

1953
10.11.53

THE ANALYST

A Monthly Publication
dealing with all branches
of Analytical Chemistry:
the Journal of the Society
of Public Analysts and
Other Analytical Chemists

Editor: F. L. OKELL, F.R.I.C.

7-8, IDOL LANE, LONDON, E.C.3

Telephone: MANsion House 6608

Published for the Society by

W. HEFFER & SONS LTD., CAMBRIDGE, ENGLAND

Volume 78

Price 6s 6d

Subscription Rate, inclusive of Abstracts C, 80/- per annum, Post Free

No. 933, Pages 685-740

December, 1953

WHATMAN FILTER PAPERS

STOCK SIZES: Circles from 4.25 — 62.25 cms. diameter Standard sheets, $18\frac{1}{8}" \times 22\frac{1}{2}"$, but other sizes can be supplied to order.

FOR MICRO-ANALYTICAL WORK: Smaller circles from 1.3 to 4.25 cms. diameter.

CHROMATOGRAPHY: Specially selected papers in any of the above sizes; also in reels from $1\frac{1}{8}" \times 100$ yds. and strips of any desired resonable width and length.

Made by

W. & R. BALSTON LTD., Maidstone, Kent

Sole Mill Representatives

H. REEVE ANGEL & CO., LTD., 9, Bridewell Place, London, E.C.4



A group of Alumina 609 Mortars and Pestles

MORTARS and PESTLES

Analysts everywhere need these new Thermal Alumina 609 mortars and pestles. Suitable for grinding extremely hard materials, they have an octagonal shape, and four different sizes are available.

THE THERMAL SYNDICATE LTD

Head Office: Wallsend, Northumberland.

London Office: 12-14 Old Pye Street, S.W.1.

IMPORTANT NOTICE TO SUBSCRIBERS

(Other than Members of the Society)

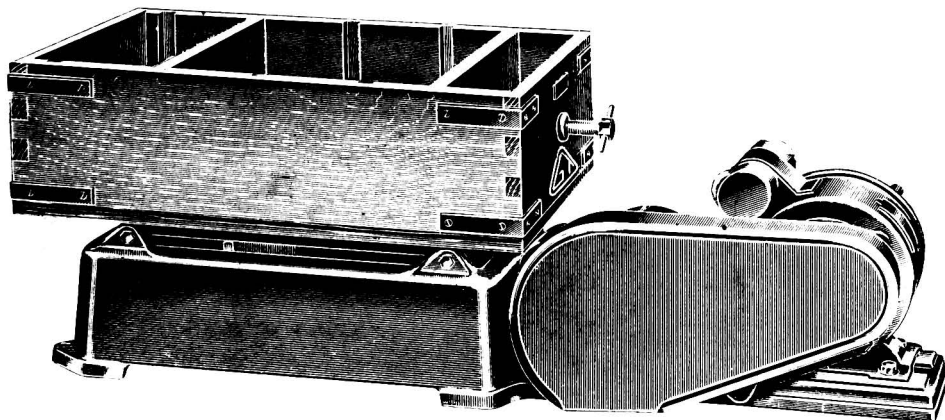
All Subscriptions and renewals to the *Journal*, including Abstracts C., should be sent through a Bookseller or direct to

W. HEFFER & SONS LTD., CAMBRIDGE, ENGLAND.

Price 6 6, postage 2d.; or to Subscribers in advance post free £4 per annum

N.B.—Members send their subscriptions to the Hon. Treasurer

G and T Silent Shaker



B.45-470

200/250 Volt. A.C. 50 cycles

Price £47 10s. 0d.

This Shaker was the first really good bottle shaker and Kahn-Test Agitator.

Before this shaker was offered to the market there was nothing to compare with it, and it is still in advance of anything else of its kind. You will be glad that you bought it. It takes any bottles up to two Winchester Quarts—shaking simultaneously, of course.

GRIFFIN AND TATLOCK LTD

LONDON

Kemble Street, W.C.2

GLASGOW

45, Renfrew Street, C.2

MANCHESTER

19, Cheetham Hill Road, 4

SHEFFIELD

95, Clarkehouse Road, 10



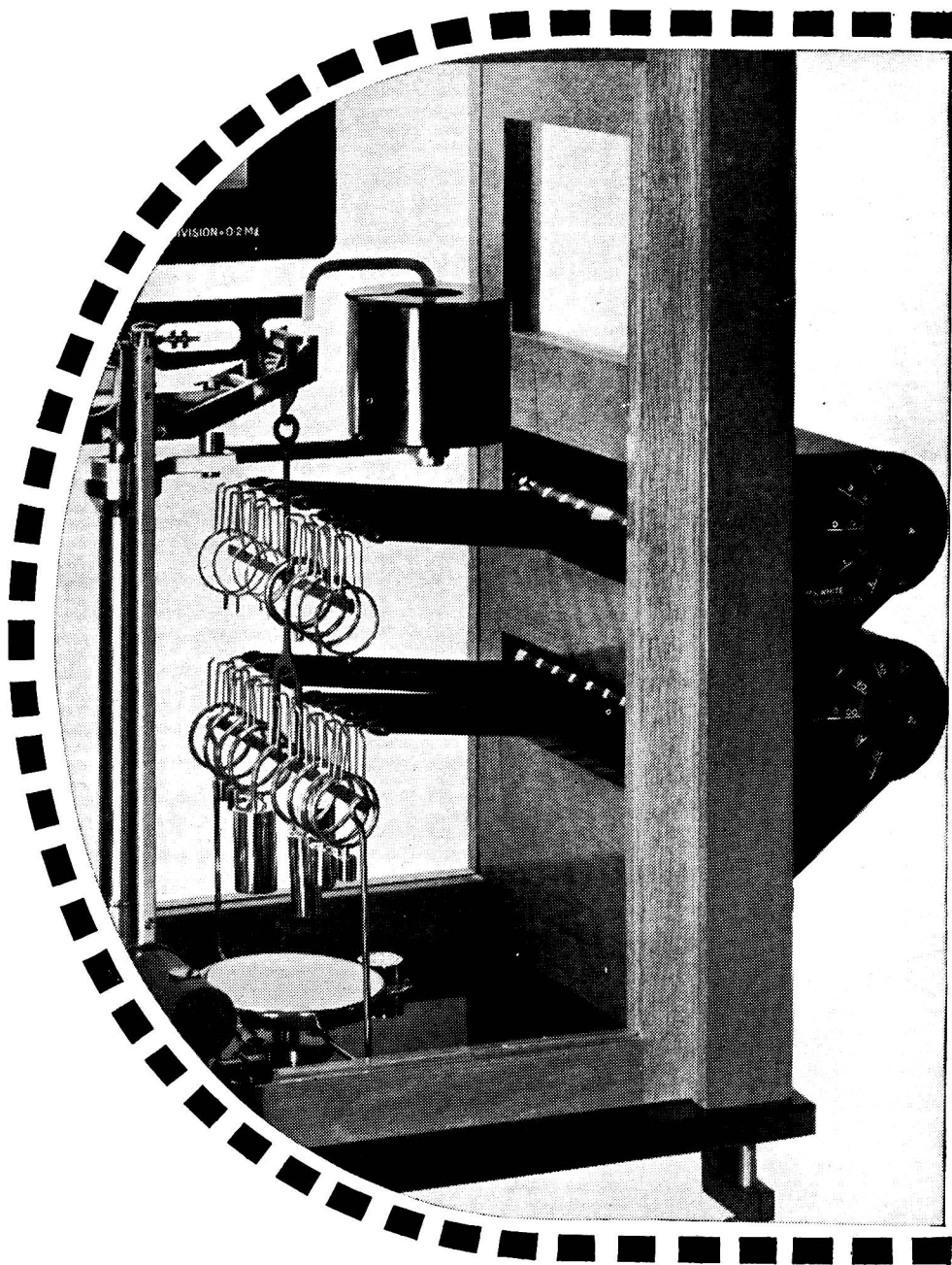
EDINBURGH

8, Johnston Terrace, 1

BIRMINGHAM

Standley Belcher & Mason, Ltd., Church St., 3

When ordering please mention this Journal



BUILT BY THE PEOPLE WHO KNOW BALANCES

***L. Oertling Ltd., Cray Valley Road, St. Mary Cray,
Orpington, Kent.***

Telephone: Orpington 5771 (5 lines)

PIONEERS IN BALANCE DESIGN FOR OVER 100 YEARS

200 GRAMS AT YOUR FINGER TIPS !

★ **TWO KNOBS ONLY** control automatic weight loading from 100 mg. to 200 grams.

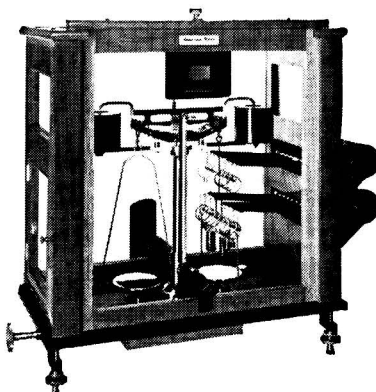
★ 0-100 mg. read direct from illuminated scale with 500 divisions. Each division represents 0.0002 grams.

★ Weights are adjusted to NPL Class A tolerances.

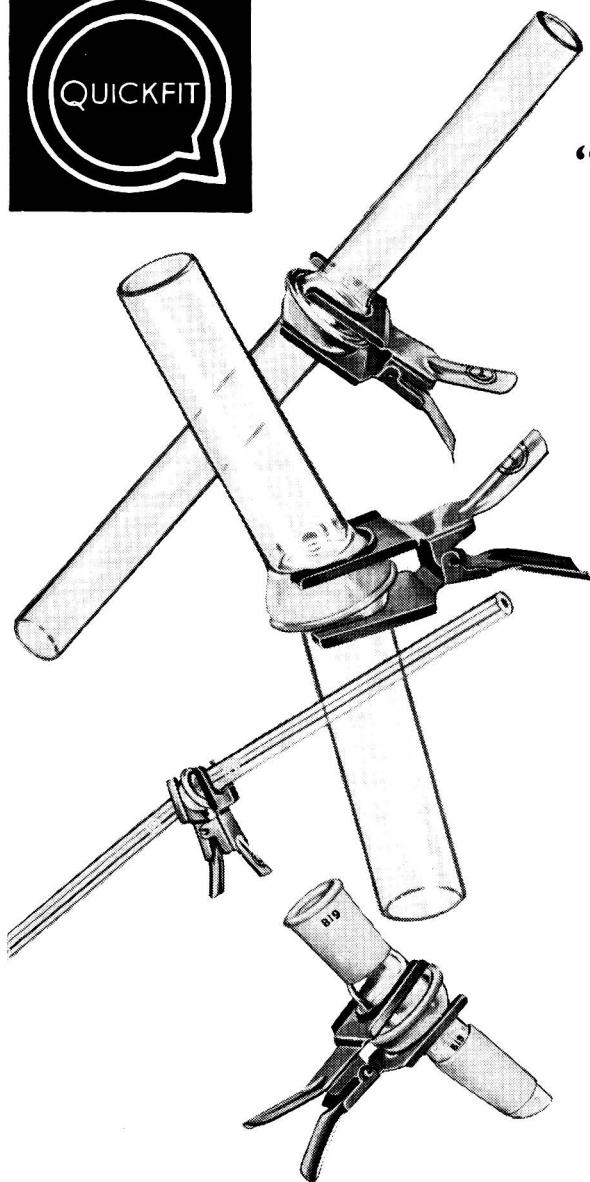
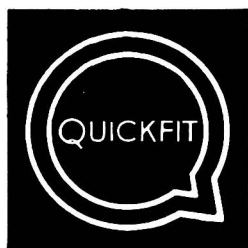
★ Ample room on right-hand pan for tare when required.

**PLUS *RELEAS-O-MATIC* for
SAFE WEIGHING!**

MODEL 126 A fine precision instrument that you can afford to use for everyday routine work! Knife edges, fully protected by *RELEAS-O-MATIC*, cannot be damaged by quick release — hence **PRECISION** combined with **GREAT SPEED**. Write now for full specification.



Oertling



“QUICKFIT” Interchangeable SPHERICAL Ground Glass Joints

The use of one or two Spherical Joints in a conical jointed assembly of glass apparatus provides a useful degree of flexibility. Such joints also overcome problems of seizure, and are, for example, extremely useful in complex high vacuum lines. The reliable “Quickfit” standard of grinding accuracy and interchangeability is maintained to the full in “Quickfit” Spherical Joints. Conical-Spherical adaptors are available to simplify the insertion of a Spherical Joint into assemblies of standard glassware.

QUICKFIT & QUARTZ LTD

INTERCHANGEABLE LABORATORY GLASSWARE

Orders and Enquiries to

Dept. O.P., “Quickfit” Works, Heart of STONE, Staffs

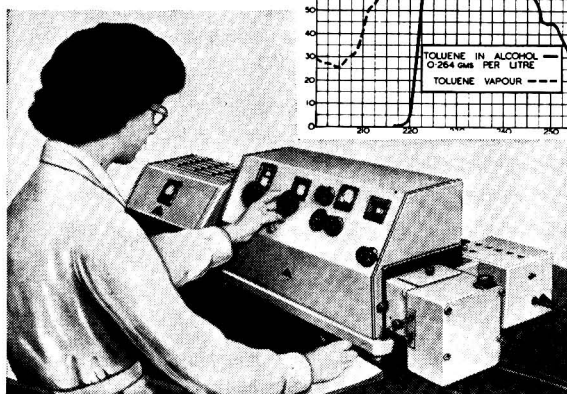
Phone: Stone 481

UNICAM

SPECTROPHOTOMETERS

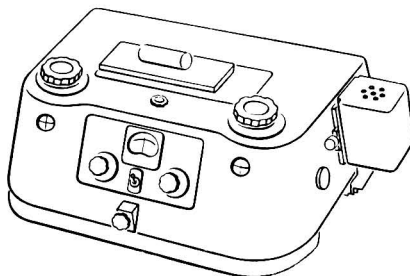
SP.500

The well-known Unicam Quartz Spectrophotometer makes possible analysis by light absorption measurement at all wavelengths from $2,000\text{\AA}$ to $10,000\text{\AA}$. The quality of its performance and simplicity of operation combine to make the instrument ideally suitable for research, process control or routine determinations.



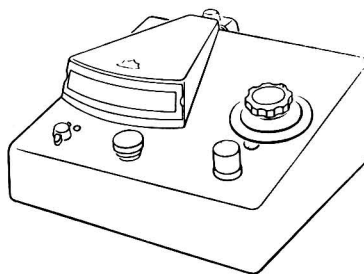
SP.600

This new spectrophotometer offers at moderate price the many advantages of up-to-date analytical methods. The high resolution of the glass prism monochromator combined with a reliable electronic system gives exceptional performance in the visible range ($3,600\text{--}10,000\text{\AA}$).



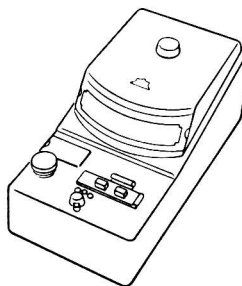
SP.350

Chemists in clinical or biochemical laboratories appreciate the diffraction grating fitted in this spectrophotometer which allows them to follow an end point in a changing system. A use will be found for this reliable instrument in almost every laboratory.



SP.300

The demand for a simple, robust filter-type colorimeter giving a consistently high performance over long periods is amply met by the Unicam SP.300 Colorimeter. New applications are continually being found for this reliable, inexpensive instrument that is strongly built and easy to operate.



Illustrated leaflets describing any of these instruments in detail will gladly be sent on request.



Complexones

These useful reagents, first developed by G. Schwarzenbach, are being extensively employed in analytical and applied chemistry. Their valuable property of sequestering alkaline earth and other metals is no doubt capable of further development. The following members of the series are now available :—

Anthranilic acid diacetic acid

Iminodiacetic acid

Methyliminodiacetic acid

Nitrilotriacetic acid

Propylenediamine tetra-acetic acid

Sequestic acid (*Ethylenediamine tetra-acetic acid*)

Uramldiacetic acid

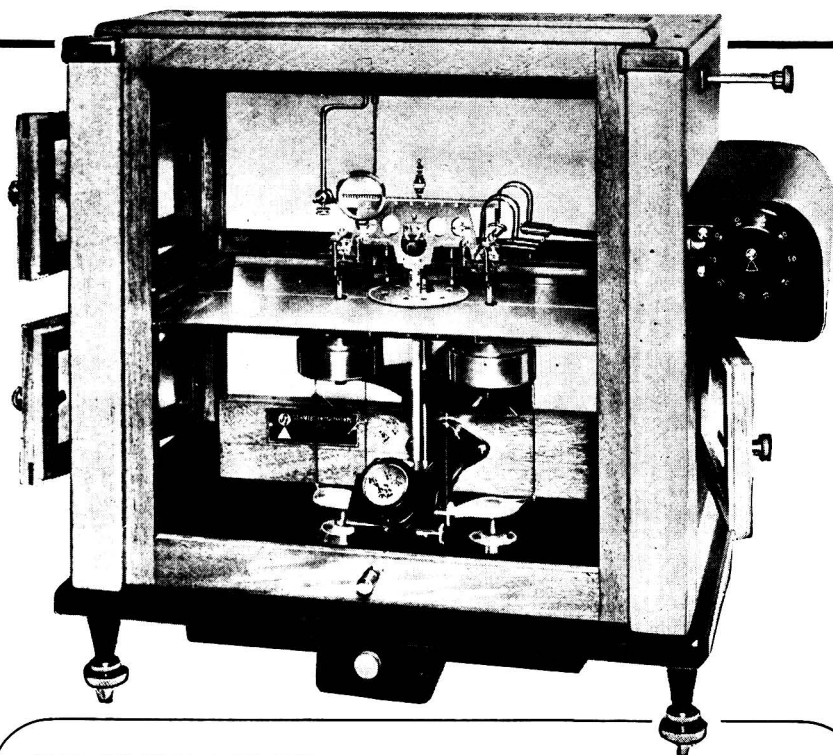
Hopkin & Williams Ltd. will send free on request a monograph of the analytical uses of Sequestic Acid, and also welcome enquiries for further compounds of the series.

HOPKIN & WILLIAMS LTD

Manufacturers of pure chemicals for research and analysis

FRESHWATER ROAD, CHADWELL HEATH, ESSEX

Britain's Finest Micro-Balance



STANTON *Aperiodic Micro-Chemical Balance*

Capacity 20g. Sensitivity 0.01 mg.

Graticule Readings can be taken over a range of 1 mg.

100 divisions either side of a central zero, each division representing 0.01 mg. and estimation to 0.005 mg. can be carried out without difficulty.

**Model
M.C.I.A.**

Fitted with a weight loading attachment up to 0.1 g.

Has a built in cut-out switch, green filter, absorption tube supports and a separate beam chamber.

Stanton serves the Laboratories of the World



STANTON

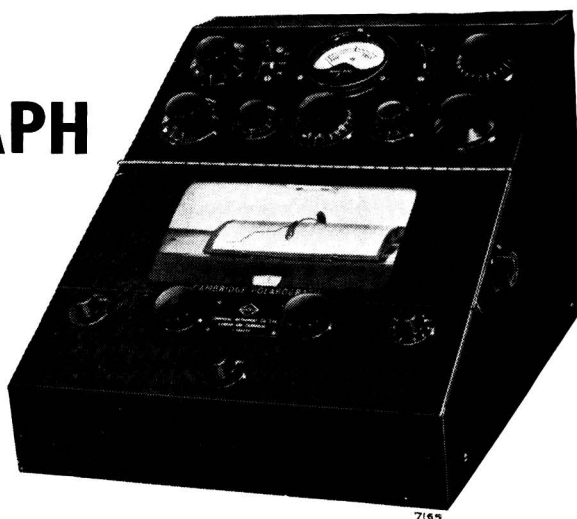
PRECISION BALANCES

Stanton Instruments Ltd., 119 Oxford St., London, W.1. Tel. Gerrard 7533/4

CAMBRIDGE POLAROGRAPH

Direct Writing Model

For the rapid electro-chemical analysis of solutions by the Heyrovsky dropping mercury electrode method, enabling traces of metals and many acid radicals and organic substances to be determined. The polarograms produced are of exceptional legibility, and are immediately available for examination.

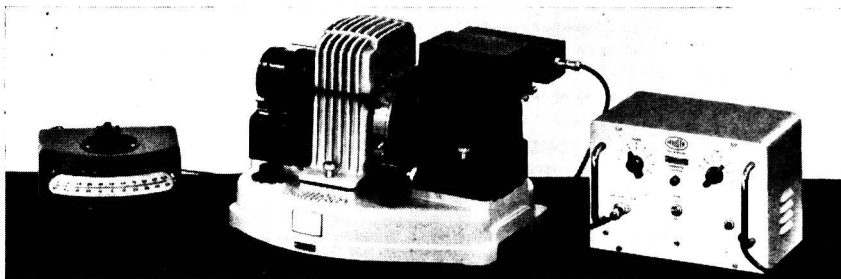


Write for **SHEET 302-U**

Details of the UNIVECTOR polarograph attachment, giving simpler records with up to 20 times the sensitivity, are given in SHEET No. 313-U

CAMBRIDGE INSTRUMENT COMPANY LIMITED

13 GROSVENOR PLACE - LONDON - SW1



A New and Sensitive Fluorimeter

Made by

HILGER & WATTS LTD.
HILGER DIVISION
98 ST. PANCRAS WAY
LONDON, N.W.1

The sensitivity of fluorimetric determination is many times increased by the new design of Hilger Spekker Fluorimeter with photomultiplier cell. A compact and simple mains-operated power supply provides all power requirements.

Please write for full description.

Member of the Scientific Instrument Manufacturers' Association and of SCIEX

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

AN Ordinary Meeting of the Society was held at 7 p.m. on Wednesday, October 7th, 1953, in the Meeting Room of the Chemical Society, Burlington House, London, W.1. The Chair was taken by the President, Dr. D. W. Kent-Jones, F.R.I.C., and about 170 members and visitors were present.

At this meeting there was a discussion on "Destruction of Organic Matter." The subject was introduced by Dr. G. Roche Lynch, O.B.E., M.B., B.Sc., D.P.H., F.C.G.I., L.M.S.S.A., F.R.I.C., and the following papers were presented and discussed: "The Preparation of Biological Material for the Determination of Trace Metals. Part II. A Method for the Destruction of Organic Matter in Biological Material," by G. Middleton, B.Sc., F.R.I.C., and R. E. Stuckey, B.Sc., Ph.D., F.R.I.C., Ph.C.; "Determination of Lead in Foodstuffs," by H. C. Lockwood, Ph.D., F.R.I.C.

NEW MEMBERS

Raymond Leslie Bass, B.Sc. (Lond.), A.R.I.C.; David Richmond Brown, B.Sc. (Lond.), A.R.I.C.; James Dennis Burton, B.Sc. (Lond.), A.R.I.C.; Denys Irvine Coomber, B.Sc., Ph.D. (Lond.), A.R.I.C.; John Mervyn Davies, B.Sc. (Wales); Guillermo V. Freile Gagliardo, B.Biol.Sci. (Rocafuerte), D.Chem. (Guayaquil); Marian Norden Gibbons, B.Sc. (Lond.), A.R.I.C.; Cecil Alfred Johnson, B.Pharm., B.Sc. (Lond.), Ph.C., A.R.I.C.; John Edwin Morrison Moxley, B.A. (Oxon.); Stanley Newton, B.Sc. (Lond.); Peter Stross, B.Sc. (Lond.).

DEATH

WE regret to record the death of

William Charles Hughes

Micro-analysis of Silicate Rocks

Part IV. The Determination of Alumina

By CHRISTINA C. MILLER AND ROBERT A. CHALMERS

(Presented at the meeting of the Society in Glasgow on Wednesday, May 6th, 1953)

A new procedure is prescribed for the separation and direct determination of alumina in 5-mg samples of silicate rocks. Silica is volatilised by heating with hydrofluoric and sulphuric acids, and the residue is fused with potassium bisulphate and extracted with *N* hydrochloric acid. Iron, titanium, vanadium and zirconium are removed together by precipitation with cupferron and extraction with *o*-dichlorobenzene. Acetylacetone is added to the aqueous phase, and aluminium and beryllium acetylacetonates are extracted from the buffered solution at a pH of 6 to 7 by means of diethyl ether. From the ether extract aluminium and beryllium are withdrawn into 6 *N* hydrochloric acid, and the aluminium alone is precipitated and weighed as aluminium 8-hydroxyquinolate.

The method has been applied in the presence of all the elements commonly found in silicate rocks. In the analysis of rocks, the results obtained are relatively 1 per cent. lower than those based on the classical procedure.

IN the analysis of silicate rocks by the classical procedure, the component determined with the least certainty, and only after a considerable expenditure of time, is alumina. Its indirect determination depends on the weight of seldom fewer than five oxides ("mixed oxides") and the separate determination of all except alumina, which is then found by difference. On the micro-scale the inherent difficulties and uncertainties of the method are increased. Slow-filtering hydroxide precipitates can be very troublesome, and the hygroscopicity of alumina, and the many high-temperature ignitions, which significantly affect the weight of platinum crucibles, are sources of error. In microchemical work especially, one would like to abolish the hydroxides precipitation and also determine alumina more directly. Hitherto, a potent reason for retaining the precipitation of the hydroxides has been that their ability to carry down some residual silica has enabled the determination of silica to be completed. We have, however, recently proposed a method for the direct determination of total silica in a separate portion of rock.¹ Precipitation of the hydroxides is not essential for the subsequent determination of iron and titanium, which are commonly determined at this stage although they are easily determined separately, but it facilitates the determination of calcium and magnesium.

Early attempts to avoid the precipitation of the hydroxides in micro-analyses of silicate rocks were made by Schoklitsch² and by Hecht,³ both of whom freed the rocks from silica, fused the residues with potassium bisulphate, precipitated together the 8-hydroxyquinolates of iron, aluminium and titanium, and from them obtained, after destruction of organic matter, a solution of metal chlorides. Schoklitsch then precipitated and removed by filtration the cupferron complexes of iron and titanium, destroyed organic matter in the filtrate and precipitated aluminium with 8-hydroxyquinoline. Hecht, on the other hand, precipitated iron as iron sulphide in an ammoniacal tartrate solution, thus separating it from aluminium and titanium, which were then precipitated together and weighed as the 8-hydroxyquinoline complexes. After a somewhat complicated procedure, he then determined titanium as titanium dioxide and found alumina by difference.

8-Hydroxyquinoline can certainly be used to simplify the determination of alumina in silicate rocks of low complexity. McLennan⁴ jointly determined iron, aluminium and titanium (TiO_2 , 2 per cent.) gravimetrically as their 8-hydroxyquinolates with a positive error of less than 0.5 per cent. One to 2 per cent. of calcium oxide and magnesium oxide, 0.5 per cent. of phosphoric oxide and 0.2 per cent. of manganous oxide were without influence at pH 5, but larger amounts of manganous oxide divided, and large (10 per cent.) amounts of calcium and magnesium oxides caused significant positive errors. As various minor constituents would accompany aluminium, one may conclude that the substitution of 8-hydroxyquinoline for ammonia is not in itself the solution of the problem when rocks of greater complexity are under consideration.

The object of our investigation was therefore to devise a method for the direct determination of alumina in 5-mg samples of more complex silicate rocks, preferably one that would not prevent the subsequent determination of calcium and magnesium, and perhaps manganese, in the same sample. The following method has been evolved.

After volatilising silica by heating with hydrofluoric and sulphuric acids, and fusing the residue with potassium bisulphate, we have quantitatively removed iron and titanium and some other elements from a *N* hydrochloric acid solution of the melt by means of cupferron and *o*-dichlorobenzene. Aluminium has then been converted into the acetylacetone complex at pH 6 to 7 and extracted quantitatively with diethyl ether, from which it has been withdrawn into 6 *N* hydrochloric acid and, after suitable adjustment of the conditions, precipitated and weighed as aluminium 8-hydroxyquinolate. The method has been applied to some silicate rocks.

EXPERIMENTAL

QUANTITATIVE SEPARATION OF IRON, TITANIUM AND SIMILAR ELEMENTS FROM ALUMINIUM BY MEANS OF CUPFERRON—

Cupferron is frequently used in conjunction with an organic solvent for removing iron^{III} and titanium^{IV} from mineral acid solutions before determining small amounts of aluminium. When aluminium is a major component, care is required because aluminium cupferronate is extractable from solutions of low acidity. Hence the acid concentration should be reasonably high and the concentration of cupferron and the volume of organic solvent should be the minimum that permits the complete removal of iron, titanium and similar elements. The efficiency of the common solvents, diethyl ether, carbon tetrachloride, chloroform and benzene for separating iron and aluminium in *N* hydrochloric acid solution was barely adequate, and superior results were obtained with *o*-dichlorobenzene. In order to facilitate separation of the liquid phases and to minimise washings, specially designed stoppered centrifuge tubes (Fig. 1A), made water-repellent by treatment with a silicone solution, were used. The wide necks were required to prevent loss when the silicone-treated stoppers were rinsed into the tubes. As it was impossible on the micro-scale to judge when the formation of cupferronates was complete, and slight loss of aluminium occurred when more than 2 to 3 mg of cupferron in excess of theoretical requirements was used, it was necessary for maximum accuracy to know the approximate amounts of iron and titanium present, so that the quantity of cupferron added could be controlled. For maximum extraction of iron a shaking time of 2 minutes was required to overcome a slight holding effect of potassium bisulphate. Under the conditions prescribed below, about 2 parts per 1000 of iron^{III} were left in the aqueous phase.

As iron^{II} was only 85 per cent. extracted, and some is formed when hydrochloric acid solutions of bisulphate melts containing iron^{III} are held in platinum, it was necessary to limit the period of contact at 100° C to 5 minutes, or else to re-oxidise the solution before extracting. Titanium (TiO₂, 250 μg), zirconium (ZrO₂, 50 μg) and vanadium (V₂O₅, 25 μg) were completely transferred to the organic phase, whereas calcium (CaO, 500 μg), magnesium (MgO, 500 μg), manganese (MnO, 50 μg), beryllium and cerium (BeO or Ce₂O₃, 25 μg) and nickel, cobalt, chromium and platinum (NiO, CoO, Cr₂O₃ or PtO₂, 300 μg) were completely held in the aqueous layer. The presence of phosphate (P₂O₅, 150 μg) had no significant effect on the results.

QUANTITATIVE SEPARATION OF ALUMINIUM FROM CALCIUM, MAGNESIUM AND SOME OTHER ELEMENTS BY MEANS OF ACETYLACETONE—

In the analysis of potable waters, Stene⁵ used acetylacetone and extraction with carbon tetrachloride or chloroform to separate aluminium, iron, beryllium, cerium and copper from calcium, magnesium, manganese, titanium, cobalt, nickel, zinc, uranium and so on, which were not extracted at a pH of 4.5 to 7.5. Abrahamczik⁶ similarly separated iron, aluminium and manganese in weakly alkaline solution from magnesium and calcium. If, in silicate rock analyses, iron, titanium, vanadium and zirconium were initially removed by means of cupferron, then it seemed that aluminium might be separable from calcium and magnesium, which were the main elements to be considered at this stage, and also, by suitable control of pH, from manganese and various other subsidiary elements, excluding beryllium.

Some experiments were first made on the precipitation of aluminium with other diketones and extraction with other solvents. A reagent superior to acetylacetone was not found,

but diethyl ether proved to be a more efficient solvent. Mechanical loss was again minimised by the use of appropriately designed extraction tubes, as shown in Fig. 1B. Water-repellent coatings were not permissible because of the solvent action of ether, and therefore the necks of the tubes could be narrower than before.

The effect of variations in pH on the extraction of aluminium acetylacetonate, under conditions comparable to those prescribed on p. 691, was examined for 1-mg amounts of alumina in the presence of 100 mg of potassium bisulphate. After the complex had been given time to form and the pH had been measured with a pH meter, the three extractions with ether were made and the residual aqueous layer was tested by means of aurine-tricarboxylic acid. The results, which are shown in Fig. 2, indicate that extraction is essentially complete over the pH range 6 to 7.

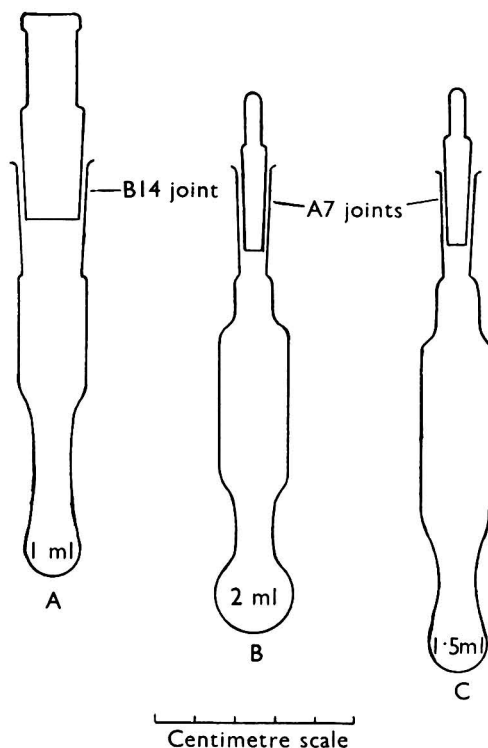


Fig. 1. Extraction tubes

The behaviour of several other elements in the procedure was investigated at a pH of 6 to 7. Calcium and magnesium (CaO or MgO , 500 μg), manganese (MnO , 50 μg), nickel and platinum (NiO or PtO_2 , 250 μg) and cerium (Ce_2O_3 , 25 μg) were not significantly removed by acetylacetone and ether. About 3 per cent. of cobalt and chromium (CoO or Cr_2O_3 , 300 μg) were extracted by ether. These elements are minor components of rocks, and would not be expected to interfere. Beryllium accompanied aluminium. The presence of phosphate (P_2O_5 , 150 μg) caused no obvious precipitation with any of the above elements except cerium and chromium. No loss of aluminium by co-precipitation with phosphate precipitates need be feared with the small amounts of these elements likely to be present in silicate rocks.

FINAL DETERMINATION OF ALUMINIUM AS ALUMINIUM 8-HYDROXYQUINOLINATE—

Attempts made to determine aluminium (in absence of beryllium) as aluminium acetylacetonate simply by evaporating the ethereal extract containing it and then heating the residue at 120° C were not a success, apparently because slow volatilisation of the metal complex occurred. The diketone had to be broken down by treating the ethereal extracts with acid and restoring the aluminium to the aqueous phase for determination. If it were precipitated with 8-hydroxyquinoline in a solution containing acetic acid, no interference

from beryllium would result. A reagent superior to 8-hydroxyquinoline for the determination of aluminium was not found.

Quantitative disruption of aluminium acetylacetonate was effected by shaking the ethereal solution vigorously for 6 minutes with 6 *N* hydrochloric acid in extraction tubes of the special type shown in Fig. 1C. Thereafter it was immaterial whether the ether was removed by evaporation in a current of air or by direct withdrawal after separation of the phases by centrifugation.

Much has been published on the determination of aluminium by means of 8-hydroxyquinoline. Its determination in faintly acid solution was required, so that, if beryllium were present, or small amounts of calcium and magnesium inadvertently accompanied aluminium,

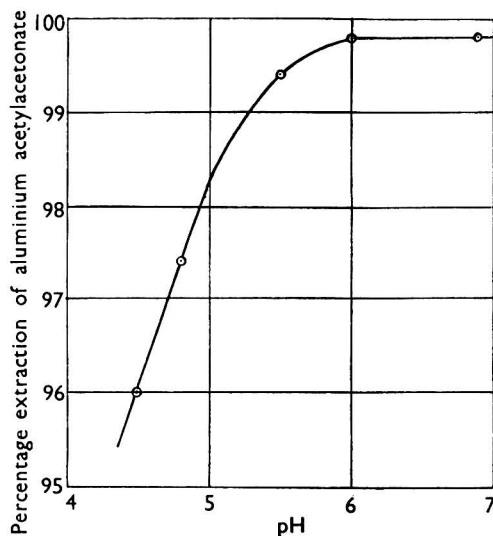


Fig. 2. Effect of pH value on the extraction of aluminium acetylacetonate with ether

they would be prevented from interfering. Got⁶⁷ gave the range of complete precipitation of aluminium 8-hydroxyquinolate as pH 4.15 to 9.80. Borrel⁸ showed that precipitation began at pH 3.85 and was quantitative at pH 4.7. The Chemical Analysis Sub-Committee of the British Ceramic Research Association⁹ obtained consistent results in the determination of aluminium over the pH range 4.80 to 5.14, and results distinctly lower below pH 4.48. As positive errors were prevalent in our early work, it was necessary to examine the conditions of the precipitation somewhat fully.

It is customary to add an excess of an acetic acid solution of 8-hydroxyquinoline to the slightly acid solution containing aluminium and to effect precipitation in the heated solution by adding an excess of ammonium acetate to raise the pH appropriately.

In experiments made with 13 to 57 mg of alumina in a final volume of 120 ml, the pH after precipitation could be varied from 4.4 to 6.7 without significant effect, and it mattered not whether the solution was filtered hot or cold. Too great a rate of addition of ammonium acetate solution caused significant positive errors, the percentage errors for the same amount of added 8-hydroxyquinoline being greater for smaller amounts of alumina. When ammonium acetate solution was added very slowly, dropwise with constant stirring, the precision of the results was greatly improved, as has also been shown by Stumpf.¹⁰ The main feature of the results was a roughly linear relationship, as shown in Fig. 3, between the weight of the excess of 8-hydroxyquinoline used and the percentage error in the weight of precipitate, from which it was deduced that a 250-mg excess was required.

On the micro-scale, one-thirtieth of all quantities was required and an excess of 8 mg of 8-hydroxyquinoline was indicated. Despite the fact that the ammonium acetate solution was added extremely slowly from a fine-tipped horizontal micro-burette¹¹ while the solution was vigorously stirred by a magnetic stirrer, an average error of +0.2 per cent. was incurred

in determining alumina. Reduction of the excess of 8-hydroxyquinoline to 6 mg eliminated the error, under the conditions of precipitation described on p. 691. The average pH of the solution after precipitation was about 4.7. The presence of a little acetylacetone did not affect the results.

METHOD

APPARATUS—

Use Pyrex or similar glassware throughout.

REAGENTS—

Reagents should as far as possible be of recognised analytical quality.

o-Dichlorobenzene—Distil technical grade *o*-dichlorobenzene before use and collect the fraction that boils between 177° and 178° C.

Diethyl ether—Free the ether from peroxide and distil.

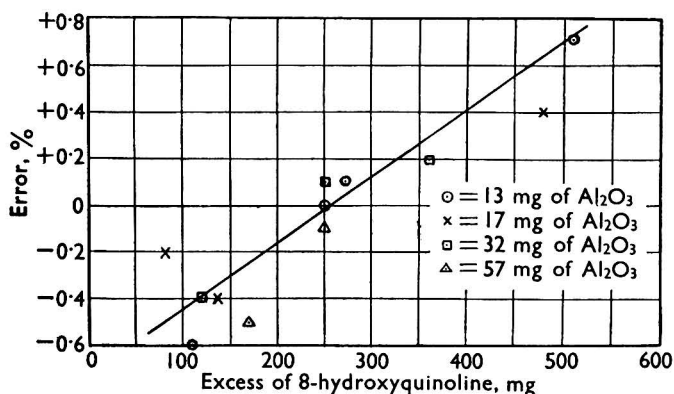


Fig. 3. Relationship between weight excess of 8-hydroxyquinoline and percentage error

Acetylacetone solution—Distil acetylacetone and collect the fraction boiling between 136° and 138° C. Mix 10 ml with 20 ml of ethanol and 70 ml of water.

Cupferron solution—Prepare freshly a cold, 5 per cent. w/v aqueous solution.

8-Hydroxyquinoline solution—Prepare a 2.5 per cent. w/v solution in 4 per cent. v/v acetic acid.

Ammonium hydroxide, 6 N—Prepare from cylinder ammonia and store in a polythene bottle.

Hydrochloric acid, 6 N—Prepare by distillation from the concentrated acid.

PROCEDURE FOR ROCKS—

With the aid of a stoppered weighing stick, weigh into a 1-ml platinum crucible about 5 mg of the dried material. Add 0.15 ml of 40 per cent. hydrofluoric acid and then, 5 minutes later, 0.15 ml of 10 N sulphuric acid. Evaporate to dryness on a steam-bath and cautiously expel sulphuric acid over a micro-bunsen flame. Next fuse the residue with 100 mg of potassium bisulphate, cool, place the crucible in a small porcelain capsule, and dissolve the melt in 1 ml of N hydrochloric acid. Heat on a steam-bath for *not more than 5 minutes* and transfer the solution, by means of a fine capillary attached to a suction apparatus, to a silicone-coated centrifuge tube of the type shown in Fig. 1A. Wash both capsule and crucible four times with 0.25-ml portions of N hydrochloric acid and finally rinse the end of the capillary with a drop of acid. Centrifuge the contents of the tube and proceed with the cupferron treatment.

Add to the solution 0.05 ml more cupferron solution than is theoretically required for removal of iron, titanium, vanadium and zirconium (0.1 ml \equiv 0.8 mg of Fe₂O₃) and 1 ml of *o*-dichlorobenzene. Shake the stoppered tube for 2 minutes at the rate of four shakes per second, then unstopper it, centrifuge and transfer the aqueous layer via a micro-filter and capillary to the appropriate centrifuge tube, Fig. 1B. The filter removes a slight opalescence that sometimes appears in the aqueous phase. Rinse the stopper into the extraction tube with 0.5 ml of N hydrochloric acid, insert it, and shake the tube for 15 seconds

before centrifuging and removing the upper layer as before. Rinse the stopper again with 0.5 ml of acid, but do not shake the tube. Centrifuge and remove the acid. Finally wash the walls with 0.5 ml of acid, centrifuge and remove the acid.

Evaporate the solution containing the aluminium in a current of air, while heating the centrifuge tube in a bath of boiling water, until the volume is about 0.5 ml. Shake the tube to dissolve separated solids and add 0.5 ml of acetylacetone solution, followed by 0.5 ml of 5 *N* ammonium acetate solution and bromocresol purple as indicator. Mix, and then add 6 *N* ammonium hydroxide until the indicator shows blue-green (in daylight), or grey-green if some aluminium acetylacetonate precipitates. Rinse the neck of the tube with 0.1 ml of water, allow 5 minutes for complex formation and then readjust the colour of the indicator, if necessary. Add 3 ml of ether, moisten the stopper of the tube with a drop of water and insert it. Shake the tube for 3 minutes as before, unstopper it, centrifuge and transfer the upper layer by slow suction via a dry capillary to the third centrifuge tube of the type shown in Fig. 1C. Rinse the stopper into the extraction tube with 3 ml of ether and proceed as before. Repeat with 1 ml of ether and shaking for 30 seconds and then with 0.25 ml and no shaking.

To the combined ether extracts add 1 ml of 6 *N* hydrochloric acid and moisten the stopper of the tube with a drop of acid. Shake the stoppered tube as usual for 6 minutes and then remove and rinse the stopper with a little water into a 6-ml beaker. Rinse down the neck of the extraction tube with 6 drops of 6 *N* hydrochloric acid, centrifuge and discard the ether layer. Place the tube in a bath of cold water and gradually heat to boiling, while a slow current of air plays on the surface of the acid and removes residual ether. Centrifuge and transfer the contents by capillary to the 6-ml beaker. Wash the tube three times with 0.75-ml portions of water, always centrifuging briefly before transferring the liquid to the beaker.

Evaporate the solution in the beaker to about 1 ml, and adjust the pH to about 3 by means of 6 *N* ammonium hydroxide and dilute hydrochloric acid, using bromophenol blue as indicator. Add 0.25 ml more of the 8-hydroxyquinoline solution than is theoretically required for aluminium (0.5 ml \approx 1.5 mg of Al_2O_3) and, if a precipitate forms, just redissolve it with a minimum of 0.2 *N* hydrochloric acid. Dilute the solution to about 4 ml., heat to 80° to 90° C and, while vigorously stirring it by means of a magnetic stirrer, add 1 ml of 2 *N* ammonium acetate solution *very slowly* from a horizontal burette. Next digest the precipitate on a steam-bath for 5 minutes, and then filter it hot on a sintered-glass filter stick of No. 4 porosity. Wash the beaker, filter, stirrer and precipitate with six 1-ml portions of hot water. A similar beaker brought up to weight by the addition of pieces of Pyrex-glass rod should be available as a tare. Wipe both beakers with damp flannelette and then with chamois leathers that have been kept in a desiccator containing saturated calcium nitrate solution. Heat both in an oven at 160° C for 1 hour, cool in aluminium blocks, using no desiccant, and weigh. Repeat the heating until the weight is constant; then dissolve the precipitate in chloroform, withdraw the solution, and dry and reweigh the beaker, filter stick and stirrer. The precipitate is $\text{Al}(\text{C}_9\text{H}_6\text{ON})_3$; multiply the weight of the precipitate by 11.10 and divide by the weight of the sample to get the percentage of alumina (Al_2O_3).

RESULTS

In all the results quoted, correction has been made, where necessary, for aluminium in the reagents.

ALUMINIUM AS ALUMINIUM 8-HYDROXYQUINOLINATE—

A standard solution of aluminium chloride in *N* hydrochloric acid was prepared from Hilger and Watts's "H.H.P." aluminium (99.98 per cent.) by dissolving it in hydrochloric acid. Alumina was determined in weighed portions by the procedure given for silicate rocks, except that in half of the experiments an 8-mg excess of 8-hydroxyquinoline was added instead of 6 mg. The smaller amount gives better results, as shown—

8-mg Excess of 8-hydroxyquinoline—

Al_2O_3 taken, μg	494	534	526	1170	1216	1919	1987
Error, μg	+1	0	+1.5	+4.5	+6	+4	+4

6-mg Excess of 8-hydroxyquinoline—

Al_2O_3 taken, μg	549	533	1041	1400	1878	2059	
Error, μg	+1.5	0	-1	0	+2	0	

It is evident that, for accuracy to 2 or 3 parts per 1000 in the determination of alumina, the amount of 8-hydroxyquinoline added must be under control to the nearest 2 mg, that is, the alumina content of a 5-mg sample must be known to the nearest 4 per cent.

ALUMINA IN THE AQUEOUS PHASE FROM THE CUPFERRON - DICHLOROBENZENE TREATMENT—

Weighed aliquots of the standard solution of aluminium chloride, to which were added 100 mg of potassium bisulphate, were submitted to treatment with 0.05 ml of 5 per cent. aqueous cupferron solution and dichlorobenzene, as in the analysis of rocks, and the alumina content of the aqueous phase was determined by precipitating aluminium 8-hydroxyquinolate. The results were as follows—

Al ₂ O ₃ taken, μ g	..	546	530	512	551	1916	1897	1899	2139
Error, μ g	..	-2	-1	+1	+3	+2	+5	+4	-3

ALUMINA EXTRACTED BY ACETYLACETONE AND DIETHYL ETHER—

Weighed aliquots of the standard solution of aluminium chloride, to which were added 100 mg of potassium bisulphate, were suitably prepared and treated with acetylacetone and diethyl ether. The ether extracts were shaken with 6 *N* hydrochloric acid and the aluminium was determined with 8-hydroxyquinoline, all as described for rock analysis. The following results were obtained—

Al ₂ O ₃ taken, μ g	..	525	579	1169	1154	1888	1937
Error, μ g	..	-0.5	-0.5	+1	-4.5	0	+2

ALUMINA IN PRESENCE OF OTHER ELEMENTS—

As satisfactory results were obtained in the three preceding steps, experiments were next made on the determination of various amounts of alumina in synthetic mixtures containing selected combinations of the elements that might be found in silicate rocks. All the test solutions contained 100 mg of potassium bisulphate and the procedure was that prescribed for rocks, except that the initial treatment with hydrofluoric acid and the fusion were omitted. The results given below indicate that in a 5-mg sample of rock it should be possible to determine 0 to 40 per cent. of alumina, with a reasonable degree of accuracy, in the presence of a considerable amount of all the usual components and small amounts of several others.

(a) *Other elements present:* CaO, 500 μ g; MgO, 500 μ g; P₂O₅, 150 μ g; MnO, 50 μ g; BeO, 25 μ g; Ce₂O₃, 25 μ g—

Al ₂ O ₃ taken, μ g	..	502	542	1969	1893
Error, μ g	..	+2.5	+1	-0.5	-3.5

(b) *Other elements present:* Fe₂O₃, 1000 μ g; TiO₂, 250 μ g; P₂O₅, 150 μ g; ZrO₂, 50 μ g; V₂O₅, 25 μ g—

Al ₂ O ₃ taken, μ g	..	546	635	1953	1910
Error, μ g	..	+0.5	+4*	+2	-7*

* These results are corrected for 1 μ g of Fe₂O₃ in the precipitate.

ALUMINA IN SILICATE ROCKS—

Finally, a number of rock samples were submitted to analysis through the whole procedure. As the method is primarily intended for use in the analysis of more complex materials, where the number and the amount of other components of the "mixed oxides" are large, and the standard samples available were lacking in this respect, it was necessary to include two synthetic samples in the series of rocks analysed. They were prepared by adding accurately weighed portions of U.S. Bureau of Standards Feldspar No. 99 (dried) to the ignited residues from the evaporation of appropriate amounts of solutions containing the elements required.

The results of the analyses are shown in Table I. The feldspars used were as issued by the U.S. Bureau of Standards. The flint clay was from the part that had passed through a 300-mesh sieve. As the percentage of silica found in this part agreed excellently with the certificate value, it was assumed that comparison with the certificate value would be valid for alumina. The olivine-basalt was part of the original sample used by Guthrie and Miller¹² and was less finely divided. All the samples, save the flint clay, which was dried at 140° C, were dried at 105° to 110° C before use.

In general, the results are about 1 per cent. lower than those shown in the last column. An attempt was made to obtain figures for comparison by other than the classical procedure. As the feldspars contained insignificant amounts of iron, titanium, manganese, calcium and magnesium, it was practicable to omit the use of cupferron and acetylacetone, precipitate

together the 8-hydroxyquinolates of aluminium, iron and titanium, and then correct for the iron and titanium content of the precipitates on the assumption that $\text{Fe}(\text{C}_9\text{H}_6\text{ON})_3$ and

TABLE I
DETERMINATION OF ALUMINA IN SILICATE ROCKS

Silicate	Approximate weight taken, mg	Amounts of certain components							Al ₂ O ₃ by micro-method, %	Al ₂ O ₃ by classical method or calculation, %
		SiO ₂ , %	Fe ₂ O ₃ , %	TiO ₂ , %	P ₂ O ₅ , %	CaO, %	MgO, %	MnO, %		
Feldspar No. 70*	4.0	67	0.03	<0.01	0.01	0.07	0.01	<0.01	17.86	18.03†
	6.3								17.71	
Feldspar No. 99*	4.8	69	0.07	0.02	0.14	0.36	0.05	<0.01	18.94	19.06†
	6.3								18.77	
Flint clay No. 97*	4.6	43	0.98	2.38	0.08	0.10	0.26	<0.01	38.23	38.77†
	4.5								38.43	
Synthetic rock A	"4.7"	40	15.0	5.0	3.0	10.0	6.0	—	11.13	11.13§
	"4.5"								10.59	10.75§
Synthetic rock B	"4.9"	40	10.0	3.0	1.0	6.0	6.0	—	25.43	25.69§
	"4.7"								25.31	25.55§
Olivine-basalt	4.2	44	13.0	2.4	0.5	10.0	13.0	0.2	12.59	12.59‡
	3.7								12.68	

* U.S. Bureau of Standards sample.

† U.S. Bureau of Standards certificate value.

‡ Guthrie and Miller's figure for semi-micro analyses.¹²

§ Calculated from the weight of Feldspar No. 99 (Al₂O₃ = 19.06 per cent.) in A, and the weight of feldspar (≈ 2.8 mg) plus additional Al₂O₃ in B.

|| Also ZrO₂, 0.23; Cr₂O₃, 0.08; and V₂O₅, 0.04 per cent.

TiO(C₉H₆ON)₂ were present (*cf.* McLennan⁴). In experiments with 70 to 90-mg samples, the results obtained were—

Feldspar No. 70—(17.60), 17.78, 17.79 and 17.81 per cent.

Feldspar No. 99—18.82 and 18.85 per cent.

which seem to suggest that the figures obtained by the classical procedure tend to be rather high. It is of interest that Guthrie and Miller,¹² working with the same sample of Feldspar No. 70, found 17.94 per cent. of alumina by a semi-micro classical procedure. The method described above was inapplicable to the flint clay on account of the complexity of the "mixed oxides."

If these figures for the feldspars are accepted as being correct, and the composition of the synthetic samples is recalculated accordingly, then the errors for the four materials, calculated as weight of alumina, are as follows—

Silicate	Feldspar No. 70		Feldspar No. 99		Synthetic "A"		Synthetic "B"	
	(i)	(ii)	(i)	(ii)	(i)	(ii)	(i)	(ii)
Al ₂ O ₃ calculated, μg ..	714	1116	908	1196	476	515.5	1185	1262.5
Al ₂ O ₃ found, μg ..	717	1111	913	1191	474	521.0	1180	1256.0
Error, μg ..	+3	-5	+5	-5	-2	+5.5	-5	-6.5

bringing them into line with those found for the determination of alumina in complex mixtures. Should, however, the certificate figures be correct and a negative error of 1 per cent. be genuine, it is still possible to say that the errors in terms of the rock total do not greatly exceed the limits normally allowed for duplicate analyses of gram samples by the same analyst.¹³ Moreover, they are considerably less than those evident in the recent co-operative investigation of precision and accuracy in the chemical analysis of silicate rocks,¹⁴ and rather less than those shown in a later co-operative analysis of a synthetic glass of known composition.¹⁵ In the new micro-method, it is almost certain that the results are for alumina only and, as far as we can judge without actual experimentation, it should be possible to determine calcium and magnesium by a standard procedure in the aqueous extract left after the removal of aluminium by means of acetylacetone and ether.

We gratefully acknowledge a maintenance grant to one of us (R. A. C.) from the Department of Scientific and Industrial Research, and grants from Imperial Chemical Industries Limited and the Trustees of the Ritchie Bequest. We are indebted to Dr. M. Borrel of Lyons University for a copy of his thesis.

REFERENCES

1. Miller, C. C., and Chalmers, R. A., *Analyst*, 1953, **78**, 24.
2. Schoklitsch, K., *Mikrochemie*, 1936, **20**, 247.
3. Hecht, F., *Mikrochim. Acta*, 1937, **2**, 188.
4. McLennan, I. C., Thesis, Edinburgh University, 1940.
5. Stene, S., *Chem. Zentralbl.*, 1939, **110** (1), 3433.
6. Abrahamczik, E., *Mikrochemie*, 1948, **33**, 209.
7. Gotô, H., *Sci. Rep. Tôhoku Imp. Univ.*, 1937, **26**, 391.
8. Borrel, M., Thesis, Université de Lyons, 1952.
9. Chemical Analysis Sub-Committee of the British Ceramic Research Association, *Trans. Brit. Ceram. Soc.*, 1952, **51**, 438.
10. Stumpf, K. E., *Z. anal. Chem.*, 1953, **138**, 30.
11. Lacourt, A., *Metallurgia*, 1948, **38**, 355.
12. Guthrie, W. C. A., and Miller, C. C., *Min. Mag. Lond.*, 1933, **23**, 405.
13. Groves, A. W., "Silicate Analysis," Second Edition, George Allen and Unwin Ltd., London, 1951, p. 228.
14. Schlecht, W. G., *Anal. Chem.*, 1951, **23**, 1568.
15. Fairbairn, H. W., and Schairer, J. F., *Amer. Min.*, 1952, **37**, 744.

NOTE—Reference 1 is to part II of this series.

CHEMISTRY DEPARTMENT
THE UNIVERSITY, EDINBURGH, 9
THE UNIVERSITY SCIENCE LABORATORIES
SOUTH ROAD, DURHAM

June 3rd, 1953

DISCUSSION

MR. R. C. CHIRNSIDE said that he had followed with interest this paper by Dr. Miller and Mr. Chalmers, as indeed he had all of Dr. Miller's papers on the subject. Those concerned with silicate and rock analysis would know that the techniques had changed very little since the time of Berzelius, and attempts to determine some of the constituents directly, as instanced in this paper, were therefore very welcome.

Some work along the same lines had been done by the Analytical Committee of the British Ceramic Research Association, and he would like to ask Dr. Miller if she was acquainted with this work, which had been published last year in the *Journal of the Ceramic Society*.

In this connection, he wished to mention that, with certain refractories where free alumina in the form of corundum was known to occur, it had been found impossible to get complete decomposition of the sample except by using a flux of sodium carbonate and borax. He wondered whether some of the low results to which Dr. Miller had referred could have arisen from incomplete decomposition of the sample.

He also wanted to mention that during the co-operative work carried out by the B.C.R.A. Committee, the cause of some apparently low results for alumina was traced to dilution of the sample with silica removed from agate mortars during the grinding of the sample.

MR. CHALMERS replied that they had read the account of the work done by the Analytical Committee of the British Ceramic Research Association, and had considered the use of sodium carbonate for the fusion of the residue left after the removal of silica by means of hydrofluoric acid, but had rejected it, partly because of the risk of loss by spattering in the subsequent dissolution in acid and partly because the bisulphate fusion was easier and seemed quite satisfactory. It was possible that the presence of free alumina might account for the apparent negative error, but it was not likely. There seemed no reason why other fluxes should not be used instead of potassium bisulphate.

MR. A. F. WILLIAMS mentioned that sodium peroxide was in universal use for breaking down ores. The technique was devised by Rafter, of New Zealand, and could be carried out in a platinum crucible at a temperature of about 400° C. The method was rapid and there was no difficulty caused by spraying. There might be difficulty in getting the sodium peroxide free from alumina, but suitable brands should be available.

MR. CHALMERS said that they were acquainted with Rafter's paper and thought that the considerations given in the previous reply would apply here also.

DR. MILLER added, in a written reply to Mr. Chirnside's comments, that the methods described by the British Ceramic Research Association did not fully lend themselves to their purpose, which was to determine alumina in association with all the elements, including some that were commonly neglected, that might be present in igneous silicate rocks, and afterwards to determine calcium and magnesium in the same sample.

