THE ANALYST

A Monthly Publication
dealing with all branches
of Analytical Chemistry:
the Journal of the Society
for Analytical Chemistry

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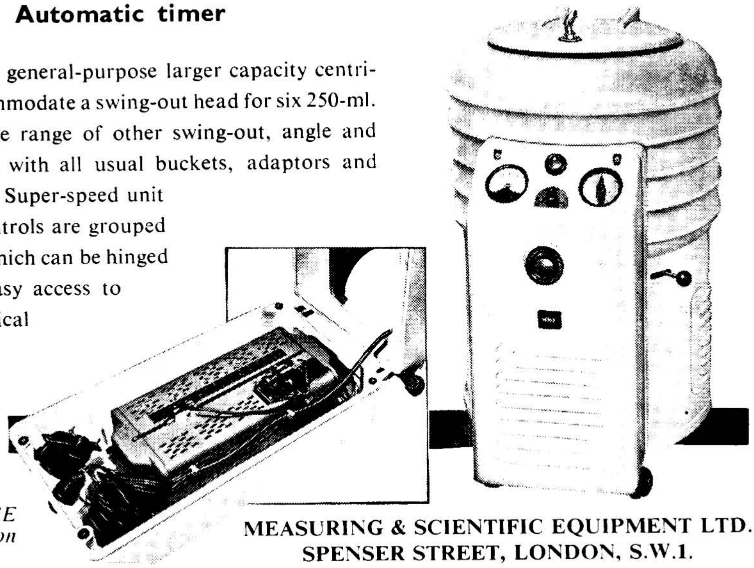

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No. 24 December, 1954

THE SOCIETY FOR ANALYTICAL CHEMISTRY

BULLETIN

FORTHCOMING MEETINGS

Ordinary Meeting of the Society, January 28th, 1955

AN Ordinary Meeting of the Society, organised by the Microchemistry Group, will be held at 7.30 p.m. on Friday, January 28th, 1955, at the School of Pharmacy, Bloomsbury Square, London, W.C.1.

Special Meeting of the Society, February 2nd, 1955

A SPECIAL Meeting of the Society will be held at 6 p.m. on Wednesday, February 2nd, 1955, in the Lecture Theatre of the Royal Institution, 21, Albemarle Street, London, W.1.

A lecture with practical demonstrations will be given by Professor G. Schwarzenbach, Ph.D., of Zurich University, on "The Complexones and their Analytical Application."

Annual General Meeting of the North of England Section, January 29th, 1955

THE Annual General Meeting of the North of England Section will be held at 2 p.m. on Saturday, January 29th, 1955, at the Engineers' Club, **Manchester.**

This will be followed immediately by an Ordinary Meeting of the Section, at which the following paper will be presented—

"The Importance of Analysis in Industry," by J. Haslam, D.Sc., F.R.I.C.

Annual General Meeting of the Scottish Section, January 19th, 1955

THE Annual General Meeting of the Scottish Section will be held at 12.45 p.m. on Wednesday, January 19th, 1955, at Macvitties (Charlotte Rooms), 136, Princes Street, **Edinburgh.**

Joint Meeting of the Scottish Section with the Society of Glass Technology and the Stirlingshire and District Sections of the Royal Institute of Chemistry and the Society of Chemical Industry, February 8th, 1955

A JOINT Meeting of the above bodies will be held at 6.30 p.m., on Tuesday, February 8th, 1955, at the Golden Lion, **Stirling.**

The following paper will be presented—

"Some Properties of Glasses in Relation to Composition," by Professor H. Moore, D.Sc., A.R.C.S., F.Inst.P.

The meeting will be preceded by an afternoon visit to the Alloa Glass Works.

Joint Meeting of the Western Section with the Local Sections of the Royal Institute of Chemistry, the Chemical Society and the Society of Chemical Industry, January 27th, 1955

A JOINT Meeting of the above bodies will be held on Thursday, January 27th, 1955, at Bristol University, Woodland Road, **Bristol.**

The following paper will be presented—

"Recent Advances in Bacteriological Examination of Water Supplies," by Dr. E. Windle Taylor.

Annual General Meeting of the Microchemistry Group, January 28th, 1955

THE Annual General Meeting of the Microchemistry Group will be held at 7 p.m. on Friday, January 28th, 1955, at the School of Pharmacy, Bloomsbury Square, London, W.C.1, and will be followed by the Retiring Chairman's Address.

Ordinary Meeting of the Physical Methods Group, January 18th, 1955

AN Ordinary Meeting of the Physical Methods Group will be held at 6.30 p.m. on Tuesday, January 18th, 1955, at the Iron and Steel Institute, 4, Grosvenor Gardens, London, S.W.1.

The subject of the meeting will be "Solvent Extraction."

COMMUNICATIONS ACCEPTED FOR PUBLICATION IN *THE ANALYST*

THE following communications 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.

"The Fractionation and Determination of Corticosteroids in Urine," by E. R. Cook, Barbara Dell and D. J. Wareham.

"A Method for Determining the Freezing Points of Biological Fluids," by G. R. Hervey.

"The Determination of Dissociation Constants of Dibasic Acids," by H. Irving, Mrs. H. S. Rossotti and G. Harris.

"The Determination of Pectin Grade with the Ridgelimeter," by H. C. Lockwood. (Note.)

"The Estimation of Amino-acids with Ninhydrin," by E. W. Yemm and E. C. Cocking.

"An Improved Apparatus for the Determination of Gaseous Elements in Metals by Vacuum Fusion on a Micro Scale," by J. N. Gregory and D. Mapper.

"The Determination of Oxygen in Beryllium by the Vacuum Fusion Method on a Micro Scale (with a Note on the Determination of Oxygen in Zirconium)," by J. N. Gregory and D. Mapper.

"Simultaneous Absorptiometric Determination of Tantalum and Niobium in Ores," by A. E. O. Marzys.

"The Spectrophotometric Identification and Estimation of Parathion," by A. I. Biggs.

"The Use of Ion-exchange Resins in the Analysis of Coal Ash," by F. Ellington and L. Stanley. (Note.)

"A Colorimetric Method for the Estimation of Methyl Bromide in Air," by G. A. Lugg.

"The Determination of Methylpentoses," by M. N. Gibbons.

"Determination of Small Quantities of Thorium by Radioactivation," by E. N. Jenkins.

"Recovery of Chloroform used in Dithizone Extraction," by J. B. Mullin and J. P. Riley. (Note.)

"Application of Paper-chromatographic Methods of Analysis to Geochemical Prospecting," by E. C. Hunt, A. A. North and R. A. Wells.

"Determination of Trimethylene Glycol in Crude Glycerine," by W. Lazarus and T. H. Newlove.

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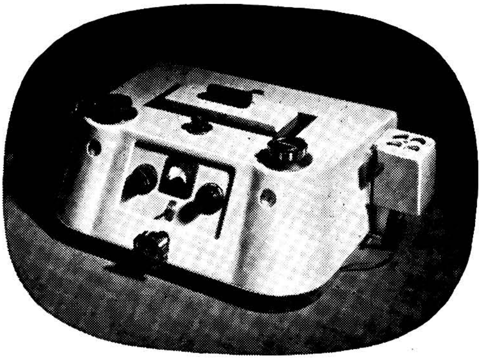

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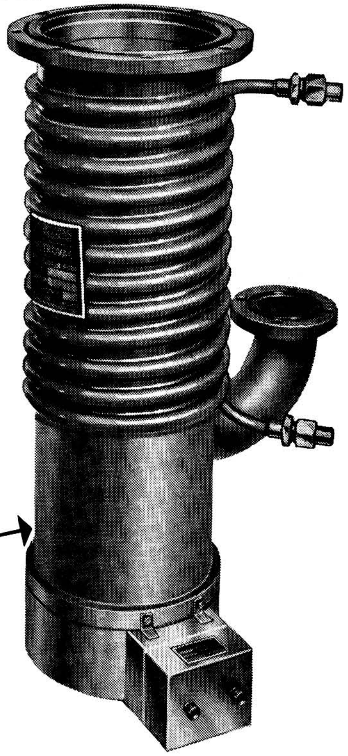


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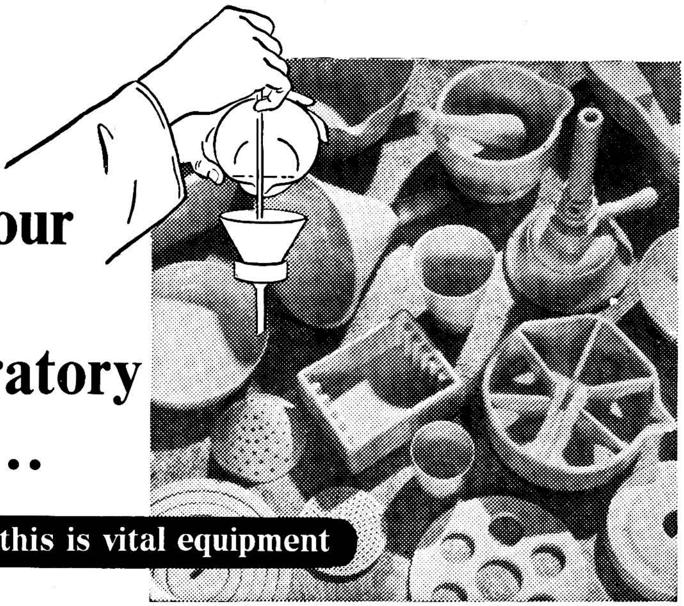

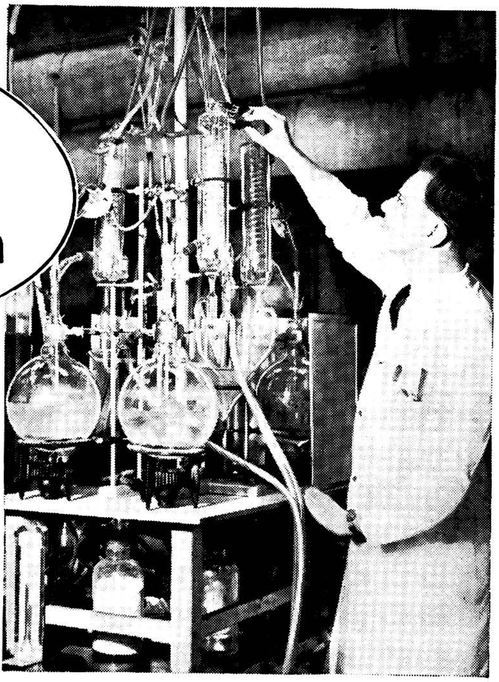

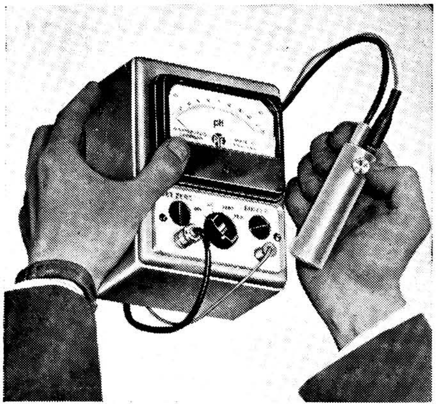

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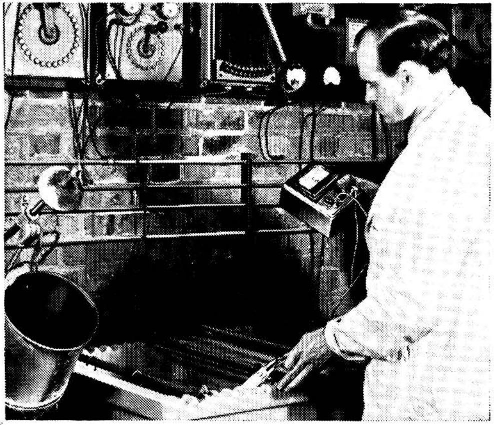

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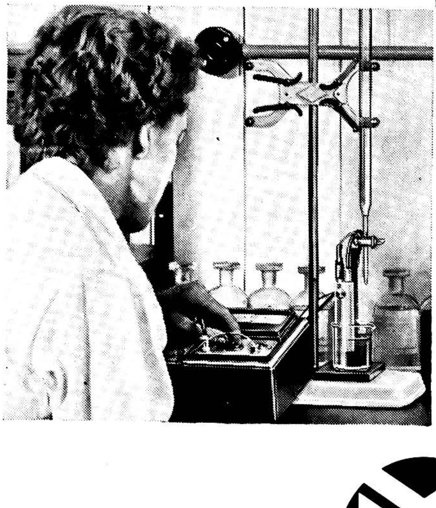

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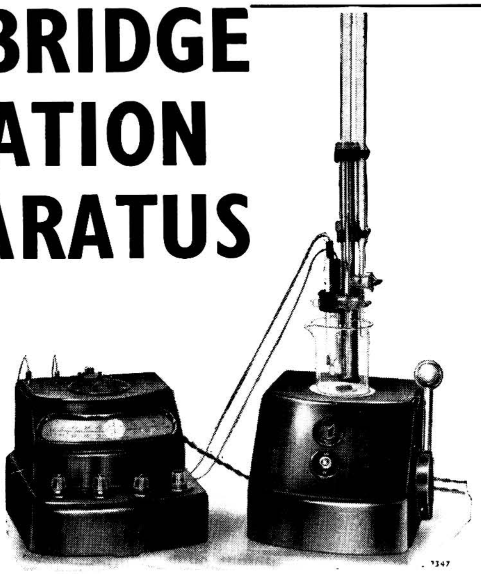

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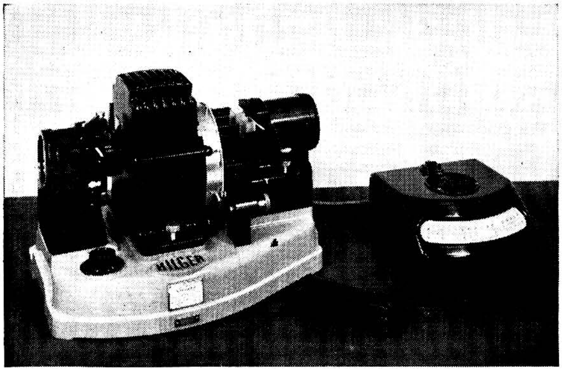

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DECEMBER, 1954 Vol. 79, No. 945

THE ANALYST

PROCEEDINGS OF THE SOCIETY FOR ANALYTICAL CHEMISTRY

ORDINARY MEETING

An Ordinary Meeting of the Society, organised by the Biological Methods Group, was held at 6.45 p.m. on Wednesday, November 3rd, 1954, 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.

The subject of the meeting was "The Biological Evaluation of the Purity of Water and Effluents" and the following papers were presented and discussed: "Introduction" by B. A. Southgate, Ph.D., D.Sc., F.R.I.C.; "Measurement of Toxicity to Fish," by D. W. M. Herbert, B.Sc., M.I.Biol.; "Some Aspects of the Biology of Polluted Rivers," by J. E. Forrest, B.Sc., Ph.D., M.I.Biol.; "Determination of the Safety of Water," by E. Windle Taylor, M.A., M.D., D.P.H., M.R.C.S., L.R.C.P., Barrister-at-Law.

JOINT MEETING

A Joint Meeting of the Society with the Oils and Fats Group of the Society of Chemical Industry was held at 7 p.m. on Wednesday, December 1st, 1954, in the meeting room of the Chemical Society, Burlington House, London, W.1. The Chair was taken by the President of the Society for Analytical Chemistry, Dr. D. W. Kent-Jones, F.R.I.C., who was accompanied on the platform by Dr. K. A. Williams, F.R.I.C., A.Inst.P., Chairman of the Oils and Fats Group.

The subject of the meeting was "Methods for the Chemical Determination of Vitamin A" and the following papers were presented and discussed: "Comment on the Determination of Vitamin A in Natural Products and especially Cod-liver Oil," by Professor R. A. Morton, Ph.D., D.Sc., F.R.I.C., F.R.S., and F. Bro-Rasmussen, M.Sc.; "Chromatographic Separation of Vitamin-A-active Compounds in Cod-liver Oil," by F. Bro-Rasmussen, M.Sc., W. Hjarde and Olga Porotnikoff; "A Modified Method for the Spectrophotometric Estimation of Vitamin A in Margarine," by J. W. Lord, M.Sc., F.R.I.C., and Pauline M. Bradley, B.Sc.

NEW MEMBERS

Ordinary Members

James Borrowdale, B.Sc. (Lond.); Gareth Samuel Llewellyn, B.Sc. (Wales); Kenneth Neville Reed, B.Sc. (Lond.).

Junior Member

George Russell Nugent.

NORTH OF ENGLAND SECTION AND MICROCHEMISTRY GROUP

A Joint Meeting of the North of England Section, the Microchemistry Group and the Sheffield, South Yorkshire and North Midlands Section of the Royal Institute of Chemistry was held at 7.30 p.m. on Friday, October 15th, 1954, at The University, Sheffield. Dr. D. H. Peacock, M.A., F.R.I.C., Chairman of the Sheffield, South Yorkshire and North Midlands Section of the Royal Institute of Chemistry, presided.

The following papers were presented and discussed: "The Microchemical Determination of Nobium and Tantalum," by A. A. North, B.Sc., A.R.I.C.; "A Statistician's Approach to Sampling Problems," by D. R. Read, B.Sc., A.R.I.C. A film entitled "The Technique of Sampling" was shown by courtesy of Imperial Chemical Industries, Ltd.

SCOTTISH SECTION

An Ordinary Meeting of the Section was held at 7.15 p.m. on Wednesday, October 27th, 1954, in the Central Station Hotel, Glasgow. The Chair was taken by Mr. R. S. Watson, F.R.I.C., and visitors included representatives of the Scottish Beekeepers' Association.

The following papers were presented and discussed: "The Determination of Sodium Carboxymethyl Cellulose," by K. Sporek, M.A., and A. F. Williams, B.Sc., F.R.I.C.; "The Properties of Ling (Heather) Honey," by T. J. Mitchell, Ph.D., A.R.T.C., A.M.I.Chem.E., F.R.I.C.

WESTERN SECTION

An Ordinary Meeting of the Section was held at 2.30 p.m. on Saturday, November 13th, 1954, at the premises of Messrs. Treharne & Davies, Merton House, Bute Crescent, Cardiff. The Chair was taken by Mr. H. J. Evans, B.Sc., F.R.I.C.

The following paper was presented and discussed: "Gas-phase Chromatography as an Analytical Technique," by C. J. Hardy, B.Sc. (see summary below).

Gas-phase Chromatography as an Analytical Technique

Mr. C. J. Hardy described the development of gas-phase chromatography as a technique for the separation and estimation of volatile substances. The basic principles were briefly outlined and apparatus and practical methods of analysis were described in detail. Separations of substances by differential adsorption on, or desorption from, charcoal and similar adsorbents were compared with those obtained by the newer James and Martin technique on gas - liquid partition columns.

Intensive research by many workers during the last two years on the development of apparatus and the semi-micro analysis of gases and liquids was summarised. Examples to illustrate the methods and results were given from the author's own work on the separation and determination of halogenated hydrocarbons, aliphatic hydrocarbons and alkyl esters. Gas-phase chromatography has been shown to be applicable to specific problems such as the analysis of intermediates and final products in gaseous reactions. An example of the analysis of products in a gaseous reaction between ethyl nitrite and nitrogen dioxide was given in detail.

Gas-phase chromatography was compared with fractional distillation and mass and infra-red spectrometry as a technique for the separation of closely related compounds and complex mixtures. Its use for the preparation of certain pure substances was considered. The advantages of gas-phase chromatography over other analytical methods and its potential applications in many fields were discussed.

PHYSICAL METHODS GROUP

The Forty-seventh Ordinary Meeting of the Group was held at 7.15 p.m. on Friday, October 22nd, 1954, at the Physical Chemistry Laboratory, South Parks Road, Oxford. The meeting was held jointly with the London Section of the Royal Institute of Chemistry. The Chairman of the Group, Mr. A. A. Smales, B.Sc., F.R.I.C., presided.

The subject of the meeting was "Radiochemistry" and the following papers were presented and discussed: "Assay Equipment for a Radiochemical Laboratory," by J. E. Johnston, Ph.D. (see summary below); "The Determination of Gamma Isomer in Crude Benzene Hexachloride by a Carbon-14 Isotope Dilution Method," by D. E. Palin, B.Sc., Ph.D., A.Inst.P.; "The Physical and Analytical Control of the Radioactive Effluent from A.E.R.E., Harwell," by R. H. Burns, B.Sc., F.R.I.C. (see summary below). The meeting was preceded by a visit to A.E.R.E., Harwell.

Assay Equipment for a Radiochemical Laboratory

Dr. J. E. Johnston briefly outlined the measurement of radioactive isotopes by ionisation chambers, proportional counters, Geiger counters and scintillation counters. The features of each of these instruments of possible interest to the analytical chemist were described, such as sensitivity, stability and specialised applications, as well as a list (including cost) of the electronic equipment required for each of the measuring methods. Three years' experience in the Isotope School with equipment supplied by the various manufacturers was described.

THE PHYSICAL AND ANALYTICAL CONTROL OF THE RADIOACTIVE EFFLUENT FROM A.E.R.E., HARWELL

MR. R. H. BURNS gave details of the permissible levels of activity in the effluent from the Atomic Energy Research Establishment. These levels were based on the internationally accepted drinking-water tolerances for radioactive isotopes. In order to ensure that the limits laid down were not exceeded, it was necessary to estimate the radium and other alpha emitters, and calcium and strontium and the other beta-active isotopes in the liquid wastes. Details of the methods developed for this analytical control were given.

The main source of activity in the effluent was derived from the isotopes formed during the fission of uranium. These fission products were numerous and very varied in chemical character. Brief details were given of the treatment processes used to decontaminate the effluent before discharge to the river Thames.

Physical control of the waste material was accomplished by the design of special containers, a separate active drainage system and treatment in a plant erected for this purpose. A description was given of the controls exercised at each stage of the disposal system from the laboratories to the Thames.

The Analysis of Analogues

A Development of the General Theory of Partition and its Application to the Determination of Cyanocobalamin and Hydroxocobalamin in Mixtures

BY J. G. HEATHCOTE AND P. J. DUFF

A unique relationship exists between the composition of a mixture of analogues and the apparent partition coefficient, as measured by some common property. When combined with a spectrophotometric assay technique, this relationship provides a quick and easy method of determining the percentages of hydroxocobalamin and cyanocobalamin present in mixtures. There is good agreement between the experimental results and the theoretical values.

METHODS of determination of cyanocobalamin, in its mixtures with hydroxocobalamin, by spectrophotometric assay at 361 mμ suffer from the serious disadvantage that the chief absorption peaks of the two vitamins are close together.[1,2,3] A method recently described[4] is based on the destruction of hydroxocobalamin by heating it with ascorbic acid, the residual cyanocobalamin being assayed microbiologically. The accurate determination of small percentages of the hydroxo analogue in this way, however, requires tedious replication. The majority of samples of vitamin B_{12} (cobalamin) prepared commercially for clinical use should be capable of more precise and simple physico-chemical determination provided only that the property measured discriminates sufficiently between these different forms of the vitamin. The partition coefficient between a suitable solvent and water has been used for the detection of traces of hydroxocobalamin in cyanocobalamin,[5] and a preliminary application of this property to the quantitative determination of either component in their mixtures has now been reported.[6]

The technique of partition and analysis of the separated phases is, of itself, not new. It was applied, for instance, by Werkman *et al.*[7,8,9,10] to the determination of two or more acids together in solution and also by Wright and Grove[11] to the determination of penicillins K and G. In this paper, however, the theory of partition has been developed in a novel way to give an exact formula capable of general application to the determination of analogues in their mixtures. The only assumption that has been made, apart from the conditions usually considered necessary for the determination of partition coefficients, is that each component of the mixture is distributed independently of any other component. The following theoretical treatment deals with mixtures of two components only, a similar, although slightly more complicated, treatment being applicable to mixtures of three components.

THEORETICAL CONSIDERATIONS

Let x g of A and y g of B be distributed between V_S litres of solvent (saturated with water) and V_W litres of water (saturated with solvent). Let the equilibrium concentrations in the two phases be $[A]_S$, $[A]_W$, $[B]_S$ and $[B]_W$, where the subscripts refer to the phases.

If the two compounds A and B are not associated (or dissociated) in solution, the partition coefficients will, generally, be constant and independent of concentration at low concentrations. If, in the mixture, they are also partitioned independently, then—

$$[A]_S = K_A[A]_W \text{ and } [B]_S = K_B[B]_W,$$

but $x = V_S[A]_S + V_W[A]_W = (V_SK_A + V_W)\,[A]_W$

hence, $[A]_W = \dfrac{x}{V_SK_A + V_W}$ and $[A]_S = \dfrac{K_A\,x}{V_SK_A + V_W}$.

Similarly, $[B]_W = \dfrac{y}{V_SK_B + V_W}$ and $[B]_S = \dfrac{K_By}{V_SK_B + V_W}$.

The apparent partition coefficient for the mixture is defined as—

$$K = \frac{\text{Concentration of mixed solute in solvent}}{\text{Concentration of mixed solute in water}} = \frac{[A]_S + [B]_S}{[A]_W + [B]_W}$$

$$= \frac{\dfrac{K_A\,x}{V_SK_A + V_W} + \dfrac{K_B\,y}{V_SK_B + V_W}}{\dfrac{x}{V_SK_A + V_W} + \dfrac{y}{V_SK_B + V_W}}.$$

If the percentages of A and B in the original mixture are, respectively—

$$X = \frac{100x}{x + y} \text{ and } Y = \frac{100y}{x + y},$$

$$\text{then } K = \frac{\dfrac{K_AX}{\dfrac{V_S}{V_W}K_A + 1} + \dfrac{K_BY}{\dfrac{V_S}{V_W}K_B + 1}}{\dfrac{X}{\dfrac{V_S}{V_W}K_A + 1} + \dfrac{Y}{\dfrac{V_S}{V_W}K_B + 1}}$$

from which it will be seen that, for a given solvent to water ratio, there is a unique relationship between the apparent partition coefficient and the composition of the original mixture.

Previously,[5] values were reported for the partition coefficients of cyanocobalamin and hydroxocobalamin between benzyl alcohol and water at 21° to 22° C and at pH 6·2 of 0·78 and 0·055, respectively. From these values (or other values determined under other standard conditions) the apparent partition coefficient, K, can be calculated for any given mixture. These calculated values of K can be plotted against the percentage of one or other cobalamin to give a curve from which other values can be interpolated.

EXPERIMENTAL

ANALYSIS—

All analyses were conducted in subdued light. A Unicam SP500 quartz spectrophotometer was used.

