paul (Analyst, 1921, **46**, 238) in his examination of oil of oats:

Rye Oils.

Oil source.	Sp.gr. at 15°/15°C.	Acid value.	Iodine value, Wijs.	Bromine value (vapour method).	Iodine value calculated from Br value.	Saponification value.	Unsaponifiable matter. Per Cent.	Livache test. 28 days. Per Cent.
Rye A	0·9374	27·8	118·3	74·2	117·8	178·7	11·2	2·7
Rye B	0·9412	10·7	129·9	78·6	124·8	173·4	9·05	3·0
Ryvita	—	20·0	110·7	69·8	110·8	187·0	8·9	—
Rye flour	0·9283	24·0	126·8	80·0	127·0	186·0	8·2	2·75
Rye (R. Meyer, 1903)	0·9334	—	81·8	—	—	196·0	—	—
Oats (Paul, 1921)	0·9250	68·9	114·2	—	—	189·8	1·3	—

FATTY ACIDS FROM RYE OILS.

Fatty acids. Source.	Melting Point °C.	Iodine value.	Bromine value (vapour method).	Iodine value calc. from Br value.	Thio-cyanogen value calc. as iodine value.	Calculated composition.	Per Cent.	Mean molec. weight.	Pb. salt-alcohol solid acid. Per Cent.
Oil from Rye A	33	122·7	77·05	122·3	66·0	Linolic	62·65	291	—
						Oleic	10·35		
						Solid	27·0		
,, Rye B	40·5	138·3	86·3	137·0	75·7	Linolic	69·2	312·5	—
						Oleic	14·55		
						Solid	16·25		
,, "Ryvita"	37·0	115·3	72·45	115·1	69·8	Linolic	50·27	286	—
						Oleic	27·0		
						Solid	22·73		
,, Rye flour	—	128·5	81·4	129·2	72·75	Linolic	61·6	309	17·1
						Oleic	19·0		
						Solid	19·4		
,, Rye (R. Meyer)	—	113·0	—	—	—	—		—	—
,, Oats (Paul, 1921)	27·5	127·1	—	—	—	—		284·8	—

It will be seen that there is a somewhat unusual variation in the figures obtained for the oils from different sources, and the unsaponifiable matter in each case is notably higher than that of other oils, with the exception of shea butter. In the case of the oil extracted from rye flour, I was able to obtain a larger quantity (I extracted 45 grms. from 20 lbs. of flour), and consequently could obtain the following additional figures: Hehner value, 94·6; insoluble bromides (calculated upon the oil), 0·65 per cent. (equivalent to a linolenic acid content of 0·24 per cent.). Bolton and Williams' test, 84·7; Reichert Meissl value, 2·62; Polenske value, 0·41. There was not sufficient oil remaining for a test for lecithin content, although, as traces of phosphorus were found in the oil, this substance is probably present, as in the case of maize, wheat, and oat oils. The iodine value obtained by using the Wijs method for a period of one hour with rye oil, compares very well with that obtained by the bromine vapour method, and evidently there is no material quantity of acids present in this oil in which the unsaturated carbon atoms occur next to the carboxyl groups (*cf* ANALYST, 1929, **54**, 445).

The unsaponifiable portions from the first three oils were mixed. The mixture was a yellowish-brown, wax-like, definitely crystalline solid, with a melting point of 107° C. It was acetylated and recrystallised three times from alcohol and then melted at 126° C.; from this it would appear that phytosterol is present. Previously the unsaponifiable matter from each oil had been tested with the antimony trichloride reagent for the detection of vitamin *A*, and in each case a greenish coloration was obtained, instead of the deep blue colour presumed to be associated with vitamin *A* in this test.

A further sample of unsaponifiable matter from the oil of rye flour weighing about a gram was extracted, dried in carbon dioxide at 70° C., and sent to Professor Drummond, who had kindly offered to make biological tests on rats with it. He made a series of tests with this material for both vitamins *A* and *D*. In doses of one to ten mgrms. there were no signs of appreciable amounts of vitamin *A*, nor did the substance give any of the colour tests associated with this vitamin. Doses of five to ten mgrms. effected no apparent cure of experimental rickets in rats. From this valuable evidence it can be concluded that there is little or none of either vitamin present in rye oil.

It is interesting to compare the figures I have obtained with those of R. Meyer, and also with those for oat oil obtained by E. Paul (*loc. cit.*). Both rye and oat oils are semi-solid at 15° C., and each contains a large proportion of free fatty acids (oat oil having even a higher figure than rye oil.) Their iodine values are similar, and the only pronounced difference in the constants is the much higher proportion of unsaponifiable matter present in rye oil.

I had a sufficient quantity of the oil of *rye flour* to carry out a lead salt and alcohol separation, which gave a solid acid percentage of 17·1. (Meyer obtained an iodine value of 113 for the liquid fatty acids from his rye oil). An analysis of the various samples of rye, etc., in the usual manner applied to cereals, gave the results shown in the following table. It should be noted that these analyses are not really complete, as the carbohydrates were estimated by difference, but they are full enough for the present purpose.

Rye.	Oil. Per Cent.	Water. Per Cent.	Ash. Per Cent.	Proteins. Per Cent.	Fibre. Per Cent.	Carbohydrates (by difference). Per Cent.
Rye A	2·05	13·9	—	—	—	—
Rye B	1·80	12·80	2·56	8·56	1·33	72·95
Ryvita	0·83	9·33	1·90	9·09	1·32	77·53
Rye flour	0·97	14·87	0·50	6·15	Traces	77·51

SUMMARY AND CONCLUSIONS.—It will be gathered, from the foregoing figures, that rye oil can be classified as a semi-drying oil, although its consistence is more nearly solid than that of any other oil in this class, with the exception of oat oil.

Rye oil has an abnormally high unsaponifiable content. There is little or no vitaminic activity in rye oil. There are very little, if any, fatty acids present in rye oil in which the unsaturated carbon atoms occur next to the carboxyl group.

In conclusion, I would like to thank Professor Drummond for the biological tests he carried out for me, Professor Hilditch for extracting a quantity of rye, and Dr. Mitchell for his help and interest throughout the work, which was carried out almost entirely at the Technical College, East Ham.

DISCUSSION.

The PRESIDENT said he would like to thank Mr. Croxford for this accumulation of data.

Mr. BOLTON thought that the author had found a want and had supplied that want. One small point that occurred to him was that the iodine value of the rye

oil examined was so very low that he would like to have seen the oxidised acids determined. He suggested that, although the test which he and Mr. Williams had put forward a little time ago might possibly afford information, in spite of the fact that it had only been applied to oils with less than 2 per cent. unsaponifiable matter.

Dr. KENT-JONES said that supplies of suitable material for the preparation of rye oil might be obtained from rye mills, and he thought that it might be interesting to see the difference in ryes from different parts of the world. If rye milling were similar to flour milling, stocks of very nearly pure germ would probably be available, giving a high yield of oil.

Mr. A. T. BACHARACH said that since rye oil contained so high a proportion of unsaponifiable matter, it would probably be worth while to submit a sample to spectroscopic examination.

Mr. K. A. WILLIAMS pointed out that there was a distinct relationship between the specific gravity of most fatty oils and their other physical constants (*e.g.*, refractive index and viscosity) and between all these constants and the iodine value. That relationship seemed to be entirely absent in this oil. If the high specific gravity figures were substantiated by further examination of samples, he thought that the relationship between iodine value and specific gravity for rye oil should prove very valuable.

Scientific Evidence Relating to Firearms, with Special Reference to a Recent Murder Trial.

BY G. W. BAKER, F.I.C.

(*Read at the Meeting, October* 1, 1930.)

THE FACTS OF THE CRIME.—On August 26th of last year it was discovered that two families living in adjacent rooms in Jaffa had been murdered during the rioting on the evening of the previous day. The doors had been forced open, and one of these had dents upon it such as might have been made with the butt of a rifle.

It was obvious that firearms had been used by the assailants. Some twenty empty cartridge cases and numerous fragments of bullets from pistol ammunition were scattered on the floors. One empty 303 mark VII cartridge case was found in a pool of clotted blood in one of the rooms, and three pieces of rifle bullets were found in another room. The nose of a rifle bullet was extracted from one of the bodies during the post-mortem examination.

The investigating officer noted that this 303 case was polished bright, and that remnants of metal polish were present in the groove round the cap, and from this he reasoned that it had recently been in possession of a policeman. Acting

on this suspicion, he visited the neighbouring police post and questioned the two policemen on duty. One of these policemen stated that he had fired his rifle on the previous day and handed in six empty cases. The investigating officer then took possession of the two rifles, and later, after further evidence had been collected, the policeman who had handed in the empty cases was arrested on the charge of murder.

A piece of wood cut from the door, two rifles, fragments of bullets, the empty 303 cartridge case found on the scene and others handed in by the accused were submitted to me for examination some weeks after the committal of the crime, and my investigations then commenced, and were continued until the case came up for trial in February.

QUESTIONS FOR INVESTIGATION.—The points at issue were:—

(*a*) Could the dents on the door have been made with the butt of a rifle?

(*b*) Were the pieces of rifle bullets portions of British ammunition such as is used by the police?

(*c*) Had those bullets been fired from the accused's rifle?

(*d*) Had the 303 cartridge case been fired in the accused's rifle?

To elucidate these points, firing tests were carried out with the assistance of the police armourer, during which some 50 rounds of S.A.A. were fired from the accused's rifle and from others, and a careful systematic examination and comparison of bullets and cases was then begun.

In this investigation use was made of microscopical examination under different conditions of magnification and illumination, special attention being given to extractor marks and pin impressions on the cases, to lands and grooves on the bullets, and to the firing pins and extractors of the rifles. The internal structure of the bullets was examined by X-ray and compared with 32 different makes of police ammunition, while the composition of the bullet casings was tested by specific gravity determinations and chemical analysis, and compared with English, German and Turkish bullet casings. Wherever possible, microphotographs of significant features were taken as permanent records for production in Court.

CONCLUSIONS.—Briefly stated, the findings, which were supported by fourteen photographs, were as follows:—

(*a*) Careful measurements of the dents on the piece of wood from the door showed that at least one of these could have been made with the butt of a service rifle.

(*b*) The pieces of bullets were all portions of British ammunition, such as is used by the Police; and, furthermore, the peculiar internal structure of the piece found in one of the bodies limited it to one of three makes, one of which (R.17.W) was the make of the cartridge case found on the scene.

(*c*) There was not sufficient evidence to identify the bullets as having been fired from the accused's rifle, but they were fired either from that rifle or others of the same calibre and rifling.

(*d*) The cartridge case found on the scene was certainly fired in the accused's rifle. This was conclusively proved by the presence of at least 14 marks in the pin impression, all of which were found on the firing pin of the rifle. The extractor mark was also a very characteristic feature.

(*e*) It was also demonstrated, from the shape and position of the blood stain on the cartridge case, that it must have been on the floor when the pool of blood flowed up to it, and that it could not, therefore, have been dropped in the room some time after the crime had been committed.

It is now known that the expert engaged by the defence, who also examined the exhibits and who was shown all my findings, but was not called to give evidence, was in agreement on all points. Counsel for the defence tried to argue that, if many more rifles were examined, one other might be found having a pin and extractor producing exactly the same marks as those of the accused's rifle. Actually the chances are so remote as to constitute a virtual impossibility in this case.

The defence tried also to break the chain of evidence by questioning the proper custody of the exhibits and by suggesting that one of the bullets found during the post-mortem might never have been in the body, because it was actually found in a clot of blood which had detached itself from a wound. In this connection the production in court of official *pro forma* describing the exhibits and giving the date and hour of their receipt in the Government Laboratory was of considerable value.

The Court found the accused (with other persons unknown) to be guilty of murder.

THE SCIENTIFIC EVIDENCE.—Having now stated the main facts of the case, I think that the following further comments, with special reference to the photographs, may be of interest :

X-RAY PHOTOGRAPHY.—So far as I am aware, X-ray photography has not previously been used in the examination of bullets for forensic purposes. In this instance the peculiar nose filling of lead and fibre was readily detected by this means without mutilation of the exhibit (Fig. II. SM/1). Comparison with thirty-two different makes of 303 ammunition (see Fig. I.) helped to establish the fact that the piece of bullet found in one of the bodies was from British ammunition. It is understood that this nose-filling was introduced during the war as a substitute for aluminium, and that, later on, plain fibre was found to answer the purpose.

CARTRIDGE CASES.—The 303 cartridge case found on the scene of the crime was compared with six others handed in by the accused as having been fired by him, and with others fired by me in the accused's rifle and in other rifles.

Three characteristic features common to the exhibit and to all other cases fired

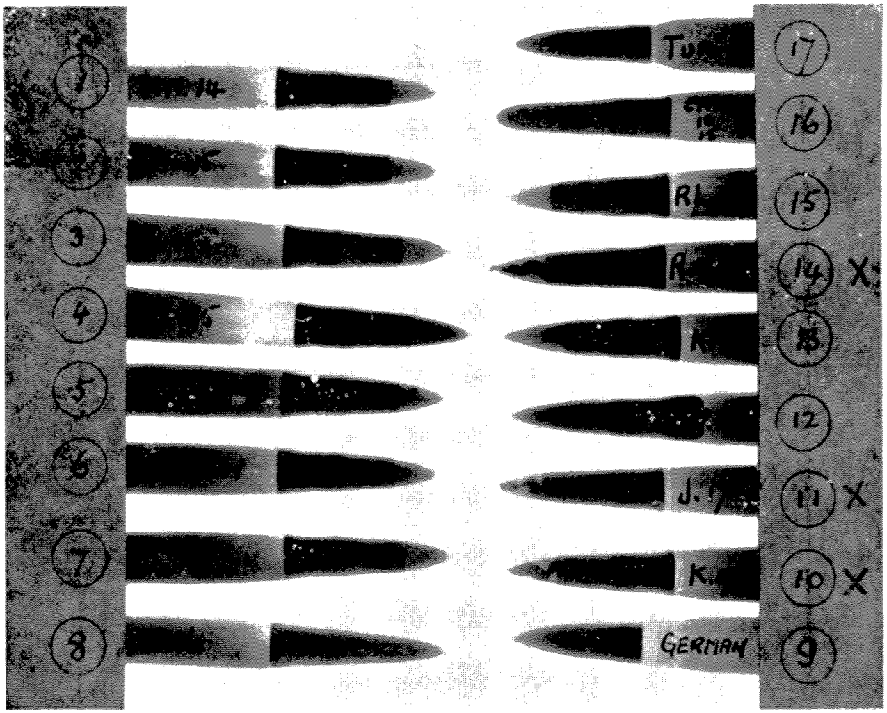


Fig. I. X-Ray Photographs of various makes of ammunition, about 2/3 natural size.