With reference to the apparent negative errors recorded for the rocks, some chemical procedures tended to give biased results, and alumina determinations, as normally made, gave results that were more often too high than too low. It might be of significance that, in the co-operative analysis of the synthetic glass of known composition, the average percentage of alumina found was 16.19 instead of 15.78—a difference of 0.4. All their results for the standard samples were lower than the certificate values, the average deviation being 0.3.

The standard samples were not further ground and could not therefore be diluted with silica.

Ultra-violet Spectrophotometric Estimation of the Quality of Mineral Oils Extracted from Bread

BY M. A. COOKSON, J. B. M. COPPOCK AND R. SCHNURMANN

(Presented at the meeting of the Society on Wednesday, May 20th, 1953)

A method has been devised for determining in bread the degree of refining of a mineral oil that has been absorbed by the dough during bread-making. The method consists basically in sulphating, under controlled conditions, the unsaponifiable fraction of the total oils extracted from the bread, so that the natural saponifiable matter is destroyed without completely removing the unsaturated hydrocarbon constituents inherent in mineral oils refined to different degrees. The recovered mineral oil is then examined by ultra-violet absorption spectrophotometry to determine its quality. A spectrophotometric criterion, based on the absorption intensity of the sulphated oils at 2600 Å, is suggested for the quality of mineral oils that can be regarded as satisfactory for the lubrication of plant used in bread production. Some properties of the naturally occurring unsaponifiable oils of bread are also described.

IN recent times bread-making has become a highly mechanised process. The nature of bread dough is such that lubrication is necessary at various stages of production to prevent adherence of the dough to metallic equipment. This is particularly so at the dough divider, where the mass of dough is separated into unit pieces, and also when the dough pieces are tinned before baking. White oils are amongst the lubricants that have been used for these purposes. Since mineral oil may also be absorbed by doughs as a result of accidental contamination from faulty machinery, it is important to maintain a reasonable standard of quality for the oil.

Mineral oils absorbed by doughs can be extracted by a solvent from the finished bread, but the naturally occurring flour-oil and other edible fats and oils added as bread ingredients are extracted at the same time.

It was recommended¹ in 1949 that mineral oils should be permitted as lubricants and greasing-aids in bread-making provided that (a) the quantity absorbed did not exceed 0.2 per cent. of the weight of the bread (this quantity is now the legally permitted limit²) and (b) the degree of refining of the oil was not less than that of liquid paraffin, B.P.

A method for determining the quantity of mineral oil in bread has been published,³ and it was seen to be desirable to form some estimate of the original quality of the oil, *i.e.*, its quality before it was absorbed by the dough.

The development of a suitable method was complicated by (i) the small amounts of oil available for analysis, unless very large quantities of bread were laboriously extracted, (ii) the miscibility of mineral oils with the unsaponifiable fraction of bread-oil (*i.e.*, the natural oils in bread), which makes separation difficult, and (iii) the lack of suitable methods for determining mineral oil quality.

The sulphuric acid test of the British Pharmacopoeia for aromatic hydrocarbons in medicinal liquid paraffin is only suitable for fairly large amounts of white oils, and the conventional interpretation of its results has been criticised.⁴ Ultra-violet absorption spectrophotometry, however, appears to be a sufficiently discriminating technique for determining the various ranges of quality of mineral oils after separating the unsaponifiable matter from bread, and it was the method chosen for this work.

EXPERIMENTAL

Five mineral oils (see Table I) of different and known degrees of refining were used in most of the experimental work.

For spectrophotometric examination, all the specimens were dissolved in purified *iso*-octane and the absorption characteristics of the solutions studied in the wavelength region 2100 to 4500 Å (particularly 2400 to 3400 Å). Most of the spectra were recorded by means

of a medium Hilger quartz spectrograph, fitted with a Spekker photometer, at the Physics Department of the Manchester Oil Refinery Ltd.

The materials examined were prepared at the British Baking Industries Research Association, the experimental procedure being as follows. Bread doughs, to which small quantities (0.1 to 0.5 per cent.) of the mineral oils listed in Table I were added during the dough-making, were baked, care being taken that they were not otherwise contaminated with extraneous mineral oil from tins or machinery. About 18 hours after baking, the cooled bread was crumbled and digested with carbon tetrachloride for 24 hours at room

TABLE I
THE CHARACTERISTICS OF THE MINERAL OILS USED IN THE EXPERIMENTS

Oil	Appearance	$E_{1\text{ cm}}^{1\%}$ values at 2600 Å	
		As refined	After treatment with 96 per cent. w/w H_2SO_4 at 100° C
Liquid paraffin, B.P. . .	Colourless (white oil)	0.030	0.051
Technical white oil . . .	Colourless (white oil)	0.270	0.435
Transformer oil . . .	Fluorescent, straw colour	37.0	17.4
An acid and clay treated Edeleanu extract of a lubricating-oil distillation fraction	Fluorescent, brown colour	49.0	14.2
Spindle oil* . . .	Fluorescent, ruby colour	220.0	21.5

* This term is not connected with the spindle dough moulder; it is derived from spindling in the manufacture of textiles.

temperature to extract the combined mineral and natural oils. These mixed oils were saponified and the unsaponifiable matter was extracted by standard procedures.³

As the oil from bread to which no mineral oil has been added contains about 10 per cent. of unsaponifiable matter, further separation is required to isolate the mineral oil as free as possible from other unsaponifiable material. Two methods were tried for this purpose: (a) percolation of a light petroleum solution of the unsaponifiable matter through a column of activated alumina and (b) heating the unsaponifiable matter with sulphuric acid to remove sterols, tocopherols and hydrocarbon compounds other than those constituting the mineral oil addition, as described below.

Both methods suffer the disadvantage that some of the constituents of the less refined mineral oils are removed and part of the natural unsaponifiable oil is retained in the end product. However, the working conditions for method (b) have been so adjusted that a sufficiently satisfactory separation can be effected.

The separation of the oil—About 0.5 g of the unsaponifiable matter is dissolved in ether and transferred to a Babcock milk-test bottle. After removal of the solvent, 5 ml of carefully standardised 85 per cent. w/w sulphuric acid are added and the bottle is vigorously shaken for 30 minutes in a water-bath at 50° C. After cooling, sufficient 85 per cent. sulphuric acid is added to bring the liquid level into the graduated neck of the bottle, which is then centrifuged until the oil layer separates. The oil is then removed with a fine pipette for spectrophotometric examination.

RESULTS

MINERAL OILS—

Absorption values for the five oils studied are shown in the third column of Table I.

It was necessary to ensure that decomposition, particularly of liquid paraffin and technical white oil, did not occur under baking conditions, thereby making some adventitious contribution to the spectra of the total oils extracted from bread containing mineral oil. This was done by heating liquid paraffin and technical white oil at baking oven temperatures (about 250° C; apart from the crust, bread itself during baking rarely exceeds a temperature of 100° C) for baking times (about 30 minutes). It was found that even in an atmosphere of air, not of carbon dioxide and steam as in bread baking, no marked change in absorption spectra for these two oils was observed.

NATURAL UNSAPONIFIABLE OILS—

As previously stated, about 10 per cent. of the oil that can be extracted from bread, made from flour, yeast, salt and water only, is unsaponifiable. Most, if not all, of the bread-oil is derived from the flour, modified during fermentation and baking, and possibly containing traces of esters and other organic compounds produced in bread-making. It is well known that flour is a variable material, depending on the wheats from which it is made (*i.e.*, the grit), the condition of the wheats when milled, the extraction rate of the flour and the age of the flour when used. Moreover, the method of fermentation and baking produces marked differences in the nature of the resultant bread. It is not surprising, therefore, that the unsaponifiable fraction of bread-oil has been found to be of variable composition.

In Fig. 1 the two unbroken lines show the extremes of the range of absorption spectra observed over a period of 4 years from unsaponifiable oils prepared from breads produced

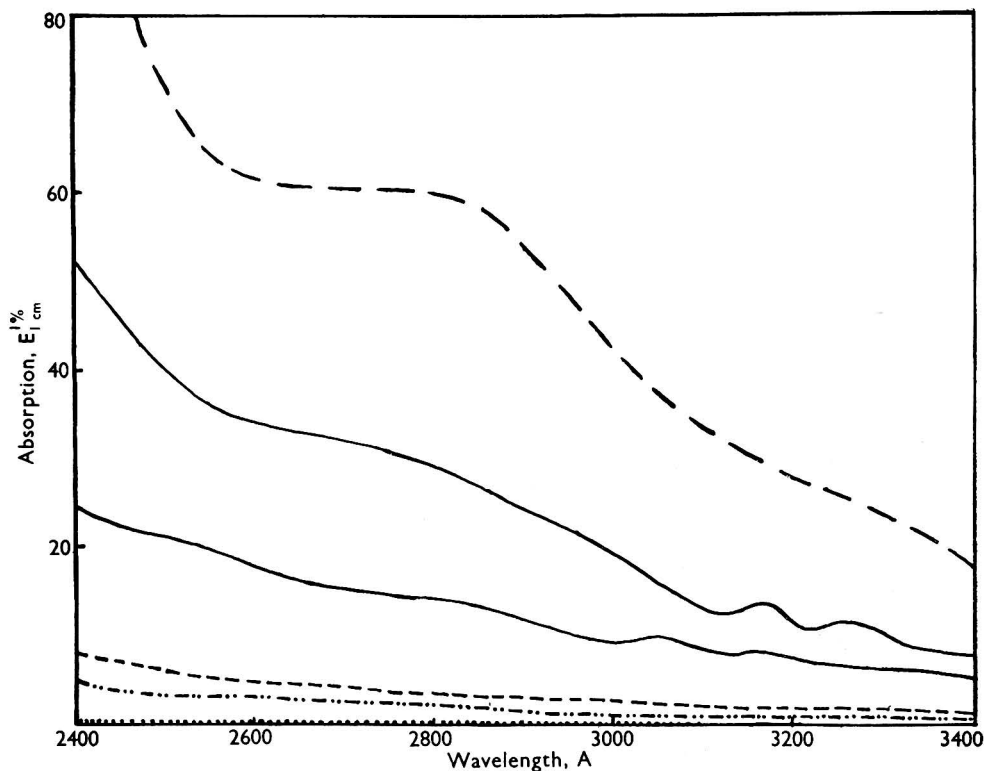


Fig. 1. Absorption spectra of (i) the unsaponifiable oils of breads made from flours of different extraction rate; (ii) the unsaponifiable oils from bread containing 0.26 per cent. of liquid paraffin, and of the chromatographed extract of this oil; and (iii) liquid paraffin—the basis for comparison.

- Unsaponifiable oil from a wholemeal bread (highest flour extraction rate).
- Unsaponifiable oils from National breads (lower flour extraction rates, 80 to 85 per cent.).
- - - - Unsaponifiable oil from National bread containing 0.26 per cent. of liquid paraffin.
- Chromatographed extract of the unsaponifiable oil from National bread containing 0.26 per cent. of liquid paraffin.
- Liquid paraffin

from National flour, bleached and treated, of 81 or 85 per cent. extraction, by the same formula and baking technique. Fig. 1 also shows the spectrum of the unsaponifiable oil from a wholemeal bread; the effect of the higher extraction rate of the flour is apparent from the higher absorption intensity of the oil from this flour.

A comparison of these absorption characteristics with those in Table I for the five mineral oils studied reveals that the light absorption in the region of the ultra-violet explored is much

greater for these unsaponifiable bread-oils than for white mineral oils, and that these naturally occurring unsaponifiable oils have absorption intensities similar to those of some mineral oils that have not undergone the same degree of refining as white oils. As compared with liquid paraffin, B.P., these less refined oils show 100 to 10,000 times as much absorption. The variations in the intensity of absorption of unsaponifiable oil from different batches of flour might be attributed to the presence of various amounts of a highly absorbing component in the unsaponifiable part of the natural oil.

Fractionation of the natural unsaponifiable oil on a column of activated alumina has indicated the existence of several components, some of which, separated with light petroleum-benzene mixtures as eluants, absorb much more strongly than those shown in Fig. 1, *e.g.*, at 2600 Å one fraction shows a maximum $E_{1\text{cm}}^{1\%}$ value of 480, and a second fraction crosses this peak and rises to an $E_{1\text{cm}}^{1\%}$ value of 750 at 2400 Å. It is hoped that further details about this particular phenomenon will be published later.

UNSAPONIFIABLE OILS CONTAINING MINERAL OILS—

The maximum permitted quantity² of mineral oil that may be present in a food as a result of its use as a processing aid is 0.2 per cent. by weight of the food, but the desirable quality of the mineral oil is not legally defined.

The method of oil extraction described in the experimental section, when applied to a bread containing no mineral oil, yields approximately 0.4 per cent. of oil, expressed on the bread weight, and approximately 0.53 per cent. when a bread containing 0.2 per cent. of mineral oil is extracted. After saponification of these oils the remaining unsaponifiable oils are approximately 0.04 and 0.11 per cent., respectively, expressed on the bread weight, *i.e.*, about two-thirds of the latter is attributable to mineral oil. It is, therefore, in the light of the various spectra discussed, and having regard to the high intensity of the bread's natural unsaponifiable oil, clearly not to be expected that the quality of the mineral oil originally used as a lubricant can always be ascertained by merely examining the spectrum of the total unsaponifiable oil; the interference would be too great. The spectra in Fig. 1 of liquid paraffin and of the unsaponifiable oils from National breads with and without liquid paraffin, *i.e.*, the first, third and fifth lines from the abscissa, illustrate this point. However, when the mineral oil is of rather low quality, a detectable increase in absorption intensity of the combined unsaponifiable matter might be expected. This has been found to occur in practice, provided there is present about 0.2 per cent. of oil of a quality approximately equivalent to an acid and clay-treated Edeleanu extract of a lubricating oil distillation fraction, but if less than 0.1 per cent. is present, it is very difficult to detect.

A further factor to be taken into consideration is that the proportion of natural to mineral oil in the total unsaponifiable oil will not be constant, so that, in the absence of any characteristic band structure connected with the mineral oil in the combined oil, quantitative measurements would be difficult to apply in estimating the quality of the mineral oil.

It is seen, therefore, that because of the relatively large absorption and heterogeneity of the naturally occurring unsaponifiable matter, and also the variation in the proportion of mineral oils in the unsaponifiable oil, a separation of the mineral oil fraction of the total unsaponifiable oil must be achieved in order to appraise the quality of the mineral oil in the mixture.

THE ATTEMPTED SEPARATION OF MINERAL OIL FROM UNSAPONIFIABLE MATTER BY CHROMATOGRAPHIC METHODS—

The use of adsorption columns of activated alumina for the separation of hydrocarbons has been known for some time. This technique has been suggested as a way of treating the unsaponifiable fraction in a general method for determining mineral oil in saponifiable oils,⁵ and particularly for determining mineral oil in bread.⁶ However, it was reported in our paper⁶ that a small fraction of the natural unsaponifiable matter was eluted immediately without being adsorbed on the separation column. If mineral oil is also present in the unsaponifiable material, it is possible that not only is the same natural non-adsorbed fraction (probably hydrocarbon in nature) eluted, but that further fractions of the natural oil may be eluted at the same time by a solvent action of the mineral oil.

It has been found that the eluates containing mineral oil obtained by this method are still sufficiently variable and intense in absorption to make difficult the discernment of the quality of the mineral oil. Although the absorption intensities of these chromatographed

fractions are not so great as those exhibited by the total unsaponifiable matters containing mineral oils, they are still appreciably greater than those of the original mineral oils.

The second and third lines from the abscissa in Fig. 1 show typical results given by the unsaponifiable matter from bread oil mixed with liquid paraffin, B.P., before and after removal of some of the absorbing materials by chromatographic separation. The liquid paraffin was present at a concentration of 0.26 per cent. in the bread.

More efficient separation than that provided by this particular chromatographic technique would therefore be necessary to determine the original quality or degree of refining of a mineral oil that has been extracted from a loaf of bread. Although greater efficiency might be attained by varying the adsorbent in the chromatographic column and the eluting solvent, separation of the mineral fraction by sulphation appeared to hold more promise.

SEPARATION OF MINERAL OIL BY SULPHATION OF UNSAPONIFIABLE MATTER—

The A.O.A.C. method⁷ for determining mineral oil in fats is based on heating the unsaponifiable fraction with concentrated sulphuric acid of not less than 94 per cent. w/w at 100° C for 30 minutes with occasional shaking. The apparatus used for this digestion is a Babcock milk-test bottle into which the fat is introduced in ether solution, the ether then being evaporated before the acid is added. After sulphation, further acid is added to the cooled digest until the liquid rises well into the graduated neck of the bottle, which is then centrifuged to bring the oil layer to the surface, where its volume is determined. This technique has been recommended⁸ for determining mineral oil in the unsaponifiable matter from bread oil, but it was suggested that the results might be unreliable if a low grade of mineral oil was present, owing to removal of impurities in the oil.

In a preliminary experiment, the five mineral oils of various degrees of refining were treated as described above, and the ultra-violet absorption spectra of the sulphated oils were examined. The results in the fourth column of Table I indicate appreciable differences from the spectra of the corresponding materials before acid treatment.

Although the more impure oils showed considerable reaction, as evidenced by loss of colour and appreciable reduction in absorption intensity, the white oils exhibited an increased absorption, particularly in the wavelength region below 2600 Å, the $E_{1\%}^{1\text{cm}}$ values at 2400 Å being 0.19 for liquid paraffin, B.P., and 0.9 for a technical white oil. The corresponding values for the non-sulphated oils were 0.05 and 0.65. The increase in absorption intensity may be caused by the formation of sulphonic acids and the dehydrogenation of reactive naphthenes by this intense treatment with sulphuric acid; an alcohol extraction and clay treatment would probably be necessary to remove these absorbing compounds.

Less drastic conditions of sulphation of the unsaponifiable matter from the total oil extracted from bread containing mineral oil were investigated. A temperature of 50° C was arbitrarily chosen for the reaction, and the acid concentration reduced until separation of the mineral oil either no longer occurred or was difficult. This was found to occur with 80 per cent. sulphuric acid. The use of 85 per cent. acid, however, permitted a separation of oil after the reaction. Under these conditions the less refined oils usually still retained some of their colour after treatment and this in itself allowed a rapid distinction to be drawn between them and white oils. If the layer above the acid in the Babcock bottle is solid or semi-solid in reasonable quantity and not a thin black sludge, as occurs when no mineral oil is present, it is safe to assume that a paraffin wax or petroleum jelly has been absorbed into the bread dough.

Fig. 2 shows the spectra given by separated mineral oils after sulphation of the total unsaponifiable matters from breads containing these oils with 85 per cent. w/w sulphuric acid at 50° C for 30 minutes. These results have been obtained in duplicate by operatives working in different laboratories, and in one case using a Uvispek absorption spectrophotometer.

The spectra of the extracted mineral oils after this sulphation treatment closely approximated to those of the original oils, except for some fall in the extinction values for the least refined oil. The main difficulty with this technique is that the spectra of the recovered two white oils are not readily distinguishable, although the figures in Table I show that in relation to each other measurable differences exist. The reason for this may, in part, lie in a reaction between sulphuric acid and quite highly refined oils, with the formation of substances having different absorption characteristics.

A further series of experiments was made with 90 per cent. instead of 85 per cent. sulphuric acid, and although the resultant spectra of the extracted oils still permitted distinction between oils of different purities, they were not as sensitive as those obtained after treatment with 85 per cent. acid.

The wavelength region between 2100 and 2400 Å was examined in the hope that discrimination would be possible between two white oils of different quality after extraction from bread. Liquid paraffin and a technical white oil were examined, but the spectra of these oils after extraction from bread could not be readily distinguished at these lower wavelengths.

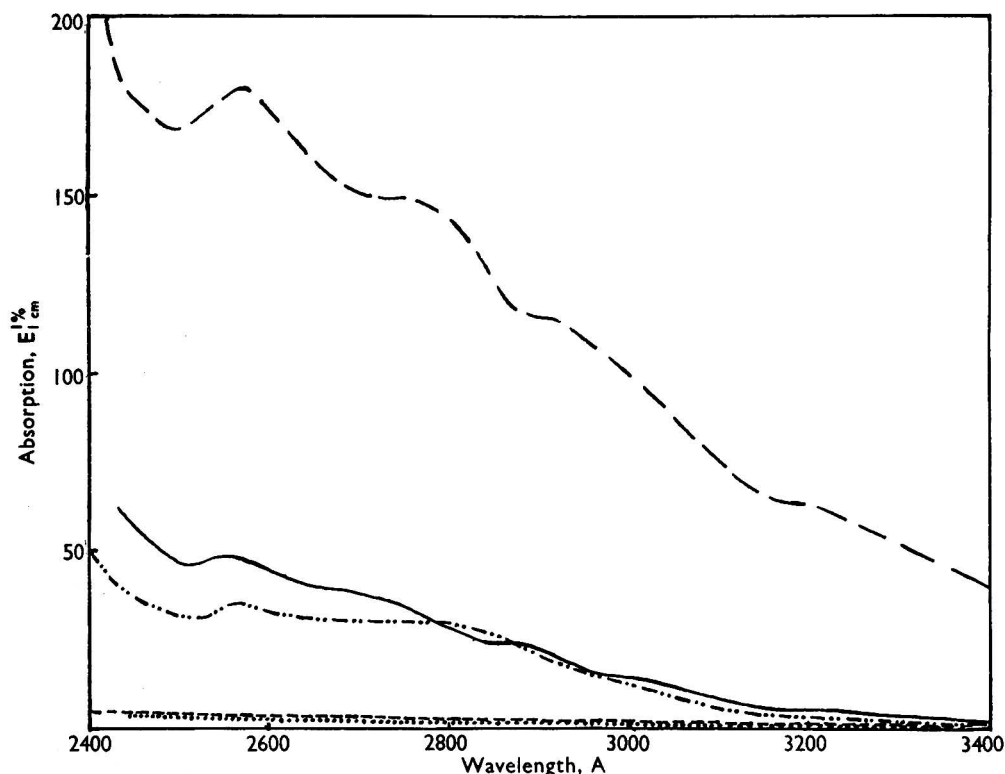


Fig. 2. Absorption spectra of mineral oils separated from the unsaponifiable matter of bread oils with 85 per cent. sulphuric acid at 50° C.

..... Liquid paraffin.
 ----- Technical white oil.
 - · - · - Transformer oil.
 ——— An acid and clay treated Edeleanu extract of a lubricating oil distillation fraction.
 ——— Spindle oil

The results recorded and used for correlation purposes in the original quantitative work on mineral oils in bread³ have not been amended by this new sulphation technique, as the re-determinations would be very tedious and would not significantly alter or increase the sensitivity of the quantitative method. None the less, the technique now described is preferable for routine use if a quality determination is also required.

CONCLUSIONS

For the lubricating oils used in bread-making, the question arises whether liquid paraffin, B.P., and some other white oils are so significantly different in physical characteristics that only the former, as has previously been maintained, should be used. It is suggested that Table II, which shows the $E_{1\text{cm}}^{1\%}$ values at 2600 Å for the various oils examined before use and after extraction from bread, will supply the answer to this question. From the

figures shown in this table, it is clear that medicinal liquid paraffin, B.P., and some other

TABLE II

Mineral oil	$E_{1\text{cm}}^{1\%}$ at 2600 A	
	Before use	Extracted from bread
Liquid paraffin, B.P.	0.030	2.6
Technical white oil	0.270	4.0
Transformer oil	37.0	33.0
An acid-treated Edeleanu extract of a lubricating-oil distillation fraction	49.0	44.0
Spindle oil	220.0	173.0

white oils, even though they may not be of B.P. quality, fall into a related group when compared with the less refined colour-bearing mineral oils (see Fig. 2). The absorption intensities of the mineral oils when compared with the natural unsaponifiable oils from bread, are (i) lower for the white oils, (ii) usually greater for the colour-bearing mineral oils and (iii) lower for all the mineral oils examined when compared with some of the components that have been isolated from natural unsaponifiable matter of the oil in bread. Mineral oils can be recovered from bread by the suggested procedure comparatively unchanged spectroscopically, and the relationship of their absorption intensities to that of the natural unsaponifiable oil of bread remains as summarised here.

Provided, as is desirable on both pharmacological⁸ and technical grounds, that the viscosity of the oil is not abnormally low, then, it seems to us for the above reasons that mineral oils for which the $E_{1\text{cm}}^{1\%}$ value at 2600 A does not exceed 0.5 before use, or 5.0 after extraction from bread by the procedure suggested above, could be regarded as satisfactory for the lubrication of plant used in bread production.

The authors express their thanks to Mr. W. F. Maddams, M.Sc., Miss P. M. Martin, M.Sc., and Mr. R. Mayoh, B.Sc., for their assistance with the spectrographic work.

REFERENCES

1. Coppock, J. B. M., and Cookson, M. A., *Brit. Med. J.*, 1949, 73.
2. Statutory Instrument, 1949, No. 614, The Mineral Oil in Food Order, H.M. Stationery Office, 1949.
3. Cookson, M. A., and Coppock, J. B. M., *J. Sci. Food Agric.*, 1951, 2, 434.
4. Schnurmann, R., Martin, P. M., and Maddams, W. F., *J. Pharm. Pharmacol.*, 1951, 3, 298.
5. Williams, K. A., *J. Ass. Off. Agric. Chem.*, 1949, 32, 668; *Analyst*, 1946, 71, 261.
6. Coppock, J. B. M., and Cookson, M. A., *J. Soc. Chem. Ind.*, 1949, 68, 274.
7. "Official Methods of Analysis of the Association of Official Agricultural Chemists," The Association of Official Agricultural Analysts, Washington, D.C., 1950, p. 209.
8. Coppock, J. B. M., *Brit. J. Nutr.*, 1951, 5, 383.

BRITISH BAKING INDUSTRIES RESEARCH ASSOCIATION
CHORLEYWOOD, HERTS.

PHYSICS DEPARTMENT
MANCHESTER OIL REFINERY LIMITED
MANCHESTER

First submitted, March 12th, 1953
Amended, June 6th, 1953

DISCUSSION

THE PRESIDENT, Dr. D. W. Kent-Jones, said he thought that many of the chemists who specialised in the examination of oils might have important observations to make, as they probably had experience of such matters in other connections.

As many analysts did not possess spectrophotometers, he wished to know if it was possible to use the ordinary Spekker absorptiometer with the ultra-violet light attachment. He was interested to hear that wholemeal bread, as opposed to white bread, apparently contained something in the unsaponifiable portion of the bread oil that had very high absorption in the ultra-violet spectrum, and he wondered if the authors had any information as to what these substances were.

MR. COOKSON replied that preliminary experiments indicated that mineral oils of various qualities could be differentiated with a Spekker absorptiometer adapted to measure the transmission of ultra-violet light through the oil. With suitable filters to exclude as much as possible of the visible light, increasingly large-scale deflections were obtained as the quality of the mineral oil decreased. However, more recent work had indicated that further experimentation was required before such a technique might be found to be successful.

The compounds naturally present in the unsaponifiable fraction of bread oils and responsible for high absorption in the ultra-violet spectrum had not as yet been identified. Adsorption chromatography had enabled a separation of some constituents of unsaponifiable flour and bread oils to be made, and the similarity between these oils from various sources had been shown by ultra-violet spectrophotometry. The reason for the greater absorption intensity of the unsaponifiable oil from wholemeal bread was unknown, but it might be due to the presence in this oil of compounds that are removed when flours are milled to a lower extraction rate. The very high extinction coefficients found at wavelengths below 3000 Å were, however, of some interest.

DR. J. R. NICHOLLS said that white petroleum oils were prepared commercially by the somewhat drastic treatment of coloured oils with sulphuric acid, whereby many other impurities were removed with the colour. The better grades were used for pharmaceutical purposes and were required by the British Pharmacopoeia to conform to a maximum limit of carbonisable substances. From the absorption curves illustrated it appeared clear that technical white oils contained only slightly greater amounts of impurities than liquid paraffin, B.P., and that the proposed method was adequate for distinguishing white oils from cruder products.

The only difficulty in permitting all grades of white oils to be used for lubrication of plant in bread-making was the possibility that some grades might contain carcinogens. Bearing in mind the purification that all white oils had undergone and the maximum amount of oil that was allowed to be present in the finished bread, any danger appeared to be small. But it was desirable that adequate tests should be made.

MR. COOKSON said that extensive surveys of the literature and discussions with authorities on this subject had failed to produce the slightest evidence of carcinogenicity from the oils included in their specification.

DR. K. A. WILLIAMS said he understood that both white and medicinal oils were made from naphthenic-base stocks by treatment first with fuming sulphuric acid and then with alcohol. The hydrocarbons present included a fairly high proportion of cycloparaffins mainly derived from cyclopentane and cyclohexane.

MR. N. L. ALLPORT referred to a sample of medicinal liquid paraffin sent to him because it had been reported against as failing the British Pharmacopoeia's sulphuric acid test. In point of fact it quite safely passed the B.P. requirement, and in the course of subsequent discussion it transpired that the first analyst had used concentrated sulphuric acid of 98 to 99 per cent., whereas the Pharmacopoeia specified a special reagent described in the First Appendix as consisting of 96 per cent. by weight of nitrogen-free sulphuric acid.

When presenting the paper Dr. Coppock had interposed that with certain unsaponifiable fractions the extinction coefficient as measured by a photo-electric type of spectrophotometer was different from that recorded by a photographic type of instrument. He asked if the authors could say which instrument gave the true value.

DR. SCHNURMANN replied that in the wavelength region between 2400 and 3000 Å it did not matter whether a photographic plate instrument or a photo-electric instrument was used. With the same type of prism in either of these instruments, results were almost identical. Difficulties might arise, however, at wavelengths shorter than 2400 Å when photo-electric instruments were used because at these shorter wavelengths the emission intensity of the hydrogen discharge tube fell off, the sensitivity of the photo-cell decreased, the reflectivity of the mirrors became poorer, and the proportion of scattered radiation increased; hence with photo-electric instruments the indicated absorption intensity might appear to decrease in the region between 2400 and 2000 Å on approaching the shorter wavelength end even for samples for which it could be proved with the help of photographic plate instruments that the absorption intensity increased towards shorter wavelengths. The photographic plate instrument, in addition to supplying a permanent record, had the advantage that it could be safely used over the whole region over which the photographic plate was sensitive.

DR. W. W. MYDDLETON, in a written contribution, said he wondered what use was to be made of the results described in the paper, and asked whether there was to be a drive in favour of using medicinal paraffin or technical white oils and, if so, on what grounds.

He asked if it was claimed that poorly refined oils endowed the bread with an objectionable taste, smell or colour, because if so, the results of the spectrophotometric method could but confirm the evidence of the senses.

He said that at one point in the presentation of the paper they were left to infer that, because medicinal paraffin and a technical white oil could be heated above 200°C for some time without any effect on the ultra-violet absorption spectrum, a similar state of affairs would exist in the baking of the loaf. He found this inference difficult to draw, for the oil in the bread was probably in thin films and was in contact with many different components of the bread and, in addition, some of it was in contact with metallic and metallic oxide surfaces during baking. There was therefore a probability of catalysed reactions occurring and involving, perhaps, dehydrogenation, polymerisation or pyrolysis.

If after being baked the product was cleaned by extraction and controlled sulphation, it was doubtful what was in fact produced as a result of heating the mineral oil, or even medicinal paraffin. There was no evidence; it had not been looked for and, in fact, it may have been destroyed.

As an example of the variable behaviour of mineral oils under different conditions, Dr. Myddleton referred to his own experience of the changes that occurred in a technical white oil, similar to the one heated to 200° C by the authors, when exposed to sunlight for three months (*J. Inst. Pet.*, 1951, **37**, 57). The maximum temperature was about 62° C for about 1 hour on bright days. The minimum on the ultra-violet absorption curve was raised by 64 per cent. and the maximum by 14.5 per cent. The ratio of the maximum to the minimum was lowered from 1.63 to 1.18 and the position of the maximum was shifted from 2710 to 2650 Å. He therefore felt diffident in accepting the assumption that even highly refined white oils remained unchanged in the process of bread baking.

DR. COPPOCK, in a written reply, said he wished to emphasise that the main purpose of the paper was to describe a method whereby the original quality of oils used for lubrication in bread-making could be determined after use. In reply to the first question raised by Dr. Myddleton, he pointed out that the Mineral Oil in Food Order restricted the quantity of mineral oils present in foods as a result of processing operations to not more than 0.2 per cent. by weight, but did not define the quality of oil regarded as desirable.

The authors had advocated previously that the quality of mineral oil used for lubricating bread-making machinery should be that of liquid paraffin, B.P., but in view of the spectrophotometric evidence presented in this paper they now suggested that certain white oils might also be used. The pharmacology of mineral oils had naturally been taken into account, and there was no evidence that the oils included in the proposed specification could in any way be harmful when used for this purpose.

Secondly, the smell, colour or taste of poorly refined oils could not be detected at levels of 0.2 per cent. in bread, and therefore more scientific methods would be required to show their presence. Thirdly, the heating of white oils to 200° C in air caused negligible change in the oils as determined by their ultra-violet absorption spectra; Dr. Myddleton's experience of the effect of sunlight on mineral oils would appear irrelevant to a consideration of the baking process, but the above conditions of heating were considerably more drastic than would be found in bread-baking, for which the temperature, except at the crust, did not exceed 100° C and the atmosphere was preponderantly carbon dioxide. Further, none of the components of bread would be expected to decompose mineral oil, even if the oil was dispersed in thin films. The possibility of decomposition at the baking-tin surface could not be ignored, but it was doubtful whether the conditions were sufficient for this to occur; it should also be noted that the commercial use of mineral oils for tin greasing was almost negligible. In any event, a vegetable oil could be just as prone, and possibly even more prone, to adverse reactions; *e.g.*, their breakdown products under certain conditions of heating were believed to be carcinogenic (see, *e.g.*, Peacock, P. R., and Beck, S., *Nature*, 1948, **162**, 252) and there were inherent dangers should vegetable oils become rancid (Frazer, A. C., *Trans. Roy. Soc. Trop. Med. Hyg.*, 1952, **46**, 576).

Fourthly, it was extremely unlikely that the extraction procedure removed any decomposition products formed in mineral oils on baking in bread and, as just stated, the authors did not believe that any decomposition occurred. It should be remembered that white oils were refined by far more drastic sulphuric acid treatments than were used to separate the mineral oil from the natural unsaponifiable oil in bread, and the sulphonation conditions were selected to be just sufficient to achieve this. The similarity of the ultra-violet absorption spectra of the mineral oils of different quality, before use and after recovery from bread, were striking, and did not suggest that any marked changes in the mineral oils had occurred, or that the controlled sulphonation was sufficiently vigorous to destroy any decomposition products that may have formed. The much more pronounced absorption intensities of the natural unsaponifiable oils in bread compared with those of white oils should also be noted in this connection.

Finally, with regard to Dr. Myddleton's statement of the effect of daylight exposure of a technical white oil which by spectrophotometric evidence was not stable, experiments recorded elsewhere (Schnurmann, R., paper presented at the Réunion Internationale de Spectroscopie Moléculaire, Paris, July, 1953) have shown that medicinal liquid paraffins that obeyed the spectrophotometric quality and stability criterion showed no significant changes of their spectra after exposure to an ultra-violet light source *provided* that the oils were contained in a glass vessel.

The Spectrophotometric Determination of Long-chain Fatty Acids Containing Ketonic Groups

With particular reference to Licanic Acid

By A. MENDELOWITZ AND J. P. RILEY

A spectrophotometric procedure, based on the alkaline 2:4-dinitrophenylhydrazone method of Lappin and Clark, is described for the determination of ketonic groups in long-chain compounds. The effect of a number of variables on the final intensity of the colour has been investigated. The method has been applied to the determination of licanic acid in the presence of other saturated and unsaturated fatty acids and shows a standard deviation of 0.8 per cent.

In the course of a study¹ of oils containing licanic acid (4-oxo-octadeca-9:11:13-trienoic acid), a method was required for its determination in the presence of elaeostearic acid (octadeca-9:11:13-trienoic acid), with which it is usually associated. As the ultra-violet absorption spectra of the two acids are almost identical,² measurement of the absorption at 270.5 $m\mu$ gives only a measure of the total conjugated trienoic unsaturation. On the other hand, licanic acid can be estimated by determination of the ketone value.

Several volumetric procedures have been described for the estimation of ketonic groups in long-chain fatty-acid derivatives,^{2,3,4,5} mainly adaptations of the hydroxylamine method originally introduced by Brochet and Cambrier,⁶ or its modification in which pyridine is used to ensure complete reaction.⁷ These methods suffer from the disadvantage that the end-point is always somewhat indistinct, particularly in the presence of pyridine.

A number of workers have used phenylhydrazine and its derivatives for the gravimetric⁸ or volumetric^{9,10,11} determination of ketonic compounds. These procedures depend on either the quantitative recovery of the phenylhydrazone of the ketone or the determination of unreacted phenylhydrazine. All were found to be inapplicable to long-chain fatty-acid derivatives owing to the difficulty of separating the phenylhydrazones in a pure state; for instance, attempts to prepare the 2:4-dinitrophenylhydrazone of methyl 12-oxostearate resulted in the production of an oily product containing much unreacted 2:4-dinitrophenylhydrazine, which could only be removed with great difficulty.

Observations by Bamberger¹² and Gnehm and Benda¹³ that nitrophenylhydrazones form dark-coloured water-soluble compounds on treatment with alkali have been made the basis of colorimetric procedures for the estimation of ketonic compounds. The earlier applications of this method^{14,15,16,17,18} were to water-soluble ketonic compounds, and they are therefore unsuitable for use with the higher fatty acids and their derivatives. Lappin and Clark¹⁹ have reported an extension of the method to the determination of traces of aldehydes and ketones in water, organic solvents or organic reaction products. They reported that both the wavelength of maximum absorption and the molecular extinction were independent of the structure of the ketonic compound.

Poole and Kloose²⁰ have described a spectrophotometric procedure for the estimation of monoketonic compounds in rancid foods. The 2:4-dinitrophenylhydrazones of the ketones were formed in benzene solution in a column of activated alumina. Alcoholic potassium hydroxide was added to the eluate from the column and the intensity of the resultant red colour was determined at 435 $m\mu$. The colour was not stable, however, and faded rather rapidly.

In view of the poor results that were attained with the hydroxylamine method for the determination of methyl 12-oxostearate, the procedure of Lappin and Clark seemed to offer the advantages of accuracy and simplicity. These workers¹⁹ treated 1 ml of a methanolic solution of the ketone with 1 ml of a 0.06 per cent. solution of 2:4-dinitrophenylhydrazine in methanol and 1 drop of concentrated hydrochloric acid. This was heated in a loosely stoppered test tube at 100° C for 5 minutes and then 5 ml of 10 per cent. methanolic potassium hydroxide were added. The absorption of the solution was measured at 480 $m\mu$ against a blank determination made with 1 ml of methanol instead of the ketone solution.

Preliminary experiments made with *cyclohexanone* and methyl 12-oxostearate revealed five defects in their procedure—

- (i) Much evaporation took place during the heating at 100° C, and often no methanol remained at the end of the reaction period. In order to reduce evaporation to a minimum, a temperature of 60° C was used for the condensation in all subsequent work and ethanol was substituted for methanol.
- (ii) Small variations in the amount of hydrochloric acid added as catalyst in the condensation reaction produced large differences in the intensity of the colour.

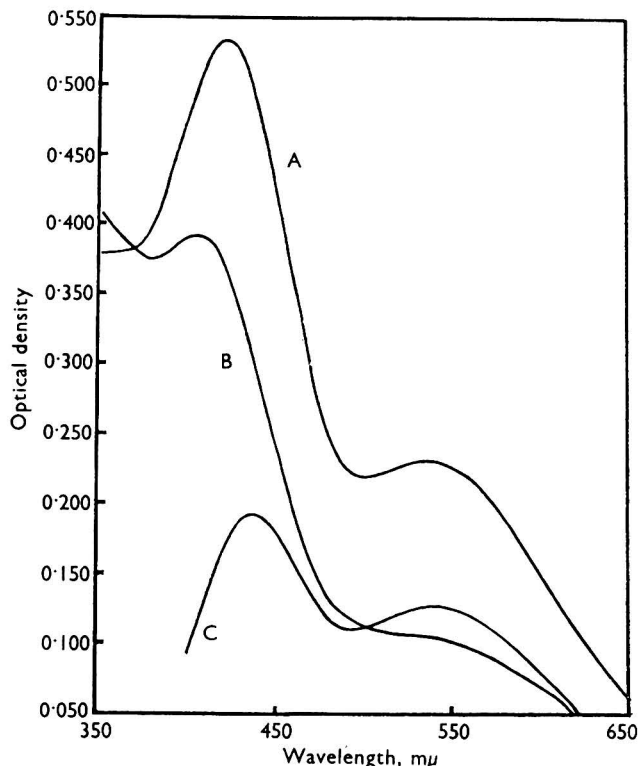


Fig. 1. Comparison of absorption curves of solution and blank solution. Curve A, methyl oxostearate; curve B, reagent blank; curve C, methyl oxostearate less reagent blank

- (iii) Potassium chloride was precipitated after the addition of alcoholic potassium hydroxide and it was difficult to obtain absolutely clear solutions.
- (iv) The wavelength of maximum absorption was not 480 $m\mu$, but varied with the nature of the ketone. Long-chain aliphatic ketones and *cyclohexanone* exhibited maximum absorption at 435 $m\mu$ (with a secondary maximum at 535 $m\mu$, see Fig. 1), whereas with cinnamaldehyde the maximum absorption was at 485 $m\mu$.
- (v) Owing partly to the difference in structure of the dinitrophenylhydrazones and partly to the equilibrium nature of the condensation reactions, the molecular extinction at the wavelength of maximum absorption depended markedly on the nature of the ketonic compound. For example, *cyclohexanone* and methyl oxostearate gave ϵ (at 435 $m\mu$) of 17,900 and 11,400, respectively. A sample of pure *cyclohexanone*-2:4-dinitrophenylhydrazone in the same strength of alcoholic potassium hydroxide had an ϵ of 19,530 at 435 $m\mu$, showing that the condensation of *cyclohexanone* with the reagent had only proceeded to the extent of about 92 per cent.

During preliminary work it was found that high reagent blanks resulted when measurements were made at 435 $m\mu$. Fig. 1 shows the absorption curves for a determination with

methyl oxostearate and for the reagent blank, both measured against water in the compensator cell. The rather high absorption of the reagent blank at 435 $m\mu$ was almost entirely due to the shoulder of the absorption band at about 395 $m\mu$ of alkaline 2:4-dinitrophenylhydrazine.²¹ On the other hand, at the secondary absorption maximum at 535 $m\mu$, the absorption of the blank was quite low and remained fairly constant over a considerable range of wavelengths; for these reasons all further measurements were made at this wavelength.

The effect of variation in hydrochloric acid concentration on the condensation reaction was examined. Some 1-ml samples of a 0.0065 per cent. ethanolic solution of methyl oxostearate were treated with 1-ml portions of dinitrophenylhydrazine reagent containing different strengths of acid (0.06 g of dinitrophenylhydrazine with 1 ml of hydrochloric acid made up to 100 ml with ethanol). The mixture was heated at 60° C for 50 minutes; then, after cooling, 5 ml of 10 per cent. alcoholic potassium hydroxide were added and the solution was diluted to 10 ml with water. The results are shown below—

Normality of acid	2	3	4.6	6
$E_{1\text{cm}}^{1\%}$ at 535 $m\mu$ *	227	344	352	361

* After deduction of reagent blank.

To minimise variations caused by changes of acid concentration, all subsequent work was performed with a reagent containing 1 ml of constant-boiling hydrochloric acid per 100 ml.

With this reagent, the time for attainment of equilibrium in the initial reaction was determined for licanic acid and oxostearic acid and its methyl ester, with the results shown in Table I. In each determination the condensation was complete within 50 minutes at 60° C.

TABLE I
RATE OF REACTION OF LONG-CHAIN KETONIC COMPOUNDS WITH
2:4-DINITROPHENYLHYDRAZINE

Time, minutes	$E_{1\text{cm}}^{1\%}$ at 535 $m\mu$ for		
	oxostearic acid	methyl oxostearate	licanic acid
30	361	346	—
35	370	355	—
40	376	359	—
45	378	362	265
50	378	362	270
55	—	—	268
60	377	362	269

Small changes in the amount of alcoholic potassium hydroxide solution added produced little alteration in the intensity of the red colour; for instance, use of 7.5 ml of this reagent instead of 5 ml increased the final optical density by only 1.5 per cent. The substitution of water for alcohol in the dilution to the final volume had only a slight effect on the final colour and prevented the precipitation of potassium chloride that had been encountered when the method of Lappin and Clark was used.

With the modified conditions it was found that, although highly reproducible results were attained (for methyl oxostearate) if the same sample of alcohol was used throughout, fresh batches of alcohol gave rather different values. That this was due to the presence during the condensation reaction of variable amounts of water in the alcohol was shown by the addition of known amounts of water to the alcohol used as solvent for the ketonic compound.

Water in alcohol, per cent.	0	0.1	0.5	1.0
$E_{1\text{cm}}^{1\%}$ at 535 $m\mu$	377	377	362	356

To overcome this difficulty a control test must be made simultaneously on the pure ketone being determined, or if this is not stable, on some ketonic compound of a similar nature that has been calibrated against the unstable ketonic compound. In the present work, oxostearic acid was used as a control for licanic acid and for methyl oxostearate, with the results shown in Table II. The constancy of the extinction coefficients of both methyl oxostearate and licanic acid, when related to an arbitrary fixed value of 360 for the extinction coefficient for oxostearic acid, is proof of the validity of this procedure.

TABLE II

$E_{1\text{cm}}^{1\%}$ VALUES FOR SIMULTANEOUS RUNS OF OXOSTEARIC ACID WITH METHYL OXOSTEARATE AND WITH LICANIC ACID

Oxostearic acid	Methyl oxostearate		Oxostearic acid	Licanic acid	
	found	calculated to oxostearic acid = 360		found	calculated to oxostearic acid = 360
370	362	352	358	270	272
368	360	352	373	280	273
364	358	354	363	275	270
367	360	353			
		Mean 353			Mean 272

It will be noted that the value found for licanic acid is very much lower than those of either oxostearic acid or its methyl ester. This emphasises the fact that the method can only be applied to a particular ketonic compound after standardisation with that compound.

Unsuccessful attempts were made to replace the alcohol used in the condensation reaction by other solvents, such as benzene or cyclohexane, which are more readily obtained free from ketonic compounds. The red colour, if it was produced at all, was very weak and ephemeral.

METHOD

STANDARD SUBSTANCES—

Methyl 12-oxostearate—Methyl ricinoleate (purity 99.4 per cent.)²² was hydrogenated with Raney-nickel catalyst.²³ The resulting methyl hydroxystearate was treated with a 100 per cent. excess of a 10 per cent. solution of chromium trioxide in glacial acetic acid. After being allowed to stand at room temperature for 2 hours the mixture was poured into water and filtered. The precipitate was dissolved in ether and the ethereal solution was washed with 5 per cent. potassium carbonate solution and with water. The crude ester, recovered by evaporation of the solvent, was crystallised three times from acetone at 0° C. The purified methyl 12-oxostearate consisted of white plates, m.p. 46.8° to 47.5° C; previous workers have reported 45° to 46° C²⁴ and 44.5° to 45° C.²⁵

12-Oxostearic acid—Crude methyl oxostearate was saponified with alcoholic potassium hydroxide. The recovered acid was crystallised three times from acetone at 0° C. The pure acid had m.p. 81.5° to 82° C; previously recorded values are 80° to 81° C²⁴ and 81° C.²⁵

Licanic acid—The mixed acids obtained from a commercial oiticica oil were dissolved in 20 ml of boiling light petroleum, boiling range 40° to 60° C, per gram of mixed acids. The boiling solution was filtered through a warm funnel to remove small amounts of resinous oxidation products. On cooling, the filtrate deposited crude licanic acid, which was re-crystallised five times from light petroleum. The purified licanic acid was pressed well on the filter and dried in a high vacuum for not longer than 10 minutes. The products from a number of preparations all melted in the range 74.5° to 75.5° C, and gave the following values of $E_{1\text{cm}}^{1\%}$ at 270.5 μ : 1711, 1703, 1724, 1702, 1725, 1729, mean 1716; Rose and Jamieson² record 74.5° to 75.5° C and 1784.

REAGENTS—

Ketone-free ethanol—Distil absolute alcohol from zinc dust and potassium hydroxide. Heat 1.5 litres of the distillate under reflux with 10 g of 2:4-dinitrophenylhydrazine and 10 ml of concentrated sulphuric acid. Distil the ketone-free ethanol after 2 hours and store it in a tightly stoppered bottle in the dark. The alcohol, which is slightly coloured yellow owing to the volatility of 2:4-dinitrophenylhydrazine, is stable for about a week.

2:4-Dinitrophenylhydrazine reagent—Add 1 ml of constant-boiling hydrochloric acid to 0.06 g of 2:4-dinitrophenylhydrazine, add 80 ml of ketone-free ethanol and warm until all is dissolved. Cool and dilute to 100 ml with ketone-free ethanol. The reagent is stable for at least 3 days if stored in the dark.

Alcoholic potassium hydroxide—Dissolve 10 g of potassium hydroxide in 20 ml of water and dilute to 100 ml with ethanol (which need not be ketone-free).

PROCEDURE—

Weigh about 0.1 g of the substance to be examined, dissolve it and make the solution up to 100 ml with ketone-free ethanol. Dilute this solution accurately with the same alcohol to give a solution approximately 0.001 per cent. with respect to the ketonic group ($>C=O$). Place 1 ml of the diluted solution in a 10-ml calibrated flask and add 1 ml of dinitrophenylhydrazine reagent. Stopper the flask loosely and heat it to 60° C in a thermostatically controlled bath. After 1 minute press the stopper home to minimise evaporation. After 50 minutes, cool the flask in water for 2 minutes. Add 5 ml of alcoholic potassium hydroxide rapidly and dilute to 10 ml with water. Measure the optical density of the solution in a 1-cm cell at the secondary absorption maximum (535 $m\mu$ for 12-oxostearic and licanic acids), using water in the compensating cell. Determine the reagent blank in the same manner with 1 ml of purified ethanol instead of the solution of the ketone. Carry out control estimations with the pure ketone simultaneously with the determination. If the ketonic compound to be determined is unstable, *e.g.*, licanic acid, make simultaneous calibration tests with a related, but stable, ketone, *e.g.*, oxostearic acid, and with the unstable compound. Use the stable ketone in place of the unstable compound as a control in all determinations.

CALCULATION FOR UNSTABLE KETONES—

Calculate the extinction coefficients ($E_{1\text{cm}}^{1\%}$) of the sample and standards from the optical densities of the solutions after deduction of the reagent blanks.

Let a and b be the extinction coefficients, determined simultaneously for the pure unstable ketone and the stable standard, respectively. If x and y are the extinction coefficients of the sample containing the unstable ketone and of the stable ketone respectively, then the percentage of the unstable ketone in the sample is—

$$\frac{b \times x \times 100}{a \times y}$$

DETERMINATION OF KETONES IN THE PRESENCE OF FATTY ACIDS—

Known mixtures of ketones with non-ketonic compounds were analysed with the results shown in Table III. The non-ketonic compounds used included the mixed acids of tung oil, in order to ensure that such acids would not interfere in the determination of licanic acid in oils such as oiticica. Each result is the mean of four determinations showing an average standard deviation of 0.8 per cent.

TABLE III
ANALYSIS OF MIXTURES

Ketone	Non-ketonic compound	$E_{1\text{cm}}^{1\%}$ of mixture	$E_{1\text{cm}}^{1\%}$ of control	Ketone in mixture	
				Found, %	Calculated, %
Methyl oxostearate	Methyl stearate	154	350*	44.0	43.6
Methyl oxostearate	Palmitic acid	183	350*	52.3	52.5
—	Palmitic acid	0	350*	0	0
—	Tung mixed acids	2.4	378†	0.8‡	0
Oxostearic acid	Tung mixed acids	176	378†	46.5	46.3
Licanic acid	Tung mixed acids	164	373†	58.6	58.1

* Methyl oxostearate. † Oxostearic acid.

‡ As licanic acid, but probably due to oxidation products.

CONCLUSIONS—

The method described is by no means limited to the fatty acids, but could be applied after suitable standardisation to many other types of ketonic compounds. It presents the advantages of rapidity and accuracy, and can be very readily adapted for use with micro-samples.

The authors wish to thank Professor T. P. Hilditch, C.B.E., F.R.S., for his interest and valued criticism.

REFERENCES

1. Mendelowitz, A., unpublished work.
2. Rose, W. G., and Jamieson, G. S., *Oil and Soap*, 1943, **20**, 227.
3. Leithe, W., *Fette u. Seif.*, 1938, **45**, 615.
4. Kaufmann, H. P., Funke, S., and Liu, F. Y., *Ibid.*, 1938, **45**, 616.
5. Feuill, A. J., and Skellon, J. H., *Analyst*, 1953, **78**, 135.
6. Brochet, A., and Cambrier, R., *Compt. Rend.*, 1895, **120**, 449.
7. Bryant, W. M. D., and Smith, D. M., *J. Amer. Chem. Soc.*, 1935, **57**, 57.
8. Iddles, H. A., and Jackson, C. A., *Ind. Eng. Chem., Anal. Ed.*, 1934, **6**, 454.
9. Strache, H., *Monatsh. Chem.*, 1891, **12**, 514.
10. Schöninger, W., and Lieb, H., *Mikrochemie*, 1951, **38**, 165.
11. Ardagh, E. G. R., and Williams, J. G., *J. Amer. Chem. Soc.*, 1925, **47**, 2976.
12. Bamberger, E., *Ber.*, 1893, **26**, 1306.
13. Gnehm, R., and Benda, L., *Ibid.*, 1896, **29**, 2017; *Annalen*, 1898, **299**, 101.
14. Neuberg, C., and Strauss, E., *Arch. Biochem.*, 1945, **7**, 211.
15. Neuberg, C., and Kobel, M., *Biochem. Z.*, 1928, **203**, 467; 1929, **210**, 466; 1929, **216**, 493; 1930, **219**, 490; 1930, **229**, 255; 1932, **256**, 481; *Ber.*, 1930, **63**, 1886.
16. Case, E. M., *Biochem. J.*, 1932, **26**, 753.
17. Lu, G. D., *Ibid.*, 1939, **33**, 249.
18. Friedemann, T. E., and Haugen, G. E., *J. Biol. Chem.*, 1943, **147**, 415.
19. Lappin, G. R., and Clark, L. C., *Anal. Chem.*, 1951, **23**, 541.
20. Poole, M. F., and Kloose, A. A., *J. Amer. Oil Chem. Soc.*, 1951, **28**, 215.
21. Roberts, J. D., and Green, C., *J. Amer. Chem. Soc.*, 1946, **68**, 214.
22. Riley, J. P., *Analyst*, 1951, **76**, 40.
23. Adkins, H., and Billica, H. R., *J. Amer. Chem. Soc.*, 1948, **70**, 695.
24. Grummitt, O., and Siedschlag, K. G., *J. Amer. Oil Chem. Soc.*, 1949, **26**, 690.
25. Perotte, R., *Compt. Rend.*, 1934, **199**, 358.

DEPARTMENT OF OCEANOGRAPHY
UNIVERSITY OF LIVERPOOL
LIVERPOOL, 3

June 3rd, 1953

Design and Operating Technique of a Vacuum Drying Oven

Part I. Design of the Oven

By S. D. GARDINER

A symmetrical vacuum drying oven of circular plan, large heat capacity and uniform temperature distribution has been designed. It has a shallow, hollow but thick metal base, which holds six sample dishes with the minimum of free space. Controlled dry air bleed is incorporated.

A STANDARD reference method for determining water in sugar products, as distinct from quick routine methods, requires the use of a vacuum oven free from the many faults in design inherent in almost all of the ovens currently available, such as those mentioned in the *Proceedings of the International Commission for Uniform Methods of Sugar Analysis*, 1949. To overcome the difficulties in drying such labile products as sugar syrups and molasses containing fructose, the oven described here was designed and constructed in the Tate and Lyle Research Laboratories. Its use need not be limited to the products mentioned—it can be more generally utilised whenever exact and reproducible results of experiments on drying losses from many other materials are required.

The design incorporates features of the oven reported to the author by a colleague, who visited the National Gas Board Laboratories. Attention has also been paid to the recommendations made by Iles and Sharman,¹ although these refer to atmospheric pressure ovens.