As the determination of the apparent partition coefficient in the present method is based on spectrophotometric absorption, the determination of the total cobalamin in each phase must be performed at the wavelength at which both components possess the same extinction. This (isobestic) point was determined experimentally to be at 355·6 mμ, but it was found that no appreciable error was introduced if measurements were made at 356 mμ. At this wavelength the specific extinction coefficient for each cobalamin was found to be 174. The same value was determined for cyanocobalamin in the solvent phase. The exact determination of the specific extinction coefficient of hydroxocobalamin in the solvent phase proved to be more difficult owing to the extremely slow dissolution of the compound. The determination is complicated further by a slow but gradual decomposition of the vitamin as it dissolves, even in the absence of light, owing no doubt to traces of some reducing agent (possibly benzaldehyde) from which it is difficult to free the benzyl alcohol entirely. However, provided that readings are taken reasonably soon ($\frac{1}{2}$ to 1 hour) after preparing the solution, the value for $E^{1\%}_{1\,cm}$ at 356 mμ (160) is not much different from that (174) obtaining in the aqueous phase. In practice, the low solubility of hydroxocobalamin in the solvent phase ensures that no significant error arises from this source during the determination of the partition coefficient. The total cobalamin concentration in each phase is evaluated by $\frac{\text{E at 356}}{0{\cdot}0174}$ μg per ml and the apparent distribution coefficient for any particular mixture, therefore, is given experimentally by—

$$K = \frac{\text{E at 356 (solvent)}}{\text{E at 356 (water)}}$$

As the isobestic point lies on the sides of the respective wave bands of the two vitamins, any shift in the calibration of the spectrophotometer such as might result from variations in ambient temperature could, theoretically, be important with certain mixtures. The calibration should, therefore, be checked from time to time on the peak of the cobalamin band at 361 mμ, a linear correction for any shift being then applied, if necessary. In practice the results at each end of the binary concentration range were remarkably good (see Table I).

DETERMINATION OF THE APPARENT PARTITION COEFFICIENT—

Solutions were first assayed for their total cobalamin content at 356 mμ, a suitable concentration range for accuracy being 50 to 80 μg per ml. Three 20-ml aliquots of the solution to be assayed were transferred by pipette into 100-ml separating funnels; the solutions were shaken for two minutes with 20 ml of dry benzyl alcohol and allowed to stand for 5 minutes. The shaking and standing were repeated twice to ensure equilibration of the phases. Then 15 ml of the lower, solvent, phase were transferred to a centrifuge tube and 15 ml of the aqueous phase to another tube. After centrifugation, 4 ml of each phase were transferred by pipette to test tubes and clarified with 1 ml of ethanol. The optical densities of the two phases were read at 356 mμ against the appropriate blanks, the temperature being maintained in the range 20° to 22° C. Solid samples were more conveniently dissolved in a measured quantity of water that had previously been brought to equilibrium with benzyl alcohol, the solvent then used being benzyl alcohol equilibrated with water. In this way, the correction to find the true ratio of the phase volumes was obviated. The effect of alteration in the phase to volume ratio is discussed below. When a ratio of unity is used, solutions containing about 20 μg of mixed vitamin per ml are approaching the lower limit of the method for accuracy and at this concentration the extinction of the benzyl alcohol becomes the limiting factor.

PURITY OF THE BENZYL ALCOHOL—

Commercial benzyl alcohol frequently contains both benzaldehyde and benzoic acid, the former having some absorption at around 360 mμ. Distillation, even under reduced pressure, did not always produce the desired reduction in optical density, but a simple washing technique proved to be quite effective. The benzyl alcohol was shaken twice with a saturated solution of sodium bisulphite and then, after washing with water, with a 15 per cent. w/v solution of sodium bicarbonate. The benzyl alcohol was finally washed well with distilled water. When low concentrations of the vitamin (20 μg per ml or less) are being examined, the extinction, $E_{1\,cm}$ at 356 mμ, of the benzyl alcohol should not exceed 0·1 and when, in addition, the samples contain high percentages of hydroxocobalamin, this figure should not be greater than 0·05.

RESULTS AND DISCUSSION

Two solutions, one of cyanocobalamin and the other of hydroxocobalamin, were made in distilled water to exactly the same concentration (80 μg per ml). From these standards, a series of solutions of mixed cobalamins was prepared. It will be seen from Table I that there is excellent agreement between the experimentally determined values of the apparent partition coefficient and the theoretically derived values.

TABLE I

ANALYSIS OF CYANOCOBALAMIN AND HYDROXOCOBALAMIN IN MIXTURES

Cyanocobalamin taken in mixture, %	Partition coefficient		Cyanocobalamin found in mixture, %
	Found	Calculated*	
90	0·667	0·662	90·4
80	0·550	0·559	79·0
70	0·472	0·469	70·2
60	0·388	0·389	59·9
50	0·320	0·318	50·4
40	0·267	0·254	42·2
30	0·195	0·197	29·6
20	0·147	0·145	20·2
10	0·096	0·098	9·8

* Equal volumes of dry benzyl alcohol and water were taken; under these conditions $V_S/V_W = 1{\cdot}11$.

As hydroxocobalamin is a weak base,[12] its partition coefficient is affected by alterations in pH (see, for example, Smith, Ball and Ireland[13]) and, if the determinations are performed under different conditions, the partition coefficients of the pure vitamins should be re-determined. This is, however, of little practical significance over the pH range 3 to 6·5 unless the concentration of the hydroxocobalamin in the mixture is greater than 50 per cent. The effect of salt concentration on the partition ratios is worth further investigation in view of the modern tendency to make up clinical samples in this way.

Throughout the foregoing work, a phase volume ratio (V_S/V_W) of unity—or unity corrected for mutual solubility of the solvents—has been chosen for simplicity and convenience. Bush and Deusen[14] deduced from empirical considerations, however, that in order to get each solute well separated from the other V_S/V_W should be adjusted so that the fraction of solute A in the solvent phase is equal to the fraction of solute B in the aqueous phase, then—

$$\frac{V_S}{V_W} = \sqrt{\frac{1}{K_A K_B}}.$$

Applying this to cyanocobalamin and hydroxocobalamin under the present conditions, the most efficacious phase volume ratio is that for which $V_S/V_W = \sqrt{\frac{1}{0{\cdot}78 \times 0{\cdot}055}}$ or about 5 to 1. This factor can be made use of with advantage when the concentration of hydroxocobalamin is low.

In practice, the method has proved useful in checking the purity of samples of cyanocobalamin and in determining the percentage of hydroxocobalamin in samples of theoretical purity as judged by the standard method of assay for cyanocobalamin.[15,16] The latter method, however, is not strictly applicable in the presence of hydroxocobalamin. Elsewhere,[5] attention has been drawn to the anomalous position of this analogue, which, despite similar clinical activity[17,18,19] and freedom from toxicity, is nevertheless excluded from the pharmacopoeias of both America and Britain.

The authors thank the Directors of the Distillers Company (Biochemicals) Limited for permission to publish.

REFERENCES

1. Kaczka, E., Wolf, D. E., and Folkers, K., *J. Amer. Chem. Soc.*, 1949, **71**, 1514.
2. Veer, W. L. C., Edelhauser, J. H., Wijmenga, H. G., and Lens, J., *Biochim. Biophys. Acta*, 1950, **6**, 225.
3. Pierce, J. V., Page, A. C., Stokstad, E. L. R., and Jukes, T. H., *J. Amer. Chem. Soc.*, 1949, **71**, 2952.
4. Campbell, J. A., McLaughlan, J. M., and Chapman, D. G., *J. Amer. Pharm. Ass., Sci. Ed.*, 1952, **41**, 479.
5. Heathcote, J. G., *J. Pharm. Pharmacol.*, 1952, **4**, 641.
6. ——, *Chem. & Ind.*, November 7th, 1953, No. 45, 1203.
7. Werkman, C. H., *Ind. Eng. Chem., Anal. Ed.*, 1930, **2**, 302.
8. Osburn, O. L., and Werkman, C. H., *Ibid.*, 1931, **3**, 264.
9. Osburn, O. L., Wood, H. G., and Werkman, C. H., *Ibid.*, 1933, **5**, 247.
10. ——, ——, ——, *Ibid.*, 1936, **8**, 270.
11. Wright, W. W., and Grove, D. C., *J. Amer. Pharm. Ass., Sci. Ed.*, 1948, **37**, 115.

12. Cooley, G., Ellis, B., Petrow, V., Beaven, G. H., Holiday, E. R., and Johnson, E. A., *J. Pharm. Pharmacol.*, 1951, **3**, 607.
13. Smith, E. L., Ball, S., and Ireland, D. M., *Biochem. J.*, 1952, **52**, 395.
14. Bush, M. T., and Deusen, P. M., *Anal. Chem.*, 1948, **20**, 121.
15. British Pharmacopoeia, 1953, p. 173.
16. United States Pharmacopoeia, Fourteenth Edition, 1950, p. 660.
17. Lichtman, H., Watson, J., Ginsberg, V., Pierce, J. V., Stokstad, E. L. R., and Jukes, T. H., *Proc. Soc. Exp. Biol. Med.*, 1949, **72**, 643.
18. Schilling, R. F., Harris, J. W., and Castle, W. B., *J. Haematol.*, 1951, **6**, 228.
19. Meyer, L. M., Sawitsky, A., Ritz, N. D., and Brahin, C., *Ibid.*, 1953, **8**, 358.

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February 25th, 1954

The Determination of Lead in Cocoa with a Square-wave Polarograph

BY D. J. FERRETT, G. W. C. MILNER AND A. A. SMALES

The rapid determination of less than 1 p.p.m. of lead in a sample of cocoa is described. The residues produced when 2 to 3-g samples of cocoa are ashed at 500° C are dissolved in hydrochloric acid and the lead in solution is determined directly by means of a square-wave polarograph. The blanks in this procedure are equivalent to 0·005 p.p.m. or less of lead. Although the blanks are greater when a 0·5-g sample of cocoa is wet oxidised with nitric and perchloric acids, the results confirm those determined by the dry-ashing method.

THE standard techniques for the determination of lead in foodstuffs require the use of considerable quantities of a large number of reagents. This is especially true when wet-oxidation techniques are used. The problem of controlling the amount of lead in the reagents used makes difficult the determination of 1 p.p.m. or less of lead. A sample of cocoa has been recently submitted to these techniques by a number of analysts and the blanks in the determinations were almost as great as the lead content reported.[1]

One method of overcoming these difficulties would be to dry-ash the cocoa, dissolve the ash in hydrochloric acid and determine the lead polarographically. Unfortunately, unless large quantities of sample are taken, normal polarographic techniques are insufficiently sensitive for this method. Barker and Jenkins[2] have shown recently, however, that concentrations of reversibly reducible metal ions of about 10^{-7} M can be determined with a square-wave polarograph. Such determinations have the additional advantage over normal polarographic techniques that they are not so seriously interfered with by the presence of ions with more positive half-wave potentials. During an investigation of the applications of square-wave polarography in analytical chemistry, we determined the lead in this cocoa sample.

EXPERIMENTAL

REAGENTS—

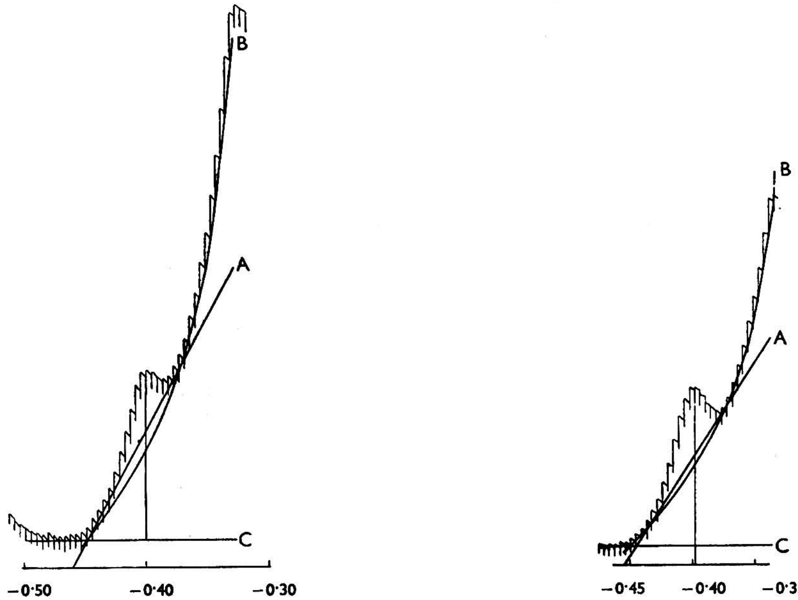
Nitric acid, 70 *per cent. and perchloric acid*, 60 *per cent.*—These acids were the B.D.H. "lead-free" grade, *i.e.*, they contained less than 0·005 p.p.m. of lead.

Redistilled water—This was further purified by passage through a mixed-bed ion-exchange column followed by a further distillation from potassium permanganate solution, the final product being collected in polythene containers. (Water from the column was found initially to give no lead step on the square-wave polarograph. When it was boiled with lead-free nitric acid, however, a lead step appeared. Some lead presumably passed out of the column in the form of an organic complex that was not detectable with the square-wave polarograph, this complex being decomposed on boiling with acid.)

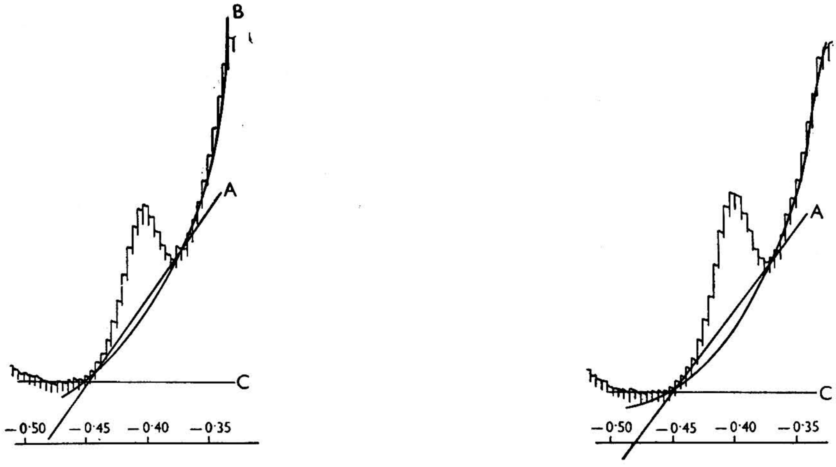
Hydrochloric acid—A constant-boiling mixture, distilled into a polythene container from a (1 + 1) mixture of the purified water and Polaritan hydrochloric acid.

PROCEDURE FOR DRY OXIDATION—

The sample of cocoa was dried at 105° C for three hours and about 3 g were weighed into a flat-bottomed silica dish of 7 cm diameter, which had been previously cleaned by fusing potassium bisulphate in it followed by treatment with boiling nitric acid. This cleaning treatment was found to be essential for removing the last traces of lead from the surface of the silica. The cocoa was then heated in a muffle furnace controlled at 500° C. A good supply of air was maintained during the combustion. A silica plate was held 1 cm above the dishes to prevent entry of dust. After the sample had been heated for an hour, a few drops of nitric acid (totalling less than 0·5 ml) were added to aid the oxidation of the last traces of



(*a*) Potential against mercury-pool anode, volts (*b*) Potential against mercury-pool anode, volts



(*c*) Potential against mercury-pool anode, volts (*d*) Potential against mercury-pool anode, volts

Fig. 1. Current - voltage curves: (*a*) for 2-ml aliquot of solution from dry-ashed cocoa (sample D in Table II). Lines A, B and C represent three possible base lines (see text). (*b*), (*c*) and (*d*) are similar curves with 0·1, 0·3 and 0·5 ml, respectively, of standard lead solution, 1·11 μg per ml, added

organic matter. The cocoa was then reheated at 500° C until the residue was colourless (usually for a further 20 minutes). After the dish had cooled, 1 ml of hydrochloric acid and 1 ml of water were added, and the ash was dissolved by gentle warming. The solution was then washed into a 10-ml calibrated flask. An identical procedure was carried out simultaneously on an empty dish to serve as a blank. Then 2 ml of the solution were transferred by pipette to the polarographic cell. Nitrogen was bubbled through the cell for 10 minutes and the current - voltage curves were recorded automatically between — 0·30 and —0·50 volt against the mercury-pool anode.

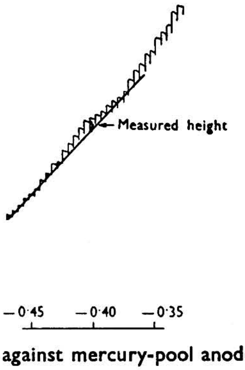


Fig. 2. Current - voltage plot for blank solution; sensitivity is 4 times greater than in Figs. 1 (*a*), (*b*), (*c*) and (*d*)

A typical polarogram for a cocoa sample (D in Table II) is shown in Fig. 1*a*. The derivative nature of the instrument produces a peak that is superimposed on a steeply falling background owing to the reduction of an impurity with a more positive half-wave potential. This peak height has to be corrected for the background current, and there are a number of methods by which this correction may be estimated.

Line A represents the best straight line that can be drawn through the nearest points on both sides of the peak.

Line B represents a fitted standard curve intercepting the maximum number of points on both sides of the peak.

Line C represents the background current when there is no overlap from the preceding peak.

Figs. 1*b*, 1*c* and 1*d* show the effect of additions of 0·1 ml, 0·3 ml and 0·5 ml of a standard lead solution containing 1·11 μg of lead per ml to the cocoa sample. From Table I, which shows the corrected increments, it can be seen that Line A gives the most consistent incremental results.

TABLE I

EQUIVALENT INCREMENTS OF PEAK HEIGHT

Scale divisions per 0·1 ml of standard lead solution (corrected for increase in volume)

Line A	Line B	Line C
2·36	1·82	−0·95
2·51	2·08	+0·69
2·55	2·26	+1·50

Fig. 2 shows a typical polarogram of a blank solution. This has been recorded at 4 times the sensitivities selected for the polarograms reproduced in Fig. 1, as interferences in the blank solution are considerably less.

Results obtained by the technique described above are shown in Table II.

In view of the agreement between the above four results and as the blanks were only about 5 per cent. of the total lead found, it was considered that the figure of 0·69 p.p.m. represented the true lead content of the cocoa, but this will only be correct if no lead is lost during the dry ignition. Such losses, *e.g.*, by volatilisation, have been suggested in the literature,[3] and it was therefore decided to confirm this value for lead by the use of a wet-oxidation technique to destroy the organic matter present. With such a technique, there is no risk of loss of lead by volatilisation, but the lead in the blanks would be much higher than the values reported in Table I, as relatively large volumes of acid are necessary for the wet oxidation. However, these quantities were kept to a minimum by decreasing the sample weight to 0·5 g.

TABLE II

RESULTS FOR LEAD IN COCOA BY A DRY-IGNITION TECHNIQUE

Sample	Weight of cocoa taken, g	Total lead found, p.p.m.	Lead in blank, p.p.m.	Lead in cocoa, p.p.m.
A	4·30	0·67	0·03	0·64
B	2·90	0·73	0·05	0·68
C	2·90	0·76	0·03	0·73
D	2·90	0·76	0·05	0·71
			Average value =	0·69

PROCEDURE FOR WET OXIDATION—

A 0·5-g sample of the dried cocoa was weighed into a 250-ml Hysil-glass beaker, 2 ml of perchloric acid and 3 ml of nitric acid were added and the solution was heated. Entry of dust was prevented by a watch-glass. When fumes of perchloric acid appeared in the beaker, the oxidation proceeded rapidly and it proved necessary to make further additions of nitric acid (totalling about 7 ml) at this stage to prevent carbonisation. The solution was then evaporated to dryness. The residue was coloured, indicating that the oxidation was incomplete. Then 2 ml of nitric and 2 ml of perchloric acids were added, the sides of the beaker being washed with these acids to remove any condensed organic vapours, and the solution was again evaporated to dryness. After this second evaporation the residue was colourless. Then 1 ml of hydrochloric acid and 1 ml of water were added and the residue was dissolved by gentle warming. The solution was washed into a 10-ml calibrated flask and polarograms were recorded as described above. An idential procedure was carried out simultaneously upon an empty beaker to serve as a blank.

TABLE III

RESULTS FOR LEAD IN COCOA BY WET-OXIDATION TECHNIQUE

Weight of cocoa taken, g	Total lead found, p.p.m.	Lead in blank, p.p.m.	Lead in cocoa, p.p.m.
0·50	1·17	0·45	0·7(2)
0·50	1·12	0·38	0·7(4)
		Average value =	0·7(3)

CONCLUSIONS

About 1 p.p.m. of lead in cocoa can be determined rapidly and reasonably accurately after dry ignition, by means of a square-wave polarograph. The small quantities of reagents needed make the blanks in such determinations extremely small compared with standard methods of lead analysis.

The confirmation of these results, by the wet-oxidation technique, proves the reliability of the dry-oxidation method. The agreement between the two sets of results shows that lead losses from the cocoa sample heated at 500° C are negligible.

REFERENCES

1. Analytical Methods Committee: Report of the Lead Panel, *Analyst*, 1954, **79**, 397.
2. Barker, G. C., and Jenkins, I. L., *Ibid.*, 1952, **77**, 685.
3. See, for example, Lynch, G. Roche, *Ibid.*, 1954, **79**, 137.

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An Impurity Compensated Polarographic Method for the Determination of the Gamma Isomer in Technical Benzene Hexachloride

BY J. WATT

A method is described for the determination of the biologically active gamma isomer in technical benzene hexachloride. The apparent amount of gamma isomer in the material is determined by a normal polarographic procedure, and a correction is made for interference by impurities present in the technical material. This correction is obtained from a polarographic comparison of two saturated solutions of the gamma isomer. One solution contains only the pure isomer and the other, being prepared from a known weight of the technical sample together with an excess of the gamma isomer, contains in addition those impurities that interfere with the determination of the apparent gamma isomer content. The results agree closely with those by other methods, including chromatographic, infra-red and isotope-dilution analysis. The precision of a single determination is about ±5 per cent. expressed as 95 per cent. confidence limits. The method is applicable to any technical mixture of benzene hexachloride.

OF the methods available for the determination of the gamma isomer of benzene hexachloride (γ-hexachloro*cyclo*hexane), polarographic analysis[1] is one of the most convenient for routine work. However, it has been recognised for some time that the polarographic determination of the gamma isomer in technical material gives high results owing to the presence of impurities that produce polarographic waves that partly coincide with the wave for the gamma isomer. Several methods[2,3,4,5,6] have been proposed for eliminating this interference, each method being based on the behaviour of certain isomers of heptachloro- and octachloro-*cyclo*hexanes that have been identified in the crude product. Although these substances are probably principally responsible for the interference, no evidence has been recorded to support this, nor has the possible effect of other impurities been considered.

The method described in this paper consists of two determinations, one of the apparent gamma isomer by a straightforward polarographic technique and the other of the total interference due to impurities normally present in the technical material. Besides being suitable for crude benzene hexachloride, the method is also applicable, with some slight modifications, to other grades of technical material. The types of technical material for which it has been used are as follows—

Crude benzene hexachloride, which contains 8 to 15 per cent. of gamma isomer and 50 to 70 per cent. of alpha isomer.

Gamma-isomer concentrate, which contains 20 to 65 per cent. of gamma isomer and 10 to 30 per cent. of alpha isomer.

Gamma benzene hexachloride, which contains 80 to 100 per cent. of gamma isomer.

Alpha - beta residues, which contain 0 to 2 per cent. of gamma isomer and 90 to 100 per cent. of alpha isomer.

The supporting medium used for polarographic examination is that recommended by Ingram and Southern,[1] and consists of a solution of 1 per cent. of potassium iodide and 0·005 per cent. of gelatin in a mixture of equal volumes of absolute ethanol and water. This was also found to be a suitable solvent for use in assessing the interference by impurities. The measurement of the polarographic wave for the gamma isomer is performed by observing the diffusion current at two fixed potentials, and thus the determination may be made with a simple and inexpensive manually operated instrument.

In the determination of the apparent amount of gamma isomer in crude benzene hexachloride and alpha - beta residues, it has been shown that certain precautions must be taken to ensure that there is complete extraction of the gamma isomer and interfering impurities and furthermore that allowance must be made for effects due to the presence of a preponderance of the alpha isomer, which, in the solvent used, is considerably less soluble than the gamma isomer.

The underlying principle of the method by which the interference due to impurities is assessed is that described by Thorp[7]; this method was subsequently used by Willermain[8] in a gravimetric - solubility method for the isomeric analysis of benzene hexachloride. If an excess of gamma isomer is thoroughly shaken with the above supporting medium, at a controlled temperature, and the resulting solution is examined polarographically, a wave for the gamma isomer is observed, of height corresponding to a saturated solution of the gamma isomer. If a second solution, prepared by shaking an excess of gamma isomer and also a small but known quantity of technical benzene hexachloride with the supporting medium, is then examined, the height of the wave for the gamma isomer exceeds that of the first solution by an amount proportional to the interfering impurities dissolved from the technical material.

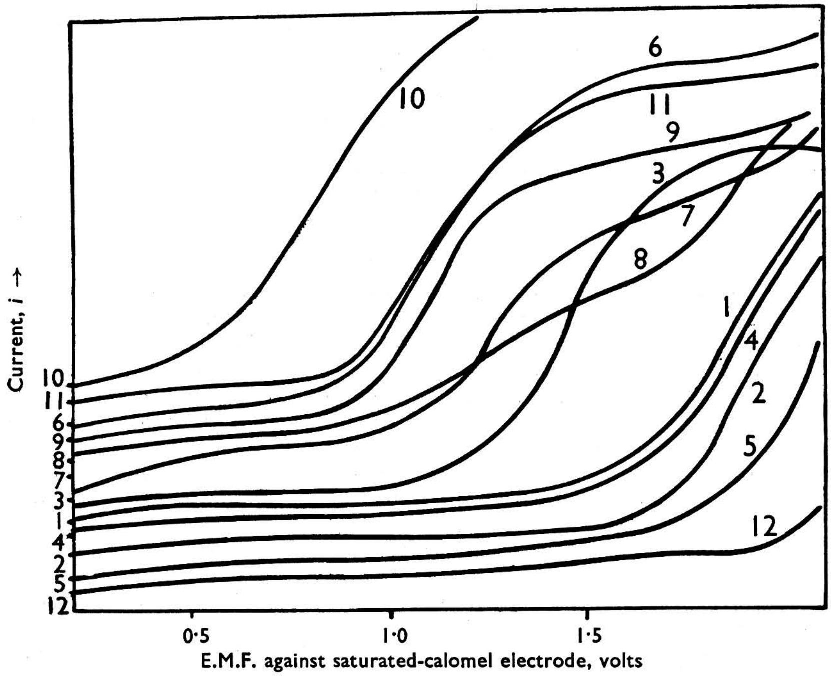

Fig. 1. Polarograms of the isomers of benzene hexachloride (hexachloro*cyclo*hexane) and related compounds as 0·01 per cent. w/v solutions in methanol, A.R., as solvent, with 0·05 *M* tetramethylammonium bromide as supporting electrolyte. Curve 1, alpha benzene hexachloride; curve 2, beta benzene hexachloride; curve 3, gamma benzene hexachloride; curve 4, delta benzene hexachloride; curve 5, epsilon benzene hexachloride; curve 6, heptachloro*cyclo*hexane (m.p. 85° C); curve 7, heptachloro*cyclo*hexane (m.p. 146° C); curve 8, heptachloro*cyclo*hexane (m.p. 155° C); curve 9, heptachloro*cyclo*hexane (m.p. 260° C); curve 10, octachloro*cyclo*hexane (m.p. 147° C); curve 11, octachloro*cyclo*hexane (m.p. 261° C); curve 12, supporting medium

The efficacy of this method of assessing the total interference by impurities is based on two assumptions: first, that the solubility of the gamma isomer is unaffected by the presence, in solution, of other isomers and interfering impurities and, secondly, that this interference is linearly proportional to the amount of such impurities present in the sample of technical benzene hexachloride. Experimental results confirming these assumptions are considered below.