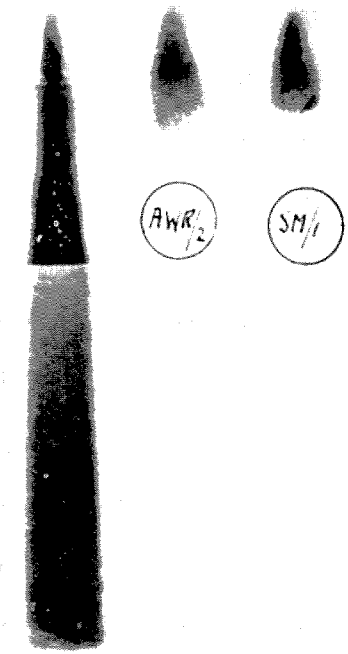
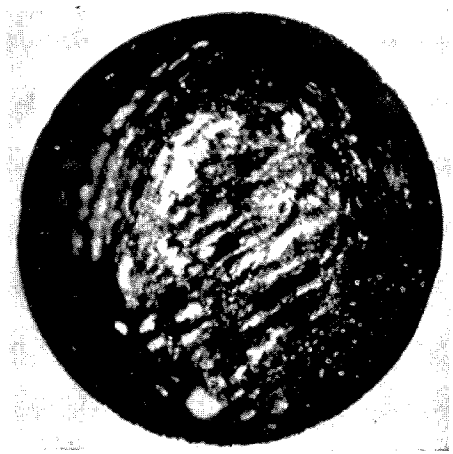


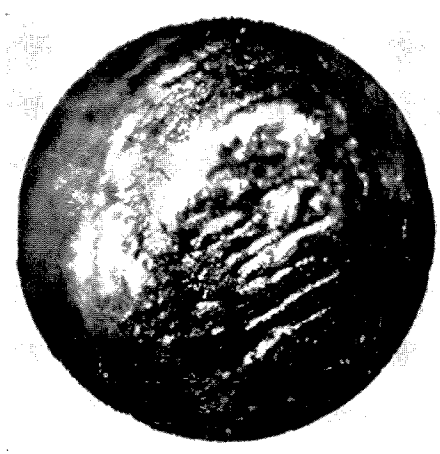
Fig. II. X-Ray Photographs of two bullets produced, and one round of R17W ammunition. Natural size.

Fig. III. Photomicrographs of Pin Impression on Cartridge Case and Firing Pin of Rifle — approx. 40 diameters.

(*a*) Key to pin impression, showing 14 marks.



(*b*) Pin impression on cartridge found on scene of crime.



(*c*) Firing pin of rifle of the ac

in the accused's rifle were demonstrated: (*a*) *The Bulge.*—A bulge on the side of the case diametrically opposite the extractor mark, and extending from the top to the solid portion some 5 mm. from the rim—due obviously to expansion of the case to fill the breach. For demonstrating this bulge a photograph was shown in which white strips represented the shadows of the cases when placed in front of the condenser of an enlarging lantern. This feature is of minor importance, and it was found that, measured with a micrometer gauge, the degree of bulge might vary when using the same rifle, and that with all the rifles tested it was, if present, diametrically opposite the extractor mark.

(*b*) *Extractor Marks.*—The second feature is an extractor mark characterised by a series of deep cuts with rounded ends parallel to the rim, which are produced when the bolt is pushed home and at right angles to these a series of long scratches produced on extraction. A section of these marks was shown photographically, and, if necessary, a series of such photographs showing the whole mark would have been prepared. There were, of course, considerable variations in the extractor mark on every case fired from the suspected rifle, but, in my opinion, there was sufficient evidence in this instance to identify the rifle by the extractor mark. Photograph 1 shows irregularities on the extractor of the rifle such as might produce the extractor mark.

(*c*) *Firing Pin Impression.*—The third feature is a characteristic pattern of at least fourteen marks at the bottom of the pin impression in the cap of the cartridge case. This was easier to demonstrate than the extractor mark, and also more convincing, as in the nature of things the firing pin of a rifle is the part most likely to leave a good impression, which is protected, to some extent, from damage by subsequent handling of the exhibit. The photographs in Fig. III. show pin impressions on the exhibited case and another fired in the accused's rifle, together with the firing pin of that rifle—proving conclusively that the cartridge case found on the scene of the crime was fired in the accused's rifle. In my control firing tests with twelve service rifles I noted that the depth of the pin impression was not always the same each time with any one rifle, and that there was a general tendency for the impression to be out of centre towards the extractor mark. The general shape of the impressions and their surface markings were, however, sufficiently characteristic to enable me to identify the firing pins of all the rifles. One photograph shows what appear to be file marks upon the pin of one of the rifles, and the impression produced on a cartridge case.

Questions were asked in Court as to whether the extractor marks could not have been produced by practice loading and unloading. Of all the marks, the pin impression is likely to be of most forensic value, because it can have been produced only by the act of firing.

The photomicrographs of the pin impressions were the most difficult to obtain. They were taken through a one-inch objective. The stage of the microscope was removed so that the cartridge case could be held vertically in a clamp and slowly rotated in a slanting beam of light. It seems probable that the use of a

microscope having a telescopic attachment would greatly facilitate this kind of work by giving greater depth of focus and plenty of working space for illumination. Some method of rotating the object with precision in any desired plane is very desirable.

The comparator eyepiece facilitated comparisons considerably, especially in the case of the extractor marks. Use was also found for the camera lucida and micrometer eyepiece. I have recently had an opportunity of seeing the equipment in use for such work at Woolwich Arsenal and intend to make more use of direct photography with a short focus lens, as advocated by Mr. Perry.

In the firing tests difficulty was experienced in recovering the bullets in a fit state for comparison. Finally, a special box was used for the purpose. This box is 6 feet long, 18 inches wide and 18 inches deep, and is divided into six equal compartments by removable cardboard partitions. The first two compartments I filled with cotton waste and the others with fine bran. The position of the bullet could be traced by the perforations in the partitions, and I found that at about 4 yards' range the rifle bullets came to rest in the fourth and fifth compartments with little distortion.

I am indebted to Mr. G. J. F. Millar, the Police Armourer, Palestine, for valued assistance in the control firing tests; and to Mr. J. Rock, of the *X*-ray Department, Government Hospital,. Jerusalem.

GOVERNMENT CENTRAL LABORATORIES,
JERUSALEM.

DISCUSSION.

Mr. G. H. PERRY (the War Department Chemist), in dealing with the scientific evidence, said that with regard to the problem of relating some of the component parts of a fired cartridge, the cartridge case and bullet, to the ammunition concerned and the weapon from which they had been fired, until recent years all that had been attempted was to identify the types of ammunition and weapon. This was done from the weight, dimensions, construction and chemical composition of the specimens, and the major markings, stamped letters and figures and rifling marks, on their surface. There were also possibilities that peculiarities of chemical composition might serve to establish relationship between these cartridge cases and bullets and a particular batch of ammunition.

Within the last few years, however, it had been possible to go further and to identify conclusively these cartridge components with the particular weapon used, by a process of comparison of minor surface markings on specimen cartridge components with those produced on components fired from the weapon in question. Direct comparison between the cartridge component and the weapon part was also possible in some instances.

The parts of a weapon were necessarily closely dimensioned and their surfaces frequently finished off by filing. They also suffered wear, and were sometimes damaged either mechanically or by rusting. When cartridges were fired the case was forced, by the very high pressures developed, against certain of these tool-marked surfaces of the weapon and the markings were faithfully reproduced on the soft metal of the case, particularly on the copper cap shell. Similarly, the

bullet "set-up" or expanded near its base into the barrel of the weapon and its surface was marked not only by the rifling lands and grooves, but also by the marks of the minutely-toothed edge of the bit used in rifling. Distortion and surface damage to the bullet on striking hard objects might obliterate some of these markings.

These secondary markings were frequently minute and only visible under considerable magnification with clear definition and very acute angle of lighting. The ordinary microscope, the stereoscopic microscope, microscope with telescope attachment, and particularly the comparator microscope, were all useful in the examination, but photographic enlargement under definite conditions and comparison of the prints had decided advantages. Super-imposition of a photographic negative on glass of a feature on the specimen on a positive on glass from the feature to be compared afforded incontrovertible evidence of coincidence. In view of the delicacy of some of these markings, it was of the greatest importance that every care should be taken to protect the specimens from damage, even by handling, after they are recovered.

Mr. Perry illustrated his remarks with specimens and photographs taken in connection with a case with which he had been connected. Some of these were similar in character to those shown by Mr. Baker, but additional features illustrated were file mark and mechanical damage reproductions on the cap shell of a fired case and the minor grooving-bit markings of a weapon reproduced on the surface of a lead-alloy bullet. The latter were shown to be very characteristic and capable of proving direct association with a particular weapon if the correspondence of the markings on a few consecutive facets of the bullets were considered.

Sir WILLIAM WILLCOX was sure that everyone would like to congratulate Mr. Baker on his admirable paper, and also Mr. Perry on the interesting account which he had given of another very important case. The work described at this meeting was quite new. Sir Bernard Spilsbury and he had made experiments on shot-guns and revolver wounds, but their experiments (done some 5 or 10 years ago) were really of quite a different nature—their object was to get evidence of the effect which revolver, rifle or shot-gun produced, and to compare this with the wounds produced on the skin and, if they were fortunate enough to find a bullet, to measure its diameter and to estimate the bore of the weapon from which it was fired. But that was as far as their experiments went. The work at present under discussion was quite new, and it was very refined work—analogous to finger-print detection which was used so largely to-day. This work, he continued, was based on actual experiment, and that was the only type of evidence of value. Nowadays, it was necessary to have actual experimental proof confirming anything said in the witness box. He was sure that all had admired the thought and skill with which these experiments and observations had been carried out. This case and that in which Mr. Perry was concerned were most interesting, and they would be most valuable in indicating new methods for the elucidation of truth and the detection of crime. There was a third case in which very interesting experiments of a similar nature had been carried out—that of the murder of the Sirdar in Egypt.

Sir BERNARD SPILSBURY said there was little of value he could add to what Sir William Willcox had said, but he would like again to emphasise the interest and importance of the investigation. One point in the Jaffa case had puzzled him very much—bullets were referred to as "lying about in fragments"; how had they been so smashed up? Was it suggested that they had struck the walls or floors after passing through the bodies? Another question was the value of the bullets

themselves in cases of crime, and what should be done with bullets to preserve their usefulness in this connection. To what extent could bullets be employed after, for instance, they had passed through such substances as bone, walls or wood? To what extent were the markings modified or lost by subsequent passage through these objects? He gathered that the cases were more important than the bullets themselves, but that the character of the bullet made a difference.

Mr. PERRY, replying (on behalf of the author), stated with regard to the Jaffa case that, as he had said, this particular make of bullet, particularly with the fibre end, did break at that position very readily, and when the velocity was taken into consideration (2,500 feet per second) it was not surprising that the bullets were smashed to fragments. It was a small room and the bullets must have hit the walls or floor after passing through the bodies. One very rarely received a bullet which would show one very much, but if one could only get two or three undamaged consecutive facets, they should give sound evidence ; the minor markings on hard- surfaced bullets were not interfered with so much as one might expect by passage through hard substances. He had heard that the United States Bureau of Standards were doing work on this particular subject and had promised a publication, which he was awaiting with interest.

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.

THE USE OF ULTRA-VIOLET LIGHT FOR THE DETECTION OF SOLVENT-EXTRACTED COCOA BUTTER.

FOLLOWING an article in the *Gordian*, abstracts have appeared from time to time in the technical literature on the use of ultra-violet light for the distinction of solvent-extracted from mechanically expressed cocoa butters.

For the examination of this method a Kelvin, Bottomley and Baird fluorescence cabinet has been used. With the solid or liquid butter the colour interferes with the observation by preventing the incident rays from penetrating. Examination of a solution in a non-fluorescent solvent, as recommended by Dr. Fincke (*Die Kakaobutter und ihre Verfälschungen*), is, therefore, much to be preferred. Of the solvents usually available, redistilled low-boiling petroleum spirit, although not quite optically inert, was the best.

METHOD.—The fat under examination was melted by gentle heating, and 0·1 grm. weighed directly into a thin-walled test tube. (N.B.—Some thick-walled tubes showed a strong greenish fluorescence.) Ten c.c. of petroleum spirit were then added; the melted fat dissolved almost immediately, giving a 1 per cent. solution. The tubes were supported under the lamp in an ordinary test-tube rack fairly close to the lamp. As the illumination does not strike uniformly, it is necessary to move the tubes about under the lamp.

FINDINGS.—The butter expressed by mechanical means or solvent-extracted by petroleum spirit from raw beans was only very slightly fluorescent. Commercial cocoa butters, presumably genuine, were more fluorescent and showed some variation with different manufacturers—some known to be mechanically expressed and of irreproachable quality had a distinct fluorescence, whilst others of equal quality were but slightly fluorescent. A special doubly-refined extracted butter, which as regards taste and odour was the best extracted butter available, gave an intense fluorescence. Admixture of this butter with raw butter, *i.e.* from raw beans, in as small an amount as 5 or 10 per cent. could be detected on direct comparison. The fluorescence of some good commercial butters was, however, equivalent to about 10 per cent. of added extracted fat, and thus one could not detect with any certainty less than, say, 25 per cent. of extracted butter. Such a percentage of any extracted fat examined in this Laboratory would be detected by taste, and the test, therefore, fails where it is most needed.

The fluorescence exhibited by different butters may differ in quality, as well as in intensity, so that solutions cannot be diluted to match a standard fluorescence and the dilution used as a comparative measure of fluorescence.

THE FLUORESCENCE OF MECHANICALLY EXPRESSED BUTTERS.—An exploration of the whole process of butter expression during cocoa manufacture showed that the increase in fluorescence, as compared with that of raw butter, takes place practically entirely during the roasting of the bean, and is not due to accidental contamination during manufacture. Roasting being essentially a heating process, the effect of heat on raw butter was next examined. A temperature of 150° C. for about 1 hour gave a fluorescence similar to that shown by the butter from roasted beans, whilst at 100° C. no appreciable change was observed. It is suggested that this increased fluorescence is due to slight oxidation, although it must not be overlooked that roasting may merely concentrate the fluorescent factor by driving off a volatile non-fluorescent portion.

To summarise the results of the examination of a number of cocoa butters, it may be stated that:

(1) A non-fluorescent butter is almost certainly not solvent extracted, and is obtained from very low roast beans.

(2) A strongly fluorescent butter is either solvent-extracted or contains mineral oil from some other source. (It is well known that mere traces of mineral oil, such as may occur in cocoa butter pressed from factory wastes, give rise to a strong fluorescence.)

(3) With regard to a slightly fluorescent butter, no definite conclusions can be drawn. Observation of the fluorescence can only be used to support evidence from other sources.

Although investigation does not support the extravagant claims made in some quarters for the fluorescent method of detection of solvent-extracted butter, the test is a useful addition to the methods available for the examination of suspected butters.

In conclusion, I wish to thank Messrs. Cadbury Bros., Ltd., for permission to publish the results of this work carried out in their Laboratory, and Mr. A. W. Knapp for advice during the progress of the investigation.

W. T. FIELD.

RESEARCH LABORATORY,
BOURNVILLE.

OILS UNDER ULTRA-VIOLET LIGHT.

THE observations recorded in the abstract on p. 773 are substantially correct; but, unfortunately, the use of the ultra-violet lamp, for the purposes named, has proved, in practice, liable to lead to inaccurate conclusions. In the first place, a number of oils used for the adulteration of olive oil—notably tea-seed oil—may give a similar fluorescence to virgin olive oil. In the second place, certain modern methods of refining include treatments that can remove from the refined oils the particular characteristics referred to in the paper; and the presence of refined oils is, therefore, not detected. Hence, while one is left with observations that are interesting from the scientific point of view, they have very little diagnostic value; and no analyst can assert with any certainty either that an olive oil is a virgin oil, or that it is free from refined or foreign oils, as a result of observations under ultra-violet light.

E. R. BOLTON.

INKS AND ULTRA-VIOLET LIGHT.

ULTRA-VIOLET light is particularly useful for detecting erasures of lead pencil writing, the residual particles of graphite, more or less buried in the fibres of the paper, reflecting the rays and becoming visible. It has also been claimed that it is possible, by the use of ultra-violet light, to differentiate between different inks in writing and even to distinguish between inks of different age.