GENERAL DESCRIPTION—

Fig. 1 shows that the oven consists primarily of a shallow, hollow, but thick, metal base, which holds six sample dishes with minimum free space. The lid is shown resting

in the off position (at the rear of the figure), where it is out of the way and suspended so that it can be kept clean and undamaged. The vacuum seal is produced with the aid of a wing-nut that clamps down the heavy lid over the central control spigot, the raised annulus of the base sealing the oven by pressing on to the grease-free silicone-rubber ring, inserted into the complementary circular groove in the lid. At the same time, provided sufficient pressure is applied, good thermal contact is made with the base. Vacuum sealing at the spigot is

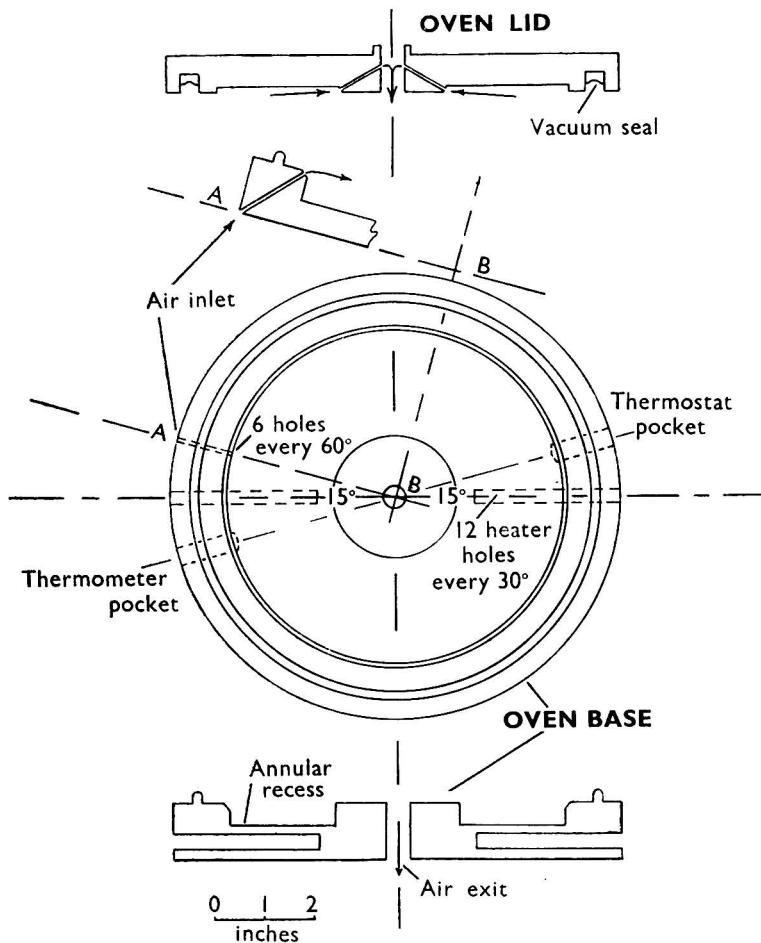


Fig. 3. Design of vacuum oven

ensured by a copper washer under the wing nut. The washer is held in position by a circular projection in the lid and is normally lubricated with a little grease. To conform with the shape of the oven the air flow has been arranged symmetrically. Fig. 2 shows the under-side of the oven with the air-distributing leads from the central supply connected by thick rubber tubes to six entry ports situated around the perimeter of the base of the oven. Fig. 3 shows that these ports, which are inclined at an angle of 45° , project the air up against the oven lid immediately over the sample dishes, from whence it passes over their surfaces to be drawn out through six exit ports arranged symmetrically in the lid near the centre. These exit ports inclined at 30° force the air upwards through the centre hole enclosing the spigot, and so out of the oven. The angles are chosen to facilitate removal of water vapour from above the sample. The air flow is adjustable to between 0.5 to 100 ml per minute by means of an Edwards needle-control valve (type LB1), after the air has been dried through activated alumina (6 to 8 mesh) and over barium oxide lumps. The alumina is arranged in a long narrow tube (Fig. 1) in order to reduce the water content of the air to 0.005 mg per litre; the



Fig. 1. General view of the oven

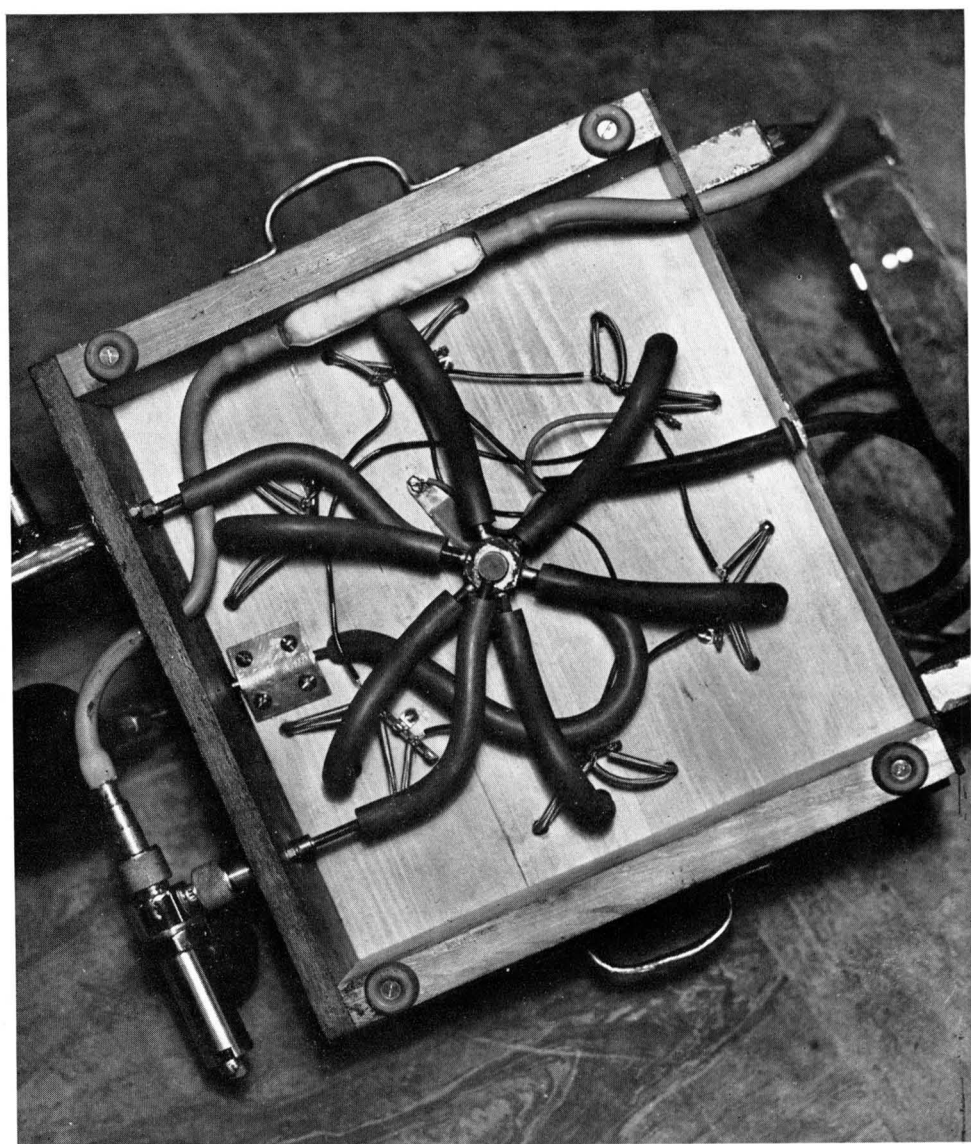


Fig. 2. View of the underside of the oven and the air distributing leads

lumps further reduce the water to 0.0007 mg per litre. The drying train is attached to the oven and moved with it. Iles and Sharman state that in the absence of the steaming effect (catalysis of glucose and amino-acid condensation) about 2 ml of air per minute per gram of water are required. The new vacuum oven is certainly free from this effect. Six samples each holding a maximum of 2.5 g of water (including added water) would have a maximum total of 15.0 g of water to be removed, which according to Iles and Sharman would need a minimum of 30 ml of air per minute, but as this amount is well within the limit of control by the needle valve, the steaming effect is safeguarded against on two counts. A simple liquid-detergent film air-flow meter may be suspended from the vertical drying tube.

Fryd and Kiff² found experimentally that the heat from the air flow contributes only slowly to the rise in temperature of the sample; conduction and radiation supply most of the heat required. The oven described here gives the same degree of heat transfer by radiation and conduction at all points in its interior. As the surface of the recess is ground flat, heat transfer is facilitated to such a degree that it is essential to commence heating at a low temperature and to heat to operational temperature by resetting the thermostatic control. This procedure assists in preventing crust formation. Uniform temperature distribution is attained by using twelve electric heaters inserted radially around the base and penetrating to near the centre. The total wattage is 80, each heater being deliberately run at one-tenth of its normal rating. An indicator lamp assists in setting the thermostatic control.

Two methods of temperature control were attempted. One oven was controlled to within $\pm 0.5^{\circ}\text{C}$ by a Sunvic adjustable bimetal thermostat (T.S.1) in an oil-filled pocket, in conjunction with a Sunvic hot-wire vacuum relay (F.102-3M). Another oven was regulated to within $\pm 1.0^{\circ}\text{C}$ by an electronic capacity-switch, operating off the 0° to 110°C thermometer. The former method is cheaper and, in the long run, simpler to maintain. Temperatures are read on an ordinary 0° to 110°C thermometer, inserted in an oil-filled pocket drilled into the base. Two thermometers diagonally situated give identical temperatures; these comply with the suggestion of Hayes³ and show agreement with the surface temperature of the interior of the oven. Quadruplicate drying tests with cane molasses agree to within less than ± 0.05 per cent. loss, and with sucrose solution to less than ± 0.02 per cent. The oven is designed to operate efficiently from 50° to 90°C . For drying at atmospheric pressure the temperature may be raised to 105°C as long as the air flow is sufficient to prevent rusting. As an additional precaution the exposed metal surfaces can be phosphate treated.

Once an efficient oven has been constructed there remain other problems in the determination of water in thermally sensitive materials. Choice of absorbent, the effect of extra water, sample to absorbent ratio, particle size, deliberate formation of degradation products are all being investigated. Sugar syrups and molasses containing fructose and organic non-sugars require especial care.

Tests are being made in drying golden syrup and molasses, and the results are being utilised to find a correction for refractometer solids based on ash content, after correction has been made for the effect of invert sugar. The refractometer is so useful in the sugar industry that the oven recommended here is not intended to replace it for routine use, especially as the time required for drying is too long for this purpose, although it is considerably shorter than has been needed for other vacuum ovens of less suitable design. The results of the work, particularly on cane molasses, will be dealt with in further papers.

REFERENCES

1. Iles, G., and Sharman, C. F., *J. Soc. Chem. Ind.*, 1949, **68**, 174.
2. Fryd, C. F. H., and Kiff, P. R., *Analyst*, 1951, **76**, 25.
3. Hayes, F. W., in *Proc. I.C.U.M.S.A.*, 1949, 42.

TATE AND LYLE RESEARCH LABORATORY
WESTERHAM ROAD
KESTON, KENT

April 27th, 1953

The Determination of Total Phosphatide in Commercial Lecithin

BY H. H. HUTT, H. WEATHERALL AND T. CULSHAW

In a previous publication it has been suggested that the difference between acetone-insoluble matter and petrol-insoluble matter can be made the basis of a means for assessing the phosphatide content of commercial lecithin for works-control purposes. A modified method, which has improved precision, is now described for the determination of acetone-insoluble matter; it is compared experimentally with previously published methods. The ratio of phosphatide to non-phosphatide removed at various stages of washing is examined.

The difference between acetone-insoluble matter determined by the method proposed and petrol-insoluble matter is suggested as giving a suitable measure of phosphatide content.

Two general methods for the determination of the total phosphatide content of commercial lecithin¹ are (i) by determination of the phosphorus content, the found value being multiplied by an arbitrary factor (usually 26.31 for commercial purposes) and (ii) by determination of the difference between acetone-insoluble content and the amount of matter insoluble in petroleum spirit of boiling range 40° to 60° C.

The rapid works-control method for the determination of acetone-insoluble matter by direct elution previously described¹ was based mainly upon experience with commercial ground-nut lecithin. Examination of a large number of specimens from various botanical sources has subsequently afforded us the opportunity of modifying the method and of making it more acceptable as a means of comparatively assessing the total phosphatide content of all types of material, when this is defined as acetone-insoluble content minus petrol-insoluble content.

Results recorded in the literature for phosphatides of different botanical origins show that the phosphorus contents of carefully prepared petrol-soluble acetone-insoluble materials are lower than expected for di-oleyl glycerophosphoryl choline (3.94 per cent.) or di-oleyl glycerophosphoryl ethanolamine (4.16 per cent.). Typical specimens of total phosphatide mixtures from soya-bean lecithin have been reported² as containing 3.4 per cent. of phosphorus, and from cotton-seed lecithin,³ 2.9 per cent. of phosphorus, and we have found 3.0 per cent. of phosphorus in total phosphatides of ground-nut lecithin prepared in the manner indicated.

The lower phosphorus content found in the oil-free preparations from the naturally occurring phosphatides, as compared with the theoretical contents of the chemical compounds mentioned above, is caused mainly by combined and closely associated carbohydrates.^{2,3,4,5} As these carbohydrates appear always to be present in the natural substances and as it is probable that their hydroxyl groups have a function in most of the industrial purposes for which commercial phosphatides are used, it seems permissible to include them in any comparative assessments of total-phosphatides. They in no way constitute any deleterious addition to, or indicate any incomplete purification of, the material under examination. Moreover, phosphorus determination is a lengthy and somewhat expensive procedure. We therefore suggest that a method that will give reasonably reproducible results for matter insoluble in acetone can be used as a basis for assessing total phosphatide.

Of methods proposed for this determination, other than the one already mentioned, that of dissolving the material in light petroleum and precipitating and eluting with acetone⁶ is probably the most widely used. The comparative merits of this method and the proposed modified method are reported upon below.

Error in the method formerly proposed for production control analysis¹ was largely caused by entrainment of insoluble matter during decantation—this was particularly noticeable with cotton-seed lecithin—and by the solubility of phosphatide in the oil-acetone solution on the first wash—observed especially with soya-bean lecithin. Preliminary centrifugation eliminates the error caused by entrained material and the solubility error can be minimised by first washing the sample in the cold.

METHOD

PROCEDURE—

Weigh accurately 3.0 ± 0.5 g of sample into a centrifuge tube and add 40 ml of acetone. Stir thoroughly, kneading the lecithin in the acetone, and then setting the tube aside for 10 minutes at room temperature. Centrifuge for 3 to 5 minutes at a relative centrifugal force of approximately 1000 *g* and decant the supernatant liquid. Extract twice more with boiling acetone and cool to 0° C before centrifuging. For soya-bean lecithin, only one of these further extractions is required. After completion of the washing, remove the bulk of the residual acetone from the insoluble material on a water-bath in a current of air and dry the residue in a vacuum oven at 100° C and at a pressure of approximately 40 cm of mercury.

EXPERIMENTAL

Acetone conforming to analytical reagent standards was used for all determinations.

Table I shows a comparison of results by the earlier method¹ with those by the same procedure corrected for matter recovered from the decanted solutions by centrifugation and with those found by the proposed modified method.

TABLE I
ACETONE-INSOLUBLE CONTENTS OF COMMERCIAL LECITHINS

Type of lecithin	Acetone-insoluble material		
	Former method, ¹ %	Former method ¹ corrected for material recovered by centrifugation, %	Proposed method, %
Unrefined ground-nut	50.7	53.4	53.5
	43.6	47.0	47.7
	48.3	50.3	50.7
	49.9	53.0	54.6
	53.6	56.2	56.8
Refined ground-nut	63.4	65.1	65.6
	66.1	67.6	68.4
	66.4	68.2	67.8
	66.0	66.3	67.5
	67.5	68.3	69.1
Soya bean	53.9	56.2	61.0
	61.5	63.5	66.6
	54.3	62.6	66.2
	45.2	62.6	66.1
	23.5	33.5	35.1
Cotton-seed	49.8	52.5	53.6
	49.4	51.2	52.5
	51.3	54.7	55.4
	47.6	52.6	52.2
	47.3	50.3	51.2

NOTE—Results are the means of duplicate determinations.

Table II compares results obtained by this modified elution method with those obtained by the method of precipitation and elution.⁶

TABLE II
COMPOSITION OF COMMERCIAL LECITHINS

Lecithin	Acetone-insoluble content*		Moisture, %	Benzene-insoluble content, %	Petrol-insoluble content, %
	Proposed method, %	A.O.C.S. method, ⁶ %			
Unrefined ground-nut (L 3) ..	57.8, 57.9	59.4, 60.0	2.50	0.29	0.47
Refined ground-nut (L 49) ..	69.8, 69.9	71.1, 71.6	0.73	0.39	0.76
Bleached soya bean (D 41) ..	66.1, 66.2	65.5, 65.7	1.30	trace	trace
Unbleached soya bean (D 84) ..	66.2, 65.9	67.0, 66.6	0.32	trace	trace
Cotton-seed (L 50)	56.7, 56.8	60.9, 60.8	0.58	0.14	0.26

* No correction was made for petrol-insoluble or benzene-insoluble matter.

Phosphatides, which are known to be slightly soluble in acetone in the presence of fatty acids,⁷ have been found to be slightly soluble in pure acetone (see Table IV).

To assess and compare the solubility errors, acetone-soluble matter was weighed separately in individual washes (see Table III) obtained by the proposed method and the A.O.C.S. method, phosphorus being determined in these individual extracts (see Table IV).

TABLE III
REMOVAL OF ACETONE-SOLUBLE MATTER BY SUCCESSIVE WASHING

Lecithin	Method	Amount of acetone-soluble material removed by wash number							
		1, %	2, %	3, %	4, %	5, %	6, %	7, %	8, %
Unrefined ground-nut (L 3)	Proposed	29.8	7.83	2.50	1.00	0.76	0.46	0.54	0.62
	A.O.C.S.	33.3	4.46	1.26	0.81	0.73	0.42	0.43	0.52
Refined ground-nut (L 49)	Proposed	22.7	5.30	1.45	1.14				
	A.O.C.S.	24.5	3.40	0.94	—				
Bleached soya bean (D 41)	Proposed	27.8	6.27	2.04	1.20	1.10	1.09	0.78	0.75
	A.O.C.S.	29.7	3.49	1.29	1.01	0.92	0.90	0.60	0.85
Unbleached soya bean (D 84)	Proposed	27.8	6.59	2.38	1.36				
	A.O.C.S.	30.2	2.69	1.48	—				
Cotton-seed (L 50)	Proposed	32.1	8.05	2.12	1.36				
	A.O.C.S.	34.5	4.00	1.05	—				

TABLE IV
PHOSPHATIDE IN ACETONE-SOLUBLE MATTER REMOVED BY SUCCESSIVE WASHING

Lecithin	Method	Amount of phosphatide* removed by wash number							
		1, %	2, %	3, %	4, %	5, %	6, %	7, %	8, %
Unrefined ground-nut (L 3)	Proposed	0.40	0.35	0.22	0.21	0.20	0.19	0.20	0.23
	A.O.C.S.	0.48	0.15	0.11	0.32	0.32	0.15	0.18	0.23
Refined ground-nut (L 49)	Proposed	0.41	0.36	0.25	0.26				
	A.O.C.S.	0.29	0.19	0.15	—				
Bleached soya bean (D 41)	Proposed	0.69	0.87	0.60	0.37	0.40	0.45	0.37	0.36
	A.O.C.S.	0.66	0.74	0.43	0.37	0.38	0.40	0.28	0.43
Unbleached soya bean (D 84)	Proposed	0.74	1.27	0.91	0.68				
	A.O.C.S.	1.31	0.35	0.32	—				
Cotton-seed (L 50)	Proposed	0.43	0.48	0.29	0.40				
	A.O.C.S.	0.69	0.16	0.09	—				

* Calculated by multiplying the figure found for phosphorus by the factor 26.31.

For the phosphorus determinations, the acetone-soluble matter was saponified and ignited to ash; the ash was dissolved in water and the phosphorus measured on a Spekker absorptiometer after development of a blue colour by an adaptation of Zinzadze's method,^{8,9,10} by means of molybdic anhydride reduced with molybdenum metal.

The results shown in Table IV are expressed as the percentage of phosphatide. This was calculated by multiplying the figure for phosphorus by the arbitrary factor 26.31, which is at present used in the commercial assessment of phosphatide on the basis of phosphorus determination. As pointed out earlier, the factor necessary for converting phosphorus to phosphatide would be higher than this for the total mixed phosphatides and it is probably much higher for phosphatides removed in the fourth and subsequent washes. Nevertheless, this variation does not greatly alter the interpretation of the figures.

A more important consideration is whether or not the phosphorus-containing material removed during the acetone washing is definitely phosphatide. It is evident that it is because (i) the phosphorus content of the acetone washes can be suppressed by using acetone previously saturated with oil-free phosphatide (the addition being allowed for), (ii) the material removed in the later washes cannot be redissolved in a smaller volume of acetone and (iii) if larger amounts of acetone are used, correspondingly larger amounts of phosphorus-containing material are dissolved. Further, as only minor amounts of water-soluble phosphorus were

present in the acetone washes, the acetone-soluble phosphorus derivatives could not be free phosphoric or glycerophosphoric acids.

There does not appear to be any other suitable fat solvent in which the phosphatides are less soluble generally than acetone. In attempts to find ways of suppressing the existing solubility, other than by temperature control, it was found that acetone saturated with phosphatide or magnesium chloride gives a lowering that is insufficient to warrant its use. Examination of the existing techniques was therefore continued. To assess the balance between phosphatide removed from the sample by acetone and oleaginous non-phosphatide left in, at given stages of the washing, the calculations summarised in Table V were made.

TABLE V
BALANCE OF ERRORS IN ESTIMATING ACETONE-INSOLUBLE MATERIAL

Lecithin	Method	Factor*	Wash number					
			2, %	3, %	4, %	5, %	6, %	7, %
Unrefined ground-nut (L 3)	Proposed	I	0.40	0.75	0.97	1.18	1.38	1.57
		II	7.48	2.28	0.79	0.56	0.27	0.34
		III	+7.08	+1.53	-0.18	-0.62	-1.11	-1.23
Unrefined ground-nut (L 3)	A.O.C.S.	I	0.48	0.63	0.74	1.06	1.38	1.53
		II	4.31	1.15	0.49	0.41	0.27	0.25
		III	+3.83	+0.52	-0.25	-0.65	-1.11	-1.28
Refined ground-nut (L 49)	Proposed	I	0.41	0.77	1.02			
		II	4.94	1.20	0.88			
		III	+4.53	+0.83	-0.14			
Refined ground-nut (L 49)	A.O.C.S.	I	0.29	0.48				
		II	3.21	0.79				
		III	+2.92	+0.31				
Bleached soya bean (D 41)	Proposed	I	0.69	1.56	2.16	2.53	2.93	3.38
		II	5.40	1.44	0.83	0.70	0.64	0.41
		III	+4.71	-0.12	-1.33	-1.83	-2.29	-2.97
Bleached soya bean (D 41)	A.O.C.S.	I	0.66	1.40	1.83	2.20	2.58	2.98
		II	2.75	0.86	0.64	0.54	0.50	0.32
		III	+2.09	-0.54	-1.19	-1.66	-2.08	-2.66
Unbleached soya bean (D 84)	Proposed	I	0.74	2.01	2.92			
		II	5.32	1.47	0.68			
		III	+4.58	-0.54	-2.24			
Unbleached soya bean (D 84)	A.O.C.S.	I	1.31	1.66				
		II	2.34	1.16				
		III	+1.03	-0.50				
Cotton-seed (L 50)	Proposed	I	0.43	0.91	1.20			
		II	7.57	1.83	0.96			
		III	+6.94	+0.92	-0.24			
Cotton-seed (L 50)	A.O.C.S.	I	0.69	0.85				
		II	3.84	0.96				
		III	+3.15	+0.11				

* I: Total phosphatide removed by preceding washes.

II: Non-phosphatide removed by wash enumerated.

III: Difference between I and II, after previous wash.

DISCUSSION OF RESULTS

The errors in these calculations, which are caused by using the factor 26.31 for converting phosphorus to phosphatide, are small compared with the amounts involved and, further, their direction is such as to contribute to the arguments in their favour. This being so, certain useful deductions can be made. The most important of these is that the least error is incurred in either method by washing no further after the second operation for soya-bean lecithin and after the third for the other lecithins.

If a higher conversion factor were used, the estimates of phosphatide removed (I) would be higher and of non-phosphatide removed (II) correspondingly lower. The increased disparity would be such as to require an earlier discontinuation of the washing. The diminishing amounts of oleaginous non-phosphatide removed, however, suggest that this would be undesirable.

The persistent removal of what appeared to be non-phosphatide in the fourth and subsequent washes is probably caused by a different type of phosphatide with a lower phosphorus content, but this, at present, is not certain. If, however, these substances were glycerides, the amount removed would diminish at a greater rate.

As already shown in Table I, centrifugation reduces the entrainment almost completely. It is also evident that a cold initial wash reduces the amount of phosphatide dissolved in the oil-acetone solution.

Statistical treatment of the duplicate analyses, of which averages are given in Table I, shows that the standard deviation of the modified method is significantly lower than that of the former method (see Table VI).

From a limited number of results (five pairs, representative of different types of lecithin—see Table II) it seems that the standard deviation (0.114) of the proposed method, calculated on the same range of duplicates, is significantly lower than that (0.286) of the elution method.

The improved precision in measuring acetone-insoluble matter is still reflected in the ultimate estimation of the difference between acetone-insoluble matter and petrol-insoluble matter when the standard deviation of the method of determining the latter (0.0186) is taken into account. The standard deviation of the difference is 0.164 for the proposed method compared with 0.321 for the earlier method,¹ even when the results for soya bean lecithin, which has been shown to have a significantly higher variance than other types, are excluded.

TABLE VI

PRECISION OF METHODS OF DETERMINING ACETONE-INSOLUBLE MATTER

Type of lecithin	Standard deviation	
	Former method ¹	Proposed method
Unrefined ground-nut	0.366	0.134
Refined ground-nut	0.265	0.190
Soya bean	0.975	0.118
Cotton-seed	0.326	0.195
(Over-all)	0.321*	0.163

* Excluding soya-bean lecithin, for which variance was shown to be significantly greater than it was for other lecithins.

In the light of the results presented and discussed, we suggest that the difference between acetone-insoluble matter and petrol-insoluble matter gives an adequate measure of the phosphatide content of commercial lecithin, acetone-insoluble matter being determined by the method described in this paper and petrol-insoluble matter being determined as described previously.¹

Other criteria may need to be referred to occasionally, and, as the modified method gives higher results than the earlier procedure, standards will need to be revised for given qualities, when those standards have been based upon acetone-insoluble content.

We are indebted to other colleagues of our Analytical Department for considerable assistance, to Mr. J. W. Lord for statistical analysis of results and to J. Bibby & Sons, Ltd. for permission to publish this work.

REFERENCES

1. Hutt, H. H., and Weatherall, H., *Analyst*, 1944, **69**, 39.
 2. Scholfield, C. R., Dutton, H. J., Tanner, F. W., jun., and Cowan, J. C., *J. Amer. Oil Chem. Soc.*, 1948, **25**, 368.
 3. Olcott, H. S., *Science*, 1944, **100**, 226.
 4. Hutt, H. H., Malkin, T., Poole, A. G., and Watt, P. R., *Nature*, 1950, **165**, 314.
 5. Scholfield, C. R., Dutton, H. J., and Dimler, R. J., *J. Amer. Oil Chem. Soc.*, 1952, **29**, 293.
 6. American Oil Chemists Society's Committee on Analysis of Commercial Fats and Oils, *Ibid.*, 1947, **24**, 77.
 7. Linteris, L., and Handschumaker, E., *Ibid.*, 1950, **27**, 260.
 8. Zinzadze, C., *Ind. Eng. Chem., Anal. Ed.*, 1935, **7**, 227.
 9. Gerritz, H. W., *J. Ass. Off. Agric. Chem.*, 1940, **23**, 321.
 10. Schricker, J. A., and Dawson, P. R., *Ibid.*, 1939, **22**, 167.
- J. BIBBY AND SONS LIMITED
KING EDWARD STREET
LIVERPOOL, 3

June 16th, 1953

The Determination of Small Amounts of Potassium, Calcium and Magnesium in Sodium and its Compounds

BY LOUIS SILVERMAN AND K. TREGO

Preliminary concentration is necessary before potassium can be determined in the presence of large amounts of sodium ion.

Metallic sodium, sodium hydroxide and sodium salts are converted to chloride. Measured, cold, saturated aqueous chloride solutions are treated with dry hydrogen chloride gas, which separates the bulk of the sodium chloride. The residual potassium, calcium and magnesium can then be determined by the usual methods.

Calcium and magnesium may also be determined with or without preliminary sodium chloride separation.

THE potassium content of commercial sodium metal is about 0.005 per cent. This is below the limits for direct quantitative determination of potassium in sodium^{1,2,3,4} and necessitates preliminary separation of sodium or concentration of potassium.

In the preliminary separation as sodium chloride, the initial weight of test sample is reduced from 5 to 50 g of sodium chloride to 0.2 to 0.5 g of concentrate, containing all of the potassium, calcium and magnesium with incidental sodium chloride. This concentrate may contain 0.5 mg or more of potassium perchlorate, which can be conveniently determined by the ethyl acetate extraction procedure.⁴

This paper describes the precipitation of the bulk of the sodium chloride from aqueous solution by hydrogen chloride gas^{5,6} and results are presented to demonstrate the effectiveness of the separation of the potassium from the bulk of the sodium chloride. Potassium (as well as calcium and magnesium) is not retained by the sodium chloride precipitate.

A method for the direct determination of calcium^{7,8} in sodium chloride has been reported, but none has been described for magnesium.⁹ These direct determinations may be used as check methods against the pre-concentration procedure for the determination of calcium and magnesium.

METHOD

REAGENTS—

Acetic acid solution of 8-hydroxyquinoline—Place 5 g of 8-hydroxyquinoline and 10 ml of glacial acetic acid in a beaker. Warm to 50° to 60°C for several minutes to complete solution and dilute to 200 ml with water. Discard after 2 weeks.

Alcoholic solution of 8-hydroxyquinoline—Dissolve 5 g of 8-hydroxyquinoline in 200 ml of ethyl or methyl alcohol. Discard after 2 weeks.

Ammonium oxalate solution—Dissolve 10 g of the salt in 100 ml of water.

Saturated hydrochloric acid solution—Saturate hydrochloric acid, sp.gr. 1.2, with dry hydrogen chloride gas, at 0°C.

Wash water, pH 10—Add ammonium hydroxide, sp.gr. 0.880, to distilled water until the pH is 10.

PROCEDURE FOR DIRECT DETERMINATION OF POTASSIUM—

Metallic sodium—Weigh the sodium metal plus container. Open the container and transfer it and the sample to a weighed glass beaker. Treat the sample with discrete portions of anhydrous methyl or ethyl alcohol until the metallic sodium has been converted to the alcoholate. Small portions of water may be added cautiously to accelerate the action. Remove the container and wash, dry and weigh it in order to find the amount of metallic sodium taken.

Add water to the alcoholate solution to increase its volume by a quarter and mix thoroughly. The sodium content if as low as 1 to 3 g may also be determined at this point

by titration with *N* hydrochloric acid solution. Add concentrated hydrochloric acid to the alcoholate solution until the solution turns litmus paper red, and add about 5 ml of hydrochloric acid in excess. Evaporate the solution nearly to dryness, then bake the residue for 4 to 12 hours in an oven at 110° to 130° C. Cool and weigh. The gain in weight is equivalent to the amount of sodium chloride. This weight of sodium chloride may be used to calculate or check the weight of metallic sodium taken for analysis.

Divide the weight of sodium chloride in grams by 0.35 (the solubility of sodium chloride in water is 35 per cent.) and add this calculated amount of water to the beaker containing the sodium chloride. Warm the solution to dissolve the salt completely. Cool the beaker in the refrigerator to 0° C and weigh the beaker and contents cold.

Sodium hydroxide—Weigh the sodium hydroxide and transfer the sample to a beaker, weighed to the nearest tenth of a gram, containing five times as much water as sodium hydroxide by weight. Add a measured amount of hydrochloric acid to make the solution just acid. Cool. Calculate the weight of sodium chloride formed. Evaporate the solution until the beaker contains a saturated solution of sodium chloride.

Sodium chloride—Weigh the sodium chloride, transfer the sample to a weighed beaker and add sufficient water to make a saturated solution of the sodium chloride.

Separation of salt—Pass dry hydrogen chloride gas through a train into the cold solution. If a tank of gas is available, successively pass the gas through a wash bottle of sulphuric acid, a safety bottle and an inverted funnel or thistle tube into the aqueous sodium chloride solution. The funnel or thistle tube should be well below the surface of the salt solution. Set the cold sodium chloride solution in an ice-bath and maintain the ice in the bath throughout the action. Pass in dry hydrogen chloride gas slowly at first, then increase the flow of gas until bubbles pass through the sodium chloride solution. If the amount of sample is large the salt solution may become hot, but it will cool when the absorption of gas is complete. The volume will increase by about 30 per cent. When the absorption seems complete and the liquid is fuming, remove the beaker from the system and weigh it. The action is complete when the salt solution has increased in weight by 47 to 51 per cent.—roughly, twice the weight of the dry sodium chloride. Set the covered beaker in the refrigerator until it is ready for filtration.

Decant the cold fuming solution through a cold dry 500-ml sintered-glass crucible, transfer the solid to the crucible and wash it with 20 ml of cold hydrochloric acid saturated with hydrogen chloride. Discard the residue. Evaporate the filtrate to a small volume, transfer the solution to a 150-ml beaker weighed to the nearest hundredth of a gram and evaporate the liquid nearly to dryness; dry the residue in an oven at 110° to 130° C. Cool and weigh. The gain in weight (about 0.2 g) represents sample concentrate, and the amount of ethyl acetate to be used will depend on this weight.

Separation of potassium from sodium—Add 5 ml of nitric acid, sp.gr. 1.4, and 5 ml of 72 per cent. perchloric acid and evaporate nearly to dryness, leaving only sufficient perchloric acid to prevent formation of insoluble ferric oxide. Cool to room temperature. Add 6 ml of anhydrous ethyl acetate (5 ml will dissolve the equivalent of 0.3 g of sodium chloride) and heat. Complete the potassium determination by the procedure of Willard and Smith.⁴

PROCEDURE FOR SEQUENCE DETERMINATION OF CALCIUM, MAGNESIUM AND POTASSIUM—

Preliminary separation from sodium—Proceed as described for direct potassium determination with sodium metal, sodium hydroxide or sodium chloride to the point where the dried sample concentrate would be treated with nitric and perchloric acids. Instead add 50 ml of water to the 150-ml beaker containing the sample concentrate of sodium chloride. Warm to dissolve the salts.

Separation of calcium and magnesium from potassium and sodium—Add 15 ml of the alcoholic solution of 8-hydroxyquinoline and warm to 60° to 80° C. Slowly add ammonium hydroxide, sp.gr. 0.880, until the pH is raised to between 9.5 and 10.5. A pH meter or thymolphthalein indicator may be used. Heat to incipient boiling and then cool to room temperature. Check the pH reading, adding ammonium hydroxide, if necessary. Filter the solution through a 12.5-cm Whatman No. 40 filter-paper into a 250-ml beaker. Wash the beaker with an aqueous solution of ammonium hydroxide adjusted to a pH value of 10. Wash the precipitate and paper several times with the wash-water. Reserve the filtrate for potassium determination.⁴

Calcium determination—Return the paper and its contents to the beaker and add 15 ml of nitric acid, sp.gr. 1.4, and 8 ml of 72 per cent. perchloric acid. Mix and evaporate the solution to heavy fumes of perchloric acid. Cool. Dilute to 50 ml with water and boil for 1 minute. If the solution appears cloudy, filter, wash and discard the residue.

Precipitate the calcium as oxalate, filter, reserve the filtrate for magnesium determination, and complete the calcium determination by titrating with 0.02 *N* potassium permanganate solution in the usual manner.

Magnesium determination—Heat the filtrate from the calcium oxalate precipitation to boiling and slowly add 15 ml of the solution of 8-hydroxyquinoline in acetic acid. If iron is present a black precipitate forms and the supernatant liquid should be yellow. Additional reagent is added, if necessary, to make the solution yellow. Stir the solution until the black precipitate coagulates, then filter off the iron precipitate on a Whatman No. 40 filter-paper containing paper pulp and collect the filtrate in a 400-ml beaker. Do not allow the black particles to creep above the paper. Wash the paper and precipitate with 0.2 per cent. v/v acetic acid solution. Discard the paper and the iron precipitate.

If the solution is not yellow, add 5 to 10 ml of alcoholic 8-hydroxyquinoline reagent to the filtrate and then warm to 60° to 80° C.

Complete the magnesium determination by the procedure of Berg.⁹

Potassium determination—To the reserved potassium solution, add 15 ml of nitric acid, sp.gr. 1.4, and 8 ml of 72 per cent. perchloric acid, evaporate to heavy fumes of perchloric acid and continue heating until nearly all the perchloric acid has been evaporated. Heat the beaker over an uncovered bunsen burner flame to volatilise ammonium salts.

Complete the potassium determination by the procedure of Willard and Smith.⁴

PROCEDURE FOR DIRECT DETERMINATION OF APPRECIABLE AMOUNTS OF CALCIUM AND MAGNESIUM⁸—

Convert the metallic sodium, sodium hydroxide or other sodium samples to sodium chloride. Adjust the volume of solution so that the salt content is 20 to 25 per cent. and proceed according to the section on separating calcium and magnesium from potassium and sodium (p. 718).

DISCUSSION OF RESULTS

The effective separation of the bulk of the sodium chloride is shown in Table I. The first column shows the weight of sodium chloride (weighed or calculated), the second the

TABLE I
SEPARATION OF SODIUM CHLORIDE BY HYDROGEN CHLORIDE GAS

NaCl, g	Water added, ml	HCl absorbed, g	Gain in weight from HCl, %	Weight of sample concentrate, g
38.8	110.9	37.8	25	0.80
47.8	136.8	80.5	44	0.70
69.7	194.3	57.8	22	0.50
116.5	329.0	62.1	14	14.8
117.2	334.7	180.5	40	3.3
122.3	349.4	240.0	51	0.45
149.9	428.3	285.0	50	0.62
69.45	198.4	138.3	52	0.24
61.8	176.7	112.8	48	0.22
22.45	64.1	40.5	47	0.12
57.35	163.9	105.3	48	0.20
10.75	30.7	19.2	47	0.10
4.85	13.9	9.0	48	0.09
9.75	27.9	18.1	48	0.05
84.95	242.7	156.3	48	0.30
56.65	161.9	100.0	46	0.24
70.5	201.4	135.4	50	0.19
54.2	154.9	98.5	47	0.18
44.65	127.6	87.4	51	0.15
40.0*	114.0	72.2	47	0.15
40.0*	114.0	74.1	48	0.15
40.0*	114.0	71.9	47	0.14

* Synthetic sample.

amount of water added and the third the weight of hydrogen chloride gas absorbed. The fourth column shows the percentage gain in weight of the solution and the fifth shows that all but a small amount of salt is precipitated from solution.

It is evident from Table I that a sample weight of 15 to 20 g of sodium metal (40 g of sodium chloride) can be used, and that all but about 0.2 g of salt can be precipitated from solution when the salt solution has absorbed nearly 50 per cent. of its weight of hydrogen chloride.

For the results in Table II, a 40-g sample of sodium chloride was used, and blank determinations of potassium, calcium and magnesium were made. After this, known amounts of the three elements were added to 40-g samples of the same batch of C.P. sodium chloride, and the three elements were determined. It should be noted that the C.P. sodium chloride sample itself contains 0.002 per cent. of potassium, 0.0002 per cent. of calcium and 0.00016 per cent. of magnesium, and these are the blank values that must be subtracted, as mentioned in a footnote to Table II.

TABLE II
SYNTHETIC SOLUTIONS OF POTASSIUM, CALCIUM AND MAGNESIUM IN 40 g
OF SODIUM CHLORIDE

		By preliminary hydrogen chloride separation								
		Potassium			Calcium			Magnesium		
Elements added		Added, mg	Found, mg	%	Added, mg	Found, mg	%	Added, mg	Found, mg	%
None.	C.P. sample	—	0.80	0.0020	—	0.082	0.00020	—	0.065	0.00016
	used for blank values	—	0.84*	0.0021	—	0.10	0.00025	—		
	K	0.3	0.3†	0.00075						
	K, Ca	0.30	0.40*†	0.0010	0.40	0.48†	0.0012			
	Ca				0.30	0.25†	0.0006			
	Ca, Mg							0.40	0.36†	0.0009
	Ca, Mg							0.40	0.43†	0.0011
		By direct 8-hydroxyquinoline separation								
		Calcium			Magnesium					
Elements added		Added, mg	Found, mg	%	Added, mg	Found, mg	%			
None.	C.P. sample	—	0.08	0.0002	—	0.05	0.00013			
	used for blank values									
	K									
	K, Ca									
	Ca									
	Ca, Mg	0.20	0.20†	0.0005	0.40	0.35†	0.0009			
	Ca, Mg	0.40	0.35†	0.0009	0.40	0.35†	0.0009			

* No potassium found in re-worked precipitate.

† Corrected for blank values.

The solubility of the potassium, calcium and magnesium in the mixture of cold saturated hydrochloric acid and sodium chloride solution is important. The calcium and magnesium form anionic complexes that are soluble. Only the potassium has limited solubility, but in the technique described no error need be expected. For example, consider the synthetic sample in Table I. For 40 g of sodium chloride, 114 ml of water are used, and after the addition of hydrogen chloride the volume has increased by 30 ml. From this precipitation 0.15 g of sample concentrate was left in solution. From the point of view of the common ion effect, this residuum could be divided as 80 per cent. of sodium and 20 per cent. of potassium, *i.e.*, 30 mg of potassium chloride (15 mg of potassium) would remain in solution. Since the obvious application of this analytical technique is to separate small amounts, or at the most 5 mg, of potassium from large amounts of sodium, the technique is well-founded.

It is also necessary to prove that no potassium is occluded by sodium chloride. This is shown experimentally in Table II. Here, the precipitated sodium chloride was filtered from the sample concentrate, dissolved in water, precipitated as before with hydrogen chloride gas and a second sample concentrate was collected. No potassium was found in this second

sample concentrate, and it is then stated that no potassium is found in the re-worked precipitate.

TABLE III
DETERMINATION OF POTASSIUM IN SODIUM METAL

Number of experiment*	Type of metal	Amount of metal, g	Potassium found, mg	Potassium, %
1	As received	33.4	2.9	0.009
1	As received	39.8	4.7	0.012
1	As received	31.5	7.0	0.022
2	Distilled†	15.7	1.4	0.009
2	Distilled†	17.2	1.4	0.008
3	Treated‡	23.75	0.72	0.003
3	Treated‡	21.95	0.41	0.002
3	Treated‡	17.7	0.51	0.003
4	Treated‡	29.8	0.68	0.002
4	Treated‡	29.4	1.53	0.004
5	Treated‡	38.8	0.3	0.001
5	Treated‡	24.5	0.2	0.001
5	Treated‡	22.4	0.6	0.003

* Samples with the same number are considered as nearly duplicate samples.

† Loftness, Ruebsamen and Coultas.¹⁰

‡ Ruebsamen.¹¹

This procedure has particular application to the determination of small changes of potassium metal content in treated and untreated sodium metal (see Table III). Samples received from the manufacturer contain 0.01 to 0.02 per cent. of potassium. Vacuum distillation reduces the figure, but treatment with graphite chips⁴ reduces the amount of potassium to about 0.001 per cent. The sets of treated samples are considered as duplicates from the same operation.

To check the results for calcium and magnesium the method described for direct 8-hydroxyquinoline separation (p. 719) was used, as shown in Table II. It was thought that the results obtained from the preliminary hydrogen chloride separation (p. 718) would be higher, but this was not so.

This report is based on studies conducted for the Atomic Energy Commission under contract AT-40-1-GEN-1064.

REFERENCES

1. Adams, M. F., and St. John, J. L., *Ind. Eng. Chem., Anal. Ed.*, 1945, **17**, 435.
2. Adie, R. H., and Wood, T. B., *J. Chem. Soc.*, 1900, **77**, 1076.
3. Kolthoff, I. M., and Bendix, G. H., *Ind. Eng. Chem., Anal. Ed.*, 1939, **11**, 94.
4. Willard, H. H., and Smith, G. F., *J. Amer. Chem. Soc.*, 1922, **44**, 2819; 1923, **45**, 286.
5. Mellor, J. W., "Inorganic and Theoretical Chemistry," Volume 2, Longmans, Green & Co. Ltd., London, 1946, p. 542.
6. Seidell, A., "Solubilities," Volume I, D. Van Nostrand Co. Inc., New York, 1940.
7. Berg, R., "Die analytische Verwendung von O-Oxyquinoline und seiner Derivate," Ferdinand Enke, Stuttgart, Germany, Second Edition, 1938, p. 40.
8. Rynasiewicz, J., and Polley, M. E., *Anal. Chem.*, 1949, **21**, 1398.
9. Berg, R., *op. cit.*, p. 30.
10. Loftness, R. L., Ruebsamen, W. C., and Coultas, T., NAA-SR-126, November 20th, 1951.
11. Ruebsamen, W. C., "The Removal of Small Amounts of Potassium from Sodium," NAA-SR-139, July 27th, 1951.

ATOMIC ENERGY RESEARCH DEPARTMENT
NORTH AMERICAN AVIATION INC.
DOWNEY, CALIFORNIA, U.S.A.

First submitted, August 25th, 1952
Amended, June 22nd, 1953

An Isopiestic Method for the Micro-determination of Molecular Weights

By Miss J. E. MORTON, A. D. CAMPBELL* AND T. S. MA

A simple method is described for the determination of the molecular weight of non-volatile compounds; it is based on Sinclair's isopiestic method for the determination of vapour pressures. A solution of the compound in a volatile solvent is rocked at constant temperature in a small desiccator with a solution, in the same solvent, of a substance of known molecular weight. When the two solutions are isopiestic, the molecular weight of the compound is calculated from the molecular weight of the standard substance and the concentrations (by weight) of the two solutions. For 3 to 7-mg samples concordant results have been obtained with a number of solvents.

IN microchemical work there is a need for a simple yet accurate method for the determination of molecular weights on 3 to 7-mg samples. Both the ebullioscopic and cryoscopic methods at present in use require extremely careful control by a skilled worker before accuracy can be attained.

When two solutions containing different solutes are placed in a closed space, vapour will distil from the solution of higher vapour pressure (*i.e.*, the more dilute solution) and condense in the one having the lower vapour pressure until equality of vapour pressure is attained. This isopiestic principle has been used for the determination of approximate molecular weights by Barger,¹ who placed drops, separated by small air spaces, of the two solutions in a capillary tube. By observing the direction of the distillation of the solvent under a microscope he could determine which solution had the greater vapour pressure, and by varying the concentration of one of the solutions he was able to determine when the two solutions were isopiestic. From the concentrations by weight of the isopiestic solutions and the molecular weight of one solute, the molecular weight of the other could be determined. In spite of many variations by later workers the probable error is high, as it is not possible to avoid completely some contamination of one solution by the other. Higher accuracy was attained by Clark,² but his method required a 20-mg sample. The two solutions were placed one in each arm of an H-shaped apparatus, from which air was evacuated. The concentration of the two solutions was determined by inverting the apparatus so that the solutions ran into accurately calibrated measuring tubes.

Most isopiestic methods suffer from the disadvantage that equilibrium is attained only slowly, but this was overcome by Sinclair^{3,4} who, when making extremely accurate vapour pressure determinations, placed the two solutions in gold-plated silver dishes in close contact on a copper block in an evacuated desiccator, which was rocked in a thermostat. In the method described here Sinclair's method has been adapted to the determination of the molecular weights of a number of organic compounds in various volatile solvents.

A more elaborate apparatus has been described by Claesson⁵; it was designed particularly for the determination of molecular weights between 1000 and 10,000. It was similar to the apparatus used by Scatchard⁶ for the determination of activities, but had an additional attachment for closing the silver beakers containing the solutions before the apparatus was opened. Wright⁷ described a very simple apparatus in which the solutions to be equilibrated were adsorbed on filter-papers in two aluminium trays that could be fitted together for equilibration, but the order of accuracy was not high.

METHOD

APPARATUS—

The solutions are contained in two platinum crucibles of 1.3 ml capacity that fit into two holes in a brass block weighing about 250 g placed inside a small vacuum desiccator of approximately 250 ml capacity (see Fig. 1). To prevent loss of volatile solvent, the platinum crucibles are weighed inside small weighing bottles with tightly fitting ground-glass stoppers.

* Present address: Chemistry Department, The University, Glasgow, Scotland.

PROCEDURE—

Weigh the sample, which should be between 3 and 7 mg (a larger sample must be taken for compounds of very high molecular weight), in one of the platinum crucibles and weigh the standard substance in the other. If the approximate molecular weight of the sample is known, the attainment of equilibrium can be speeded by taking similar molar quantities of sample and standard. Add the solvent, *e.g.*, about 0.3 ml of chloroform, to each of the two crucibles and then weigh them inside the counterpoised weighing bottles fitted with

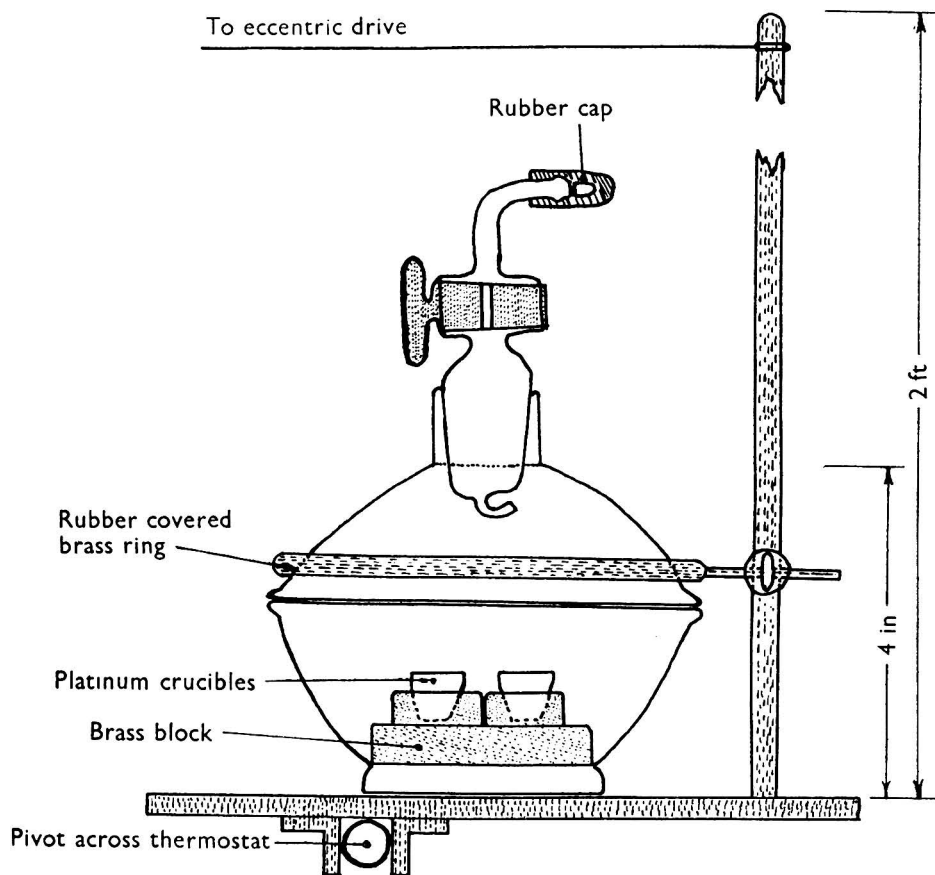


Fig. 1. Micro vacuum desiccator mounted in thermostatically controlled bath

ground-glass stoppers. Place the crucibles in the holes in the brass block inside the desiccator. Pour a small quantity of solvent (about 0.5 ml) on the brass block and then evacuate air from the desiccator under controlled conditions so that each crucible still contains 100 to 200 mg of solvent at the end of the experiment. This is achieved by connecting the desiccator through a large bottle, of capacity about $2\frac{1}{2}$ litres, to a water pump. With the connection between the bottle and the desiccator closed, reduce the pressure in the bottle to 200 mm of mercury; then close the connection between the bottle and the pump and open that between the bottle and the desiccator and allow the pressure to equalise. Disconnect the desiccator and rock it at 10 strokes per minute through an angle of 30° in a water-bath at 25°C for 24 hours. At the end of this time open the desiccator and quickly place the crucibles inside the weighing bottles and weigh them. Then return the crucibles to the desiccator, place a few drops of solvent inside the jar and evacuate the apparatus as before. After a further 24 hours of rocking in the water-bath, again weigh the crucibles.

The solutions are assumed to be isopiestic when the ratio of their concentrations (by weight) is constant for two consecutive readings at 24-hour intervals. The molecular weight of the sample is then calculated from the formula—

$$M_1 = \frac{W_1 S_2 M_2}{W_2 S_1},$$

where M_1 and M_2 are the molecular weights of the unknown and standard substance, respectively, W_1 and W_2 are the weights of the unknown and standard substance, and S_1 and S_2 are the weights of solvent in the crucible containing the unknown and standard substance, respectively.

The approach to equilibrium with time is shown by a comparison of the weight ratios of the standard and unknown solutions, *i.e.*, by determination of W_1/S_1 divided by W_2/S_2 . When equilibrium is reached this ratio becomes constant. The approach to constant weight of the solution of the unknown cannot be used as a criterion of equilibrium because the breaking of the vacuum and subsequent re-evacuation of air alters the total amount of solvent in the system. In preliminary work the same solute, azobenzene, was used in both crucibles.

RESULTS AND DISCUSSION

The results shown in Table I indicate the high order of accuracy attainable by this method for a number of compounds and various volatile solvents.

TABLE I
MOLECULAR WEIGHT DETERMINATIONS

Compound	Weight of compound, mg	Solvent	Weight of solvent at equilibrium, mg	Molecular weight		Error, %
				Found	Theoretical	
<i>p</i> -Phenyl-phenacyl bromide	6.441	Chloroform	81.2	278	275	+1.1
Azobenzene (m.w. 182) ..	3.369		64.8			
<i>p</i> -Phenyl-phenacyl bromide	6.516	Chloroform	112.2	278	275	+1.1
Azobenzene (m.w. 182) ..	3.438		90.4			
2:4-Dinitrochlorobenzene ..	5.443	Benzene	90.6	194	192.5	+0.8
Phenyl benzoate (m.w. 198)	5.521		90.3			
Salicylic acid ..	3.040	Acetone	95.3	137	138	-0.7
Azobenzene (m.w. 182) ..	3.967		93.6			
Adipic acid ..	3.102	Acetone	113.4	144	146	-1.4
Azobenzene (m.w. 182) ..	3.874		112.3			
Phenacetin ..	5.020	Methanol	125.2	167	169	-1.2
Azobenzene (m.w. 182) ..	4.877		111.2			
Adipic acid ..	4.675	Methanol	122.6	143	146	-2.1
Phenacetin (m.w. 179) ..	5.248		116.4			
Salicylic acid ..	5.308	Ethanol	85.2	135	138	-2.2
Benzoic acid (m.w. 122) ..	4.855		85.90			
Salicylic acid ..	5.671	Ethanol	150.2	139	138	+0.7
Benzoic acid (m.w. 122) ..	4.957		149.3			
Methyl oleate ..	6.110	Chloroform	191.5	302	292	+3.4
Azobenzene (m.w. 182) ..	5.563		288.6			
Sesquiterpene nitrosate ..	6.922	Chloroform	114.6	679	684 or 342	-0.7
Azobenzene (m.w. 182) ..	4.671		288.6			
Sesquiterpene nitrosate ..	12.365	Benzene	102.5	716	684 or 342	+4.7
Phenyl benzoate (m.w. 198)	4.890		146.7			
Caffeine ..	4.342	Chloroform	108.5	194	196	-1.0
Azobenzene (m.w. 182) ..	4.846		128.7			
Standard deviation ..						±1.6

NOTE—The second compound of each pair was used as the reference substance of known molecular weight.

Solutions in chloroform, acetone and benzene attain equilibrium in 24 hours and therefore the first two determinations at 24-hour intervals are in agreement, but with ethanol and methanol a third determination is necessary before agreement is attained. The quantity

of solvent placed in the desiccator and in the crucibles depends on the nature of the solvent used, the pressure to which the desiccator is evacuated and the size of the desiccator. The quantities of solvent and the pressure quoted in the procedure have been found suitable when chloroform is used as solvent. With chloroform and other very volatile solvents a correction may have to be made for loss of solvent from the weighing bottles during the weighing operation, but it is possible to grind the stoppers so that this loss is negligible. The choice of standard substance and solvent is governed by the solubility at room temperature, the stability and a consideration of the possibility of association in the solvent concerned. The limitations of the method are determined by the well-known deviations from Raoult's law.

As residual air in the apparatus has been found to slow up the equilibration,⁶ the controlled evacuation should be repeated once or twice for solvents that are less volatile than chloroform.

MACRO-DETERMINATION OF MOLECULAR WEIGHTS

With readily available laboratory apparatus and an ordinary analytical balance, the isopiestic principle can be used to determine the molecular weights of compounds on a 0.2-g sample. This is no great disadvantage, as the sample is readily recovered from the solution at the end of the experiment.

The results in Table II were recorded with glass weighing bottles, of diameter 3 cm, as containers for the two solutions placed inside a glass jar of 500 ml capacity that was adapted for evacuation and was rocked in a thermostatically controlled bath. The jar was evacuated to a pressure of 50 mm of mercury by water pump, controlled evacuation being unnecessary on the macro scale.

TABLE II
MOLECULAR WEIGHT DETERMINATIONS ON A MACRO SCALE

Compound	Weight of compound, g	Solvent	Weight of solvent at equilibrium, g	Molecular weight		Error, %
				Found	Theoretical	
Phenyl benzoate ..	0.2010	Chloroform	1.593	198	198	0.0
Azobenzene (m.w. 182) ..	0.1999		1.728			
Benzoic acid ..	0.2326	Acetone	1.604	123	122	+0.8
Azobenzene (m.w. 182) ..	0.3030		1.410			
Glutaric acid ..	0.2162	Methanol	1.8442	130	132	-1.5
Succinic acid (m.w. 118) ..	0.2186		2.0626			
Salicylic acid ..	0.2436	Methanol	1.1676	139	138	+0.7
Glutaric acid (m.w. 132) ..	0.2264		1.142			
Adipic acid ..	0.2498	Methanol	1.9282	145	146	-0.7
Succinic acid (m.w. 118) ..	0.2526		2.3910			
Standard deviation ..						±0.5

The authors thank Professor F. G. Soper for suggesting the subject of this investigation and for his interest and helpful advice. They also acknowledge grants from the Mellor Research Fund.

REFERENCES

1. Barger, G., *J. Chem. Soc.*, 1904, **85**, 286.
2. Clark, E. P., *Ind. Eng. Chem., Anal. Ed.*, 1941, **13**, 820.
3. Sinclair, D. A., *J. Phys. Chem.*, 1933, **37**, 495.
4. Sinclair, D. A., and Robinson, R. A., *J. Amer. Chem. Soc.*, 1934, **56**, 1830.
5. Claesson, S., *Ark. Kemi*, 1949, **1**, 81.
6. Scatchard, G., Hamer, W. J., and Wood, S. E., *J. Amer. Chem. Soc.*, 1938, **60**, 3061.
7. Wright, R., *Analyst*, 1948, **73**, 387.