EXPERIMENTAL

THE POLAROGRAPHIC BEHAVIOUR AND SOLUBILITIES OF THE ISOMERS OF BENZENE HEXACHLORIDE AND OTHER RELATED SUBSTANCES—

As has been pointed out by Hasselbach and Schwabe,[4] if a suitable supporting electrolyte is selected, all the isomers of benzene hexachloride give observable polarographic waves. In Fig. 1 the waves of these isomers and other substances believed to be present in technical benzene hexachloride are shown. The figure shows that, when examining a mixture of these materials, it is not possible to select two potentials between which the height of wave for the gamma isomer is unaffected by the waves of the other constituents. Even if such a mixture is examined by a derivative technique, *i.e.*, by measuring the slope or first derivative of the normal polarographic wave, the interference by impurities has been shown to be only partly eliminated, which indicates that this interference occurs over the whole range of the wave for the gamma isomer.

It has been found that, by the use of certain supporting media other than that used in the determination of the polarograms shown in Fig. 1, the relative positions of the polarograms remain unchanged. In order to assess the correction for impurities by the proposed technique, it is desirable to use a supporting medium in which a saturated solution of the gamma isomer gives a measurable polarographic wave; 50 per cent. v/v aqueous ethanol containing 1 per cent. w/v of potassium iodide as electrolyte and 0·005 per cent. w/v of gelatin as maximum suppressor was found to fulfil this requirement, the solubility of the gamma isomer in this solvent being approximately 0·067 per cent. at 20° C. With potassium iodide as supporting electrolyte, the waves for alpha, beta and delta benzene hexachloride are masked by the potassium wave, but the wave for the gamma isomer is not affected in this way. Unless gelatin is present in solutions containing more than 0·01 per cent. of gamma isomer, pronounced polarographic maxima are usually observed. A mercury-pool anode has been found to be suitable as the reference electrode. It has been shown that dissolved oxygen, the polarographic wave of which interferes with that for the gamma isomer, can be adequately removed by bubbling nitrogen, previously saturated with solvent vapour, through the cell solution at a rate of 2 litres per hour for several minutes.

Of the available methods for the measurement of the wave for the gamma isomer, the method in which the difference, Δi, in diffusion current between two fixed potentials, $-0{\cdot}8$ volt and $-1{\cdot}1$ volts against a mercury-pool anode, is determined was chosen as being the most convenient and possibly the most precise method. The precision of this method, when a simple manual polarograph is used, has been calculated from observed values of Δi for ten or more successive measurements of the wave for the gamma isomer given by different portions of the same standard solution of gamma isomer and has been found to be better than ± 2 per cent., expressed as 95 per cent. confidence limits for a single measurement.

The solubilities at 20° C of the various substances referred to in Fig. 1 have been determined in the supporting medium by a polarographic technique. Expressed as per cent. w/v, they are approximately as follows—

Benzene hexachloride isomers: alpha, 0·01; beta, <0·005; gamma, 0·07; delta, 0·16; epsilon not determined.
Heptachloro*cyclo*hexanes: m.p. 146° C, 0·04; m.p. 261° C, <0·005; m.p. 155° C, 0·10; m.p. 85° C, 0·05.
Octachloro*cyclo*hexanes: m.p. 147° C, 0·01; m.p. 260° C, <0·005.

THE DETERMINATION OF THE APPARENT GAMMA-ISOMER CONTENT OF TECHNICAL BENZENE HEXACHLORIDE—

Basically the method by which the apparent gamma-isomer content of a sample of technical benzene hexachloride is assessed is to compare polarographically a solution of the crude material with a standard solution of the gamma isomer. Certain factors must be considered in dealing with the various grades of technical material.

Crude benzene hexachloride—Owing to the low solubility of the alpha isomer (approximately 0·01 per cent. w/v) compared with that of the gamma isomer (approximately 0·07 per cent. w/v) in a mixture of equal volumes of ethanol and water and the fact that most crude materials contain about four times as much alpha as apparent gamma isomer, it is impracticable to examine crude benzene hexachloride as a solution in which all the alpha isomer is dissolved. If, therefore, all the alpha isomer is not to be in solution, either the gamma isomer in the crude material must be extracted with the supporting medium or the sample must first be completely dissolved in ethanol and the aqueous supporting electrolyte and maximum suppressor added subsequently, when the alpha isomer is gradually precipitated. The latter procedure has been found to be the more convenient and effective, but it has been shown conclusively by examination of the precipitated alpha isomer that a significant amount of gamma isomer is co-precipitated and that the quantity of gamma isomer removed from solution in this way depends on both the amount of alpha isomer precipitated and the concentration of gamma isomer in solution. Not only the gamma isomer but also the interfering impurities are co-precipitated to a small extent by this method. When 0·1 g of crude benzene hexachloride was dissolved in 50 ml of ethanol and an equal volume of the aqueous part of the supporting medium was added, it was found that the alpha isomer that was precipitated overnight contained as much as one-tenth of the apparent amount of gamma isomer in the sample.

This effect is counteracted to a certain extent by the contribution that the alpha isomer makes to the wave for the gamma isomer, a contribution that for reasons described below is not included in the correction for impurities. The contribution to Δi made by 0·01 to 0·015 per cent. w/v of alpha isomer (*i.e.*, as is present in a saturated or slightly supersaturated solution of the alpha isomer) was found to be equivalent to a concentration of gamma isomer of approximately 0·0002 per cent. w/v. For example, when the value of Δi of a 0·01 per cent. standard solution of gamma isomer was 100 arbitrary units, that of the supporting medium was approximately 7 units and that of a saturated solution of alpha isomer was 9 units. It was established that this contribution by the alpha isomer to the wave for the gamma isomer was due to the alpha isomer and not to a small amount of gamma isomer present as impurity in the alpha isomer used.

With a solution of suitable concentration of the crude benzene hexachloride sample, it was found possible to achieve almost complete compensation of the opposing errors due to co-precipitation and the contribution that the alpha isomer makes to the wave for the gamma isomer. However, in the method described below, it is recommended that the standard solution of gamma isomer (0·01 per cent. w/v), with which the solution of crude gamma isomer (0·07 per cent. w/v) is compared, be prepared with the appropriate amount of alpha isomer (0·04 per cent. w/v) present.

Gamma-isomer concentrate and gamma isomer—Unlike crude benzene hexachloride, these materials may conveniently be examined as solutions in which the entire sample remains dissolved. Difficulties due to the precipitation of alpha isomer do not arise. A suitable concentration of gamma isomer was found to be about 0·02 per cent. w/v, the standard solution of gamma isomer being exactly of this concentration.

Alpha - beta residues—In order to prepare a solution of alpha - beta residues containing a suitable concentration of gamma isomer (0·01 per cent. w/v), it is necessary to dissolve 0·6 g of material in 50 ml of ethanol and to add to this solution 50 ml of the aqueous part of the supporting medium, when most of the alpha and beta isomers in the residue are precipitated. Owing to co-precipitation of the gamma isomer, the recovery of this isomer is only 80 to 90 per cent. To compensate for this effect, the standard solution of the gamma isomer with which the sample solutions are compared is prepared with an appropriate amount of alpha isomer present.

DETERMINATION OF THE CORRECTION FOR INTERFERING IMPURITIES—

Crude benzene hexachloride—As stated above, the proposed method for assessing the total interference by impurities is based on the validity of two assumptions, the first of which is that the solubility of the gamma isomer in the chosen supporting medium is unaffected by the presence, in solution, of other isomers and related substances. It was shown that, within the limits of experimental error, the solubility of the gamma isomer (approximately 0·067 per cent. w/v) was unaffected by the presence of the following concentrations of interfering impurities—

> 0·05 per cent. w/v of delta benzene hexachloride; 0·05 per cent. w/v of heptachloro*cyclo*hexane, m.p. 155° C; 0·05 per cent. w/v of heptachloro*cyclo*hexane, m.p. 85° C; and a mixture of 0·16 per cent. w/v of delta benzene hexachloride and 0·12 per cent. w/v of crude heptachloro*cyclo*hexane (*i.e.*, chlorinated monochlorobenzene).

The second assumption is that the interference by impurities is linearly proportional to the amount present in the quantity of crude benzene hexachloride taken. Various amounts of finely ground and sieved (44 B.S.S.) crude benzene hexachloride, excess (approximately 0·04 g) of pure gamma isomer and 10 ml of supporting medium were sealed into 6-inch × $\frac{3}{4}$-inch test tubes and the mixture was shaken overnight at 20° C. The values of Δi for the waves of the apparent gamma isomer of the resulting solutions saturated with gamma isomer were determined and are shown in Fig. 2.

For each set of results the best straight line was calculated; this is shown in Fig. 2. From the equations of these straight lines ($y = c + mx$), the percentage interference due to impurities was calculated by means of the expression—

$$\text{Interference, \%} = \frac{m \times \text{Concentration of gamma isomer standard} \times 10}{\Delta i\ (\text{gamma isomer standard} - \text{blank})}$$

and was found to be as follows—

A, line of best fit, $y = 40 + 230x$: interference = 3·9 per cent.
B, line of best fit, $y = 40 + 200x$: interference = 3·4 per cent.
C, line of best fit, $y = 40 + 165x$: interference = 2·8 per cent.
D, line of best fit, $y = 40 + 5x$: interference = 0·1 per cent.

For the second assumption to be valid, it is necessary that the amount of each interfering impurity present in the quantity of crude benzene hexachloride used to determine the percentage interference should not exceed its solubility in the volume of supporting medium

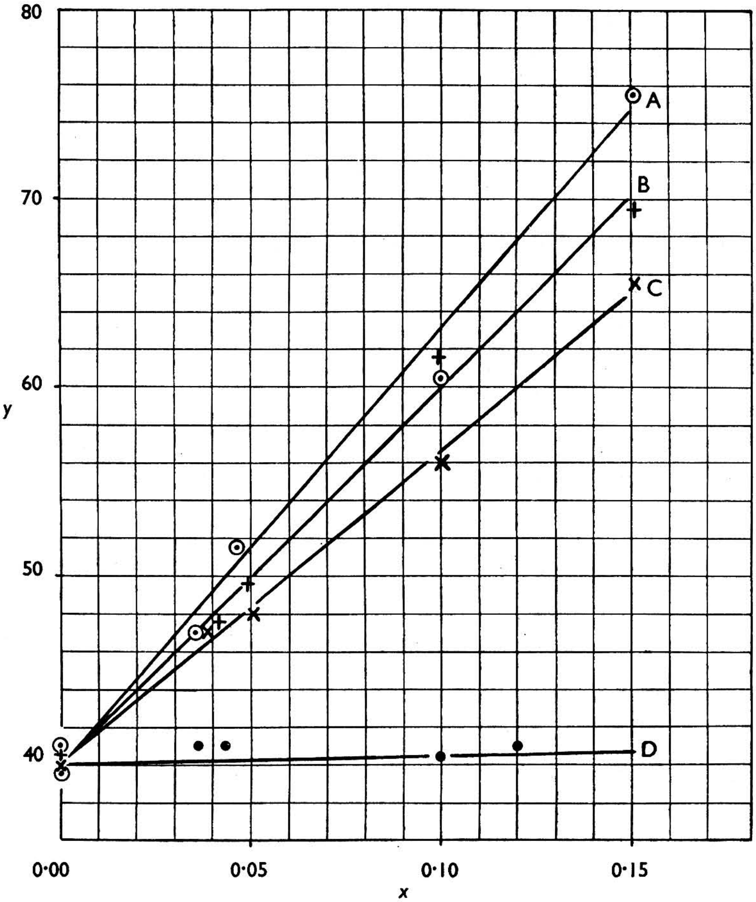

Fig. 2. Interference with wave for gamma benzene hexachloride caused by impurities in technical benzene hexachloride. Axis $y = \Delta i$ (− 0·8 to − 1·1 volts) for apparent wave for gamma benzene hexachloride in solution saturated with the gamma isomer: axis x = weight in grams of technical benzene hexachloride shaken with excess of gamma benzene hexachloride *plus* 10 ml of supporting medium. Sensitivity such that a standard solution of 0·05 per cent. w/v of gamma benzene hexachloride has $\Delta i = 30$; supporting medium blank has $\Delta i = 0{\cdot}5$.

Curves A, B and C, miscellaneous solutions of technical benzene hexachloride; curve D, synthetic mixture of isomers of benzene hexachloride simulating technical benzene hexachloride with which it is shaken. From a consideration of the normal concentrations of impurity in crude benzene hexachloride and the list of solubilities given above, it has been shown that, if 0·10 g of crude benzene hexachloride and an excess of pure gamma isomer are shaken with 10 ml of supporting medium, this condition is fulfilled except for the alpha and beta isomers. Special allowance for the effect of the alpha isomer is however made in the determination of apparent gamma isomer, and interference by the beta isomer can be neglected owing to its insolubility. It has been found that the recovery of interfering impurities by the extraction procedure described above is only about 90 per cent. complete; consequently, the correction for impurities is about 10 per cent. low and the final corrected value is correspondingly high. Extraction of the impurities by shaking the sample with the supporting medium is more effective than by dissolving the entire sample in ethanol and diluting the solution with an equal volume of the aqueous part of the supporting medium, when the greater part of the alpha and beta isomers is precipitated.

If any doubt exists as to the correctness of the value for the interference by impurities determined by the method given below, a more exhaustive analysis of the material should be performed; from the results, a diagram of the type shown in Fig. 2 can be constructed and it will generally indicate whether or not the value is in error. As already stated, the method is dependent on a straight-line relationship between x and y.

Gamma-isomer concentrate—The proposed method for assessing the impurity correction was shown to be applicable to gamma-isomer concentrates by experiments similar to those outlined in the previous section. Diagrams of the kind shown in Fig. 2 were constructed for a variety of gamma-isomer concentrates. It was found that the preparation of saturated solutions from gamma-isomer concentrates could be performed most conveniently and effectively by diluting 5 ml of a 0·8 per cent. w/v solution of the concentrate in ethanol with an equal volume of the aqueous part of the supporting medium, with the addition of 0·35 ml of supporting medium (to allow for volume contraction on adding water to ethanol) and a small amount of pure gamma isomer, and shaking the mixture at 20° C in a sealed vessel until equilibrium was established. The recovery of the interfering impurities was 95 per cent. by this method. The same ethanolic solution of the gamma-isomer concentrate can be used for the determination of the apparent gamma isomer and the impurity correction. The correction for impurities does not include the slight error caused by the presence of the alpha isomer (about +0·2 per cent.). If the amount of alpha isomer present is known approximately, allowance can be made when determining the apparent gamma isomer by having an appropriate amount of alpha isomer in the gamma-isomer standard.

Gamma isomer—A correction for impurities such as that applied to crude benzene hexachloride and gamma-isomer concentrate is not generally necessary for gamma isomer of 80 to 100 per cent. purity. The impurity is usually a mixture of alpha and beta isomers and, if particular accuracy is required, the slight error that arises owing to their presence may be allowed for by having approximately the same amount of impurity in the standard solution of gamma isomer.

Alpha - beta residues—In the same way that the gamma isomer tends to remain in alpha - beta residues after repeated washing, the interfering impurities are usually present as well. The correction for these impurities may be determined by the technique proposed for crude benzene hexachloride, except that a slightly larger amount of sample should be taken, *i.e.*, 0·4 g and not 0·1 g. The recovery of interfering impurities is usually about 65 per cent.

METHOD

This method is recommended for the examination of crude benzene hexachloride of the approximate composition: 12·5 to 17·5 per cent. of apparent gamma isomer and 50 to 70 per cent. of alpha isomer. For other types of technical benzene hexachloride, the procedure, the quantities of material and the composition of the standards must be adjusted in accordance with the observations made above in the experimental section. The solutions used for the determination of the apparent gamma-isomer centent, and the saturated solutions used to assess the interference by impurities may, if necessary, be examined after not less than 3 hours, but for routine analysis, it will usually be convenient to allow them to come to equilibrium overnight.

APPARATUS—

Vessels and shaker for the preparation of saturated solutions—A simple horizontal shaker having an amplitude of about three inches and a period of about half a second is recommended. The saturated solutions are prepared in capsules made from 6-inch × $\frac{3}{4}$-inch test tubes, which are clamped to a plate. This plate is immersed in a thermostatically controlled bath maintained at 20° C and attached to the shaker in a position such that the length of the capsules is in the plane of motion.

Polarographic equipment—The Cambridge cell assembly and thermostatically controlled bath are suitable; the thermostatically controlled bath is essential for accurate work. It should be set at 25° C and should not vary by more than $\pm 0{\cdot}2°$ C. Any type of polarograph may be used; a simple manual instrument incorporating a heavily damped Cambridge spot galvanometer has been found particularly suitable.

REAGENTS—

Ethanol, absolute (99 *per cent.*).

Potassium iodide, 2 *per cent. w/v*—An aqueous solution of analytical-reagent grade material.

Gelatin, 0·25 *per cent. w/v*—An aqueous solution of the purest gelatin available. (Use immediately.)

Potassium iodide - gelatin solution—Mix 960 ml of 2 per cent. w/v potassium iodide solution with 40 ml of 0·25 per cent. w/v gelatin solution. (Retain not longer than one week.)

Supporting medium—Add one volume of ethanol to one volume of potassium iodide - gelatin solution.

Gamma benzene hexachloride—Lindane quality containing more than 99 per cent. of gamma isomer.

Alpha benzene hexachloride—Particular care must be taken to ensure that the material used is free from gamma isomer, although the presence of small amounts of the other isomers is unimportant. The apparent gamma-isomer content, as determined by the procedure described below, should be less than 0·3 per cent.

Gamma benzene hexachloride, 0·200 *per cent. w/v*—A stock solution in ethanol.

Alpha benzene hexachloride, 1·00 *per cent. w/v*—A stock solution in ethanol.

PROCEDURE FOR POLAROGRAPHIC EXAMINATION—

Remove oxygen from the cell solution by passing a stream of nitrogen, previously saturated with supporting medium vapour, for six minutes at a rate of 2 litres per hour. At a suitable sensitivity, measure the difference, Δi, in diffusion current between $-0{\cdot}8$ volt and $-1{\cdot}1$ volts against a mercury-pool anode. Allow two minutes at each fixed potential for the reading to become steady. It has been found convenient to observe the maxima of the oscillations caused by the formation and fall of the mercury drops.

PROCEDURE FOR THE DETERMINATION OF APPARENT GAMMA ISOMER—

It will be noted that the concentrations of both standards and sample, although designated as percentages, are g per 96·5 ml, as there is a 3·5-ml volume contraction when 50 ml of ethanol and 50 ml of water are mixed.

Preparation of standard solution of gamma isomer, 0·01 *per cent. w/v*—To 5 ml of the stock solution of gamma benzene hexachloride (0·200 per cent. in ethanol) add 45 ml of ethanol and 50 ml of potassium iodide - gelatin solution.

Preparation of standard solution of alpha - gamma isomers, 0·01 *per cent. w/v of gamma isomer and* 0·04 *per cent. w/v of alpha isomer*—To 5 ml of the stock solution of gamma benzene hexachloride (0·200 per cent. in ethanol) and 4 ml of the stock solution of alpha isomer (1·00 per cent. in ethanol), add 41 ml of ethanol and 50 ml of potassium iodide - gelatin solution.

Preparation of solution of crude benzene hexachloride—Dissolve an accurately weighed amount, W_1 g, about 0·07 g, of the finely ground and sieved (44 B.S.S.) crude benzene hexachloride in 50 ml of ethanol and add 50 ml of potassium iodide - gelatin solution.

After allowing the standard and sample solutions to stand overnight, determine Δi for each solution and also a blank value for the supporting medium. Provided that the purity of the reagents is satisfactory, the blank should be about 7 per cent. of Δi for the 0·01 per cent.

gamma-isomer standard, and the recovery of gamma isomer in the alpha - gamma-isomer standard should be approximately 98 per cent.

Calculate the apparent gamma isomer content of the sample as follows—

$$\text{Apparent gamma isomer, \%} = \frac{\Delta i \text{ of sample solution} - \text{blank}}{\Delta i \text{ of alpha - gamma standard} - \text{blank}} \times \frac{1{\cdot}00}{W_1}$$

PROCEDURE FOR THE DETERMINATION OF INTERFERENCE BY IMPURITIES—

Preparation of standard solution of gamma isomer, 0·05 per cent. w/v—Measure 25 ml of the stock solution of gamma isomer (0·200 per cent. in ethanol), 25 ml of ethanol and 50 ml of potassium iodide - gelatin solution into a 100-ml calibrated flask and dilute to the mark with the supporting medium.

Preparation of saturated solution A (sample + excess of gamma isomer)—Weigh accurately W_2 g, approximately 0·1 g, of the ground and sieved crude benzene hexachloride into a 6-inch × $\frac{3}{4}$-inch test tube and add about 0·04 g of pure gamma isomer. Constrict the tube about one inch from the open end, introduce 10 ml of supporting medium, seal the tube and allow the contents to be shaken overnight at 20° C.

Preparation of saturated solution B (gamma isomer alone)—As for saturated solution A, but with no crude benzene hexachloride present.

Remove the tubes from the thermostatically controlled bath as required and filter the solutions into a polarographic cell through a Whatman No. 40 filter-paper. Determine Δi for the standard, for saturated solutions A and B and also for the supporting medium as a blank. In selecting a suitable sensitivity for the polarographic measurement, it must be remembered that Δi for saturated solution B may be more than twice that for the 0·05 per cent. gamma-isomer standard.

First calculate the gamma-isomer content of saturated solution B—

$$\text{Gamma benzene hexachloride, \% w/v} = \frac{\Delta i \text{ of saturated solution B} - \text{blank} \times 0{\cdot}05}{\Delta i \text{ of gamma-isomer standard} - \text{blank}}.$$

A reproducible value of approximately 0·067 per cent. w/v should be found; then calculate the percentage interference due to impurities as follows—

$$\text{Interference, \%} = \frac{\Delta i \text{ of saturated solution A} - \Delta i \text{ of saturated solution B}}{\Delta i \text{ of standard solution} - \text{blank}} \times \frac{0{\cdot}5}{W_2} \times 1{\cdot}1$$

(1·1 = correction to allow for incomplete recovery of impurities).

Corrected value for the concentration of gamma isomer—The difference between the apparent gamma-isomer content and the percentage interference is the corrected value.

RESULTS

Synthetic mixtures of the isomers of benzene hexachloride and heptachloro*cyclo*hexane of composition corresponding to various grades of technical material have been examined, and the results have been found to be substantially correct within experimental limits. These results, however, are not adequate verification of the method, as the principal difficulty with regard to the polarographic examination of benzene hexachloride is the determination of the gamma isomer in technical materials of uncertain composition. A more important consideration is, perhaps, the measure of agreement between results for the determination of gamma isomer in miscellaneous technical mixtures by the proposed method and other independent techniques. The results in Table I are those determined polarographically and by the other methods of analysis that are used or have been investigated in this Research Department; values for crude benzene hexachloride, gamma-isomer concentrate, and gamma isomer are compared, but comparative results for alpha - beta residues are not available. All results quoted are the mean of replicate determinations, usually four determinations. Details of the Dragt-polarographic,[2] solubility,[8] adsorption-chromatographic[9] and isotope-dilution[10] methods of analysis are described in the literature, but the infra-red method, as used for these determinations, and the differential-refractometric method[11] have not yet been published.

The repeatability of the method, expressed as 95 per cent. confidence limits for a single determination, has been calculated from replicate determinations on a number of samples and found to be—

Crude benzene hexachloride containing 8 to 15 per cent. of gamma isomer, ±0·6.
Gamma-isomer concentrate containing 20 to 65 per cent. of gamma isomer, ±1·4 to 1·8.
Gamma benzene hexachloride containing 80 to 100 per cent. of gamma isomer, ±2·5.
Alpha - beta residues containing 0 to 2 per cent. of gamma isomer, ±0·1 to 0·2.

The limits of precision for a single analysis of 80 to 100 per cent. pure gamma isomer may be narrowed to less than ±1·0 by certain refinements in the polarographic equipment used; in particular, by the use of a more sensitive and precise instrument for measuring diffusion currents than the usual galvanometer, and by controlling the temperature of the cell solution to within ±0·05° C.

TABLE I

COMPARATIVE RESULTS FOR THE DETERMINATION OF THE PERCENTAGE OF GAMMA ISOMER IN TECHNICAL BENZENE HEXACHLORIDE BY VARIOUS METHODS

Polarograph		Isotope dilution, %	Infra-red, %	Dragt polarograph, %	Adsorption chromatograph, %	Solubility, %	Differential refractometer, %
Apparent, %	Corrected, %						
13·4	8·9	9·2		8·6	9·4		
14·0	10·9	10·5			11·3		
14·6	11·7	11·8					
15·1	13·8	14·6					
14·9	12·7	13·0			12·7		
15·5	14·1				13·9		
16·0	14·5				14·5		
15·6	14·1				13·9	14·3	
14·9	12·8			13·2			
15·3	11·7				12·1		
13·7	8·9				10·3		
15·3	13·4				13·4		
15·2	13·4				13·0		
23·4	22·7	22·6		21·5			
23·5	22·8	23·3					
36·5	27·5	28·3	25·9				
42·2	33·9	35·8	33·5	31·2			
40·8	35·1	36·1	34·8				
38·2	32·0	34·2	31·2				
50·7	49·7	50·1	48·5	45·4			
50·8	49·8	50·0	48·8				
50·2	49·2	49·0	47·4				
43·3	42·6	43·1	41·4				
51·9	50·8	46·2	48·2				
64·8	62·4	62·3	63·0				
54·5	54·2		54·2		50·2		
54·2	54·0		54·8		51·4		
98·6	98·5						98·0
99·6	99·6						99·2
99·9	99·9						99·8
98·5	98·4						98·3

CONCLUSIONS

The method described provides a suitably rapid and precise method for the routine determination of gamma isomer in technical benzene hexachloride; one operator can examine about eight samples per day. The technique whereby the interference due to impurities is assessed has been verified for benzene hexachloride and heptachloro*cyclo*hexane isomers, nevertheless, it is theoretically valid for any interfering impurity, provided that suitable quantities of material are examined.

Results by the method described agree well with those by other methods of analysis.

With suitable adjustments of the quantities of material taken and the concentrations of the standards, the method may be applied to any grade of technical benzene hexachloride.

The technique whereby correction for impurities is made is one that would possibly be applicable to the determination of other components of technical benzene hexachloride and also to the analysis of other mixtures of closely related organic compounds whose polarographic waves coincide.