With the object of testing these claims I have made a series of tests with the Hanovia lamp upon writing done with the various constituents of inks, with inks of known composition, and with certain commercial inks.

It is well known (see ANALYST, 1925, **50**, 641; 1926, **51**, 54, 481) that extracts of various tannin-containing substances differ in their appearance beneath the ultra-violet rays, and I have found that this is also true of their behaviour when applied in dilute solutions to paper and allowed to dry. Under these conditions, gall-extract showed a pale violet-brown fluorescence, myrobalans a bright yellow; and writing, in pyrogallol solution, which was barely visible in ordinary light, fluoresced dark brown when brought beneath the lamp.

When, however, these three extracts were made into inks by the addition of ferrous sulphate they produced writings which could not be differentiated by ultra-violet light.

An ink made experimentally from tannin, gallic acid and copperas, with the addition of aniline (soluble blue) dye, was also indistinguishable in writing, when examined by the lamp, from the gall or myrobalans ink without the dye.

In some cases, inks which, on paper, were manifestly different to the eye, could not be distinguished from one another in ultra-violet light. For example, an ink containing aniline black (nigrosine) and finely divided carbon, looked like a blue black (Swan) ink, although in ordinary light the two were obviously different. Winsor and Newton's Artists' Black Ink showed no difference from Stephens' blue-black ink beneath the lamp. Various blue-black inks (Field's, Stephens', Swan) could not be distinguished from one another beneath the rays; but an American aniline ink, containing a dye agreeing in its reactions with anthracene chrome black, showed a yellow-brown fluorescence in ultra-violet light. It could, however, also be readily distinguished from ordinary blue-black inks in ordinary light.

Having regard to these results, it did not seem probable that ultra-violet light would be of much use for distinguishing between writing of different ages in the same kind of ink, and experiments showed this to be the fact. A blue-black ink made on October 28th, 1929, and oxidised by exposure to the air, appeared identical with ink (Swan) made on April 4, 1929, but not oxidised, and with freshly-made ink of the same manufacture (Swan). Similar negative results were obtained in the comparison of writing in blue-black ink of the same manufacture over a period of years. Even when differences could be observed in ordinary light, they disappeared beneath the ultra-violet rays. Hence, statements about the differentiation of ink in writing by means of ultra-violet light should be critically examined before being accepted.

I am indebted to the British Hanovia Lamp Co., Ltd., for their courtesy in placing one of their lamps at my disposal for the purpose of these experiments.

C. AINSWORTH MITCHELL.

THE DETECTION OF VISCOGEN IN CREAM.

THE detection of viscogen (calcium saccharate) in cream presents some difficulties. The usual methods depend upon the recognition of cane sugar, and the presence of an abnormally high proportion of calcium in the ash. As the addition of cane sugar to cream is legal, the burden of proof of viscogen thickening is really thrown on some form of ash analysis. This is troublesome and, at times, difficult to interpret, for the amounts of lime in the form of viscogen which are capable of thickening cream need not exceed about 0·04 per cent. of calcium oxide, and, therefore, may not bring the calcium content of the cream or ash beyond the values sometimes found in normal samples.

Viscogen has, of course, other obvious chemical effects besides its influence on the composition of milk ash. For instance, it is strongly alkaline; and as it is usually added in amounts sufficient to neutralise from one-half to two-thirds of the titratable acidity, it will cause the cream to display an unusually low acidity and an increased *p*H value. The second effect is probably the more important, from the point of view of detecting viscogen, as the initial *p*H value of a normal milk or cream is much more constant than its titratable acidity. Bromphenol blue is a suitable indicator to use; its colour in fresh cream is yellow, and varies from yellowish-green to greenish-blue, or even blue, in cream to which viscogen has been added, according to the quantity present. Taken in conjunction with a positive cane sugar reaction, this test is useful. If, however, the cream has undergone a slight subsequent acidification, through natural souring or otherwise, *p*H determinations may give little information.

It seems probable that in many cases tests for viscogen, based on the physical and chemical changes it produces in milk or cream, would be more useful than those just discussed. I have shown (*J. Agric. Sci.*, 1929) that viscogen owes its thickening properties mainly to the interaction of its lime with the soluble milk phosphates, from which there is formed a gelatinous precipitate of tricalcic phosphate, which carries down a certain amount of casein. It is not very easy to demonstrate the existence of this precipitate in cream by staining methods, but its presence may be shown indirectly. Thus, the addition of a soluble oxalate to milk or cream, previously treated with viscogen, is found to lead to the conversion of the gelatinous calcium phosphate into crystalline calcium oxalate, and to bring about a re-solution, presumably as alkali caseinate, of the co-precipitated casein.

That is, the thickening action of viscogen can be largely reversed by the subsequent addition of a soluble oxalate. This suggests a means of examining a sample of cream suspected to contain viscogen by comparing its viscosities before and after addition of, say, potassium oxalate. I have examined in this way a number of samples of pasteurised cream, to which viscogen had been added, and have obtained very promising results. The following typical values were obtained with a sample of pasteurised cream containing 50 per cent. of fat:

Cream. c.c.	2 *N* Viscogen. c.c.	Water. c.c.	Saturated neutral potassium oxalate. c.c.	Relative viscosity. Seconds.
40	0·175	1	—	41
40	0·175	—	1	18
40	—	1	—	15·5
40	—	—	1	17·5

The first two lines illustrate the considerable reduction in the viscosity of a viscogen-thickened cream brought about by the addition of oxalate, and the next two show the influence of oxalate on unthickened cream. The slight increase in viscosity found here is no doubt due to the partial conversion of colloidal calcium caseinate into the more viscous alkali caseinate. Since the effect of adding oxalate is, in general, to increase slightly the viscosity of unthickened cream, it will probably be safe to assume that a cream which, on addition of oxalate in the proportion used above, shows any decided fall in viscosity has been thickened by viscogen. This should, of course, be confirmed by a test for cane sugar. The viscosity of the oxalated sample must naturally be compared with that of one in which the oxalate solution is replaced by an equal amount of water.

A few precautions must be observed in order that the viscosities may bear a proper interpretation. The cream examined must be free from lumpiness, air bubbles, etc., and should, if any doubt exists about this, be warmed to 30°–35° C., mixed thoroughly without incorporating any air, and cooled to ordinary temperature before making the determinations. Further, as the viscosity of a rich cream is very sensitive to slight variations in fat content, it is essential that the oxalate solution and water are thoroughly mixed in. This may be ensured by tinting these liquids with some highly coloured substance like methylene blue. Another and a less obvious precaution is to see that the two samples compared have somewhat similar *p*H values. The addition of oxalate to a milk or cream increases its alkalinity, and in markedly alkaline solutions (*p*H > 8·5) the viscosity of casein is considerably altered. To ensure that the test is not complicated by this factor, it is advisable to add first of all a little phenolphthalein to the suspected cream. Two equal samples are then weighed out, and to one is added one-fortieth of its volume of saturated potassium oxalate. If it now turns pink (as usually happens if much viscogen is present), a few drops of a roughly *N* acetic acid solution (containing enough sodium acetate to reduce its hydrogen ion concentration to about *p*H 5) should be added until the pink colour disappears. A volume of water equal to the sum of the volume of oxalate and acetic acid mixture is then added to the other sample. After an interval of about 15 minutes, to allow the oxalate to act completely, the samples are ready for the viscosity measurements.

For these determinations a simple type of capillary viscometer is sufficiently accurate. This may be constructed from any ordinary, say 20 c.c., pipette, which has been cut off a little below the bulb, and to which the appropriate one of a series of capillary tubes of various diameters can be closely attached by rubber tubing.

With this simple apparatus, and the precaution mentioned before, I have found the method to work very satisfactorily on any sample examined up to the present.

It is necessary to point out, however, that this test cannot be used to prove definitely the presence of viscogen in a sample of cream; it will show the existence of a thickening produced by viscogen, and the two are not the same. Viscogen is a very specific thickener, depending mainly for its efficacy on the presence of soluble phosphates and of protein, such as calcium caseinate, which are readily carried down by freshly precipitated calcium phosphate. Samples of milk and cream occur occasionally in which the soluble phosphates content is so low that only very slight precipitation follows the addition of viscogen. In these cases viscogen may have little or no thickening power. Such samples will, however, probably be rarely met with among commercial creams from mixed herds.

DEPARTMENT OF DAIRY CHEMISTRY,
UNIVERSITY COLLEGE, CORK.

G. T. PYNE.

ABNORMAL SWEETENED CONDENSED MILK.

IN view of the recent attention drawn to the possibility of the presence of invert sugar in sweetened condensed milk by Dr. Monier-Williams (*Reports on Public Health and Medical Subjects*, No. 57; ANALYST, 1930, 573) the following details of samples examined by us may not be without interest.

Sample No. 1 was submitted for analysis in September, 1929. Upon opening the tin there was no evidence of gas-formation, but a little mould was observed on the under-surface of the top of the tin. The milk was of an unusually thick and gelatinous consistence, the appearance being quite sufficient to suggest that the sample was in an abnormal condition. Some of the milk was taken immediately under sterile conditions for a bacteriological examination. The remainder was transferred to a glass jar, when it was noticed that a number of reddish button-like masses were present in the milk. The chemical and bacteriological results are set out below:—

CHEMICAL ANALYSIS.

	No. 1. Per Cent.	No. 2. Per Cent.
Total solids	72·80	73·20
Milk-fat	0·59	0·51
Proteins ($N \times 6{\cdot}38$)	9·54	9·38
Reducing sugars (calculated as hydrated lactose)	18·47	20·06
Cane sugar	41·15	40·72
Mineral matter (ash)	2·42	2·41
Sugar reducing Barfoed's solution calculated as invert sugar	2·5	3·9
Acidity as lactic acid	0·30	0·32

BACTERIOLOGICAL EXAMINATION.

No. 1.

Total organisms growing at 37° C. agar aerobic	$<$	5,000 per grm. (chiefly micrococci)
Total organisms growing at 37° C. agar anaerobic	$<$	3,000 ,, ,,
Total organisms growing at 20° C. gelatin		
(*a*) moulds	$>$	200,000 ,, ,,
(*b*) other organisms	$<$	30,000 ,, ,,
Total anaerobic spores	$<$	100 ,, ,,
Cane sugar fermenters	$<$	1,000 ,, ,,

B. coli absent from 1 grm.
B. Welchii absent from 1 grm.

The chemical analysis shows that Sample No. 1 closely resembled that examined by Dr. Monier-Williams, though the amount of fat present is somewhat high for a machine-skimmed condensed milk. Unfortunately a polarimeter was not available to confirm the amount of invert sugar found by reduction of Barfoed's solution, for which determination control tests were made by using a normal sample of the same brand to which invert sugar had been added. There was no excess of acidity in the sample.

The bacteriological and mycological examination showed the presence of moulds of the *Aspergillus* and *Penicillium* species in large numbers. Yeasts and gas-forming bacilli were rare, and there was no abnormal growth of other organisms.

Sample No. 2 was examined in November, 1929. This milk appeared to be in much the same condition as No. 1; moulds and "buttons" were present, and the sample had a similar thick gelatinous appearance. No bacteriological examination was made, but the chemical analysis showed that approximately 3·9 per cent. of invert sugar was present.

The more frequent changes recorded as occurring in sweetened condensed milk due to the growth of micro-organisms are: (1) Gas-formation usually due to the development of yeasts, (2) thickening, and (3) formation of "buttons." We are concerned here with the last two changes.

Whilst thickening may be only a physical change, produced by keeping condensed milk for a long time or at too high a temperature, Rice and Downs (*J. Dairy Science*, **1923, 6**, 532) have shown that in some instances it is due to the growth of a micrococcus capable also of inverting sucrose, and which is particularly active at temperatures above normal room temperature.

Rogers, Wahlberg and Evans (*J. Dairy Science*, **1920, 3, 122**) have studied the cause of "buttons" in condensed milk. These are reddish-brown lumps of curd of a firm and cheesy consistency, and were found to be due to *Aspergillus repens* and other moulds. The growth of the moulds ceases as soon as the oxygen present in the tin has been used up, but the harmful effects can continue, since the proteolytic enzymes present in the moulds may digest the mycelium itself and then act on the surrounding medium. These investigators found that buttons did not develop in milk kept at 20° C., and hence their formation can be prevented either by excluding oxygen from the tin or by storing in a cool place.

Moulds of the *Aspergillus* genus possess strong inverting power which they are able to exert in highly concentrated sugar solutions of varying reaction. *Aspergillus repens* has been found to be the most active of the moulds causing deterioration of raw sugars (Owen, *Inter. Sugar J.*, **1923, 25, 371**).

The results of the bacteriological and mycological examination appear, therefore, to indicate that in these two instances the physical and chemical changes in the milk have been brought about by the growth of moulds induced by storage at too high a temperature.

Studies by Thom and Ayers (*J. Agric. Res.*, **1916, 6, 153**) have shown that practically all mould spores in milk are destroyed by heating at 63° C. for 30 minutes. The entire process of making condensed milk gives a very thorough heating of the milk, presumably much greater than that which is necessary for destruction of all mould spores. It is, therefore, highly probable that the moulds gained access after the processes of pasteurisation, admixture with boiled cane sugar and evaporation *in vacuo*.

Whilst, as a rule, care is taken by shippers to avoid perishable articles being unduly heated during transit, enquiry shows that, occasionally, consignments of

condensed milk are to be found in close proximity to the boiler room, and the temperature may be sufficient to promote active growth of moulds in milk infected with their spores.

Although the milk may be unsaleable, these moulds or their products are not likely to be injurious to health, and the samples were therefore returned as of inferior quality.

S. DIXON.
J. H. SUGDEN.

INSTITUTE OF PREVENTIVE MEDICINE,
CARDIFF.

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 OF LEEDS.

ANNUAL REPORT OF THE CITY ANALYST FOR 1929.

THE work commenced in the Municipal Laboratory, in June, 1928, which marked the advent of a whole-time Analyst, has been continued with success. In the year 1929, analyses have been made, in all, for eight Corporation Departments, *viz.* Public Health, City Coroner's, City Engineer's, City Police, Waterworks, Sewerage, Highways and Cleansing.

The actual number of samples analysed in 1929 was 2812, of which 1962 were food and drugs.

MILK.—Of the 1424 samples (1393 formal), 226 were below standard. Although it is the practice of the great majority of farmers to market the mixed product of the whole herd, certain farmers producing Grade A milk have been found to bottle the milk of individual cows, with the result that in one instance a fat deficiency of 19 per cent. was traced to this method of procedure.

FOIL-WRAPPED CHEESE.—Both tin and antimony were found in five samples of Gruyère cheese wrapped in foil containing 96·8 per cent. of tin and 3·2 per cent. of antimony. Twelve other samples of different types (Cheddar, Cheshire, Gruyère) were examined, and all the four Gruyère cheeses showed discoloration. In one of them, wrapped in pure tinfoil, the tin amounted to 1·12 grains per lb. (See ANALYST, 1930, 191.)

POTTED MEAT.—Three of the 17 samples were adulterated, one containing 0·35 per cent. of boric acid. The vendor was summoned, but the case was dismissed on the technical point that the preservative had not been knowingly and wilfully added. The other two samples contained 4·2 and 4·6 per cent. of starch. Such products should be sold, not as "potted meat," but as "meat paste."

POTTED SALMON.—One sample was found to contain 12 per cent. of starch; it should have been sold as "salmon paste."

SWEET SPIRIT OF NITRE.—Of 5 samples analysed, 3 were 10 per cent., 19·7 per cent., and 100 per cent. deficient, respectively, in the minimum amount (1·52

per cent.) of ethyl nitrite required by the British Pharmacopoeia. The sample in which ethyl nitrite was entirely absent was an imitation product consisting of ammonium acetate, sugar, alcohol, and water. The vendors were all warned by letter from the Medical Officer of Health.