CHEMISTRY DEPARTMENT
UNIVERSITY OF OTAGO
DUNEDIN, NEW ZEALAND

February 9th, 1953

Notes

THE LIPASE ACTIVITY OF CERTAIN CEREAL PRODUCTS

SOMETIMES off-flavours in finished baked products can be caused by the action of lipase originally present in the flour or cereal product used in their manufacture. This effect is well-known in baked oat products in which the oatmeal has not had sufficient treatment to destroy the lipase originally present in the grain. Methods of treatment designed to destroy lipase activity have been described by Moran.¹

Hutchinson and Martin² have published a method for assessing lipase activity in oats and oatmeals, and a modification mentioned by these authors seemed the most promising as a means of assessing the activity of malt flours and other flours. The method was tried and the results may be of interest to analysts investigating off-flavours in foodstuffs.

The method has been used by us in a large number of investigations and the results have proved informative. As an example, the results of an examination of five samples of malted barley flour, a malted wheat flour and two wholemeal wheat flours are quoted below. Some of the samples were known to have been used in products about which complaints of off-flavour had been made. Comparison was also made with two fine and medium-ground oatmeals, known to have caused a soapy flavour in oatcakes.

TABLE I
LIPASE ACTIVITY OF SAMPLES OF FLOUR AND OATMEAL

Sample	Amount of oil hydrolysed after incubation for				
	2 hours, %	3 hours, %	4 hours, %	5 hours, %	6 hours, %
1. Barley malt flour	0.68	1.24	1.06	1.09	1.44
2. "	0.51	0.72	1.18	1.27	1.43
3. "	0.53	0.55	0.72	0.92	0.93
4. "	1.28	2.34	2.23	2.88	2.87
5. "	0.35	0.56	0.88	0.65	1.26
6. Wheat malt flour	0.64	0.78	0.95	0.94	1.76
7. Wholemeal flour	0.10	0.09	0.03	0.08	0.07
8. "	1.24	1.63	1.64	1.67	2.33
9. Oatmeal					29.08
10. "					41.49

The flours were defatted by washing with light petroleum as described by Hutchinson and Martin,² so that no complications from differences in substrate were involved.

Six portions of 0.5 g of the defatted malt flour were weighed and placed in 2-inch \times $\frac{1}{2}$ -inch tubes, 7 drops (about 0.2 g) of olive oil and 0.2 ml of water were added to five of the tubes, and the time at which the water was added was noted. The mixture was stirred into a smooth dough with a glass rod and the tubes were tightly corked. The tubes were placed in a bath at 37° C and incubated for 2, 3, 4, 5 or 6 hours.

When incubation was completed for any tube, it was removed from the bath and the oil and free fatty acid were extracted from the dough by stirring vigorously with a glass rod with successive small portions of light petroleum (A.R. grade boiling between 40° and 60° C) until 25 ml had been used. The light petroleum extract was centrifuged to remove the solid matter and then decanted into a tared flask. The light petroleum was distilled off, and the oil and free fatty acid were dried for an hour in an air oven at 100° C and weighed.

The oil and free fatty acid were dissolved in 25 ml of a neutral (1 + 1) mixture of alcohol and benzene, brought to the boil on the water-bath and titrated with 0.02 *N* alcoholic sodium hydroxide, with phenolphthalein as indicator.

The experimental blank was determined on the sixth portion by adding 7 drops of olive oil, but no water, and then treating it exactly as the others but without incubation.

By assuming the free fatty acid to be oleic acid, the proportion of oil hydrolysed was calculated as a percentage of the total oil.

Each assessment was carried out three times and the means of the three results are shown in Table I. The amount of olive oil hydrolysed after incubation for 6 hours seemed to give the most useful indication of lipase activity, the differences between replicates being least at this period.

Two experiments with doughs of higher water content were made in an attempt to reduce the time required for the assessment, but the quantities of oil recovered from the mixtures were too small for a reliable test.

Finally, three 6-hour incubations were run simultaneously and "worked up" together. The results were of the same order as those that had been obtained separately.

It is evident that there was a distinct difference between samples 3 and 4, sample 4 being known from other experiments to have more lipase activity than is desirable in a malt flour. Sample 6, the wheat malt flour, was known to have an undesirably high lipase activity, having given a soapy taste to biscuits, and a higher percentage of olive oil was hydrolysed by this flour than by sample 5, which was not suspect. Of the two wholemeal flours, sample 7 had almost no lipase activity, whilst sample 8 had sufficient to impart distinctly adverse odours and flavours to the flour. Both the oatmeals were highly active, between thirty and forty times as active as any of the other flours examined. Oatcakes made with these meals had a very unpleasant taste.

From the results it would appear that a malt or wholemeal wheaten flour with sufficient lipase activity to hydrolyse rather more than 1.5 per cent. of olive oil in 6 hours might not be safe to use, but much depends on the proportion of malt flour in the goods and the time that elapses between mixing and baking.

Hard and fast conclusions cannot be drawn from the figures shown, but they give an indication of the state of the flour and whether or not it is responsible for the soapy taste in samples of baked goods.

We wish to express our appreciation and thanks to Mr. H. F. Martin of the Cereals Research Station, St. Albans, for his help in showing us the latest modification to his method, and we are indebted also to the Council of the Research Association for permission to publish this note.

REFERENCES

1. Moran, T., *Food Manuf.*, 1952, **27**, 73.
2. Hutchinson, J. B. and Martin, H. F., *J. Sci. Food Agric.*, 1952, **3**, 312.

BRITISH BAKING INDUSTRIES RESEARCH ASSOCIATION
CHORLEYWOOD, HERTS.

W. H. TEMPLETON
B. R. CARPENTER
June 12th, 1953

TESTS FOR NITRITE AND NITRATE APPLICABLE OVER WIDE CONCENTRATION RANGES

THE long-established method for identifying nitrite in solution by diazotisation has given rise to many investigations into the possibility of estimating very small amounts. The method of Ilosvay¹ makes use of sulphanilic acid and α -naphthylamine as the two reagents to be coupled; this reaction can be used for quantitative colorimetric analysis if certain precautions are observed.² Many other reagents have been suggested for attaining increased sensitivity and a more stable colour, e.g., dimethyl- α -naphthylamine and sulphanilic acid,^{3,4} dimethylaniline and sulphanilic acid,⁵ and N- α -naphthylethylenediamine hydrochloride and sulphanilamide^{6,7}; comparative tests have also been carried out.⁸

A good qualitative colorimetric test should give (a) a positive indication at any concentration likely to be encountered when the test is applied under standard conditions and (b) an approximate indication of the concentration of the test solution (nitrite in this instance) according to the intensity of the colour produced. It is never safe to assume that a test that is perfectly satisfactory for solutions of molarity 10^{-5} or less will be equally satisfactory when applied to solutions that may be molar. On this basis the Ilosvay test is clearly unsuitable for use in routine analysis, since it gives its maximum colour intensity with about 0.01 M nitrite (with the added objection that this colour is unstable and soon changes to a brown deposit); with concentrated solutions only a very pale yellow colour is produced. An excess of nitrite is capable of destroying the red colour even after it has been formed—presumably because the possibilities for resonance are much more restricted when the amine is converted into a diazonium salt. This latter difficulty can be avoided by the use of a tertiary, instead of a primary, amine as coupling agent; but aromatic tertiary amines readily give nitroso derivatives with free nitrous acid, and the nitrite test in concentrated solution gives a muddy coloured unstable precipitate.

For routine identification of nitrite, phenols in alkaline solution are preferable to amines in acid solution. Results are good with sulphanilic acid and α -naphthol (for which a concentration

limit of 1 part in 10^7 was claimed⁹ in 1887); the diazotisation is carried out in acetic acid solution and the product is poured into an alkaline solution of α -naphthol. Benford and Ingold¹⁰ have recommended a somewhat similar procedure for the quantitative determination of traces.

METHOD FOR THE IDENTIFICATION OF NITRITE

PROCEDURE—

To 1 ml of the suspected nitrite solution, which should be neutral, add 0.5 ml of sulphanilic acid solution (1 per cent. w/v in 5 *N* acetic acid). Shake well, allowing at least 10 seconds for the diazotisation to proceed; then pour into 2 ml of a fresh solution of α -naphthol in 2 *N* sodium hydroxide or 2 *N* ammonium hydroxide. The concentration of this α -naphthol solution is not critical, but an approximately 0.5 per cent. w/v solution is recommended. A deep red colour, which is stable, and the intensity of which depends on the original concentration of nitrite, is produced. If traces are being sought, diazotisation should take about 10 minutes to reach completion; the solution is then made alkaline. The reagents used should all be of analytical reagent quality.

INTERFERING RADICALS—

Sulphate, nitrate, fluoride, chloride, bromide, silicate, borate, phosphate, arsenate and the common organic acids do not interfere. The slight colour found by Odekerken¹¹ in the Ilosvay test with fluoride, chloride, sulphate, nitrate and carbonate can almost certainly be ascribed to traces of nitrite present as impurity. Iodide, chromate, sulphite, thiosulphate and ferrocyanide all interfere to some extent; addition of lead acetate precipitates the corresponding lead salt, and there is no need to filter, as these precipitates are not appreciably soluble in the dilute acetic acid of the sulphanilic acid reagent and are redissolved from the diazotised solution when it is poured into the sodium hydroxide. Ferricyanide also interferes and can be removed similarly by the addition of zinc sulphate solution. Cyanide and thiocyanate interfere if present in large amounts; they can be precipitated by careful addition of silver sulphate solution (or nitrate, if this radical is not being sought) until a faint permanent precipitate is just produced for the cyanide, or until precipitation is complete for the thiocyanate. If the alkali used for making the α -naphthol solution is ammonium hydroxide, any excess of silver ion will be taken back into solution as the ammine. As the solubility of silver nitrite is only about 0.015 moles per litre at room temperature, any nitrite in excess of this value will be removed along with the interfering radicals. Coloured ions can be allowed for by the use of a comparison solution, or they can be removed by precipitation.

EXTENSION OF THE METHOD TO THE IDENTIFICATION OF NITRATE

The main problem in extending the method to nitrates is to find a suitable reducing agent for the reduction of nitrate to nitrite. The following are all unsuitable for various reasons: magnesium, zinc needles or wool, cadmium, iron, tin or mercury in acid solution; sodium or zinc amalgam; salts such as hypophosphite and organic reducing agents such as formaldehyde. Many textbooks recommend the addition of Ilosvay's reagent to the suspected nitrate, followed by reduction with zinc dust. This procedure is unsatisfactory, as the reduction easily goes much further than the nitrite stage in solutions of such high acidity—the efficiency may easily be much less than 1 per cent.—and the desired azo-compound is itself easily reduced by zinc dust in acetic acid. Consequently, no colour is produced unless the zinc dust is added with extreme caution. Odekerken¹¹ suggests that hydrogen peroxide stabilises the colour produced; it presumably also reduces the rate of reduction, but the method is open to the same objections in a lesser degree. The only satisfactory method is to control the acidity within the range pH 4 to 5, to use only a slight excess of zinc, and to remove this excess by filtration before testing for nitrite in the usual way.

PROCEDURE FOR THE IDENTIFICATION OF NITRATE—

To 1 ml of the suspected nitrate solution, which should be neutral, add 3 ml of acetate buffer (50 ml of 2 *N* acetic acid and 25 ml of 2 *N* sodium hydroxide diluted to 100 ml). Add 40 to 50 mg of zinc dust, shake the mixture for about 30 seconds and then filter. Test the filtrate for nitrite as before. Under these conditions, measurements with a Spekker photo-electric absorptiometer show that the efficiency of reduction varies from about 50 per cent. at 0.25 *M* to about 40 per cent. at 0.0001 *M*, *i.e.*, the reduction is efficient and nearly independent of the concentration.

PROCEDURE IF BOTH NITRITE AND NITRATE ARE PRESENT—

The usual method is to identify the nitrite, destroy it, and then to proceed to the identification of nitrate by reduction to nitrite. Of the three methods commonly used for the destruction of nitrite, Odekerken¹¹ recommends the use of sodium azide in acetic acid solution. Although this method is certainly more rapid than the use of urea in acid solution, both methods are objectionable in so far as a little nitrate is inevitably introduced into the solution during the destruction of the nitrite; consequently the interpretation of any subsequent test is rendered uncertain. Moreover, Odekerken omits to mention the need for removing the excess of azide after the destruction of the nitrite. Clearly this is important, as its reaction with nitrite is fairly rapid even in the cold; the excess of azide can be removed by precipitation with silver sulphate. The precipitated silver azide is not dangerous if thoroughly wet, but it should be destroyed chemically. One would hesitate to recommend such a test to inexperienced students.

Nitrite is best destroyed in *neutral* solution by boiling it with an equal volume of 4 *M* ammonium chloride solution. During the 10 minutes required any loss of nitrate is quite negligible (measurements on the Spekker absorptiometer showed that losses at 0.25 *M* and at 0.0001 *M* are not greater than 5 per cent. and may well be less). There is also the further advantage that any excess of ammonium chloride does not interfere with the subsequent test for nitrite, as this test is carried out in the cold.

University students, when using textbook methods for the detection of nitrite or nitrate, or both, often obtained erroneous results, and their attempts to judge relative amounts were generally unsound. The difficulties have been surmounted by the introduction of the tests described above, which have been found to give reliable indications in terms of such rough quantitative categories as large, small or trace.

REFERENCES

1. Ilosvay, L., *Bull. Soc. Chim. France*, 1889, [3] 2, 388.
2. Rider, B. F., and Mellon, M. G., *Ind. Eng. Chem., Anal. Ed.*, 1946, 18, 96.
3. Wallace, G. I., and Neave, S. L., *J. Bact.*, 1927, 14, 377.
4. Germuth, F. G., *Ind. Eng. Chem., Anal. Ed.*, 1929, 1, 28.
5. Giblin, J. C., and Chapman, G., *Analyst*, 1936, 61, 686.
6. Bratton, A. C., Marshall, E. K., Babbitt, D., and Hendrickson, A. R., *J. Biol. Chem.*, 1939, 128, 537.
7. Shinn, M. B., *Ind. Eng. Chem., Anal. Ed.*, 1941, 13, 33.
8. Barnes, H., and Volkard, A. R., *Analyst*, 1951, 76, 599.
9. Zambelli, L., *J. Chem. Soc.*, 1887, 52, 533.
10. Benford, G. A., and Ingold, C. K., *Ibid.*, 1938, 929 (particularly pp. 933-4).
11. Odekerken, J. M., *Z. anal. Chem.*, 1950, 131, 171.

THE UNIVERSITY
SHEFFIELD, 10

P. WOODWARD
June 15th, 1953

PREPARATION OF A MATERIAL RICH IN "CADION" SUITABLE FOR THE
DETECTION OF CADMIUM

Dwyer's "cation,"¹ *p*-nitrodiazoaminoazobenzene ($C_6H_5.N:N.C_6H_4.NH.N:N.C_6H_4.NO_2$), is probably the most satisfactory reagent for the detection of cadmium. Unfortunately, the use of cation has been limited, because its preparation by Dwyer's procedure¹ or by that of van Nieuwenburg² is rather involved. This note describes a simplified method for preparing a satisfactory grade of cation.

When synthesising cation from *p*-nitrodiazoaminobenzene ($C_6H_5.NH.N:N.C_6H_4.NO_2$) by van Nieuwenburg's procedure,² we observed that crude *p*-nitrodiazoaminobenzene prepared in this laboratory gave a satisfactory spot test for cadmium. The sensitivity to cadmium of this crude *p*-nitrodiazoaminobenzene was essentially the same as that of cation made by the procedures of Dwyer and van Nieuwenburg. Purification of the crude *p*-nitrodiazoaminobenzene by Dwyer's method^{3,4} produced two fractions. One is composed of fairly pure *p*-nitrodiazoaminobenzene; it is inactive with respect to cadmium. The other fraction, consisting of impure cation, gives an excellent spot test for cadmium. This indicates that cation is the active principle in the crude *p*-nitrodiazoaminobenzene. Apparently, the high acid content of the preparation medium induces the formation of a large amount of cation by Earl's reaction.^{5,6,7}

PREPARATION OF CRUDE *p*-NITRODIAZOAMINO BENZENE RICH IN CADION—

This synthesis is based upon that of Nölting and Binder.⁸ Dissolve 2.8 g of *p*-nitroaniline in 60 ml of diluted hydrochloric acid (1 + 1) by heating the mixture to 70° to 80° C. Cool the

solution to room temperature, add 1.9 g of aniline and cool to 0° C. Some *p*-nitroaniline hydrochloride is usually precipitated on cooling, but it dissolves when nitrite solution is added. Stir the cold mixture while slowly adding a solution of 1.4 g of sodium nitrite in 25 ml of ice-water; keep the temperature of the solution below 6° C. Place the mixture in a bath of cold water for approximately 5 minutes. Then add an ice-cold solution of 32 g of anhydrous sodium acetate in 100 ml of water; this causes crude *p*-nitrodiazoaminobenzene that is rich in cation to precipitate. Filter the cold slurry under suction. Wash the residue with approximately 500 ml of ice-water, and dry it in an oven at 35° to 40° C.

PREPARATION OF CADION SOLUTION—

Dissolve 40 mg of the crude *p*-nitrodiazoaminobenzene that is rich in cation in 100 ml of 95.6 per cent. w/w alcohol. This solution remains stable for over six months.

SELECTION OF SPOT-TEST PAPER—

Filter-papers differ greatly in their suitability for use in this cadmium test. In general, semi-crepe qualitative papers are superior to other filter-papers; highly retentive quantitative papers are usually unsatisfactory.

PROCEDURE FOR DETECTION OF CADMIUM—

Place a drop of the test solution, which is either neutral or faintly acidic with acetic acid and which is free of interfering substances,^{1,2} on a strip of filter-paper. Add a drop of cation solution to the spot, and then a drop of 2 *M* potassium hydroxide solution. If cadmium is present, the spot becomes pink-orange when the alkali is added. In the absence of cadmium, the added alkali causes the spot to turn red-purple.

REFERENCES

1. Dwyer, F. P., *J. Aust. Chem. Inst.*, 1937, **4**, 26.
2. Van Nieuwenburg, C. J., in Wenger, P. E., and Duckert, R., "Reagents for Qualitative Inorganic Analysis," Elsevier Publishing Co. Inc., New York, London, Amsterdam and Brussels, 1948, p. 30.
3. Dwyer, F. P., *J. Soc. Chem. Ind.*, 1937, **56**, 70r.
4. —, *J. Aust. Chem. Inst.*, 1938, **5**, 67.
5. Earl, J. C., *Proc. Royal Soc. N.S.W.*, 1929, **63**, 89.
6. —, *Ibid.*, 1930, **64**, 96.
7. —, *Ber.*, 1930, **63**, 1666.
8. Nölting, E., and Binder, F., *Ibid.*, 1887, **20**, 3004.

CHEMISTRY DEPARTMENT
MEMPHIS STATE COLLEGE
MEMPHIS 11, TENNESSEE, U.S.A.

E. FISHER
B. T. ESTES
J. E. ROSE
May 18th, 1953

ADSORPTION OF THIAMINE ON GLASSWARE

BIRD¹ has reported that, under certain conditions, there can be considerable adsorption of quinine sulphate from solutions of the sulphate in 0.1 *N* sulphuric acid solution on the glass walls of the containing vessel. This is very serious when the quinine sulphate solution is used as a reference standard in the determination of thiamine, but the phenomenon can be prevented if the glassware is first treated for 1 minute with boiling 30 per cent. sodium hydroxide solution.

When a non-linear graph was obtained for a series of standard thiamine solutions, it seemed probable that, in spite of Bird's claim that thiochrome is not adsorbed, thiamine might be, under circumstances similar to the quinine sulphate adsorption. This has been found to be true.

EXPERIMENTAL

Thirty millilitres of thiamine solution containing 0.1 μ g of thiamine hydrochloride per ml were swirled for 20 seconds in each of a number of glass vessels in succession. From the last vessel in the series, three 5-ml aliquots were removed by means of a pipette for the thiamine assay by the thiochrome procedure; 0.1 per cent. potassium ferricyanide solution was used as the oxidising agent and *n*-butyl alcohol as the extractant. The butyl alcohol phase was clarified with ethyl alcohol before the fluorescence was read on a Coleman, Model 12A, photofluorimeter.

The loss of thiamine was expressed as a percentage of the thiamine present in the untreated stock solution.

In Table I results are set out for a number of glass vessels. All, with the exception of the 100-ml calibrated flasks (series 8), were from normal laboratory stocks and had been in general use. The 50-ml Erlenmeyer flasks had served many times to hold the solutions of thiochrome in butyl alcohol immediately before the fluorescence was measured in the final stage of the thiamine assay. The 100-ml calibrated flasks had never been used before. All glassware was used only once in these and in most of the other experiments carried out.

TABLE I
ADSORPTION OF THIAMINE BY GLASSWARE

Series No.	Kind of glassware	No. in Series	Loss of thiamine, %
1	50-ml Erlenmeyer flasks	7	22
2	50-ml "	5	34
3	50-ml "	5	31
4	50-ml "	6	42
5	250-ml calibrated cylinders	5	21
6	500-ml calibrated flasks	5	25
7	25-ml stoppered cylinders	6	44
8	100-ml calibrated flasks	3	34
9	100-ml Erlenmeyer flasks	7	27

That this loss was due largely to adsorption of thiamine on the glass was shown by the recovery of 75 per cent. of it by washing the glassware with alkaline ferricyanide solution.

Thiochrome was not adsorbed either from aqueous solutions or from the mixture of butyl and ethyl alcohols used in making the final reading. On the other hand, fluorescence, shown by treatment with nitric acid and by irradiation in ultra-violet light not to be thiochrome, was picked up from the glass by the mixture of butyl and ethyl alcohols.

Of special interest in these laboratories are assays on yeast extract, in which solutions containing 0.10 to 0.15 g of yeast extract per 100 ml are acidified to pH 1 with concentrated hydrochloric acid and washed by thorough shaking with butyl alcohol to remove interfering fluorescence before the thiamine is assayed. Another series of experiments showed that under these conditions of high acidity thiamine is not adsorbed on glass.

Bird¹ claims that when glassware showing quinine sulphate adsorption is boiled for 1 minute in 30 per cent. sodium hydroxide solution, it can be then used without fear of further adsorption. Solutions rinsed successively through five alkali-treated flasks showed, within an experimental error of ± 5 per cent., no loss of thiamine. Further experimental work showed that Bird's procedure prevented adsorption of thiamine, and, in addition, prevented fluorescent material from being removed from the glass by the mixed alcohol solutions.

It was found that the preventive action of the alkali treatment is not permanent, adsorption causing losses of thiamine greater than experimental error after two or three uses. Nevertheless, it must be remembered that the adsorption is exaggerated by swilling the test solutions through six flasks before assaying, and, in addition, that it is almost completely avoided in untreated flasks if strongly acidified solutions are used.

The elution of the fluorescent material from glass could lead to higher results, and the errors involved could be well outside experimental error. This is more serious than the loss of thiamine by adsorption, for although the latter can be prevented in strongly acidic solutions, or recovered by oxidation to thiochrome, pick-up of foreign fluorescence from the glass can only be prevented by previous treatment of the glassware. In the authors' laboratory, acidified solutions are always used, and the glassware is treated with alkali every 3 months.

This work has mainly been on glassware from one maker, *viz.*, Crown Crystal Glass Pty. Ltd., Sydney. We have yet to prove whether the same phenomena would be observed with glassware from other manufacturers, although a few flasks made elsewhere have behaved similarly.

REFERENCE

1. Bird, L. H., *N.Z. J. Sci. Tech.*, 1949, **30**, 344.

KRAFT FOODS LIMITED
MELBOURNE, AUSTRALIA

K. T. H. FARRER
W. C. J. HOLLENBERG
May 6th, 1953

THE DETERMINATION OF ACETYLENE IN AIR

IMPROVEMENTS have been made to the method of Purser.¹ A standard solution of acetylene is used as a reference sample for each batch of reagents. Other analytical details have been amended as a result of a long series of experiments.

CALIBRATION—

Slight differences in the technique of preparing the cuprous chloride reagent can produce slightly different shades of colour when the reagent is added to the acetylene solution and this may cause the calibration to vary for each batch of reagent. Various ways of standardising the technique were tried without success and it was found necessary to prepare a reference solution of acetylene for each batch of reagent, as follows.

Fill a small calibrated gas-sampling tube of approximately 10 ml capacity with acetylene. Connect this tube to a bead bubbler, as used in the procedure, containing 40 ml of acetone and cooled in solid carbon dioxide. Absorb the acetylene by drawing air through the tube into the bubbler for a few minutes at a rate of 50 ml per minute. Analyse aliquots from this bubbler by the procedure described. Satisfactory readings are given by 0.5, 1.0, 1.5 and 2.0-ml aliquots of this bubbler solution in a 1-cm cell. A calibration factor can be calculated if the temperature and pressure at the time of filling the sampling tube are known.

PROCEDURE—

Store all glass vessels in a bath of ice-water before use. Keep the pipettes cool by storing them in a glass cylinder cooled in an ice-bath.

Empty the contents of the bubbler into a suitable vessel, such as a 25-ml cylinder, kept in the ice-water bath and quickly add exactly 10 ml of this into a cooled 50-ml cylinder already charged with 10 ml of acetone. Add 20 ml of cooled water, then 2 ml of reagent and mix the whole by means of a glass-rod plunger. Place this cylinder in a water-bath at 20° C for 5 minutes and measure the resulting colour in a Spekker absorptiometer using the appropriate cell (4 or 1-cm) to suit the depth of colour.

If the colour appears to be too deep for the 1-cm cell, quickly add a smaller aliquot by means of a pipette and repeat the determination in another cooled 50-ml cylinder filled with sufficient acetone to bring the volume to 20 ml after the aliquot has been added. When small aliquots are used, allow the end of the pipette to dip beneath the layer of acetone in the 50-ml cylinder.

Arrange to have the reagent added to the correct aliquot of acetylene solution no later than 2 minutes after the removal of the bubbler from the solid carbon dioxide.

The author thanks the Chief Scientist, Ministry of Supply, for permission to publish this note.

REFERENCE

1. Purser, B. J., *Analyst*, 1949, **74**, 237.

MINISTRY OF SUPPLY

CHEMICAL DEFENCE EXPERIMENTAL ESTABLISHMENT
PORTON, WILTS.

B. J. PURSER
May 18th, 1953

A QUALITATIVE TEST FOR MONOSACCHARIDES

When glucose is heated for a few minutes at 100° C in diluted sulphuric acid (3 + 1), a pink colour develops.¹ The colour has been used for the estimation of glucose in blood² and of glycogen in tissue.³ It was recently shown⁴ that the colour was due to condensation of 5-hydroxymethyl furfural, a dehydration product of glucose, with the groups of carbon atoms 2 and 3 in the sugar molecule. A more intense pink colour was produced when pure 5-hydroxymethyl furfural was added to the glucose before heating. Differences in the resulting colours produced by other sugars with added 5-hydroxymethyl furfural have made possible a simple procedure for their identification.

METHOD

REAGENTS—

Sulphuric acid—AnalaR grade, sp.gr. 1.840.

5-Hydroxymethyl furfural—Prepare by the method of Haworth and Jones⁵ and twice distil at a pressure of 0.1 mm of mercury. Make up the reagent to contain 1 mg per ml in distilled water.

PROCEDURE—

After showing by the conventional colour tests that pentoses, polysaccharides and ketohexoses are absent, distinguish the three aldohexoses as follows.

Place 1 ml of 5-hydroxymethyl furfural solution in a 6-inch \times $\frac{3}{4}$ -inch test tube by means of a pipette and add 1 ml of the sugar solution. The concentration of the sugar should be within the range 0.2 to 4.0 mg per ml. Place the tube in a basin of running cold water or, better, in ice-water. Add about 2 ml of sulphuric acid from a burette with constant swirling, and continue to swirl the tube until it has cooled. Add a further 4 ml of acid, swirling the tube in the cold bath all the time.

Examine the solution immediately the operation is complete. If it is coloured pink, the sugar is mannose. If it is colourless, immerse the tube for about 3 minutes in a bath of boiling water and then cool. If the solution is coloured pink, the sugar is glucose, if brown, it is galactose.

OTHER SUGARS—

Fructose and sorbose exhibit a pale yellow colour immediately the sulphuric acid is added. On heating as above, fructose becomes yellow-pink and sorbose becomes brown.

Both the pentoses, ribose and lyxose give pink colours in the cold, which are visible at concentrations above 0.3 and 0.2 mg per ml, respectively, and which deepen on standing. Xylose shows no colour immediately after the acid is added, apart from a slight darkening at concentrations above 5 mg per ml; after 30 minutes at room temperature, a slight pink colour develops. Arabinose shows no colour at all, even after heating.

Oligosaccharides and polysaccharides behave like mixtures of the individual sugars, so the reaction is unsatisfactory for their characterisation.

Sorbitol and mannitol produce a slight pinkish colour and galacturonic acid becomes deep pink on heating; gluconic acid and glucosamine show no colour at all, apart from the slight colour of the 5-hydroxymethyl furfural. Rhamnose becomes greenish-brown on heating.

Proteins interfere with the reaction, but if trichloroacetic acid is added, the protein-free filtrate reacts satisfactorily.

CONCLUSIONS—

The reaction is useful when only small amounts of hexose are available, *e.g.*, in eluates from chromatograms. Much less sugar is required than in the osazone test, and mannose can be distinguished from glucose. The colours produced by pentoses can often be used as confirmatory tests, although alone they are insufficiently specific to identify the sugars with certainty.

The work described in this paper was carried out as part of the programme of the Food Investigation Organisation of the Department of Scientific and Industrial Research.

REFERENCES

1. Mendel, B., and Bauch, M., *Klin. Wschr.*, 1926, **5**, 1329.
2. Mendel, B., and Hoogland, P. L., *Lancet*, 1950, **ii**, 16.
3. Van der Kleij, B. J., *Biochim. Biophys. Acta*, 1951, **7**, 481.
4. Love, R. M., *Biochem. J.*, 1953, **55**, 126.
5. Haworth, W. N., and Jones, W. G. M., *J. Chem. Soc.*, 1944, 667.

DEPARTMENT OF SCIENTIFIC AND
INDUSTRIAL RESEARCH (FOOD INVESTIGATION)
TERRY RESEARCH STATION
ABERDEEN

R. M. LOVE
June 15th, 1953

THE DETERMINATION OF ZINC BY TITRATION WITH DI-SODIUM ETHYLENEDIAMINETETRA-ACETATE

A RECENT paper¹ describes the amperometric titration of small amounts of zinc with di-sodium ethylenediaminetetra-acetate after appropriate removal or masking of interfering cations.

The object of the present note is to draw attention to the fact that the same micro-scale titration can be carried out with at least the same accuracy, and much more simply and expeditiously, to a visual end-point by the method of Biedermann and Schwarzenbach,² who used the dyestuff Colour Index No. 203 (Society of Dyers and Colourists), obtainable commercially as Solochrome Black W DFA (I.C.I. Ltd.), as indicator. Biedermann and Schwarzenbach showed that the end-point with zinc is particularly sharp, being better than with magnesium and calcium.

Experiments in this laboratory have indicated, for example, that 0.2 mg of zinc ions in a volume of 10 to 15 ml can be titrated with 0.01 *M* di-sodium ethylenediaminetetra-acetate in presence of an ammonium hydroxide and ammonium chloride buffer solution, by means of a 0.5-ml micrometer syringe burette, to an accuracy of within $\pm 2 \mu\text{g}$. As Pickles and Washbrook claim, the presence of calcium can be masked completely by the addition of sodium fluoride.

REFERENCES

1. Pickles, D., and Washbrook, C. C., *Analyst*, 1953, **78**, 304.
2. Biedermann, W., and Schwarzenbach, G., *Chimia*, 1948, **2**, 56.

DIVISION ANALYTICAL DEPARTMENT
IMPERIAL CHEMICAL INDUSTRIES LIMITED
DYESTUFFS DIVISION
HEXAGON HOUSE, BLACKLEY
MANCHESTER, 9

N. STRAFFORD
June 8th, 1953

Apparatus

A MODIFIED ALL-GLASS APPARATUS FOR THE DETERMINATION OF NITROGEN BY THE MICRO-KJELDAHL METHOD

KJELDAHL's¹ original apparatus for the determination of total nitrogen in body fluids, tissues and excretions, has undergone many changes since its inception in 1883. These modifications have been largely concerned with devising an efficient method for the distillation of the ammonia. Of the various distillation methods, Pregl's steam distillation technique is undoubtedly the safest and smoothest, and the most reliable, especially when considerable amounts of concentrated sulphuric acid have to be used for the digestion process.

In 1942, Markham² introduced an all-glass steam distillation apparatus for the distillation of volatile fatty acids. It has also proved successful in the determination of nitrogen by the micro-Kjeldahl method. The apparatus, although different in some structural details from Pregl's, is basically similar in its method of distillation and of generating the steam. As the apparatus is essentially for steam distillation, the digestion process is carried out in a separate Kjeldahl flask or tube. The digest is then transferred and washed into the distillation apparatus, as completely as possible, with distilled water through a cup-shaped container that is also used for the sodium hydroxide. When similar determinations are made with the new apparatus, this procedure is unnecessary. As a result, no material is lost through transference and the amount of liquid in the Kjeldahl flask is reduced to a minimum (approximately 25 ml) before distillation of the ammonia.

EXPERIMENTAL—

Preliminary chemical tests and trials indicated that superheated steam, which is "on tap" in most clinical laboratories, might be used for this purpose. More extensive experiments proved that there was no difference between the superheated steam "blank," and the blank value obtained by the more orthodox procedure. Although an iron steam-pipe corrodes to a slight extent, it was found that any iron contaminating the steam does not interfere with the Kjeldahl determinations.

There are many advantages in using steam under pressure, provided it is clean and free from chemical impurities. It accelerates the rate of distillation, aids in the decomposition of amino or ammino-complexes that may be formed, requires no preparation (the conventional method does) and occupies little space. Moreover, it is much safer, easier to control and manipulate, requires little or no attention, and is always available for use. It is possible, moreover, to make any number of determinations simultaneously, the only limiting factor being the ability of the individual to regulate the rate of distillation, which is controlled by a steam-release valve connected to the main steam-pipe.

The condensed water in the superheated steam is trapped in a wide-mouthed bolt-head flask that is also used as a steam reservoir. Thus only dry steam, *i.e.*, steam unmixed with fine drops of water, is used for the distillation process. A simple siphoning system fitted to the bolt-head flask keeps the flask free from water. The incoming steam produces the necessary force to automatically siphon off any water that otherwise collects in the flask. The steam from the steam reservoir can be cut off from the distillation apparatus by a Pyrex-glass tap as necessary. This

device is especially useful when distillation is finished, as it eliminates any interference with the main steam supply. It also helps to maintain a constant head of pressure, and so minimises slight fluctuations in steam pressure.

APPARATUS—

The apparatus is made of hard glass and is constructed in four sections (A, B, C and D in Fig. 1). Each section is detachable and can be easily replaced or repaired.

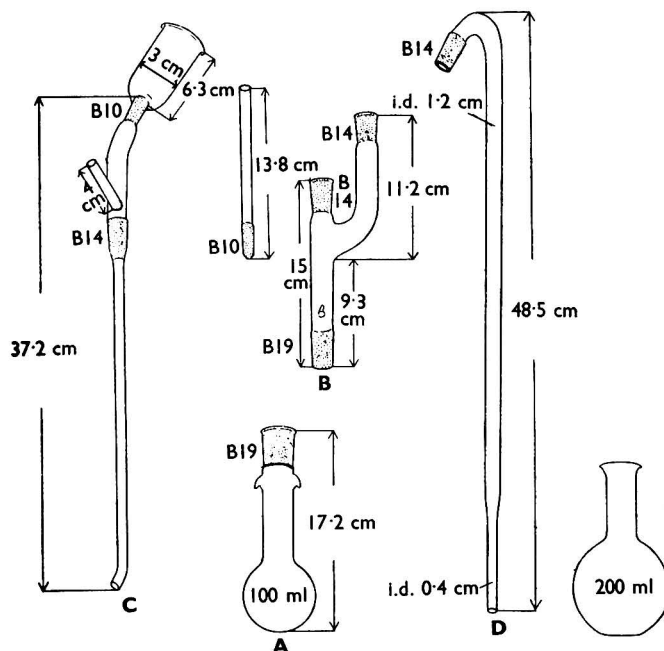


Fig. 1. View of individual section of apparatus. A, 100-ml Kjeldahl flask; B, "Claisen-like" still head; C, reservoir and steam inlet tube; D, air condenser and flask

For the distillation a 100-ml Kjeldahl flask, A, the neck of which carries a B19 standard joint, is attached to a B19 cone on the "Claisen-like" still head, the main arm of which is used to support the whole apparatus. This arm is clamped at an angle, as shown in Fig. 2, to prevent the strongly alkaline solution in the flask from being splashed into the entrance of the air condenser, and so being blown over mechanically into the condenser. This design allows the flask to be taken off or attached quite freely, without interference to any other part of the apparatus. The Kjeldahl flask is firmly held in position by a spring clip, which attaches it by means of hooks to the still-head.

The "Claisen-like" still-head, B, besides being used as an attachment for the Kjeldahl flask, the air condenser by a B14 standard joint, and the reservoir and steam inlet tube, also serves as an efficient splash-head. Any spray is trapped in the side-arm of the still-head and returned as a steady stream of liquid to the Kjeldahl flask. It also offers the minimum obstruction to the flow of the steam and does not trap pools of distillate.

Into the B14 socket of the main arm of the "Claisen-like" still-head a long glass tube, C, reaching to the bottom of the Kjeldahl flask, is fitted by a B14 cone. Above the B14 cone this glass tube branches off to a steam delivery tube and a cup-shaped reservoir for the 40 per cent. sodium hydroxide solution. The cup-shaped reservoir is plugged by a closed B10 standard cone.

A long air condenser, D, made of relatively wide bore glass tubing, has its lower end drawn out to the thickness of an ordinary glass-tubing, internal diameter 4 mm, and to approximately 9 cm long. It dips under the standard acid and is constructed to prevent any disastrous suck-back.

Recovery experiments proved that it is unnecessary to use a water condenser, as the air condenser, provided it is long enough to dip below the surface of the acid, is just as efficient. The

air condenser is in many ways preferable, since it considerably simplifies the apparatus and prevents the steam from condensing on the sides of the condenser. For the collection of the ammonia, a 200-ml flask is used, which stands on a wooden block 6 inches high and 4 inches wide. A half-inch groove in this wooden block accommodates the flask and holds it firmly in position. This facilitates the easy withdrawal of the flask, which is necessary for subsequent manipulations.

In twenty-four different determinations on solutions of specially purified ammonium sulphate containing 4.71 mg per 5 ml (1 mg of nitrogen per 5 ml) a recovery of 99 to 100 per cent. was obtained in 2 minutes. For use with the micro-method, 10 ml of N/70 sulphuric acid was back-

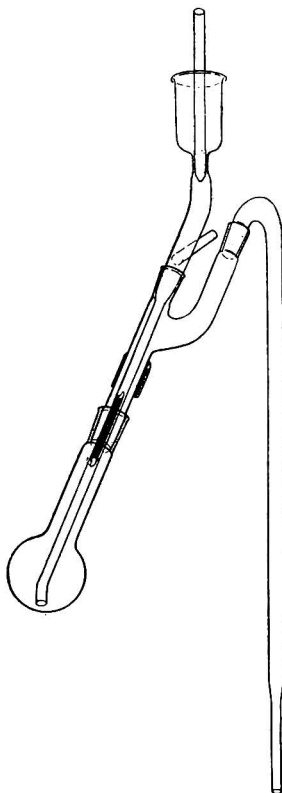


Fig. 2. View of assembly of apparatus

titrated with N/70 sodium hydroxide solution, a mixed indicator being used (3 parts 0.1 per cent. of bromocresol green in alcohol, and 1 part 0.2 per cent. of methyl red in alcohol). This covers the range of 0 to 2 mg of nitrogen.

ADVANTAGES AND OTHER USES—

No rubber connections, which are always a potential source of trouble and error, are used in its assembly, and it can be mounted on a single retort stand requiring only one clamp to keep it in position at an angle of approximately 20° to the air condenser.

The digestion and distillation processes are done in one Kjeldahl flask, which is always much safer than the transfer of the digest to another distillation flask, especially when small quantities of nitrogen are to be determined. This also saves time and effort.

The apparatus can also be used for the purification of many water-insoluble compounds, both solid and liquid, provided that the compound is volatile and the impurities non-volatile. Further, it can be used for the separation and determination of volatile poisons and for allied procedures. It could also be used, without making any structural alterations to the apparatus, for the determination of nitrogen by the macro-Kjeldahl method by substituting a much larger flask (not necessarily a Kjeldahl flask) carrying a B19 standard joint, in the digestion and distillation procedures.

I wish to express my thanks to Mr. D. MacDonald whose glass-blowing skill and co-operation made possible the new apparatus.

The apparatus can now be obtained from Messrs. Quickfit & Quartz Ltd.

REFERENCES

1. Kjeldahl, J., *Z. anal. Chem.*, 1883, **22**, 366.
2. Markham, R., *Biochem. J.*, 1942, **36**, 790.

DEPARTMENT OF CLINICAL CHEMISTRY
UNIVERSITY OF EDINBURGH
ROYAL INFIRMARY, EDINBURGH

F. J. SCANDRETT
June 24th, 1953

Ministry of Food

STATUTORY INSTRUMENTS*

1953—No. 1609. The Public Health (Condensed Milk) (Amendment) Regulations, 1953. Price 2d.

*These Regulations, which came into operation on November 11th, 1953, amend the Public Health (Condensed Milk) Regulations, 1923 (S.R. & O., 1923, No. 509; Analyst, 1923, **48**, 272), as amended, by permitting for a limited period the sale of full cream unsweetened condensed milk, imported by the Minister of Food, containing not less than 7·8 per cent. of milk-fat and not less than 25·5 per cent. of all milk-solids including fat.*

1953—No. 1610. The Public Health (Preservatives etc. in Food) (Amendment) Regulations, 1953. Price 2d.

*These Regulations, which came into operation on November 11th, 1953, amend the Public Health (Preservatives etc. in Food) Regulations, 1925 (S.R. & O., 1925, No. 775; Analyst, 1925, **50**, 455), as amended. They—*

- (i) *Provide for the sale and importation of certain specified dehydrated vegetables that contain sulphur dioxide not exceeding prescribed limits, as follows—*

Food		Preservative		Parts per million
Dehydrated Vegetables:				
(a)	Potatoes	Sulphur dioxide	550	
(b)	Runner beans, turnips, spinach, swedes, peas, parsnips, carrots, mixed vegetables.	Sulphur dioxide	2,000	
(c)	Cabbage	Sulphur dioxide	3,000	

- (ii) *Increase the maximum permitted sulphur dioxide content of jam from 40 to 100 parts per million;*
- (iii) *Permit the presence of diphenyl in citrus fruit imported in wrappers treated with diphenyl; and*
- (iv) *Permit, for a limited period, the addition of borax to margarine, the amount of boron preservative (expressed as boric acid) not to exceed 2,500 parts per million.*

1953—No. 1618. The Public Health (Condensed Milk) (Scotland) Amendment Regulations, 1953. Price 2d.

These Regulations, which came into operation on November 16th, 1953, amend the Public Health (Condensed Milk) Regulations (Scotland), 1931, as amended, in the same manner as S.I., 1953, No. 1609 (see above) amends the corresponding Regulation for England.

CIRCULAR MF 20/53

This circular (price 2d.), dated November 11th, 1953, draws attention to S.I., 1953, Nos. 1609 and 1610 (see above). It states that these amending Regulations re-enact substantially the provisions of Regulation 60 CAA of the Defence (General) Regulations, which will be allowed to lapse on December 10th, 1953, unless revoked at an earlier date. The provision whereby meat may contain sulphur dioxide is no longer necessary and is not re-enacted.

* Obtainable from H.M. Stationery Office. Italics indicate changed wording.

British Standards Institution

DRAFT SPECIFICATIONS

A FEW copies of the following draft specifications, issued for comment only, are available to members of the Society, and can be obtained from the Secretary, Society of Public Analysts and Other Analytical Chemists, 7-8, Idol Lane, London, E.C.3.

Draft Specification prepared by Sub-Committee LBC/1/1—Dean and Stark and Ubbelohde Apparatus.

CR(LBC)6796—Draft B.S. for Ubbelohde Apparatus for Flow and Drop Points. (Revision of B.S. 894.)

Draft Specification prepared by Technical Committee LBC/12—Dairy Apparatus.

CR(LBC)6270—Draft B.S. for Centrifuge Tubes and Sedimentation Vessels for the Determination of Visible Dirt in Milk. (Revision of B.S. 736.)

Book Reviews

INORGANIC THERMOGRAVIMETRIC ANALYSIS. By CLÉMENT DUVAL. Pp. xvi + 531. Amsterdam, London and New York: Elsevier Publishing Co. Ltd. London: Cleaver-Hume Press Ltd. 1953. Price 60s.

The many papers on this subject published by Professor Duval and his collaborators over the last five years in *Analytica Chimica Acta* have now been collected in this single volume. It is noted in the preface that "this preliminary work" has so stimulated the interest of analysts that nearly two thousand requests for reprints of the papers have been received. One must indeed admire the industry that has gone to the recording of such a vast amount of potentially useful data comprising as it does "the pyrolysis curves of 967 precipitates proposed up to July, 1951, for inorganic gravimetric analysis."

For any who may be unfamiliar with this work it should be explained that a "pyrolysis curve" records the constancy, loss or gain in weight of a substance during a heating cycle in a furnace, the crucible containing the substance under observation being permanently attached to a continuously recording sensitive balance. For these studies, Professor Duval has used the Chevenard thermobalance, of which a detailed description is given in Chapter 2 of the book; a short historical review of thermobalances is given in the first chapter.

Chapter 3 deals with "Applications of the Thermobalance," Chapter 4 with "Automatic Gravimetric Analysis" and, save for a one-page "Postscript," the remainder of the book is given over to "The Thermogravimetric Investigation of Analytical Precipitates." In this, the main section, are recorded data on compounds of 62 elements, from lithium to uranium. The pattern is similar throughout—a number (sometimes as many as 25) of precipitants for the element in question are listed; the conditions of each precipitation, or the appropriate references, are given; the pyrolysis curves are reproduced; a tabular summary is given showing the precipitating reagent, form weighed and temperature limits; and finally, there is a list of references.

This specific information is potentially of very great value, and the techniques by which it has been obtained are clearly elegant, but it is doubtful if the author is justified in presenting these pyrolysis curves as absolute data. The impression is given that the weight of a precipitate is solely a function of temperature; the possible influence of rate of increase of temperature (an arbitrary figure governed by the gearing of the rheostat) and of the many other variables appear to be ignored. In the categorical acceptance or rejection of a particular precipitant, the thermogravimetric data are interpreted on the assumption that the thermobalance can and will be used. For normal methods of ignition and weighing, other factors, not disclosed by the thermogravimetric studies, must be taken into account.

That Professor Duval has not done this derives no doubt from his expressed opinions of analytical practice. He contends that in most analytical laboratories precipitates are heated for too long and at too high a temperature; this is probably true, but does he seriously believe that competent inorganic analysts have not improved on the techniques of Berzelius and Rose and on those of Winkler and Treadwell, singled out for criticism on p. 41? It is unfortunate that the author has sought, unnecessarily, to justify his work by arguments that are invalid because they are out of date.

On the interpretation of the pyrolysis curves, the reviewer would have found some of the deductions more convincing had they been backed by complementary information from X-ray diffraction studies of the pyrolysis products. This view is expressed in the knowledge that some of the deductions are at complete variance with established X-ray data. For example, in the

table on p. 116, minimum ignition temperatures are given for the precipitates obtained by treating solutions of aluminium with 24 different precipitants. Only two of these temperatures are higher than 950° C, and one is as low as 280° C. The most recent studies show that when hydrated alumina is heated to a temperature of 400° C, the alumina is wholly in the form of the alpha or beta monohydrate, while the gamma anhydrous form, which is strongly hygroscopic, is likely to persist up to at least 950° C. The pyrolysis curve for hydrargillite (a trihydrate) on p. 117 is in such disagreement with published verifiable crystallographic data as to raise some doubts as to the exact meaning of these curves. Again, although the curve for the zinc - oxine complex is in reasonable agreement with the reviewer's published X-ray studies, that for the magnesium complex is not. It may be that the explanation lies in the author's own comment that "the form of curve obtained with this substance depends very much on the method of precipitation and on the rate of heating."

In the tabular summaries at the end of each chapter, the methods suitable for the automatic determination of the element concerned are indicated by means of an asterisk placed before the precipitating reagent. It is this concept of "automatic gravimetric analysis" and the philosophy behind it that calls for most comment. Techniques for single substances and for binary mixtures are described in Chapter 4. In the first, the weight of the precipitate is obtained by subsequent linear measurement of the height of the "horizontal" (the measuring scale is graduated in half millimetres) and by comparison with a calibrated "horizontal," say from a 50-mg weight. It is frankly difficult to see in what way this becomes usefully more automatic than by a normal weighing on the latest type of rider-loaded air-damped balance.

A mixture of calcium and magnesium oxalates is used to illustrate the application to binary mixtures. Suffice it to say that by recording the levels at 500° C and 900° C and comparing the heights above the calibration line, "two first degree equations can be set up with the unknowns x and y as the weights of calcium and magnesium and with m and n as the known weights of the mixtures present at 500° C and 900° C"!

So much for the technique, but is it properly named? Surely, it is only the ignition and weighing that can be considered automatic. In gravimetric analysis, the major and most difficult part comprises the separations that must precede the final determination. It is not sufficiently realised, even now, that in applied analysis those operations that make the determination possible are often more important than those that attend the determination itself.

As to the philosophy: as before, Professor Duval uses arguments to justify the techniques that suggest that he is out of touch both with the methods and the problems of the industrial inorganic analyst.

It would seem that we have only two choices open to us: to pay for a thermobalance plus the salary of "a non-specialised laboratory technician who is capable of changing the substance to be determined every 15 minutes" or "to pay the emoluments of a graduate chemist who with inexpensive equipment carries out evaporations, separations, precipitations, filtrations through paper, ignitions of paper, who heats crucibles at ill-defined temperatures for indeterminate periods of time which are generally too long, who weighs, re-heats, re-weighs and risks making mistakes in the weight, and who, having done all this, has no evidence of any kind as to whether in the event of the determination being incorrect the mistake has been voluntary or involuntary."

It seems odd that in the next (and shorter) sentence the author should feel able to declare that "the role of the modern graduate chemist is to create and not to perform throughout his whole career the same analytical operations more or less with distaste" and, it is pertinent to ask if the ability to carry out the simpler and repetitive operations of analytical chemistry really well is a handicap in later essays in creation or, to use a popular word, research.

Fortunately, these misapprehensions will not deter the discerning reader from looking for the real merit in the author's work. The extent and nature of the reviewer's comments and reservations are not intended to detract from the large amount of potentially valuable data, which should be of interest to all analysts. It is to be hoped that Professor Duval will extend his work to a more fundamental and detailed study of the promising compounds he has examined in this preliminary survey.

It remains to add that the translation by Mr. N. G. Bissett is well done, only the word "docimasy" seems out of context, and that the book is extremely well produced.

R. C. CHIRNSIDE

POLYSACCHARIDE CHEMISTRY. By R. L. WHISTLER and C. L. SMART. Pp. xvi + 493. New York: Academic Press Inc. 1953. Price \$10.80; 86s. 6d.

Until relatively recently a book under the present title would have dealt almost exclusively with cellulose, starch and pectins. Current investigations in carbohydrate chemistry have greatly extended our knowledge of polymolecular structures based on sugars and allied substances. We now know that polysaccharides constitute approximately three-quarters of the dry weight of the higher plants and sea weeds. They form the basis of the structural material of plants, the exoskeleton of insects and crustaceans and the protective sheaths and capsular material of bacteria; they contribute largely to fuel reserves stored as cell inclusions; and they protect surfaces against desiccation and they seal wounds.

Plant tissue is a matrix of giant polysaccharide molecules, sometimes parallel to each other as in rigid structural tissue and sometimes weaving and entangling together as in thickening material, the whole fabric being permeated and held together by many other substances, including lipids, proteins, salts and colouring matter. This close association, with many secondary valence links, makes the separation and purification of molecules of one type very difficult.

Glycogen used to be considered almost the sole animal polysaccharide, but hyaluronic acid is important as intercellular cement and chitin as a structural support for lower animals. Specific polysaccharides play a significant part in synovial and other body fluids, contribute to blood group specificity and immunological reactions, and have protective and lubricative functions on mucous surfaces.

The first two chapters of the present volume deal with occurrence, nomenclature and classification and with methods used in general characterisation and proof of structure. Then follow 33 chapters each dealing with a particular type of polysaccharide. Cellulose, starch and pectins each occupy about 10 per cent. of the space, an amount that is admitted to be insufficient to cover the whole chemistry of such important substances, but adequate for the particular purposes of this book. A complete literature survey is claimed for all the other polysaccharides, and the only omission that the reviewer has noticed is under lignin, where no indication is given of its nature or even if it is a polysaccharide.

The book is a valuable compendium on the occurrence, structure and properties of the lesser-known polysaccharides. Their analytical chemistry is not in a very advanced state, so that the few methods of analysis given are largely of academic interest. The information should stimulate analysts to devise methods for the determination of these important substances.

J. R. NICHOLLS

Publications Received

THE IDENTIFICATION OF TEXTILE FIBRES. QUALITATIVE AND QUANTITATIVE ANALYSIS OF FIBRE BLENDS. By BRUNO LUNIAK. Pp. xiv + 177. London: Sir Isaac Pitman & Sons Ltd. 1953. Price 45s.

STANDARD X-RAY DIFFRACTION POWDER PATTERNS. Volume I. National Bureau of Standards Circular 539. By HOWARD E. SWANSON and ELEANOR TATGE. Pp. ii + 95. Washington: U.S. Government Printing Office. 1953. Price 45 cents.

STANDARD X-RAY DIFFRACTION POWDER PATTERNS. Volume II. National Bureau of Standards Circular 539. By HOWARD E. SWANSON and RUTH K. FUYAT. Pp. ii + 65. Washington: U.S. Government Printing Office. 1953. Price 45 cents.

MISES AU POINT DE CHIMIE ANALYTIQUE PURE ET APPLIQUÉE ET D'ANALYSE BROMATOLOGIQUE. Publiées sous la direction de J.-A. GAUTIER. Pp. iv + 172. Paris: Masson et Cie. 1953. Price 1400 fr.

ION EXCHANGE RESINS. Pp. 36. Poole, Dorset: The British Drug Houses Ltd. 1953. Gratis.

THE CHEMICAL INDUSTRY: PAST AND PRESENT. By T. I. WILLIAMS, M.A., B.Sc., D.Phil., F.R.I.C. London: Penguin Books. 1953. Price 2s.

CHEMISTRY OF THE LANTHANONS. By R. C. VICKERY, D.Sc., Ph.D. Pp. viii + 296. London: Butterworths Scientific Publications. 1953. Price 35s.

SOAP MANUFACTURE. Volume I. By J. DAVIDSOHN, E. J. BETTER and A. DAVIDSOHN. Pp. xii + 525. London and New York: Interscience Publishers Inc. 1953. Price \$12.50; 95s.

ANALYTICAL CHEMIST required for Research Laboratory. Applicants should have had extensive industrial analytical experience and should be capable of devising new methods of approach to a wide variety of analytical problems. Applications to be sent to Chief Chemist, International Paints Ltd., Felling-on-Tyne, Co. Durham, giving full details of qualifications, experience and salary required.

EVANS MEDICAL SUPPLIES LTD. Vacancies occur in the Analytical Department of the Pharmaceutical Research and Development Unit, for Analysts with A.R.I.C., B.Sc., or suitable Pharmaceutical qualifications. Experience in analysis of Pharmaceuticals would be an advantage. Superannuation scheme and 5 day week in operation. Write giving age, qualifications, and experience to the Pharmaceutical Development Director, Evans Medical Supplies Ltd., Speke, Liverpool 19.

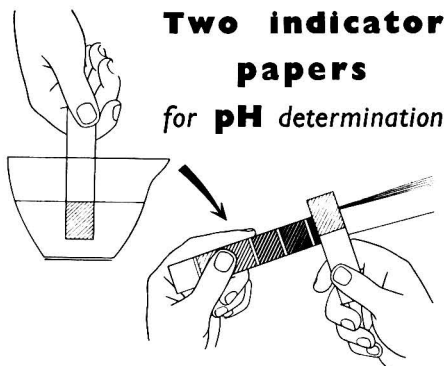
IMPERIAL CHEMICAL INDUSTRIES LIMITED has vacancies for Assistant Analysts (men) in the Pharmaceutical Control Laboratory. Applicants should preferably be experienced in the testing of synthetic products and formulated products and should possess at least Inter. B.Sc. or equivalent qualification; age up to 30. Applications in writing to Staff Department, Hexagon House, Blackley, Manchester, 9.

REQUIRED Volumes 51-76 of *The Analyst*. Write Box No. 3853 THE ANALYST, 47, Gresham Street, London, E.C.2

HEFFER'S OF CAMBRIDGE

publish from time to time catalogues and lists of books on various subjects, and announcements of individual new books of special importance. Let us add your name to our mailing list.

W. HEFFER & SONS, LIMITED
3 & 4 PETTY CURY, CAMBRIDGE



Two indicator papers for pH determination

UNIVERSAL One paper covering completely the range from 1 to 10 enabling pH values to be checked to within 0.5 pH.

COMPARATOR Four separate books for work requiring greater accuracy. With these papers the pH value of any solution can be ascertained to within 0.3 pH.