REFERENCES

1. Ingram, G. B., and Southern, H. K., *Nature*, 1948, **161**, 437.
2. Dragt, G., *Anal. Chem.*, 1948, **20**, 737.
3. Nakajima, S., *et al.*, *Botyu Kagaka*, March 11th, 1949, p. 3, and September 13th, 1949, p. 14.
4. Hasselbach, H., and Schwabe, K., *Z. anal. Chem.*, 1951, **132**, 94.
5. Wolf, S., Munster, C., and Sarfert, E., *Ibid.*, 1953, **140**, 25.
6. Streuli, C. A., and Cooke, W. D., *Anal. Chem.*, 1954, **26**, 970.
7. Thorp, D., *J. Soc. Chem. Ind.*, 1946, **65**, 414.
8. Willermain, M., *Anal. Chim. Acta*, 1949, **3**, 206.
9. Granger, G., and Zwilling, J. P., *Bull. Soc. Chim. France*, 1950, 873.
10. Hill, R., Jones, A. G., and Palin, D. E., *Chem. & Ind.*, February 6th, 1954, No. 6, 162.
11. Hill, R., and Jones, A. G., *Analyst*, in the press.

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GENERAL CHEMICALS DIVISION
WIDNES

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A Continuous Recorder for Dissolved Oxygen in Water

BY R. BRIGGS, G. KNOWLES AND L. J. SCRAGG

Winkler's method for oxygen in water has been mechanised and adapted to automatic working. The optical densities of the iodine solutions produced are measured by a photocell, the current from which is amplified, rectified and automatically recorded. Inherent absorption of the samples and reagents is recorded after addition of sodium sulphite to remove the iodine colour. The oxygen content is then calculated from the difference between the two readings. In laboratory tests on water containing 2 to 8 p.p.m. of oxygen by weight the maximum error was 0·05 p.p.m.

IN studies of the ecology of rivers, including investigations of the effects of pollution, it is desirable to obtain a continuous record of fluctuations in the content of dissolved oxygen. Even in a comparatively unpolluted river there may be wide variations, partly because the effect of photosynthesis by green plants is to increase the content of dissolved oxygen during daylight and partly because of factors, such as wind, that affect the rate of solution of oxygen through the surface. Butcher, Pentelow and Woodley[1] found a diurnal variation of several p.p.m. by weight in rivers.

Search of the literature revealed no apparatus that could function unattended for long periods with an error of no more than 0·1 p.p.m. by weight, which is the maximum allowable error. Adaptation of existing methods was considered, but none seemed suitable. A method that consists in removing the oxygen from water by hydrogen and recording the thermal conductivity of the resulting gas mixture has been applied only to boiler-feed waters of very low dissolved oxygen content, and apart from other difficulties it appears unsuitable for river waters, which may yield such gases as carbon dioxide, hydrogen sulphide and methane. The simple electrochemical method developed by Tödt *et al.*[2] is accurate for boiler-feed water, but, as has been fully discussed by Ohle,[3] is adversely affected by such factors as deposition of carbonate from water containing bicarbonate and by variations in the concentration of inorganic salts. Polarography has been applied to some extent, but the adverse comments of Ippen, Yoseph and Posthill[4] and Ippen, Campbell and Carver[5] indicate that trouble is likely both with dropping-mercury and platinum electrodes. Another disadvantage of polarography is that variable concentrations of other reducible substances may contribute to the recorded current; Seaman and Allen[6] attempted to overcome this by measuring the current before and after blowing an inert gas through the sample but, as Rand and Heukelekian[7] indicated, other gases or volatile compounds capable of contributing to the current can be removed by the inert gas, in addition to the oxygen. In a colorimetric procedure used by Tuve, White and Luke[8] and Tuve[9] for recording dissolved oxygen, sensitised pyrogallol is used, but for the present purpose much work would be required on the effect of various factors on the rate of formation and stability of the colour.

EXPERIMENTAL

As none of the methods described was suitable, it was decided to adapt the Winkler procedure to automatic recording. This is the most widely used manual method for the determination of oxygen dissolved in water and is fully described by the American Public Health Association.[10] A solution of manganous sulphate and an alkaline solution of potassium iodide are added to the sample, so forming a precipitate of manganous hydroxide, part of which is oxidised by all the dissolved oxygen present. On acidification the precipitate dissolves and the oxidised portion releases a quantity of iodine equivalent to the dissolved oxygen originally present.

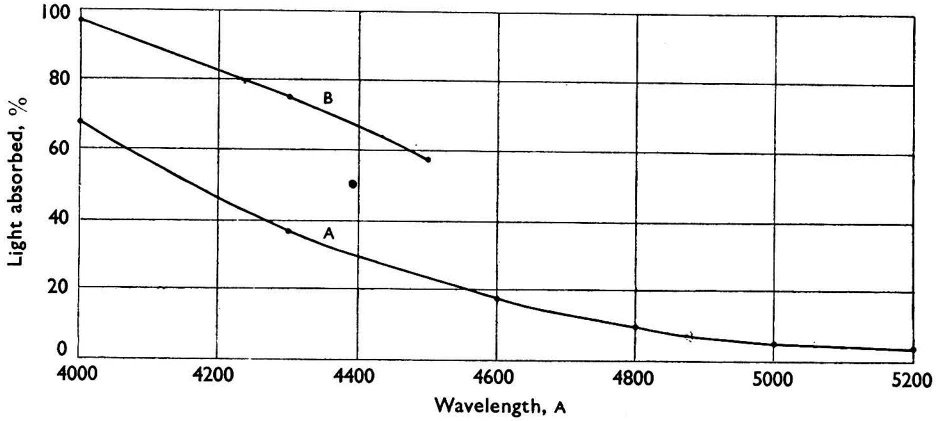

Fig. 1. Relation between the wavelength and percentage of light absorbed by a 1-cm thick iodine solution. Curve A, 9·45 p.p.m. of iodine; curve B, 113 p.p.m. of iodine. Concentrations of reagents were as in the Winkler method, except that the concentration of potassium iodide was 16 per cent. less for curve B; 100 per cent. transmission is that of the corresponding solution, free from iodine, for each curve (distilled water also gave 100 per cent. transmission)

In this manual method the liberated iodine is usually determined by titration, but it was thought better in the automatic method to determine its colour photo-electrically; it is of

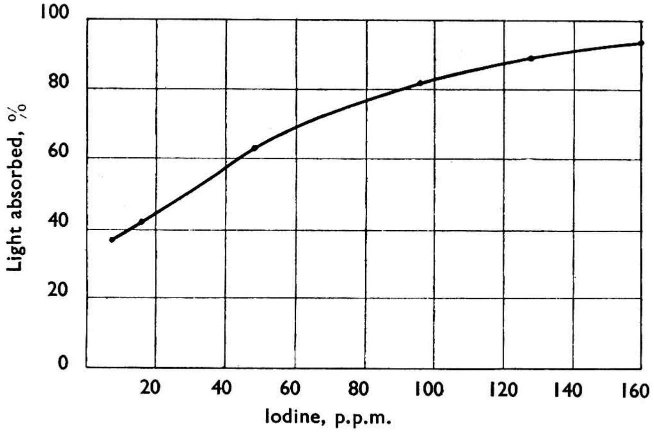

Fig. 2. Relationship between concentration of iodine and percentage of light at 4300 A absorbed by a 1-cm thick iodine solution. Concentrations of reagents were as in the Winkler method; 100 per cent. transmission corresponds to the same solution, with no iodine, for every reading. Distilled water also gave 100 per cent. transmission

interest, however, that an apparatus making use of automatic titration of iodine was used by Gluckauf, Heal, Martin and Paneth[11] in a continuous recorder for ozone in air.

As it was thought that variation in the concentration of potassium iodide might affect the amount of light absorbed by a solution of iodine, an experiment was made to determine how much variation in the dose of this reagent could be permitted. Solutions containing 8 p.p.m. of iodine, Winkler reagents other than potassium iodide in the usual concentrations and either 600 (the usual concentration), 480 or 720 p.p.m. of potassium iodide were prepared. The percentages of light of wavelength 4300 A absorbed by the three solutions were 37·7, 36·9 and 36·9, respectively. As this 20 per cent. variation in concentration of potassium iodide had so little effect, it was concluded that no errors would be introduced if the precision of dosing was about ±5 per cent.

For determining the intensity of the colour of the iodine in a recorder for oxidising substances in air, Littman and Benoliel[12] used ultra-violet illumination of about 3550 A, where there is a maximum in the absorption spectrum, but for our present purpose the use of visible light was preferred. To determine the most suitable wavelength, absorption spectra (shown in Fig. 1) were determined for two iodine solutions, with the usual amounts of Winkler reagents, in which the concentrations of iodine corresponded to concentrations of dissolved oxygen of about 0·6 and 7·0 p.p.m. by weight. From these spectra the best available filter appeared to be Ilford No. 806, which passes only light of about 4500 A; this filter was used in the final apparatus and has proved suitable.

Fig. 2 shows the percentage of light absorbed at 4300 A by iodine solutions, the concentrations of which correspond to the range of 0 to 10 p.p.m. of oxygen by weight, in presence of the usual concentrations of Winkler reagents as specified in "Standard Methods for the Examination of Water and Sewage."[10]

METHOD

REAGENTS—

Manganous sulphate solution—Dissolve 480 g of manganous sulphate, $MnSO_4.4H_2O$, in water and dilute to 1 litre.

Alkaline potassium iodide solution—Dissolve 500 g of sodium hydroxide and 150 g of potassium iodide in water and dilute to 1 litre.

Alkaline potassium iodide - azide reagent—As for the alkaline iodide solution with the addition of 10 g of sodium azide per litre.

Sulphuric acid, concentrated.

Sodium sulphite - EDTA *solution*—Dissolve 125 g of sodium sulphite, $Na_2SO_3.7H_2O$, and 10 g of disodium ethylenediaminetetra-acetate (EDTA) in water and dilute to 1 litre.

APPARATUS—

A general view of the apparatus used is shown in Fig. 3, and Fig. 4 shows the arrangement for taking a sample of water, adding the reagents and transferring the solutions to the photometric cell in which their optical densities are recorded. The sequence of operations is controlled by a Post Office Uniselector stepping switch whose rotating contact arm works repeatedly through 25 positions, being moved forward one position every half-minute on the closing of a micro-switch by the Synclock motor in the recorder. The flow of liquids is controlled by a series of electromagnetic valves* each of which allows liquid to flow only when the valve is energised.

The units for automatic addition of the reagents are of the type described by Eden, Downing and Wheatland,[13] and one such unit is shown at the left of Fig. 4. When the valve A is opened, reagent runs into the dosing pipette from a stock bottle until its level is that of the foot of a Mariotte tube in the bottle, and when A is then closed and B opened the reagent in the dosing pipette runs into the water in the reaction cell.

The volume of the water sample in the reaction cell is 106 ml and to this are added 2·2 ml of alkaline iodide - azide solution, 1·25 ml of manganous sulphate solution and 3·5 ml of sulphuric acid. All the reagent concentrations are adjusted so that these doses result in the usual additions of chemicals specified for the Winkler method, *i.e.*, 1 ml of each reagent is added per 250 ml of sample. The azide modification[10] was adopted because nitrite may be present in streams of which the concentration of dissolved oxygen is to be recorded.

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* Valves are type LF/VA made by Londex Ltd.

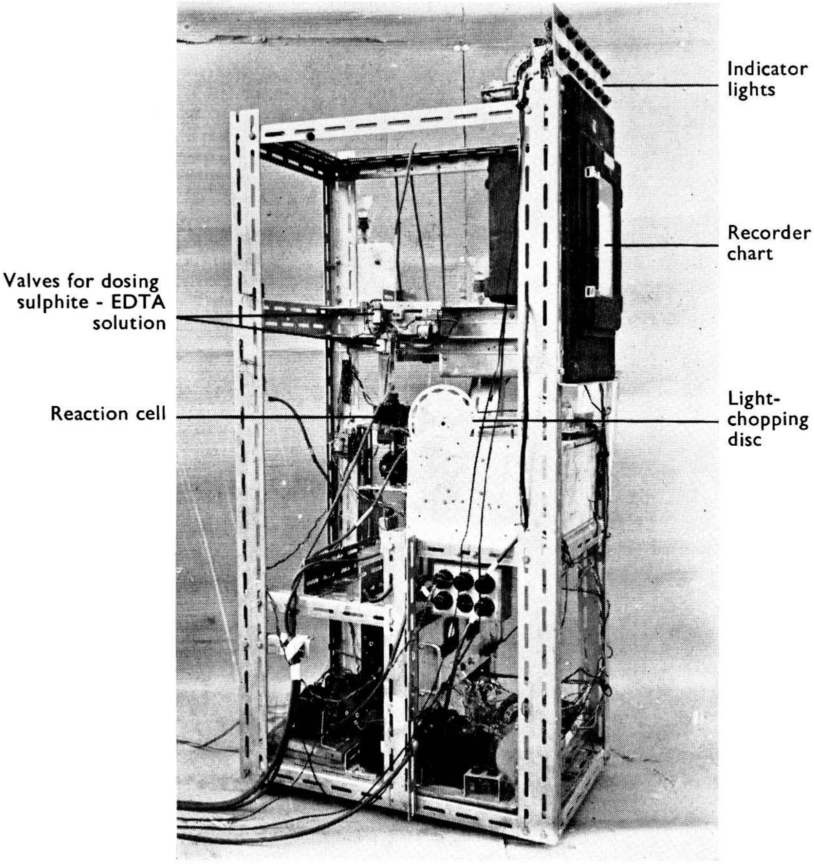

Fig. 3. Continuous recorder for dissolved oxygen in water

When the optical density of the iodine solution has been recorded, the iodine in a portion (about 70 ml) of the iodine solution remaining in the reaction cell is destroyed by addition of 1·3 ml of sodium sulphite - EDTA solution. The accuracy of this dose is not critical so long as excess of sulphite is present. The optical density of this solution is then determined in the photometric cell. The EDTA is included merely to keep the cells and tubes free from deposits.

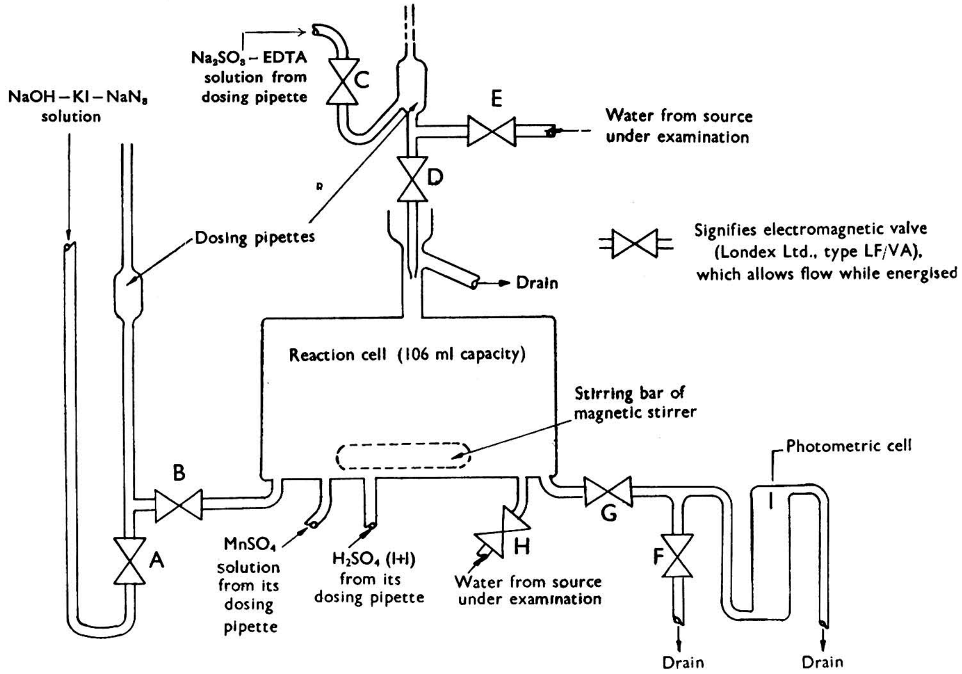

Fig. 4. Arrangement for sampling, dosing and transference to photometric cell for recording of intensity of iodine colour

The recorder chart has two lines of points, and a curve showing the concentration of dissolved oxygen against time is plotted by reading, from Fig. 5, the values of dissolved oxygen concentration corresponding to the two points determined in each cycle and subtracting that for colour and turbidity due to the raw water from that for the solution also containing iodine. Values for the calibration curve shown in Fig. 5 were determined by supplying the apparatus with clear tap water whose concentrations of dissolved oxygen were simultaneously determined by the manual Winkler method, the azide modification being used.

APPARATUS FOR RECORDING THE OPTICAL DENSITY—

To record the optical density, light from a 6-volt, 24-watt, car headlamp bulb run at 4 volts a.c. (supplied by the mains via an Advance constant-voltage transformer) passes through a pair of $1\frac{1}{2}$-inch diameter photographic condensing lenses and through a 1-cm path length of the solution in the photometric cell, coming to a focus as an image of the filament in a slit, $\frac{1}{2}$ inch by 1/10 inch, cut in metal (Fig. 6). Diverging again from the focus, the light passes through an Ilford No. 806 filter and falls on the cathode of a vacuum photocell, but before reaching the filter and photocell it is periodically interrupted by a disc, driven at about 1425 r.p.m. by a 1/20 h.p. induction motor; as a result the intensity of illumination of the photocell cathode rises from zero to a value dependent on the optical density of the solution and falls to zero again, the intensity varying with time in approximately the form of half a sine wave, this half wave lasting $\frac{1}{600}$ second. No light reaches the photocell in the next $\frac{1}{600}$ second, at the end of which the next slot passes the next flash of illumination to the photocell. The frequency of illumination of the photocell is thus 300 cycles per second. The photocell is

V_1 in Fig. 7 and the voltage of the 300 cycles per second signal it develops across resistance R_3 is proportional to the intensity of the light transmitted by the liquid in the photometric cell.

In this manner the optical density of the liquid is expressed in terms of the amplitude of a voltage wave of frequency 300 cycles per second across the high resistance R_3. By means of a cathode-follower stage, including electrometer valve V_2, this voltage is converted to a corresponding one across the small resistance R_4, which is then amplified, still as an a.c. of 300 cycles per second, by the 4-stage a.c. amplifier that follows; finally the signal passes through the transformer, T, to the recorder circuit in which there is a germanium diode to convert the current to a d.c. current for recording on the d.c. recorder of full-scale deflection 0·6 mA and resistance 650 ohms. The design of the a.c. amplifier is largely based on a published circuit.[14]

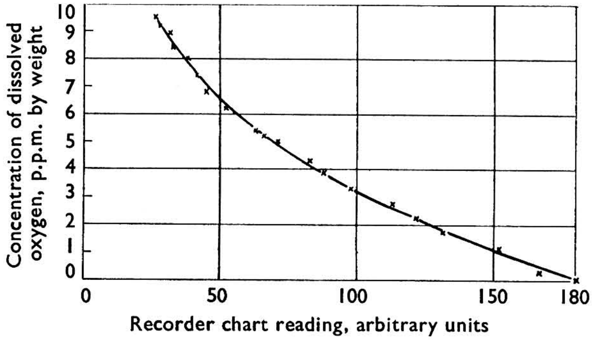

Fig. 5. Relation between recorder chart reading and concentration of dissolved oxygen in a tap water in which concentration of dissolved oxygen was varied. The con-concentrations of dissolved oxygen shown were determined manually by the azide modification of the Winkler method

The reason for the use of the light-chopping disc to obtain an a.c. output from the photocell is that unless power is converted to an alternating current before amplification it is impossible to obtain a sufficiently constant amplification factor (for any given photocell output) over long periods of time. Davenport[15] used a similar arrangement for short-period recording, but owing to the absence of any feedback circuit in his a.c. amplifier, it must have

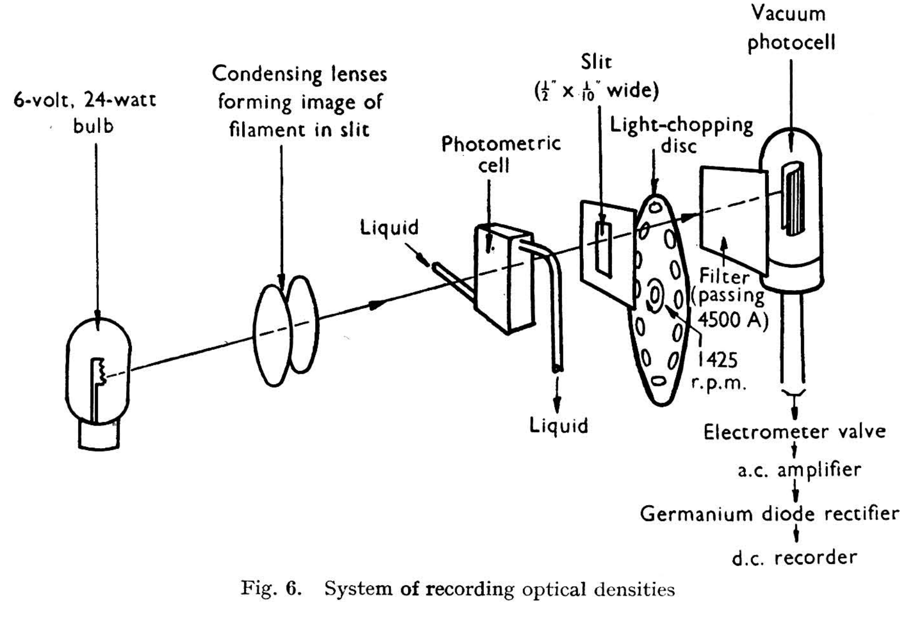

Fig. 6. System of recording optical densities

been far less stable than that in Fig. 7, of which the gain appears to be constant to within ±0·1 per cent. during a period of many weeks and on which no adjustment is necessary at any time. The power for the whole circuit, shown in Fig. 7, is from the mains via an Advance constant-voltage transformer and the electronically stabilised Ediswan power pack type R.1095.

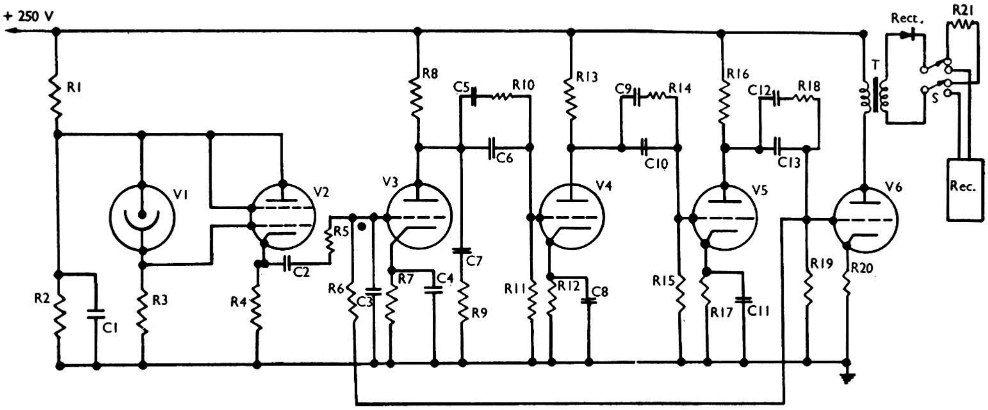

Fig. 7. Photocell, input stage, A.C. amplifier, rectifier and recorder circuit

C_1 = 1 μF
C_2 = 0·1 μF
C_3 = 680 $\mu\mu$F
C_4 = 2 μF
C_5 = 0·1 μF
C_6 = 0·005 μF
C_7 = 0·006 μF
C_8 = 2 μF
C_9 = 0·1 μF
C_{10} = 0·02 μF
C_{11} = 1 μF
C_{12} = 0·1 μF
C_{13} = 0·05 μF
R_1 = 22,000 ohms
R_2 = 4700 ohms
R_3 = 2 megohms
R_4 = 20,000 ohms
R_5 = 130,000 ohms
R_6 = 550,000 ohms
R_7 = 2000 ohms
R_8 = 220,000 ohms
R_9 = 560 ohms
R_{10} = 5·1 megohms
R_{11} = 300,000 ohms
R_{12} = 2000 ohms
R_{13} = 220,000 ohms
R_{14} = 5·1 megohms
R_{15} = 75,000 ohms
R_{16} = 100,000 ohms
R_{17} = 6800 ohms
R_{18} = 5·1 megohms
R_{19} = 1 megohm
R_{20} = 1000 ohms
R_{21} = 650 ohms

Rec. = Recorder, full-scale deflection 60 mA, resistance 650 ohms
Rect. = Germanium rectifier, type GEX56 (General Electric Co. Ltd.)
S = 2-pole, 2-way switch operated by motor of recorder
T = Step-down transformer (8 to 1)
V_1 = Mullard 90AV vacuum photocell
V_2 = Mullard ME 1400 electrometer valve
V_3, V_4, V_5, V_6 = 6C4

Exact measurement has not been made of the intensity of light falling on the photocell cathode, but it is equivalent to an intensity, at the peaks, of about 0·03 lumen if the photometric cell contains clear water; the corresponding peak voltage of the waveform developed across R_3 is about 1·4 volts and across R_{19} is about 5 volts.

POWER SUPPLY—

The power required for the whole equipment is derived entirely from the mains, the total consumption being about 250 watts, including 40 watts for the pump.

CYCLE OF OPERATIONS—

The cycle of events is as follows (lettering refers to Fig. 4)—

Minutes

0 At this stage the sampling pump is still running from the preceding cycle; it delivers the water under examination to a constant-head tube of the simple type used by Spoor[16] to prevent the exposure of the sample to air. Valve H opens to allow water to run into the reaction cell from the constant-head tube. E, D, G and F were the only valves open at the end of the preceding cycle and E, G and F close as H opens.

$\frac{1}{2}$ H closes. G opens to wash the photometric cell.

1 F opens to complete draining of water from reaction cell.

1½ D, F and G close. H opens to supply water to reaction cell.

2 H closes. G opens to complete washing of photometric cell.

2½ F opens to complete draining of water from reaction cell.

3 H opens and F closes simultaneously, at 3 minutes. For the following 1½ minutes H remains open, so filling the reaction cell with water and passing many changes of water upwards through it, the surplus leaving by the drain tube at the top.

4½ H closes. Pump switched off. Magnetic stirrer in reaction cell switched on. A, C and other valves supplying dosing pipettes open.

5 A, C and other valves supplying dosing pipettes close. B opens to allow dose of alkaline iodide - azide to run into the sample of water in the reaction cell. The corresponding exit valve on the dosing pipette for manganous sulphate also opens, allowing this solution to run into the sample of water. Sampling pump switched off.

5½ Water in reaction cell, now containing the flocculent precipitate of manganese hydroxides, is stirred. Light for photometric measurement is switched on. Light-chopping disc switched on. Exit valves on the dosing pipettes for alkaline iodide and manganous sulphate close.

6 Exit valve on the dosing pipette for sulphuric acid opens, allowing this solution to run into the reaction cell where it brings the precipitate into solution, liberating iodine.

6½ G opens to allow the iodine solution to run through the photometric cell. Exit valve on the dosing pipette for sulphuric acid closes.

7 G closes. About one-third of the contents of the reaction cell have run to the photometric cell, which now remains full of the iodine solution whose optical density is to be recorded. D opens to allow the dose of sodium sulphite - EDTA solution to run into the iodine solution remaining in reaction cell.

7½ D closes. Optical density of iodine solution in photometric cell is recorded by closure of recorder switch (S on Fig. 7).

8 Recorder switch opens. G opens to allow the solution from the reaction cell to flow through the photometric cell; all iodine in this solution has been destroyed by the sulphite.