C. H. MANLEY.

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 BISMUTH TABLETS: A QUESTION OF WARRANTY.

A FORMAL sample of bismuth tablets was recently certified "not genuine." The tablets were inferior in quality generally, but in particular were deficient to the extent of 32 per cent. of the amount of bismuth compound claimed to be present. Proceedings were taken against the retailer, who gave notice of his intention to plead warranty. At the hearing the warranty and other conditions were proved and the case dismissed.

It was, however, found impossible to proceed against the warrantor for false warranty, as such proceedings are subject to a time limitation of six months, imposed by the Summary Jurisdiction Act, 1848, and in this case the warranty had been given and dated more than six months previous to the date of the original proceedings.

This seems to be the first case of the kind in this area (Leicester), and it exposes the anomaly that the Food and Drugs (Adulteration) Act protects the warrantee, and the 1848 Act protects the warrantor, but the public is entirely without protection where stock is more than six months old, if such stock was originally supplied under warranty.

F. C. BULLOCK.

LINIMENT OF TURPENTINE.

ON October 14, a firm of druggists was summoned, at Old Street Police Court, for having sold liniment of turpentine deficient to the extent of 50 per cent. in camphor and turpentine.

The certificate of the Public Analyst (Mr. A. E. Parkes) stated that the sample contained "soft soap 8·5 grms. per cent.; camphor and turpentine, 35 c.c. per cent.; water, 56·5 c.c. per cent. The combined camphor and turpentine in the said sample were deficient to the extent of 50 per cent. Turpentine liniment, according to the British Pharmacopoeia, should contain not less than 70 per cent. of combined camphor and turpentine."

Mr. Glyn-Jones, for the defence, at the outset took exception to the form of the certificate on the ground that it did not distinguish between the camphor and the turpentine, either or both of which might have been deficient.

Mr. Jenkins, for the Bethnal Green Borough Council, asked for an adjournment in order that the Public Analyst might be called.

Mr. Glyn-Jones would not agree to this, and the Magistrate (Mr. Clarke Hall) suggested that the prosecution should be withdrawn.

The case was accordingly dismissed.

DERMATITIS FROM A HAT LINING.

ON July 24, at the Middlesex Guildhall, a radiologist claimed damages for injury received through poisoning by a dyestuff in the lining of a hat. Evidence was given that after wearing the hat he had developed a rash upon the forehead, which was diagnosed as dermatitis, attributable to the defective dyeing of the hat lining. For three months the plaintiff had suffered a loss of professional reputation, since doctors and dentists were unwilling to send patients to him.

The defendants, who had supplied the hat, admitted liability for the plaintiff's temporary discomfort, but denied that he had suffered professional loss.

The Court awarded £10 damages, including £1 for special damages.

ALLEGED MENTHOL DERMATITIS.

ON October 24, a claim was made at the Marylebone County Court for injury alleged to have been caused by the use of an excessive amount of menthol in a shampoo.

The plaintiff, a hairdresser's assistant, said that for three years, prior to June, 1928, he had used shampoos without any ill effect. The defendant (the hairdresser) had then bought certain shampoos to which he added crystals of menthol, whenever a customer asked for a cooler shampoo.

Medical evidence for the plaintiff was to the effect that the complaint had been diagnosed as dermatitis produced by menthol, which ought not to be added indiscriminately to a shampoo.

Dr. Jane Scobell, physician at the London Skin Hospital, said that she had never heard of dermatitis from menthol prior to this case.

Chemical evidence was also called for the defence to prove that the menthol used was of good quality and answered to the requirements of the British Pharmacopoeia. As much as 15 per cent. of menthol was used in some of the B.P. ointments and liniments. The amount of menthol used in this case was about 0·5 grm. per pint bottle. Witness admitted that the alcohol in the shampoo would remove fat from the skin, and that the menthol would then come into closer contact and be more potent.

Judge Snagge said that he sympathised with the sufferings of the plaintiff, but that, so far as the common law action was concerned, judgment must be given for the defendants with costs.

Straits Settlements.

REPORT OF THE GOVERNMENT ANALYST FOR THE YEAR 1929.

THERE are two branches of the Government Analysts' Department, one with three laboratories at Singapore, and one, a single laboratory, in Penang. The staff comprises the Government Analyst (Mr. J. C. Cowap), four European chemists in Singapore, and one in Penang, together with locally trained Asiatic assistants.

During the year alteration was made in the Schedule of the Deleterious Drugs Ordinance (to bring this into line with the Home provisions) whereby the esters of morphine and other similar habit-forming drugs were brought under control.

Additions were also made to the Schedule of the Poisons Ordinance. The necessity for this arose from a proposed local trade effort to popularise the use of drugs which were considered undesirable, and resulted in the classification of the compounds of barbituric acid such as veronal, together with urethanes, etc., as poisons. Rules under the same Ordinance were made for the storage and sale of ethyl petrol.

The number of samples examined was 14,406, as against 12,870 in 1928.

TOXICOLOGICAL.—Of the 55 specimens examined, no less than 40 were found to contain poison. The poisons included opium, caustic soda, formaldehyde, metacetaldehyde, aconite, tuba root, cresol and alcohol. Thirty-nine specimens of urine were examined for lead, mainly in connection with an investigation into the incidence of lead poisoning in local printing offices. All these were cases where a medical diagnosis of lead poisoning had been made, and chemical confirmation was obtained in nearly all the specimens.

DELETERIOUS DRUGS.—The majority of cases, apart from seizures of pure drugs by the Monopolies Department, concern anti-opium preparations containing opium or morphine itself. These appear to be imported, but are occasionally local pharmaceutical products. A disconcerting feature sometimes associated with the latter is that definite brands are found at one time to be innocuous and at another time to contain morphine. In 7 of the 74 cases, novocaine was found; this is not scheduled as a deleterious drug, but is included in the Poisons Schedule.

COUNTERFEIT COINS.—Six lots of exhibits in counterfeit coining cases were received. In two of these complete coining outfits were seized, and in each case the coins were made from a tin-lead alloy. In all the cases where no apparatus for coining was found the coins were made of brass coated with silver, and were remarkably good imitations of genuine currency. They appeared to be imported.

ASPHYXIATION BY WET RUBBER.—At the request of the Master Attendant an investigation was undertaken into the circumstances in which a Revenue officer was asphyxiated while searching a ship. It was demonstrated that the wet native rubber which was contained in the hold was capable of rendering air irrespirable by the substitution of carbon dioxide for oxygen.

Hong Kong.

REPORT OF THE GOVERNMENT ANALYST FOR THE YEAR 1929.

THE Government Analyst (Mr. E. R. Dovey) reports that 3710 analyses were made during the year, as compared with 3060 in 1928. The work included the examination of food and drugs, waters, dangerous goods, building materials, and samples submitted under the Pharmacy Ordinance.

TOXICOLOGICAL WORK.—In 26 of the 46 cases, evidence of poisoning was obtained, opium being found in eleven cases, animal toxins in five, and alcohol and acetaldehyde in six.

Gelsemium poisoning.—In August, the viscera from a young Chinese woman were received from the Kowloon mortuary. The woman was supposed to have committed suicide by taking some Chinese herbs. The alkaloid gelsemidine, the active principle of *Gelsemium elegans*, was isolated from the stomach contents,

from the walls of the stomach itself, and from the liver. The total quantity isolated was 1·09 grains.

CRIMINOLOGICAL WORK.—Two pieces of cotton tape were submitted by the police. They had been removed from a body, and it was suspected that they had previously been used as part of a driving belt on a pulley in a barber's shop. Chemical examination showed the presence of oily stains, which tended to confirm the suspicion.

In January, a cotton jacket worn by a man, on whose back corrosive fluid had been thrown, was examined. The stains were found to contain nitric acid.

In December, a passport was examined, the presence of erasures was proved, and an opinion as to the nature of the writing previously present was given.

DETECTION OF SEA-WATER IN DROWNING CASES.—During the year there have been several cases in which samples of the stomach contents, of the blood, or of fluid from the thorax, have been submitted. The total chlorides have been determined by both Whitehorn's and Smirk's method, but chief reliance has been placed on a method, worked out in the laboratory, of determining admixture of seawater by means of the electrical conductivity. Osmotic requirements of the body necessitate the maintenance of the concentration of electrolytes within comparatively narrow limits, and variations in most of the organic constituents of bodily fluids have no effect on the conductivity. The admixture of 5 per cent. of sea-water with blood will give an increase in the conductivity figure of about 2000 reciprocal megohms.

A modification of the ortho-tolidine method is being worked out for the determination of total chlorine in a single drop of blood. In cases of supposed drowning, where it is of great importance to know the relative concentrations of chlorine in the right and left heart, such a method would be invaluable.

ACID FOR SUBMARINE BATTERIES.—A certain amount of work was also carried out on the determination of minute amounts of chlorine in acid for the batteries of submarines. Specifications for such acid require the chlorine percentage to be not greater than 0·0005 per cent. Accurate determinations of such amounts are not easy without special apparatus and exceptionally pure reagents.

Department of Scientific and Industrial Research.

THE INVESTIGATION OF ATMOSPHERIC POLLUTION.*

REPORT ON OBSERVATIONS IN THE YEAR ENDING 31ST MARCH, 1929. (FIFTEENTH REPORT).

IN addition to the actual data collected during the year 1928–1929, the report of the Superintendent of Observations thereon, and an account of the work of the Research Committee, the present volume contains the report of the Standing Conference of Co-operating Bodies set up to maintain contact between the central organisation and local authorities or other bodies giving active or financial assistance. The general arrangement follows that adopted for the first time in the fourteenth report (ANALYST, 1930, **55**, 450).

* Published 28th Oct. 1930, pp. 64. Obtainable from H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 3s 6d. net.

ATMOSPHERIC POLLUTION RESEARCH COMMITTEE.—Investigations of the effect on the deposits obtained of differences of exposure of the standard deposit gauge have been continued, and certain defects in existing gauges, notably improper connection between the gauge and collecting bottle, have been remedied. Investigations, made by arrangement with the Government Chemist, showed that sulphur trioxide is not a normal constituent of the air (at Holborn), and appears only during fog. The National Physical Laboratory has designed an experimental apparatus for measuring daylight, which overcomes the difficulty of variation in the proportions of light of different wave-lengths on different days by use of two definite wave-lengths. The Laboratory has also tested 2 methods of determination of water in fog:—(*a*) The foggy air is warmed to evaporate the water, which is determined continuously by means of a wet and dry bulb hygrometer (Kohler). (*b*) Foggy air and filtered air are placed in separate vessels connected by a manometer and immersed in a water-bath, when the difference in pressure is due to evaporation of the liquid particles (Owen).

SUPERINTENDENT OF OBSERVATIONS.—During the year, 81 deposit gauges and 12 automatic filters were in operation (maintained by 33 and 7 authorities, respectively), including 6 new gauges.

The respective maximum and minimum mean monthly deposits in metric tons per 100 sq. km. were:—*Tar*: Newcastle, City Road 103, Huddersfield, Cooper Bridge 1; *Other Insoluble Carbonaceous Matter*: Newcastle, City Road, 984, Rothamsted 41; *Ash of Insoluble Matter*: Newcastle, City Road 1402, Garston 39; *Ash of Soluble Matter*: Newcastle, City Road 523, Leicester, Western Park 78; *Total Solids*: Newcastle, City Road 3188, Leicester, Western Park 266. The maximum and minimum total solids comprised SO_3 251 and 30, Cl 66 and 17, NH_3 13 and 1, respectively. For London the total solids varied from 520 (Kew) to 1290 (Golden Lane). The maximum and minimum mean monthly deposits of total solids (expressed as percentages of the general average for the stations concerned) were 141 (Marple) and 61 (Bournville, Birmingham) respectively, and for London 107 (Southwark Park) to 73 (Archbishop's Park).

Decreases and increases, respectively, of the various constituents of the deposits, compared with the average for the previous 5 years, were found as follows:—Tar, 12 and 14; other insoluble carbonaceous material, 13 and 15; insoluble ash, 15 and 13; loss on ignition, 24 and 4; soluble ash, 19 and 8; total solids, 19 and 9; SO_3, 20 and 7; Cl, 21 and 6; NH_3, 21 and 5. There are thus, in general, signs of improvement, except for Glasgow (Alexandra Park), Huddersfield (Cooper Bridge), Leeds (Headingley and Park Square), Marple and Newcastle (City Road), where the tar and SO_3 figures are higher.

There is a definite indication that the soluble matter in the deposit varies more or less directly with the rainfall, whilst the insoluble matter is independent of this factor. The washing effect, by which falling drops operate selectively on suspended matter, is considered a more potent cause of the removal of soluble material than condensation round soluble nuclei.

Automatic Filter Results.—Graphs illustrate the hourly distribution of suspended impurity, the shade numbers of the automatic filters being converted into mgrms. per cubic m. by the factor 0·3 (determined for sooty impurity only, in London). For London, on both ordinary and foggy days, South Kensington shows a higher average amount of suspended impurity than Victoria Street, the hourly distributions being similar, except that the morning and evening maxima are about 1 hour earlier for the former station. Kew gives a lower average value than Westminster, the maxima being at an hour later on foggy days, and at the same time on ordinary days. In the early morning of foggy days, however, the

air at Westminster is clearer than that at Kew. At Stoke-on-Trent the peaks occur at about 9 a.m. and 9 p.m., *i.e.* about 1 to 2 hours later than in London (neglecting the values for Kew, which appears to derive its impurities from London and to be a point of concentration of impurities by low-velocity easterly winds). There is no indication that hazy days are concentrated at any one part of the week.

The curve for the London smoke-fog of January, 1929, showed a distribution over the 24 hours similar to days without abnormal impurity.

Summer- and Winter-Time Changes.—The Stoke-on-Trent data have been used to compare the effects of summer and winter times on the hourly distribution of suspended impurity, and it is shown that both the valley in the curves due to the comparatively pure early-morning air and the morning maximum are shifted by the change to summer time as would be anticipated if their positions are due to human activities as distinct from meteorological influences. The afternoon peaks give doubtful results.

Domestic and Industrial Smoke.—If it is assumed that both industrial and domestic smoke are present on weekdays, but only the latter on Sundays, the former may be found by difference, and the ratio of the domestic to industrial smoke calculated. The mean of the values obtained for the 5 Glasgow centres is 3·59 for average week days (2·5 was previously found for London) and 5·71 for average Saturdays.

Measurement of Daylight.—The results for Salford are similar to those already obtained (*cf.* ANALYST, *loc. cit.*), and indicate the loss of daylight in the city. Conditions are similar at Leeds, where Headingley has a higher daily average than Park Square.

In pp. 9 to 11 the mean monthly deposits of the stations are summarised. Pp. 12 to 14 show the figures for the hourly variations of suspended impurity, p. 15 the incidence of foggy days during the week, and pp. 16 to 20 are curves illustrating the above conclusions. Pp. 21 to 64 are the General Deposit Tables of the stations for the year. J. G.

DETERMINATION OF AROMATIC, UNSATURATED, AND NAPHTHENE HYDROCARBONS IN LIGHT OILS AND MOTOR SPIRITS.*

IN these determinations, a small quantity of the oil, such as the light oils, b.pt. about 30–170° C., obtained during the carbonisation of coal at different temperatures and in different types of retort, is vaporised in a current of air and the vapour passed through a weighed quantity of a suitable reagent.