Descriptive leaflet sent free on request

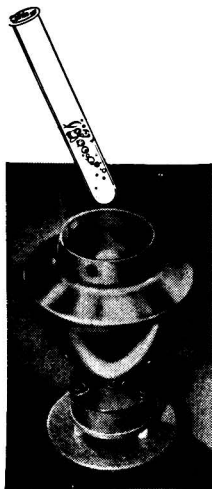


London, N.W.4

Est. 1743

clean, concentrated heat . . .
THE BUNRAY
electric bunsen burner

any container becomes a
PRECISION constant
temperature bath or
circulating system . . . if you
have a **CIRCOTHERM**

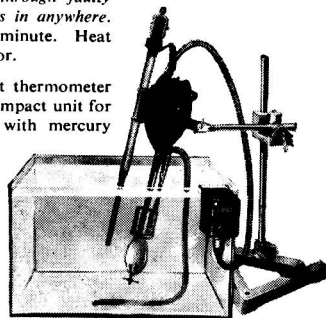


Left. This new infra-red reflex burner combines the advantages of the time-honoured Bunsen burner with the good features of electrical heating:

Cleanliness—No smells—Concentrated heat source—No heat deflection by draught—No danger of explosions through faulty connections—Can be used in any position—Plugs in anywhere. Temperatures of 800°C. reached in about 1 minute. Heat regulation effected by resistance or energy regulator.

Right. Heater, stirrer, pump, adjustable contact thermometer and variable speed motor are combined as a compact unit for clamping to any laboratory stand. Complete with mercury switch relay. Exceptionally uniform temperature through the bath; able to supply constant temperature liquid to outside apparatus.

SHANDON



SHANDON SCIENTIFIC COMPANY, 6a, CROMWELL PLACE, LONDON, S.W.7



Items from the range of
LABORATORY PREPARATIONS

- OXOID** Desoxycholate Citrate Agar. A modification of Liefson's medium for the isolation of intestinal pathogens.
- OXOID** Wilson & Blair (modified) Bismuth Sulphite Agar Medium. For the isolation and differentiation of intestinal bacilli.
- OXOID** Selenite Broth. An enrichment medium for the isolation of typhoid and salmonellæ from pathological specimens.
- OXOID** Test Tube Caps. An improvement on cotton wool for closing Test Tubes. Can be used repeatedly and ensure sterility for long periods. Plain or in eight colours to fit $\frac{1}{2}$ ", $\frac{5}{8}$ ", $\frac{3}{4}$ " and 1" rimless test tubes.



*Laboratory Preparations are obtainable from any Laboratory Supplier
or direct from*

**OXO Limited, Medical Dept,
Thames House, London, E.C.4**

Telephone: Central 9781

For your
vital
Laboratory
work...



this is vital equipment

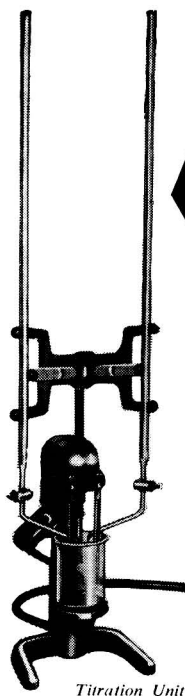
Special hard porcelain for chemical and scientific use has been produced by Royal Doulton for over 30 years.

It combines the following advantages :

- Excellent resistance to high temperatures and to thermal shock caused by temperature fluctuations.
- The glaze, which is perfectly married to the body of the ware, withstands the strain of heating up and cooling down.
- The use of first quality raw materials gives the finished product high resistance to chemical attack.
- The raw materials are carefully selected to ensure freedom from iron contamination.
- High mechanical strength is apparent even in the thin-walled articles normally used in laboratory practice.

Write for further details to
Doulton & Co. Limited,
Industrial Ceramics Division,
Dept. AJ, Doulton House,
Albert Embankment,
London, S.E.1.

Royal
DOULTON
LABORATORY PORCELAIN



Titration Unit

Potentiometric Titration Apparatus

with electron beam tuning indicator

THIS new Mullard electronic Potentiometric Titration Apparatus—designed for simplicity of operation—facilitates rapid and accurate acid/alkali and oxidation reduction titrations. A unique feature of the potentiometric apparatus is the use of an electron beam (magic eye) tuning indicator in place of the more delicate galvanometer. For routine analysis, the closing of the eye can be made to coincide with the end point of titration.

Potentiometric Unit

The titration assembly, which has been developed in conjunction with A. Gallenkamp and Co. Ltd., includes two 50 ml. graduated burettes, a two-speed stirrer motor, and five electrodes. A hot plate is available for hot titrations. The Potentiometric Titration Apparatus is one of a number of Mullard high grade electrochemical instruments. Full details of these instruments will be gladly supplied on request.

Mullard



SPECIALISED ELECTRONIC EQUIPMENT

Mullard Ltd., Equipment Division, Century House, Shaftesbury Ave., W.C.2

(M1423A)

COLORIMETERS

REFLECTOMETERS

ABSORPTIOMETERS

NEPHELOMETERS

THE



FLAME PHOTOMETER

Essential to any laboratory concerned with sodium, potassium, lithium or calcium determinations

An accurate, quick reading, simply operated, low priced photo-electric instrument that, although only recently introduced, has already proved itself invaluable.

This is a typical unsolicited appreciation:

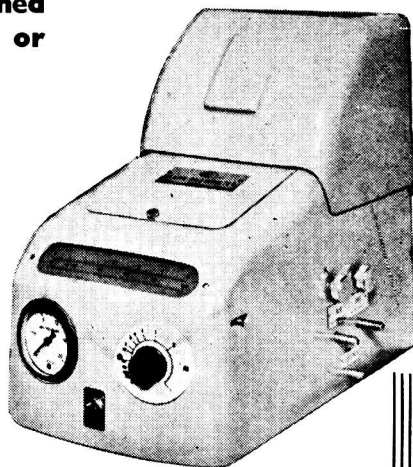
"The time taken in determination of these three alkalis in the material mentioned is not more than one hour. I have carried out eight such analyses in a working day comfortably, and if pressed could have done more. This time covers all phases of the test and means from receipt of sample to the issue of the final report. Remembering that eight samples by the old methods meant a month's work, this is quite astonishing."

Send for full particulars to:

EVANS ELECTROSELENIUM LTD

Sales Division 347 Harlow Essex

PHOTOMETERS • SELENIUM PHOTOCELLS • SPECTROPHOTOMETERS • DENSITOMETERS



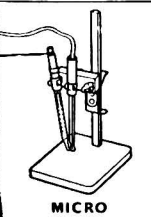
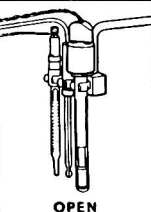
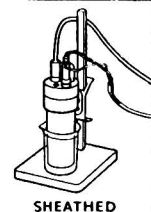


This, the fourth edition of our brochure, "M&B Laboratory Chemicals & Reagents", sets out the entire range of over 500 products together with the complete specifications of each. The specifications of 16 recent additions are given for the first time. We shall be pleased to send a copy on request.

M&B brand
**LABORATORY
 CHEMICALS
 AND REAGENTS**

MANUFACTURED BY
MAY & BAKER LTD
 DAGENHAM ENGLAND
 Telephone: ILFord 3060 Extension 40

**ELECTRODE
 ASSEMBLIES
 INCLUDED**

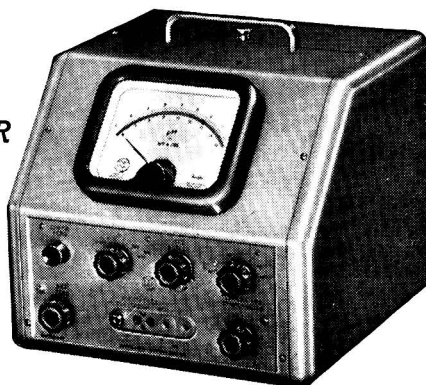


**PYE UNIVERSAL
 pH METER
 AND MILLIVOLTMETER**

This new version of the Pye pH Meter not only retains the many excellent features of the previous model such as low zero drift, stability under wide mains variations, ease of operation and automatic temperature compensation, but it now incorporates a manual temperature compensator, complete coverage 0-14 pH and a wide range of electrode assemblies.

In a wide variety of industries the Pye pH Meter is contributing towards higher productivity, the elimination of waste and improved product quality. An unskilled operator can test acidity or alkalinity of process solutions with rapidity and ease.

In laboratories covering a very wide range of research the Pye pH Meter is proving, by its convenience and consistently high accuracy, to be the outstanding instrument in its field.

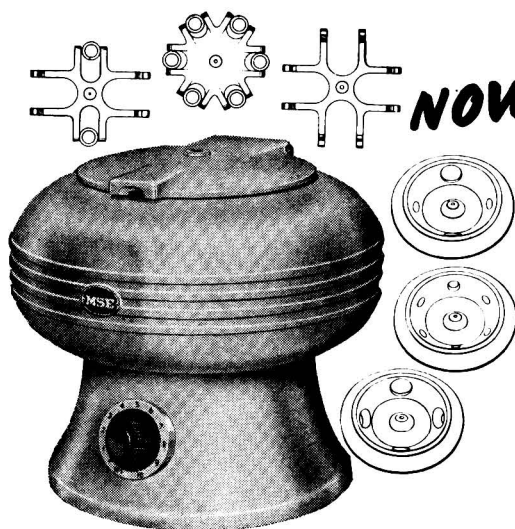


Cat. No. A. 11066

**PORTABLE
 MAINS OPERATED
 DIRECT READING 0-14pH
 RANGE OF ELECTRODE ASSEMBLIES
 DRIVES CONTROLLERS
 OR RECORDERS
 MANUAL OR AUTOMATIC
 TEMPERATURE COMPENSATION**

SCIENTIFIC  INSTRUMENTS

W. G. PYE & CO. LTD., GRANTA WORKS, CAMBRIDGE



NOW—an even better
"MINOR" Centrifuge

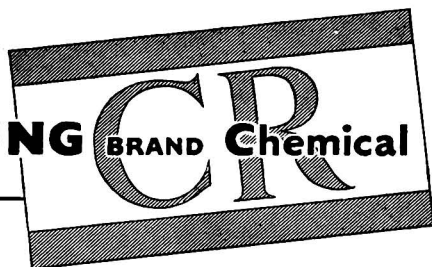
The famous MSE "Minor" Centrifuge not only has a "new look" but it now incorporates stepless speed control—an exclusive feature for this type of centrifuge. Six different "swing-out" or "angle" heads to choose from. Speeds up to 4,600 r.p.m. (approx. 3,000 × gravity). Universal AC/DC motor of great reliability.



FOR YOUR FILE: *We have just published a new edition of MSE publication 888, giving a brief review of the entire range of Centrifuges, Microtomes and Laboratory Blenders. Have you had your copy?*

MEASURING & SCIENTIFIC EQUIPMENT LTD., SPENSER STREET, LONDON, S.W.1

STERLING BRAND Chemical Reagents



"Sterling" Brand Chemical Reagents are manufactured under close laboratory supervision and are guaranteed to conform with the specification stated on the label.

These specifications have been compiled with the object of providing material suitable for all general analytical and research purposes.

"Sterling" Brand Chemical Reagents comply with the specifications of the British Pharmacopoeia, Appendix I, for analytical reagents, where such reagents are listed.

A list of prices and specifications is at your disposal from

Thomas Tyrer & Co. Ltd.

STRATFORD, LONDON, E.15

CALGITEX

ALGINATE BACTERIOLOGICAL WOOL

THE IDEAL SWABBING MATERIAL, SOLUBLE, STERILIZABLE

Calgitex Alginate Bacteriological Wool dissolves in solutions of certain Sodium salts to give solutions of normal pH., thus permitting high recovery of organisms. It is the ideal material for quantitative bacteriological work. Comparative tests¹ have shown that for staphylococci the recovery of known inocula was 50% using cotton wool and 90% using alginate wool; with *E. coli* the recoveries were 67% and 90%.

Particularly suitable for diagnostic work and for hygienic control in the food processing and distributing industries²

NOTE: Calgitex Alginate Bacteriological Wool is composed entirely of calcium salt of alginic acid and is not a cotton fibre impregnated with alginate.

REF: 1. Higgins M., *Monthly Bull. Min. of Health & Pub. Health Lab. Service. Feb. 1950 p. 49.*
2. Higgins M., & Hobbs B. *ibid p. 38.*

Calgitex Alginate Bacteriological Wool is available in tubes containing sufficient for approximately 50 swabs. Price 3s. 6d. per tube direct from the manufacturers.

MEDICAL ALGINATES LIMITED • WADSWORTH ROAD • PERIVALE • MIDDLESEX
Full technical information supplied on request

Cathode Ray POLAROGRAPH

For Accurate and Speedy Analysis

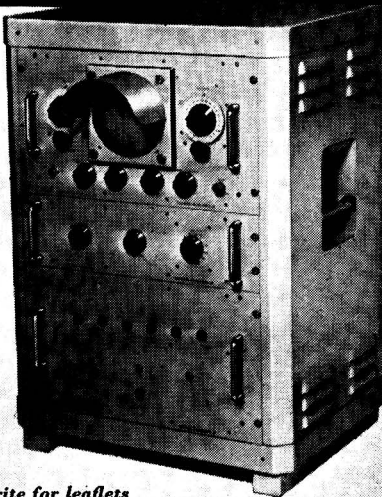
**METALLURGICAL INORGANIC
ORGANIC BIOCHEMICAL**

Since its invention by Heyrovsky in 1925 the Polarograph has established itself as an essential tool in many Analytical Laboratories all over the world.

The Cathode Ray Polarograph greatly extends the scope of the polarographic technique. Its increased sensitivity, resolution and speed reduce the amount of chemical separation and concentration required and make possible many determinations which cannot be carried out in any other way.

**QUALITATIVE QUANTITATIVE
ROUTINE SPECIAL RESEARCH**

Write for leaflets



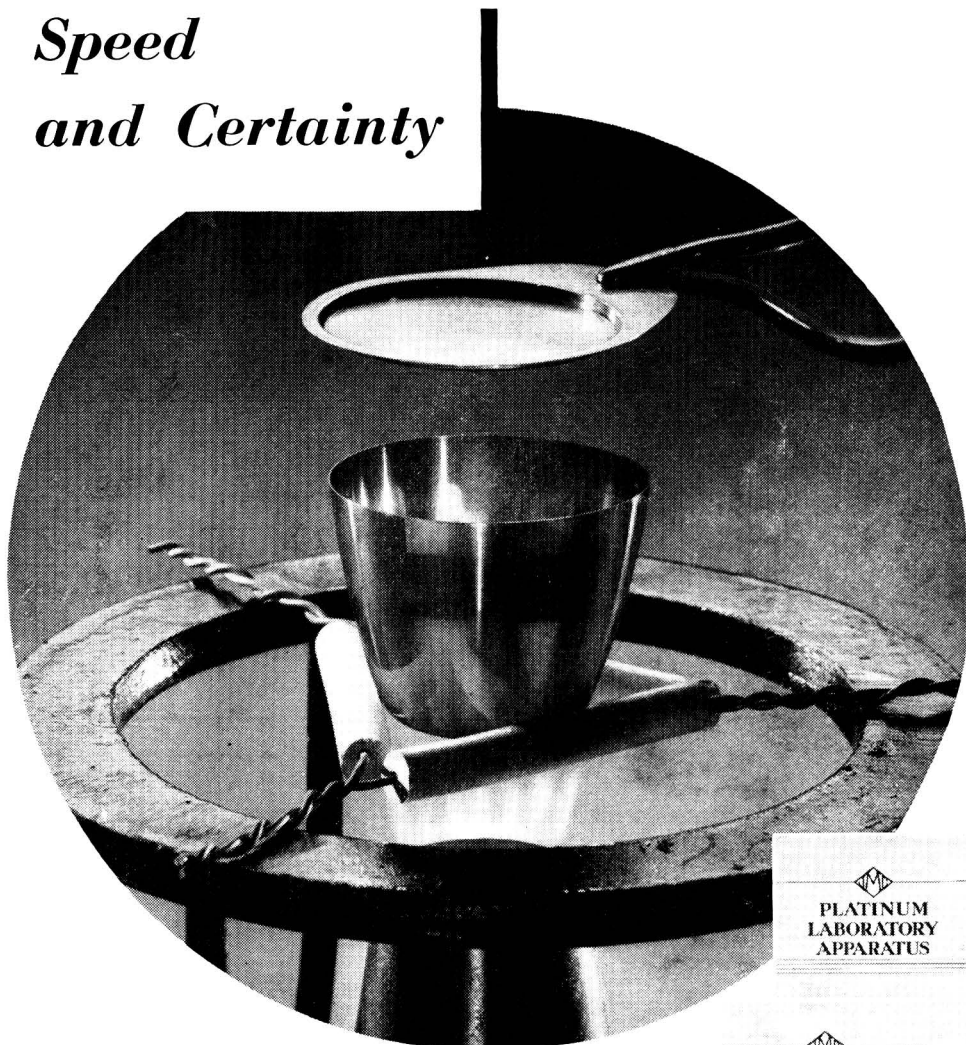
SOUTHERN INSTRUMENTS LIMITED
Special Products Division

CAMBERLEY

SURREY

Telephone : Camberley 2230 (3 lines)

Speed and Certainty



**PLATINUM
LABORATORY
APPARATUS**

**PLATINUM
ELECTRODES**

Most analysts would say that they use platinum apparatus for accurate work because of the certainty it provides—because errors caused by weight losses varying from piece to piece are completely avoided. But platinum, because of its high conductivity and very small heat capacity, enables work to be done faster, as well as more accurately. It is worth reviewing all your gravimetric work with this thought in mind.

Specialised Products of

Johnson  Matthey

Booklets 1710 "Platinum Laboratory Apparatus" and 1720 "Platinum Electrodes" contain valuable advice upon the care and maintenance of apparatus, and detail the JMC stock ranges of equipment. Both are free on request.

JOHNSON, MATTHEY & CO., LIMITED, HATTON GARDEN, LONDON, E.C.1
Telephone: HOLborn 6989. Vittoria St., Birmingham, 1. Telephone: Central 8004. 75-79 Eyre St., Sheffield, 1. Telephone: 29212

BOOKS ON THE CHEMICAL & ALLIED SCIENCES

Large Stock of Recent Editions.

Foreign Books, Select Stock. Books not in stock obtained under Board of Trade Licence. Catalogue on request.

SECONDHAND DEPT.

Select stock of recent editions of books on Science and Technology available. Back numbers of scientific journals obtainable. Books sought for and reported free of charge. Large or small collections bought.

LENDING LIBRARY

Scientific and Technical

Annual Subscription from 25s.

The Library is particularly useful to Societies and Institutions and to those engaged in research work.

Prospectus post free on request.

THE LIBRARY CATALOGUE, revised to December, 1949, containing a classified Index of Authors and Subjects. To Subscribers, 17s. 6d. net; to Non-Subscribers, 35s. net. Postage 1s. 3d. Supplement to 1950 to 1952. To subscribers 3s. net; to non-subscribers 6s. net; postage 6d.

Bi-monthly List of New Books and New Editions sent post free to subscribers on request.

H. K. LEWIS & Co. Ltd.

LONDON:

136, GOWER STREET, W.C.1.

Telephone: EUSton 4282

One of sixty
reagents
specially
prepared for
the Analoid
System of
Metallurgical Analysis.

"ANALOID" Reagent
COMPRESSED REAGENTS

Reacting Constituent

POT. PERIODATE 0.3g.

No. 39 Analoids

RIDSDALE & CO LTD Middlesbrough, Eng

Detailed List supplied on request

RIDSDALE & CO. LTD.

NEWHAM HALL, MIDDLESBROUGH

ANALYTICALLY STANDARDISED SAMPLES

Now available

For Universities, and Technical Colleges,
Special Series which includes Carbon and
alloy steels; cast irons; non-ferrous alloys
and assorted minerals

List No. 351A free on request from

**BUREAU OF ANALYSED SAMPLES
LTD.**

NEWHAM HALL, MIDDLESBROUGH

L. LIGHT & CO LTD

Items from our current stock—

3-Aminophthalhydrazide (luminol) ..	45/- D
p-Amino-acetophenone ..	58/- H
Anthrone ..	9/- D
p,p'-Azo-dimethyl aniline ..	20/- G
p-Bromomandelic acid ..	35/- D
N-Bromo-methyl phthalimide ..	55/- D
Calcium- α -naphthyl phosphate ..	32/- G
Calcium- β -naphthyl phosphate ..	32/- G
Celite, analytical filter aid ..	77/- lb
m-Cresoxy-acetic acid ..	17/- D
1,1'-Dianthrime ..	36/- D
Di-iodo phenol sulphonphthalein ..	10/- G
2,9-Dimethyl-1,10-phenanthroline ..	46/- G
5,6-Dimethyl-1,10-phenanthroline ..	80/- G
1,1'-Dinaphthyl ..	15/- G
2,6-Dinitro-phenol ..	25/- D
1,6-Diphenyl-hexa-1,3,5-triene ..	15/- G
2,5-Diphenyl-oxazole ..	15/- G
4,7-Diphenyl-1,10-phenanthroline ..	60/- G
2,2'-Diquinolyl ..	60/- G
Erichrome Black T ..	6/- D
Ethylenediamine-tetra-acetic acid Na ₂ Salt ..	14/- H
N-Ethyl-maleimide ..	55/- G
2-(o-Hydroxyphenyl)benzimidazole ..	69/- D
2-(o-Hydroxyphenyl)benzoxazole ..	65/- D
p-Hydroxyphenyl pyruvic acid ..	56/- G
o-Iodosobenzoic acid ..	60/- D
Lithium aluminium hydride ..	164/- H
Lithium borohydride ..	120/- D
Methyliminodiacetic acid ..	56/- H
9-Methyl-2,3,7-trihydroxy-6-fluorone ..	4/- G
Morin ..	30/- D
Murexide ..	25/- D
Naphtho-resorcinol ..	12/- G
Neocupferron ..	5/- G
5-Nitro-1,10-phenanthroline ..	36/- G
Nitro-phenol phthalein ..	9/- G
Nitro phenol sulphonphthalein ..	12/- G
Nitro thymol ..	13/- G
p-Nitro phenyl phosphate, Na, Salt ..	12/- G
1,10-Phenanthroline hydrate or HCl ..	50/- D
o-Phthaldialdehyde ..	60/- D
Potassium borohydride ..	48/- D
Quinaldine acid ..	20/- D
Resazurin, sodium ..	90/- D
Sodium borohydride ..	15/- G
Sodium iodoacetate ..	10/- D
Sodium pyruvate ..	45/- D
Sodium rhodizonate ..	56/- D
Tetraphenylboron, sodium salt ..	67/- D
Thioformamide (etheral solution) ..	97/- H
Trifluoroacetic acid ..	13/- D
2,3,5-Tri-iodo-benzoic acid ..	40/- D
2,4,7-Trinitrofluorenone ..	36/- D
2,3,5-Triphenyl tetrazolium chloride or bromide ..	24/- D
Tris(hydroxymethyl) amino methane ..	18/- H
Xanthidrol ..	14/- D

POYLE TRADING ESTATE
COLNBROOK, BUCKS

THE ANALYST

THE JOURNAL OF
THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

PUBLICATION COMMITTEE

Chairman: J. R. Nicholls, C.B.E. *Members:* N. L. Allport, A. J. Amos, A. L. Bacharach, R. C. Chirnside, B. S. Cooper, L. Eynon, D. C. Garratt, J. Haslam, H. M. N. H. Irving, G. Roche Lynch, O.B.E., G. H. Osborn, J. E. Page, A. A. Smales, George Taylor, O.B.E., L. S. Theobald, Eric Voelcker, C. Whalley, E. C. Wood.

President of the Society: D. W. Kent-Jones

Hon. Secretary of the Society:
K. A. Williams

Secretary:
Mrs. D. V. Hicks

Hon. Treasurer of the Society:
J. H. Hamence

Editor: F. L. Okell, F.R.I.C.

Assistant Editor: J. B. Attrill, M.A.

CONTENTS

	<i>Page</i>
Proceedings of the Society of Public Analysts and Other Analytical Chemists	
Ordinary Meeting	685
New Members	685
Death	685
Original Papers	
Micro-analysis of Silicate Rocks—Part IV. The Determination of Alumina—Christina C. Miller and Robert A. Chalmers	686
Ultra-violet Spectrophotometric Estimation of the Quality of Mineral Oils Extracted from Bread—M. A. Cookson, J. B. M. Coppock and R. Schnurmman	695
The Spectrophotometric Determination of Long-chain Fatty Acids Containing Ketonic Groups—With particular reference to Licanic Acid—A. Mendelowitz and J. P. Riley	704
Design and Operating Technique of a Vacuum Drying Oven—Part I. Design of the Oven—S. D. Gardiner	709
The Determination of Total Phosphatide in Commercial Lecithin—H. H. Hutt, H. Weatherall and T. Culshaw	712
The Determination of Small Amounts of Potassium, Calcium and Magnesium in Sodium and its Compounds—Louis Silverman and K. Trego	717
An Isopiestic Method for the Micro-determination of Molecular Weights—Miss J. E. Morton, A. D. Campbell and T. S. Ma	722
Notes	
The Lipase Activity of Certain Cereal Products—W. H. Templeton and B. R. Carpenter	726
Tests for Nitrite and Nitrate Applicable over Wide Concentration Ranges—P. Woodward	727
Preparation of a Material Rich in "Cation" suitable for the Detection of Cadmium—E. Fisher, B. T. Estes and J. E. Rose	729
Adsorption of Thiamine on Glassware—K. T. H. Farrer and W. C. J. Hollenberg	730
The Determination of Acetylene in Air—B. J. Purser	732
A Qualitative Test for Monosaccharides—R. M. Love	732
The Determination of Zinc by Titration with Di-sodium Ethylenediaminetetra-acetate—N. Strafford	733
Apparatus	
A Modified All-glass Apparatus for the Determination of Nitrogen by the Micro-Kjeldahl Method—F. J. Scandrett	734
Ministry of Food	737
British Standards Institution	738
Book Reviews	
Inorganic Thermogravimetric Analysis. By Clément Duval	738
Polysaccharide Chemistry. By R. L. Whistler and C. L. Smart	740
Publications Received	740

THE SOCIETY OF PUBLIC ANALYSTS
AND OTHER ANALYTICAL CHEMISTS

BULLETIN

FORTHCOMING MEETINGS

Ordinary Meeting of the Society, October 23rd, 1953

AN Ordinary Meeting of the Society will be held in the Lecture Hall of **Southampton** University on Friday, October 23rd, 1953, at 5 p.m., and will be preceded by a visit to the Esso Refinery at Fawley. This meeting has been organised by the Physical Methods Group.

The subject of the meeting will be "Paper Electrophoresis," and the following papers will be presented—

"The Analysis of Inorganic Compounds by Electromigration and Electrochromatography," by F. H. Pollard, B.Sc., Ph.D.

"The Use of Paper Electrophoresis in the Study of Nucleic Acids," by Roy Markham, M.A., Ph.D.

"Paper-Strip Electrophoresis of Serum Proteins," by A. L. Latner, M.Sc., M.D., F.R.I.C.

Ordinary Meeting of the Society, November 4th, 1953

AN Ordinary Meeting of the Society will be held at 7 p.m. on Wednesday, November 4th, 1953, in the Meeting Room of the Chemical Society, Burlington House, London, W.1.

The subject of the meeting will be "The Determination of Niobium in Minerals and Mineral Dressing Products," and the following papers will be presented—

"The Absorptiometric Determination of Niobium in Some African Low-grade Minerals and Mineral Dressing Products," by G. W. C. Milner, B.Sc., F.R.I.C., A.Inst.P., and A. A. Smales, B.Sc., F.R.I.C. (A.E.R.E., Harwell).

"The Absorptiometric Determination of Niobium in Some African Low-grade Ores," by A. E. O. Marzys (Uganda Development Corporation, Ltd.).

"Spectrographic Determination of Niobium and Tantalum in Sukulu-type Soils," by C. S. Campbell, M.A., and D. Nicholas (Fulmer Research Institute, Ltd.).

"Inorganic Chromatography on Cellulose. Part XIV. A Shortened Method for the Determination of Niobium and Tantalum in Minerals and Ores," by R. A. Mercer and R. A. Wells, B.Sc., A.R.I.C. (Chemical Research Laboratory, Teddington).

"The Colorimetric Estimation of Niobium and Tantalum with Pyrogallol," by E. C. Hunt, B.Sc., A.R.I.C., and R. A. Wells, B.Sc., A.R.I.C. (Chemical Research Laboratory, Teddington).

"Inorganic Chromatography on Cellulose. Part XV. A Rapid Method for the Determination of Niobium in Low-grade Ores," by E. C. Hunt, B.Sc., A.R.I.C. (Chemical Research Laboratory, Teddington).

Annual General Meeting of the Society, March 3rd, 1954

THE Annual General Meeting of the Society will be held at 4.30 p.m. on **Wednesday, March 3rd, 1954,*** in the Meeting Room of the Royal Society, Burlington House, London, W.1.

This will be followed at 5 p.m. by the Bernard Dyer Memorial Lecture given by E. B. Hughes, D.Sc., F.R.I.C.

Meeting of the North of England Section, November 28th, 1953

AN Ordinary Meeting of the North of England Section will be held at 2 p.m. on Saturday, November 28th, 1953, at the City Laboratories, Liverpool.

At this meeting a paper on "Chromatography" will be presented by Dr. R. L. M. Synge, F.R.S.

Meeting of the Scottish Section, November 10th, 1953

AN Ordinary Meeting of the Scottish Section will be held at 7.15 p.m. on Tuesday, November 10th, 1953, at the Central Station Hotel, Glasgow.

The following papers will be presented—

"Rapid Determination of Glycerol in Fermentation Solutions: a New Chromatographic Procedure," by A. F. Williams, B.Sc., F.R.I.C.

"Field Analysis in Connection with Water Treatment Problems," by I. A. Heald.

Annual General Meeting of the Physical Methods Group, November 24th, 1953

THE Ninth Annual General Meeting of the Physical Methods Group will be held at 6 p.m. on Tuesday, November 24th, 1953, in the Meeting Room of the Chemical Society, Burlington House, London, W.1.

The Business Meeting will be followed by a Lecture by the retiring Chairman, J. Haslam, D.Sc., F.R.I.C.

PAPERS ACCEPTED FOR PUBLICATION IN *THE ANALYST*

THE following papers have been accepted for publication in *The Analyst*, and are expected to appear in the near future. It is not possible to enter into correspondence about any of them.

"A Simple Volumetric Method for the Routine Determination of Glycerol," by J. W. B. Erskine, C. R. N. Strouts, G. Walley and W. Lazarus.

Glycerol is oxidised at room temperature by a solution of sodium meta-periodate to give formaldehyde and formic acid. The formic acid, after the excess of periodate has been destroyed with ethylene glycol, is titrated with standard alkali, with phenol red as indicator, to give a measure of the glycerol present. Carbon dioxide must be excluded, and the oxidation must be carried out in the dark to minimise side reactions between sodium meta-periodate and the formaldehyde and formic acid. A number of other polyhydric alcohols and sugars interfere, but these are not likely to be present in the glycerol-containing products of soap-making and fat-splitting, such as crude glycerine, lye and soap. The most likely organic impurities in these products are trimethylene glycol, which does not react, and polyglycerols, which do not yield formic acid; these substances, therefore, do not interfere as they do in the acetin and dichromate methods. Glycols with adjacent hydroxyl groups, like ethylene glycol, react, but produce only formaldehyde.

The method has been checked against distilled glycerine, made-up soap-lye crude glycerine, soap lyes and soaps containing known added amounts of glycerol, and also on commercial crudes, soap lyes and soaps by comparison with the acetin and dichromate methods.

* The date of this meeting has been altered since the Programme of Meetings for the Session was printed.

"The Determination of *iso*Butyraldehyde in *n*-Butyraldehyde," by G. R. Primavesi.

*iso*Butyraldehyde and *n*-butyraldehyde are reduced to the corresponding alcohols by sodium borohydride. The *isobutanol* is then estimated absorptiometrically by means of a modified Komarowsky reaction for higher alcohols, of improved specificity, carried out under controlled conditions.

"The Quantitative Separation of Copper, Lead and Tin by Cathodic Deposition," by G. H. Aylward and A. Bryson.

A method is described for the quantitative separation of copper, lead and tin by cathodic deposition from phosphoric acid solution. The tin forms an anionic complex that is not reduced at the cathode and the copper is separated from the lead by deposition at a controlled potential. The lead is deposited in a metallic form and the tin is determined volumetrically. The difficulties associated with the quantitative cathodic deposition of lead have been investigated and optimum working conditions have been established to avoid these difficulties.

"Diethylammonium Diethyldithiocarbamate for the Separation and Determination of Small Amounts of Metals," by P. F. Wyatt.

A scheme for the successive separation and absorptiometric determination of copper, manganese and iron is described, whereby iron is isolated as cupferrate, and copper and manganese separately as diethyldithiocarbamates. Methods are described for the absorptiometric determination of all three elements as their coloured diethyldithiocarbamate complexes in chloroform, and alternative absorptiometric procedures are indicated.

Tests on random mixtures of the three metals over the ranges 20 to 1000 μg of iron, 5 to 70 μg of copper and 10 to 200 μg of manganese show good recoveries.

"The Photometric Determination of Phosphorus in Copper-Based Alloys Containing Tin," by H. K. Lutwak.

The method of the A.S.T.M. (E62-50T) for the determination of phosphorus in copper-based alloys has several shortcomings. An investigation has been made to obviate them and to simplify the method.

Calibration graphs that obeyed Beer's law were obtained by (a) determining the sensitivity of the phosphovanadomolybdate complex to acidity and dilution changes and choosing the most favourable conditions, and (b) increasing the rate of colour development by heating the coloured solution before dilution. Colour intensities that reach their final value at room temperature are stable; colours obtained on heating fade slowly on standing.

Separation of metastannic acid is prevented by the use of a suitable mixture and amount of nitric and hydrochloric acids in the final solution. The conditions chosen also provide optimum acidity for colour development.

The same procedure is recommended for development of calibration graphs for alloys of both low and high phosphorus contents.

"The Indirect Polarographic Determination of Calcium by Chloranilic Acid," by B. Breyer and J. McPhillips.

Calcium reacts quantitatively with chloranilic acid, precipitating calcium chloranilate. The concentration of the calcium can be determined, without removing the precipitate, by measuring the polarographic diffusion current of the residual chloranilic acid and referring the value obtained to a calibration graph. Calcium can thus be determined within a concentration range of $1.3 \times 10^{-4} M$ to $1.2 \times 10^{-2} M$ with a maximum error of ± 3 per cent.

The determination cannot be carried out in the presence of cobalt, zinc, lead, manganese, cadmium, nickel, aluminium, copper and silver ions, because they precipitate the reagent. Sodium, potassium, ammonium, magnesium, iron and mercury ions do not cause precipitation, but ferric and mercuric ions interfere if their concentrations are greater than $4.8 \times 10^{-4} M$ and $5.9 \times 10^{-4} M$, respectively. Magnesium does not interfere if present at concentrations of less than $2.4 \times 10^{-3} M$, nor do lithium, barium and chromium when their concentrations are below $5.9 \times 10^{-4} M$. Strontium does not affect the determination at concentrations up to $3.6 \times 10^{-4} M$.

Calcium has been determined in blood serum and in milk.

"The Determination of Small Amounts of Potassium, Calcium and Magnesium in Sodium and its Compounds," by Louis Silverman and K. Trego.

Preliminary concentration is necessary before potassium can be determined in the presence of large amounts of sodium ion.

Metallic sodium, sodium hydroxide and sodium salts are converted to chloride. Measured, cold, saturated aqueous chloride solutions are treated with dry hydrogen chloride gas, which separates the bulk of the sodium chloride. The residual potassium, calcium and magnesium can then be determined by the usual methods.

Calcium and magnesium may also be determined with or without preliminary sodium chloride separation.

"Determination of Borate in Presence of Silver," by S. Z. Haider.

A modification of the usual volumetric procedure for the determination of boric acid in presence of silver ions is described. The interfering effect of the silver ion is first nullified by masking it as a soluble thiosulphate complex, after which the usual procedure is applicable. The proportions of free acid and thiosulphate have to be carefully controlled.

The investigation covered a range of boric acid from 0.0805 to 0.2480 g and sodium pyroborate from 0.0560 to 0.1505 g in mixtures with different proportions of silver nitrate. Results were found to vary within an error of ± 0.7 per cent.

"The Analysis of Acid Chlorides," by C. C. T. Chinnick and P. A. Lincoln.

A method is described for the analysis of samples of organic acid chlorides containing as impurities free organic acid, anhydride and hydrochloric acid.

The procedure consists in (i) plotting a titration graph with a pH meter, from which the points corresponding to the neutralisation of hydrochloric acid and organic acid are read and (ii) determining the ester by hydrolysis with an excess of standard alkali.

Special provision is made for determining anhydride in the presence of hydrochloric acid and details of an alternative, but less convenient, titration procedure, by means of a double indicator, are also given.

Formulae for the calculation of results are included in the paper.

"Micro-analysis of Silicate Rocks," by Christina C. Miller and Robert A. Chalmers.

A new procedure is prescribed for the separation and direct determination of alumina in 5-mg samples of silicate rocks. Silica is volatilised by heating with hydrofluoric and sulphuric acids, and the residue is fused with potassium bisulphate and extracted with *N* hydrochloric acid. Iron, titanium, vanadium and zirconium are removed together by precipitation with cupferron and extraction with *o*-dichlorobenzene. Acetylacetone is added to the aqueous phase, and aluminium and beryllium acetylacetonates are extracted from the buffered solution at a pH of 6 to 7 by means of diethyl ether. From the ether extract aluminium and beryllium are withdrawn into 6 *N* hydrochloric acid, and the aluminium alone is precipitated and weighed as aluminium 8-hydroxyquinolate.

The method has been applied in the presence of all the elements commonly found in silicate rocks. In the analysis of rocks, the results attained are relatively 1 per cent. lower than the corresponding ones based on the classical procedure.

THE ANALYST

THE JOURNAL OF THE Society of Public Analysts and Other Analytical Chemists

A MONTHLY JOURNAL DEVOTED TO THE ADVANCEMENT
OF ANALYTICAL CHEMISTRY

Publication Committee :

Chairman : J. R. NICHOLLS, C.B.E., D.Sc., F.R.I.C.

N. L. ALLPORT, F.R.I.C.	G. H. OSBORN, F.R.I.C., A.M.Inst.M.M.
A. J. AMOS, B.Sc., Ph.D., F.R.I.C.	J. E. PAGE, B.Sc., Ph.D., F.R.I.C.
A. L. BACHARACH, M.A., F.R.I.C.	A. A. SMALES, B.Sc., F.R.I.C.
R. C. CHIRNSIDE, F.R.I.C.	GEORGE TAYLOR, O.B.E., F.R.I.C.
B. S. COOPER, B.Sc., F.Inst.P.	L. S. THEOBALD, M.Sc., A.R.C.S.
L. EYNON, B.Sc., F.R.I.C.	F.R.I.C.
D. C. GARRATT, B.Sc., Ph.D., F.R.I.C.	ERIC VOELCKER, A.R.C.S., F.R.I.C.
J. HASLAM, D.Sc., F.R.I.C.	C. WHALLEY, B.Sc., A.R.I.C.
H. M. N. H. IRVING, M.A., D.Phil., F.R.I.C.	E. C. WOOD, B.Sc., Ph.D., A.R.C.S., F.R.I.C.
G. ROCHE LYNCH, O.B.E., M.B., B.S., D.P.H., F.C.G.I., L.M.S.S.A., F.R.I.C.	

President of the Society :

D. W. KENT-JONES, B.Sc., Ph.D., F.R.I.C.

Hon. Secretary of the Society :

K. A. WILLIAMS, B.Sc., Ph.D., F.R.I.C.,
A.Inst.P.

Hon. Treasurer of the Society :

J. H. HAMENCE, M.Sc., Ph.D., F.R.I.C.

Editor : F. L. OKELL, F.R.I.C.

Assistant Editor :

J. B. ATTRILL, M.A.

VOL. 78

1953

PUBLISHED FOR THE SOCIETY BY

W. HEFFER & SONS LTD.

4, PFTTY CURY,

ERRATA:

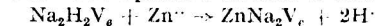
VOL. 77, 1952:

Sept., p. 463. The equation on line 4: $x(y - \text{titration})/5$ is applicable only when $x = 50$, as described in the procedure. The general expression should be: $500(y - \text{titration})/x$.

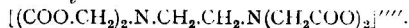
VOL. 78, 1953:

p. 219. The paper by Rây should bear the date "August 14th, 1952."

p. 309. The equations on lines 33 and 35 should read:—



where V_6 is



p. 551, title and lines 13 and 17 of synopsis; p. 552, line 9. For "esters" read "ethers."

INDEX TO VOLUME 78

INDEX TO NAMES

* Denotes authors of original papers and notes that have been published in *The Analyst*.

A

- Adams, C. A.** Random reflections on food legislation, 569.
- Adams, R.** Organic Reactions. Vol. VII. (Publication received), 684.
- Adamson, D. C. M.** Review of *Antibiotics: Survey of Their Properties and Uses*. 2nd Edn., 330.
- Ainsworth, G. C.** Medical Mycology, Introduction to Problems. (Publication received), 71.
- ***Albon, N., et al.** Large-scale chromatographic separation of sucrose-raffinose mixtures on powdered cellulose for detmng. raffinose in raw sugars, 191.
- Allport, N. L.** Review of *Handbook of Colorimetric Chemical Analytical Methods for Industrial, Research and Clinical Laboratories. Developed for Use with Lovibond Comparator*, 568.
- Amos, A. J.** Review of Barton-Wright's *Microbiological Assay of Vitamin-B Complex and Amino-Acids*, 129.
- ***Angell, F. G.** Detmng. small amounts of *m*-dinitrobenzene in nitrobenzene, 603.
- Arnold, R. T.** Organic Syntheses. Vol. 32. (Publication received), 188.
- Arthur, P., et al.** Semimicro Qualitative Analysis. 3rd Edn. (Publication received), 72; (Review), 447.
- ***Ashton, G. C., et al.** Colorimetric detmn. of dihydrostreptomycin, 581.
- Audrieth, L. F., et al.** Non-Aqueous Solvents. Applications as Media for Chemical Reactions. (Publication received), 448.
- ***Awad, S. A., et al.** Potentiometric detmn. of quadrivalent tellurium by potassium permanganate in weakly alkaline solutions, 487.
- Awbery, J. H.** University Textbook of Physics. Vol. III. Heat. 11th Edn. (Publication received), 71.
- ***Ayers, C. W.** Chromatographic separation of polyamides, 382.
- ***Aylward, G. H., et al.** Mercury-cathode cell for electrolytic separations, 386.
- *— Quantitative separation of copper, lead and tin by cathodic deposition, 651.

B

- ***Baar, S.** Colorimetric micro-detmn. of potassium in serum, 353.
- Bacharach, A. L.** Review of Finney's *Statistical Method in Biological Assay*, 505.

- ***Baker, P. R. W.** Decomposition of ammonia in sealed-tube micro-Kjeldahl digestions with selenium catalyst, 500.
- Ball, E. G.** Biochemical Preparations. Vol. II. (Publication received), 71; (Review), 188.
- Barnard, G. P.** Modern Mass Spectrometry. (Publication received), 448.
- ***Barnes, H.** Double syringe-pipette for dissolved oxygen estmns., 501.
- Barnett, W. L.** Composition of Jamaica rum, 259.
- Barrett, F. C., et al.** Practical Chromatography. (Publication received), 684.
- Barton-Wright, E. C.** Microbiological Assay of Vitamin-B Complex and Amino-Acids. (Publication received), 71; (Review), 129.
- Bass, A. M., et al.** Spectrophotometric Atlas of the $2\Sigma^+ - 2\Pi$ Transition of OH. (Publication received), 628.
- ***Benfield, D. A., et al.** Detmng. carbon dioxide and sulphur dioxide in Orsat apparatus, 320.
- Better, E. J., et al.** Soap Manufacture. Vol. I. (Publication received), 740.
- Bhattacharya, G. N., et al.** Revisers of Rangaswami and Sen's *Handbook of Shellac Analysis*. 2nd Edn. (Publication received), 72; (Review), 130.
- ***Biggs, R.** Control of anticoagulant therapy, 84.
- ***Bishop, E.** Opening of white metals for analysis by dry chlorine, 61.
- *— Replacement of standard cell and salt bridge by indicator electrodes and use of non-aqueous solutions in potentiometry. II. Iodometry and iodimetry in aqueous solution, 149.
- ***Bishop, J. R., et al.** Detmng. aluminium and zinc after chromatographic separation from tin-lead alloys, 117.
- Block, R. J., et al.** Paper Chromatography. Laboratory Manual. (Review), 388.
- Blow, C. M., et al.** Natural Rubber Latex and its Applications. No. 2. Latex Casting. (Publication received), 71.
- Blunt, R.** Appointment as Deputy Public Analyst and Deputy Official Agricultural Analyst to County Borough of Burnley, 323.
- Bolton, H. L.** Review of Friedel and Orchin's *Ultraviolet Spectra of Aromatic Compounds*, 69.
- Bose, P. K., et al.** Revisers of Rangaswami and Sen's *Handbook of Shellac Analysis*. 2nd Edn. (Publication received), 72; (Review), 130.
- Bowen, E. J., et al.** Fluorescence of Solutions. (Publication received), 684.
- ***Bradshaw, G., et al.** Volumetric detmn. of zinc with ferrocyanide in magnesium alloys, 367.

- Brantlect, C. A.** Starch: Sources, Production and Uses. (Publication received), 568.
- Brevis, J. G., et al.** Fertilizer Experiments in Natal, 1933-50. (Publication received), 506.
- ***Breyer, B., et al.** Indirect polarographic detmn. of calcium by chloranilic acid, 666.
- Bright, H. A., et al.** Revisers of Hillebrand and Lundell's *Applied Inorganic Analysis. With Special Reference to Analysis of Metals, Minerals and Rocks*. 2nd Edn. (Publication received), 448.
- Brimley, R. C., et al.** Practical Chromatography. (Publication received), 684.
- Broida, H. P., et al.** Spectrophotometric Atlas of the $^2\Sigma^+ \rightarrow ^2\Pi$ Transition of OH. (Publication received), 628.
- ***Brown, E. G.** Qualitative detcn. of tellurium in tellurium-lead alloys, 623.
- Brown, G. I.** Simple Guide to Modern Valency Theory. (Publication received), 568.
- Browning, E.** Toxicity of Industrial Organic Solvents. 2nd Edn. (Publication received), 448.
- ***Bryant, F. J., et al.** Research polarograph for photographic recording and a multipurpose polarographic cell, 373.
- ***Bryson, A., et al.** Critical examination of ferrocyanide detmn. of zinc, 291.
- *— Quantitative separation of copper, lead and tin by cathodic deposition, 651.
- *— Separation of zinc and cadmium by activated copper, 299.
- ***Burden, E. H. W. J., et al.** Modified procedure for quantitative bromine absorption of oils and fats, 619.
- ***Busfield, H., et al.** Detmng. added hexamethylenetetramine in two-stage phenol-formaldehyde resins, 617.
- ***Butler, E. J., et al.** Reversion method for absorptiometric detmn. of traces of lead with dithizone, 571.
- ***Button, D. F. H.** Adulterant of dried sage, 679.
- Appointment as Public Analyst to Metropolitan Borough of Hammersmith, 67.
- C**
- ***Cama, H. R., et al.** Spectroscopic properties of vitamin A₂. Application to assay of cod-liver oil, 74.
- Cameron, M. P., et al.** Ciba Foundation Colloquia on Endocrinology. Vol. II. Steroid Metabolism and Estmn. (Publication received), 72; Vol. V. Bioassay of Anterior Pituitary and Adrenocortical Hormones. (Publication received), 448.
- ***Campbell, A. D., et al.** Isopiestic method for micro-detmn. of molecular weights, 722.
- Carlos, A. S.** Appointment as Public Analyst and Official Agricultural Analyst to County Borough of Bournemouth, 323.
- ***Carpenter, B. R., et al.** Lipase activity of cereal products, 726.
- ***Chalmers, R. A.** Micro-analysis of silicate rocks. III. Spectrophotometric detmn. of phosphoric oxide in presence of silica, 32.
- *— et al. Micro-analysis of silicate rocks. II. Pptn. of silica as 2:4-dimethylquinoline silicomolybdate and gravimetric detmn. as silicomolybdic anhydride, 24; IV. Detmng. alumina, 686.
- ***Chinnick, C. C. T., et al.** Analysis of acid chlorides, 675.
- ***Chipperfield, E. H., et al.** Detmng. added hexamethylenetetramine in two-stage phenol-formaldehyde resins, 617.
- Chirnside, R. C.** Review of Duval's *Inorganic Thermogravimetric Analysis*, 738.
- Clark, F.** Reports on Progress of Applied Chemistry. Vol. XXXVII. (Publication received), 568.
- ***Clark, G. C., et al.** Identifying alloys and stainless steels by electrographic methods, 145.
- Clark, J. F.** Appointment as Official Agricultural Analyst to County Borough of Wallasey, 387.
- Clark, T. M.** Appointment as Additional Public Analyst to City of Glasgow, 257.
- ***Collier, R. E., et al.** Modified dead-stop end-point circuit for titrations of ferrous iron with potassium dichromate in approximately N solutions, 440.
- ***Cookson, M. A., et al.** Ultra-violet spectrophotometric estmn. of quality of mineral oils extracted from bread, 695.
- ***Cooper, R. L., et al.** Deteng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage, 170.
- Cooper, T. H., et al.** Phenylfluorone. Reagent for Germanium. (Publication received), 388.
- ***Coppock, J. B. M., et al.** Ultra-violet spectrophotometric estmn. of quality of mineral oils extracted from bread, 695.
- ***Corbett, J. A.** Detmng. magnesium and aluminium in titanium metal, 20.
- ***Coulson, E. A., et al.** Detmng. beta- and gamma-piccolines, 2:6-lutidine and 2-ethylpyridine in mixtures by infra-red spectroscopy, 114.
- ***Culshaw, T., et al.** Detmng. total phosphatide in commercial lecithin, 712.
- ***Cunningham, M. N., et al.** Detmng. unfermentable reducing substances in molasses, 321.
- *— Preservation of fermenting liquors in detmn. of reducing sugars, 320.
- Curran, S. C.** Luminescence and Scintillation Counter. (Publication received), 568.
- D**
- Dalton, N. N., et al.** Glycerol. (Publication received), 568.
- ***Davenport, J. B.** Departure from Beer's law affecting spectrophotometric detmn. of diphenyl, 558.
- Davidsohn, A., et al.** Soap Manufacture. Vol. I. (Publication received), 740.
- Davidsohn, J., et al.** Soap Manufacture. Vol. I. (Publication received), 740.
- Davies, R. E. L.** Obituary, 2, 264.
- Davies, W. C.** Review of Kolthoff and Lingane's *Polarography*. Vols. I and II. 2nd Edn., 327.
- ***Davis, D. E., et al.** Analysis of rosin size, 670.
- ***Davis, H. M., et al.** Randles-type cathode-ray polarograph, 314.
- Davis, J. G., et al.** Revisers of *Richmond's Dairy Chemistry*. 5th Edn. (Publication received), 448.
- ***de Bruin, A. S., et al.** Deteng. preservatives in beverages by fermentation test, with special reference to brominated compounds, 37.
- Dedicoat, H.** Appointment as Public Analyst to Borough of Bacup, 444.
- ***Deutschman, J., et al.** Volumetric detmn. of zinc with ferrocyanide in magnesium alloys, 367.
- ***Dickinson, L.** Evaluation of anti-viral compounds, 283.
- Dickson, W.** Obituary, 189.
- Diehl, H., et al.** Quantitative Analysis. Elementary Principles and Practice. (Publication received), 260.
- ***Diggie, W. M., et al.** Detmng. keten and acetic anhydride in atmosphere, 473.

- *Donald, G. M. S., *et al.* Detmng. unfermentable reducing substances in molasses, 321.
- *— Preservation of fermenting liquors in detmn. of reducing sugars, 320.
- Drummond, Sir Jack. Memorial Fund, 449.
- Obituary, 3, 264.
- *Dubravčić, M., *et al.* Micro-detmn. of iodides by arresting catalytic reduction of ceric ions, 594.
- *Duncan, R. E. B., *et al.* Identifying and detmng. lower straight-chain fatty acids by paper partition chromatography, 641.
- Dunlop, A. P., *et al.* Furans. (Publication received), 684.
- Duval, C. Inorganic Thermogravimetric Analysis. (Publication received), 188; (Review), 738.
- Dyche-Teague, F. C. Obituary, 134, 264.
- Dyer, B. Memorial lecture. Editorial, 261.
- Memorial medal selected for exhibition, 450.
- Photograph of Memorial medal, facing p. 261.
- E**
- *Easterbrook, W. C., *et al.* Micro-detmn. of alkoxyl values in cellulose ethers, 551; Erratum, 616.
- Eden, A. Review of Hewitt's *Sand and Water Culture Methods Used in Study of Plant Nutrition*, 329.
- Edwards, F. C. G., *et al.* Aids to Qualitative Pharmaceutical Analysis. (Publication received), 684.
- Elderfield, R. C. Heterocyclic Compounds. Vol. II. Five- and Six-Membered Polycyclic Compounds Containing One O or S Atom. (Review), 259; Vol. III. Polycyclic Derivatives of Pyrrole; Polycyclic Systems with One Nitrogen Common to Both Rings; Pyrindine and Related Compounds. Vol. IV. Quinoline, isoQuinoline and Their Benzo Derivatives. (Review), 260.
- Ellis, B. A. Review of Elderfield's *Heterocyclic Compounds*, Vol. II, 259; Vols. III and IV, 260.
- Review of Pesez and Poirier's *Méthodes et Réactions de l'Analyse Organique*. Vol. I, 70; Vol. II, 567.
- Review of Pullman and Pullman's *Théories Électroniques de la Chimie Organique*, 329.
- Review of Venkataraman's *Chemistry of Synthetic Dyes*. Vol. I, 131.
- Elvidge, W. F. Appointment as Deputy Public Analyst to County of Cumberland, County of Durham, County Borough of Carlisle, County Borough of Darlington, County Borough of South Shields, County Borough of West Hartlepool and Borough of Stockton-on-Tees, and Deputy Official Agricultural Analyst to County of Cumberland, County of Durham, County Borough of Darlington, County Borough of South Shields and County Borough of West Hartlepool, 387; as Deputy Public Analyst to County of Northumberland, 444; as Deputy Public Analyst and Deputy Official Agricultural Analyst to County of Westmorland, 626.
- *Erskine, J. W. B., *et al.* Volumetric routine detmn. of glycerol, 630.
- *Estes, B. T., *et al.* Prepg. material rich in cation suitable for detcng. cadmium, 729.
- F**
- *Fatherley, M., *et al.* Colorimetric detmn. of dihydrostreptomycin, 581.
- *Feldstein, M., *et al.* Analysis of general unknowns in toxicology, 43.
- *Fernell, W. R., *et al.* Simultaneous detmn. of pentose and hexose in sugar mixtures, 80.
- *Ferrett, D. J., *et al.* Construction of polarograph capillaries from Pyrex glass rod, 564.
- *Feuell, A. J., *et al.* Detmng. carbonyl compounds by semicarbazide and hydroxylamine, with special reference to fatty-acid oxidation products, 135.
- Findlay, A. General and Inorganic Chemistry. (Publication received), 188, 332.
- Introduction to Physical Chemistry. 3rd Edn. Revised by H. W. Melville. (Publication received), 260.
- Review of Hammett's *Introduction to Study of Physical Chemistry*, 505.
- Review of Partington's *Advanced Treatise on Physical Chemistry*. Vol. III, 331.
- Finney, D. J. Statistical Method in Biological Assay. (Publication received), 71; (Review), 505.
- *Fisher, E., *et al.* Prepg. material rich in cation suitable for detcng. cadmium, 729.
- Flett, L. H., *et al.* Maleic Anhydride Derivatives. Reactions of the Double Bond. (Publication received), 131.
- Flint, J. W. Appointment as Deputy Public Analyst to Borough of Chatham, 257.
- *Folkes, B. F. *p*-Hydroxydiphenyl test for acet-aldehyde: detmng. alanine and threonine in protein hydrolysates, 496.
- *Forster, W. A. Detmng. copper in plant material, 614.
- *— Detmng. magnesium in plant material with ethylenediamine-tetra-acetic acid, 179.
- *— Detmng. nickel in plant material in presence of other metals, 560.
- Forstner, G. E. Obituary, 189.
- Foster, G. E. Review of Velluz's *Substances Naturelles de Synthèse; Préparations et Méthodes de Laboratoire*. Vols. I-IV, 446.
- *Foster, M. C., *et al.* Colorimetric detmn. of dihydrostreptomycin, 581.
- Francis, W., *et al.* Composition and Assaying of Minerals. (Publication received), 71.
- *Freeman, F., *et al.* Detmng. chloromethyl-phenoxyacetic acids in MCPA formulations, 205.
- *Freeman, G. G., *et al.* Detmng. unfermentable reducing substances in molasses, 321.
- *— Preservation of fermenting liquors in detmn. of reducing sugars, 320.
- *Fricker, D. J., *et al.* Modified dead-stop end-point circuit for titrations of ferrous iron with potassium dichromate in approximately *N* solutions, 440.
- Friedel, R. A., *et al.* Ultraviolet Spectra of Aromatic Compounds. (Review), 69.
- Fruton, J. S., *et al.* General Biochemistry. (Publication received), 568.
- Fuyat, R. K., *et al.* Standard X-ray Diffraction Powder Patterns. Vol. II. (Publication received), 740.
- G**
- *Gage, J. C., *et al.* Detmng. keten and acetic anhydride in atmosphere, 473.
- *Gardiner, S. D. Design and operating technique of vacuum drying oven. I. Design of oven, 709.
- *Gardner, K., *et al.* Detmng. chloromethyl-phenoxyacetic acids in MCPA formulations, 205.