8½ G closes. Optical density of the solution in the photometric cell is recorded by closure of recorder switch; this optical density is due to any colour or turbidity present in the original water.

9 Recorder switch opens. G and F open to drain reaction cell.

9½ Sampling pump switched on. E and D open to allow water to wash the sulphite dosing tubes, to prevent any drip of sulphite into the water during the next cycle of events.

10 This corresponds to 0 minutes above; the cycle starts again.

PERFORMANCE

The apparatus has been tested in the laboratory on tap water in which the concentration of dissolved oxygen had been artificially varied by passing a stream of nitrogen through it. The results are shown in Fig. 8, in which each dot represents an automatic determination of the dissolved oxygen; at each arrow a manual determination was made by the Winkler method simultaneously with the automatic determination. In no experiment did the automatic and manual values differ by more than 0·05 p.p.m. of oxygen by weight.

The apparatus is now being used for recording the concentration of dissolved oxygen in the effluent channel from a sewage works, and some results, for a 5-day period, are shown in Fig. 9. As individual determinations are too numerous to show, their averages for each 4-hour period have been plotted. There is much suspended material in the effluent channel, but the automatic analyser continues to work satisfactorily under these conditions, provided that cloth is fixed round the pump intake to remove the larger particles. The turbidity and colour of the water analysed are high at times, reducing the recorder chart reading (Fig. 5) to as low as 130 for the solution in which iodine has been destroyed by the addition of sodium sulphite, but numerous periodical checks have never shown a greater difference than 0·1 p.p.m. between automatic and manual determinations.

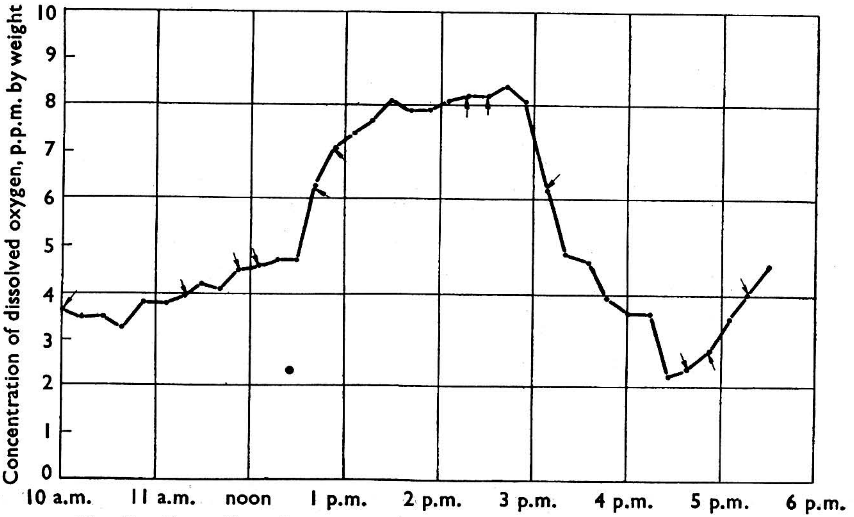

Fig. 8. Recording of concentration of dissolved oxygen in tap water
• Automatic determination
→ Manual determination, point of arrow indicating value

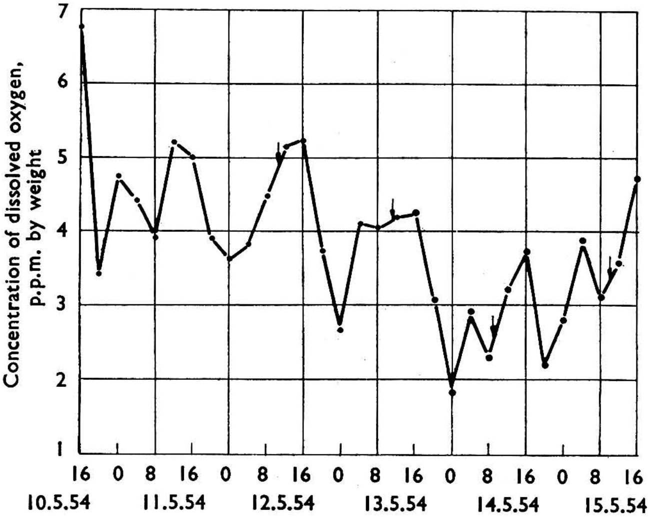

Fig. 9. Concentrations of dissolved oxygen in water of effluent channel of a sewage works, determined by the continuous recorder. Each point is the average of the determinations made by the continuous recorder during a period of 4 hours. At each time marked by an arrow, a manual determination was made on a duplicate sample. No manual determination differed by more than 0·05 p.p.m. from the corresponding automatic determination

It seems likely that this type of apparatus could be used for recording the concentrations of other substances for which colorimetric determinations are available.

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References

1. Butcher, R. W., Pentelow, F. T. K., and Woodley, J. W. A., *Int. Rev. Hydrobiol.*, 1930, **24**, 47.
2. Freier, R., Tödt, F., and Wickert, K., *Chem.-Ing.-Tech.*, 1951, **23**, 325.
3. Ohle, W., *Vom Wasser*, 1952, **19**, 99.
4. Ippen, A. T., Yoseph, R. S., and Posthill, B. N., U.S. Public Health Service, Public Health Project No. RG–863. Massachusetts Institute of Technology Hydrodynamics Laboratory, Technical Report No. 3, 1951.

5. Ippen, A. T., Campbell, L. G., and Carver, C. E., U.S. Public Health Service, Public Health Project No. RG–863. Massachusetts Institute of Technology Hydrodynamics Laboratory, Technical Report No. 7, 1952.
6. Seaman, W., and Allen, W., *Sewage Ind. Wastes*, 1951, **23**, 1141.
7. Rand, M. C., and Heukelekian, H., *Ibid.*, 1951, **23**, 1141.
8. Tuve, R. L., White, J. C., and Luke, E. L., U.S. Patent 2,430,895.
9. Tuve, R. L., U.S. Patent 2,440,315.
10. American Public Health Association, "Standard Methods for the Examination of Water and Sewage," Ninth Edition, New York, 1946, pp. 127–130.
11. Gluckauf, E., Heal, H. G., Martin, G. R., and Paneth, F. A., *J. Chem. Soc.*, 1944, 1.
12. Littman, F. E., and Benoliel, R. W., *Anal. Chem.*, 1953, **25**, 1480.
13. Eden, G. E., Downing, A. L., and Wheatland, A. B., *J. Instn. Wat. Engrs*, 1952, **6**, 511.
14. Valley, G. E., and Wellman, H., *Editors*, "Vacuum Tube Amplifiers," McGraw-Hill Book Co. Ltd., New York, p. 381.
15. Davenport, T. B., *J. Sci. Instrum.*, 1944, **21**, 84 and 188.
16. Spoor, W. A., *Science*, 1948, **108**, 421.

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June 10*th*, 1954

The Determination of Potentially Ionic Fluorine in Non-aqueous Solvents

BY H. F. LIDDELL

The thorium lake of Chromotrope 2B is a bluish-violet dispersion in ethanol containing ethyl cellulose, and this colour is changed to the intense bright red of the original dye by potentially ionic fluorine. The method can be used quantitatively and is sensitive to 0·02 p.p.m. of fluorine. This change can be determined absorptiometrically or visually; some refinements of technique are described.

IT has been found that the thorium lake of chromotrope 2B (Colour Index No. 45), which forms a black colloidal dispersion in water under some conditions, will produce a bluish-violet dispersion in ethanol containing ethyl cellulose. In ethanol or other organic solvent this violet colour is changed to the intense bright red of the original dye in the presence of potentially ionic fluorine and 0·02 p.p.m. of fluorine can be readily detected. The absorption curves of the two colours are shown in Fig. 1. The change of colour can be determined with a suitable

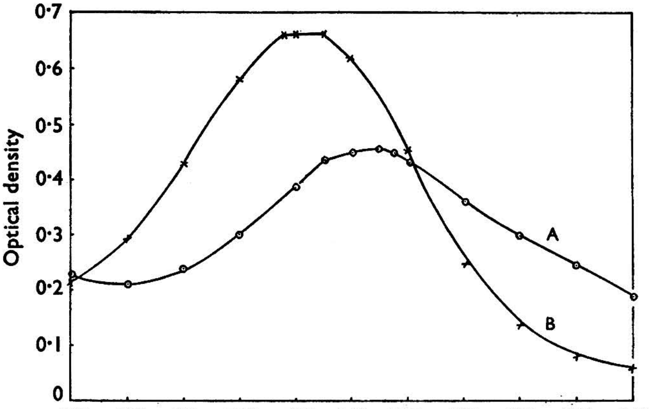

Fig. 1. Absorption curves for chromotrope 2B. Curve A, thorium lake dispersed in ethanol; curve B, diphenylguanidine salt dissolved in ethanol

absorptiometer or, at great dilution, it can be determined visually in Nessler cylinders. Alternatively, when toluene is the main solvent, the unchanged lake dispersion can be precipitated with triethylamine phthalate, the precipitate being separated by filtration and the red colour of the dyestuff alone observed. It will be seen from the calibration curves shown in Fig. 2 that the precision of the method is not ideal, but the great sensitivity by visual determination makes it of some value.

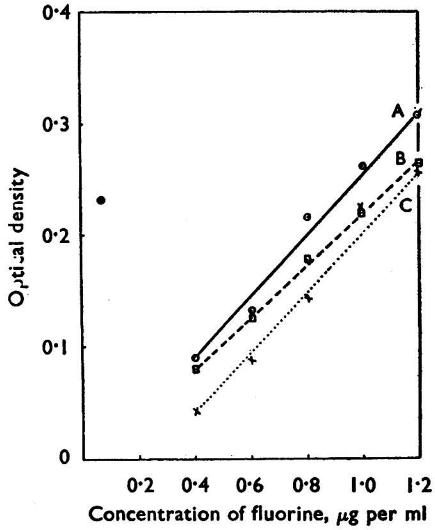

Fig. 2. Comparison of solvents with Spekker absorptiometer and Ilford No. 604 filter. Curve A, toluene; curve B, *iso*propanol; curve C, ethanol

As Chromotrope 2B (sodium salt of *p*-nitrophenylazo-1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid) is only slightly soluble in ethanol, the diphenylguanidine salt is used for the preparation of the stock solution of the dye in this solvent. The trifuryldihydroglyoxaline salt is also suitable, but the free acid and the *cyclo*hexylamine and di*cyclo*hexylamine salts are too soluble in water to be conveniently isolated.

METHOD

PREPARATION OF THE DIPHENYLGUANIDINE SALT OF CHROMOTROPE 2B—

Dissolve 6·3 g of Chromotrope 2B in 400 ml of boiling water and cool the solution to 20° C. To it add 35 ml of a 10 per cent. solution of diphenylguanidine hydrochloride, the solutions being maintained at less than 20° C. Remove the solid by filtration at below 10° C, wash it with water and dry it in a desiccator under reduced pressure. The yield is 5·4 g.

REAGENTS—

Dispersed thorium lake of Chromotrope 2B (*lake dispersion*)—Dissolve 0·1 g of the diphenylguanidine salt in 5·0 ml of hot ethanol. Filter the solution and add to it 2·0 ml of a 1 per cent. solution of ethyl cellulose, E.C. 250, in ethanol. Then add 2·6 ml of solution containing 0·01 g-mol. of AnalaR thorium nitrate in 400 ml of ethanol, and then add a further 1 ml of the ethyl cellulose solution. Shake the mixture, dilute it to 100 ml with ethanol and add 66 ml of anhydrous toluene.

Triethylamine phthalate—Dissolve 17·8 g of phthalic anhydride in 30 ml of boiling ethanol and then add 90 ml of toluene. Cool the solution and add 11·1 g of anhydrous triethylamine and 15 ml of toluene. Dilute to 200 ml with toluene to give the stock solution. Prepare the working solution by diluting 2·8 ml of the stock solution to 100 ml with toluene.

PROCEDURE—

Place 1 ml of lake dispersion in a 25-ml calibrated flask, add to it the solution containing fluorine and dilute to the mark with the chosen solvent. At the same time and in the same solvent, prepare a range of standard solutions for comparison, these solutions being made by diluting a solution of hydrofluoric acid or ammonium fluoride in ethanol; the concentration of this solution is determined by precipitating the fluoride as lead chlorofluoride. Allow the solution to stand for 1 hour and then determine the optical density, using either a Spekker absorptiometer with Ilford No. 604 green filters or a Unicam spectrophotometer at 515 mμ. Examples of curves prepared in this way when the greater part of the solvent used was ethanol, toluene or *iso*propanol are shown in Fig. 2. When toluene is the main solvent, at least 2 ml of ethanol should be present in each 25 ml of toluene so that the diphenylguanidine salt of the dye formed in the reaction remains in solution.

Provided that a sufficient volume of solution is available, very low concentrations of fluoride can be determined by comparison in Nessler cylinders with standard solutions. For example, to 100 ml of the unknown solution of fluoride in toluene containing 5 per cent. of ethanol, add 0·2 ml of lake dispersion. Mix the solution well and allow it to stand for ½ to 2 hours. The colour is compared visually with those produced by a known set of standard solutions. Thus a solution containing 0·02 μg of ionic fluorine per ml can be readily distinguished from a blank.

The alternative method in which the unchanged lake dispersion in toluene solution is precipitated and separated is as follows. Add 4 ml of lake dispersion to not more than 20 ml of the fluoride solution in toluene. Allow the mixture to stand for half an hour at room temperature. Add 1 ml of triethylamine phthalate working solution and dilute to 25 ml with toluene, if necessary. Allow the mixture to stand for a further half an hour and filter it. If the precipitation of the unchanged lake is not complete, the solution should be heated to 60° C for half an hour. Determine the optical density of the red filtrate against a blank, using a Spekker absorptiometer and Ilford No. 604 green filters or a Unicam spectrophotometer at 515 mμ.

Many water-soluble substances that interfere with standard methods do not interfere with this method owing to their insolubility in organic solvents. However, under conditions when the optical density of a solution containing 0·8 μg of fluorine per ml was 0·033, that of a solution containing 32 μg of hydrochloric acid per ml was 0·021.

The author thanks the Chief Scientist, Ministry of Supply, for permission to publish this paper, Miss S. Williams who has performed much of the practical work and Mr. F. E. Charlton for the analysis of the primary standards.

MINISTRY OF SUPPLY
CHEMICAL DEFENCE EXPERIMENTAL ESTABLISHMENT
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A Rapid Flame-photometric Method for the Determination of Calcium in Coal Ash and Coke Ash

By L. J. EDGCOMBE and D. R. HEWETT

A method for the rapid determination of calcium in coal ash and coke ash is described. The silica is first removed and the ash is dissolved in sulphuric and hydrofluoric acids. The extent to which the commoner elements present in the resulting solution, and the acid radicles introduced, interfere with the flame-photometric determination of calcium has been determined. Methods are described for eliminating the interfering elements or for making allowance for their presence.

CALCIUM can be determined in solutions by measuring its emission in a flame photometer. The presence of other elements occurring in coal ash and coke ash and the acids used to bring the ash into solution may affect this emission, and so it is necessary to determine the degree of interference of each.

An EEL flame photometer[1] (Evans Electroselenium Ltd.) was used in this investigation and the results apply only to this particular instrument.

INTERFERENCE BY OTHER ANIONS AND CATIONS

INTERFERENCE BY ANIONS—

Nitric, hydrochloric, sulphuric and phosphoric acids were examined in concentrations covering the range 0·001 *N* to 5 *N*. First, a solution containing about 1000 p.p.m. of calcium oxide was made by dissolving reagent-grade calcium oxide in distilled water free from carbon dioxide. From this solution, a reference solution containing one-tenth of the amount of calcium oxide was made by dilution. Both solutions were kept in polythene containers. For each experiment, 10 ml of the stronger solution were transferred by pipette to a 100-ml calibrated flask and sufficient of the acid under test was added to give a required final concentration, and then the contents were diluted to the mark. The emission of this solution was compared with that of the reference solution by means of the flame photometer. As each solution contains the same amount of calcium, any difference in the emission must be due to the presence of the acid.

The results are shown in Fig. 1. Whereas the interference of hydrochloric and phosphoric acids is variable and depends on their concentrations, that of nitric and sulphuric acids, in certain ranges of concentration, is a definite fixed value. The range in which sulphuric acid produces this constant interference is much greater than the range for nitric acid. All subsequent work was therefore done with solutions in sulphuric acid within this concentration range. New standard solutions were prepared, the stronger solution containing 1000 p.p.m. of calcium oxide in 0·5 *N* sulphuric acid, and the reference standard containing 100 p.p.m. of calcium oxide in 0·5 *N* sulphuric acid.

INTERFERENCE BY CATIONS—

Various metal sulphates were added to a solution of calcium sulphate in 0·5 *N* sulphuric acid. The salts examined were the sulphates of the metals normally found in coal ashes, *i.e.*, iron, aluminium, titanium, manganese, magnesium, sodium and potassium sulphates, and ammonium sulphate. The effect of these salts was determined at concentrations of 100 p.p.m. and 1000 p.p.m., while the calcium oxide content of the solution was maintained at 100 p.p.m. The solutions were prepared as described above, and the extent of the interference is shown in Table I.

A further test showed that when the 0·5 *N* sulphuric acid was neutralised with ammonium hydroxide, which gave a concentration of ammonium ion of about 60,000 p.p.m., there was still no interference.

The group 3A metals, which cause a large error, can easily be removed, and corrections can be applied for the effects of sodium and magnesium.

TABLE I

INTERFERENCE EFFECT OF VARIOUS CATIONS

Interfering cation	Concentration of cation, p.p.m.	Error in determination of 100 p.p.m. of calcium oxide, %
Sodium	100	+ 2·5
	1000	+36·0
Potassium	100	0
	1000	+ 1·0
Iron	100	+46·4
	1000	+67·2
Aluminium	100	−45·6
	1000	−88·8
Titanium	100	−48·0
	1000	−67·6
Magnesium	100	+ 8·0
	1000	+27·2
Manganese	100	+ 1·6
	1000	+27·6
Ammonium	100	0
	1000	0

CORRECTIONS FOR SODIUM AND MAGNESIUM—

Solutions containing 100 p.p.m. of calcium oxide and from 0 to 100 p.p.m. of either magnesium oxide or sodium oxide in approximately 0·5 *N* sulphuric acid were prepared. It was found that the error produced by the sodium oxide was directly proportional to the amount present and that 100 p.p.m. of sodium oxide gave an error of +2·2 p.p.m. of calcium oxide, irrespective of the amount of calcium oxide present. But the error due to magnesium oxide was found to be proportional to both the amount of magnesium oxide and the amount of calcium oxide present; 100 p.p.m. of magnesium oxide produced an error of +8·0 per cent. of the calcium oxide present.

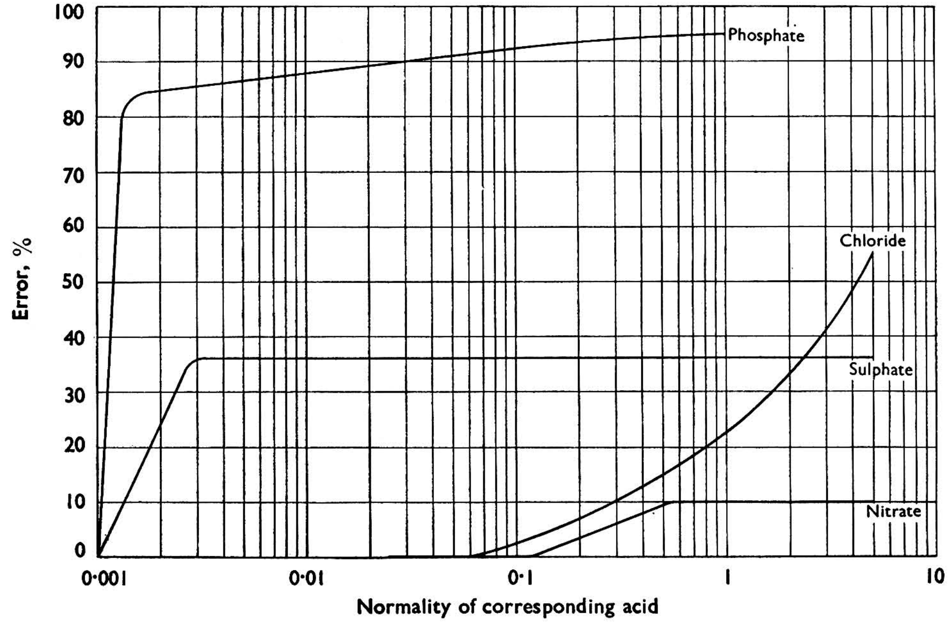

Fig. 1. Error in the determination of 100 p.p.m. of calcium oxide in the presence of acid radicles

REMOVAL OF GROUP 3A METALS—

A coal ash was examined by the usual methods and found to contain: Fe_2O_3, 20·99 per cent.; Al_2O_3, 13·46 per cent.; TiO_2, 0·63 per cent.; MgO, 6·11 per cent.; Na_2O, 0·57 per cent.; P_2O_5, 0·27 per cent.; and CaO, 13·83 per cent. Approximately 0·1 g of this ash was accurately weighed and dissolved in sulphuric and hydrofluoric acids (see method, below). The solution was heated and treated dropwise with ammonium hydroxide until it was just alkaline to methyl red. The suspension was cooled to room temperature, transferred to a 100-ml calibrated flask and diluted to the mark. The solution was mixed and about 20 ml were poured into a centrifuge tube and spun at about 3000 r.p.m. and 10 cm radius for 2 minutes. The supernatant liquid was decanted, and the calcium oxide was determined. The calcium oxide found, after correction for magnesium oxide and sodium oxide, was 12·74 per cent.

To determine whether any calcium oxide was co-precipitated with the group 3A metals, a further 0·1 g of ash was dissolved in acid as described above and the solution was diluted to 100 ml. Four 20-ml aliquots of this solution were transferred by pipette into centrifuge tubes and the metals were precipitated by the following four procedures—

(*a*) single precipitation, without washing the precipitate,
(*b*) single precipitation, washing the precipitate,
(*c*) double precipitation, without washing, and
(*d*) double precipitation, with an intervening wash.

The decanted solution was diluted to 50 ml and the calcium oxide was determined. After correction, the following percentages of calcium oxide were found—

(*a*) 11·35, (*b*) 13·20, (*c*) 13·82 and (*d*) 13·80.

It was evident that a double precipitation was necessary to recover all the calcium.

METHOD

REAGENTS—

Sulphuric acid, 20 per cent. v/v.
Hydrofluoric acid, 40 per cent. w/v—AnalaR.
Ammonium hydroxide, sp.gr. 0·880—Laboratory-reagent grade.
Standard calcium solution—Prepared from AnalaR calcium carbonate and equivalent to 50 p.p.m. of calcium oxide in approximately 0·5 *N* sulphuric acid.

PROCEDURE—

Accurately weigh about 0·1 g of ash into a platinum crucible. Add 5 ml of dilute sulphuric acid and 5 ml of hydrofluoric acid. Cover the crucible with its lid and heat it in a crucible air-bath[2] until copious white fumes are evolved. Allow the crucible to cool and place it in a 250-ml beaker containing about 50 ml of distilled water. Heat the water nearly to boiling and then remove the beaker from the source of heat. Remove the crucible and lid, rinse them and collect the rinsings in the solution; take particular care to include any traces of adhering material. Replace the beaker on the source of heat and boil until the contents are as clear as possible. (If barium is present in the ash, the solution will not clear completely.) Remove the beaker and cool it to room temperature. Wash the contents into a 100-ml calibrated flask and dilute to the mark. Mix the solution and, with a pipette, transfer 20 ml to a suitable centrifuge tube. Add 2 drops of methyl red indicator, stir the solution with a jet of air and add ammonium hydroxide dropwise until the solution just turns yellow. Place the tube in a centrifuge and spin it at 2000 to 3000 r.p.m. and 10 cm radius for 2 to 3 minutes. Remove the tube and decant the clear supernatant liquid into a 50-ml calibrated flask. Add 5 ml of dilute sulphuric acid to the centrifuge tube, rinse the sides of the tube with distilled water and dilute the solution to about 15 ml. Add 1 drop of methyl red indicator and repeat the precipitation and centrifugation as before. Decant the clear supernatant liquid into the same 50-ml calibrated flask and dilute to the mark. Using the EEL flame photometer, determine the calcium oxide in this solution by direct comparison with the standard solution containing 50 p.p.m. of calcium oxide.

CALCULATION OF RESULTS—

If s = the flame photometer reading for the standard, and
x = the flame photometer reading for the unknown.

$$\text{Then calcium oxide content (uncorrected)} = \frac{5x}{4s \times \text{sample weight in grams}} \text{ per cent.}$$

Error due to sodium oxide = 0·05 per cent. of calcium oxide for each 1 per cent. of sodium oxide.

Error due to magnesium oxide = 0·8 per cent. of the calcium oxide per cent. for each 1 per cent. of magnesium oxide.

Then calcium oxide content (corrected), per cent. =

$$\frac{\text{Percentage of calcium oxide (uncorrected)} \times 100}{100 + (0{\cdot}8 \times \text{percentage of magnesium oxide})} - 0{\cdot}05 \times \text{percentage of sodium oxide.}$$

RESULTS

Ten ashes of known composition were examined by the above procedure, and the results are shown in Table II.

TABLE II

DETERMINATION OF CALCIUM IN ASHES OF KNOWN CALCIUM CONTENT

Normal method of analysis			Flame-photometric method	
Magnesium oxide, %	Sodium oxide, %	Calcium oxide, %	Calcium oxide (uncorrected), %	Calcium oxide (corrected), %
0·40	1·19	9·86	10·02	9·95
2·82	0·74	7·63	7·83	7·61
1·81	0·84	1·00	1·06	1·01
2·25	0·88	4·03	4·02	3·92
0·28	0·14	3·38	3·42	3·40
0·34	0·18	6·68	6·56	6·54
0·09	0·09	0·62	0·66	0·66
0·76	0·12	4·45	4·52	4·48
0·59	0·07	3·43	3·45	3·43
0·25	0·04	5·75	5·82	5·81

In this series of results the maximum divergence of the uncorrected flame-photometric result from the result by the usual volumetric methods is 0·20 per cent. and the maximum divergence of the corrected result is 0·11 per cent. The ash with the abnormally high magnesium oxide content of 6·11 per cent. gave maximum divergencies of 0·83 and 0·01 per cent., respectively, from the values by the volumetric method.

DISCUSSION—

For most of the ashes encountered, the uncorrected value for calcium oxide by the flame-photometric method is satisfactory, as the normal magnesium oxide content is only 1 to 2 per cent. However, abnormal ashes are encountered and then, or with any doubtful sample, the magnesium oxide and sodium oxide content must be determined. The magnesium oxide can be determined rapidly with a polarograph and the sodium oxide with the flame photometer.[1]

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REFERENCES

1. Collins, G. C., and Polkinhorne, H., *Analyst*, 1952, **77**, 430.
2. "Improved Methods for the Quantitative Analysis of Coal Ash and Coke Ash," Fuel Research Survey Paper No. 50, H.M. Stationery Office, London, 1949.