In determining aromatic hydrocarbons in mixtures containing no unsaturated hydrocarbons, addition of 2–3 per cent. of silver sulphate to 98 per cent. sulphuric acid greatly accelerates sulphonation of the benzene and its homologues, and does not affect the slight action of the acid on saturated hydrocarbons, which are absorbed to the extent of 0·1–1·0 per cent. The use of a second set of absorbing bulbs, through which only the saturated hydrocarbons pass, permits a correction to be made for this absorption. The method works well for the determination of aromatic hydrocarbons in motor spirits which do not contain cracked distillates, or of paraffins in technical benzol, toluol, and xylol.

To determine both unsaturated and aromatic hydrocarbons in hydrocarbon mixtures, both are absorbed in a suitable nitrating mixture, such as 16 per cent.

* By A. B. Manning and F. M. E. Shepherd. Technical Paper No. 28. Published September, 1930, pp. 14, under the authority of H.M. Stationery Office. Price 4d.

of potassium nitrate in 98 per cent. sulphuric acid, a second absorber being used to correct for the attack on the saturated hydrocarbons. To determine the aromatic hydrocarbons separately, they are then isolated as nitro-derivatives. The solution in the acid mixture, preferably with the addition of a little more nitric acid, is heated in a water-bath for 2–3 hours to complete the nitration of the aromatic hydrocarbons and the oxidation of the unsaturated hydrocarbons, the cooled solution being poured into excess of water and the nitro-compounds extracted with three 50 c.c. portions of benzene. The benzene solution is extracted with 10 per cent. sodium hydroxide solution, washed with water, evaporated nearly to dryness in a tared flask, and dried in a current of air at room temperature, and finally in a vacuum desiccator. Under these conditions, benzene, toluene, and *m*-xylene give, respectively, dinitrobenzene, trinitrotoluene, and trinitro-*m*-xylene, all in very nearly 96 per cent. yield. The factors for converting these to the original hydrocarbons are 0·484, 0·422, and 0·458, the mean being 0·455. Other aromatic hydrocarbons, possible in light oil, mostly yield trinitro-compounds, and only exceptionally would occur in sufficient amount to affect this factor appreciably. The unsaturated hydrocarbons are oxidised practically completely, but the products contaminate the aromatic nitro-compounds. The error thus caused may be diminished by removing some of the unsaturated hydrocarbons, before passing the vapour into the nitrating mixture, by interposition of a vessel charged with 70 per cent. sulphuric acid. It is best to run two experiments in parallel, one for determining the unsaturated and aromatic hydrocarbons together, and the other the aromatics alone. A more accurate value for the percentage of aromatic hydrocarbons and some knowledge of the proportions of the constituents are obtained by careful fractionation of the oil, cutting at 95° and 124° C. Saturated aqueous mercuric acetate solution absorbs about 70 per cent. of the unsaturated hydrocarbons in light oils obtained by low temperature carbonisation, and its use prior to the nitrating mixture might be useful when the unsaturated hydrocarbons are high and the aromatics low in amount.

The naphthene content of a light oil may be determined roughly by ascertaining the "aniline point" of the residual oil freed from unsaturated and aromatic hydrocarbons, this residual oil being recovered by passing the issuing air through a tube immersed in solid carbon dioxide and acetone. More accurate results are obtainable by dehydrogenation of these naphthenes, either pure or mixed with paraffins, when vaporised in a current of nitrogen or hydrogen and passed over palladium black at 300–350° C. The dehydrogenation may be rendered more complete by re-circulating the vapour over the catalyst and removing the toluene (from methylcyclohexane) as formed.

T. H. P.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Detection of Heated Milk and a New Method for the Detection of Pasteurisation. S. Rothenfusser. (*Z. Unters. Lebensm.*, 1930, **60**, 94–109.)—The author's method (ANALYST, 1909, **34**, 37) is revised as follows:—The "lead-serum" of the milk is prepared by addition to 100 c.c. of the sample of the minimum excess of basic lead acetate solution (5 to 6 c.c.) required to produce

complete precipitation. The mixture is shaken well, and filtered, and the filtrate should then contain no casein, fat, phosphates or citrates, but only chlorides, sulphates and acetates of sodium, potassium, calcium and magnesium, and a trace of lead. The *p*H value is 6·6 to 6·7, and the acidity is equivalent to about 1 c.c. of 0·25 *N* sodium hydroxide solution, and in serum from fresh milk both peroxidase and diastase should be present. For milk heated at a relatively high temperature *p*-tetrol sulphite (stable in the dark) is used as reagent. It is prepared by adding 1·45 c.c. of a fresh 20 per cent. solution of pure sodium bisulphite to 100 c.c. of a mixture containing 1 grm. of *p*-phenylenediamine hydrochloride in 12 c.c. of water, and 4 grms. of crystalline guaiacol in 100 c.c. of 96 per cent. alcohol, diluted to 150 c.c. with 96 per cent. alcohol. To 10 c.c. of serum (or milk) are added 4 drops of 3 per cent. hydrogen peroxide, and 10 drops of reagent. Raw milk, or milk heated for 30 minutes below 70° C., gives a violet ring immediately, milk heated for a short period above 79° C. or for a long period at 70 to 75° C. gives no colour, whilst milk heated for 30 minutes at 75° C. gives a colour after 1 minute. Storage in ice for 14 days after heating does not affect the nature of the colour. With milk pasteurised at 63° C. for half-an-hour the action of the peroxidase is upset by changes in acidity, and it is necessary to utilise the diastatic activity of the serum. A stable standard solution of starch is prepared by grinding 10 grms. of soluble starch with 10 c.c. of water, and adding 500 c.c. of boiling water. The mixture is boiled gently for 10 minutes, 150 c.c. of pure glycerin (sp. gr. 1·23) added, boiling continued for 10 minutes, and 6 grms. of sodium chloride in 50 c.c. of boiled water and 5 c.c. of 0·25 *N* sodium hydroxide solution stirred in. The hot mixture is filtered, 250 c.c. of 96 per cent. alcohol added in 50 c.c. portions, and the mixture diluted to 1 litre with boiled water, cooled, and decanted from any sediment when required. For the test, 30 c.c. of milk are shaken with 1·6 c.c. of the lead reagent in a conical centrifuge tube, 2 c.c. of hydrochloric acid-free trichlorethylene (or chloroform) added, and the mixture again shaken and centrifuged. The clear serum (10 c.c.) is then mixed with 1 c.c. of starch solution, and after 15 hours at room-temperature (or 3½ hours at 40° C.) 1·5 c.c. is poured into a small tube and 1·5 c.c. of 0·002 *N* iodine solution added. A yellow colour in the mixture denotes a slight degree of heating insufficient to destroy the character of raw milk; brown indicates a short period of heating at 60 to 63° C., or a long period at 55° C.; red-violet, 30 minutes at 60 to 63° C., violet, prolonged heating at 63° C. ("permanent" pasteurisation); and, a full blue colour, the complete absence of active diastase. Both methods are influenced by substances or treatments other than heating which affect the activities of the enzymes, and these are discussed.

J. G.

Groups of Extractives in Coffee. Ciupka. (*Chem. Ztg.*, 1930, **54**, 803.)—If a raw coffee is completely freed from alcohol-, ether- and water-soluble extractives by extraction with each solvent for 4 hours, and the residual raw fibre is dried and roasted, further extraction yields further amounts of extractives. Hence, with a roasted coffee, distinction must be made between natural and pyrogenetic extractives. The latter class comprises principally those substances

which condition the type and quality of a coffee. Experiments with Costa Rica coffee show that roasting may result in diminution of the natural extractives, although an increase in the total extractives occurs; in interpreting the results, allowance must be made for the loss in weight of the raw coffee caused by the roasting. T. H. P.

Maltol and its Colorimetric Determination in Malt Coffee. T. Merl. (*Z. Unters. Lebensm.*, 1930, **60**, 216–217.)—Maltol was prepared by heating a preparation of malt diastase (1:100) at 180 to 200° C., and was separated by steam-distillation and extraction of the distillate with ether. The ethereal extract was dried over sodium sulphate, evaporated under reduced pressure, and the maltol obtained as crystals, m.pt. 153–155° C. Other plant materials (*e.g.* larch and other barks) give it if heated, especially if collected in the spring. It gives the characteristic reactions of a phenol, *e.g.* a violet colour with ferric chloride solution, dark blue on addition of anhydrous phosphomolybdic acid, followed by 1 drop of 10 per cent. ammonia to a solution in chloroform (Brauer, ANALYST, 1926, **51**, 534), and a red precipitate with uranyl nitrate in acid-free alcohol (Aloy-Rabaud). The last reaction has a sensitiveness of 1:5,000 and, from the weight of U_3O_8 produced after ignition of the precipitate, the formula $C_6H_6O_3$ (2-methyl-3-oxypyrone) was established. It is preferable to warm 10 c.c. of an aqueous solution of maltol with 3 c.c. of 0·25 *N* sodium hydroxide solution at 50° C., 10 c.c. of 0·1 *N* iodine solution then being added, and, on the following day, 0·5 grm. of potassium iodide, and 10 c.c. of 0·25 *N* sulphuric acid, the excess of free iodine being titrated with 0·02 *N* sodium thiosulphate solution. The resulting iodoform may then be filtered off and washed and weighed with suitable precautions, or else dissolved in 5 c.c. of 20 per cent. silver nitrate solution containing nitric acid, and the silver iodide formed after 2 hours on the water-bath filtered off and weighed (0·01 grm. maltol $\equiv$ 0·0147 grm. $CHI_3 \equiv$ 0·0241 grm. AgI). With malt-coffees, 50 grms. are ground so as to pass a 0·5 mm. sieve, 10 grms. digested with 5 c.c. of water for 1 hour, and then mixed with 3 c.c. of water, 15 grms. of sand and 1·5 grms. of blood charcoal, and extracted for 5 hours with carbon tetrachloride in a Soxhlet apparatus. The extract is shaken with 100 c.c. of water containing 3 drops of 10 per cent. ferric chloride solution and 2 c.c. of *N* hydrochloric acid, and the colour matched in a Duboscq colorimeter against that produced in the same way from a standard solution of salicylic acid. Then 1 mgrm. of maltol per 100 c.c. in a layer 54 mm. deep matches 1·75 mgrms. of salicylic acid per 100 c.c. in a layer 25 mm. deep. Tests with a number of malt-coffees prepared by different processes gave maltol contents of from 1 to 9 mgrms. per 10 grms., those made from dried and roasted barley, as distinct from normally-malted barley, always giving less than 6 mgrms. per 10 grms. This figure is, therefore, proposed as a basis of a test for the specification for malt-coffee, namely, that it should contain at least 70 per cent. of half-grown barley corns, the figures actually obtained for artificial mixtures containing 25, 50 and 75 per cent. of dried roasted barley being 5·6, 4·2 and 2·7 mgrms. per 10 grms., respectively. The biological function of maltol (as an anti-oxygen for oils and fats) is discussed. J. G.

Component Glycerides of Stillingia (Chinese Vegetable) Tallow. T. P. Hilditch and J. Priestman. (*J. Soc. Chem. Ind.*, 1930, **49**, 397–400T.)—The waxlike covering of the fruits of *Stillingia sebifera* from China, Florida and Texas was examined with regard to the fatty acid and glyceride structure. The fatty acids consisted of:—(*a*) U.S. stillingia tallow neutralised, (*b*) Chinese tallow (neutralised): lauric (?), 1·2, 2·5; myristic, 2·9, 3·6; palmitic, 63·1, 57·6; stearic, 3·2, 1·8; and oleic acids, 29·6, 34·5 per cent. The tallow usually appears to contain 7 to 10 per cent. of the total acids as other than palmitic and oleic, and the presence of acids of lower molecular weight than myristic has not been definitely proved. Small quantities of non-fatty matter of a semi-volatile ester nature were present with the fat. The neutralised tallow was examined in detail by permanganate oxidation of the acetone solution, and the greater part was found to consist of mono-oleodisaturated glycerides (over 60 per cent.), whilst as much as 25–35 per cent. of fully saturated glycerides may be present. Since at least 90 per cent. of the saturated fatty acids are palmitic, these classes of fat must consist mainly of oleodipalmitins and tripalmitin. The relations of fully saturated glyceride content and the molecular ratio of saturated to oleic acids in the whole fat are similar to those found in palm oils (ANALYST, 1930, 701). The solidification temperatures of the fats studied varied from 25·6° C. for the neutral Chinese fat to 48·2° C. for the American sample. The fat is considered suitable for candlemaking and soap, but less so for confectionery purposes, owing to the high proportion of tripalmitin. D. G. H.

Dhupa Kernels (*Vateria indica*) and Oil from India. (*Bull. Imp. Inst.*, 1930, **28**, 279–281.)—The light to dark brown or purplish pieces of kernel were hard and brittle, with a faint aromatic odour. They contained 7·6 per cent. of moisture and, on extraction with petroleum spirit (b.pt. 40–60° C.), yielded 21·4 per cent. of a greenish-white, fairly hard fat. Further successive extractions of the residue gave, with ether, 3 per cent., and with acetone 7·2 per cent. (on the kernels) of brittle resinous substances. Extraction with petroleum spirit, b.pt. 90–110° C., gave 22·8 per cent. of fat, and with trichlorethylene, 22·6 per cent. The petroleum spirit extracted fat had sp. gr. 100/15° C., 0·8585, $n_D^{40°}$ C., 1·4588; m.pt. (open tube method), 34·2° C.; saponification value, 190·3; iodine value (Wijs, 3 hours), 45·9; unsaponifiable matter, 0·9 per cent.; acid value, 1·0, and solidifying pt. of fatty acids, 52·7° C. The light brown residual meal had a strong bitter taste, but no cyanogenetic glucosides or alkaloids were present. It consisted of moisture, 8·8; ether extract, 4·1; crude protein, 6·2; carbohydrates (by difference), 73·8; crude fibre, 5·0; and ash, 2·1 per cent. D. G. H.

American Cherry Kernel Oil. G. S. Jamieson and S. I. Gertler. (*Oil and Fat Ind.*, 1930, **7**, 371–372.)—The cherry-kernel oil, expressed from the mechanically separated kernels, was dark golden yellow, with a nut-like odour and slightly bitter taste, but, after refining, it was of a pale straw colour and had a bland flavour. The oil had the following characteristics, the second set of

values referring to the crude oil:—Sp. gr. 25/25°, 0·9183, 0·9176; $n_D^{25°}$ C., 1·4740, 1·4742; saponification value, 190·7; iodine value (Hanus), 115·8, 118·7; Reichert-Meissl value, 0·5; Polenske value, 0·2; acid value, 0·09, 4·39; unsaponifiable matter, 0·5, 0·66 per cent.; saturated acids, corrected, 7·7 per cent.; unsaturated acids, 87·0 per cent., with iodine value, 127·9. The calculated iodine value of the unsaturated acids was 133·1, and their composition was calculated to be: oleic acid, 46·85; linolic, 40·11 per cent., on the oils separated. The saturated acids were esterified, the esters fractionated, and eventually six fractions were analysed and their composition determined from the data obtained. The percentages of acids in the oil were:—Myristic, 0·19; palmitic, 4·04; stearic, 2·79; and arachidic, 0·72 per cent. D. G. H.