- Gardner, W. H., et al.** Maleic Anhydride Derivatives. Reactions of the Double Bond. (Publication received), 131.
- Garside, J. E., et al.** Textbook of Pure and Applied Chemistry. (Publication received), 332.
- *Gaskin, J. G. N., et al.** Precipitate error in detmng. sugar polarisations, 334.
- Gaudenzi, N.** Guida Bibliografica Internazionale per il Chimico, Libri e Riviste. (Publication received), 71.
- Gautier, J.-A.** Mises au Point de Chimie Analytique Pure et Appliquée et d'Analyse Bromatologique. (Publication received), 740.
- Gaydon, A. G.** Dissociation Energies and Spectra of Diatomic Molecules. 2nd Edn. (Publication received), 332.
- *Gerritsma, K. W., et al.** Detmng. theobromine in cocoa residues, 201.
- Gilbard, J. F. H.** Obituary, 264.
- Gilman, H.** Organic Chemistry. Advanced Treatise. Vols. III and IV. (Publication received), 568.
- *Glastonbury, H. A.** Apparatus for dead-stop end-point titrations with acoustic indication of end-point, 682.
- Goodwin, T. W.** Comparative Biochemistry of Carotenoids. (Publication received), 71.
- Gore, W. L.** Statistical Methods for Chemical Experimentation. (Publication received), 131.
- *Gregory, J. N., et al.** Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method, 414.
- *Grigg, J. L.** Rapid detmn. of molybdenum in soils, 470.
- *Grindley, D. N., et al.** Modified procedure for quantitative bromine absorption of oils and fats, 619.
- *Gross, D., et al.** Large-scale chromatographic separation of sucrose-raffinose mixtures on powdered cellulose for detmng. raffinose in raw sugars, 191.
- *Grossman, S., et al.** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers, 92.
- *Grünpeter, A., et al.** Modification of van Gulik detmn. of fat in soft cheese, 621.
- H**
- *Haider, S. Z.** Detmng. borate in presence of silver, 673.
- *Hale, E. E., et al.** Identifying alloys and stainless steels by electrographic methods, 145.
- *Hales, J. L., et al.** Detmng. beta- and gamma-picolines, 2:6-lutidine and 2-ethylpyridine in mixtures by infra-red spectroscopy, 114.
- *Hall, A., et al.** Detmng. calcium in plants and soils, 106.
- Hamence, J. H.** Appointment as Official Agricultural Analyst to County of Wiltshire, 387.
- *Hamilton, J. B., et al.** Micro-detmn. of alkoxyl values in cellulose ethers, 551; Erratum, 616.
- Hammett, L. P.** Introduction to Study of Physical Chemistry. (Publication received), 131; (Review), 505.
- *Hands, G. C., et al.** Precipitate error in detmng. sugar polarisations, 334.
- *Harris, F. J. T.** Colorimetric detmn. of fructose and sorbose, 287.
- Harris, M. M., et al.** Organic Chemistry. (Publication received), 71.
- Harris, T.** Appointment as Public Analyst and Official Agricultural Analyst to County Borough of Stockport, 323; as Public Analyst and Official Agricultural Analyst to County Borough of Wallasey, 387; as Public Analyst to Borough of Crewe and Borough of Glossop, 444; as Public Analyst to County Borough of Birkenhead, Borough of Crosby and Borough of Swinton and Pendlebury, and as Official Agricultural Analyst to County Borough of Birkenhead, 626.
- *Harrison, G. E., et al.** Detmng. microgram amounts of calcium, 528.
- *Hart, H. V., et al.** Separating rodent hairs and insect fragments from oat products, 439.
- *Harvey, D. G., et al.** Detmng. *p*-nitrophenol in urine and blood by indophenol reaction, 63.
- Harvey, E. H., et al.** Elements of Food Engineering. Vol. I. (Publication received), 332.
- *Harvey, H. W.** Micro-detmn. of phosphorus in biological material, 110.
- *Haslam, J., et al.** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers, 92.
- *—** Detmng. amount and composition of free phenols in phenol-formaldehyde and cresol-formaldehyde resins and moulding powders, 340.
- *—** Detmng. arsenic by B. S. Evans's method, with observations on separating arsenic and antimony, 390.
- *Hassall, C. H., et al.** Colorimetric detmn. of cardiac glycosides, 126.
- *Heighton, A. M., et al.** Separating rodent hairs and insect fragments from oat products, 439.
- Herd, M.** Appointment as Public Analyst to City of Glasgow, 67.
- *—** Paper-strip method of examining fuel oils suspected of being identical, 383.
- *Herington, E. F. G.** Inorganic complexes in colour reactions for organic compounds. I. Detmng. isonicotinic acid, 174.
- *Hermon, S. E., et al.** Separating small amounts of zirconium with mandelic acid, 256.
- Hewitt, E. J.** Sand and Water Culture Methods used in Study of Plant Nutrition. (Publication received), 71; (Review), 329.
- *Heyns, W. K., et al.** Detmng. ammonia in presence of hydrazine, 177.
- Hicks, D. V.** See Wilson, D. V.
- Hillebrand, W. F., et al.** Applied Inorganic Analysis. With Special Reference to Analysis of Metals, Minerals and Rocks. 2nd Edn. Revised by Lundell et al. (Publication received), 448.
- Hoffman, J. I., et al.** Revisers of Hillebrand and Lundell's *Applied Inorganic Analysis. With Special Reference to Analysis of Metals, Minerals and Rocks.* 2nd Edn. (Publication received), 448.
- *Holden, M.** Comparison of applicability to plant extracts of three methods of detmng. deoxyribonucleic acid, 542.
- *Hollenberg, W. C. J., et al.** Adsorption of thiamine on glassware, 730.
- Holness, H.** Inorganic Qualitative Analysis. Semi-Micro Apparatus and Technique. (Publication received), 388.
- *— et al.** Scheme of semi-micro qualitative analysis for 39 elements, 356.
- *Houlihan, J. E., et al.** Thiocyanate detmn. of iron with hydrogen peroxide, 559.
- House, C. J.** Appointment as Public Analyst to County Borough of Bury, 387; as Official Agricultural Analyst, 445.
- Hughes, W. C.** Obituary, 685.

- ***Hunter, J. G., et al.** Detmng. calcium in plants and soils, 106.
 ***Hutt, H. H., et al.** Detmng. total phosphatide in commercial lecithin, 712.

I

- Ingold, C. K.** Structure and Mechanism in Organic Chemistry. (Publication received), 684.
 ***Irving, H. M., et al.** Reversion method for absorptiometric detmn. of traces of lead with dithizone, 571.
 ***Issa, I. M., et al.** Potentiometric detmn. of quadrivalent tellurium by potassium permanganate in weakly alkaline solutions, 487.

J

- ***Jackson, C.** Detmng. potassium and traces of sodium in potassium salts, 599.
Jackson, L. C. Translator of Ketelaar's *Chemical Constitution. Introduction to Theory of Chemical Bond*. 1st English Edn. (Publication received), 448.
 ***Jackson, W. H.** Guard valve for tin detmns. by iodimetric procedure, 443.
Jacobs, M. B., et al. Chemical Analysis. Vol. VII. Chemical Analysis of Industrial Solvents. (Publication received), 332.
Jacobson, C. A. Encyclopedia of Chemical Reactions. Vol. V. (Publication received), 388.
James, G. V. Appointment as Additional Public Analyst to County Borough of Newport, County of Monmouthshire, and Urban District of Pontypool, 67; as Deputy Official Agricultural Analyst to County Borough of Newport, 67; as Deputy Official Agricultural Analyst to County of Monmouth, 257.
 ***Jefferies, J. P., et al.** Detmng. ergosterol in yeast. I. Ultra-violet absorption of purified ergosterol, 509; II. Detmn. by saponification and ultra-violet spectroscopy, 514; III. Corrections for irrelevant absorption in solutions of ergosterol, 519; IV. Short method based on ultra-violet absorption, 524.
 ***Jewsbury, A.** Detmng. copper with sodium diethyldithiocarbamate in presence of nickel and other interfering elements, with particular reference to traces of copper in sodium hydroxide, 363.
 ***Jones, A., et al.** Plate-assay technique for biotin, nicotinic acid and pantothenic acid, 15.
Jordan, L. A. Review of Rangaswami and Sen's *Handbook of Shellac Analysis*. 2nd Edn., 130.

K

- ***Kent-Jones, D. W., et al.** Detmng. benzoyl peroxide in flour and bread, 467.
 ***Kershaw, F. G., et al.** Separating small amounts of arsenic, copper and bismuth from lead and zinc by diethylammonium diethyldithiocarbamate, 624.
Ketelaar, J. A. A. Chemical Constitution. Introduction to Theory of Chemical Bond. 1st English Edn., trans. by L. C. Jackson. (Publication received), 448.
 ***Khundkar, M. H., et al.** Modified procedure for dissolving chromite ores, 623.
 ***King, H. K., et al.** Simultaneous detmn. of pentose and hexose in sugar mixtures, 80.

- King, J.** Review of Tomiček's *Chemical Indicators*, 331.
Kirk, P. L. Crime Investigation. Physical Evidence and the Police Laboratory. (Publication received), 260.
 ***Kirkpatrick, H. F. W.** Detmng. iodine in blood serum, 348.
Kleinberg, J., et al. Non-Aqueous Solvents. Applications as Media for Chemical Reactions. (Publication received), 448.
 ***Klendshoj, N. C., et al.** Analysis of general unknowns in toxicology, 43.
 ***Knight, R. A., et al.** Detmng. benzoyl peroxide in flour and bread, 467.
 ***Knowles, G., et al.** Detcng. end-point in titrating iodine with thiosulphate, 159.
 ***Koers, J., et al.** Detmng. theobromine in cocoa residues, 201.
 ***Kolthoff, I. M., et al.** Amperometric titration of traces of ammonia with hypobromite at rotated platinum wire electrode, application to detmn. of nitrogen in organic compounds, 405.
 — Polarography. Vols. I and II. 2nd Edn. (Publication received), 71; (Review), 327.
 — Textbook of Quantitative Inorganic Analysis. 3rd Edn. (Publication received), 188.

L

- ***Lawford, D. J., et al.** Detmng. *p*-nitrophenol in urine and blood by indophenol reaction, 63.
 ***Lawrence, K. R., et al.** Scheme of semi-micro qualitative analysis for 39 elements, 356.
 ***Lazarus, W., et al.** Volumetric routine detmn. of glycerol, 630.
Lederer, M. Progr's Récents de la Chromatographie. II. Chimie Minérale. (Publication received), 188.
Lees, A. Obituary of J. R. Stubbs, 570.
LeRosen, A. L., et al. Qualitative Analysis and Analytical Chemical Separations. (Publication received), 332.
LeStrange, R., et al. Paper Chromatography. Laboratory Manual. (Review), 388.
 ***Lewis, J. A., et al.** Detmng. gold in solution by adsorption extraction and spectrography, 385.
 ***Liddell, H. F.** Colorimetric detmn. of small amounts of fluorine, 494.
 ***Liebmann, H., et al.** Detmng. aluminium and zinc after chromatographic separation from tin-lead alloys, 117.
 ***Lincoln, P. A., et al.** Analysis of acid chlorides, 675.
Lingane, J. J., et al. Polarography. Vols. I and II. 2nd Edn. (Publication received), 71; (Review), 327.
 ***Linke, K., et al.** Analysis of rosin size, 670.
 ***Lippman, A. E., et al.** Colorimetric detmn. of cardiac glycosides, 126.
 ***Longstaff, J. V. L., et al.** Bromometric detmn. of formic acid and nitrite, 491.
 ***Love, R. M.** Qualitative test for monosaccharides, 732.
 ***Loveday, S. F., et al.** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers, 92.
 ***Lowden, G. F., et al.** Detcng. end-point in titrating iodine with thiosulphate, 159.
 ***Lowy, S. L., et al.** Separation of zinc and cadmium by activated copper, 299.
Lundell, G. E. F., et al. Applied Inorganic Analysis. With Special Reference to Analysis of Metals, Minerals and Rocks. 2nd Edn. Revised by Lundell et al. (Publication received), 448.

- Luniak, B.** Identification of Textile Fibres. Qualitative and Quantitative Analysis of Fibre Blends. (Publication received), 740.
- ***Lutwak, H. K.** Photometric detmn. of phosphorus in copper-based alloys containing tin, 661.

M

- ***Ma, T. S., et al.** Isopiestic method for micro-detmn. of molecular weights, 722.
- ***McAllister, R. A.** Quantitative colour reaction for metallic palladium, 65.
- ***McCormick, H.** Continuous differential refractometer for chromatographic analysis, 562.
- McDonald, F. J., et al.** Revisers of *Richmond's Dairy Chemistry*. 5th Edn. (Publication received), 448.
- ***McMullen, M. J.** Detcng. boron in treated timber, 442.
- McOmie, J. F. W., et al.** Chromatographic Methods of Inorganic Analysis: with Special Reference to Paper Chromatography. (Publication received), 568.
- ***McPhillips, J., et al.** Indirect polarographic detmn. of calcium by chloranilic acid, 666.
- ***Madley, D. G., et al.** Apparatus for micro-analysis of gas samples, 122.
- Mallinder, R.** Appointment as Public Analyst to County of West Riding of Yorkshire, 67; as Official Agricultural Analyst, 257.
- Mann, F. G., et al.** Practical Organic Chemistry. 3rd Edn. (Publication received), 71.
- ***Mapper, D., et al.** Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method, 414.
- ***Mayer, A., et al.** Volumetric detmn. of zinc with ferrocyanide in magnesium alloys, 367.
- ***Mayne, J. E. O., et al.** Detmng. zinc by sodium diethyldithiocarbamate, 625.
- Melville, H. W.** Reviser of Findlay's *Introduction to Physical Chemistry*. 3rd Edn. (Publication received), 260.
- ***Mendelowitz, A., et al.** Spectrophotometric detmn. of long-chain fatty acids containing ketonic groups, with particular reference to licanic acid, 704.
- ***Middleton, G., et al.** Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures, 532.
- ***Miller, C. C.** Anhydrous calcium oxalate as weighing form for calcium, 186.
- *— **et al.** Micro-analysis of silicate rocks. II. Pptn. of silica as 2:4-dimethylquinoline silicomolybdate and gravimetric detmn. as silicomolybdic anhydride, 24; IV. Detmng. alumina, 686.
- ***Mills, E. C., et al.** Separating small amounts of zirconium with mandelic acid, 256.
- Miner, C. S., et al.** Glycerol. (Publication received), 568.
- Minor, R. G.** Appointment as Deputy Public Analyst to Metropolitan Borough of Hammsmith, 67.
- ***Mitra, S. N.** Characterisation of vinegar by albuminoid ammonia value, 499.
- *— **et al.** Albuminoid ammonia value in analysis of fruit juices, squashes and cordials, 681.
- Moeller, T.** Inorganic Chemistry. Advanced Textbook. (Publication received), 72; (Review), 447.
- ***Monk, P. R.** Survey of development of electrographic analysis, with special reference to recent British apparatus, 141.
- ***Morgan, R. H.** Ascorbic acid content of preserved lemon juice, 323.
- ***Morren, L., et al.** Amperometric titration of traces of ammonia with hypobromite at rotated platinum wire electrode, application to detmn. of nitrogen in organic compounds, 405.
- ***Morries, P., et al.** Chromatographic detmn. of glutamic acid in wheat gluten and gluten hydrolysates, 636.
- ***Morris, S., et al.** Plate-assay technique for biotin, nicotinic acid and pantothenic acid, 15.
- ***Morton, J. E., et al.** Isopiestic method for micro-detmn. of molecular weights, 722.
- ***Morton, R. A., et al.** Spectroscopic properties of vitamin A₂. Application to assay of cod-liver oil, 74.
- ***Mossel, D. A. A., et al.** Detcng. preservatives in beverages by fermentation test, with special reference to brominated compounds, 37.
- Myers, J.** Obituary, 189.

N

- ***Newlands, G., et al.** Detmng. amount and composition of free phenols in phenol-formaldehyde and cresol-formaldehyde resins and moulding powders, 340.
- Newton, L. O.** Obituary, 74, 264.
- Nicholas, D. J. D.** Chemical Tissue Tests for Detmng. Mineral Status of Plants in the Field. (Publication received), 628.
- Nicholls, J. R.** Address of retiring President, 271.
- Aids to Analysis of Food and Drugs. 7th Edn. (Publication received), 72; (Review), 328.
- Obituary of Sir Jack Drummond, 3.
- Review of Whistler and Smart's *Polysaccharide Chemistry*, 740.
- ***Noordhof, G. H., et al.** Detmng. zinc by sodium diethyldithiocarbamate, 625.

O

- Okell, F. L.** Review of *British Pharmacopoeia* 1953, 627.
- Review of Nicholls' *Aids to Analysis of Food and Drugs*. 7th Edn., 328.
- Orchin, M., et al.** Ultraviolet Spectra of Aromatic Compounds. (Review), 69.
- ***Oshorn, G. H.** Bibliography on analytical applications of ion-exchange resins, 221.
- *— Detmng. free chlorine and combined chlorine in boron trichloride, 65.
- *— Ion-exchange resins in analytical chemistry. Application of ion-exchange resins to analysis of insoluble substances, 220.

P

- ***Palmer, J. F., et al.** Apparatus for automatic control of cathode potential in electro-analysis, 428.
- Parker, M. E., et al.** Elements of Food Engineering. Vol. I. (Publication received), 332.
- Parkes, A. E.** Appointment as Public Analyst to Metropolitan Borough of Stepney, 323.
- Parkes, H. A.** Appointment as Deputy Public Analyst to Metropolitan Borough of Bethnal Green, 67; as Additional Public Analyst to Metropolitan Borough of Stepney, 323.

- Partington, J. R.** Advanced Treatise on Physical Chemistry. Vol. III. Properties of Solids. (Publication received), 71; (Review), 331.
- ***Perkins, M., et al.** Polarographic detmn. of iron and chromium, 480.
- Pesetz, M., et al.** Méthodes et Réactions de l'Analyse Organique. Vol. I. (Review), 70; Vol. II. (Publication received), 260; (Review), 567.
- Peters, F. N., et al.** Furans. (Publication received), 684.
- ***Phillips, C. S. G., et al.** Construction of polarograph capillaries from Pyrex glass rod, 564.
- Phillips, R. F., et al.** Textbook of Pure and Applied Chemistry. (Publication received), 332.
- ***Pickles, D., et al.** Detmng. zinc in lubricating oils by amperometric titration. I. Amperometric titration of zinc with versene, 304; Erratum, 439.
- Pigott, E. C.** Ferrous Analysis. Modern Practice and Theory. 2nd Edn. (Publication received), 332.
- Pike, E. R.** Appointment as Deputy Official Agricultural Analyst to County Borough of Leicester, 257.
- ***Pinsky, A., et al.** Modification of van Gulik detmn. of fat in soft cheese, 621.
- Poirier, P., et al.** Méthodes et Réactions de l'Analyse Organique. Vol. I. (Review), 70; Vol. II. (Publication received), 260; (Review), 567.
- Pollard, F. H., et al.** Chromatographic Methods of Inorganic Analysis: with Special Reference to Paper Chromatography. (Publication received), 568.
- ***Porteous, J. W., et al.** Identifying and detmng. lower straight-chain fatty acids by paper partition chromatography, 641.
- Priestman, J., et al.** Aids to Qualitative Pharmaceutical Analysis. (Publication received), 684.
- ***Primavesi, G.** Detmng. isobutyraldehyde in *n*-butyraldehyde, 647.
- ***Prince, R. H.** Apparatus for simplifying titration in controlled atmosphere. Application to detmng. moisture in transformer oil with Fischer reagent, 607.
- ***Pritchard, H.** Selection of methods for routine assays for members of vitamin-B complex, 460.
- ***Pugh, W., et al.** Detmng. ammonia in presence of hydrazine, 177.
- Pullman, A., et al.** Théories Électroniques de la Chimie Organique. (Review), 329.
- Pullman, B., et al.** Théories Électroniques de la Chimie Organique. (Review), 329.
- ***Purser, B. J.** Detmng. acetylene in air, 732.
- Q**
- ***Quadir, S. J., et al.** Modified procedure for dissolving chromite ores, 623.
- Quenouille, M. H.** Associated Measurements. (Publication received), 71; (Review), 130.
- Design and Analysis of Experiment. (Publication received), 332.
- R**
- Radley, J. A.** Starch and its Derivatives. Vol. I. 3rd Edn. (Publication received), 332.
- Rakshit, P. C., et al.** Organic Chemistry. 7th Edn. (Publication received), 628.
- Rangaswami, M., et al.** Handbook of Shellac Analysis. 2nd Edn. Revised by Bhattacharya and Bose. (Publication received), 72; (Review), 130.
- ***Rây, H. N.** Detmng. molybdenum by ammonium thiosulphate and sodium hypophosphite, 217; Erratum, 313.
- ***Raymond, W. H. A., et al.** Detmng. microgram amounts of calcium, 528.
- Rayner, A.** Obituary, 264, 265.
- Reeves, H. G.** Obituary, 2, 264, 265.
- Reynolds, C. V.** Appointment as Deputy Official Agricultural Analyst to County Borough of Plymouth, 67.
- ***Reynolds, G. F., et al.** Polarographic detmn. of iron and chromium, 480.
- *— Randles-type cathode-ray polarograph, 314.
- *— Research polarograph for photographic recording and a multi-purpose polarographic cell, 373.
- ***Richardson, M. R., et al.** Critical examination of ferrocyanide detmn. of zinc, 291.
- ***Riley, J. P., et al.** Spectrophotometric detmn. of long-chain fatty acids containing ketonic groups, with particular reference to licanic acid, 704.
- ***Ripley-Duggan, B. A.** Detmng. small quantities of boron, 183.
- Robinson, F. A.** Review of *Biochemical Preparations*. Vol. II, 188.
- Review of Trease's *Textbook of Pharmacognosy*. 6th Edn., 628.
- Rodd, E. H.** Chemistry of Carbon Compounds. Vol. II, Part A. Alicyclic Compounds. (Publication received), 568.
- ***Rogina, B., et al.** Micro-detmn. of iodides by arresting catalytic reduction of ceric ions, 594.
- ***Roman, W., et al.** Colorimetric detmn. of indene, 679.
- Rooksby, H. P.** Review of Smith's *Visual Lines for Spectroscopic Analysis*, 567.
- ***Rose, J. E., et al.** Prepng. material rich in cation suitable for detcng. cadmium, 729.
- ***Roy, S. C., et al.** Albuminoid ammonia value in analysis of fruit juices, squashes and cordials, 681.
- S**
- ***Sachs, G.** Feigl's micro-test for mercury in organic substances, 185.
- ***Salt, H. B.** Micro-analytical methods for proteins in blood plasma. Critical review, 4.
- Samuelson, O.** Ion Exchangers in Analytical Chemistry. (Publication received), 684.
- Sandell, E. B., et al.** Textbook of Quantitative Inorganic Analysis. 3rd Edn. (Publication received), 188.
- Sarkar, P. B., et al.** Organic Chemistry. 7th Edn. (Publication received), 628.
- Saunders, B. C., et al.** Practical Organic Chemistry. 3rd Edn. (Publication received), 71.
- ***Scandrett, F. J.** Modified all-glass apparatus for detmng. nitrogen by micro-Kjeldahl method, 734.
- Scheffan, L., et al.** Chemical Analysis. Vol. VII. Chemical Analysis of Industrial Solvents. (Publication received), 332.
- ***Schnurmann, R., et al.** Ultra-violet spectrophotometric estmn. of quality of mineral oils extracted from bread, 695.
- Sen, H. K., et al.** Handbook of Shellac Analysis. 2nd Edn. Revised by Bhattacharya and Bose. (Publication received), 72; (Review), 130.

- ***Serin, P. A., et al.** Detmng. gold in solution by adsorption extraction and spectrography, 385.
- ***Seyfang, A. P., et al.** Detmng. uranium-235 in mixtures of naturally occurring uranium isotopes by radioactivation, 394.
- ***Shaw, W. H. C.** Colorimetric detmn. of 3:5-diiodothyronine, 253.
- *— **et al.** Detmng. ergosterol in yeast. I. Ultra-violet absorption of purified ergosterol, 509; II. Detmn. by saponification and ultra-violet spectroscopy, 514; III. Corrections for irrelevant absorption in solutions of ergosterol, 519; IV. Short method based on ultra-violet absorption, 524.
- Sherratt, J. G.** Appointment as Public Analyst and Official Agricultural Analyst to County Borough of St. Helens, 257; as Official Agricultural Analyst to County Borough of Blackpool, 445; as Public Analyst, 626.
- ***Silverman, L., et al.** Detmng. small amounts of potassium, calcium and magnesium in sodium and its compounds, 717.
- Simmonds, S., et al.** General Biochemistry. (Publication received), 568.
- ***Singer, K., et al.** Bromometric detmn. of formic acid and nitrite, 491.
- ***Skellon, J. H., et al.** Detmng. carbonyl compounds by semicarbazide and hydroxylamine with special reference to fatty-acid oxidation products, 135.
- Slater, R. H.** Obituary, 134, 264, 265.
- Smales, A. A.** Review of Inst. of Petroleum's *Mass Spectrometry*, 70.
- *— **et al.** Detmng. uranium-235 in mixtures of naturally occurring uranium isotopes by radioactivation, 394.
- Smart, C. L., et al.** Polysaccharide Chemistry. (Publication received), 332; (Review), 740.
- ***Smith, D. C., et al.** Reaction between periodic acid and polyhydroxy compounds, with particular reference to colorimetric detmn. of formaldehyde with chromotropic acid, 209.
- Smith, D. M.** Visual Lines for Spectroscopic Analysis. 2nd Edn. (Publication received), 188; (Review), 567.
- Smith, G. F., et al.** Quantitative Analysis. Elementary Principles and Practice. (Publication received), 260.
- ***Smith, M., et al.** Colorimetric detmn. of indene, 679.
- Smith, O. M., et al.** Semimicro Qualitative Analysis. 3rd Edn. (Publication received), 72; (Review), 447.
- Smuts, I. J., et al.** Agronomy Experiments, 1927-46, at Kroonstad Agricultural Research Station. (Publication received), 506.
- Fertilizer Experiments, 1927-46, at Kroonstad Agricultural Research Station. (Publication received), 506.
- Spalding, R. C.** Appointment as Deputy Official Agricultural Analyst to County of Kent, 257; as Deputy Public Analyst, 323; as Deputy Public Analyst to Borough of Beckenham, Borough of Bexley, Borough of Bromley, Borough of Chatham, Borough of Dartford, Borough of Tunbridge Wells, and Urban District of Chislehurst and Sidcup, 387; as Deputy Public Analyst to Borough of Erith, Borough of Gillingham and Urban District of Orpington, 444.
- ***Squirrell, D. C. M., et al.** Detmng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers, 92.
- Stateler, E. S., et al.** Elements of Food Engineering. (Publication received), 332.
- ***Steinbergs, A.** Rapid turbidimetric detmn. of small amounts of sulphur in plant material, 47.
- Stewart-Remington, J., et al.** Composition and Assaying of Minerals. (Publication received), 71.
- Stokes, S. C., et al.** Natural Rubber Latex and its Applications. No. 2. Latex Casting. (Publication received), 71.
- ***Strachan, K. G., et al.** Detmng. carbon dioxide and sulphur dioxide in Orsat apparatus, 320.
- ***Strafford, N.** Detmng. zinc by titration with di-sodium ethylenediaminetetra-acetate, 733.
- *— **et al.** Separating small amounts of arsenic, copper and bismuth from lead and zinc by diethylammonium diethyldithiocarbamate, 624.
- ***Strickland-Constable, R. F., et al.** Apparatus for micro-analysis of gas samples, 122.
- ***Stricks, W., et al.** Amperometric titration of traces of ammonia with hypobromite at rotated platinum wire electrode, application to detmn. of nitrogen in organic compounds, 405.
- ***Strouts, C. R. N., et al.** Volumetric routine detmn. of glycerol, 630.
- Stubbs, J. R.** Obituary, 262, 570.
- ***Stuckey, R. E., et al.** Chromatographic detmn. of glutamic acid in wheat gluten and gluten hydrolysates, 636.
- *— Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures, 532.
- Swanson, H. E., et al.** Standard X-ray Diffraction Powder Patterns. Vols. I and II. (Publication received), 740.

T

- Tatge, E., et al.** Standard X-ray Diffraction Powder Patterns. Vol. I. (Publication received), 740.
- Taylor, A. J., et al.** Fertilizer Experiments in Natal, 1933-50. (Publication received), 506.
- Taylor, G.** Review of *Fertilisers. Methods of Analysis Used in O.E.F.C. Countries*, 445.
- ***Templeton, W. H., et al.** Lipase activity of cereal products, 726.
- Theobald, L. S.** Review of Moeller's *Inorganic Chemistry. Advanced Textbook*, 447.
- Thompson, F. E.** Obituary, 264, 265.
- Thorp, W.** Obituary, 333.
- Tomichek, O.** Chemical Indicators. Trans. by A. R. Weir. (Review), 331.
- ***Tompsett, S. L., et al.** Reaction between periodic acid and polyhydroxy compounds with particular reference to colorimetric detmn. of formaldehyde with chromotropic acid, 209.
- Trease, G. E.** Textbook of Pharmacognosy. 6th Edn. (Publication received), 131; (Review), 628.
- ***Trego, K., et al.** Detmng. small amounts of potassium, calcium and magnesium in sodium and its compounds, 717.
- ***Trinder, P.** Titrimetric detmn. of sodium in biological fluids, 180.
- Turner, E. E., et al.** Organic Chemistry. (Publication received), 71.

V

- van der Merwe, W., et al.** Agronomy Experiments, 1938-45, at Vaal-Hartz Agricultural Research Station. (Publication received), 506.
- van Garderen, J., et al.** Agronomy Experiments, 1927-46, at Kroonstad Agricultural Research Station. (Publication received), 506.

- Agronomy Experiments, 1938–45, at Vaal-Hartz Agricultural Research Station. (Publication received), 506.
- Fertilizer Experiments, 1927–46, at Kroonstad Agricultural Research Station. (Publication received), 506.
- Fertilizer Experiments in Natal, 1933–50. (Publication received), 506.
- Velluz, L.** Substances Naturelles de Synthèse. Préparations et Méthodes de Laboratoire. Vols. I–IV. (Publication received), 71; (Review), 446.
- Venkataraman, K.** Chemistry of Synthetic Dyes. Vol. I. (Review), 131; Vol. II. (Publication received), 72.
- Vick, M. M., et al.** Qualitative Analysis and Analytical Chemical Separations. (Publication received), 332.
- Vickery, R. C.** Chemistry of Lanthanons. (Publication received), 740.
- Voelcker, E.** Appointment as Public Analyst to Metropolitan Borough of Hampstead, 257.
- Vogel, A. I.** Textbook of Quantitative Inorganic Analysis, Theory and Practice. 2nd Edn. (Review), 69.
- *— **et al.** Apparatus for automatic control of cathode potential in electro-analysis, 428.

W

- Wagner, R. B., et al.** Synthetic Organic Chemistry. (Publication received), 568.
- Walby, B. J.** Review of Vogel's *Textbook of Quantitative Inorganic Analysis, Theory and Practice*. 2nd Edn., 69.
- ***Walley, G., et al.** Volumetric routine detmn. of glycerol, 630.
- Walter, F. G.** Manufacture of Compressed Yeast. 2nd Edn. (Publication received), 628.
- ***Washbrook, C. C.** Detmng. neutralisation and saponification values of used lubricants, 254.
- *— **et al.** Detmng. zinc in lubricating oils by amperometric titration. I. Amperometric titration of zinc with versene, 304; Erratum, 439.
- ***Weatherall, H., et al.** Detmng. total phosphatide in commercial lecithin, 712.
- ***Wedgwood, P., et al.** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage, 170.
- Weir, A. R.** Translator of Tomiček's *Chemical Indicators*. (Review), 331.
- West, P. W., et al.** Qualitative Analysis and Analytical Chemical Separations. (Publication received), 332.
- ***Whettem, S. M. A., et al.** Detmng. amount and composition of free phenols in phenol-formaldehyde and cresol-formaldehyde resins and moulding powders, 340.
- Whistler, R. L., et al.** Polysaccharide Chemistry. (Publication received), 332; (Review), 740.
- ***Wilkinson, N. T.** Detmng. antimony in aromatic organic compounds containing nitrogen, chlorine and antimony, 165.
- *— **et al.** Detmng. arsenic by B. S. Evans's method, with observations on separating arsenic and antimony, 390.

- Williams, K. A.** Review of Block, LeStrange and Zweig's *Paper Chromatography*, 388.
- ***Williams, R. J. P.** Systematic approach to choice of organic reagents for metal ions, 586.
- Williams, T. I.** Chemical Industry, Past and Present. (Publication received), 740.
- Wilson, C. L.** Review of Arthur and Smith's *Semi-micro Qualitative Analysis*. 3rd Edn., 447.
- Wilson, D. V.** Notice of marriage, 569.
- Wokes, F., et al.** Fluorescence of Solutions. (Publication received), 684.
- Wolff, G., et al.** Méthodes d'Analyse et de Contrôle Industriel des Matières Grasses. (Publication received), 628.
- Wolff, J. P., et al.** Méthodes d'Analyse et de Contrôle Industriel des Matières Grasses. (Publication received), 628.
- Wolstenholme, G. E. W., et al.** Ciba Foundation Colloquia on Endocrinology. Vol. II. Steroid Metabolism and Estmn. (Publication received), 72; Vol. V. Bioassay of Anterior Pituitary and Adrenocortical Hormones. (Publication received), 448.
- ***Wood, A. A. R.** Absorptiometric detmn. of chromium in steels and alloys, 54.
- ***Wood, E. C.** Efficient planning of microbiological assays, illustrated by assays of cobalamin, 451.
- Review of Quenouille's *Associated Measurements*, 130.
- Woodhead, J. E.** Appointment as Public Analyst to Metropolitan Borough of Wandsworth, 444.
- ***Woodward, J. A., et al.** Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method, 414.
- ***Woodward, P.** Tests for nitrite and nitrate applicable over wide concentration ranges, 727.
- ***Wooldridge, H. V., et al.** Mercury-cathode cell for electrolytic separations, 386.
- ***Wyatt, P. F.** Diethylammonium diethyldithiocarbamate for separating and detmng. small amounts of metals. I. Successive detmn. of small amounts of copper, manganese and iron in organic compounds, 656.
- *— **et al.** Separating small amounts of arsenic, copper and bismuth from lead and zinc by diethylammonium diethyldithiocarbamate, 624.
- Wyckoff, R. W. G.** Crystal Structures. Vol. III, with Suppl. II. (Publication received), 568.

Y

- Yardley, J. T., et al.** Phenylfluorone, Reagent for Germanium. (Publication received), 388.
- ***Young, R. S., et al.** Detmng. carbon dioxide and sulphur dioxide in Orsat apparatus, 320.

Z

- Zook, H. D., et al.** Synthetic Organic Chemistry. (Publication received), 568.
- Zweig, G., et al.** Paper Chromatography. Laboratory Manual. (Review), 388.

INDEX TO SUBJECTS

* Denotes original papers and notes that have been published in *The Analyst*.

A

Abstracts: Analytical ——. New journal. Society of Public Analysts and Other Analytical Chemists. Editorial, 629.

***Acenaphthene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.

***Acetaldehyde:** *p*-Hydroxydiphenyl test for ——. Detmng. alanine and threonine in protein hydrolysates. Folkes, 496.

***Acetates:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Acetic acid:** Identifying and detmng. lower straight-chain fatty acids by paper partition chromatography. Duncan and Porteous, 641.

***Acetic anhydride:** Detmng. keten and — in atmosphere. Diggle and Gage, 473.

***Acetone:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Acetylene:** Detmng. — in air. Purser, 732.

***Acid(s):** chlorides: Analysis of ——. Chinnick and Lincoln, 675.

*aliphatic: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*fatty: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*fatty: Identifying and detmng. lower straight-chain fatty — by paper partition chromatography. Duncan and Porteous, 641.

*fruit: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

organic: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Spectrophotometric detmn. of long-chain fatty — containing ketonic groups with particular reference to licanic acid. Mendelowitz and Riley, 704.

***Acrylate polymers:** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.

***Actinium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Adenosine polyphosphates:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Adrenaline:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Agene:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Agglutinating Value: Prepng. Standard Sand as Used in British Standard Method of Detmng. — of Coal (B.S. 705:1936). D.S.I.R. Fuel Research Technical Paper No. 56. (Publication received), 71.

***Aglycones:** Colorimetric detmn. of cardiac glycosides. Hassall and Lippman, 126.

***Agriculture:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

See also **Ministry of Agriculture and Fisheries.**

Agronomy Experiments, 1927-46, at Kroonstad Agricultural Research Station. Smuts and van Garderen. (Publication received), 506.

Experiments, 1938-45, at Vaal-Hartz Agricultural Research Station. van der Merwe and van Garderen. (Publication received), 506.

***Air:** Detmng. acetylene in ——. Purser, 732.

*Detmng. keten and acetic anhydride in atmosphere. Diggle and Gage, 473.

***Alanine:** *p*-Hydroxydiphenyl test for acetaldehyde. Detmng. — and threonine in protein hydrolysates. Folkes, 496.

***Albumin:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Micro-analytical methods for proteins in blood plasma. Critical review. Salt, 4.

***Albuminoid ammonia** value: Characterising vinegar by —. Mitra, 499.

*value in analysis of fruit juices, squashes and cordials. Mitra and Roy, 681.

***Alcohols:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Aldehydes:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Detmng. carbonyl compounds by semicarbazide and hydroxylamine, with special reference to fatty-acid oxidation products. Feuell and Skellon, 135.

***Algae:** Micro-detmn. of phosphorus in biological material. Harvey, 110.

***Alkali metals:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.

***Alkaline earths:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.

***Alkaloids:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Alkoxyl values:** Micro-detmn. of — in cellulose ethers. Easterbrook and Hamilton, 551; Erratum, 616.

***Alloys:** Absorptiometric detmn. of chromium in steels and —. Wood, 54.

*Detmng. aluminium and zinc after chromatographic separation from tin-lead —. Bishop and Liebmann, 117.

*Identifying — and stainless steels by electrographic methods. Clark and Hale, 145.

*Opening of white metals for analysis by dry chlorine. Bishop, 61.

*Photometric detmn. of phosphorus in copper-based — containing tin. Lutwak, 661.

*Qualitative detcn. of tellurium in tellurium-lead —. Brown, 623.

*Quantitative separation of copper, lead and tin by cathodic deposition. Aylward and Bryson, 651.

*Survey of development of electrographic analysis. Monk, 141.

*Volumetric detmn. of zinc with ferrocyanide in magnesium —. Mayer, Bradshaw and Deutschman, 367.

***Alumina:** Micro-analysis of silicate rocks. IV. Detmng. —. Miller and Chalmers, 686.

***Aluminium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Detmng. — and zinc after chromatographic separation from tin-lead alloys. Bishop and Liebmann, 117.

Aluminium—continued

- *Detmng. magnesium and — in titanium metal. Corbett, 20.
- *Micro-analysis of silicate rocks. IV. Detmng. alumina. Miller and Chalmers, 686.
- *Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Survey of development of electrographic analysis. Monk, 141.
- ***Amino-acids:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Microbiological Assay of Vitamin B Complex and —. Barton-Wright. (Publication received), 71; (Review), 129.
- p-Aminobenzoic acid:** Approved name unchanged. International Union of Pure and Applied Chemistry, 72.
- *Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.
- ***5-Amino-caproic acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Ammonia:** Albuminoid — value in analysis of fruit juices, squashes and cordials. Mitra and Roy, 681.
- *Amperometric titration of traces of — with hypobromite at rotated platinum wire electrode. Application to detmn. of nitrogen in organic compounds. Kolthoff, Stricks and Morren, 405.
- *Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Decomposition of — in sealed-tube micro-Kjeldahl digestions with selenium catalyst. Baker, 500.
- *Detmng. — in presence of hydrazine. Pugh and Heyns, 177.
- *value: Characterising vinegar by albuminoid —. Mitra, 499.
- ***Ammonium thiosulphate:** Detmng. molybdenum by — and sodium hypophosphite. Rây, 217; Erratum, 313.

Ampoules. B.S. 795:1953, 326.

***Anaesthetics:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Analysis: Aids to — of Food and Drugs. Nicholls. 7th Edn. (Publication received), 72; (Review), 328.

Aids to Qualitative Pharmaceutical —. Priestman and Edwards. (Publication received), 684.

Applied Inorganic —, with Special Reference to — of Metals, Minerals and Rocks. Hillebrand and Lundell. 2nd Edn. (Publication received), 448.

*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Chemical —. Vol. VII. Chemical — of Industrial Solvents. Jacobs and Scheffan. (Publication received), 332.

Chromatographic Methods of Inorganic —, with Special Reference to Paper Chromatography. Pollard and McOmie. (Publication received), 568.

Ferrous —. Modern Practice and Theory. Pigott. 2nd Edn. (Publication received), 332.

Fertilisers. Methods of — used by O.E.E.C. Countries. Organisation for European Economic Co-operation. (Publication received), 188; (Review), 445.

Analysis—continued

Handbook of Colorimetric Chemical Analytical Methods for Industrial, Research and Clinical Laboratories. Developed for Use with Lovibond Comparator. Tintometer, Ltd. (Publication received), 332; (Review), 568.

Handbook of Shellac —. Rangaswami and Sen. Revised by Bhattacharya and Bose. 2nd Edn. (Publication received), 72; (Review), 130.

*Identification of Textile Fibres. Qualitative and Quantitative — of Fibre Blends. Luniak. (Publication received), 740.

Inorganic Qualitative —. Semi-micro Apparatus and Technique. Holness. (Publication received), 388.

Inorganic Thermogravimetric —. Duval. (Publication received), 188; (Review), 738.

*Ion-exchange resins in analytical chemistry. Applications of ion-exchange resins in — of insoluble substances. Osborn, 220.

Ion Exchangers in Analytical Chemistry. Samuelson. (Publication received), 684.

Méthodes d'Analyse et de Contrôle Industriel des Matières Grasses. Wolff and Wolff. (Publication received), 628.

Méthodes et Réactions de l'Analyse Organique. Vol. I. Pesetz and Poirier. (Review), 70; Vol. II. (Publication received), 260; (Review), 567.

Mises au Point de Chimie Analytique Pure et Appliquée et d'Analyse Bromatologique. Gautier. (Publication received), 740.

*Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.

Qualitative — and Analytical Chemical Separations. West, Vick and LeRosen. (Publication received), 332.

Quantitative —. Elementary Principles and Practice. Diehl and Smith. (Publication received), 260.

Reference substances for use in organic micro—. Society of Public Analysts and Other Analytical Chemists, Microchemistry Group, 258.

*Scheme of semi-micro qualitative — for 39 elements. Holness and Lawrence, 356.

Semimicro Qualitative —. Arthur and Smith. 3rd Edn. (Publication received), 72; (Review), 447.

*Systematic approach to choice of organic reagents for metal ions. Williams, 586.

Textbook of Quantitative Inorganic —. Kolthoff and Sandell. 3rd Edn. (Publication received), 188.

Textbook of Quantitative Inorganic —, Theory and Practice. Vogel. 2nd Edn. (Review), 69.

Visual Lines for Spectroscopic —. Smith. 2nd Edn. (Publication received), 188; (Review), 567.

Analyst: Reprint from 1876 to 1951. Editorial, 73.

Analytical Abstracts. New journal. Society of Public Analysts and Other Analytical Chemists. Editorial, 629.

Analytical chemistry: Publication of Proceedings of First International Congress on —. Editorial, 133.

Aneurine: Approved name now thiamine. International Union of Pure and Applied Chemistry, 72.

***Anthanthrene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.

- ***Anthracene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- ***Antibiotics:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221. Survey of Properties and Uses. 2nd Edn. (Review), 330.
- ***Anticoagulant therapy:** Control of —. Biggs, 84.
- ***Antimony:** Detmng. — in aromatic organic compounds containing nitrogen, chlorine and antimony. Wilkinson, 165.
- *Detmng. arsenic by B. S. Evans's method, with observations on separating arsenic and —. Haslam and Wilkinson, 390.
- *Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- Antioxidants:** Ministry of Food, Food Standards Committee, Preservatives Sub-Committee, 504.
- ***Anti-viral compounds:** Evaluation of —. Dickinson, 283.
- ***Apparatus:** Adsorption of thiamine on glassware. Farrer and Hollenberg, 730.
- *Analysis of "general unknowns" in toxicology. Feldstein and Klendshoj, 43.
- *Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Continuous differential refractometer for chromatographic analysis. McCormick, 562.
- Dean and Stark —. B.S. 756:1952. 128.
- Density bottles. B.S. 733:1952. 68.
- Density hydrometers and specific gravity hydrometers. B.S. 718:1953. 445.
- *Design and operating technique of vacuum drying oven. I. Design of oven. Gardiner, 709.
- *Detcng. end-point in titrating iodine with thiosulphate. Knowles and Lowden, 159.
- *Detmng. ergosterol in yeast. IV. Short method based on ultra-violet absorption. Shaw and Jefferies, 524.
- *Detmng. iodine in blood serum. Kirkpatrick, 348.
- *Detmng. keten and acetic anhydride in atmosphere. Diggie and Gage, 473.
- *Detmng. zinc in lubricating oils by amperometric titration. I. Amperometric titration of zinc with versene. Pickles and Washbrook, 304; Erratum, 439.
- Dispensing measures for pharmaceutical purposes (imperial units). B.S. 1921:1953. 387; Dispensing measures for pharmaceutical purposes (metric units). B.S. 1922:1953. 387.
- *Double syringe-pipette for dissolved oxygen estmns. Barnes, 501.
- Flasks for detmng. distillation range. B.S. 571: 1953. 257.
- Flasks with graduated necks. B.S. 676:1953. 326.
- *for automatic control of cathode potential in electro-analysis. Palmer and Vogel, 428.
- *for dead-stop end-point titrations with acoustic indication of end-point. Glastonbury, 682.
- *for micro-analysis of gas samples. Madley and Strickland-Constable, 122.
- *for simplifying titration in controlled atmosphere. Application to detmng. moisture in transformer oil with Fischer reagent. Prince, 607.
- Glass filter funnels. B.S. 1923:1953. 326.
- Graduated pipettes and one-mark cylindrical pipettes. B.S. 700:1952. 68.
- Apparatus—continued**
- *Guard valve for tin detmns. by iodimetric procedure. Jackson, 443.
- Haemacytometer counting chambers and dilution pipettes. B.S. 748:1953. 505.
- Halogens and sulphur combustion train (micro-Grote). Part A4; Nitrogen detmn. — (micro-Kjeldahl). Part B1; Crucibles for microchemical analysis. Part E1; Combustion boats, sheath and contact stars for microchemical analysis. Part II. B.S. 1428:1953. 326.
- *Improved Randles-type cathode-ray polarograph. Reynolds and Davis, 314.
- Inorganic Qualitative Analysis. Semi-micro — and Technique. Holness. (Publication received), 388.
- *Isopiestic method for micro-detmn. of molecular weights. Morton, Campbell and Ma, 722.
- Kohlrausch flasks. B.S. 615:1953. 627.
- *Mercury-cathode cell for electrolytic separations. Aylward and Wooldridge, 386.
- *Micro-analysis of silicate rocks. III. Spectrophotometric detmn. of phosphoric oxide in presence of silica. Chalmers, 32; IV. Detmng. alumina, 686.
- *Micro-detmn. of alkoxyl values in cellulose ethers. Easterbrook and Hamilton, 551; Erratum, 616.
- *Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method. Gregory, Mapper and Woodward, 414.
- *Modified all-glass — for detmng. nitrogen by micro-Kjeldahl method. Scandrett, 734.
- *Modified dead-stop end-point circuit for titrations of ferrous iron with potassium dichromate in approximately *N* solutions. Collier and Fricker, 440.
- One-mark bulb pipettes. B.S. 1583:1950. Amendment slip, 387.
- *Opening of white metals for analysis by dry chlorine. Bishop, 61.
- Ostwald-Folin pipettes. B.S. 773:1953. 326.
- *Polarograph capillaries from Pyrex glass rod. Ferrett and Phillips, 564.
- *Research polarograph for photographic recording and multipurpose polarographic cell. Bryant and Reynolds, 373.
- Secondary reference thermometers (Centigrade scale). B.S. 1900:1952. 68; Amendment slip, 187.
- Sugar flasks. B.S. 675:1953. 326.
- *Survey of development of electrographic analysis, with special reference to recent British —. Monk, 141.
- Tests for performance characteristics of sintered filters. B.S. 1969:1953. 627.
- *Volumetric detmn. of zinc with ferrocyanide in magnesium alloys. Mayer, Bradshaw and Deutschman, 367.
- Applied Chemistry:** Reports on Progress of —. Society of Chemical Industry. Vol. XXXVII. (Publication received), 568.
- Textbook of Pure and —. Garside and Phillips. (Publication received), 332.
- Appointments,** official: 67, 257, 323, 387, 444, 445, 626.
- ***Arabinose:** Qualitative test for monosaccharides. Love, 732.
- **d*:- Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- ***Arsenic:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Arsenic—continued

- *Detmng. — by B. S. Evans's method, with observations on separating — and antimony. Haslam and Wilkinson, 390.
- *Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Separating small amounts of —, copper and bismuth from lead and zinc by means of diethylammonium diethyldithiocarbamate. Trafford, Wyatt and Kershaw, 624.
- ***4-(o-Arsonophenylazo)-N-(1-naphthyl)-ethylenediamine**, thorium salt of: Colorimetric detmn. of small amounts of fluorine. Liddell, 494.
- Ascorbic acid**: Approved name for vitamin C. International Union of Pure and Applied Chemistry, 72.
- *content of preserved lemon juice. Morgan, 323.
- *Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- ***Aspartic acid**: Chromatographic detmn. of glutamic acid in wheat gluten and gluten hydrolysates. Morris and Stuckey, 636.
- Associated Measurements**. Quenouille. (Publication received), 71; (Review), 130.
- Association of British Chemical Manufacturers**: British Chemicals and their Manufacturers. (Publication received), 188.
- ***Atmosphere**: Detmng. keten and acetic anhydride in —. Diggle and Gage, 473.
- ***1:2-Benzanthracene**: Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- ***Benzoic acid**: Detmng. benzoyl peroxide in flour and bread. Knight and Kent-Jones, 467.
- ***Benzoyl peroxide**: Detmng. — in flour and bread. Knight and Kent-Jones, 467.
- ***1:12-Benzperylene**: Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- ***3:4-Benzpyrene**: Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- ***Beryllium**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- Bethnal Green**: Appointment of H. A. Parkes as Deputy Public Analyst to Metropolitan Borough of —, 67.
- ***Beverages**: Detcng. preservatives in — by fermentation test, with special reference to brominated compounds. Mossel and de Bruin, 37.
- Bexley**: Appointment of R. C. Spalding as Deputy Public Analyst to Borough of —, 387.
- Bibliographic Guide**: Guida Bibliografica Internazionale per il Chimico, Libri e Riviste. Gaudenzi. (Publication received), 71.
- Biochemical Preparations**. Vol. II. Ball. (Publication received), 71; (Review), 183.
- Biochemistry**: General —. Fruton and Simmonds. (Publication received), 568.
- Biological Assay**: Statistical Method in —. Finney. (Publication received), 71; (Review), 505.
- *fluids: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *fluids: Titrimetric detmn. of sodium in —. Trinder, 180.
- *material: Prepng. — for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- Biotin**: Approved name unchanged. International Union of Pure and Applied Chemistry, 72.
- *Plate-assay technique for —, nicotinic acid and pantothenic acid. Morris and Jones, 15.
- *Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.
- Birkenhead**: Appointment of T. Harris as Public Analyst and Official Agricultural Analyst to County Borough of —, 626.
- Biscuits**: Labelling of Food Order, 1953. 324.
- ***Bismuth**: Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Separating small amounts of arsenic, copper and — from lead and zinc by diethylammonium diethyldithiocarbamate. Trafford, Wyatt and Kershaw, 624.
- Blackberry jam**: Food Standards (Preserves) Order, 1953. 324.
- Blackcurrant jam**: Food Standards (Preserves) Order, 1953. 324.
- Blackpool**: Appointment of J. G. Sherratt as Official Agricultural Analyst to County Borough of —, 445; as Public Analyst, 626.
- ***Blood**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

B

- ***Bacteriology**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Bacup**: Appointment of H. Dedicoat as Public Analyst to Borough of —, 444.
- Baking powder**: Fluorine content of acidic phosphates used for food purposes. Ministry of Food, Food Standards Committee, Metallic Contamination Sub-Committee, 504.
- ***Barium**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. microgram amounts of calcium. Harrison and Raymond, 528.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Systematic approach to choice of organic reagents for metal ions. Williams, 586.
- ***Barium succinate**: Ion-exchange resins in analytical chemistry. Application of ion-exchange resins to analysis of insoluble substances. Osborn, 220.
- ***Barium sulphate**: Ion-exchange resins in analytical chemistry. Application of ion-exchange resins to analysis of insoluble substances. Osborn, 220.
- ***Bases**, organic: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Beans**, runner: Sulphur dioxide in imported dehydrated vegetables. Ministry of Food, 128.
- Beckenham**: Appointment of R. C. Spalding as Deputy Public Analyst to Borough of —, 387.
- Beef sausages**: Labelling of Food Order, 1953. 324. sausages: Meat Products (No. 3) Order, 1952. 187.
- Beet**: Sugar — Cultivation. Ministry of Agriculture and Fisheries. (Publication received), 628.

Blood—continued

- *clotting: Control of anticoagulant therapy. Biggs, 84.
 - *Colorimetric micro-detmn. of potassium in serum. Baar, 353.
 - *detmng. carbon monoxide in: Quantitative colour reaction for metallic palladium. McAllister, 65.
 - *Detmng. iodine in — serum. Kirkpatrick, 348.
 - *Detmng. microgram amounts of calcium. Harrison and Raymond, 528.
 - *Detmng. *p*-nitrophenol in urine and — by indophenol reaction. Lawford and Harvey, 63.
 - *plasma: Micro-analytical methods for proteins in —. Critical review. Salt, 4.
 - *Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
 - *serum: Indirect polarographic detmn. of calcium by chloranilic acid. Breyer and McPhillips, 666.
- Bone grease:** Home-produced technical —. B.S. 1483:1948. Amendment slip, 68.

Book reviews:

- Arthur and Smith. Semimicro Qualitative Analysis. 3rd Edn., 447.
- Ball. Biochemical Preparations. Vol. II, 188.
- Barton-Wright. Microbiological Assay of Vitamin-B Complex and Amino-Acids, 129.
- Block, LeStrange and Zweig. Paper Chromatography, 388.
- Duval. Inorganic Thermogravimetric Analysis, 738.
- Elderfield. Heterocyclic Compounds. Vol. II, 259; Vols. III and IV, 260.
- Finney. Statistical Method in Biological Assay, 505.
- Friedel and Orchin. Ultraviolet Spectra of Aromatic Compounds, 69.
- Hammett. Introduction to Study of Physical Chemistry, 505.
- Hewitt. Sand and Water Culture Methods used in Study of Plant Nutrition, 329.
- Institute of Petroleum. Mass Spectrometry, 70.
- Kolthoff and Lingane. Polarography. 2nd Edn. Vols. I and II, 327.
- Moeller. Inorganic Chemistry. Advanced Text-book, 447.
- Nicholls. Aids to Analysis of Food and Drugs. 7th Edn., 328.
- O.E.E.C. Fertilisers. Methods of Analysis Used in O.E.E.C. Countries, 445.
- Partington. Advanced Treatise on Physical Chemistry. Vol. III. Properties of Solids, 331.
- Pesez and Poirier. Méthodes et Réactions de l'Analyse Organique. Vol. I, 70; Vol. II, 567.
- Pullman and Pullman. Théories Électroniques de la Chimie Organique, 329.
- Quenouille. Associated Measurements, 130.
- Rangaswami and Sen. Handbook of Shellac Analysis. 2nd Edn., 130.
- Smith. Visual Lines for Spectroscopic Analysis. 2nd Edn., 567.
- Tintometer, Ltd. Handbook of Colorimetric Chemical Analytical Methods for Industrial, Research and Clinical Laboratories, Developed for Use with Lovibond Comparator, 568.
- Tomiček. Chemical Indicators, 331.
- Trease. Textbook of Pharmacognosy. 6th Edn., 628.
- Velluz. Substances Naturelles de Synthèse; Préparations et Méthodes de Laboratoire. Vols. I-IV, 446.
- Venkataraman. Chemistry of Synthetic Dyes, Vol. I, 131.

Book Reviews—continued

- Vogel. Textbook of Quantitative Inorganic Analysis, Theory and Practice. 2nd Edn., 69.
- Whistler and Smart. Polysaccharide Chemistry, 740.
- Antibiotics. Survey of Properties and Uses. 2nd Edn., 330.
- British Pharmacopoeia 1953. 627.
- Books:** Guida Bibliografica Internazionale per il Chimico, Libri e Riviste. Gaudenzi. (Publication received), 71.
- ***Borate:** Detmng. — in presence of silver. Haider, 673.
- ***Borax:** Detcng. boron in treated timber. McMullen, 442.
- Public Health (Preservatives, etc., in Food) (Amendment) Regulations, 1953. 737.
- ***Boric acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detcng. boron in treated timber. McMullen, 442.
- *Detmng. borate in presence of silver. Haider, 673.
- *Detmng. small quantities of boron. Ripley-Duggan, 183.
- ***Borofluorides:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Ion-exchange resins in analytical chemistry. Application of ion-exchange resins to analysis of insoluble substances. Osborn, 220.
- ***Boron:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detcng. — in treated timber. McMullen, 442.
- *Detmng. small quantities of —. Ripley-Duggan, 183.
- ***Boron trichloride:** Detmng. free and combined chlorine in —. Osborn, 65.
- Bottles:** Capacity of cylindrical glass milk —. B.S. 1925:1953. 257.
- Bournemouth:** Appointment of A. S. Carlos as Public Analyst and Official Agricultural Analyst to County Borough of —, 323.
- Brains:** Offals in Meat Products Order, 1953. 187.
- Bramble jam:** Food Standards (Preserves) Order, 1953. 324.
- ***Bread:** Detmng. benzoyl peroxide in flour and —. Knight and Kent-Jones, 467.
- Order, 1953. 566.
- *Ultra-violet spectrophotometric estmn. of quality of mineral oils extracted from —. Cookson, Coppock and Schnurmann, 695.
- ***Brewing:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- British Abstracts C.** Cessation of publication. Editorial, 629.
- British Drug Houses Ltd.:** Ion Exchange Resins. (Publication received), 740.
- British Medical Association:** National Formulary 1952. First Amendment 1953. (Publication received), 568.
- British Pharmacopoeia 1953.** General Medical Council. (Publication received), 260; (Review), 627.
- British Standard Specifications:**
 - 188:1937. Method for detmng. viscosity of liquids in absolute (C.G.S.) units. Amendment slip, 567.
 - 541:1934. Detmng. Rideal-Walker coefficient of disinfectants. Amendment slip, 505.
 - 571:1953. Flasks for detmng. distillation range, 257.
 - 615:1953. Kohlrausch flasks, 627.
 - 627:1953. Sampling fats and fatty oils, 326.
 - 675:1953. Sugar flasks, 326.