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May 13*th*, 1954

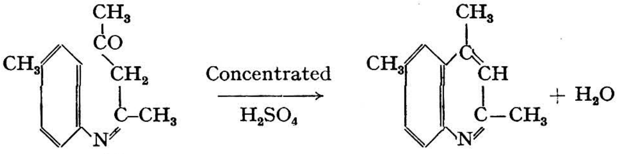
The Absorptiometric Determination of Acetylacetone with Ferric Iron

By T. G. BONNER and Miss M. THORNE

An absorptiometric method is described for the determination of acetylacetone in aqueous solution by converting it to the soluble red ferric salt. The influence of acidity and sodium sulphate on the colour intensity was investigated and the method was shown to be applicable to the determination of a Schiff's base of acetylacetone.

A CONVENIENT method of synthesising 2 : 4-dimethylquinolines in concentrated sulphuric acid by cyclisation of Schiff's bases prepared from primary aromatic amines and acetylacetone was developed by Combes,[1,2] and later the influence of nuclear substituents in the anilino portion of the Schiff's base on the yield and course of the cyclisation reaction was studied by Turner and Roberts.[3]

The cyclisation reaction is an electrophilic substitution, and a method was sought for following the rate of cyclisation in sulphuric acid - water mixtures of those Schiff's bases, such as methyl β-(*p*-toluidino)propenyl ketone (**I**), that are converted to the corresponding quinoline compound in nearly 100 per cent. yield.



(I)

As the Schiff's base (**I**) is quantitatively hydrolysed to acetylacetone and *p*-toluidine in dilute sulphuric acid, the reaction mixture can be examined by determining the acetylacetone liberated when an aliquot portion is run into water.

Pulsifer[4] showed that the red-coloured compound formed by acetylacetone and ferric iron could be used for the colorimetric determination of small quantities of ferric iron in aqueous solutions; this method has been recommended as one of the most satisfactory by Wenger and Duckert.[5] The intensity of the colour developed was shown to depend on the acidity of the solution, but not on the presence of small amounts of salts.

When the method was adapted for the determination of acetylacetone, the influence of acidity and of concentration of ferric salt and sodium sulphate was investigated, and so the optimum conditions for the determination were established.

EXPERIMENTAL

REAGENT—

Ferric ammonium sulphate solution—A 6·5 per cent. w/v solution in 0·1 *N* sulphuric acid.

PROCEDURE FOR THE DETERMINATION OF ACETYLACETONE IN AQUEOUS SOLUTION—

To 5 ml of the ferric ammonium sulphate solution, add a measured volume (1 to 15 ml) of the acetylacetone solution, which must be 0·1 *N* with respect to sulphuric acid, and then add an appropriate volume of 0·1 *N* sulphuric acid so that the final volume is 20 ml. Allow the solution to stand for 15 minutes, when the colour develops its full intensity. Prepare a blank solution by substituting an equal volume of 0·1 *N* sulphuric acid for the acetylacetone solution. Measure the colour in a Spekker photo-electric absorptiometer, using an Ilford No. 603 blue-green filter. There should be no alteration of colour intensity after the solution has stood for a further hour.

ABSORPTION SPECTRUM OF THE FERRIC - ACETYLACETONE COMPOUND—

This was studied with a Uvispek spectrophotometer. The colour was developed as described above, except that only 1 ml of the reagent was used; the final solution contained

1·75 mg of acetylacetone in 20 ml ($8·73 \times 10^{-4}$ moles per litre). The results are shown graphically in Fig. 1, the maximum absorption being at 484 mμ.

INFLUENCE OF THE ACIDITY AND AMOUNT OF REAGENT ON COLOUR PRODUCTION—

By the procedure described above and with the final solution always containing 2·00 mg of acetylacetone, the acidity was varied over the range 0·001 to 0·300 *N* with respect to sulphuric acid; the optical density of the different solutions was found to decrease continuously from 0·832 to 0·477 with increase of acidity. It is therefore essential to maintain the acidity at some arbitrarily chosen value for the standard procedure, and 0·1 *N* sulphuric acid was used for this purpose.

The optical density depends on the concentration of the ferric reagent. In preliminary experiments with a 5 per cent. w/v ferric ammonium sulphate solution in 0·1 *N* sulphuric acid, variations in the volume of reagent solution used, between 1 and 9 ml in a total volume of 20 ml, resulted in a change of optical density from 0·260 to 0·656. The variation was least marked in the region of 6 to 6·5 ml of this ferric solution. These experiments led to the use of 5 ml of a 6·5 per cent. w/v ferric ammonium sulphate solution in a total volume of 20 ml for the proposed procedure.

TABLE I

EFFECT OF ADDED SODIUM SULPHATE ON COLOUR PRODUCTION

	Optical density		
Acetylacetone, mg	No added sodium sulphate	1·04 g of sodium sulphate in 20 ml of final solution	0·39 g of sodium sulphate in 20 ml of final solution
2·45	0·768	0·748	
2·25	0·719	0·689	0·689
2·04	0·650	0·632	
1·83	0·584	0·574	0·573
1·63	0·524	0·511	
1·43	0·461	0·452	0·452
1·23	0·395	0·388	
1·02	0·325	0·327	

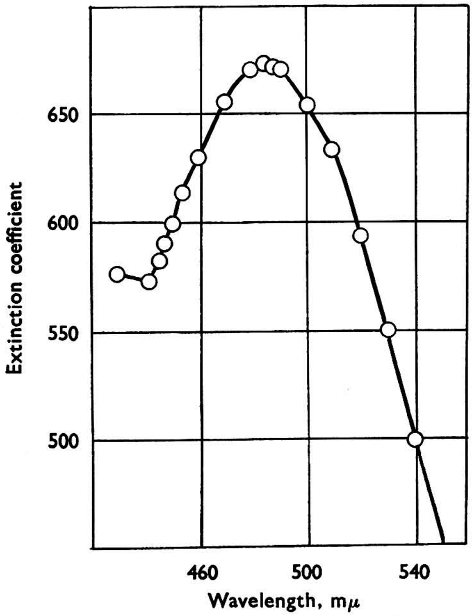


Fig. 1. Absorption curve for the ferric - acetylacetone complex

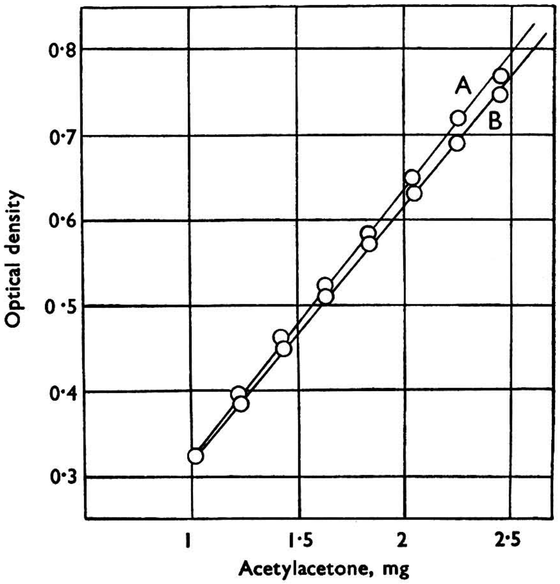


Fig. 2. Calibration curves for the ferric salt of acetylacetone: curve A, no added sodium sulphate; curve B, 1·04 g of sodium sulphate in 20 ml of final solution

EFFECT OF SODIUM SULPHATE ON COLOUR PRODUCTION—

As the acidity of the acid solutions of acetylacetone used in the kinetic experiments had to be adjusted by the addition of sodium hydroxide solution, the final solution to be examined absorptiometrically contained considerable amounts of sodium sulphate (about 0·5 g of sodium sulphate per 5-ml aliquot of the acetylacetone solution).

The effect of the presence of this salt on the colour production is shown in Fig. 2. The linear relationship between the optical density and concentration of acetylacetone is maintained, but with a slightly altered slope. Further, as indicated in Table I, it appears that the influence of the sodium sulphate is constant within the range of 0·40 to 1·00 g of salt per 20 ml of final solution.

TABLE II

DETERMINATION OF THE SCHIFF'S BASE (I) BY HYDROLYSIS

Aliquots used, ml	Amount of Schiff's base (I), g	Acetylacetone Calculated, mg	Acetylacetone Found, mg	Recovery, %
5	0·3485	1·84	1·84	100
10	0·2003	2·12	2·10	99·1
15	0·1006	1·60	1·56	97·5

APPLICATION OF THE METHOD TO THE DETERMINATION OF METHYL β-(*p*-TOLUIDINO)PROPENYL KETONE (I)—

Various weights of the Schiff's base (**I**) were dissolved in 100-ml portions of *N* sulphuric acid and the solutions were allowed to stand for 2 hours to effect complete hydrolysis to acetylacetone. The acid solutions were neutralised by titration with *N* sodium hydroxide solution, universal indicator paper being used to establish the end-point. Then 50 ml of *N* sulphuric acid were added and the solution was diluted to 500 ml with water. Aliquot portions were removed for the determination of acetylacetone; the results are shown in Table II and indicate that the method is applicable to the analysis of Schiff's bases of acetylacetone.

In kinetic experiments 1 g of the Schiff's base was dissolved rapidly in 25 ml of the concentrated sulphuric acid medium at 25° C at zero time and 2-ml aliquots were withdrawn at fixed time intervals and run into 20 ml of water. This dilution reduced the acidity to about 2 *N* and, on standing for 2 hours, complete hydrolysis of the unchanged Schiff's base occurred. The solution was neutralised as described above and, after filtration, was diluted exactly to 100 ml with water after the addition of 10 ml of *N* sulphuric acid, to give an exactly 0·1 *N* acid solution. A 5-ml or 10-ml aliquot portion of this solution was then removed and its acetylacetone content was determined by the standard procedure. The rate constants determined by this method were satisfactory.

Acknowledgments are made to Imperial Chemical Industries Limited and to the Central Research Fund for financial assistance.

REFERENCES

1. Combes, A., *Compt. Rend.*, 1887, **106**, 142.
2. ——, *Bull. Soc. Chim. France*, 1888, **49**, 90.
3. Turner, E. E., and Roberts, E., *J. Chem. Soc.*, 1927, 1832.
4. Pulsifer, H. B., *J. Amer. Chem. Soc.*, 1904, **26**, 967.
5. Wenger, P., and Duckert, R., *Helv. Chim. Acta*, 1944, **27**, 757.

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June 8th, 1954

Absorptiometric Determination of Magnesium in Titanium and its Alloys

BY H. J. G. CHALLIS AND D. F. WOOD

Procedures have been established for the determination of magnesium in titanium and its alloys. For the examination of titanium metal, the method involves the preliminary separation of titanium by extraction with cupferron - chloroform followed by the absorptiometric determination of magnesium, making use of Titan Yellow. Aluminium, chromium and copper interfere, but modifications are described so that interference due to the amounts likely to be present is avoided. Tests have established that the Titan Yellow method is reliable and reproducible for magnesium contents between 0·02 and 1·0 per cent. and is more rapid and convenient for routine batch analysis than the conventional phosphate method, particularly for amounts of magnesium below 0·1 per cent.

In the analysis of titanium alloys, removal of titanium and alloying elements is necessary and is effected by separations involving the use of cupferron, zinc oxide - permanganate and sodium hydroxide - cyanide. The final determination of magnesium is accomplished by the Solochrome Cyanine R.200 method. The recoveries have been good from synthetic solutions and a number of alloys.

MAGNESIUM is a likely impurity in titanium sponge produced by the Kroll process, and although procedures for its determination have been described,[1,2] these are restricted to amounts greater than 0·1 per cent. in titanium and furthermore they are not applicable to alloys. For smaller amounts of magnesium it has been necessary, therefore, to use more sensitive colorimetric methods and to develop modifications suitable for the determination of magnesium in titanium alloys.

Part I. Determination of Magnesium in Titanium

SEPARATION OF TITANIUM—

Colorimetric reactions for magnesium are not specific and so it was considered essential to effect preliminary separation of titanium before applying such reactions. The extraction by the cupferron - chloroform technique was selected as the most suitable method of separating titanium from small amounts of magnesium in sponge and ingots. In sulphuric and hydrochloric acid solutions (10 per cent. by volume) this extraction removes titanium and impurities such as iron, tin and vanadium, so that the magnesium, together with any traces of aluminium, chromium, copper, manganese, molybdenum, nickel and alkali or alkaline-earth metals, remains in the aqueous phase.[3,4] Next, a sensitive colour reaction for the absorptiometric determination of magnesium was sought.

ABSORPTIOMETRIC DETERMINATION WITH TITAN YELLOW—

Many organic reagents depending on the formation of coloured lakes of colloidal magnesium hydroxide are available for the colorimetric determination of magnesium.[5,6,7,8] Titan Yellow was selected for trial as much information has been published about this reagent.

Initial experiments with Titan Yellow made use of hydroxylamine hydrochloride as the stabilising reagent,[5] but above concentrations of 3·2 p.p.m. of magnesium, the complex coagulated after 10 minutes. By using starch and glycerol as protective colloids,[5,6] in addition to hydroxylamine hydrochloride, it was found that at concentrations of 1 to 4 p.p.m. of magnesium and with the particular dye used (British Drug Houses Ltd.), the complex formed after 5 minutes was stable for at least two hours.

Based on information in the literature,[5,6] a calibration curve was prepared as described on p. 767. Synthetic solutions containing 0·5 g of refined titanium (van Arkel) and magnesium equivalent to 0·02 to 0·10 per cent. were prepared. After removal of the titanium as the complex with cupferron, the magnesium in the aqueous extract was determined by the

Titan Yellow method. The results were satisfactory over the range tested and were as follows—

Magnesium added, %	0·0	0·020	0·040	0·060	0·080	0·100	0·200
Magnesium recovered, %	0·002	0·022	0·041	0·060	0·078	0·100	0·195

EFFECT OF IMPURITIES ON THE TITAN YELLOW METHOD—

The effect of various impurities on the apparent magnesium content, at a concentration of 1 to 4 p.p.m. of magnesium in the final solutions, was ascertained, and the results are summarised as follows—

Aluminium. When present up to 2 p.p.m., it has only a slight effect, but 4 and 8 p.p.m. cause a marked decrease in optical density in the lower range of magnesium concentrations, as shown in Fig. 1. In the recommended procedure, however, aluminium is removed as the sparingly soluble sulphate by filtration, so that the amount of aluminium remaining in solution has a negligible effect on the determination (see results in Table I). Alternatively, the interference due to aluminium can be corrected by precipitating the aluminium with ammonium hydroxide, iron being used as a collector,[5,6] and removing any ammonia or ammonium salts by boiling with sodium hydroxide.

TABLE I

EFFECT ON INTERFERENCE BY ALUMINIUM OF FUMING WITH SULPHURIC ACID AND REMOVING ALUMINIUM SULPHATE BY FILTRATION

Magnesium added,* %	Aluminium added, %	Time fumed with sulphuric acid, minutes	Magnesium found by Titan Yellow method, %
0·02	Nil	10	0·022
0·02		10	0·022
0·10		10	0·10
0·10		10	0·095
0·02	0·1	Nil	Nil
0·02		10	0·018
0·10		Nil	0·08
0·10		10	0·10
0·04		Nil	0·02
0·04		10	0·04

* The magnesium was added to 0·5-g samples of refined titanium (van Arkel).

Chromium. It has an effect similar to that of aluminium, but the amount present is unlikely to exceed 1 p.p.m.

Copper. This interferes at concentrations as small as 0·01 per cent., but in the presence of potassium cyanide, the interference is negligible at concentrations less than 1 p.p.m. (0·025 per cent.).

Iron. At 1 p.p.m. it causes a slight decrease in absorption values.

Manganese. In amounts up to 8 p.p.m. (0·2 per cent.) the effect is slight, but 20 p.p.m. cause an appreciable increase in absorption.

Nickel and vanadium. They have a negligible effect at concentrations below 8 p.p.m.

Titanium. After separation with cupferron, a residue of 6 p.p.m. of titanium causes only a slight increase in absorption, but at 17 p.p.m. the effect is significant, particularly with more than 2 p.p.m. of magnesium.

Ammonium and sodium sulphates. Information on the effect of these two salts was required in connection with the proposed separation of aluminium as hydroxide. The absorption progressively increases with concentration of ammonium sulphate, whereas sodium sulphate in concentrations up to N has no effect on 1·6 p.p.m. of magnesium.

EFFECT OF TEMPERATURE ON THE TITAN YELLOW METHOD—

The results shown in Table II indicate that over the range of magnesium concentrations tested, control of the temperature at 25° ± 5° C is adequate.

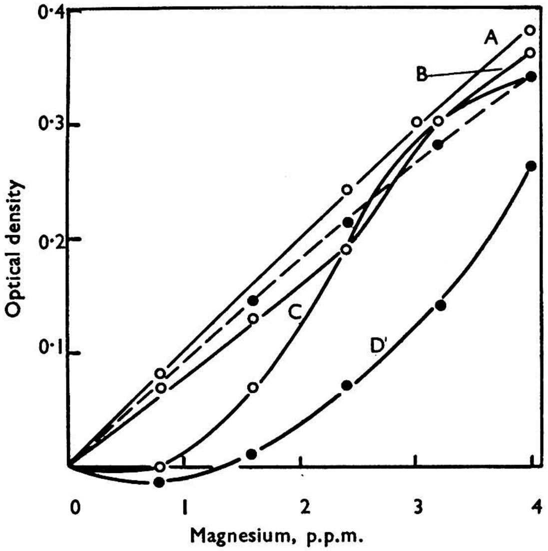

Fig. 1. Effect of aluminium on the determination of magnesium with Titan Yellow. Curve A, magnesium plus 1 p.p.m. of aluminium; curve B, magnesium plus 2 p.p.m. of aluminium; curve C, magnesium plus 4 p.p.m. of aluminium; curve D, magnesium plus 8 p.p.m. of aluminium; dotted line, magnesium alone. All concentrations calculated on final volume of 50 ml

APPLICATION OF TITAN YELLOW METHOD—

The colorimetric method described below was used for a number of samples of titanium and the results are compared in Table III with those determined by the gravimetric phosphate method. Furthermore, samples of titanium were selected for reproducibility tests and four determinations were made by the Titan Yellow method on each sample, these results being shown in Table IV.

TABLE II

EFFECT OF TEMPERATURE ON TITAN YELLOW METHOD

	16° C		25° C		35° C	
Magnesium added, %	Optical density	Magnesium (less blank), %	Optical density	Magnesium (less blank), %	Optical density	Magnesium (less blank), %
0	0·13	—	0·14	—	0·14	—
0·02	0·20	0·02	0·21	0·02	0·215	0·022
0·04	0·27	0·04	0·28	0·04	0·28	0·04
0·08	0·40	0·08	0·41	0·08	0·42	0·082

The results in Tables III and IV indicate that the precision of the Titan Yellow method is satisfactory. It is more suitable than the phosphate procedure for magnesium contents below about 0·1 per cent.

COLORIMETRIC DETERMINATION WITH SOLOCHROME CYANINE R.200—

While the Titan Yellow method was being investigated, a new colorimetric method for the determination of magnesium in aluminium alloys was described by Bacon,[9] who claimed that the reagent Solochrome Cyanine R.S. is less susceptible to interference from impurities. Aluminium and copper had less effect on the reaction of magnesium with Solochrome Cyanine

R.200 (see Fig. 2) than on that with Titan Yellow. Calcium interfered and its effect was compensated by the addition of an equal amount to the blank (see results in Table V). However, when applying the test to samples, it was necessary, after the initial separation of titanium as the complex with cupferron, to precipitate the magnesium with sodium hydroxide and then to dissolve the precipitate in a standard volume of *N* sulphuric acid in order to eliminate the effect of variation of the concentration of salts. This procedure gave satisfactory recoveries of magnesium (0·02 to 1·0 per cent.) from synthetic solutions. Determinations were performed on solutions of 0·5-g samples of refined titanium (van Arkel), and the results by the Solochrome method were as follows—

TABLE III

COMPARISON OF PHOSPHATE AND COLORIMETRIC METHODS WHEN APPLIED TO TITANIUM SPONGE AND INGOTS

Magnesium by phosphate method, %	Magnesium by Titan Yellow method, %	Magnesium by Solochrome Cyanine method, %
0·15	0·13	0·14
0·21	0·21	
0·14	0·13	0·14
0·86	0·80	
1·28	1·32	1·20
∼0·04	0·052	
∼0·06	0·05	
∼0·03	0·03	0·032
∼0·04	0·04	0·037
∼0·04	0·035	
∼0·04	0·06	
∼0·05	0·060	
0·27	0·25	
0·78	0·73	
∼0·08	0·082	0·084
∼0·08	0·076	0·084
0·18	0·20	0·18
0·17	0·20	0·19
0·32	0·32	0·33
0·07	0·076	0·081
0·22	0·18	0·22
—	0·015	0·013
—	0·014	0·014

TABLE IV

REPRODUCIBILITY OF TITAN YELLOW METHOD FOR DETERMINATION OF MAGNESIUM IN TITANIUM

Sample	Test 1. Magnesium, %	Test 2. Magnesium, %	Test 3. Magnesium, %	Test 4. Magnesium, %
A	0·28	0·27	0·28	0·28
B	0·31	0·31	0·29	0·30
C	0·18	0·18	0·19	0·18
D	0·045	0·045	0·042	0·048
E	0·52	0·50	0·54	0·54
F	0·13	0·12	0·12	0·14

Magnesium added, % ..	0	0·02	0·04	0·06	0·08	0·10	0·20	0·40	0·60	0·80	1·00
Magnesium recovered, % ..	0·002	0·022	0·042	0·060	0·078	0·096	0·20	0·38	0·60	0·79	1·02

The results for various samples were in agreement with those determined by the phosphate and Titan Yellow methods (see Table III). Although it was found that the colorimetric method with Solochrome Cyanine R.200 was simpler and quicker than that with Titan Yellow, this advantage was outweighed by the necessity for separating magnesium as hydroxide and accurate control of the temperature when measuring the absorption due to the magnesium complex. Titan Yellow is therefore preferred for the determination of magnesium in titanium metal and sponge. As indicated later, however, when aluminium, chromium, and so on,

TABLE V

INFLUENCE OF CALCIUM ON THE SOLOCHROME CYANINE METHOD FOR MAGNESIUM IN THE ABSENCE OF TITANIUM

Magnesium present,* %	Calcium added,* %	Optical density	Optical density less blank	Magnesium recovered, %
Nil	Nil	0·33	—	—
Nil	0·10	0·35	—	—
0·02	0·10	0·43	0·08	0·02
0·04	0·10	0·51	0·16	0·04
0·08	0·10	0·67	0·32	0·08
0·10	0·10	0·76	0·41	0·10
Nil	Nil	0·33	—	—
Nil	0·4	0·41	—	—
0·02	0·4	0·49	0·08	0·02
0·04	0·4	0·57	0·16	0·04
0·08	0·4	0·735	0·325	0·082
0·10	0·4	0·825	0·415	0·102

* Calculated on 0·25-g sample.

are present in alloying amounts, separation of magnesium with sodium hydroxide is essential for both colorimetric methods, so that the Solochrome Cyanine method can then be applied advantageously.

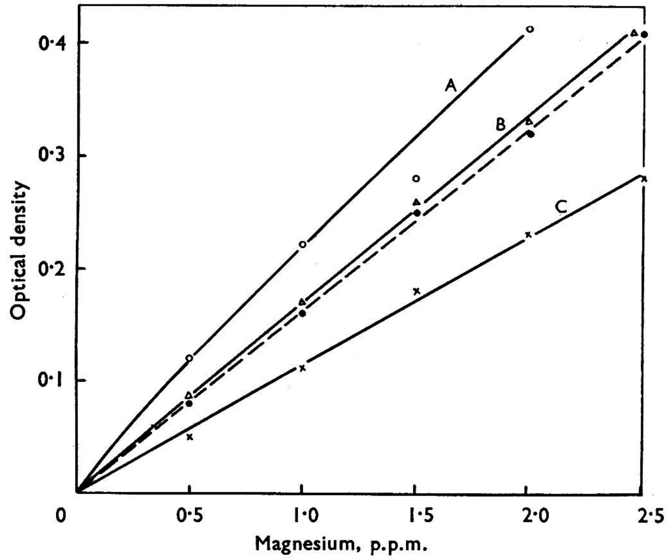

Fig. 2. Effect of copper, iron and aluminium on the determination of magnesium with solochrome cyanine R.200. Curve A, magnesium plus 4 p.p.m. of copper; curve B, magnesium plus 4 p.p.m. of aluminium; curve C, magnesium plus 5 p.p.m. of iron; dotted line, magnesium alone. All concentrations calculated on final volume of 100 ml

TITAN YELLOW METHOD FOR THE DETERMINATION OF MAGNESIUM IN TITANIUM METAL OR SPONGE

REAGENTS—

Hydrofluoric acid, 10 *per cent.*—Dilute 10 ml of analytical-reagent grade hydrofluoric acid to 100 ml and store it in a polythene container.

Hydrogen peroxide, 100-*volume.*—Analytical-reagent grade.

Sulphuric acid, sp.gr. 1·84.

Cupferron solution, 9 per cent.—A freshly prepared aqueous solution, filtered before use.

Chloroform.

Nitric acid, sp.gr. 1·42.

Sodium hydroxide, 20 *per cent. w/v aqueous solution.*

Hydroxylamine hydrochloride, 5 *per cent. aqueous solution.*

Calcium chloride solution, 5·5 *per cent.*—Dissolve 5·5 g of analytical-reagent grade calcium chloride, $CaCl_2.6H_2O$, in 100 ml of 0·01 *N* hydrochloric acid.

Starch solution, 1 *per cent.*—Make a paste of 1 g of soluble starch with cold water, pour it into 80 ml of boiling water, boil the solution for a few minutes, cool it and dilute it to 100 ml. This solution should be freshly prepared and filtered immediately before use.

Glycerol solution—Dilute 50 ml of glycerol with 50 ml of water.

Potassium cyanide solution, 0·05 *per cent.*—Dissolve 0·05 g of potassium cyanide in 100 ml of water.

Titan Yellow solution, 0·1 *per cent.*—Dissolve 0·1 g of Titan Yellow in 100 ml of water. This solution must not be kept longer than one week. As different batches of Titan Yellow may vary in reactivity, it is essential to prepare a fresh calibration curve for each batch of reagent.

Standard magnesium solution—Dissolve 0·1 g of pure magnesium in a mixture of 9 ml of water and 1 ml of sulphuric acid, sp.gr. 1·84, and dilute to 1 litre.

PROCEDURE—

Preparation of calibration curve for 0 *to* 4 *p.p.m. of magnesium*—Take 0·4, 0·8, 1·2, 1·6 and 2-ml portions of the standard magnesium solution and place them in 50-ml calibrated flasks, with an additional flask for the blank. Dilute each solution to about 20 ml and add the six reagents described on p. 768, commencing with the hydroxylamine hydrochloride. Neutralise the solution with sodium hydroxide solution and then add a further 1 ml of the sodium hydroxide. Dilute the solution to the mark, mix it well by shaking and measure the optical density using a Spekker absorptiometer with a tungsten lamp, a 2-cm cell and an Ilford No. 605 filter. From the readings construct a calibration graph.