***Moringa aptera* Seed and Oil from Egypt.** (*Bull. Imp. Inst.*, 1930, **28**, 276–279.)—The *Moringa aptera* seeds were obtained from a small wild Egyptian tree related to *Moringa pterygosperma*, the seeds of which yield ben oil. The sample consisted of seeds averaging $\frac{5}{8}$ in. long and $\frac{1}{2}$ in. broad, triangular in cross section, about 50 per cent. being greyish-brown and the rest cream coloured and usually covered with green patches, averaging 0·5 grm. in weight. The thin brittle shell (51·2 per cent.) enclosed cream-coloured or greenish kernels (48·8 per cent.) which contained 4·9 per cent. moisture and 50·0 per cent. of oil. The golden-yellow extracted oil had sp. gr. 15/15° C., 0·9151; $n_D^{40°}$ C., 1·461; saponification value, 188·2; iodine value (Wijs, 3 hours), 71·2; unsaponifiable matter, 0·5 per cent.; acid value, 0·5; and solidifying point of fatty acids, 28·1° C. These figures, except the last, are very similar to those of ben oil, and the cream coloured residual meal had the following composition: Moisture, 8·7; crude proteins, 48·6; fat, 2·6; carbohydrates (difference), 28·0; crude fibre, 6·6; and ash, 5·5 per cent. The meal, free from cyanogenetic glucosides, appeared to contain an alkaloidal substance and a saponin. It was bitter in taste. D. G. H.

Brazil Nut Oil. H. A. Schuette, R. W. Thomas and M. Duthey. (*J. Amer. Chem. Soc.*, 1930, **52**, 4114–4117.)—The oil from 12 kilos. of shelled Brazil nuts, expressed in a manual press, was of a pale yellow colour, and the residual pulp when extracted with petroleum spirit yielded a dark brown oil which, in contradistinction to the expressed oil, yielded no deposit of glycerides at 5–10° C. The oils had the following characteristics:—(1) Expressed, and (2) residual:—Sp. gr. 25/25° C., 0·9150, 0·9143; $n_D^{20°}$ C., 1·4678, 1·4683; saponification value, 194·0, 198·0; iodine value (Wijs), 99·92, 95·21; Reichert-Meissl value, 0·0, 0·31; Polenske value, 0·0, 0·32; free fatty acids (as oleic), 0·006, 0·02 per cent.; unsaponifiable matter, 0·64 and 0·68 per cent.; acetyl value, 12·3, 12·3; "titre" test, — and 33·3°; soluble acids as butyric, 0·87, 0·56; insoluble acids, 94·16, 93·88; unsaturated acids (corrected), 73·0, 70·1, of iodine value 129·18 and 127·92 respectively, and saponification value, 199·6 and 201·2; saturated acids (corrected), 20·29, 21·36. The composition of the unsaturated acid fraction was found by bromination of the acids to be (as percentage in the oil), oleic 51·26, linolic 18·84.

The saturated fraction, by separation of the methyl esters into 5 fractions, was found to consist of myristic 1·70, palmitic 12·92, and stearic acid 2·47 per cent. in the oil. D. G. H.

New Reaction of Diallylmalonylurea (Dial). F. Lagarce. (*J. Pharm. Chim.*, 1930, **122**, 364–365.)—A fresh solution of 1 per cent. of vanillin in sulphuric acid gives, on warming with dial, a stable cherry-red colour. A few crystals of dial may be treated with a few drops of the reagent on a watchglass, or the reagent may be poured into an aqueous solution of dial, when 0·5 mgrm. gives a distinct reaction. If no colour results after addition of 2 or 3 times the volume of reagent, the liquid is warmed, and under these conditions alcoholic or ethereal solutions give a green colour in the absence of dial. Although colour reactions are similarly given by other products, such as allyl alcohol, terpenes, menthol and camphor, no colour is formed by the alkyl derivatives of malonylurea, for example isopropyl allylmalonylurea (numal), and the reaction is useful for identifying dial in toxicological work, particularly when the quantity isolated is too small to allow of the m.pt. being determined, or when a difficult mixture of crystals is being dealt with. D. G. H.

New Colour Reaction of Ephedrine. J. Siradgian. (*J. Pharm. Chim.*, 1930, **122**, 266–269.)—To 4 c.c. of a solution of hydrogen peroxide containing 4 per cent. of sodium chloride a few mgrms. of an amino alcohol of the ephedrine group are added (if the salt of the base is being used 6 drops of a 0·1 *N* sodium hydroxide solution are added) and the mixture boiled. A red colour is formed, deepening to red-violet on cooling. If no sodium chloride is present the molecule is destroyed and an odour of benzaldehyde is apparent. In the molecule of compounds of this series the simultaneous presence of the two secondary amine and alcohol radicles is necessary for production of the reaction, and this is also the case with the biuret reaction. Ephedrine and its superior homologues give intense red-violet colours, but the pseudo-forms take an orange yellow colour. Pyramidon gives a blue coloration. D. G. H.

Diastatic Power of Malt and Malt Extract. C. T. Bennett and F. C. L. Bateman. (*Quart. J. Pharm. and Pharmacol.*, 1930, **3**, 349–353.)—The following standard values have been fixed by the National Mark scheme for the diastatic powers of various grades of malt extract : (1) Pharmaceutical malt extract, not below 25. (2) Baker's malt extract (white bread), not below 40. (3) Baker's malt extract (brown bread), no fixed minimum. (4) Veterinary malt extract, not below 15. The method of determination is fully described in Appendix 3 of the marketing leaflet No. 14, issued by the Ministry of Agriculture.

For a sample of malt extract and one of ground malt supplied to a number of laboratories, widely divergent values were obtained for the diastatic powers. The soluble starches sold for analytical purposes appear to vary considerably, and most workers prepare their own, but no standard is available and no means of checking soluble starch exists. The authors consider that the temperature of

70° F., originally proposed by Lintner for the starch conversion, might, with advantage, be changed to 40° or 45° C., at which the diastatic power is much greater.

It is questionable to what extent diastatic power is necessary for a pharmaceutical malt extract. If this is administered for its diastatic activity, its ability to convert starch into sugar is limited by the amount given and by the action of the digestive juices, whilst if it is given as a food or as a vehicle for cod-liver oil, no test for diastase appears necessary. The authors favour Silbernagel's suggestion that the use of soluble starch for testing diastatic activity be replaced by that of refined potato starch, which is more nearly a standard product (*Ind. Eng. Chem.* [*Anal. Ed.*], 1930, **2**, 31). T. H. P.

Biochemical.

Determination of Inorganic Sulphate in Serum. R. S. Hubbard. (*J. Biol. Chem.*, 1930, **88**, 663–668.)—The reagents used in this method (ANALYST, 1929, **54**, 300) are: (1) 20 per cent. solution of sulphate-free trichloroacetic acid in sulphate-free water; (2) a stock solution of 4·015 grms. of pure benzidine hydrochloride per litre, 1 c.c. of this corresponding with 0·5 mgrm. of sulphur. This is diluted to give solutions of which 1 c.c. is equivalent to 0·1, 0·01 and 0·001 mgrm. of sulphur; these are fairly stable, but should be rejected if they develop much colour. (3) One per cent. solution of high grade benzidine in high grade acetone; this is discarded when it becomes highly coloured, and is best prepared daily; (4) The same grade of acetone for washing purposes; it is redistilled if coloured or contaminated with inorganic matter; (5) *N* and 0·2 *N* hydrochloric acid; (6) 3 per cent. hydrogen peroxide solution of pharmacopoeia quality freshly diluted with 9 parts of water; bottles which have been opened for a long time should not be used. (7) Aqueous 2·5 per cent. ferric chloride solution. All glassware used must be clean and free from sulphate.

The protein is precipitated by adding 1 part of reagent (1) to 1 part of the serum, and the liquid centrifuged. The sulphate is precipitated by adding 2 c.c. of the supernatant liquid (=1 c.c. of serum) to 5 c.c. of reagent (3) in a sharp-pointed, conical, 15 c.c. centrifuge tube, the liquid being mixed and the tube capped. After 15 minutes the tube is centrifuged at high speed; if no powerful centrifuge is available, the precipitate may be caused to flock out by immersion of the tube in ice-water for an hour or more. The liquid is decanted off, the precipitate allowed to drain for 5 minutes, and the inside of the lip of the tube wiped. The precipitate is mixed with 15 c.c. of acetone, centrifuged, and drained as before. A second washing is usually unnecessary. The precipitate is warmed with 2 c.c. of 0·2 *N* hydrochloric acid and dissolves easily if less than 0·5 mgrm. of sulphur is present. Otherwise 4·6 c.c. of *N* hydrochloric acid is added, the liquid being warmed, if necessary, and made up to 25 c.c. with water; 2 c.c. is measured into a 15 c.c. centrifuge tube for the final determination.

Standards containing benzidine equivalent to amounts of sulphur ranging

from 0·001 to 0·05 mgrm. are prepared, no one of these being more than twice as strong as the next lower one. Each of these is mixed with 2 c.c. of 0·2 *N* hydrochloric acid and diluted to 10 c.c. The solution to be tested, containing 2 c.c. of 0·2 *N* hydrochloric acid, is also diluted to 10 c.c. Each solution is then mixed with 1 c.c. of the freshly diluted hydrogen peroxide solution, treated with 0·5 c.c. of 2·5 per cent. ferric chloride solution, and again mixed. After the lapse of at least 10, and not more than 30 minutes, the colorimetric comparison is made. From the readings obtained with the standard and serum solutions the proportion of sulphur in the serum is easily calculated.

If more than 0·7 mgrm. of sulphur is present in 1 c.c. of serum, it is usually necessary, owing to the appearance of an interfering brown pigment, to repeat the determination with a smaller aliquot of the 25 c.c. of the benzidine sulphate solution. In such case, 0·2 *N* hydrochloric acid is added to make the total volume 2 c.c. before diluting for the colour comparison. T. H. P.

Determination of Peptic Activity: Examination and Application of the Gates Method of Proteolytic Enzyme Titration. A. Gilman and G. R. Cowgill. (*J. Biol. Chem.*, 1930, **88**, 743–752.)—In this method (*Proc. Soc. Exp. Biol. and Med.*, 1926–1927, **24**, 936), a photographic film is reduced by exposure and subsequent development, an almost opaque substrate being thus obtained. Digestion of the gelatin liberates reduced silver with a progressive change from opacity to transparency, depending in degree on the extent of the digestion. The change in intensity of the light penetrating the film before and after digestion determines the proteolytic activity.

A pile of six Eastman's commercial or, better, commercial ortho films (10 by 8 inches) is exposed at a target range of 24 inches for 2 minutes to Röntgen rays at 25,000 volts. and 10 milliamperes, the films being then developed under standard conditions (so as to ensure constant opacity) and fixed in plain hypo without hardener. Re-washing of the developed films with distilled water and again drying before use is advantageous in order to "set" the film, and thus render subsequent wettings without effect. The films are cut into pieces, $\frac{3}{4}$ by 1 inch, which are digested in small cells holding about 0·5 c.c., made by mounting rings of No. 14 copper wire, 0·5 inch across, on glass squares of 1 inch side by means of paraffin wax. The cell is filled with the solution under examination so that the meniscus projects above the copper ring, the film with gelatin layer down being placed on the top of the cell and in intimate contact with the liquid. Another glass square is put over the film and the whole cell, held together with a clothes-pin and thus made water-tight, is immersed in a water-bath at $25 \pm 0{\cdot}02°$ C., for a definite time (10 mins.). Even at this low temperature, the films would be completely digested by the concentration of pepsin usual in gastric juice, so that dilution of the liquid is necessary; this is best effected by Sörensen's glycine, sodium chloride and hydrochloric acid buffer solution, which gives the optimum *p*H 2. At the conclusion of the digestion the films are washed free of enzyme by immersion in the water-bath and dried in a current of air.

The light penetrating the film before and after digestion is measured by means of a Klett or other colorimeter of Duboscq type. The films are read against a gelatin-silver suspension prepared by dissolving the gelatin layer from two films in about 2 c.c. of hot water, filtering through filter-paper to remove any large heavy particles, and suspending this black silver emulsion in sufficient glycerol to fill the cup of the colorimeter; no settling of the silver occurs during several hours. Below this cup of the colorimeter is placed one of the films from which the gelatin and silver have been removed. The other cup contains glycerol to which has been added the same proportional amount of water as is present in the gelatin-silver suspension just described, and below it is placed the film to be measured. The light traversing the latter is determined by regulating the depth of the suspension on the opposite side of colorimeter until the light intensities match. The films are carried between two thin layers of sheet brass lacquered to prevent internal reflexion and having a central half-inch hole; these carriers are clamped to the rack of the colorimeter with a paper-clip so that the exposed part of the film is directly under the cup.

Despite careful exposure and development, the films vary in their initial readings, and films with the same reading should be used for any series of determinations. T. H. P.

Quantitative Differentiation of Vitamins *A* and *D*. II. H. C. Sherman and H. K. Stiebeling. (*J. Biol. Chem.*, 1930, **88**, 683–693.)—When rats with considerable bodily stores of vitamins *A* and *D* are used for the quantitative determination of these vitamins under conditions in which lack of the vitamin in question is the first dietary deficiency, gain in weight under suitably controlled conditions is the best measure for vitamin *A* intake, and the degree of calcification the most practical measurement of vitamin *D* intake (in test animals not showing rickets). For vitamin *A*, it is best to determine the amounts of materials necessary to produce the same limited gain in weight (3 or 4 grms. per week) in suitably standardised rats receiving a basal diet free from vitamin *A* but otherwise adequate. As regards vitamin *D*, determination is made of the amounts of food (or other) materials necessary to induce a degree of calcification midway between the maximum and minimum values obtainable under controlled conditions. T. H. P.

Anti-Scorbutic Vitamin in Apples. M. F. Bracewell, E. Hoyle and S. S. Zilva. (Issued by Medical Research Council, His Majesty's Stationery Office, 1930.)—Of a number of varieties of apples tested for the anti-scorbutic vitamin *C*, Bramley's Seedling was found to be decidedly more active than all the other varieties, which differed among themselves very little in their vitamin *C* content. There were no indications in the results that the anti-scorbutic activity of the apple is influenced by the character of the soil, or the age of the tree, or the season. Bramley's Seedlings, picked from the tree 14 days before the normal crop, were approximately of the same anti-scorbutic activity as those of the normal crop, and little loss in activity occurred with apples stored for 3 months either in air at 34° F., or in a mixture of carbon dioxide, nitrogen, and oxygen

at 50° F.; the gas-stored apples showed, however, a definitely greater deterioration in the vitamin. With various imported dessert apples the activity was highest for those most recently picked; no very marked differences corresponding with differences in variety was noticed. The baking of Bramley's Seedlings in their skins scarcely affected their anti-scorbutic activity. T. H. P.

Agricultural.

Determination of the Acid-Base Balance in the Ash of Plants. D. E. Frear. (*J. Biol. Chem.*, 1930, **88**, 675–681.)—As a reagent to be added to plant material prior to ignition, magnesium nitrate gives good results, as it possesses sufficient oxidising power and a suitable alkaline element, and does not yield a coloured solution or a highly adsorptive precipitate on neutralisation of the calcined material. In a porcelain beaker, 2 grms. of the finely ground plant material are thoroughly wetted with about 10 c.c. of water and then mixed with 25 c.c. of a magnesium nitrate solution containing 25 grms. of the hydrated salt per 100 c.c. The mass is then left to simmer on an electric hot plate until a quite dry, but not explosive, residue is obtained (3 to 4 hours); the duration of drying is best regulated by experience. The beaker is then covered and placed in an electric furnace heated to about 250° C.; after the lapse of 15 minutes, or when it is evident that the first reaction is over, the temperature is raised to 500°, which is maintained for 30 minutes or for a longer period if particles of unburnt carbon remain. When the residue is white or nearly so, it is allowed to cool, and carefully and thoroughly wetted with about 10 c.c. of water. After addition of 60 c.c. of *N* nitric acid from a burette, the liquid is digested on a hot plate just below the boiling point for 3 hours. The solution and any undissolved matter are then transferred to an Erlenmeyer flask, the excess of acid being titrated with *N* sodium hydroxide solution in presence of methyl red. A blank determination with 1 grm. of sugar is made in the same manner.