British Standard Specifications—continued

- 676:1953. Flasks with graduated necks, 326.
700:1952. Graduated pipettes and one-mark cylindrical pipettes, 68.
718:1953. Density hydrometers and specific gravity hydrometers, 445.
733:1952. Density bottles, 68.
748:1953. Haemacytometer counting chambers and dilution pipettes, 505.
756:1952. Dean and Stark apparatus, 128.
773:1953. Ostwald - Folin pipettes, 326.
795:1953. Ampoules, 326.
1428:1953. Part A4, Halogens and sulphur combustion train (micro-Grote), 326; Part B1, Nitrogen detmn. apparatus (micro-Kjeldahl), 326; Part E1, Crucibles for microchemical analysis, 326; Part I1, Combustion boats, sheath and contact stars for microchemical analysis, 326.
1482:1948. Home-produced technical tallow. Amendment slip, 68.
1483:1948. Home-produced technical bone grease. Amendment slip, 68.
1583:1950. One-mark bulb pipettes. Amendment slip, 387.
1900:1952. Secondary reference thermometers (Centigrade scale), 68; Amendment slip, 187.
1910:1953. Carbolio soap, 326.
1911:1953. Genuine hard soap, 326.
1912:1953. Soap flakes, 326.
1913:1953. Soft soap, 326.
1914:1953. Toilet soap, 326.
1921:1953. Dispensing measures for pharmaceutical purposes (imperial units), 387.
1922:1953. Dispensing measures for pharmaceutical purposes (metric units), 387.
1923:1953. Glass filter funnels, 326.
1925:1953. Capacity of cylindrical glass milk bottles, 257.
1939:1953. Sunflower seed oil, 257.
1940:1953. Ethyl methyl ketone, 257.
1941:1953. *iso*Butyl methyl ketone, 257.
1969:1953. Tests for performance characteristics of sintered filters, 627.
1992:1953. Butyl acetylricinoleate, 627.
1993:1953. *sec*Butyl alcohol, 627.
1994:1953. Dichloromethane, 627.
1995:1953. Di-2-ethylhexyl phthalate, 627.
1996:1953. Dimethyl phthalate, 627.
1997:1953. Glycerol triacetate (triacetin), 627.
1998:1953. Triphenyl phosphate, 627.
1999:1953. Tritolyl phosphate (tricresyl phosphate), 627.
2005:1953. Glossary of terms applicable to fillings and stuffings, 627.
British Standards Institution: Change of address, 505.
Yearbook, 1953. 568.
***Bromide:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
***Bromine absorption:** Modified procedure for quantitative — of oils and fats. Burden and Grindley, 619.
See also **Halogens.**
Bromley: Appointment of R. C. Spalding as Deputy Public Analyst to Borough of —, 387.
***Bromoacetic esters:** Detcng. preservatives in beverages by fermentation test, with special reference to brominated compounds. Mossel and de Bruin, 37.
Burnley: Appointment of R. Blunt as Deputy Public Analyst and Deputy Official Agricultural Analyst to County Borough of —, 323.

- Bury:** Appointment of C. J. House as Public Analyst to County Borough of —, 387; as Official Agricultural Analyst, 445.
Butanol: See **Butyl alcohol.**
Butyl acetylricinoleate. B.S. 1992:1953. 627.
***Butyl alcohol:** Detmng. *isobutylaldehyde* in *n*-butylaldehyde. Primavesi, 647.
sec —. B.S. 1993:1953. 627.
Butylated hydroxyanisole: Antioxidants. Ministry of Food, Food Standards Committee, Preservatives Sub-Committee, 504.
***Butylaldehyde:** Detmng. *iso* — in *n* —. Primavesi, 647.
***Butyric acid:** Identifying and detmng. lower straight-chain fatty acids by paper partition chromatography. Duncan and Porteous, 641.

C

- Cabbage:** Sulphur dioxide in imported dehydrated vegetables. Ministry of Food, 128.
***Cadion:** Prepng. material rich in — suitable for detcng. cadmium. Fisher, Estes and Rose, 729.
***Cadmium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
*Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
*Prepng. material rich in cadion suitable for detcng. —. Fisher, Estes and Rose, 729.
*Research polarograph for photographic recording and multipurpose polarographic cell. Bryant and Reynolds, 373.
*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
*Separating zinc and — by activated copper. Bryson and Lowy, 299.
*Survey of development of electrographic analysis. Monk, 141.
*Systematic approach to choice of organic reagents for metal ions. Williams, 586.
***Caesium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
Calciferol. Approved name now ergocalciferol. International Union of Pure and Applied Chemistry, 72.
***Calcium:** Anhydrous calcium oxalate as weighing form for —. Miller, 186.
*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
*Detmng. — in plants and soils. Hunter and Hall, 106.
*Detmng. microgram amounts of —. Harrison and Raymond, 528.
*Detmng. small amounts of potassium, — and magnesium in sodium and its compounds. Silverman and Trego, 717.
*Indirect polarographic detmn. of — by chloranilic acid. Breyer and McPhillips, 666.
*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
*Systematic approach to choice of organic reagents for metal ions. Williams, 586.
***Calcium carbonate:** Ion-exchange resins in analytical chemistry. Application of ion-exchange resins to analysis of insoluble substances. Osborn, 220.
***Calcium oxalate:** Anhydrous — as weighing form for calcium. Miller, 186.

- Cancer:** Excerpta Medica. Section XVI. — (Experimental and Clinical). Vol. I, No. 1, July, 1953. (Publication received), 628.
- Canned Corned Meat (Prices) Order, 1953.** 187.
- foods: Tin in —. Ministry of Food, Food Standards Committee, Metallic Contamination Sub-Committee, 187.
- fruit: Labelling of Food Order, 1953. 324.
- vegetables: Labelling of Food Order, 1953. 324.
- ***Capillaries:** Polarograph — from Pyrex glass rod. Ferrett and Phillips, 564.
- Carbolic soap.** B.S. 1910:1953. 326.
- ***Carbon:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Carbon dioxide:** Apparatus for micro-analysis of gas samples. Madley and Strickland-Constable, 122.
- *Detmng. — and sulphur dioxide in Orsat apparatus. Young, Benfield and Strachan, 320.
- ***Carbon monoxide:** Apparatus for micro-analysis of gas samples. Madley and Strickland-Constable, 122.
- *detmng.: Quantitative colour reaction for metallic palladium. McAllister, 65.
- ***Carbonyl compounds:** Detmng. — by semicarbazide and hydroxylamine, with special reference to fatty-acid oxidation products. Feuell and Skellon, 135.
- ***Cardiac glycosides:** Colorimetric detmn. of —. Hassall and Lippman, 126.
- Carlisle:** Appointment of W. F. Elvidge as Deputy Public Analyst to County Borough of —, 387.
- Carotenoids:** Comparative Biochemistry of —. Goodwin. (Publication received), 71.
- Carrots:** Sulphur dioxide in imported dehydrated vegetables. Ministry of Food, 128.
- ***Casein:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Catalysts:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Catechol monobenzoate:** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate - ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.
- ***Cathode potential:** Apparatus for automatic control of — in electro-analysis. Palmer and Vogel, 428.
- ***Cellulose ethers:** Micro-detmn. of alkoxyl values in —. Easterbrook and Hamilton, 551; Erratum, 616.
- *Large-scale chromatographic separation of sucrose - raffinose mixtures on powdered — for detmn. of raffinose in raw sugars. Gross and Albon, 191.
- ***Cereal products:** Lipase activity of —. Templeton and Carpenter, 726.
- ***Cerium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Micro-detmn. of iodides by arresting catalytic reduction of ceric ions. Rogina and Dubravčić, 594.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- Chatham:** Appointment of J. W. Flint as Deputy Public Analyst to Borough of —, 257.
- Appointment of R. C. Spalding as Deputy Public Analyst to Borough of —, 387.
- ***Cheese:** Modification of van Gulik method for detmng. fat in soft —. Pinsky and Grün-peter, 621.
- Chemical(s) Constitution.** Introduction to Theory of the Chemical Bond. Ketelaar. 1st English Edn. translated by Jackson. (Publication received), 448.
- Industry, Past and Present. Williams. (Publication received), 740.
- British — and their Manufacturers. Assoc. of British Chemical Manufacturers. (Publication received), 188.
- Chemical Society:** Annual Reports on Progress of Chemistry for 1952. (Publication received), 628.
- Chemistry:** Advanced Treatise on Physical —. Vol. III. Properties of Solids. Partington. (Publication received), 71; (Review), 331.
- Annual Reports on Progress of — for 1952. Chemical Society. (Publication received), 628.
- General and Inorganic —. Findlay. (Publication received), 188, 332.
- Heterocyclic Compounds. Vol. II. Elderfield. (Review), 259; Vols. III and IV, 260.
- Inorganic —. Advanced Textbook. Moeller. (Publication received), 72; (Review), 447.
- Introduction to Physical —. Findlay. 3rd Edn. Revised by Melville. (Publication received), 260.
- of Carbon Compounds. Vol. II, Part A. Alicyclic Compounds. Rodd. (Publication received), 568.
- Organic —. Sarkar and Rakshit. 7th Edn. (Publication received), 628.
- Organic —. Turner and Harris. (Publication received), 71.
- Organic —. Advanced Treatise. Gilman. Vols. III and IV. (Publication received), 568.
- Polysaccharide —. Whistler and Smart. (Publication received), 332; (Review), 740.
- Practical Organic —. Mann and Saunders. 3rd Edn. (Publication received), 71.
- Reports on Progress of Applied —. Society of Chemical Industry. Vol. XXXVII. (Publication received), 568.
- Structure and Mechanism in Organic —. Ingold. (Publication received), 684.
- Synthetic Organic —. Wagner and Zook. (Publication received), 568.
- Textbook of Pure and Applied —. Garside and Phillips. (Publication received), 332.
- Théories Électroniques de la Chimie Organique. Pullman and Pullman. (Review), 329.
- Chislehurst and Sidcup:** Appointment of R. C. Spalding as Deputy Public Analyst to Urban District of —, 387.
- Chitterlings:** Offals in Meat Products Order, 1953. 187.
- ***Chloranilic acid:** Indirect polarographic detmn. of calcium by —. Breyer and McPhillips, 666.
- ***Chloride(s):** Analysis of acid —. Chinnick and Lincoln, 675.
- *Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Chlorine:** Detmng. free and combined — in boron trichloride. Osborn, 65.
- *Opening of white metals for analysis by dry —. Bishop, 61.
- See also **Halogens.**
- ***Chloromethylphenoxyacetic acids:** Detmng. — in MCPA formulations. Freeman and Gardner, 205.
- Cholecalciferol.** Approved name for vitamin D₃. International Union of Pure and Applied Chemistry, 72.
- Choline:** Approved name unchanged. International Union of Pure and Applied Chemistry, 72.

Choline—*continued*

*Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.

Chromatography: Chromatographic Methods of Inorganic Analysis, with Special Reference to Paper —. Pollard and McOmie. (Publication received), 568.

Paper —. Laboratory Manual. Block, LeStrange and Zweig. (Review), 388.

Practical —. Brimley and Barrett. (Publication received), 684.

Progrès Recents de la Chromatographie. Part 2. Chimie Minérale. Lederer. (Publication received), 188.

***Chromic acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Chromite ores:** Modified procedure for dissolving —. Khundkar and Quadir, 623.

***Chromium:** Absorptiometric detmn. of — in steels and alloys. Wood, 54.

*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Identifying alloys and stainless steels by electrographic methods. Clark and Hale, 145.

*Polarographic detmn. of iron and —. Perkins and Reynolds, 480.

*Research polarograph for photographic recording and multipurpose polarographic cell. Bryant and Reynolds, 373.

*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.

*Survey of development of electrographic analysis. Monk, 141.

*Systematic approach to choice of organic reagents for metal ions. Williams, 586.

***Chromotropic acid:** Reaction between periodic acid and polyhydroxy compounds, with particular reference to colorimetric detmn. of formaldehyde with —. Tompsett and Smith, 209.

***Chrysene:** Deteng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.

Ciba Foundation Colloquia on Endocrinology. Vol. II. Steroid Metabolism and Estmn. Wolstenholme and Cameron. (Publication received), 72; Vol. V. Bioassay of Anterior Pituitary and Adrenocortical Hormones, 448.

***Cistus:** Adulterant of dried sage. Button, 679.

***Citric acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Clays:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Clotting, blood:** Control of anticoagulant therapy. Biggs, 84.

***Coagulation, blood:** Control of anticoagulant therapy. Biggs, 84.

Coal: Prepng. Standard Sand as Used in British Standard Method for Detmng. Agglutinating Value of —. (B.S. 705:1936.) D.S.I.R. Fuel Research Technical Paper No. 56. (Publication received), 71.

*tar: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Cobalamin. Approved collective name for vitamins possessed of B₁₂ activity. International Union of Pure and Applied Chemistry, 72.

*Efficient planning of microbiological assays illustrated by assays of —. Wood, 451.

*Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.

***Cobalt:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.

*Survey of development of electrographic analysis. Monk, 141.

*Systematic approach to choice of organic reagents for metal ions. Williams, 586.

***Cocoa residues:** Detmng. theobromine in —. Gerritsma and Koers, 201.

Coconut oil: Oils and Fats (No. 2) Order, 1953. 565.

***Cod-liver oil:** Spectroscopic properties of vitamin A₂, application to assay of —. Cama and Morton, 74.

Coffee: Labelling of Food Order, 1953. 324.

Combustion boats, sheath and contact stars for microchemical analysis. B.S. 1428:1953, Part II, 326.

train: Halogens and sulphur — (micro-Grote). B.S. 1428:1953, Part A4, 326.

***Complexes:** Use of inorganic — in colour reactions for organic compounds. I. Detmng. isonicotinic acid. Herington, 174.

Condensed Milk: Public Health (—) (Amendment) Regulations, 1953. 737.

Public Health (—) (Scotland) Amendment Regulations, 1953. 737.

Congress: Publication of Proceedings of First International — on Analytical Chemistry. Editorial, 133.

Contact stars: Combustion boats, sheath and — for microchemical analysis. B.S. 1428:1953, Part II, 326.

***Contamination:** Public health hazards and the analytical chemist. Nicholls, 272.

***Convallatoxin:** Colorimetric detmn. of cardiac glycosides. Hassall and Lippman, 126.

***Copper:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Detmng. — in plant material. Forster, 614.

*Detmng. — with sodium diethyldithiocarbamate in presence of nickel and other interfering elements, with particular reference to traces of — in sodium hydroxide. Jewsbury, 363.

*Diethylammonium diethyldithiocarbamate for separating and detmng. small amounts of metals. I. Successive detmn. of small amounts of —, manganese and iron in organic compounds. Wyatt, 656.

*Identifying alloys and stainless steels by electrographic methods. Clark and Hale, 145.

*Photometric detmn. of phosphorus in — based alloys containing tin. Lutwak, 661.

*Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.

*Quantitative separation of —, lead and tin by cathodic deposition. Aylward and Bryson, 651.

*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.

*Separating small amounts of arsenic, — and bismuth from lead and zinc by means of diethylammonium diethyldithiocarbamate. Strafford, Wyatt and Kershaw, 624.

*Separating zinc and cadmium by activated —. Bryson and Lowy, 299.

*Systematic approach to choice of organic reagents for metal ions. Williams, 586.

- *Cordials:** Albuminoid ammonia value in analysis of fruit juices, squashes and ——. Mitra and Roy, 681.
- Corned Meat:** Canned — (Prices) Order, 1953. 187.
- *Coronene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- *Corrosion:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Cotton seed oil:** Oils and Fats (No. 2) Order, 1953. 565.
- Counting chambers:** Haemocytometer — and dilution pipettes. B.S. 748:1953. 505.
- Cream and Use of Milk (Revocation) Order, 1953. 324.**
- *Cresol:** Detmng. amount and composition of free phenols in phenol-formaldehyde and — formaldehyde resins and moulding powders. Haslam, Whettem and Newlands, 340.
- *Cresyl esters:** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.
- Crews:** Appointment of T. Harris as Public Analyst to Borough of —, 444.
- Crime Investigation.** Physical Evidence and Police Laboratory. Kirk. (Publication received), 260.
- Crosby:** Appointment of T. Harris as Public Analyst to Borough of —, 626.
- Crucibles for microchemical analysis.** B.S. 1428:1953. Part E1, 326.
- *Cryoglobulins:** Micro-analytical methods for proteins in blood plasma. Critical review. Salt, 4.
- Crystal Structures.** Wyckoff. Vol. III and Suppl. II. (Publication received), 568.
- Culture Methods:** Sand and Water — Used in Study of Plant Nutrition. Hewitt. (Publication received), 71; (Review), 329.
- Cumberland:** Appointment of W. F. Elvidge as Deputy Public Analyst and Deputy Official Agricultural Analyst to County of —, 387.
- Cyanocobalamin.** Approved name for vitamin B₁₂. International Union of Pure and Applied Chemistry, 72.
- D**
- Dairy Chemistry:** Richmond's —. Davis and MacDonald. 5th Edn. (Publication received), 448.
- Damson jam:** Food Standards (Preserves) Order, 1953. 324; (Amendment) Order, 1953. 626.
- Darlington:** Appointment of W. F. Elvidge as Deputy Public Analyst and Deputy Official Agricultural Analyst to County Borough of —, 387.
- Dartford:** Appointment of R. C. Spalding as Deputy Public Analyst to Borough of —, 387.
- *Deacetylanhydro-oleandrin:** Colorimetric detmn. of cardiac glycosides. Hassall and Lippman, 126.
- Dean and Stark apparatus.** B.S. 756:1952. 128.
- Dehydrated vegetables:** Public Health (Preservatives, etc., in Food) (Amendment) Regulations, 1953. 737.
- vegetables: Sulphur dioxide in imported —. Ministry of Food, 128.
- Density bottles:** B.S. 733:1952. 68.
- hydrometers and specific gravity hydrometers. B.S. 718:1953. 445.
- *Deoxyribonucleic acid:** Comparison of applicability to plant extracts of three methods of detmng. —. Holden, 542.
- *Reaction between periodic acid and polyhydroxy compounds.** Tompsett and Smith, 209.
- Department of Scientific and Industrial Research:** Prepg. Standard Sand as Used in British Standard Method for Detmng. Agglutinating Value of Coal (B.S. 705:1936). — Fuel Research Technical Paper No. 56. (Publication received), 71.
- *Dianhydrogitoxinigenin:** Colorimetric detmn. of cardiac glycosides. Hassall and Lippman, 126.
- *Dibutyl phthalate:** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.
- Dichloromethane:** B.S. 1994:1953. 627.
- *4:6-Dichloromethylphenoxycetic acid:** Detmng. chloromethylphenoxycetic acids in MCPA formulations. Freeman and Gardner, 205.
- *Dichromate:** Modified dead-stop end-point circuit for titrations of ferrous iron with potassium — in approximately *N* solutions. Collier and Fricker, 440.
- *Dichromic acid, transmittancy curves:** Absorptiometric detmn. of chromium in steels and alloys. Wood, 54.
- *Diethylammonium diethyldithiocarbamate** for separating and detmng. small amounts of metals. I. Successive detmn. of small amounts of copper, manganese and iron in organic compounds. Wyatt, 656.
- *Separating small amounts of arsenic, copper and bismuth from lead and zinc by —.** Strafford, Wyatt and Kershaw, 624.
- Di-2-ethylhexyl phthalate.** B.S. 1995:1953. 627.
- *Digitoxin:** Colorimetric detmn. of cardiac glycosides. Hassall and Lippman, 126.
- *9:10-Dihydroanthracene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- *Dihydrostreptomycin:** Colorimetric detmn. of —. Ashton, Foster and Fatherley, 581.
- *Dihydroxyacetone:** Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- *2:4-Dihydroxybenzophenone:** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.
- *3:5-Di-iodothyronine:** Colorimetric detmn. of —. Shaw, 253.
- Dimethyl phthalate.** B.S. 1996:1953. 627.
- *2:4-Dimethylquinoline silicomolybdate:** Micro-analysis of silicate rocks. II. Pptn. of silica as — and gravimetric detmn. as silicomolybdic anhydride. Miller and Chalmers, 24.
- **m*-Dinitrobenzene:** Detmng. small amounts of — in nitrobenzene. Angell, 603.
- *Diphenyl:** Departure from Beer's law affecting spectrophotometric detmn. of —. Davenport, 558.
- Public Health (Preservatives, etc., in Food) (Amendment) Regulations, 1953. 737.
- Disinfectants:** Detmng. Rideal-Walker coefficient of —. B.S. 541:1934. Amendment slip, 505.
- *Di-sodium ethylenediaminetetra-acetate:** Detmng. zinc by titration with —. Strafford, 733.
- See also Versene.

- Dissociation Energies** and Spectra of Diatomic Molecules. Gaydon. 2nd Edn. (Publication received), 332.
- Distillation** range: Flasks for detmng. —. B.S. 571:1953. 257.
- ***Dithizone**: Reversion method for absorptiometric detmn. of traces of lead with —. Irving and Butler, 571.
- Documents**: Protective Display Lighting of Historical —. U.S. National Bureau of Standards. (Publication received), 448.
- Dried fruit**: Mineral Oil in Food (Amendment) Order, 1952. 68.
- ***Drinks**, soft: Detcng. preservatives in beverages by fermentation test, with special reference to brominated compounds. Mossel and de Bruin, 37.
- Drugs**: Aids to Analysis of Food and —. Nicholls. 7th Edn. (Publication received), 72; (Review), 328.
- Drummond Memorial Fund**, 449.
- ***Drying oven**: Design and operating technique of vacuum —. I. Design of oven. Gardiner, 709.
- Durham**: Appointment of W. F. Elvidge as Deputy Public Analyst and Deputy Official Agricultural Analyst to County of —, 387.
- Dyer**: Bernard — Memorial Medal, 261; Medal for exhibition, 450.
- ***Dyes**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
Chemistry of Synthetic. Vol. I. Venkataraman. (Review), 131; Vol. II. (Publication received), 72.

E

- E605**: See **Parathion**.
- ***Effluents**: Detcng. and detmng. traces of polynuclear hydrocarbons in industrial — and sewage. Wedgwood and Cooper, 170.
- ***Electro-analysis**: Apparatus for automatic control of cathode potential in —. Palmer and Vogel, 428.
- ***Electrodes**: Replacement of standard cell and salt bridge by indicator — and use of non-aqueous solutions in potentiometry. II. Iodometry and iodimetry in aqueous solution. Bishop, 149.
- ***Electrographic** analysis: Survey of development of —, with special reference to recent British apparatus. Monk, 141.
*methods: Identifying alloys and stainless steels by —. Clark and Hale, 145.
- ***Electrolysis** cell: Mercury-cathode cell for electrolytic separations. Aylward and Wooldridge, 386.
- Electronic Theory**: Théories Électroniques de la Chimie Organique. Pullman and Pullman. (Review), 329.
- Emulsifying salts**: Labelling of Food Order, 1953. 324.
- Encyclopedia** of Chemical Reactions. Vol. V. Jacobson. (Publication received), 388.
- Endocrinology**: Ciba Foundation Colloquia on —. Vol. II. Steroid Metabolism and Estmn. Wolstenholme and Cameron. (Publication received), 72; Vol. V. Bioassay of Anterior Pituitary and Adrenocortical Hormones. (Publication received), 448.
- Engineering**: Elements of Food —. Vol. I. Parker, Harvey and Stateler. (Publication received), 332.

- ***Enzymes**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Ergocalciferol**. Approved name for vitamin D₂ and calciferol. International Union of Pure and Applied Chemistry, 72.
- ***Ergosterol**: Detmng. — in yeast. I. Ultra-violet absorption of purified —. Shaw and Jefferies, 509; II. Detmn. by saponification and ultra-violet absorption spectroscopy, 514; III. Corrections for irrelevant absorption in solutions of —, 519; IV. Short method based on ultra-violet absorption, 524.
- Erith**: Appointment of R. C. Spalding as Deputy Public Analyst to Borough of —, 444.
- ***Erythritol**: Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- ***Esters**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Ethanolamine**: Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- ***Ethyl bromoacetate**: Detcng. preservatives in beverages by fermentation test, with special reference to brominated compounds. Mossel and de Bruin, 37.
- ***Ethyl cellulose**: Micro-detmn. of alkoxyl values in cellulose ethers. Easterbrook and Hamilton, 551; Erratum, 616.
- ***Ethylenediaminetetra-acetate** ammonium salt: Detmng. copper in plant material. Forster, 614.
*Detmng. zinc by titration with di-sodium —. Strafford, 733.
sodium: See also **Versene**.
- ***Ethylenediaminetetra-acetic acid**: Detmng. magnesium in plant material by —. Forster, 179.
- Ethyl methyl ketone**: B.S. 1940:1953. 257.
- ***2-Ethylpyridine**: Detmng. β - and γ -picolines, 2:6-lutidine and — in mixtures by infrared spectroscopy. Coulson and Hales, 114.
- Excerpta Medica**. Section XVI. Cancer (Experimental and Clinical). Vol. I, No. 1, July, 1953. New journal. (Publication received), 628.

F

- Faraday Society**: Letters exchanged between — and Society of Public Analysts and Other Analytical Chemists on 50th Anniversary of foundation of —, 450.
- ***Fat(s)**: Modified procedure for quantitative bromine absorption of oils and —. Burden and Grindley, 619.
*Modified van Gulik method for detmng. — in soft cheese. Pinsky and Grūnpeter, 621.
Oils and — Order, 1953. 187; (No. 2) Order, 1953. 565.
Sampling — and fatty oils. B.S. 627:1953. 326.
- ***Fatty acids**: Identifying and detmng. lower straight-chain — by paper partition chromatography. Duncan and Porteous, 641.
- ***Spectrophotometric** detmn. of long-chain — containing ketonic groups with particular reference to licanic acid. Mendelowitz and Riley, 704.
- Fatty Substances**: Méthodes d'Analyse et de Contrôle Industriel des Matières Grasses. Wolff and Wolff. (Publication received), 628.

- Feeding Stuffs** (Revocation) Order, 1953. 565.
- Feet:** Offals in Meat Products Order, 1953. 187
- *Fermentation,** inhibition of: Preservation of fermenting liquors in detmn. of reducing sugars. Donald, Freeman and Cunningham, 320.
- *test:** Detcng. preservatives in beverages by —, with special reference to brominated compounds. Mossel and de Bruin, 37.
- *Ferricyanide:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Ferrocyanide:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Critical examination of —** detmn. of zinc. Richardsen and Bryson, 291.
- *Volumetric detmn. of zinc with —** in magnesium alloys. Mayer, Bradshaw and Deutschman, 367.
- *Ferro-molybdenum:** Detmng. molybdenum by ammonium thiosulphate and sodium hypophosphite. Røy, 217; Erratum, 313.
- Fertiliser** Experiments, 1927-46, at Kroonstad Agricultural Research Station. van Garderen and Smuts. (Publication received), 506.
- Experiments in Natal, 1933-50. van Garderen, Brevis and Taylor. (Publication received), 506.
- Methods of Analysis used in O.E.E.C. Countries. Organisation for European Economic Co-operation. (Publication received), 188; (Review), 445.
- Fibres:** Identification of Textile —. Qualitative and Quantitative Analysis of — Blends. Luniak. (Publication received), 740.
- *Fibrinogen:** Micro-analytical methods for proteins in blood plasma. Critical review. Salt, 4.
- Field tests:** Chemical Tissue Tests for Detmng. Mineral Status of Plants in the Field. Nicholas. (Publication received), 628.
- Fig** and lemon jam: Food Standards (Preserves) (Amendment) Order, 1953. 626.
- Fillings:** Glossary of terms applicable to — and stuffings. B.S. 2005:1953. 627.
- Filter(s)** funnels: Glass —. B.S. 1923:1953. 326.
- Tests for performance characteristics of sintered —. B.S. 1969:1953. 627.
- *Fischer reagent:** Apparatus for simplifying titration in controlled atmosphere. Application to detmng. moisture in transformer oil with —. Prince, 607.
- *Fission products:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Flasks** for detmng. distillation range. B.S. 571: 1953. 257.
- Kohlrausch —. B.S. 615:1953. 627.
- Sugar —. B.S. 675:1953. 326.
- with graduated necks. B.S. 676:1953. 326.
- *Flavonoids:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Flour** confectionery: Labelling of Food Order, 1953. 324.
- *Detmng. benzoyl peroxide in — and bread.** Knight and Kent-Jones, 467.
- *Lipase activity of cereal products.** Templeton and Carpenter, 726.
- Order, 1953. 566.
- self-raising: Fluorine content of acidic phosphates used for food purposes. Ministry of Food, Food Standards Committee, Metallic Contamination Sub-Committee, 504.
- *Fluoranthene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- *Fluorene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- Fluorescence of Solutions.** Bowen and Wokes. (Publication received), 684.
- *Fluoride(s):** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Ion-exchange resins in analytical chemistry.** Application of ion-exchange resins to analysis of insoluble substances. Osborn, 220.
- *Fluorine:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Colorimetric detmn. of small amounts of —.** Liddell, 494.
- content of acidic phosphates used for food purposes. Ministry of Food, Food Standards Sub-Committee, Metallic Contamination Sub-Committee, 504.
- See also **Halogens.**
- *Folic acid:** Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.
- Food(s):** Aids to Analysis of — and Drugs. Nicholls. 7th Edn. (Publication received), 72; (Review), 328.
- Antioxidants. Ministry of —, — Standards Committee, Preservatives Sub-Committee, 504.
- Artificial Sweeteners in — Order, 1953. 626.
- *Bibliography on analytical applications of ion-exchange resins.** Osborn, 221.
- Elements of — Engineering. Vol. I. Parker, Harvey and Stateler. (Publication received), 332.
- Fluorine content of acidic phosphates used for — purposes. Ministry of —, — Standards Committee, Metallic Contamination Sub-Committee, 504.
- Labelling of — Order, 1953. 324.
- legislation: Random reflections on —. Adams, 569.
- List of Current Statutory Instruments and Statutory Rules and Orders relating to —. Ministry of —, 257, 505, 683.
- Mineral Oil in — (Amendment) Order, 1952. 68.
- Public Health (Preservatives, etc., in —) (Amendment) Regulations, 1953. 737.
- Standards (Ice-Cream) Order, 1953. 387.
- Standards (Preserves) Order, 1953. 324; (Amendment) Order, 1953. 626.
- Standards (Saccharin Tablets) Order, 1953. 626.
- Standards (Suet) Order, 1952. 128.
- Tin in canned —. Ministry of —, — Standards Committee, Metallic Contamination Sub-Committee, 187.
- *Formaldehyde:** Reaction between periodic acid and polyhydroxy compounds, with particular reference to colorimetric detmn. of — with chromotropic acid. Tompsett and Smith, 209.
- *Formic acid:** Bromometric detmn. of — and nitrite. Longstaff and Singer, 491.
- Formulary:** National — 1952. First Amendment 1953. (Publication received), 568.
- Fries:** Offals in Meat Products Order, 1953. 187.
- *Fructose:** Colorimetric detmn. of — and sorbose. Harris, 287.
- *Qualitative test for monosaccharides.** Love, 732.
- *Reaction between periodic acid and polyhydroxy compounds.** Tompsett and Smith, 209.
- *Simultaneous detmn. of pentose and hexose in mixtures of sugars.** Fernell and King, 80.
- Fruit, canned:** Labelling of Food Order, 1953. 324.
- citrus: Public Health (Preservatives, etc., in Food) (Amendment) Regulations, 1953. 737.

Fruit—continued

- curd: Food Standards (Preserves) Order, 1953. 324.
 dried: Mineral Oil in Food (Amendment) Order, 1952. 68.
 *juices: Albuminoid ammonia value in analysis of —, squashes and cordials. Mitra and Roy, 681.
 ***Fuel oils**: Paper-strip method of examining — suspected of being identical. Herd, 383.
Funnels: Glass filter —. B.S. 1923: 1953. 326.
Furans. Dunlop and Peters. (Publication received), 684.

G

- ***Galactose**: Qualitative test for monosaccharides. Love, 732.
 *Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
 *Simultaneous detmn. of pentose and hexose in mixtures of sugars. Fernell and King, 80.
 ***Galacturonic acid**: Qualitative test for monosaccharides. Love, 732.
 ***Galénicals**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
 ***Gas(es)** analysis: Apparatus for micro-analysis of gas samples. Madley and Strickland-Constable, 122.
 *Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method. Gregory, Mapper and Woodward, 414.
 ***Gelatin**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
 Detmng. — in meat extract and meat stocks. Interim report. Society of Public Analysts and Other Analytical Chemists, Analytical Methods Committee, Meat Extracts Sub-Committee, 134; Reprint available, 332.
General Medical Council: British Pharmacopoeia 1953. (Publication received), 260.
 ***Geo-chemistry**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
Germanium: Phenylfluorone, Reagent for —. Cooper and Yardley. (Publication received), 388.
 ***Gillingham**: Appointment of R. C. Spalding as Deputy Public Analyst to Borough of —, 444.
Gingelly oil: Oils and Fats (No. 2) Order, 1953. 565.
Ginger marmalade: Food Standards (Preserves) Order, 1953. 324.
Glasgow: Appointment of M. Herd as Public Analyst to City of —, 67.
 Appointment of T. M. Clark as Additional Public Analyst to City of —, 257.
 ***Glassware**: Adsorption of thiamine on —. Farrer and Hollenberg, 730.
 ***Globulins**: Micro-analytical methods for proteins in blood plasma. Critical review. Salt, 4.
Glossop: Appointment of T. Harris as Public Analyst to Borough of —, 444.
 ***Gluconic acid**: Qualitative test for monosaccharides. Love, 732.
 ***Glucosamine**: Qualitative test for monosaccharides. Love, 732.
 *hydrochloride: Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
 ***Glucose**: Qualitative test for monosaccharides. Love, 732.

Glucose—continued

- *Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
 *Simultaneous detmn. of pentose and hexose in mixtures of sugars. Fernell and King, 80.
 ***Glucose-1-phosphate**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
 ***Glutamic acid**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
 *Chromatographic detmn. of — in wheat gluten and gluten hydrolysates. Morries and Stuckey, 636.
 ***Glutamine**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
 ***Gluten**: Chromatographic detmn. of glutamic acid in wheat — and — hydrolysates. Morries and Stuckey, 636.
 ***Glyceric acid**: Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
Glycerol. Miner and Dalton. (Publication received), 568.
 *Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
 *Simple volumetric routine detmn. of —. Erskine, Strouts, Walley and Lazarus, 630.
Glycerol triacetate. B.S. 1997: 1953. 627.
 * **β -Glycerophosphate**, sodium: Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
 ***Glycosides**: Colorimetric detmn. of cardiac —. Hassall and Lippman, 126.
 ***Gold**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
 *Detmng. — in solution by adsorption extraction and spectrography. Lewis and Serin, 385.
 *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
Golden raising powder: Fluorine content of acidic phosphates used for food purposes. Ministry of Food, Food Standards Committee, Metallic Contamination Sub-Committee, 504.
Gooseberry and raspberry jam: Food Standards (Preserves) Order, 1953. 324; (Amendment) Order, 1953. 626.
 and strawberry jam: Food Standards (Preserves) Order, 1953. 324; (Amendment) Order, 1953. 626.
 jam: Food Standards (Preserves) Order, 1953. 324.
Grease, bone: Home-produced technical —. B.S. 1483: 1948. Amendment slip, 68.
Greengage jam: Food Standards (Preserves) Order, 1953. 324.
Ground nut oil: Oils and Fats (No. 2) Order, 1953. 565.
Gut: Offals in Meat Products Order, 1953. 187.

H

- Haemacytometer** counting chambers and dilution pipettes. B.S. 748: 1953. 505.
 ***Hafnium**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
 ***Hairs**: Separating rodent — and insect fragments from oat products. Hart and Heighton, 439.
 ***Halogens**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
Hammersmith: Appointment of D. F. H. Button as Public Analyst and of R. G. Minor as Deputy Public Analyst to Metropolitan Borough of —, 67.

- Hampstead:** Appointment of E. Voelcker as Public Analyst to Metropolitan Borough of —, 257.
- Hard soap:** Genuine —. B.S. 1911: 1953. 326.
- *Hazards:** Public health — and the analytical chemist. Nicholls, 272.
- Heat:** University Textbook of Physics. Vol. III. —. Awbrey. 11th Edn. (Publication received), 71.
- *Heparinic acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Herring oil:** Oils and Fats (No. 2) Order, 1953. 565.
- *Heteropoly acids:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Hexamethylenediamine dihydrochloride:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Hexamethylenetetramine:** Detmng. added — in two-stage phenol-formaldehyde resins. Chipperfield and Busfield, 617.
- *Reaction between periodic acid and polyhydroxy compounds.** Tompsett and Smith, 209.
- *Hexanoic acid:** Identifying and detmng. lower straight-chain fatty acids by paper partition chromatography. Duncan and Porteous, 641.
- *Hexone bases:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Hexose:** Simultaneous detmn. of pentose and — in mixtures of sugars. Fernell and King, 80.
- *Histamine:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Hormones:** Ciba Foundation Colloquia on Endocrinology. Vol. II. Steroid Metabolism and Estmn. Wolstenholme and Cameron. (Publication received), 72; Vol. V. Bioassay of Anterior Pituitary and Adrenocortical —. (Publication received), 448.
- *Hydrazine:** Detmng. ammonia in presence of —. Pugh and Heyns, 177.
- *Hydrocarbons:** Detcng. and detmng. traces of polynuclear — in industrial effluents and sewage. Wedgwood and Cooper, 170.
- *Hydrogen:** Apparatus for micro-analysis of gas samples. Madley and Strickland-Constable, 122.
- *Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method.** Gregory, Mapper and Woodward, 414.
- *Hydrogen peroxide:** Thiocyanate detmn. of iron with —. Houlihan and Farina, 559.
- Hydrometers:** Density — and specific gravity —. B.S. 718: 1953. 445.
- *Hydroquinone monobenzoate:** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.
- Hydroxocobalamin.** Approved name for vitamin B₁₂. International Union of Pure and Applied Chemistry, 72.
- *p-Hydroxydiphenyl test for acetaldehyde.** Detmng. alanine and threonine in protein hydrolysates. Folkes, 496.
- *Hydroxylamine:** Detmng. carbonyl compounds by semicarbazide and —, with special reference to fatty-acid oxidation products. Feuell and Skellon, 135.
- *Hypobromite:** Amperometric titration of traces of ammonia with — at rotated platinum wire electrode. Application to detmn. of nitrogen in organic compounds. Kolthoff, Stricks and Morren, 405.
- Ice-Cream:** Food Standards (—) Order, 1953. 387.
- *Indene:** Colorimetric detmn. of —. Roman and Smith, 679.
- Indicators:** Chemical —. Tomiček. Translated by Weir. (Review), 331.
- *Indium:** Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Scheme of semi-micro qualitative analysis for 39 elements.** Holness and Lawrence, 356.
- *Indole:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Indophenol:** Detmng. *p*-nitrophenol in urine and blood by — reaction. Lawford and Harvey, 63.
- Industry:** Chemical —, Past and Present. Williams. (Publication received), 740.
- Infra-red spectra.** Acceptance of report of Joint Committee on Reproduction of Infra-red Absorption Data, 684.
- Inorganic Analysis:** Textbook of Quantitative —. Kolthoff and Sandell. 3rd Edn. (Publication received), 188.
- Chemistry. Advanced Textbook.** Moeller. (Publication received), 72; (Review), 447.
- Chemistry: General and —.** Findlay. (Publication received), 188, 332.
- Thermogravimetric Analysis.** Duval. (Publication received), 188.
- *Inositol:** Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.
- *Insect fragments:** Separating rodent hairs and — from oat products. Hart and Heighton, 439.
- Institute of Petroleum.** Mass Spectrometry. (Review), 70.
- International Union of Pure and Applied Chemistry.** Approved nomenclature of vitamins, 72.
- *Iodides:** Micro-detmn. of — by arresting catalytic reduction of ceric ions. Rogina and Dubravčić, 594.
- Iodimetry:** Replacement of standard cell and salt bridge by indicator electrodes in potentiometry and use of non-aqueous solutions in potentiometry. II. Iodometry and — in aqueous solution. Bishop, 149.
- *Iodine:** Detcng. end-point in titrating — with thiosulphate. Knowles and Lowden, 159.
- *Detmng. — in blood serum.** Kirkpatrick, 348. See also **Halogens**.
- *Iodometry:** Replacement of standard cell and salt bridge by indicator electrodes and use of non-aqueous solutions in potentiometry. II. — and iodimetry in aqueous solution. Bishop, 149.
- Ion Exchange Resins.** British Drug Houses Ltd. (Publication received), 740.
- *Bibliography on analytical applications of —.** Osborn, 221.
- *in analytical chemistry.** Application of — to analysis of insoluble substances. Osborn, 220.
- Ion Exchangers in Analytical Chemistry.** Samuelson. (Publication received), 684.
- *Ionophoresis:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Iron:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Diethylammonium diethyldithiocarbamate for separating and detmng. small amounts of metals. I. Successive detmn. of small amounts of copper, manganese and — in organic compounds.** Wyatt, 656.

Iron—continued

- Ferrous Analysis.** Modern Practice and Theory. Pigott. 2nd Edn. (Publication received), 332.
- *Identifying alloys and stainless steels by electrographic methods. Clark and Hale, 145.
- *Modified dead-stop end-point circuit for titrations of ferrous — with potassium dichromate in approximately *N* solutions. Collier and Fricker, 440.
- *pig.: Detmng. arsenic by B. S. Evans's method, with observations on separating arsenic and antimony. Haslam and Wilkinson, 390.
- *Polarographic detmn. of — and chromium. Perkins and Reynolds, 480.
- *Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Research polarograph for photographic recording and multi-purpose polarographic cell. Bryant and Reynolds, 373.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Survey of development of electrographic analysis. Monk, 141.
- *Systematic approach to choice of organic reagents for metal ions. Williams, 586.
- *Thiocyanate detmn. of — with hydrogen peroxide. Houlihan and Farina, 559.
- ***isoButyl alcohol:** Detmng. *isobutyraldehyde* in *n*-butyraldehyde. Primavesi, 647.
- isoButyl methyl ketone:** B.S. 1941:1953. 257.
- ***isoButyraldehyde:** Detmng. — in *n*-butyraldehyde. Primavesi, 647.
- ***isoNicotinic acid:** Inorganic complexes in colour reactions for organic compounds. I. Detmng. —. Herington, 174.

J

- Jam:** Food Standards (Preserves) Order, 1953. 324.
- Jamaica rum:** Composition of —. 259.
- Journal(s):** Guida Bibliografica Internazionale per il Chimico, Libri e Riviste. Gaudenzi. (Publication received), 71.
- new: Analytical Abstracts. Society of Public Analysts and Other Analytical Chemists. Editorial, 629.
- new: Excerpta Medica. Section XVI. Cancer (Experimental and Clinical). Vol. I, No. 1, July, 1953. (Publication received), 628.
- new: Leybold Polarographische Berichte. Band I, Heft 1/2, Oktober, 1952, and Heft 3, Januar, 1953. (Publication received), 448.
- ***Juices:** Albuminoid ammonia value in analysis of fruit —, squashes and cordials. Mitra and Roy, 681.

K

- Kapok seed oil:** Oils and Fats (No. 2) Order, 1953. 565.
- Kent:** Appointment of R. C. Spalding as Deputy Official Agricultural Analyst to County of —, 257; as Deputy Public Analyst, 323.
- ***Keten:** Detmng. — and acetic anhydride in atmosphere. Diggle and Gage, 473.
- ***Ketones:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. carbonyl compounds by semicarbazide and hydroxylamine, with special reference to fatty-acid oxidation products. Feuill and Skellon, 135.

- ***Kjeldahl digestions:** Decomposition of ammonia in sealed-tube micro- — with selenium catalyst. Baker, 500.
- *Modified all-glass apparatus for detmng. nitrogen by micro- — method. Scandrett, 734.
- Kohlrausch flasks.** B.S. 615:1953. 627.
- Kroonstad Agricultural Research Station:** Agronomy Experiments, 1927-46, at —. Smuts and van Garderen. (Publication received), 506.
- Fertiliser Experiments, 1927-46, at —. van Garderen and Smuts. (Publication received), 506.

L

- Labelling of Food Order,** 1953. 324.
- Lacine XX:** Approved oxidising and preservative agents. Ministry of Food, 326.
- ***Lactose:** Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- ***Lanthanons:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Chemistry of —. Vickery. (Publication received), 740.
- ***Lanthanum:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. microgram amounts of calcium. Harrison and Raymond, 528.
- Lard:** Oils and Fats (No. 2) Order, 1953. 565.
- Latex:** Natural Rubber — and its Applications. No. 2. — Casting. Blow and Stokes. (Publication received), 71.
- ***Lauryl mercaptan:** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.
- ***Lead:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. aluminium and zinc after chromatographic separation from tin — alloys. Bishop and Liebmann, 117.
- *Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Qualitative detcn. of tellurium in tellurium — alloys. Brown, 623.
- *Quantitative separation of copper, — and tin by cathodic deposition. Aylward and Bryson, 651.
- *Reversion method for absorptiometric detmn. of traces of — with dithizone. Irving and Butler, 571.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Separating small amounts of arsenic, copper and bismuth from — and zinc by diethylammonium diethyldithiocarbamate. Strafford, Wyatt and Kershaw, 624.
- ***Lead acetylsalicylate:** Ion-exchange resins in analytical chemistry. Application of ion-exchange resins to analysis of insoluble substances. Osborn, 220.
- ***Leather:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Lecithin:** Detmng. total phosphatide in commercial —. Hutt, Weatherall and Culshaw, 712.
- ***Legislation:** Random reflections on food —. Adams, 569.
- Leicester:** Appointment of E. R. Pike as Deputy Official Agricultural Analyst to County Borough of —, 257.

- ***Lemon juice**: Ascorbic acid content of preserved —. Morgan, 323.
- ***Licanic acid**: Spectrophotometric detmn. of long-chain fatty acids containing ketonic groups with particular reference to —. Mendelowitz and Riley, 704.
- Lighting**: Protective Display — of Historical Documents. U.S. National Bureau of Standards. (Publication received), 448.
- ***Lipase** activity of cereal products. Templeton and Carpenter, 726.
- Liquors**: Labelling of Food Order, 1953. 324.
- Lites**: Offals in Meat Products Order, 1953. 187.
- ***Lithium**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- ***Liver oils**: Spectroscopic properties of vitamin A₂, application to assay of cod —. Cama and Morton, 74.
- Loganberry** jam: Food Standards (Preserves) Order, 1953. 324.
- Lovibond Comparator**: Handbook of Colorimetric Chemical Analytical Methods for Industrial, Research and Clinical Laboratories. Developed for Use with —. Tintometer, Ltd. (Publication received), 332; (Review), 568.
- ***Lubricants**: Detmng. neutralisation and saponification values of used —. Washbrook, 254.
- *Ultra-violet spectrophotometric estmn. of quality of mineral oils extracted from bread. Cookson, Coppock and Schnurmann, 695.
- ***Lubricating oils**: Detmng. zinc in — by amperometric titration. I. Amperometric titration of zinc with versene. Pickles and Washbrook, 304; Erratum, 439.
- Luminescence** and Scintillation Counter. Curran. (Publication received), 568.
- ***2:6-Lutidine**: Detmng. β - and γ -picolines, — and 2-ethylpyridine in mixtures by infra-red spectroscopy. Coulson and Hales, 114.
- ***Lyxose**: Qualitative test for monosaccharides. Love, 732.
- M**
- ***Magnesium** alloys: Volumetric detmn. of zinc with ferrocyanide in —. Mayer, Bradshaw and Deutschman, 367.
- *Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. — and aluminium in titanium metal. Corbett, 20.
- *Detmng. — in plant material by ethylene-diamine-tetra-acetic acid. Forster, 179.
- *Detmng. small amounts of potassium, calcium and — in sodium and its compounds. Silverman and Trego, 717.
- *Systematic approach to choice of organic reagents for metal ions. Williams, 586.
- Maize oil**: Oils and Fats (No. 2) Order, 1953. 565.
- Maleic Anhydride** Derivatives. Reactions of the Double Bond. Flett and Gardner. (Publication received), 131.
- ***Maltose**: Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- *Simultaneous detmn. of pentose and hexose in mixtures of sugars. Fernell and King, 80.
- ***Mandelic acid**: Separation of small amounts of zirconium with —. Mills and Hermon, 256.
- ***Manganese**: Diethylammonium diethyldithiocarbamate for separating and detmng. small amounts of metals. I. Successive detmn. of small amounts of copper, — and iron in organic compounds. Wyatt, 656.
- *Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- Manifolds**: Offals in Meat Products Order, 1953. 187.
- ***Mannitol**: Qualitative test for monosaccharides. Love, 732.
- *Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- ***Mannose**: Qualitative test for monosaccharides. Love, 732.
- Margarine**: Public Health (Preservatives, etc., in Food) (Amendment) Regulations, 1953. 737.
- Marmalade**: Food Standards (Preserves) Order, 1953. 324.
- Martindale**: Extra Pharmacopoeia (—). Vol. I. 23rd Edn. (Publication received), 71.
- Mass Spectrometry**. Institute of Petroleum. (Review), 70.
- Modern —. Barnard. (Publication received), 448.
- Maws**, pig's: Offals in Meat Products Order, 1953. 187.
- ***MCPA formulations**: Detmng. chloromethyl-phenoxyacetic acids in —. Freeman and Gardner, 205.
- Measurements**: Associated —. Quenouille. (Publication received), 71; (Review), 130.
- Meat**: Detmng. gelatin in — extract and — stocks. Interim report. Society of Public Analysts and Other Analytical Chemists, Analytical Methods Committee, — Extracts Sub-Committee, 134; Reprint available, 332.
- Products (No. 3) Order, 1952. 187.
- Products: Offals in — Order, 1953. 187.
- sulphur dioxide in: Circular MF 20/53. Ministry of Food, 737.
- Medal**: Bernard Dyer Memorial —, 261; for exhibition, 450.
- Medical Abstracts**. Excerpta Medica. Section XVI. Cancer (Experimental and Clinical). Vol. I, No. 1, July, 1953. (Publication received), 628.
- ***Medicinals**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Melon** and ginger jam: Food Standards (Preserves) Order, 1953. 324.
- and lemon jam: Food Standards (Preserves) Order, 1953. 324.
- and pineapple jam: Food Standards (Preserves) Order, 1953. 324.
- Melts**: Offals in Meat Products Order, 1953. 187.
- Memorial Fund**: Drummond —, 449.
- Medal: Bernard Dyer —, 261; for exhibition, 450.
- ***Mercaptans**: Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrel and Loveday, 92.
- ***Mercuric chloride**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Mercury-cathode** cell for electrolytic separations. Aylward and Wooldridge, 386.
- *Feigl's micro-test for — in organic substances. Sachs, 185.

Mercury—continued

- *Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Systematic approach to choice of organic reagents for metal ions. Williams, 586.
- ***Metallurgy**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Metal(s)**: Applied Inorganic Analysis, with Special Reference to Analysis of —, Minerals and Rocks. Hillebrand and Lundell. 2nd Edn. (Publication received), 448.
- *Micro-detmn. of traces of gaseous elements in — by vacuum fusion method. Gregory, Mapper and Woodward, 414.
- *Opening of white — for analysis by dry chlorine. Bishop, 61.
- *Prepng. biological material for detmn. of trace —. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Systematic approach to choice of organic reagents for — ions. Williams, 586.
- ***Methacrylate polymers**: Detcng. and detmn. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.
- ***Methane**: Apparatus for micro-analysis of gas samples. Madley and Strickland-Constable, 122.
- ***Methyl cellulose**: Micro-detmn. of alkoxy values in cellulose ethers. Easterbrook and Hamilton, 551; Erratum, 616.
- Methylene chloride**. B.S. 1994:1953. 627.
- Methyl ethyl ketone**. B.S. 1940:1953. 257.
- Methyl isobutyl ketone**. B.S. 1941:1953. 257.
- ***2-Methylphenoxyacetic acid**: Detmng. chloromethylphenoxyacetic acids in MCPA formulations. Freeman and Gardner, 205.
- ***Methyl salicylate**: Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.
- Micro-analysis**: Reference substances for use in organic —. Society of Public Analysts and Other Analytical Chemists, Microchemistry Group, 258.
- ***Microbiological assay(s)**: Efficient planning of — illustrated by assays of cobalamin. Wood, 451.
- of Vitamin B Complex and Amino-Acids. Barton-Wright. (Publication received), 71; (Review), 129.
- *Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.
- ***Milk**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- bottles: Capacity of cylindrical glass —. B.S. 1925:1953, 257.
- Cream and Use of — (Revocation) Order, 1953. 324.
- (Great Britain) Order, 1953. 326.
- *Indirect polarographic detmn. of calcium by chloranilic acid. Breyer and McPhillips, 666.
- Public Health (Condensed —) (Amendment) Regulations, 1953. 737.
- Public Health (Condensed —) (Scotland) Amendment Regulations, 1953. 737.
- Mincemeat**: Food Standards (Preserves) Order, 1953. 324.

- Mineral(s)**: Applied Inorganic Analysis, with Special Reference to Analysis of Metals, — and Rocks. Hillebrand and Lundell. 2nd Edn. (Publication received), 448.
- Chemical Tissue Tests for Detmng. — Status of Plants in the Field. Nicholas. (Publication received), 628.
- Composition and Assaying of —. Stewart-Remington and Francis. (Publication received), 71.
- Oil in Food (Amendment) Order, 1952. 68.
- *oils: Ultra-violet spectrophotometric estmn. of quality of — extracted from bread. Cookson, Coppock and Schnurmann, 695.
- Ministry of Agriculture and Fisheries**: Sugar Beet Cultivation. (Publication received), 628.
- Ministry of Food**, Food Standards Committee, Metallic Contamination Sub-Committee. Fluorine content of acidic phosphates used for food purposes, 504.
- Food Standards Committee, Metallic Contamination Sub-Committee. Tin in canned foods, 187.
- Food Standards Committee, Preservatives Sub-Committee. Antioxidants, 504.
- List of Current Statutory Instruments and Statutory Rules and Orders relating to Food, 257, 505, 683.
- Ministry of Food Orders, etc.:**
- Artificial sweeteners, 626.
- Beef sausages, 187.
- Bread, 566.
- Canned corned meat, 187.
- Condensed milk, 737.
- Cream, 324.
- Fats, 187, 565.
- Feeding stuffs, 565.
- Flour, 566.
- Flour confectionery, 324.
- Food standards, 128, 324, 387, 626.
- Fruit curd, 324.
- Ice-cream, 387.
- Jam, 324, 626.
- Labelling of food, 324.
- Marmalade, 324.
- Meat products, 187.
- Milk, 324.
- Mincemeat, 324.
- Mineral oil in dried fruit, 68.
- Offals, 187.
- Oils, 187, 565.
- Oxidising and preservative agents, 68, 326.
- Pork sausages, 187.
- Preservatives, 68, 326, 737.
- Preserves, 324, 626.
- Saccharin, 626.
- Sausages, 187.
- Suet, 128.
- Sulphur dioxide in imported dehydrated vegetables, 128.
- Sulphur dioxide in meat, 737.
- ***Moisture**: Apparatus for simplifying titration in controlled atmosphere. Application to detmng. — in transformer oil with Fischer reagent. Prince, 607.
- ***Molasses**: Detmng. unfermentable reducing substances in —. Donald, Freeman and Cunningham, 321.
- ***Molecular weights**: Isopiestic method for micro-detmn. of —. Morton, Campbell and Ma, 722.
- ***Molybdenum**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. — by ammonium thiosulphate and sodium hypophosphite. Rây, 217; Erratum, 313.

Molybdenum—continued

- *Identifying alloys and stainless steels by electrographic methods. Clarke and Hale, 145.
- *Rapid detmn. of — in soils. Grigg, 470.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Survey of development of electrographic analysis. Monk, 141.
- Monmouth:** Appointment of G. V. James as Additional Public Analyst to County of —, 67; as Deputy Official Agricultural Analyst, 257.
- ***Monosaccharides:** Qualitative test for —. Love, 732.
- ***Morgan's reagent:** Detmng. calcium in plants and soils. Hunter and Hall, 106.
- ***Morin:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Moulding powders:** Detmng. amount and composition of free phenols in phenol-formaldehyde and cresol-formaldehyde resins and —. Haslam, Whettem and Newlands, 340.
- ***Mucoproteins:** Micro-analytical methods for proteins in blood plasma. Critical review. Salt, 4.
- Mycology:** Medical —. Introduction to Problems. Ainsworth. (Publication received), 71.

N

- ***Naphthacene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- ***Naphthalene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- Natal:** Fertiliser Experiments in —, 1933–50. van Garderen, Brevis and Taylor. (Publication received), 506.
- National bread:** Bread Order, 1953. 566.
brown bread: Bread Order, 1953. 566.
flour: Flour Order, 1953. 566.
Formulary 1952: First Amendment 1953. (Publication received), 568.
- ***Neutralisation value:** Detmng. — and saponification value of used lubricants. Washbrook, 254.
- Newport:** Appointment of G. V. James as Additional Public Analyst and Deputy Official Agricultural Analyst to County Borough of —, 67.
- Niacinamide:** Approved name now nicotinamide. International Union of Pure and Applied Chemistry, 72.
- ***Nickel:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
*Detmng. — in plant material in presence of other metals. Forster, 560.
*Identifying alloys and stainless steels by electrographic methods. Clark and Hale, 145.
*Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
*Survey of development of electrographic analysis. Monk, 141.
- Nicotinamide:** Approved name for vitamin PP and niacinamide. International Union of Pure and Applied Chemistry, 72.
*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Nicotine:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Nicotinic acid:** Plate-assay technique for biotin, — and pantothenic acid. Morris and Jones, 15.