Separation of titanium—Weigh 0·5 g of titanium metal into a platinum dish and add 10 ml of water followed by 10 ml of 10 per cent. hydrofluoric acid (for the treatment of titanium sponge see Note 1). A blank should be determined for the reagents. When the sample has dissolved, add 1 ml of 100-volume hydrogen peroxide and allow the solution to cool, then add 5 ml of sulphuric acid, sp.gr. 1·84, and evaporate it until fumes are evolved. Continue heating the mixture for two minutes, cool it, dilute it to about 50 ml with water, filter the solution if necessary and transfer it to a 500-ml separating funnel.

Add 90 ml of cupferron solution, then shake the suspension for 1 minute to coagulate the precipitate and add 90 ml of chloroform. Shake the funnel for half a minute, allow the layers to separate and remove the chloroform layer. Add 40 ml of chloroform, shake the funnel for half a minute, allow the layers to separate and add a few drops of cupferron solution to the upper layer. A white flash of cupferron indicates complete precipitation of titanium. If a coloured precipitate forms, add a further 10 ml of cupferron solution and shake. Remove the chloroform layer and continue the extraction with 20-ml portions of chloroform until the two layers are colourless.

Transfer the aqueous layer to a 400-ml beaker, introduce a boiling rod and evaporate to about 10 ml. Destroy the excess of cupferron by adding 25 ml of nitric acid, sp.gr. 1·42, and evaporate the solution until fumes of sulphur trioxide are evolved. Continue the evaporation until only 1 to 2 ml of solution remain and then allow it to cool. Add 10 ml of water to dissolve magnesium and ammonium sulphates (Note 2). Cool the solution and filter it through a 9-cm Whatman No. 40 filter-paper (to remove any insoluble aluminium sulphate) into a 50-ml calibrated flask. Dilute to the mark with distilled water; this is Solution A.

Determination of magnesium with Titan Yellow—Using a pipette, transfer a suitable aliquot of Solution A to a 50-ml flask so that the final volume contains 1 to 4 p.p.m. of magnesium. For example, for amounts up to 0·1 per cent. take 20 ml of Solution A, or for amounts from 0·1 to 0·5 per cent. take 4 ml of Solution A. An aliquot equal to that taken from the sample should be taken from the blank solution and put in a 50-ml flask. If the approximate amount of magnesium is not known, make a preliminary test on a 4-ml aliquot. Amounts up to 1 per cent. can be determined by diluting a portion of Solution A and then taking an aliquot suitable for the range of the calibration graph.

Add the following reagents in the given order to both sample and blank aliquots—

1 ml of hydroxylamine hydrochloride solution,
1 ml of calcium chloride solution,
2 ml of starch solution,
2 ml of glycerol solution,
1 ml of potassium cyanide solution and
1 ml of Titan Yellow solution.

Then add sodium hydroxide solution dropwise from a burette, while shaking the solution, until the pale green colour produced just begins to turn orange.

At this point add 1 ml more of sodium hydroxide solution and dilute to the mark. Adjust the temperature of the sample and the blank to 25° ± 5° C. Mix the solution well and transfer a part to a 2-cm cell. Measure the optical density of the sample and blank with a Spekker absorptiometer, a tungsten lamp and an Ilford No. 605 yellow-green filter. Deduct the blank reading (usually about 0·08) from that for the sample, then determine the amount of magnesium in the solution from the calibration graph and convert it to percentage of magnesium.

NOTES—

1. For titanium sponge, which is heterogeneous, it is advisable to dissolve a large amount of the sample in sulphuric acid, dilute the solution to a definite volume and take an aliquot corresponding to a 0·5-g sample. For example, 25 g of sponge are dissolved in 750 ml of diluted sulphuric acid (1 + 3) and the solution is diluted to 1 litre. To a 20-ml aliquot of this solution add 1 ml of 100-volume hydrogen peroxide, evaporate the solution until fumes are evolved and then continue the procedure as described for titanium metal.

2. If chromium is present at concentrations greater than 2 p.p.m., it should be removed together with aluminium as follows. Add 1 ml of a 1 per cent. ferrous iron solution (1 g of iron wire dissolved in diluted hydrochloric acid (1 + 1) and the solution diluted to 100 ml) to reduce chromate and to act as a carrier. Oxidise the solution with nitric acid, neutralise it with ammonium hydroxide, filter it, add 10 ml of 10 per cent. sodium hydroxide and boil it to remove ammonium salts. Separate the magnesium hydroxide by filtration and dissolve it in 10 ml of sulphuric acid, dilute to 50 ml and continue as described above.

Part II. Determination of Magnesium in Titanium Alloys

REMOVAL OF ALLOYING ELEMENTS—

Previous experience in the analysis of titanium sponge suggested that the alloying metals, *e.g.*, aluminium, chromium and manganese, would make special treatment necessary. By analogy with Smith's method[10] for the determination of magnesium in aluminium alloys, it seemed probable that aluminium could be satisfactorily removed by the zinc oxide - permanganate separation and the magnesium then precipitated by the sodium hydroxide - cyanide method. In this way adequate control of salt concentration can be maintained and the determination of magnesium can be conveniently completed by the Solochrome Cyanine method.[9]

TABLE VI

DETERMINATION OF MAGNESIUM IN SYNTHETIC TITANIUM ALLOYS BY THE SOLOCHROME CYANINE METHOD

Magnesium added, %	Synthetic alloy of titanium with the addition of the following	Magnesium recovered, %
0·02 0·04 0·08	5% of Al, 5% of Mn and 5% of Cr	0·018 0·036 0·08
0·02 0·04 0·08	10% of Al and 10% of Mn	0·018 0·038 0·076
0·02 0·04 0·08 0·20 0·60 0·80	10% of Al, 5% of Mn and 5% of Cr	0·022 0·038 0·080 0·20 0·58 0·77

To test this proposed method, solutions containing 0·5 g of titanium and salts of aluminium, chromium, copper, manganese and nickel equivalent to 1 per cent. together with

0·02 to 0·10 per cent. of magnesium were analysed and the results were satisfactory. The efficiency of the separation was further confirmed by tests on a series of alloys containing amounts of aluminium and manganese up to 10 per cent. and chromium up to 5 per cent. with various amounts of magnesium. The results are shown in Table VI.

In addition, tests were made on drillings from titanium ingots containing 0·06 to 0·3 per cent. of magnesium to which were added 1 per cent. of aluminium, 2 per cent. of chromium, 1 per cent. of copper, 1 per cent. of iron and 1 per cent. of manganese as sulphates. The results were as follows—

Magnesium in titanium ingot, %	0·060	0·080	0·083	0·20	0·32
Magnesium recovered from solution containing other metals, %	0·056	0·086	0·086	0·22	0·32

These results were satisfactory and so the following method is recommended.

SOLOCHROME CYANINE METHOD FOR THE DETERMINATION OF MAGNESIUM IN TITANIUM ALLOYS

REAGENTS—

The first seven reagents used in the Titan Yellow method, with the addition of the following—

Hydrochloric acid, sp.gr. 1·18.

Sulphuric acid, diluted (1 + 4)—Carefully add 200 ml of sulphuric acid, sp.gr. 1·84, to 800 ml of water.

Potassium permanganate, approximately 0·2 N—Dissolve 6·5 g of potassium permanganate in 1 litre of water.

Zinc oxide emulsion—Mix 50 g of analytical-reagent grade zinc oxide with 300 ml of water. Shake the solution before use.

Ethanol, 90 *per cent.*—Analytical-reagent grade.

Potassium cyanide, 10 *per cent. solution*—Dissolve 10 g of potassium cyanide in 100 ml of water.

Sulphuric acid, approximately N—Carefully add 27·5 ml of sulphuric acid, sp.gr. 1·84, to 500 ml of water, cool and dilute to 1 litre.

Ammonium hydroxide, sp.gr. 0·880.

Solochrome Cyanine solution—Dissolve 0·25 g of Solochrome Cyanine R.200 in 600 ml of water containing 2 ml of 20 per cent. sodium hydroxide solution and dilute to 1 litre. Prepare a fresh solution each week.

Standard magnesium solution—Dissolve 0·25 g of magnesium in 100 ml of *N* sulphuric acid and dilute to 1 litre.

PROCEDURE—

Preparation of calibration curve—Add 0, 0·2, 0·4, 0·6, 0·8 and 1·0-ml portions of the standard magnesium solution to six 100-ml calibrated flasks. To each flask add 5 ml of *N* sulphuric acid, 25 ml of ammonium hydroxide, sp.gr. 0·880, and 20 ml of Solochrome Cyanine solution and then dilute to the mark.

Mix the solution well, transfer portions of each solution to 2-cm cells and determine extinction values with a Spekker absorptiometer, a tungsten lamp and an Ilford No. 605 filter. Deduct the blank value and plot extinction values against magnesium concentration.

Separation of metals—Weigh 0·5 g of sample into a platinum dish and proceed as described for titanium metal on p. 767 until, after extraction with chloroform, the solution is free from titanium. Transfer the aqueous layer to a 400-ml beaker and evaporate it to about 10 ml. Add 25 ml of nitric acid, sp.gr. 1·42, to oxidise organic matter and evaporate the solution just to fumes of sulphur trioxide. Cool the residue and carefully add 100 ml of water, 5 ml of hydrochloric acid, sp.gr. 1·18, mix, boil the solution for about 10 minutes to dissolve any solid salts and then filter it through a No. 41 filter-paper into a 400-ml beaker. Add sodium hydroxide solution until the solution is just alkaline to litmus paper (about 30 ml are required). Add a few drops of diluted sulphuric acid followed by 5 ml of 0·2 *N* potassium permanganate and heat to boiling. Add 30 ml of zinc oxide emulsion and continue the addition of potassium permanganate until the liquid remains pink on boiling for about 3 minutes after the last addition. Remove the beaker from the source of heat and destroy the excess of permanganate with a few drops of ethanol. Boil the solution, cool it and dilute it to 500 ml in a calibrated flask. Shake the mixture well, allow the precipitate to settle and filter 250 ml of the supernatant liquid through a No. 540 filter-paper into a calibrated flask.

Transfer 250 ml to a 400-ml beaker, add 10 ml of potassium cyanide solution, 20 ml of sodium hydoxide and a small amount of paper-pulp and boil the solution until it is free from ammonia (about 25 minutes).

Filter the solution through a No. 540 filter-paper, and wash the filter-paper and magnesium hydroxide precipitate with 0·01 *N* sodium hydroxide, carefully removing all precipitate from the beaker by means of a rubber-tipped glass rod. Dissolve the magnesium hydroxide in 10 ml of *N* sulphuric acid into a 100-ml calibrated flask, wash the filter-paper with water and dilute to the mark; this is Solution B.

Determination of magnesium at a concentration of 0 *to* 1 *per cent. with Solochrome Cyanine R.*200—Place a 10-ml aliquot of Solution B in a 100-ml calibrated flask, add 4 ml of *N* sulphuric acid, 25 ml of ammonium hydroxide, sp.gr. 0·880, and 20 ml of Solochrome Cyanine R.200 solution. Dilute to the mark, mix the solution and measure the extinction within 15 minutes at 20° ± 1° C, using a Spekker absorptiometer with a 2-cm cell, tungsten lamp and an Ilford No. 605 filter. Deduct the blank reading from that of the sample, then determine the magnesium content of the solution from the calibration graph and convert it to percentage of magnesium.

Determination of magnesium at a concentration of 0 *to* 0·2 *per cent. with Solochrome Cyanine R.*200—Place a 50-ml aliquot of Solution B in a 100-ml calibrated flask, and add 25 ml of ammonium hydroxide, sp.gr. 0·880, and 20 ml of Solochrome Cyanine solution. Dilute to the mark, measure the extinction as described above and calculate the magnesium content.

CONCLUSIONS

Following the experimental work described in Part I and II of the paper, two absorptiometric methods for magnesium are recommended. These are the use of Titan Yellow for titanium metal and sponge and of Solochrome Cyanine R.200 for titanium alloys.

Both methods are more rapid and accurate for magnesium in concentrations below 0·1 per cent. than the phosphate gravimetric procedure.[1,2]

The Titan Yellow method is satisfactory provided that only the usual impurities are present. Of these impurities, aluminium interferes seriously and its presence necessitates a modification of technique in which the aluminium is removed as sulphate or hydroxide. Separation of calcium, which is essential with the phosphate method, is not necessary with Titan Yellow; in fact, with the latter reagent, calcium is added to intensify the colour. The Titan Yellow method has now been in use in a routine laboratory for over a year and no difficulties have been encountered, even when the technique has been used by inexperienced personnel.

With titanium alloys, separation of titanium and alloying metals is necessary before application of the Solochrome Cyanine reaction. The procedure recommended for the determination of magnesium in alloys has been applied without difficulty to alloys containing aluminium, chromium, iron and manganese. Tests indicate that the method should be applicable to a wide range of alloys.

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REFERENCES

1. "Handbook on Titanium Metal," Titanium Metals Corporation of America, 1950, p. 44.
2. Corbett, J. A., *Analyst*, 1953, **78**, 20.
3. Westwood, W., and Mayer, A., "Chemical Analysis of Cast Iron and Foundry Materials," George Allen and Unwin Ltd., 1951, p. 26.
4. Sandell, E. B., "Colorimetric Determination of Traces of Metals," Second Edition, Interscience Publishers Inc., New York, 1950, p. 142.
5. ——, *op. cit.*, 1950, p. 419.
6. Johnson, E. A., "Organic Reagents for Metals—Monograph No. 4, Titan Yellow," Hopkin and Williams Ltd., Chadwell Heath, Essex, 1952.
7. Sandell, E. B., *op. cit.*, 1950, p. 425.
8. "Organic Reagents for Metals," Fourth Edition, Hopkin and Williams Ltd., London, 1943, p. 126.
9. Bacon, A., *Metallurgia*, 1951, **44**, 207.
10. Smith, G. S., *Analyst*, 1935, **60**, 812.

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Potentiometric Method for the Macro- and Micro-determination of Thallium by Oxidation with Potassium Permanganate in Alkaline Solutions

By I. M. ISSA and R. M. ISSA

A method is described for the potentiometric determination of thallium. It depends on the oxidation of thallium^{I} to thallium^{III} in alkaline solution (preferably about 0·1 *N* with respect to sodium hydroxide) with potassium permanganate. If the thallium is in the tervalent state, it is first reduced with sulphur dioxide in acid solution. The method can be used for the determination of 5 μg of thallium.

THALLIUM^{I} has been determined gravimetrically as iodide[1] or chromate,[2] the latter compound being considered the most suitable form for weighing thallium; thallium^{III} has been determined by precipitation as the oxide.[3] Thallium^{I} has been determined volumetrically by oxidation with bromate,[4] iodate,[5] ceric sulphate,[5,6] potassium permanganate[5] and hypobromite solution.[7] Some of these reagents, *e.g.*, iodide,[8] bromate[4] and ceric sulphate,[6] can be used in a potentiometric method. Another potentiometric method depended on the reduction of thallium^{III} with titanous chloride.[4]

In this investigation a new method was devised for the potentiometric determination of thallium^{I}; it depends on the oxidation of the thallium^{I} to the tervalent state in alkaline solution by means of potassium permanganate. Thallium^{III} is first reduced to the univalent state and then determined in the same way. The method can be used for the determination of micro quantities of thallium.

EXPERIMENTAL

The titration device used was that described previously.[9] A solution of the thallium salt was placed in the titration cell, made alkaline with sodium hydroxide and titrated with potassium permanganate. Sometimes, sodium chloride, ferric salts or telluric acid were added to the thallous salt. Thallium^{III} was determined by reducing it to thallium^{I} with an excess of sulphur dioxide in acid solution,[1] the unused sulphur dioxide being expelled by heating the solution and passing a stream of carbon dioxide through it; the solution was then made alkaline and titrated with potassium permanganate.

PREPARATION AND STANDARDISATION OF SOLUTIONS—

Potassium permanganate solution was prepared by the method of Stamm[10] and standardised by the acid oxalate[11] and alkaline formate[12] methods.

Laboratory-grade thallous sulphate and thallous acetate were dissolved in twice-distilled water. The thallium^{I} in the solutions was determined gravimetrically by precipitation as chromate.[2] This method is recommended by Moser and Brukl[2] as the most accurate for the determination of thallium.^{I} Five determinations were performed on 10-ml aliquots of these two solutions and the average weights of thallous chromate were 0·2846 g and 0·2039 g; the results of the determinations did not differ by more than 0·0001 g. From these results the solutions were calculated to be 0·1085 *N* and 0·0880 *N*.

Solutions of potassium permanganate and thallium^{I} of concentrations 0·0001203 *N* and 0·0000275 *N* were prepared by successive dilution with water twice distilled over alkaline potassium permanganate.

A solution of laboratory-grade thallic chloride was prepared, reduced as described above and titrated with 0·0496 *N* potassium iodate, chloroform being used in the detection of the liberated iodine. By this method the solution was found to be 0·0316 *N*, and the concentration determined by a similar reduction of the thallic chloride solution followed by precipitation of thallous chromate was also 0·0316 *N*.

ACCURACY OF THE METHOD—

Reduction of alkaline potassium permanganate proceeds in two stages; first, the permanganate ion is reduced to manganate, which is then reduced to manganese dioxide. The extent of the reduction depends on the alkalinity of the medium and the reducing agent. Potassium permanganate in weakly alkaline solution is reduced to manganese dioxide as follows—

$$3\ \mathrm{Tl}^{\cdot} + 2\ \mathrm{Mn}^{\cdot\cdot\cdot\cdot\cdot\cdot\cdot} \longrightarrow 3\ \mathrm{Tl}^{\cdot\cdot\cdot} + 2\ \mathrm{Mn}^{\cdot\cdot\cdot\cdot}$$

As a measure of the completeness of this reaction, K, the equilibrium constant, and α, the degree of completion, can be calculated by means of the redox potentials of the system. The following equations are used—

$$\log K = \frac{Z}{0{\cdot}059}\ (E_1 - E_2) \text{ and } \alpha = K^{1/(n_1+n_2)}$$

in which n_1 and n_2 represent the respective valency changes for both pairs of ions involved in the process, E_1 and E_2 are the redox potentials of the first and second systems and Z is the number of faradays necessary to complete the reaction. The values of the redox potentials of the $\mathrm{Tl}^{\cdot\cdot\cdot}$ - $\mathrm{Tl}^{\cdot}$ and $\mathrm{Mn}^{\cdot\cdot\cdot\cdot\cdot\cdot\cdot}$ - $\mathrm{Mn}^{\cdot\cdot\cdot\cdot}$ systems in alkaline media are[13] —0·05 volt and 0·588 volt, and $Z = 6$, $n_1 = 2$ and $n_2 = 3$; the values of K and α are calculated to be $1{\cdot}31 \times 10^{-65}$ and $1{\cdot}06 \times 10^{-13}$, which indicates that the reaction is quantitative.

RESULTS

From the results shown in Tables I, II and III it can be seen that there is good agreement between the potentiometric method with potassium permanganate in alkaline solution and the gravimetric chromate method.

When 0·1085 *N* thallium$^{\mathrm{I}}$ solutions are titrated with 0·1203 *N* potassium permanganate, the error is approximately 0·3 per cent.; this value is independent of the concentration of the alkali if it is between 0·25 and 2·5 *N* with respect to sodium hydroxide. When the reactants are diluted tenfold and 100-fold, the alkali concentrations being 0·1 *N*, the accuracy is the same as that with the more concentrated solution. At concentrations less than 0·001085 *N* the error is 0·69 per cent. if the alkalinity is 0·1 *N*, but is reduced to 0·52 per cent. when the alkalinity is 0·05 *N*. If 0·0088 *N* and 0·00088 *N* thallium^{1} solutions are titrated with potassium permanganate and the alkalinity of the solutions is approximately *N* and 2·5 *N*, then the errors are appreciable and the end-point is delayed.

It was found that when the more concentrated solutions were used (0·1085 *N* and 0·088 *N*), the equilibria were attained rapidly at the start of the titration. But after the addition of about one-half of the volume of potassium permanganate necessary for complete oxidation of the thallium$^{\mathrm{I}}$, the reaction became slower, about 8 minutes being required before equilibrium was attained. This time could only be shortened to about 6 to 7 minutes by the addition of catalysts, such as ferric salts or sodium chloride. When the solutions were heated to 40° C, the time required to attain equilibrium was 3 minutes. With more dilute solutions, the reaction was more rapid, only 5 minutes being required for equilibrium near the end-point.

TABLE I

TITRATION OF 10-ml ALIQUOTS OF THALLIUM$^{\mathrm{I}}$ SOLUTIONS WITH POTASSIUM PERMANGANATE

Concentration of sodium hydroxide, *N*	In presence of	Normality of thallium$^{\mathrm{I}}$ solution	Normality of potassium permanganate	Maximum inflection, mV	Thallium present, g per litre	Thallium found, g per litre	Error, %
0·25		0·1085	0·1203	130	11·0887	11·0521	—0·33
0·25	NaCl	0·1085	0·1203	74	11·0887	11·0658	—0·27
0·25	$\mathrm{Fe}^{\cdots}$	0·1085	0·1203	79	11·0887	11·0443	—0·4
2·5		0·1085	0·1203	122	11·0887	11·0521	—0·33
0·1		$1{\cdot}085 \times 10^{-2}$	$1{\cdot}203 \times 10^{-2}$	50	1·1089	1·1044	—0·4
0·1		$1{\cdot}085 \times 10^{-2}$	$1{\cdot}203 \times 10^{-2}$	53	1·1089	1·1126	0·33
0·1		$1{\cdot}085 \times 10^{-3}$	$1{\cdot}203 \times 10^{-3}$	25	0·11089	0·1111	0·18
0·1		$1{\cdot}085 \times 10^{-4}$	$1{\cdot}203 \times 10^{-4}$	12	0·011089	0·01133	0·4
0·05		$1{\cdot}1 \times 10^{-4}$	$3{\cdot}15 \times 10^{-4}$	16	0·011242	0·01130	0·52
0·1		$1{\cdot}1 \times 10^{-4}$	$3{\cdot}15 \times 10^{-4}$	18	0·011242	0·011319	0·69
0·1		$2{\cdot}75 \times 10^{-5}$	$3{\cdot}15 \times 10^{-4}$	9	0·005621	0·00566	0·69
0·05		$2{\cdot}75 \times 10^{-5}$	$3{\cdot}15 \times 10^{-4}$	9	0·005621	0·00564	0·35

The slowness of the reaction near the end-point was believed to be due to the primary reduction of permanganate to manganic oxide and manganese dioxide. If so, the effect of the permanganate would be to oxidise thallium$^{\mathrm{I}}$ to thallium$^{\mathrm{III}}$ and solid manganic oxide to manganese dioxide. However, according to Tomiček,[14] telluric acid prevents the precipitation of manganese dioxide, and it was thought probable that in its presence the reduction of the potassium permanganate might stop at the manganite state. Contrary to expectation the reaction was slower when telluric acid was present, 5 minutes being required for equilibrium after each addition of titrant, although the reaction velocity can be greatly increased by adding 1 g of sodium chloride per 10 ml of the solution to be titrated. Near the end-point the attainment of equilibrium still required 4 to 5 minutes with a platinum indicator electrode and about 6 minutes with a chromium indicator electrode.

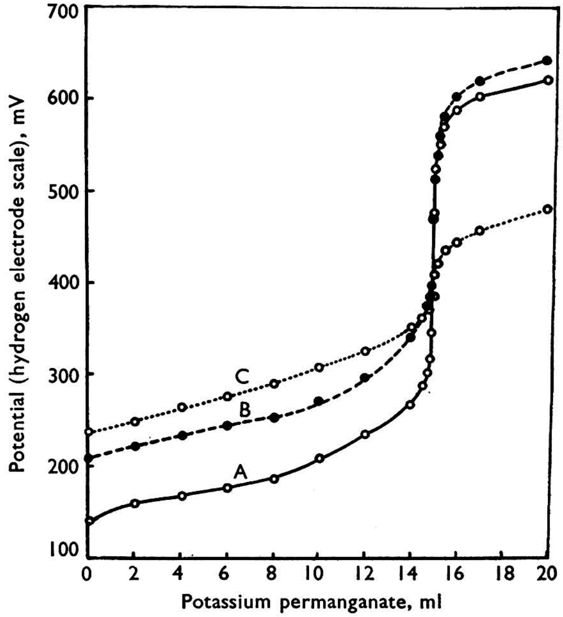

Fig. 1. Potentiometric titration of thallium^{I} with potassium permanganate in alkaline solution. Curve A, 10 ml of 0·1085 *N* thallium^{I} titrated with 0·1203 *N* potassium permanganate in 0·25 *N* sodium hydroxide; curve B, 10 ml of 0·1203 *N* thallium^{I} titrated with 0·1203 *N* potassium permanganate in presence of sodium chloride as catalyst; curve C, 10 ml of 0·001085 *N* thallium^{I} titrated with 0·001203 *N* potassium permanganate in 0·1 *N* sodium hydroxide

TABLE II

TITRATION OF 10-ml ALIQUOTS OF THALLIUM^{I} SOLUTION WITH POTASSIUM PERMANGANATE IN PRESENCE OF TELLURIC ACID IN 0·1 *N* SODIUM HYDROXIDE

Normality of thallium^{I} solution	Normality of potassium permanganate	Maximum inflection, mV	Thallium present, g per litre	Thallium found, g per litre	Error, %	Other conditions
$8·8 \times 10^{-2}$	$1·261 \times 10^{-1}$	136	8·9932	9·0238	0·34	Pt electrode
$8·8 \times 10^{-2}$	$1·261 \times 10^{-1}$	135	8·9932	9·0319	0·43	Pt electrode (NaCl added)
$8·8 \times 10^{-2}$	$1·261 \times 10^{-1}$	126	8·9932	9·0238	0·34	Cr electrode (NaCl added)
$8·8 \times 10^{-3}$	$1·261 \times 10^{-2}$	74	0·8993	0·9024	0·34	Pt electrode
$8·8 \times 10^{-3}$	$1·261 \times 10^{-2}$	104	0·8993	0·9016	0·26	Cr electrode
$8·8 \times 10^{-3}$	$1·163 \times 10^{-2}$	83	0·8993	0·9078	0·95	Pt electrode (*N* NaOH)
$8·8 \times 10^{-3}$	$1·163 \times 10^{-2}$	85	0·8993	0·9150	1·75	Pt electrode (2·5 *N* NaOH)
$8·8 \times 10^{-4}$	$1·261 \times 10^{-3}$	22	0·08993	0·09009	0·18	Pt electrode
$8·8 \times 10^{-4}$	$1·261 \times 10^{-3}$	36	0·08993	0·09033	0·44	Cr electrode
$8·8 \times 10^{-4}$	$1·163 \times 10^{-3}$	29	0·08993	0·09186	2·14	Pt electrode (*N* NaOH)
$8·8 \times 10^{-4}$	$1·163 \times 10^{-3}$	38	0·08993	0·09250	2·85	Pt electrode (2·5 *N* NaOH)
$8·8 \times 10^{-5}$	$1·261 \times 10^{-4}$	9	0·008993	0·009055	0·69	Pt electrode
$8·8 \times 10^{-5}$	$1·261 \times 10^{-4}$	11	0·008993	0·009063	0·78	Cr electrode

The titration curves for the oxidation of thallium^{I} in alkaline solution with potassium permanganate, telluric acid being absent, are characterised by distinct inflections at the end-point (see Fig. 1); these decrease in magnitude as the concentrations of the reactants are decreased. The inflection was 130 mV per 0·1 ml of 0·1203 *N* potassium permanganate when titrated with a 0·1085 *N* thallium^{I} solution and became 11 to 12 mV when the solutions were diluted 1000-fold. When a $2{\cdot}75 \times 10^{-5}$ *N* thallium^{I} solution was titrated with $1{\cdot}203 \times 10^{-5}$ *N* potassium permanganate, there was no distinct inflection and the end-point could not be determined accurately. However, with more concentrated solutions of potassium permanganate, *e.g.*, 0·000315 *N*, the inflection for the 0·00011 *N* thallium^{I} solution was increased to 16 to 18 mV and for a $2{\cdot}75 \times 10^{-5}$ *N* thallium^{I} solution was about 10 mV.