The amount of *N* acid used, less that of *N* alkali, gives the amount of acid required to neutralise the alkalinity of the crop, plus that of the magnesium oxide formed from the oxidising solution. Subtraction of the amount of *N* acid necessary for neutralisation in the blank determination now gives a number of c.c. which represents the milli-equivalent weight of excess alkalinity in 2 grms. of the plant material. Should the material have an excess of acidic elements, the result of this calculation will be of negative sign.

Test experiments show that only small losses of sulphur and chlorine occur when the above procedure is followed, and that the acid-base balance of the plant thus determined is in good agreement with the stoichiometric balance of the strong acidic and basic elements obtained by chemical analysis. T. H. P.

Organic Analysis.

Iodine Colorimetric Method for the Determination of Starch. L. Paloheimo. (*Biochem. Zeits.*, 1930, **222**, 150.)—The standard starch solution used is prepared by making 0·5 grm. of air-dried starch, of known moisture content,

into a paste with a little cold water, adding boiling water to make the volume up to 400 c.c., boiling for 15 minutes, adding 20 c.c. of *N* sulphuric acid, and continuing the boiling for a further period of 15 minutes. Of the cooled solution made up to 400 c.c., 20 c.c. are made up to 500 c.c. with water and 5 c.c. of a 5 per cent. solution of potassium iodide saturated with iodine. The solution of the unknown starch is prepared from 1 grm. of the finely ground substance and is treated as above, except that the solution is filtered hot before being cooled and remade up to 400 c.c. Of this solution an amount which contains more starch than 20 c.c. of the standard solution is mixed with 5 c.c. of the iodine solution and made up to 500 c.c. A dilute iodine solution also is prepared by diluting 5 c.c. of the original iodine solution to 500 c.c.

Two glass cylinders of the same diameter are placed side by side in a simple comparator illuminated by direct light. Into the first are poured 100 c.c. of the unknown starch and iodine solution, and into the second 150 c.c. of the corresponding standard starch solution, and the dilute iodine solution is gradually added to the first cylinder. As the dilution proceeds, the solution being tested retains the same iodine concentration, whilst its starch concentration approaches that of the standard solution and equals it when the colorations of the two liquids become identical. If the standard and unknown starches are of different kinds, a factor must be introduced into the calculation. T. H. P.

Determination of Asphaltene. F. J. Nellensteyn and N. M. Roodenburg. (*Chem. Ztg.*, 1930, **54**, 819.)—A 150 c.c. Erlenmeyer flask is weighed together with a bent rod and a filter, 2 grms. of asphalt weighed in, and 100 c.c. of ether added. The mixture is shaken, the flask stoppered, and the liquid filtered the next day through a weighed paper, the flask and paper being washed with 100 c.c. of ether, and the residual asphaltene weighed after drying the flask, rod and paper in the oven. Its ash is usually about 5 per cent. Ether is preferable to petroleum spirit, as it is constant in composition and is better as a flocculating agent and solvent for substances other than asphalt likely to be found in coal tar (especially for hydroxy-fatty acids). It is cheaper than the specially prepared "normal benzine," 5 samples of which gave results in asphaltene determinations ranging from 16·4 to 38·6 per cent., whilst 3 samples of ether gave 30·5 to 36·8 per cent. for the same sample of asphalt. J. G.

Inorganic Analysis.

Analysis of Red Phosphorus. S. A. Tolkatschoff and M. A. Portnoff. (*Z. anal. Chem.*, 1930, **82**, 122–133.)—Improved methods are given for the determination of total phosphorus, phosphorus combined with oxygen, and yellow phosphorus. *Total phosphorus:* Complete oxidation to phosphoric acid was found to be readily accomplished by a mixture of bromine and nitric acid. About 0·25 grm. of phosphorus is covered with a water layer, 2 to 3 cm. deep in a 100 c.c. flask, which is then warmed; small portions of a saturated solution of bromine in

nitric acid are cautiously added, no white fumes should form above the liquid. When solution is complete (after about 20 minutes), the content of the flask is transferred to a basin, evaporated to small bulk, and the residue twice evaporated with 5 c.c. of bromine in nitric acid. The liquid is diluted, filtered, if necessary, from insoluble matter present in the sample, made ammoniacal, filtered if necessary, and precipitated with magnesia mixture. *Phosphorus combined with oxygen*: 20 grms. are weighed into a graduated 250 c.c. flask and shaken for 12 to 15 hours with 20 c.c. of 2 *N* sulphuric acid diluted with water. The volume is made up, the liquid filtered through linen, and 50 c.c. of filtrate evaporated twice with 5 c.c. portions of bromine in nitric acid. After dilution with hot water and filtration, the solution is precipitated with ammonium molybdate. *Yellow phosphorus*: 15 to 20 grms. are digested in a 100 c.c. graduated flask with carbon disulphide for 12 to 15 hours with occasional shaking. The solution, made up to the mark, is rapidly filtered in a carbon dioxide atmosphere, and 50 c.c. pipetted off. The pipette is emptied by a carbon dioxide supply into a flask containing bromine water; the flask is shaken, and more bromine added till its colour proves presence of an excess. The carbon disulphide is distilled off, and the residual liquid heated on the water bath with 5 c.c. of bromine in nitric acid. The liquid is then transferred to a basin, evaporated, and another evaporation with bromine in nitric acid made; the solution is filtered and the filtrate precipitated with magnesia mixture. Other impurities are determined, after solution of the phosphorus, by ordinary analytical methods. Moisture is determined in a vacuum desiccator; the weight becomes constant after about two days. W. R. S.

Iodimetric Determination of Vanadium in Alloy Steels and Ferrovanadium. W. Werz. (*Z. anal. Chem.*, 1930, **81**, 448–450.)—The drillings (1 to 3 grms.), in a 500 c.c. conical flask, are boiled with 50 c.c. of syrupy phosphoric acid, 175 c.c. of water, and 5 c.c. of nitric acid (1:1) till dissolved. The solution is oxidised with nitric acid, 0·5 to 1 grm. of ammonium persulphate is added, and the excess destroyed by 15 minutes' boiling. After cooling to about 70° C., any permanganic acid is destroyed with 5 to 10 c.c. of 1 per cent. oxalic acid solution. When quite cold, the solution is shaken with potassium iodide and titrated after 5 minutes with 0·05 *N* thiosulphate (1 c.c.=0·00255 grm. V). Starch is added towards the end; total volume, about 200 c.c. *Chrome steel* may give a dark residue of carbide, which is filtered off through glass wool before oxidation with persulphate. *Ferrovanadium:* 1 grm. in a 500 c.c. flask is dissolved in 25 c.c. of nitric acid. The volume is adjusted, 50 c.c. transferred to a 500 c.c. conical flask, and treated with 75 c.c. of phosphoric acid, 100 c.c. of water, and persulphate as above. The method is claimed to be a rapid one. W. R. S.

Some Colour Reactions of Magnesium. I. M. Kolthoff. (*Mikrochemie Emich-Festschrift*, 1930, 180–190.)—The application of several dyestuffs for the detection of magnesium in strongly alkaline media is examined. In all cases, cobalt and nickel, and sometimes manganese, give the same test as magnesium, and, in the presence of these ions, special precautions must be taken.

Titan yellow is a delicate reagent for magnesium, and more specific than 1.2.5.8-oxyanthraquinone, which also reacts with beryllium, lanthanum and cadmium to give coloured compounds. For the titan yellow test, 10 c.c. of the solution are used, and 0·1–0·2 c.c. of a 0·1 per cent. solution of titan yellow G in water are added, and about 0·25 to 1 c.c. of 4 *N* sodium hydroxide solution. In the absence of magnesium the mixture is a brownish yellow colour; if 5 mgrms. per litre of magnesium are present the solution turns red; and for 1 mgrm. per litre, orange. The sensitivity* of the test is 0·2 mgrm. per litre, in a solution containing 2μ grm. Small amounts of calcium salts intensify the colour. Zinc interferes with the test, unless potassium cyanide to the extent of about double the weight of the zinc present is added. Potassium cyanide also nullifies the interfering action of cobalt and nickel, but if manganese is present, it is better to remove it as sulphide. Beryllium does not interfere in the presence of sufficient sodium hydroxide, though it decreases the sensitivity of the test for magnesium. A mixture of Titan yellow and magnesium chloride can be used as a reagent for hydroxyl ions, the sensitiveness being dependent on the magnesium concentration. This reaction may also be used for spot tests on filter paper. Brilliant yellow (diaminostilbene-diphenylsulphonic acid) behaves similarly to titan yellow, but gives a less sensitive reaction. Using the same procedure as with titan yellow, the sensitivity for magnesium is 4 mgrm. per litre, and for cobalt and nickel, 2 mgrm. per litre. The reactions with *o*-*p*-dihydroxy-azo-*p*-nitrobenzene, Congo red, "la Motte purple," turmeric, benzopurpurin, and aniline yellow S, have also been examined. Aniline yellow S (Kahlbaum) gives a very sensitive reaction with lanthanum and copper in weakly acid medium (acetate buffer, *p*H about 5·0). J. W. B.

Volumetric Determination of Silica in Silicates. N. A. Tananaeff and A. K. Babko. (*Z. anal. Chem.*, 1930, **82**, 145–150.)—The silicate (0.2 grm. SiO_2) is fused with potassium carbonate, the cake transferred to a platinum dish, and the crucible cleaned, with 20 c.c. of water. The liquid is treated with 20 c.c. of strong hydrochloric acid and 2 grms. of ammonium fluoride, and left to stand for 1 to 2 hours, with occasional stirring. The precipitated potassium fluosilicate is collected on paper in a paraffin-coated funnel, and washed 5 times with a saturated solution of potassium fluosilicate. Filter and precipitate are transferred to a conical flask with 20 c.c. of 4 *N* calcium chloride solution and water to make up about 100 c.c. The flask is warmed on a water bath, and the liquid titrated with 0·5 *N* sodium hydroxide after addition of methyl red:

$$K_2SiF_6 + 3CaCl_2 + 4NaOH = 2KCl + 4NaCl + Si(OH)_4 + 3CaF_2.$$

The end-point is indicated by either the coagulation of the precipitate or the colour change. A blank determination is always required, and the volume deducted from that found in the assay. The determination requires 3 to 3½ hours. Deviations of 0·3 to 0·4 per cent. from the results obtained gravimetrically were observed. W. R. S.

* Feigl (*Mikrochemie*, 1923, **1**, 1) defines "sensitivity" as the limit of dilution in which a test can be successfully carried out.

Determination of Lead Tetraethyl in Gasoline. L. J. Catlin and J. E. Starrett. (*Chemist-Analyst*, 1930, **19**, No. 5, 5–6.)—A 10 per cent. solution of bromine in carbon tetrachloride is added slowly, with stirring, to 100 c.c. of sample to the extent of 10 c.c. for straight run spirit and 30 c.c. for cracked spirit. After a few seconds, 5 c.c. are added in excess, the lead bromide allowed to settle, filtered off and washed by decantation with 25 c.c. of carbon disulphide. The precipitate is then washed back into the flask with, and dissolved in, 30 c.c. of hot 15 per cent. nitric acid, 5 c.c. of concentrated sulphuric acid added, and the mixture heated till white fumes appear. The lead sulphate is filtered off, washed, dissolved in hot ammonium acetate solution and titrated while hot, with ammonium molybdate solution (8·6 grms. per litre; 1 c.c. = 0·01 grm. Pb). A yellow colour with a fresh 0·5 per cent. solution of tannin, used as outside-indicator, gives the end-point.

J. G.

Arsenic in Writing Materials. G. Kappeller. (*Z. Unters. Lebensm.*, 1930, **60**, 213–215.)—Of 14 samples of violet carbon-paper, 5 were found to contain arsenic, 3 being of German origin (0·95 to 3·8 grms. of arsenic per 100 grms., or 16·1 to 59·6 mgrms. per sheet), while 2 were American (0·9 and 3·0 grms. of arsenic per 100 grms., or 19·8 and 45·4 mgrms. per sheet), corresponding with 1·9 to 7·6 and 1·8 to 6·0 grms. of arsenic per 100 grms. of colouring matter, respectively. Two (English) violet typewriter-ribbons contained 0·5 to 1·1 grms. of arsenic per 100 grms., or 5·8 and 15·4 mgrms. per metre, respectively. Violet pencils (4) and aniline ink-powders (2) were free from arsenic. The possibility of contamination through use of oxides of arsenic in the manufacture of (*e.g.*) fuchsin, methyl violet, Prussian blue, etc., is discussed. J. G.

Microchemical.

Application of the Dilution Method to Micro-analysis. J. B. Mederl, O. R. Trantz and W. J. Saschek. (*Mikrochemie Emich-Festschrift*, 1930, 219–232.)—The micro-determination of nitrogen is carried out by Pregl's or Dubsky's methods without using a micro-balance. The substance to be determined is weighed, dissolved in some solvent free from nitrogen, to give a 1 per cent. solution. The most useful solvent is carbon tetrachloride; but any other non-nitrogeneous solvent may be used, such as water, methyl and ethyl alcohol, ether, chloroform, benzene, carbon disulphide or mixtures. A weight of solution to contain 3–5 mgrms. of the substance to be analysed is weighed on an ordinary analytical balance in a tube containing about 2 c.c. of powdered copper oxide, and provided with a tightly fitting stopper. The solvent is then removed by evaporation. When carbon tetrachloride is used the tube is left unstoppered at room temperature for 24 hours. The material is then analysed in the usual way.

A micro-Dumas determination of nitrogen is described, in which uncertainty as to the length of time and the volume of carbon dioxide used in sweeping out the gases after the combustion is over is avoided by the use of a gas holder of 150–200 c.c. volume. After the combustion is at an end, 100 c.c. of carbon dioxide from

the gas holder are passed through the tube. This volume is sufficient for any micro-combustion. An advantage of the method is that a blank experiment may be carried out in which the volume of air present in 100 c.c. of the carbon dioxide may be determined, and subtracted from the results, as even the purest carbon dioxide contains some air. An arrangement of Kipp's apparatus in series for the production of pure carbon dioxide is described, the gas above the surface of the hydrochloric acid being carbon dioxide from another Kipp's apparatus. The Kipp's apparatus may be refilled with acid without any air being allowed to enter.