Nicotinic acid—continued

- *Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.
- ***Niobium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- ***Nitrate(s):** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Tests for nitrite and — applicable over wide concentration ranges. Woodward, 727.
- ***Nitrite:** Bromometric detmn. of formic acid and —. Longstaff and Singer, 491.
*Tests for — and nitrate applicable over wide concentration ranges. Woodward, 727.
- ***Nitrobenzene:** Detmng. small amounts of *m*-dinitrobenzene. Angell, 603.
- p*-Nitrodiazoaminoazobenzene:** See **Cadion**.
- ***Nitrogen:** Amperometric titration of traces of ammonia with hypobromite at rotated platinum electrode. Application to detmn. of — in organic compounds. Kolthoff, Stricks and Morren, 405.
*Apparatus for micro-analysis of gas samples. Madley and Strickland-Constable, 122.
*Decomposition of ammonia in sealed-tube micro-Kjeldahl digestions with selenium catalyst. Baker, 500.
*Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method. Gregory, Mapper and Woodward, 414.
*Modified all-glass apparatus for detmng. — by micro-Kjeldahl method. Scandrett, 734.
- ****p*-Nitrophenol:** Detmng. — in urine and blood by indophenol reaction. Lawford and Harvey, 63.
- p*-Nitrophenyl diethyl thiophosphate:** See **Parathion**.
- Nitrosocobalamin.** Approved name for vitamin B_{12c}. International Union of Pure and Applied Chemistry, 72.
- ***Nitrous oxide:** Apparatus for micro-analysis of gas samples. Madley and Strickland-Constable, 122.
- Northumberland:** Appointment of W. F. Elvidge as Deputy Public Analyst to County of —, 444.
- ***Novolaks:** Detmng. added hexamethylenetetramine in two-stage phenol-formaldehyde resins. Chipperfield and Busfield, 617.
- ***Nucleic acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
*Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- ***Nucleoproteins:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Nutrition:** Sand and Water Culture Methods Used in Study of Plant —. Hewitt. (Publication received), 71; (Review), 329.
- ***Nylon:** Chromatographic separation of polyamides. Ayers, 382.
*type polymers: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

O

- ***Oat meal:** Lipase activity of cereal products. Templeton and Carpenter, 726.
- *products: Separating rodent hairs and insect fragments from —. Hart and Heighton, 439.

Obituaries:

- Davies, R. E. L., 2, 264.
 Dickson, W., 189.
 Drummond, Sir Jack, 3, 264.
 Dyche-Teague, F. C., 134, 264.
 Forstner, G. E., 189.
 Gilbard, J. F. H., 264.
 Hughes, W. C., 685.
 Myers, J., 189.
 Newton, L. O., 74, 264.
 Rayner, A., 264, 265.
 Reeves, H. G., 2, 264, 265.
 Slater, R. H., 134, 264, 265.
 Stubbs, J. R., 262, 570.
 Thompson, F. E., 264, 265.
 Thorp, W., 333.

Offals in Meat Products Order, 1953. 187.

Oil(s) and Fats Order, 1953. 187; (No. 2) Order, 1953. 565.

- *Apparatus for simplifying titration in controlled atmosphere. Application to detmng. moisture in transformer — with Fischer reagent. Prince, 607.
 *Detmng. neutralisation and saponification values of used lubricants. Washbrook, 254.
 *Detmng. zinc in lubricating — by amperometric titration. I. Amperometric titration of zinc with versene. Pickles and Washbrook, 304; Erratum, 439.
 *Modified procedure for quantitative bromine absorption of — and fats. Burden and Grindley, 619.
 *Paper-strip method of examining fuel — suspected of being identical. Herd, 383.
 Sampling fats and fatty —. B.S. 627:1953. 326.
 *Spectroscopic properties of vitamin A₂, application to assay of cod-liver —. Cama and Morton, 74.
 Sunflower seed —. B.S. 1939:1953. 257.
 *Ultra-violet spectrophotometric estmn. of quality of mineral — extracted from bread. Cookson, Coppock and Schnurmman, 695.

***Ores:** Modified procedure for dissolving chromite —. Khundkar and Quadir, 623.

Organic analysis: Méthodes et Réactions de l'Analyse Organique. Vol. II. Pesze and Poirier. (Publication received), 260; (Review), 567.

Chemistry. Sarkar and Rakshit. 7th Edn. (Publication received), 628.

Chemistry. Turner and Harris. (Publication received), 71.

Chemistry. Advanced Treatise. Gilman. Vols. III and IV. (Publication received), 568.

Chemistry: Chemistry of Carbon Compounds. Vol. II, Part A. Alicyclic Compounds. Rodd. (Publication received), 568.

Chemistry: Heterocyclic Compounds. Vol. II. Elderfield. (Review), 259; Vols. III and IV, 260.

Chemistry: Maleic Anhydride Derivatives. Reactions of the Double Bond. Flett and Gardner. (Publication received), 131.

chemistry: Polysaccharide Chemistry. Whistler and Smart. (Publication received), 332; (Review), 740.

Chemistry: Practical —. Mann and Saunders. 3rd Edn. (Publication received), 71.

Chemistry: Structure and Mechanism in —. Ingold. (Publication received), 684.

Chemistry: Synthetic —. Wagner and Zook. (Publication received), 568.

chemistry: Théories Électroniques de la Chimie Organique. Pullman and Pullman. (Review), 329.

Organic—continued

micro-analysis: Reference substances for use in —. Society of Public Analysts and Other Analytical Chemists, Microchemistry Group, 258.

Reactions. Adams. Vol. VII. (Publication received), 684.

Syntheses. Vol. 32. Arnold. (Publication received), 188.

Organisation for European Economic Co-operation:

Fertilisers. Methods of Analysis used by — Countries. (Publication received), 188; (Review), 445.

***Orotic acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Orpington: Appointment of R. C. Spalding as Deputy Public Analyst to Urban District of —, 444.

***Oven:** Design and operating technique of vacuum drying —. I. Design of —. Gardiner, 709.

***Oxalate(s):** Anhydrous calcium — as weighing form for calcium. Miller, 186.

*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Oxalic acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Oxide(s):** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Oxidising agents: Approved — and preservative agents. Ministry of Food, 68, 326.

4-Oxo-octadeca-9:11:13-trienoic acid: See **Licanic acid**.

***Oxygen:** Apparatus for micro-analysis of gas samples. Madley and Strickland-Constable, 122.

*Double syringe-pipette for dissolved — estmns. Barnes, 501.

*Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method. Gregory, Mapper and Woodward, 414.

P

***Palladium:** Quantitative colour reaction for metallic —. McAllister, 65.

Palm kernel oil: Oils and Fats (No. 2) Order, 1953. 565.

oil: Oils and Fats (No. 2) Order, 1953. 565.

Pantothenic acid: Approved name unchanged. International Union of Pure and Applied Chemistry, 72.

*Plate-assay technique for biotin, nicotinic acid and —. Morris and Jones, 15.

*Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.

***Paper analysis:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*-strip method of examining fuel oils suspected of being identical. Herd, 383.

***Parathion:** Detmng. *p*-nitrophenol in urine and blood by indophenol reaction. Lawford and Harvey, 63.

Parsnips: Sulphur dioxide in imported dehydrated vegetables. Ministry of Food, 128.

Paunches: Offals in Meat Products Order, 1953. 187.

Peas: Sulphur dioxide in imported dehydrated vegetables. Ministry of Food, 128.

***Pectins:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Pendlebury: See Swinton and Pendlebury.

***Pentacyanoamminoferrate:** Inorganic complexes in colour reactions for organic compounds. I. Detmng. isonicotinic acid. Herington, 174.

***Pentose:** Simultaneous detmn. of — and hexose in mixtures of sugars. Fernell and King, 80.

***Peptides:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Perchlorates:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Periodic acid:** Reaction between — and polyhydroxy compounds, with particular reference to colorimetric detmn. of formaldehyde with chromotropic acid. Tompsett and Smith, 209.

***Perlon U:** Chromatographic separation of polyamides. Ayers, 382.

***Perylene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.

Pharmaceutical(s): Analysis: Aids to Qualitative —. Priestman and Edwards. (Publication received), 684.

Dispensing measures for — purposes (imperial units). B.S. 1921:1953. 387; Dispensing measures for — purposes (metric units). B.S. 1922:1953. 387.

*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Pharmaceutical Society of Great Britain: Calendar of —, 1952-53. (Publication received), 332.

Pharmacognosy: Textbook of —. Trease. 6th Edn. (Publication received), 131; (Review), 628.

***Pharmacological analysis:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Pharmacopoeia: British — 1953. General Medical Council. (Publication received), 260; (Review), 627.

Extra — (Martindale). Vol. I. 23rd Edn. (Publication received), 71.

***Phenanthrene:** Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.

***Phenol(s):** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Detmng. amount and composition of free — in — - formaldehyde and cresol - formaldehyde resins and moulding powders. Haslam, Whettem and Newlands, 340.

*- formaldehyde resins: Detmng. added hexamethylenetetramine in two-stage —. Chipperfield and Busfield, 617.

Phenylfluorone: Reagent for Germanium. Cooper and Yardley. (Publication received), 388.

***Phenyl mercuric ions:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Phenyl salicylate:** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrel and Loveday, 92.

Phosphate(s), acidic: Fluorine content of — used for food purposes. Ministry of Food, Food Standards Committee, Metallic Contamination Sub-Committee, 504.

*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Phosphatide:** Detmng. total — in commercial lecithin. Hutt, Weatherall and Culshaw, 712.

***Phosphomolybdic acid:** Quantitative colour reaction for metallic palladium. McAllister, 65.

***Phosphoric acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Phosphoric oxide:** Micro-analysis of silicate rocks. III. Spectrophotometric detmn. of — in presence of silica. Chalmers, 32.

***Phosphorus:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Micro-detmn. of — in biological material. Harvey, 110.

*Photometric detmn. of — in copper-based alloys containing tin. Lutwak, 661.

*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.

Physical Chemistry: Advanced Treatise on —. Vol. III. Properties of Solids. Partington. (Publication received), 71; (Review), 331.

Chemistry: Introduction to —. Findlay. 3rd Edn. Revised by Melville. (Publication received), 260.

Chemistry: Introduction to Study of —. Hammett. (Publication received), 131; (Review), 505.

Physics: University Textbook of —. Vol. III. Heat. Awbery. 11th Edn. (Publication received), 71.

***Picoline:** Detmng. β - and γ - —, 2:6-lutidine and 2-ethylpyridine in mixtures by infra-red spectroscopy. Coulson and Hales, 114.

***Pig-iron:** Detmng. arsenic by B. S. Evans's method, with observations on separating arsenic and antimony. Haslam and Wilkinson, 390.

Pilchard oil: Oils and Fats (No. 2) Order, 1953. 565.

***Pipette(s):** Double syringe- — for dissolved oxygen estmns. Barnes, 501.

Graduated — and one-mark cylindrical —. B.S. 700:1952. 68.

Haemocytometer counting chambers and dilution —. B.S. 748:1953. 505.

One-mark bulb —. B.S. 1583:1950. Amendment slip, 387.

Ostwald - Folin —. B.S. 773:1953. 326.

***Plant(s) analysis:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Chemical Tissue Tests for Detmng. Mineral Status of — in the Field. Nicholas. (Publication received), 628.

*Detmng. calcium in — and soils. Hunter and Hall, 106.

*extracts: Comparison of applicability to — of three methods of detmng. deoxyribonucleic acid. Holden, 542.

*material: Detmng. copper in —. Forster, 614.

*material: Detmng. magnesium in —, by ethylenediamine-tetra-acetic acid. Forster, 179.

*material: Detmng. nickel in — in presence of other metals. Forster, 560.

*material: Rapid turbidimetric detmn. of small amounts of sulphur in —. Steinbergs, 47.

Nutrition: Sand and Water Culture Methods Used in Study of —. Hewitt. (Publication received), 71; (Review), 329.

***Plasma:** Micro-analytical methods for proteins in blood —. Critical review. Salt, 4.

*Titrimetric detmn. of sodium in biological fluids. Trinder, 180.

***Plating:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Survey of development of electrographic analysis. Monk, 141.

- *Platinum:** Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- Plymouth:** Appointment of C. V. Reynolds as Deputy Official Agricultural Analyst to County Borough of —, 67.
- *Poisons:** Analysis of "general unknowns" in toxicology. Feldstein and Klendshoj, 43.
- *Polarisations:** Precipitate error in detmng. sugar —. Gaskin and Hands, 334.
- *Polarograph** capillaries from Pyrex glass rod. Ferrett and Phillips, 564.
- *Improved Randles-type cathode-ray —.** Reynolds and Davis, 314.
- *Research —** for photographic recording and multipurpose polarographic cell. Bryant and Reynolds, 373.
- *Polarographic** detmn. of iron and chromium. Perkins and Reynolds, 480.
- Polarography.** Vols. I and II. Kolthoff and Lingane. 2nd Edn. (Publication received), 71; (Review), 327.
- Leybold Polarographische Berichte. New journal. Band I, Heft 1/2, Oktober, 1952, and Heft 3, Januar, 1953. (Publication received), 448.
- Police Laboratory:** Crime Investigation. Physical Evidence and —. Kirk. (Publication received), 260.
- *Pollution:** Public health hazards and the analytical chemist. Nicholls, 272.
- *Polyamides:** Chromatographic separation of —. Ayers, 382.
- *Polycapromide:** Chromatographic separation of polyamides. Ayers, 382.
- *Polyhexamethylenediamine adipamide:** Chromatographic separation of polyamides. Ayers, 382.
- *Polyhexamethylenediamine sebacamide:** Chromatographic separation of polyamides. Ayers, 382.
- *Polymer(s):** Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrel and Loveday, 92.
- *emulsions:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Polymethyl methacrylate:** Detcng. and detmng. ultra-violet absorbers and other additives in — and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrel and Loveday, 92.
- Polysaccharide** Chemistry. Whistler and Smart. (Publication received), 332; (Review), 740.
- *Polyurethane:** Chromatographic separation of polyamides. Ayers, 382.
- Pontypool:** Appointment of G. V. James as Additional Public Analyst to Urban District of —, 67.
- Pork** sausages: Labelling of Food Order, 1953. 324.
- sausages: Meat Products (No. 3) Order, 1952. 187.
- *Potassium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Colorimetric micro-detmn. of —** in serum. Baar, 353.
- *Detmng. —** and traces of sodium in — salts. Jackson, 599.
- *Detmng. small amounts of —,** calcium and magnesium in sodium and its compounds. Silverman and Trego, 717.
- *Potassium dichromate:** Modified dead-stop end-point circuit for titrations of ferrous iron with — in approximately *N* solutions. Collier and Fricker, 440.
- *Potassium ferrocyanide:** Critical examination of ferrocyanide detmn. of zinc. Richardson and Bryson, 291.
- *Potassium permanganate:** Potentiometric detmn. of quadrivalent tellurium by — in weakly alkaline solutions. Issa and Awad, 487.
- Potatoes:** Sulphur dioxide in imported dehydrated vegetables. Ministry of Food, 128.
- *Potential:** Apparatus for automatic control of cathode — in electro-analysis. Palmer and Vogel, 428.
- *Potentiometric detmn. of quadrivalent tellurium** by potassium permanganate in weakly alkaline solutions. Issa and Awad, 487.
- *Potentiometry:** Replacement of standard cell and salt bridge by indicator electrodes and use of non-aqueous solutions in —. II. Iodometry and iodimetry in aqueous solution. Bishop, 149.
- *Preservation** of fermenting liquors in detmn. of reducing sugars. Donald, Freeman and Cunningham, 320.
- Preservative(s)** agents: Approved oxidising and —. Ministry of Food, 68, 326.
- Antioxidants. Ministry of Food, Food Standards Committee, — Sub-Committee, 504.
- *Detcng. —** in beverages by fermentation test, with special reference to brominated compounds. Mossel and de Bruin, 37.
- Public Health (—, etc., in Food) (Amendment) Regulations, 1953. 737.
- Preserves:** Food Standards (—) Order, 1953. 324; (Amendment) Order, 1953. 626.
- *Proactinium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Propionic acid:** Identifying and detmng. lower straight-chain fatty acids by paper partition chromatography. Duncan and Porteous, 641.
- Propyl gallate:** Antioxidants. Ministry of Food, Food Standards Committee, Preservatives Sub-Committee, 504.
- *Protein(s):** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *hydrolysates:** *p*-Hydroxydiphenyl test for acetaldehyde. Detmng. alanine and threonine in —. Folkes, 496.
- *Micro-analytical methods for —** in blood plasma. Critical review. Salt, 4.
- *Prothrombin test:** Control of anticoagulant therapy. Biggs, 84.
- *Public health** hazards and the analytical chemist. Nicholls, 272.
- Publications received:**
- Adams. Organic Reactions. Vol. VII, 684.
- Ainsworth. Medical Mycology, Introduction to its Problems, 71.
- Arnold. Organic Syntheses. Vol. 32, 188.
- Arthur and Smith. Semimicro Qualitative Analysis. 3rd Edn., 72.
- Assoc. of British Chemical Manufacturers. British Chemicals and their Manufacturers, 188.
- Audrieth and Kleinberg. Non-Aqueous Solvents. Applications as Media for Chemical Reactions, 448.
- Awbery. University Textbook of Physics. Vol. III. Heat. 11th Edn., 71.
- Ball. Biochemical Preparations. Vol. II, 71.
- Barnard. Modern Mass Spectrometry, 448.
- Barton-Wright. Microbiological Assay of Vitamin B Complex and Amino-Acids, 71.
- Bass and Broida. Spectrophotometric Atlas of $^{252}\text{E}^+$ — ^{252}I Transition of OH, 628.
- Blow and Stokes. Natural Rubber Latex and its Applications. No. 2. Latex Casting, 71.

Publications received—continued

- Bowen and Wokes. Fluorescence of Solutions, 684.
- Brautlect. Starch, Sources, Production and Uses, 568.
- Brimley and Barrett. Practical Chromatography, 684.
- British Drug Houses Ltd. Ion Exchange Resins, 740.
- Brown. Simple Guide to Modern Valency Theory, 568.
- Browning. Toxicity of Industrial Organic Solvents. 2nd Edn., 448.
- Chemical Society. Annual Reports on Progress of Chemistry for 1952. 628.
- Cooper and Yardley. Phenylfluorone, Reagent for Germanium, 388.
- Curran. Luminescence and Scintillation Counter, 568.
- Davidsohn, Better and Davidsohn. Soap Manufacture. Vol. I, 740.
- Davis and MacDonald. Richmond's Dairy Chemistry. 5th Edn., 448.
- Diehl and Smith. Quantitative Analysis. Elementary Principles and Practice, 260.
- D.S.I.R. Fuel Research Technical Paper No. 56. Prepng. Standard Sand as Used in British Standard Method for Detmng. Agglutinating Value of Coal (B.S. 705:1936), 71.
- Dunlop and Peters. Furans, 684.
- Duval. Inorganic Thermogravimetric Analysis, 188.
- Findlay. General and Inorganic Chemistry, 188, 332.
- Findlay. Introduction to Physical Chemistry. 3rd Edn., 260.
- Finney. Statistical Method in Biological Assay, 71.
- Flett and Gardner. Maleic Anhydride Derivatives. Reactions of the Double Bond, 131.
- Fruton and Simmonds. General Biochemistry, 568.
- Garside and Phillips. Textbook of Pure and Applied Chemistry, 332.
- Gaudenzi. Guida Bibliografica Internazionale per il Chimico, Libri e Riviste, 71.
- Gautier. Mises au Point de Chimie Analytique Pure et Appliquée et d'Analyse Bromatologique, 740.
- Gaydon. Dissociation Energies and Spectra of Diatomic Molecules. 2nd Edn., 332.
- Gilman. Organic Chemistry, Advanced Treatise. Vols. III and IV, 568.
- Goodwin. Comparative Biochemistry of Carotenoids, 71.
- Gore. Statistical Methods for Chemical Experimentation, 131.
- Hammett. Introduction to Study of Physical Chemistry, 131.
- Hewitt. Sand and Water Culture Methods Used in Study of Plant Nutrition, 71.
- Hillebrand and Lundell. Applied Inorganic Analysis, with Special Reference to Analysis of Metals, Minerals and Rocks. 2nd Edn., 448.
- Holness. Inorganic Qualitative Analysis. Semi-Micro Apparatus and Technique, 388.
- Ingold. Structure and Mechanism in Organic Chemistry, 684.
- Jacobs and Scheffan. Chemical Analysis. Vol. VII. Chemical Analysis of Industrial Solvents, 332.
- Jacobson. Encyclopedia of Chemical Reactions. Vol. V, 388.

Publications received—continued

- Ketelaar. Chemical Constitution. Introduction to Theory of the Chemical Bond, 448.
- Kirk. Crime Investigation. Physical Evidence and Police Laboratory, 260.
- Kolthoff and Lingane. Polarography. Vols. I and II. 2nd Edn., 71.
- Kolthoff and Sandell. Textbook of Quantitative Inorganic Analysis. 3rd Edn., 188.
- Lederer. Progrès Récents de la Chromatographie. Part 2. Chimie Minérale, 188.
- Luniak. Identification of Textile Fibres. Qualitative and Quantitative Analysis of Fibre Blends, 740.
- Mann and Saunders. Practical Organic Chemistry. 3rd Edn., 71.
- Miner and Dalton. Glycerol, 568.
- Ministry of Agriculture and Fisheries. Sugar Beet Cultivation, 628.
- Moeller. Inorganic Chemistry. Advanced Textbook, 72.
- Nicholas. Chemical Tissue Tests for Detmng. Mineral Status of Plants in the Field, 628.
- Nicholls. Aids to Analysis of Food and Drugs. 7th Edn., 72.
- O.E.E.C. Fertilisers. Methods of Analysis used in O.E.E.C. Countries, 188.
- Parker, Harvey and Steteler. Elements of Food Engineering. Vol. I, 332.
- Partington. Advanced Treatise on Physical Chemistry. Vol. III. Properties of Solids, 71.
- Pesetz and Poirier. Méthodes et Réactions de l'Analyse Organique. Vol. II, 260.
- Pharmaceutical Society of Great Britain. Calendar, 1952-53, 332.
- Pigott. Ferrous Analysis. Modern Practice and Theory. 2nd Edn., 332.
- Pollard and McOmie. Chromatographic Methods of Inorganic Analysis, with Special Reference to Paper Chromatography, 568.
- Priestman and Edwards. Aids to Qualitative Pharmaceutical Analysis, 684.
- Quenouille. Associated Measurements, 71.
- Quenouille. Design and Analysis of Experiment, 332.
- Radley. Starch and its Derivatives. Vol. I. 3rd Edn., 332.
- Rangaswami and Sen. Handbook of Shellac Analysis. 2nd Edn., 72.
- Rodd. Chemistry of Carbon Compounds. Vol. II, Part A, Alicyclic Compounds, 568.
- Samuelson. Ion Exchangers in Analytical Chemistry, 684.
- Sarkar and Rakshit. Organic Chemistry. 7th Edn., 628.
- Smith. Visual Lines for Spectroscopic Analysis. 2nd Edn., 188.
- Smuts and van Garderen. Agronomy Experiments, 1927-46, at Kroonstad Agricultural Research Station, 506.
- Society of Chemical Industry. Reports on Progress of Applied Chemistry. Vol. XXXVII, 568.
- Stewart-Remington and Francis. Composition and Assaying of Minerals, 71.
- Swanson and Fuyat. Standard X-ray Diffraction Powder Patterns. Vol. II, 740.
- Swanson and Tatge. Standard X-ray Diffraction Powder Patterns. Vol. I, 740.
- Tintometer Ltd. Handbook of Colorimetric Chemical Analytical Methods for Industrial, Research and Clinical Laboratories. Developed for Use with Lovibond Comparator, 332.

Publications received—continued

- Trease. Textbook of Pharmacognosy. 6th Edn., 131.
- Turner and Harris. Organic Chemistry, 71.
- U.S. National Bureau of Standards. Protective Display Lighting of Historical Documents, 448.
- van der Merwe and van Garderen. Agronomy Experiments, 1938-45, at Vaal-Hartz Agricultural Research Station, 506.
- van Garderen, Brevis and Taylor. Agronomy Experiments in Natal, 1933-50. 506.
- van Garderen and Smuts. Fertiliser Experiments, 1927-46, at Kroonstad Agricultural Research Station, 506.
- Velluz. Substances Naturelles de Synthèse. Préparations et Méthodes de Laboratoire. Vols. I-IV, 71.
- Venkataraman. Chemistry of Synthetic Dyes. Vol. II, 72.
- Vickery. Chemistry of Lanthanons, 740.
- Wagner and Zook. Synthetic Organic Chemistry, 568.
- Walter. Manufacture of Compressed Yeast. 2nd Edn., 628.
- West, Vick and LeRosen. Qualitative Analysis and Analytical Chemical Separations, 332.
- Whistler and Smart. Polysaccharide Chemistry, 332.
- Williams. Chemical Industry, Past and Present, 740.
- Wolff and Wolff. Méthodes d'Analyse et de Contrôle Industriel des Matières Grasses, 628.
- Wolstenholme and Cameron. Ciba Foundation Colloquia on Endocrinology. Vol. II. Steroid Metabolism and Estm., 72; Vol. V. Bioassay of Anterior Pituitary and Adrenocortical Hormones, 448.
- Wyckoff. Crystal Structures. Vol. III and Suppl. II, 568.
- British Pharmacopoeia 1953. 260.
- British Standards Institution Yearbook, 1953. 568.
- Excerpta Medica. Section XVI. Cancer (Experimental and Clinical). New journal, 628.
- Extra Pharmacopoeia (Martindale). Vol. I. 23rd Edn., 71.
- Leybold Polarographische Berichte. New journal, 448.
- National Formulary 1952, First Amendment 1953. 568.
- *Purines:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Pyrene:** Detmg. and detmng. traces of polynuclear hydrocarbons in industrial effluents and sewage. Wedgwood and Cooper, 170.
- *Pyrex glass:** Polarograph capillaries from — rod. Ferrett and Phillips, 564.
- *Pyridoxin:** Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.
- *Pyrimidine:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Pyroborate:** Detmng. borate in presence of silver. Haider, 673.
- Q**
- *Quinalizarin:** Detmng. small quantities of boron. Ripley-Duggan, 183.
- R**
- *Radioactivity:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Radium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Raffinose:** Large-scale chromatographic separation of sucrose - — mixtures on powdered cellulose for detmng. of — in raw sugars. Gross and Albon, 191.
- *Rare earths:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. microgram amounts of calcium.** Harrison and Raymond, 528.
- *Scheme of semi-micro qualitative analysis for 39 elements.** Holness and Lawrence, 356.
- Raspberry and gooseberry jam:** Food Standards (Preserves) Order, 1953. 324.
- and loganberry jam:** Food Standards (Preserves) Order, 1953. 324.
- and redcurrant jam:** Food Standards (Preserves) Order, 1953. 324.
- jam:** Food Standards (Preserves) Order, 1953. 324.
- Reactions:** Non-Aqueous Solvents. Applications as Media for Chemical —. Audrieth and Kleinberg. (Publication received), 448.
- Organic —.** Adams. Vol. VII. (Publication received), 684.
- *Reagents:** Systematic approach to choice of organic — for metal ions. Williams, 586.
- Redcurrant jam:** Food Standards (Preserves) Order, 1953. 324.
- *Reducing substances:** Detmng. unfermentable — in molasses. Donald, Freeman and Cunningham, 321.
- Reference substances** for use in organic micro-analysis. Society of Public Analysts and Other Analytical Chemists, Microchemistry Group, 258.
- *Refractometer:** Continuous differential — for chromatographic analysis. McCormick, 562.
- *Resins:** Bibliography on analytical applications of ion-exchange —. Osborn, 221.
- *Detmng. added hexamethylenetetramine in two-stage phenol-formaldehyde —.** Chipperfield and Busfield, 617.
- *Detmng. amount and composition of free phenols in phenol-formaldehyde and cresol-formaldehyde — and moulding powders.** Haslam, Whettem and Newlands, 340.
- Ion Exchange —.** British Drug Houses Ltd. (Publication received), 740.
- *Ion-exchange — in analytical chemistry.** Application of ion-exchange — to analysis of insoluble substances. Osborn, 220.
- Ion Exchangers in Analytical Chemistry.** Samuelson. (Publication received), 684.
- *Resorcinol monobenzoate:** Detmg. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.
- *Resorcyclic acid:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Review(s):** Guida Bibliografica Internazionale per il Chimico, Libri e Riviste. Gaudenzi. (Publication received), 71.
- papers.** Editorial, 1.
- *Rhamnose:** Qualitative test for monosaccharides. Love, 732.
- *Rhenium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Rhubarb and ginger jam:** Food Standards (Preserves) (Amendment) Order, 1953. 626.
- Riboflavin.** Approved name for vitamin B₂. International Union of Pure and Applied Chemistry, 72.

Riboflavin—*continued*

- *Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.
- ***Ribonucleic acid**: Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- ***Ribose**: Qualitative test for monosaccharides. Love, 732.
- *Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- *Simultaneous detmn. of pentose and hexose in mixtures of sugars. Fernell and King, 80.
- Richmond's Dairy Chemistry**. Davis and Macdonald. 5th Edn. (Publication received), 448.
- Rideal-Walker coefficient**: Detmng. — of disinfectants. B.S. 541:1934. Amendment slip, 505.
- Rocks**: Applied Inorganic Analysis, with Special Reference to Analysis of Metals, Minerals and —. Hillebrand and Lundell. 2nd Edn. (Publication received), 448.
- *Micro-analysis of silicate —. II. Pptn. of silica as 2:4-dimethylquinoline silicomolybdate and gravimetric detmn. as silicomolybdic anhydride. Miller and Chalmers, 24; III. Spectrophotometric detmn. of phosphoric oxide in presence of silica. Chalmers, 32; IV. Detmng. alumina. Miller and Chalmers, 686.
- ***Rodent hairs**: Separating — and insect fragments from oat products. Hart and Heighton, 439.
- ***Rosin size**: Analysis of —. Davis and Linke, 670.
- Rubber**: Natural — Latex and its Applications. No. 2. Latex Casting. Blow and Stokes. (Publication received), 71.
- ***Rubidium**: Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- Rum**: Composition of Jamaica —, 259.

S

- Saccharin**: Food Standards (— Tablets) Order, 1953. 626.
- ***Sage**: Adulterant of dried —. Button, 679.
- St. Helens**: Appointment of J. G. Sherratt as Public Analyst and Official Agricultural Analyst to County Borough of —, 257.
- ***Salicylates**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- Sand**: Prepng. Standard — as Used in British Standard Method for Detmng. Agglutinating Value of Coal (B.S. 705:1936). D.S.I.R. Fuel Research Technical Paper No. 56. (Publication received), 71.
- ***Saponification value**: Detmng. neutralisation value and — of used lubricants. Washbrook, 254.
- Sausage(s)**: Labelling of Food Order, 1953. 324.
- meat: Labelling of Food Order, 1953. 324.
- Meat Products (No. 3) Order, 1952. 187.
- Savonol** (Red Label): Approved oxidising and preservative agents. Ministry of Food, 326.
- Scintillation Counter**: Luminescence and —. Curran. (Publication received), 568.
- ***Sea water**: Double syringe-pipette for dissolved oxygen estmns. Barnes, 501.
- Seal oil**: Oils and Fats (No. 2) Order, 1953. 565.
- Seed oil**: Sunflower —. B.S. 1939:1953. 257.
- ***Selenium**: Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- Self-raising flour**: Fluorine content of acidic phosphates used for food purposes. Ministry of Food, Food Standards Committee, Metallic Contamination Sub-Committee, 504.
- ***Semicarbazide**: Detmng. carbonyl compounds by — and hydroxylamine, with special reference to fatty-acid oxidation products. Feuell and Skellon, 135.
- ***Serine**: Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.
- ***Serum**: Colorimetric micro-detmn. of potassium in —. Baar, 353.
- *Detmng. iodine in blood —. Kirkpatrick, 348.
- *Detmng. microgram amounts of calcium. Harrison and Raymond, 528.
- *Indirect polarographic detmn. of calcium by chloranilic acid. Breyer and McPhillips, 666.
- *Titrimetric detmn. of sodium in biological fluids. Trinder, 180.
- Sesame oil**: Oils and Fats (No. 2) Order, 1953. 565.
- ***Sewage**: Detcng. and detmng. traces of polynuclear hydrocarbons in industrial effluents and —. Wedgwood and Cooper, 170.
- Shea butter**: Oils and Fats (No. 2) Order, 1953. 565.
- oil: Oils and Fats (No. 2) Order, 1953. 565.
- Sheath**: Combustion boats, — and contact stars for microchemical analysis. B.S. 1428:1953. Part II, 326.
- Shellac**: Handbook of — Analysis. Rangaswami and Sen, revised by Bhattacharya and Bose. 2nd Edn. (Publication received), 72; (Review), 130.
- Sidecup**: See **Chislehurst and Sidcup**.
- ***Silica**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Micro-analysis of silicate rocks. II. Pptn. of — as 2:4-dimethylquinoline silicomolybdate and gravimetric detmn. as silicomolybdic anhydride. Miller and Chalmers, 24; III. Spectrophotometric detmn. of phosphoric oxide in presence of —. Chalmers, 32.
- *Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- ***Silicate rocks**: Micro-analysis of —. II. Pptn. of silica as 2:4-dimethylquinoline silicomolybdate and gravimetric detmn. as silicomolybdic anhydride. Miller and Chalmers, 24; III. Spectrophotometric detmn. of phosphoric oxide in presence of silica. Chalmers, 32; IV. Detmng. alumina. Miller and Chalmers, 686.
- ***Siliceous materials**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Silicofluoride**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Silicomolybdic anhydride**: Micro-analysis of silicate rocks. II. Pptn. of silica as 2:4-dimethylquinoline silicomolybdate and gravimetric detmn. as —. Miller and Chalmers, 24.
- ***Silicon**: Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- ***Silver**: Detmng. borate in presence of —. Haider, 673.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Systematic approach to choice of organic reagents for metal ions. Williams, 586.
- ***Size**: Analysis of rosin —. Davis and Linke, 670.
- ***Soap(s)**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Soap—continued

- Carbolic —. B.S. 1910:1953; genuine hard —. B.S. 1911:1953; — flakes. B.S. 1912:1953; soft —. B.S. 1913:1953; toilet —. B.S. 1914:1953. 326.
- Manufacture. Vol. I. Davidsohn, Better and Davidsohn. (Publication received), 740.
- Society of Chemical Industry.** Reports on Progress of Applied Chemistry. Vol. XXXVII. (Publication received), 568.
- *Society of Public Analysts and Other Analytical Chemists:** Address of retiring President. Nicholls, 271.
- Analytical Abstracts. New journal. Editorial, 629.
- Analytical Methods Committee, Meat Extracts Sub-Committee. Detmng. gelatin in meat extract and meat stocks. Interim report, 134; Reprint available, 332.
- Anniversary dinner, 281.
- Annual Report of Council, 264.
- Biological Methods Group, 8th A.G.M., 190.
- Committees, 1953-54. 263.
- Humble Address to Her Most Excellent Majesty Queen Elizabeth II, facing p. 333; Reply, facing p. 389.
- Letters exchanged between — and Faraday Society on occasion of 50th Anniversary of foundation of Faraday Society, 450.
- Microchemistry Group. 9th A.G.M., 190.
- Microchemistry Group. Reference substances for use in organic micro-analysis, 258.
- North of England Section. 28th A.G.M., 189.
- Physical Methods Group. 8th A.G.M., 190.
- Polarographic Discussion Panel. A.G.M., 190.
- Reprint of *The Analyst* from 1876 to 1951. 73.
- Scottish Section. 18th A.G.M., 190.
- 79th A.G.M., 262.
- *Sodium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. potassium and traces of — in potassium salts. Jackson, 599.
- *Detmng. small amounts of potassium, calcium and magnesium in — and its compounds. Silverman and Trego, 717.
- *Titrimetric detmn. of — in biological fluids. Trinder, 180.
- *Sodium chloride:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Sodium diethyldithiocarbamate:** Detmng. copper with — in presence of nickel and other interfering elements, with particular reference to traces of copper in sodium hydroxide. Jewsbury, 363.
- *Detmng. zinc by —. Mayne and Noordhof, 625.
- *Sodium hexametaphosphate:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Sodium hydroxide:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. copper with sodium diethyldithiocarbamate in presence of nickel and other interfering elements, with particular reference to traces of copper in —. Jewsbury, 363.
- *Sodium hypophosphite:** Detmng. molybdenum by ammonium thiosulphate and —. Rây, 217; Erratum, 313.
- *Soft drinks:** Detng. preservatives in beverages by fermentation test, with special reference to brominated compounds. Mossel and de Bruin, 37.
- Soft soap.** B.S. 1913:1953. 326.
- *Soils:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. calcium in plants and —. Hunter and Hall, 106.
- *Rapid detmn. of molybdenum in —. Grigg, 470.
- *Soldiers:** Detmng. aluminium and zinc after chromatographic separation from tin-lead alloys. Bishop and Liebmann, 117.
- Solvents:** Chemical Analysis. Vol. VII. Chemical Analysis of Industrial —. Jacobs and Scheflan. (Publication received), 332.
- Non-Aqueous —. Applications as Media for Chemical Reactions. Audrieth and Kleinberg. (Publication received), 448.
- Toxicity of Industrial Organic —. Browning. 2nd Edn. (Publication received), 448.
- *Sorbitol:** Qualitative test for monosaccharides. Love, 732.
- *Sorbse:** Colorimetric detmn. of fructose and —. Harris, 287.
- *Qualitative test for monosaccharides. Love, 732.
- South Shields:** Appointment of W. F. Elvidge as Deputy Public Analyst and Deputy Official Agricultural Analyst to County Borough of —, 387.
- Soya bean oil:** Oils and Fats (No. 2) Order, 1953. 565.
- Specific gravity:** Density hydrometers and — hydrometers. B.S. 718:1953. 445.
- Spectra:** Dissociation Energies and — of Diatomic Molecules. Gaydon. 2nd Edn. (Publication received), 332.
- infra-red: Acceptance of report of Joint Committee on Reproduction of Infra-red Absorption Data, 684.
- Ultraviolet — of Aromatic Compounds. Friedel and Orchin. (Review), 69.
- Spectrometry:** Mass —. Institute of Petroleum. (Review), 70.
- Modern Mass —. Barnard. (Publication received), 448.
- Spectrophotometry:** Spectrophotometric Atlas of $^2\Sigma^+ - ^2\Pi$ Transition of OH. Bass and Broida. (Publication received), 628.
- Spectroscopic Analysis:** Visual Lines for —. Smith. 2nd Edn. (Publication received), 188; (Review), 567.
- Spinach:** Sulphur dioxide in imported dehydrated vegetables. Ministry of Food, 128.
- Spinal cord:** Offals in Meat Products Order, 1953. 187.
- *Squashes:** Albuminoid ammonia value in analysis of fruit juices, — and cordials. Mitra and Roy, 681.
- *Stainless steels:** Identifying alloys and — by electrographic methods. Clark and Hale, 145.
- Standards:** Antioxidants. Ministry of Food, Food — Committee, Preservatives Sub-Committee, 504.
- Fluorine content of acidic phosphates used for food purposes. Ministry of Food, Food — Committee, Metallic Contamination Sub-Committee, 504.
- Food — (Ice-Cream) Order, 1953. 387.
- Food — (Preserves) Order, 1953. 324; (Amendment) Order, 1953. 626.
- Food — (Saccharin Tablets) Order, 1953. 626.
- Food — (Suet) Order, 1952. 128.
- Tin in canned foods. Ministry of Food, — Committee, Metallic Contamination Sub-Committee, 187.

Starch and its Derivatives. Vol. I. Radley. 3rd Edn. (Publication received), 332.

*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

edible: Labelling of Food Order, 1953. 324.

*Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.

Sources, Production and Uses. Brantlect. (Publication received), 568.

***Stas - Otto procedure**, modified: Analysis of "general unknowns" in toxicology. Feldstein and Klendshoj, 43.

Statistical methods: Associated Measurements. Quenouille. (Publication received), 71; (Review), 130.

Design and Analysis of Experiment. Quenouille. (Publication received), 332.

*Efficient planning of microbiological assays illustrated by assays of cobalamin. Wood, 451. for Chemical Experimentation. Gore. (Publication received), 131.

in Biological Assay. Finney. (Publication received), 71; (Review), 505.

***Steel(s)**: Absorptiometric detmn. of chromium in — and alloys. Wood, 54.

*Detmng. molybdenum by ammonium thio-sulphate and sodium hypophosphite. Rây, 217; Erratum, 313.

*Identifying alloys and stainless — by electro-graphic methods. Clark and Hale, 145.

*Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method. Gregory, Mapper and Woodward, 414.

*Survey of development of electrographic analysis. Monk, 141.

Stepney: Appointment of A. E. Parkes as Public Analyst and H. A. Parkes as Additional Public Analyst to Metropolitan Borough of —, 323.

Steroid: Ciba Foundation Colloquia on Endocrinology. Vol. II. — Metabolism and Estmn. Wolstenholme and Cameron. (Publication received), 72; Vol. V. Bioassay of Anterior Pituitary and Adrenocortical Hormones, 448.

Sterylclean: Approved oxidising and preservative agents. Ministry of Food, 68.

***Stilbene**: Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate - ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.

Stockport: Appointment of T. Harris as Public Analyst and Official Agricultural Analyst to County Borough of —, 323.

Stockton-on-Tees: Appointment of W. F. Elvidge as Deputy Public Analyst to County Borough of —, 387.

Strawberry and gooseberry jam: Food Standards (Preserves) Order, 1953. 324.

jam: Food Standards (Preserves) Order, 1953. 324.

***Strontium**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Detmng. microgram amounts of calcium. Harrison and Raymond, 528.

*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.

*Systematic approach to choice of organic reagents for metal ions. Williams, 586.

***Strontium sulphate**: Ion-exchange resins in analytical chemistry. Application of ion-exchange resins to analysis of insoluble substances. Osborn, 220.

Structure and Mechanism in Organic Chemistry. Ingold. (Publication received), 684.

Stuffings: Glossary of terms applicable to fillings and —. B.S. 2005:1953. 627.

***Sucrose**: Large-scale chromatographic separation of — - raffinose mixtures on powdered cellulose for detmn. of raffinose in raw sugars. Gross and Albon, 191.

*Simultaneous detmn. of pentose and hexose in mixtures of sugars. Fernell and King, 80.

Suet: Food Standards (—) Order, 1952. 128.

Sugar(s) Beet Cultivation. Ministry of Agriculture and Fisheries. (Publication received), 628.

*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Colorimetric detmn. of fructose and sorbose. Harris, 287.

flasks. B.S. 675:1953. 326.

*Large-scale chromatographic separation of sucrose - raffinose mixtures on powdered cellulose for detmn. of raffinose in raw —. Gross and Albon, 191.

*Precipitate error in detmng. — polarisations. Gaskin and Hands, 334.

*Preservation of fermenting liquors in detmn. of reducing —. Donald, Freeman and Cunningham, 320.

*Qualitative test for monosaccharides. Love, 732.

*Simultaneous detmn. of pentose and hexose in mixtures of —. Fernell and King, 80.

***Sulphate**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Sulphur**: Detcng. and detmng. ultra-violet absorbers and other additives in polymethyl methacrylate and methyl methacrylate - ethyl acrylate co-polymers. Haslam, Grossman, Squirrell and Loveday, 92.

*Prepg. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.

*Rapid turbidimetric detmn. of small amounts of — in plant material. Steinbergs, 47.

***Sulphur dioxide**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Detmng. carbon dioxide and — in Orsat apparatus. Young, Benfield and Strachan, 320. in imported dehydrated vegetables. Ministry of Food, 128.

in meat: Circular MF 20/53. Ministry of Food, 737.

Public Health (Preservatives, etc., in Food) (Amendment) Regulations, 1953. 737.

Sunflower seed oil. B.S. 1939:1953. 257.

Oils and Fats (No. 2) Order, 1953. 565.

Swedes: Sulphur dioxide in imported dehydrated vegetables. Ministry of Food, 128.

Sweetbreads: Offals in Meat Products Order, 1953. 187.

Sweeteners: Artificial — in Food Order, 1953. 626.

Swinton and Pendlebury: Appointment of T. Harris as Public Analyst to Borough of —, 626.

Symposium on Analytical Chemistry, 1954. Arrangements, 388.

Syntheses: Organic —. Vol. 32. Arnold. (Publication received), 188.

Substances Naturelles de Synthèse. Préparations et Méthodes de Laboratoire. Vols. I, II, III, and IV. Velluz. (Publications received), 71; (Review), 446.

T

Tallow: Home-produced technical —. B.S. 1482:1948. Amendment slip, 68.

Oils and Fats (No. 2) Order, 1953. 565.

- ***Tanning**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Tantalum**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- ***Tar**: Paper-strip method of examining fuel oils suspected of being identical. Herd, 383.
- ***Tartaric acid**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Tellurium**: Potentiometric detmn. of quadrivalent — by potassium permanganate in weakly alkaline solutions. Issa and Awad, 487.
- *Qualitative detcn. of — in — lead alloys. Brown, 623.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- Textile Fibres**: Identification of —. Qualitative and Quantitative Analysis of Fibre Blends. Luniak. (Publication received), 740.
- ***Thallium**: Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- ***Theobromine**: Detmng. — in cocoa residues. Gerritsma and Koers, 201.
- Thermogravimetric Analysis**: Inorganic —. Duval. (Publication received), 188.
- Thermometers**: Secondary reference — (Centigrade scale). B.S. 1900:1952. 68; Amendment slip, 187.
- ***Thiamine**: Adsorption of — on glassware. Farrer and Hollenberg, 730.
- Approved name for vitamin B₁ and aneurine. International Union of Pure and Applied Chemistry, 72.
- *Selection of methods for routine assays for members of vitamin-B complex. Pritchard, 460.
- ***Thiocyanate** cationic complexes: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *detmn. of iron with hydrogen peroxide. Houlihan and Farina, 559.
- ***Thiosulphate**: Deteng. end-point in titrating iodine with —. Knowles and Lowden, 159.
- ***Thorium**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *salt of 4-(*o*-arsonophenylazo)-N-(1-naphthyl)-ethylenediamine: Colorimetric detmn. of small amounts of fluorine. Liddell, 494.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- ***Threonine**: *p*-Hydroxydiphenyl test for acetaldehyde. Detmng. alanine and — in protein hydrolysates. Folkes, 496.
- ***Thyroxine**: Colorimetric detmn. of 3:5-di-iodo-thyronine. Shaw, 253.
- ***Timber**: Deteng. boron in treated —. McMullen, 442.
- *Detmng. small quantities of boron. Ripley-Duggan, 183.
- ***Tin**: Detmng. aluminium and zinc after chromatographic separation from — lead alloys. Bishop and Liebmman, 117.
- *Guard valve for — detmns. by iodimetric procedure. Jackson, 443.
- in canned foods. Ministry of Food, Food Standards Committee, Metallic Contamination Sub-Committee, 187.
- *Photometric detmn. of phosphorus in copper-based alloys containing —. Lutwak, 661.
- *Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.
- Tin—continued**
- *Quantitative separation of copper, lead and — by cathodic deposition. Aylward and Bryson, 651.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- *Systematic approach to choice of organic reagents for metal ions. Williams, 586.
- Tintometer Ltd.**: Chemical Tissue Tests for Detmng. Mineral Status of Plants in the Field. Nicholas. (Publication received), 628.
- Handbook of Colorimetric Chemical Analytical Methods for Industrial, Research and Clinical Laboratories. Developed for Use with Lovibond Comparator. (Publication received), 332; (Review), 568.
- ***Titanium**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- *Detmng. magnesium and aluminium in — metal. Corbett, 20.
- *Identifying alloys and stainless steels by electrographic methods. Clark and Hale, 145.
- *Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- ***Titration(s)**: Apparatus for dead-stop end-point — with acoustic indication of end-point. Glastonbury, 682.
- *Apparatus for simplifying — in controlled atmosphere. Application to detmng. moisture in transformer oil with Fischer reagent. Prince, 607.
- Tocopherols**: α -, β - and γ —. Approved names for vitamins E. International Union of Pure and Applied Chemistry, 72.
- Toilet soap**. B.S. 1914:1953. 326.
- Toxicity** of Industrial Organic Solvents. Browning. 2nd Edn. (Publication received), 448.
- ***Toxicology**: Analysis of "general unknowns" in —. Feldstein and Klendshoj, 43.
- ***Trace elements**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.
- ***Transformer oil**: Apparatus for simplifying titration in controlled atmosphere. Application to detmng. moisture in — with Fischer reagents. Prince, 607.
- Triacetin**. B.S. 1997:1953. 627.
- Tricresyl phosphate**. B.S. 1999:1953. 627.
- Tripe**: Offals in Meat Products Order, 1953. 187.
- Triphenyl phosphate**. B.S. 1998:1953. 627.
- ***Tri-sodium pentacyanoamminoferrate**: Inorganic complexes in colour reactions for organic compounds. I. Detmng. *isonicotinic acid*. Herington, 174.
- Tritolyl phosphate**. B.S. 1999:1953. 627.
- Tunbridge Wells**: Appointment of R. C. Spalding as Deputy Public Analyst to Borough of —, 387.
- ***Tungsten**: Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.
- Turnips**: Sulphur dioxide in imported dehydrated vegetables. Ministry of Food, 128.

U

- Udders**: Offals in Meat Products Order, 1953. 187.
- ***Ultra-violet** absorbers: Deteng. and detmng. — and other additives in polymethyl methacrylate and methyl methacrylate-ethyl acrylate copolymers. Haslam, Grossman, Squirrel and Loveday, 92.
- Spectra of Aromatic Compounds. Friedel and Orchin. (Review), 69.

Ultra-violet—*continued*

*spectrophotometric estmn. of quality of mineral oils extracted from bread. Cookson, Coppock and Schnurmann, 695.

***Unfermentable reducing substances**: Detmng. — in molasses. Donald, Freeman and Cunningham, 321.

U.S. National Bureau of Standards: Protective Display Lighting of Historical Documents. (Publication received), 448.

Spectrophotometric Atlas of $^2\Sigma^+ \rightarrow ^2\Pi$ Transition of OH. Bass and Broida. (Publication received), 628.

Standard X-ray Diffraction Powder Patterns. Vols. I and II. (Publications received), 740.

***Uranium**: Detmng. uranium-235 in mixtures of naturally occurring — isotopes by radioactivation. Seyfang and Smales, 394.

*Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method. Gregory, Mapper and Woodward, 414.

*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.

*Systematic approach to choice of organic reagents for metal ions. Williams, 586.

***Uranium-235**: Detmng. — in mixtures of naturally occurring uranium isotopes by radioactivation. Seyfang and Smales, 394.

***Urine**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Detmng. *p*-nitrophenol in — and blood by indophenol reaction. Lawford and Harvey, 63.

*Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.

*Titrimetric detmn. of sodium in biological fluids. Trinder, 180.

Uteri: Offals in Meat Products Order, 1953. 187.

V

Vaal-Hartz Agricultural Research Station: Agronomy Experiments, 1938-45, at —, van der Merwe and van Garderen. (Publication received), 506.

Valency: Simple Guide to Modern — Theory. Brown. (Publication received), 568.

***Valeric acid**: Identifying and detmng. lower straight-chain fatty acids by paper partition chromatography. Duncan and Porteous, 641.

***Valve**: Guard — for tin detmns. by iodimetric procedure. Jackson, 443.

***Vanadium**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.

***van Gulik method**: Modification of — for detmng. fat in soft cheese. Pinsky and Grünpeter, 621.

Vegetables, canned: Labelling of Food Order, 1953. 324.

dehydrated: Public Health (Preservatives, etc., in Food) (Amendment) Regulations, 1953. 737.

Sulphur dioxide in imported dehydrated —, Ministry of Food, 128.

Vells, calves: Offals in Meat Products Order, 1953. 187.

***Versene**: Detmng. zinc in lubricating oils by amperometric titration. I. Amperometric titration of zinc with —. Pickles and Washbrook, 304; Erratum, 439.

***Vinegar**: Characterising — by albuminoid ammonia value. Mitra, 499.

***Viruses**: Evaluation of anti-viral compounds. Dickinson, 283.

Viscosity: Method for detmng. — of liquids in absolute (C.G.S.) units. B.S. 188:1937. Amendment slip, 567.

***Vitamin A₂**: Spectroscopic properties of —, application to assay of cod-liver oil. Cama and Morton, 74.

Vitamin B Complex: Microbiological Assay of — and Amino-Acids. Barton-Wright. (Publication received), 71; (Review), 129.

*complex: Selection of methods for routine assay of members of —. Pritchard, 460.

Vitamin B₁: Approved name now thiamine. International Union of Pure and Applied Chemistry, 72.

Vitamin B₂: Approved name now riboflavin. International Union of Pure and Applied Chemistry, 72.

Vitamin B₁₂: Approved name now cyanocobalamin. Approved collective name for vitamins possessed of B₁₂ activity now cobalamin. International Union of Pure and Applied Chemistry, 72.

Vitamin B_{12ab}: Approved name now hydroxocobalamin. International Union of Pure and Applied Chemistry, 72.

Vitamin B_{12c}: Approved name now nitrosocobalamin. International Union of Pure and Applied Chemistry, 72.

Vitamin C: Approved name now ascorbic acid. International Union of Pure and Applied Chemistry, 72.

Vitamin D₂: Approved name now ergocalciferol. International Union of Pure and Applied Chemistry, 72.

Vitamin D₃: Approved name now cholecalciferol. International Union of Pure and Applied Chemistry, 72.

Vitamin PP: Approved name now nicotinamide. International Union of Pure and Applied Chemistry, 72.

Vitamins: Approved nomenclature. International Union of Pure and Applied Chemistry, 72.

*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Vitamins E: Approved names now α -, β - and γ -tocopherol. International Union of Pure and Applied Chemistry, 72.

***Voltammetry**: Amperometric titration of traces of ammonia with hypobromite at rotated platinum electrode. Application to detmn. of nitrogen in organic compounds. Kolthoff, Stricks and Morren, 405.

W

Wallasey: Appointment of T. Harris as Public Analyst and Official Agricultural Analyst and of J. F. Clark as Official Agricultural Analyst to County Borough of —, 387.

Wandsworth: Appointment of J. E. Woodhead as Public Analyst to Metropolitan Borough of —, 444.

***Waste liquors**: Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Water**: Apparatus for simplifying titration in controlled atmosphere. Application to detmng. moisture in transformer oil with Fischer reagent. Prince, 607.

*Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

Water—*continued*

- *Double syringe-pipette for dissolved oxygen estmns. Barnes, 501.

West Hartlepool: Appointment of W. E. Elvidge as Deputy Public Analyst and Deputy Official Agricultural Analyst to County Borough of —, 387.

Westmorland: Appointment of W. F. Elvidge as Deputy Public Analyst and Deputy Official Agricultural Analyst to County of —, 626.

West Riding of Yorkshire: *See* **Yorkshire**.

Whale oil: Oils and Fats (No. 2) Order, 1953. 565.

***Wheat gluten:** Chromatographic detmn. of glutamic acid in — and gluten hydrolysates. Morries and Stuckey, 636.

***White metals:** Opening of — for analysis by dry chlorine. Bishop, 61.

Wiltshire: Appointment of J. H. Hamence as Official Agricultural Analyst to County of —, 387.

***Wine:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

***Wood:** Detmg. boron in treated timber. McMullen, 442.

*Detmg. small quantities of boron. Ripley-Duggan, 183.

***Wool:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Rapid turbidimetric detmn. of small amounts of sulphur in plant material. Steinbergs, 47.

X

X-ray Diffraction Powder Patterns: Standard —, Swanson and Tatge. Vol. I; Swanson and Fuyat. Vol. II. (Publication received), 740.

***Xylose:** Qualitative test for monosaccharides. Love, 732.

*Reaction between periodic acid and polyhydroxy compounds. Tompsett and Smith, 209.

*Simultaneous detmn. of pentose and hexose in mixtures of sugars. Fernell and King, 80.

Y

***Yeast:** Detmg. ergosterol in —, I. Ultra-violet absorption of purified ergosterol. Shaw and Jefferies, 509; II. Detmn. by saponification and ultra-violet absorption spectroscopy, 514; III. Corrections for irrelevant absorption in solutions of ergosterol, 519; IV. Short method based on ultra-violet absorption, 524.

Manufacture of Compressed —, Walter. 2nd Edn. (Publication received), 628.

Yeast—*continued*

*Preservation of fermenting liquors in detmn. of reducing sugars. Donald, Freeman and Cunningham, 320.

Yorkshire: Appointment of R. Mallinder as Public Analyst to County of West Riding of —, 67; as Official Agricultural Analyst, 257.

Youngberry jam: Food Standards (Preserves) Order, 1953. 324.

***Yttrium:** Detmg. microgram amounts of calcium. Harrison and Raymond, 528.

Z

***Zinc:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Critical examination of ferrocyanide detmn. of —. Richardson and Bryson, 291.

*Detmg. aluminium and — after chromatographic separation from tin-lead alloys. Bishop and Liebmann, 117.

*Detmg. — by sodium diethyldithiocarbamate. Mayne and Noordhof, 625.

*Detmg. — by titration with di-sodium ethylenediaminetetra-acetate. Strafford, 733.

*Detmg. — in lubricating oils by amperometric titration. I. Amperometric titration of — with versene. Pickles and Washbrook, 304; Erratum, 439.

*Prepng. biological material for detmn. of trace metals. I. Critical review of existing procedures. Middleton and Stuckey, 532.

*Separating small amounts of arsenic, copper and bismuth from lead and — by diethylammonium diethyldithiocarbamate. Strafford, Wyatt and Kershaw, 624.

*Separating — and cadmium by activated copper. Bryson and Lowy, 299.

*Survey of development of electrographic analysis. Monk, 141.

*Systematic approach to choice of organic reagents for metal ions. Williams, 586.

*Volumetric detmn. of — with ferrocyanide in magnesium alloys. Mayer, Bradshaw and Deutschman, 367.

***Zirconium:** Bibliography on analytical applications of ion-exchange resins. Osborn, 221.

*Micro-detmn. of traces of gaseous elements in metals by vacuum fusion method. Gregory, Mapper and Woodward, 414.

*Scheme of semi-micro qualitative analysis for 39 elements. Holness and Lawrence, 356.

*Separation of small amounts of — with mandelic acid. Mills and Hermon, 256.

PRINTED BY
W. HEFFER & SONS LTD.,
CAMBRIDGE, ENGLAND