J. W. B.

Micro-Analysis of Steel. J. Kassler. (*Mikrochemie Emich-Festschrift*, 1930, 170–174.)—Methods are described for the determination of carbon, manganese, nickel and chromium in a total of not more than 0·5 grm. of steel. *Carbon* is determined by the Dennstedt method, using samples of 0·05 grm. of steel, mixed with an equal weight of lead peroxide. Soda-lime is used for the absorption of carbon dioxide with phosphorus pentoxide to prevent water losses. The value for a blank test (0·12 mgrm.) is subtracted from the results. For heavily alloyed steel, which is difficult to burn, about 0·05 grm. of an unalloyed steel is mixed with the test steel and the lead peroxide in the porcelain boat. *Manganese* is slightly alloyed or unalloyed steel is determined by Procter Smith's volumetric method, in 0·02;0·05 grm. of steel. The sample is weighed out into a 50 c.c. beaker and dissolved in 5 c.c. of a mixture of nitric and sulphuric acids (100 c.c. H_2SO_4, 100 c.c. HNO_3, 300 c.c. water), boiled, diluted with 10 c.c. of water, and treated with 4 c.c. of a 10 per cent. ammonium persulphate solution and 2 c.c. of a 0·5 per cent. silver nitrate solution. After boiling for one minute and diluting with 25 c.c. of water, the permanganic acid is titrated in the usual way with arsenious acid (1 grm. of As_3O_3, 5 grms. of Na_2CO_3, dissolved in 400 c.c. of hot water and diluted with 6,400 c.c. of water). The analysis takes 10 minutes. An empirical factor is used. *Nickel* is determined volumetrically by Moore's method, using an empirical factor, calculated from a steel of known nickel content, or gravimetrically, by precipitation, as dimethyl glyoxime. About 5 mgrms. of steel are used for the determination, which is carried out, using Emich's "filter stick" method of filtration. *Chromium* is determined in 0·1 grm. of steel. The steel is taken up in 1: 5 sulphuric acid containing 60 grms. per litre of sodium biphosphate, and oxidised with 1 c.c. of nitric acid with heating. After dilution with 50 c.c. of water and boiling, the chromium is oxidised while hot to chromic acid by means of a 1 per cent. potassium permanganate solution, when, after suitable dilution, after the destruction of excess permanganate, the chromic acid is titrated in the usual manner with iron ammonium sulphate and potassium permanganate. Steels which are hard to dissolve are first fused in small silver crucibles with sodium peroxide, and the chromic acid determined after solution.

J. W. B.

Potassium Antimonate Test for Sodium. W. Böttger. (*Mikrochemie Pregl-Festschrift*, 1929, 14–17.)—This test is more useful on the micro- than on the macro-scale. The solution to be tested for sodium is evaporated to dryness, a small granule of the residue placed on the slide, and a small drop of the reagent placed over it. The crystals are formed in less than a minute, the form depending largely on the concentration of the reagent. It is important that there should not be an excess of reagent present, otherwise concentration crystals are formed. The reagent is best made from about 0·05 grm. of potassium antimonate shaken for 2 or 3 minutes with 5 c.c. of water, and then warmed to 50° C. The fine particles then go into solution, and the residue should be filtered off. To dissolve the coarser grained potassium antimonate, the crystals are heated to boiling with water and rapidly cooled. The test is disturbed by the presence of magnesium, which should be removed from the solutions to be tested for sodium. J. W. B.

Physical Methods, Apparatus, etc.

Fluorescence of Olive Oil under Ultra-Violet Light. A. L. Glanz. (*Ind. Eng. Chem., Anal. Ed.*, 1930, **2**, 256–258.)—All pure virgin olive oils exhibit a yellow fluorescence under ultra-violet light, whilst all refined oils show a blue fluorescence. By this means as little as 5 per cent. of refined oil in a virgin oil can be detected. The spectrum of virgin olive oils shows a characteristic red band which is lacking in refined oils, but may be approximated by the addition of chlorophyll. The fluorescence of an oil, however, is independent of its chlorophyll content. When carotene and annatto are added to virgin oils adulterated with refined oil, the yellow fluorescence only is observed. The addition of annatto can be detected by chemical tests, but not that of carotene. The carotene content is believed to be directly responsible for the yellow fluorescence exhibited by virgin olive oils. W. P. S.

Reviews.

A Comprehensive Treatise on Inorganic and Theoretical Chemistry. Vol. X. By J. W. Mellor, D.Sc., F.R.S. Pp. x + 958. London: Longmans, Green & Co., Ltd. 1930. Price 3 guineas net.

In this volume only two elements are discussed, sulphur and selenium, and no less than two-thirds of it are devoted to sulphur. Taken as a whole, the volume compares well with the previous volumes, the high standard there reached being maintained. It is trivial on the part of a reviewer to attempt to criticise the work of so great and able an author and compiler as Mellor, especially when it is appreciated how extremely fortunate is the present age in having such a servant. No previous age in the progress of chemistry has ever had such a compiler.

Attention might, however, without any lack of gratitude to Dr. Mellor, be drawn to a few slight imperfections that appear in the present volume. Dealing on page 212 with the effect of salts on the solubility of sulphur dioxide in water, no mention is made of the latest, and probably the most satisfactory, work published by J. C. Hudson in 1925. Fig. 49 does not, as stated, represent an electrometric titration curve of sulphurous acid as obtained by Kolthoff. The curve given in his paper was plotted from figures calculated from the dissociation constants found by means of indicators. As far as the reviewer is aware, a curve obtained electrometrically has not yet been published, none of the ordinary electrodes employed for the determination of the concentration of hydrogen ions being responsive to these changes in sulphurous acid solutions. It might here be stated that Dr. R. A. Robinson, working in the reviewer's laboratory, has found that a considerable measure of success can be obtained with the antimony electrode in solutions of sulphur dioxide. Fig. 49, and its application in the use of suitable indicators in the volumetric estimation of sulphurous acid, might have been better placed with the matter relating to the dissociation constants given on an earlier page. In spite of there being over 600 pages on the chemistry of sulphur in the book, barely three-quarters of a page are given to the manufacture of sulphuric acid by the "contact process," and the problem of the catalyst receives only a casual mention.

In concluding this notice, the reviewer again avails himself of the opportunity to convey to Dr. Mellor the heartiest congratulations and thanks of the living "privates in the great army of workers in chemistry" on bringing his stupendous treatise yet another step towards completion.

HUBERT T. S. BRITTON.

MOLDS, YEASTS, AND ACTINOMYCETES. A Handbook for Students of Bacteriology. By ARTHUR T. HENRICI, M.D., Professor of Bacteriology, University of Minnesota. Pp. x + 296. New York: John Wiley & Sons, Inc.; London: Chapman & Hall, Ltd. 1930. Price 17s. 6d.

Professor Henrici has written his handbook on general lines, but has, nevertheless, managed to place in it much detailed information of a varied and valuable kind. Designed as an introduction to mycology, to supplement and correct the insufficient and often inaccurate descriptions of fungi in bacteriological text-books, it meets a definite need and should be of real value to the bacteriologist who has had no previous training in mycology.

The opening chapters deal briefly, but adequately, with the classification, morphology, cultivation, and biological characters of fungi. Here the author's technique for slide cultivation is noteworthy for its simplicity and effectiveness.

The *Mucoraceae* are next considered in some detail, but the continuity of this chapter is interrupted by the insertion of Lendner's keys to the classification of *Mucor* and *Rhizopus*. Succeeding chapters on the *Ascomycetes* and the *Fungi Imperfecti*, the *Dermatophytes*, the *Moniliae*, and the Yeasts, are arranged in a similar manner. In my opinion, in order to preserve continuity of reading, all

systems of classification and sugar fermentation tables might advantageously be placed at the end of the appropriate chapter, or be grouped together in an Appendix. In these chapters short, concise accounts are given of most of the pathogenic fungi and the diseases they produce, but there is no mention of *Rhinosporidiosis*, and no reference is made to Pijper's study of *Bronchomoniliasis* or de Magalhaes' study of *Oidium Braziliense*. These omissions are not perhaps of serious importance, although the above-mentioned studies are widely quoted by workers on the *Moniliae*, and *Rhinosporidiosis*, though rare, is probably as common in the Far East as *Coccidioidal granuloma* is in the United States.

The last forty pages are devoted to a consideration of the *Actinomycetes*, upon which subject the author obviously writes with a knowledge born of careful and intimate study.

As a whole, the book is informative, easy to read, well-illustrated (many of the best illustrations are original), well-printed in clear type, and inexpensive. The author is to be congratulated upon the ability with which he has condensed so much useful information into so small a space.

J. F. D. SHREWSBURY.

THE QUANTITATIVE ANALYSIS OF INORGANIC MATERIALS. By NORMAN HACKNEY, B.Sc., F.I.C. Pp. xv + 378, with 28 illustrations. London: Charles Griffin & Co., Ltd. 1930. Price 30s.

The preface to this book states that the author's aim "has been to give none but sound, practical, and commercially accurate methods, treating the more important estimations in considerable detail, though it is hoped that in no case has any essential been omitted," and that he has "tried to cater primarily for the university student who is reading for his final degree examination." It is also to be inferred from the preface that here, at long last, is a book on quantitative analysis which sets out to remedy defects—some real and some not so real—in existing works, at any rate from the students' point of view. The promise of the preface cannot be said to be realised satisfactory in the text.

The book comprises nine sections. The first deals with apparatus and general manipulation in a thoroughly practical manner, and the page devoted to pulp filtration is very timely, since most books entirely overlook the advantages over paper filters which these pulp filters possess in a large number of cases, *e.g.* for barium sulphate, small quantities of a precipitate, etc.

The next section, "Theoretical Considerations," deals with the mass action law; the ionic hypothesis; the mechanism of precipitation and adsorption; acids, bases, salts; hydrolysis; solubility of salts in acids; theory of indicators; *p*H values, all in twenty pages. This kind of compressed theory is particularly dangerous in analytical work, and this is being realised more now than formerly; a little more scientific reasoning in connection with some of the processes given later in the book would have been preferable.

The other sections include the determination of the commoner metals, considered in the order of the qualitative groups; the determination of the anions (non-metals); a synopsis of volumetric analysis; the separation of the metals and of the anions preparatory to their determination by the methods of the earlier sections. The book concludes with a section dealing with a variety of materials of industrial importance—iron and steel, the chief non-ferrous alloys, refractories, a few ores, water and the like (50 pp.), a collection of useful tables (20 pp.), and it closes with an index.

The methods given are in all cases old favourites, but some of these, particularly sulphide precipitations, have been stretched to cover cases to which they do not properly apply. Nearly all of the valuable work of recent years in discovering sources of error in some of these processes and recommending improvements has been ignored; in fact, this book might well have been written at least 15 years ago when very much less was expected of the analyst. Most authors of text-books realise that they cannot give sufficient information on some points of possible importance to a reader, or that their acquaintance with some features of the subject is limited; they make up this deficiency and add greatly to the value of their books by giving references to the original literature. There is not a single reference given in this book; not even a date is mentioned to provide a clue.

Although it suffers from a super-abundance of sweeping statements and there are many inaccuracies, it must not be supposed that this book is without good points; it is well written, has a strong flavour of common sense about it, and is at least as good as any of the more usual text-books of similar scope.

S. G. CLARKE.

METALLURGY OF WHITE METAL SCRAP AND RESIDUES. By EDMUND RICHARD THEWS. London: Chapman & Hall. Price 25s. net.

This is a very well-written book, with relatively few misprints, is well produced in clear readable type, and is illustrated with excellent drawings. The book contains a great deal of interesting and useful matter on a subject which must necessarily become increasingly important in these days. The importance of sampling is emphasised throughout, and this subject is well treated.

The price, however, is too high; this is a common fault with many works dealing with metallurgical matters, and puts most of the useful books out of the reach of those whom they would benefit most. The purchase price of this one could be reduced substantially by omitting several chapters, the subject-matter of which can be found adequately treated in established text-books. The book thus condensed could still be very valuable as a treatise on the "Metallurgy of White Metal Scrap and Residues."

B. JONES.

QUANTITATIVE ORGANIC MICRO-ANALYSIS. By FRITZ PREGL. Translated by ERNEST FYLEMAN. 2nd English Edition. Pp. xiv + 232. With 51 illustrations. London: J. & A. Churchill. 1930. Price 15s.

This is a translation of the third German edition published earlier in the

year and already reviewed in THE ANALYST (1930, 304). The translation has been very ably carried out, and several slight errors in the former edition have been corrected. The new edition, which contains all the methods described in the old edition and considerable new matter, has been revised and improved. The most important addition is the method for the determination of the acetyl group. Modifications and improvements in the determinations of phosphorus, arsenic and mercury are described, and also a new automatic filtration apparatus for the barium sulphate precipitate and a new device for delivering pure carbon dioxide from the Kipp's apparatus for the micro-Dumas determination of nitrogen. The calculation example at the end of each section is a further improvement.

The only flaw in this most excellent book (a fault of the original German edition) is that the index is hardly detailed enough for the book when used for rapid reference in the laboratory. The English edition is much needed, owing to the growing interest in micro-chemistry in this country, and this remains the standard text-book of organic micro-analysis.

JANET W. BROWN.

COLLOIDS. By H. R. KRUYT. Translated by H. S. VAN KLOOSTER. Pp. xiii + 286. London: Chapman & Hall, Ltd. 1930. Price 17s. 6d. net.

The first edition of this book was reviewed in THE ANALYST (1928, **53**, 116), when the present reviewer gave the opinion "that the book is well suited as an introduction to the standard work by Freundlich." The early appearance of a second edition speaks well for the favour that Kruyt's volume has found.

Although enlarged by only twenty-four pages, the author has revised the text up to the level of 1929. Special revision has been made of the discussions dealing with the electric double layer. The distinction between charge and ζ potential is handled early in the text, and a much fuller account is given of the diffuse double layer. Considerable alterations have been made in the sections describing peptisation, flocculation and its kinetics (H. Müller's investigations) both for suspensoids and for emulsoids (coacervation), gelatinising, emulsions and other subjects.

The treatment of emulsions though more elementary, is similar to that of Ramsden, to whom reference should certainly have been made on p. 269.

The four parts of the book deal with: (1) General Introduction, (2) Suspensoids, (3) Emulsoids, (4) Special Cases. The treatment of emulsoids and of proteins, in particular, is excellent, and introduces the work of Kruyt's laboratory in convincing fashion.

Altogether, as an elementary text-book, very readable and accurate, it can be recommended with every confidence. Even advanced students of colloid physics will find its reading refreshing and stimulating. The printing and binding leave nothing to be desired.

WILLIAM CLAYTON.

PLANT BIOLOGY: AN OUTLINE OF THE PRINCIPLES UNDERLYING PLANT ACTIVITY AND STRUCTURE. By H. GODWIN, M.A., Ph.D. Cambridge University Press. 1930. Price 8s. 6d. net.

Dr. Godwin's book is to be welcomed as a fresh and stimulating account of plant life. Intended primarily as a text-book for medical students, it may well appeal to a wider audience. It might, with advantage, be used in schools for supplementary reading after the matriculation standard has been reached.

Early in the book the physico-chemical background of vital phenomena is dealt with in chapters on the chief organic compounds and on crystalloids and colloids. These are linked up with sections on the cell, general metabolism, and photosynthesis. This part of the book is particularly successful. The picture of the activities thus given is extended in the chapters on yeasts, fungi and bacteria, and in these the possibilities of applications to medicine, agriculture and various industries are made clear. The remainder of the book deals with other types of plant life. The green algae provide material for the treatment of sexual and asexual reproduction. A detailed study of Fucus gives an account of the building up of a complex soma and of its relations to the environmental conditions in which the plant lives. Fucus and Funaria serve to introduce the differentiation of tissues. The final chapters deal with the structure and functions of the flowering plant.

There is much to be said for using the less familiar groups of plants as the main basis of an account of plant life. When only a short time is available for the study of botany this method does allow a better idea of the diversity of plant life to be combined with an understanding of fundamental questions. Yet it seems a pity that the more obvious and familiar biological features of the flowering plants—the biology of the flower, the fruit and the seed—representing as they do the climax activities of the plant kingdom, should be entirely neglected. This is, of course, a question of choice, and when space and time are limited something must go.

Certain minor criticisms might be made. The word "karyokinesis" might well be dropped. Loftfield, as well as Lloyd, should be considered in an account of stomatal function. Here and there the mention of a topic is so brief as to have little value, *e.g.* the reference to magnesium in chlorophyll on p. 79 and to the fraction of light utilised in photosynthesis on p. 82. These are small matters, and the main impression left after reading this book is that of an original and interesting account of plant life. The illustrations are bold and informative.

M. SKENE.