The inflections at the end-point were sharper in the presence of telluric acid, as shown in Fig. 2, and the results were accurate. As the telluric acid prevented the separation of manganese dioxide, at the start of the titration the solution was clear and bright pink. Half way to the end-point a white precipitate was formed and the solution turned brown after each addition of potassium permanganate, but it soon changed to red.

Solutions of thallium^{III} that had been reduced to thallium^{I} were made alkaline and titrated with potassium permanganate at 40° C either in the absence of telluric acid or with telluric acid present and sodium chloride as catalyst. A thallium^{III} solution was found to be 0·0316 *N* by titration with 0·0496 *N* potassium iodate and 0·0316 *N* by the gravimetric thallous chromate method. The value by the alkaline permanganate method was 0·0317 *N*, with an error of 0·3 per cent.

MICRO-DETERMINATION OF THALLIUM

The smallest quantity of thallium that had been determined by titration was 56 μg, with an error of 0·35 to 0·69 per cent., depending on the alkalinity. To determine the value

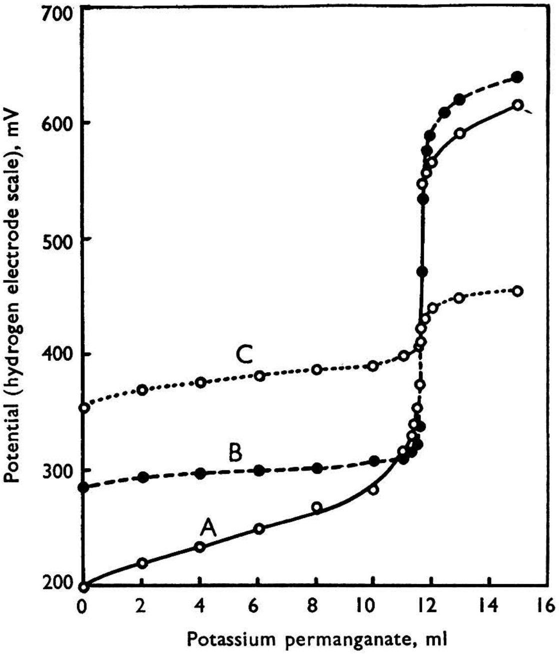

Fig. 2. Potentiometric titration of thalium^{I} with potassium permanganate in the presence of telluric acid in 0·1 *N* sodium hydroxide. Curve A, 10 ml of 0·088 *N* thallium^{I} titrated with 0·1261 *N* potassium permanganate in 0·1 *N* sodium hydroxide; curve B, 10 ml of 0·088 *N* thallium^{I} titrated with 0·1261 *N* potassium permanganate in presence of sodium chloride as catalyst; curve C, 10 ml of 0·00088 *N* thallium^{I} titrated with 0·001261 *N* potassium permanganate in 0·1 *N* sodium hydroxide

of the method for the titration of small volumes of dilute solutions, small aliquots of a $5{\cdot}5 \times 10^{-5}\,N$ thallium$^{\mathrm{I}}$ solution were titrated with $3{\cdot}15 \times 10^{-4}\,N$ potassium permanganate. The apparatus was similar to that used in the macro method; the titration vessel was a 25-ml beaker and the titrant was delivered from a 2-ml burette graduated to 0·01 ml and the solution was stirred mechanically.

The results are shown in Table III, and quantities of thallium as small as 5 µg could be determined. Although the inflection at the end-point is only about 6 mV per 0·02 ml of titrant, it is distinct.

TABLE III

TITRATION OF A $5{\cdot}5 \times 10^{-5}\,N$ THALLIUM$^{\mathrm{I}}$ SOLUTION WITH $3{\cdot}15 \times 10^{-4}\,N$ POTASSIUM PERMANGANATE

Volume of thallium$^{\mathrm{I}}$ solution, ml	Volume of potassium permangante used, ml	Volume of potassium permanganate required (theoretical), ml	Difference in volumes, ml	Thallium found, µg	Thallium present, µg	Difference	Maximum inflection per 0·02 ml, mV
5	1·445	1·455	0·010	55·8	56·2	0·6	5
2	0·588	0·582	0·006	22·7	22·5	0·2	6
1	0·295	0·291	0·004	11·4	11·2	0·2	7
0·5	0·149	0·146	0·003	5·7	5·6	0·1	6

REFERENCES

1. Treadwell, F. P., and Hall, W. T., "Analytical Chemistry," Ninth Edition, John Wiley & Sons Inc., New York, 1948, Volume II, p. 52.
2. Moser, L., and Brukl, A., *Monatsh. Chem.*, 1926, **47**, 709.
3. Mach, F., and Lepper, W., *Z. anal. Chem.*, 1926, **68**, 41.
4. Zintl, E., and Reinaker, G., *Z. anorg. Chem.*, 1926, **153**, 276.
5. Swift, E. H., and Garner, C. S., *J. Amer. Chem. Soc.*, 1936, **58**, 113.
6. Willard, H. H., and Young, P., *Ibid.*, 1930, **52**, 36.
7. Tomiček, O., and Jasek, M., *Ibid.*, 1935, **57**, 2409.
8. Kolthoff, I. M., and Furmann, N. H., "Potentiometric Titration," Second Edition, John Wiley & Sons Inc., New York, 1947, p. 198.
9. Issa, I. M., and Awad, S. A., *Analyst*, 1953, **78**, 487.
10. Stamm, H., *Z. angew. Chem.*, 1934, **47**, 791.
11. Fowler, R. M., and Bright, H. A., *J. Res. Nat. Bur. Stand.*, 1935, **15**, 493.
12. Issa, I. M., and Issa, R. M., *Anal. Chim. Acta*, 1954, **11**, 192.
13. Latimer, W. M., "Oxidation Potentials," Second Edition, Prentice Hall, New York, 1953, pp. 166, 238.
14. Tomiček, O., Procke, O., and Pavelka, V., *Coll. Czech. Chem. Commun.*, 1939, **11**, 449.

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The Determination of Fluorene in Tar Fractions

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A method is described for the determination of fluorene in coal-tar fractions. The fluorene is condensed with benzaldehyde in the presence of an alkaline catalyst, and the excess of benzaldehyde is determined volumetrically.

According to Franck,[1] the major components of high temperature tar are naphthalene, phenanthrene, fluoranthene, anthracene, chrysene, fluorene and acenaphthene in that order. As fluoranthene and chrysene are mainly left in the pitch residue on distillation of tar, it follows that fluorene is the fourth most abundant component in the distillate oils produced commercially from coke-oven and horizontal-retort tars and is concentrated in the fraction distilling at 298° C.

Owing to the increasing interest in the recovery and utilisation of pure hydrocarbons from coal tar, quantitative methods are necessary for the determination of these major components in tar oil fractions. Reliable methods exist for the determination of naphthalene,[2] phenanthrene[3] and anthracene,[4] but no suitable means have so far been developed for fluorene. Gluzman and Popova[5] suggested a method based on the insolubility of nitrofluorene in acetic acid, but as this method is confined to samples containing more than 66 per cent. of fluorene and the conversion is stated to be between 88·5 and 91 per cent., it is of limited application and doubtful value.

Fluorene differs from the components of coal tar that boil in the same range in that it possesses a reactive methylene group, which enables it to condense with esters, aldehydes, nitriles, and so on, and it was considered that a possible method of determination could be based on this property. Benzaldehyde, which is known to condense with fluorene in the presence of an alkaline catalyst,[6,7] was selected as the most suitable reagent.

Experimental

The fluorene used in the experiments to be described was derived from a sample of crude commercial fluorene by fractionation at a pressure of 50 mm of mercury in a 50-plate column to yield a fraction boiling between 190° and 194° C. This fraction was crystallised twice from ethanol to give a product melting at 114° to 115° C. This product and fluorene synthesised by the reduction of fluorenone were quantitatively compared in *cyclo*hexane solution at 3000 A. It was found that the purity of the product in terms of the synthetic fluorene was over 98 per cent.

Although benzylidenefluorene was formed when the fluorene was heated under reflux with an excess of benzaldehyde in absolute ethanol, sodium ethoxide being used as catalyst, it was found that a large proportion of the benzaldehyde underwent the Cannizzaro reaction and no attempt to determine the amount of benzaldehyde condensing with the fluorene by determining unchanged benzaldehyde could be made. Attempts to determine the benzylidenefluorene directly by bromination or by ultra-violet absorption were not encouraging.

The use of other basic catalysts was investigated and of those tested it was found that an aqueous solution of benzyltrimethylammonium hydroxide solution (Triton B) gave a consistently high yield of benzylidenefluorene with no apparent side reactions. Therefore, 5 g of fluorene and 5 ml of benzaldehyde were dissolved in 50 ml of absolute ethanol, 0·5 ml of Triton B was added, and the mixture was boiled under a reflux condenser for an hour. When the excess of benzaldehyde was determined by titration with alkali after the addition of hydroxylamine hydrochloride, there was a conversion of 84 per cent. to benzylidenefluorene.

Time of reaction—The effect of time of reaction on the conversion was studied by examining samples withdrawn at half-hourly intervals from the refluxing mixture. The results were as follows—

Time of reflux, hours	½	1	1½	2
Fluorene reacted, g	0·616	0·633	0·704	0·689

From these values it was concluded that the maximum conversion was reached in 1½ hours, and this reflux time was made standard in the subsequent experiments.

Extent of the Cannizzaro reaction—Two identical mixtures of reagents were prepared as previously described without the addition of fluorene. The benzaldehyde in one mixture was determined immediately by titration with 0·2 *N* sodium hydroxide after the addition of hydroxylamine hydrochloride. The other mixture was heated under reflux for 1½ hours and its benzaldehyde content then determined in the same way. The titre of 0·2 *N* sodium hydroxide for the second mixture was 0·5 ml less than that for the first, and this shows that the Cannizzaro reaction does not occur to any marked extent with Triton B as catalyst.

This slight reaction, which occurs on heating the mixture under reflux, can be allowed for in the determination of fluorene by performing a blank experiment.

Purity of the benzaldehyde—With some specimens of benzaldehyde, the conversions were found to be low. This was traced to the presence of benzoic acid, which neutralised the Triton B and so reduced its catalytic action.

Conversion factor—To determine the conversion factor, four determinations on pure fluorene under the conditions of the proposed method were each made by four operators, the final results in each determination being an average of two titrations. The results are shown in Table I; they have an average of 86·0 per cent. with a standard deviation of 0·40 per cent.

TABLE I

CONVERSION FACTOR FOUND WITH PURE FLUORENE

Operator	Conversion factor, %
A	85·8, 85·8, 85·8, 86·6
B	86·2, 86·2, 86·1, 86·1
C	85·4, 85·7, 85·2, 86·5
D	85·7, 86·2, 86·0, 86·6

Effect of other compounds—Naphthalene, methylnaphthalenes, acenaphthene, diphenyl, diphenylene oxide, anthracene, phenanthrene, phenols and bases are the compounds commonly found in fluorene fractions from coal tar. All these compounds were tested under the above conditions, with and without the addition of fluorene, to ascertain their effect. It was found that phenols suppressed the reaction, probably by combination with the catalyst, that primary bases reacted with the benzaldehyde and that, of the secondary amines, indole reacted but not carbazole. None of the other compounds, including tertiary amines, had any effect.

TABLE II

EFFECT OF INCREASING FLUORENE CONTENT

Fluorene added to 10 g of sample, g	Fluorene found, g	Difference (g in sample)
nil	0·311	0·311
0·2610	0·575	0·315
0·5010	0·973	0·475
0·6510	1·060	0·410
1·050	1·320	0·270
1·800	2·070	0·270
2·450	2·720	0·270

As there were no samples of methylfluorenes available, their effect was not determined. From the nature of the reaction it is expected that the method would determine methylfluorenes as fluorene.

Lower limit of determination—To discover whether the method had a lower limit of determination, a fraction of coke-oven tar boiling in the range 250° to 310° C and having a low fluorene content was washed free from tar acids and bases, and samples of increasing fluorene content were prepared from it by the addition of pure fluorene. These samples were then examined for their fluorene content and the results are shown in Table II.

It can be seen that it is possible to apply the factor of 86 per cent., but that at contents below 10 per cent. the results are of doubtful value.

METHOD

REAGENTS—

Benzaldehyde, freshly prepared—Wash pure benzaldehyde with small portions of 10 per cent. sodium carbonate solution (until no further carbon dioxide is evolved) and then with water. Steam distil the washed benzaldehyde, separate it from the water and dry it by adding anhydrous magnesium or sodium sulphate.

Hydroxylamine hydrochloride solution—Dissolve 34·7 g of the analytical-reagent grade material in about 600 ml of 60 per cent. aqueous methanol. Add 10 drops of bromophenol blue indicator and then add 0·2 *N* sodium hydroxide until the solution just becomes blue (see Note 1). Dilute to 1 litre with 60 per cent. aqueous methanol.

Triton B—The 40 per cent. solution of benzyltrimethylammonium hydroxide supplied by British Drug Houses Ltd.

Bromophenol blue—A 0·1 per cent. solution in water.

Absolute ethanol.

Industrial methylated spirit.

Hydrochloric acid, approximately 0·1 N.

Sodium hydroxide, 0·2 N.

PROCEDURE—

Sufficient sample is taken to contain from 3 to 5 g of fluorene (see Note 2) and weighed accurately. Transfer it to a dry 250-ml round-bottomed flask fitted with a ground-glass joint and add 50 ml of absolute ethanol. Add 50 ml of ethanol to a similar flask and to both flasks add 5 ml of benzaldehyde by means of a pipette, swirl gently and add by pipette 0·5 ml of Triton B.

Fit ground-glass condensers to each flask and gently heat them under reflux for 1½ hours. Allow the flasks to cool for 15 minutes and then run 5 ml of industrial methylated spirit down each condenser. Carefully transfer the contents of each flask to two 100-ml calibrated flasks, washing out the reaction flasks three times with industrial methylated spirit. Allow the calibrated flasks to cool thoroughly and dilute to the mark with industrial methylated spirit.

Using a pipette, transfer 10-ml aliquots from the calibrated flasks to 250-ml conical flasks. Add 75 ml of industrial methylated spirit to each flask and 0·5 ml of bromophenol blue solution (see Note 1). Titrate with the approximately 0·1 *N* hydrochloric acid dropwise until the yellow colour of the indicator is observed. Allow the flasks to stand for two minutes and add more acid if necessary. Add to each flask 25 ml of hydroxylamine hydrochloride solution, mix the contents well and allow the mixture to stand for 10 minutes.

Titrate the blank with 0·2 *N* sodium hydroxide until a green colour is just produced. Titrate the sample to the same colour. Allow the flasks to stand for a further 10 minutes and, if the colours have changed, add 0·2 *N* sodium hydroxide until the original green colour is produced. Note the volume of 0·2 *N* sodium hydroxide added to each flask.

Let T = difference in titre between the two flasks, ml,
f = factor of the 0·2 *N* sodium hydroxide, and
w = weight of sample taken, g.

$$\text{Then percentage of fluorene} = \frac{T \times f \times 166 \times 100}{w \times 5 \times 86}.$$

NOTES—

1. In place of an indicator, antimony - calomel electrodes may be used to indicate the end-point. The use of electrodes is essential if the sample is sufficiently coloured to interfere with the matching of the colour of the indicator in the sample and blank.

2. The sample must be free from tar acids and bases. Indole, which must also be absent, can be removed by fractionating the sample in a column of efficiency of more than 8 theoretical plates and taking a suitable fraction for examination.

RESULTS

The following is a selection of results determined on material containing fluorene.

Material	Fluorene, %
Fluorene obtained from Leicester vertical-retort neutral tar oil by fractionation in a 50-plate column at 50 mm of mercury and collection of the material boiling between 184° and 213° C	3·0, 3·8
Fluorene oil obtained from Evesham horizontal-retort neutral tar oil by fractionation in a 50-plate column at 20 mm of mercury and collection of the material boiling between 156° and 180° C	32·2, 32·2
Fluorene oil obtained from Colvilles coke-oven neutral tar oil by fractionation as above..	40·9, 40·7
Fluorene oil obtained from Normanby Park coke-oven neutral tar oil by fractionation in a 10-plate column at 12 mm of mercury and collection of the material boiling between 141° and 167° C	24·5
Commercial crude fluorene obtained from the North Thames Gas Board	40·9, 40·7
Commercial fluorene obtained from the North Thames Gas Board..	68·0, 68·2
Fluorene obtained by chromatographic separation of the above sample on a column of alumina	91·5, 91·8

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REFERENCES

1. Franck, H. G., *Brennstoff-Chemie*, 1953, **34**, 37.
2. Coggeshall, N. D., and Glessner, A. S., *Anal. Chem.*, 1949, **21**, 550.
3. Khmelevskii, V. I., and Postovskii, I. Ya., *J. Appl. Chem., U.S.S.R.*, 1944, **17**, 463.
4. "Standard Methods for Testing Tar and its Products," Third Edition, Standardisation of Tar Products Tests Committee, Gomersal, Leeds, 1950, pp. 301 to 305.
5. Gluzman, L. D., and Popova, G. M., *Khim. Referat. Zhur.*, 1941, **4**, No. 9, 87; *Chem. Abstr.*, 1944, **38**, 2808.
6. Thiele, J., *Ber.*, 1900, **33**, 851.
7. Thiele, J., and Henle, F., *Ann.*, 1906, **347**, 290.

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Notes

THE ACID INVERSION OF CANE SUGAR

IN several published analytical methods based on the well-known Clerget procedure for determining sucrose by measuring the optical rotation before and after inversion, the inversion is carried out by adding a measured volume of 6·34 *N* hydrochloric acid. This specification of the normality to two decimal places implies that it (and, therefore, also the volume taken) must be adjusted with an error of not more than one part in six hundred. Reference to the original literature shows that this strength of acid was first used by Jackson and Gillis[1] during their researches into the inversion of sucrose under varying conditions of acidity and other factors, apparently because it happened to be an exact 1 to 1 dilution of some (unusually) concentrated acid they had available. This normality of 6·34 has been copied from place to place ever since and has acquired an almost mystic significance.

Variations in the strength of the acid used for inversion, all other conditions such as time and temperature remaining unaltered, may affect the optical rotation in two ways. First, there is a small unavoidable destruction of invert sugar by the acid during the inversion; this is, of course, allowed for in the divisor of the Clerget formula. Secondly, there is a direct effect on the observed rotation in the same way that the presence of neutral salts in an invert sugar solution affects its rotation. The first effect is quite small under normal conditions of time and temperature; for example, approximately 1 per cent. of invert sugar is destroyed under the conditions specified in the S.P.A. method[2] for determining sucrose in sweetened condensed milk. An alteration in the strength of the acid used by 10 per cent. would make a completely negligible difference in this amount of destruction. The direct effect is somewhat larger; Jackson and Gillis showed that a 10 per cent. variation in the strength of hydrochloric acid present altered the rotation of the normal weight of invert sugar in 100 ml by 0·125 International Sugar Degrees.

These facts indicate that, except in the most precise research work, the strength of the acid used, or the volume taken, can be varied by (say) 5 per cent. without appreciable effect on the results. This has been confirmed by experiment. A solution was made up to contain approximately 8 per cent. of sucrose and 2·5 per cent. of lactose, these concentrations being near to those that would be present in a solution prepared by applying the S.P.A. method to a normal sweetened condensed milk. Inversion was carried out exactly as specified except that various volumes of 5 *N* hydrochloric acid were added to 40 ml of the filtrate. The volume of 5 *N* acid theoretically equivalent to 6·00 ml of 6·34 *N* acid is 7·61 ml. The volumes used in the experiments were 6·6, 7·0, 7·6, 8·0 and 8·6 ml. The inverted solution was made up to 50 ml as usual, and the optical rotation was read at 20° C in a Ventzke polarimeter that could be read to one-tenth of a sugar degree. The rotation of all five solutions was $-1{\cdot}3°$.

A sample of condensed milk was then examined by the S.P.A. method and two separate 40-ml portions of the filtrate were treated respectively with 7·0 and 8·0 ml of 5 *N* acid. In both experiments the optical rotation after inversion was $-1{\cdot}0°$.

From this it is clear that when inversions of sucrose are being carried out for ordinary routine analytical purposes, the strength of the acid used need not be adjusted precisely, a tolerance of ± 2 per cent. at least being quite permissible, and the volume taken can be measured with a measuring cylinder with ample accuracy. There is no point in making 6·34 *N* acid; the 5 *N* acid, which many laboratories use for other purposes (*e.g.*, lead determinations), can be used in equivalent volume.

REFERENCES

1. Jackson, R. F., and Gillis, C. L., *Sci. Pap. B.S.*, 1920, **16**, 141. An excellent summary of the effect of various factors on the inversion of sucrose is given in U.S. National Bureau of Standards Circular No. C440, "Polarimetry, Saccharimetry and the Sugars," Washington, 1942.
2. Report of the Milk Products Sub-Committee, Report No. 2, *Analyst*, 1930, **55**, 111.

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THE DETERMINATION OF SUCROSE IN SWEETENED CONDENSED MILK: A SIMPLIFICATION

IF, in the method[1] described in 1930 by the Milk Products Sub-Committee of this Society's Analytical Methods Committee for determining sucrose in sweetened condensed milk, the sample is always diluted to 200 ml before filtration and polarimetric rotations are always recorded in "sugar degrees" through a 2-dm tube with white light, the formula to be used becomes—

$$S = \frac{D - \frac{5}{4}I}{5{\cdot}098W} \times (200 - v) \qquad \text{(1)}$$

In this formula, *D* and *I* are the "direct" and "inverted" readings, *W* is the weight taken, and *v* is a correction for the volume occupied by the precipitate produced during clarification and is given by—

$$v = \frac{W}{100}\,(1{\cdot}08F + 1{\cdot}55P) \qquad \text{(2)}$$

The S.P.A. method directs that approximately 40 g of the sample shall be taken and accurately weighed. By taking the slightly smaller weights shown in Table I, it is possible to arrange that $5{\cdot}098\,W$ exactly equals $(200 - v)$. This simplifies the calculation considerably, as the percentage of sucrose is now given by—

$$S = D - \tfrac{5}{4}I \qquad \text{(3)}$$

Weighing the exact quantity specified in the Table takes very little extra time, particularly if an aperiodic balance is in use, and this extra time is more than saved on the calculation, with less risk of arithmetical error.

A further simplification can be achieved with little error by assuming the protein content to be equal to the fat content. Although this is only approximately true, small variations in P affect $(200 - v)$ but little, since *v* is so much smaller than 200. For instance, if $F = 10{\cdot}0$ and $P = 9{\cdot}0$, then $v = \frac{W}{100} \times 24{\cdot}75$; if $F = 10{\cdot}0$ and $P = 10{\cdot}0$, $v = \frac{W}{100} \times 26{\cdot}30$. If *W* is taken as

37·3 g, $200 - v = 190{\cdot}77$ in the first example and 190·19 in the second. A difference (absolute) of 1 per cent. in P thus alters $(200 - v)$ relatively by 0·3 per cent. If, then, the fat content is determined (which will almost always be necessary in any event) and the protein is assumed to be equal to the fat content, its analytical determination is avoided and the possible error in the result will be too small to matter for many purposes. The weight of milk to be taken is then the amount shown in bold type in Table I. Weights corresponding to intermediate fat contents can be interpolated if necessary.

TABLE I

WEIGHT OF FULL-CREAM MILK TO BE TAKEN

Fat, %	Protein, %				
	8·5	9·0	9·5	10·0	10·5
8·5	**37·58** g	37·53 g	37·48 g	37·43 g	37·38 g
9·0	37·54 g	**37·49** g	37·44 g	37·38 g	37·33 g
9·5	37·50 g	37·45 g	**37·40** g	37·34 g	37·28 g
10·0	37·47 g	37·41 g	37·36 g	**37·30** g	37·25 g
10·5	37·43 g	37·37 g	32·73 g	37·27 g	**37·22** g

For a modern sweetened condensed *separated* milk, the fat content will be very close to 0·5 per cent., whilst the protein content will usually be between 9·5 per cent. and 11·5 per cent. The weight to be taken, for the same simplification to hold good, is given by Table II. If 38·00 g were always taken, the error would be negligible in routine work.

TABLE II

WEIGHT OF SEPARATED MILK TO BE TAKEN

Protein, per cent.	9·5	10·0	10·5	11·0	11·5
Weight, g	38·08	38·03	37·98	37·93	37·87

If it becomes necessary to make a temperature correction to the result, owing to the polarisation after inversion being taken at some temperature, T, not far removed from 20° C, the result calculated by the simple formula (1) above should be multiplied by—

$$(1 + 0{\cdot}0037\,(T - 20)).$$

REFERENCE

1. Report of the Milk Products Sub-Committee, Report No. 2, *Analyst*, 1930, **55**, 111.

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A METHOD FOR THE SEPARATION AND IDENTIFICATION OF THE COMMON GROUP 2 ELEMENTS

THE methods most frequently used for the separation of the elements in group 2 involve the use of an alkaline solvent, such as ammonium sulphide, sodium hydroxide or lithium hydroxide,[1] to extract the sulphides of arsenic, antimony and tin from the sulphides of the copper sub-group.

The most commonly used reagent is ammonium sulphide and the identification of the group 2B elements is complicated by the presence of excess of sulphur. If copper is present, a coloured precipitate resembling antimony sulphide forms when the ammonium sulphide extract is acidified,[1] and this can be misleading. When lithium hydroxide is used, the separation is better and the group 2B precipitate is free from the large excess of sulphur. However, identification of these elements can be difficult if they are present in disproportionate amounts, whatever reagent is used for their isolation. Similarly in group 2A the cyanide - hydrogen sulphide test for cadmium does not give convincing results when it is applied to a precipitate containing a large excess of copper sulphide.

The disadvantages of these methods are appreciated by the teacher, for this group has to be introduced at a time when the student has had very little experience. Resorting to non-hydrogen sulphide schemes,[2] or the extensive use of organic precipitants, offers no advantage, as such schemes have much less educational value.

The method described below is straightforward in application, the separations are distinct and the confirmatory tests quite definite, even when small quantities of material are handled. For most elements well-known confirmatory tests have been used.

SEPARATION INTO GROUP 2A AND 2B

The sulphides are precipitated, separated and washed in the usual way. The precipitate is boiled with concentrated hydrochloric acid, about one-third of the volume of the original solution being a suitable quantity of acid, although this may be reduced if the precipitate is small or completely soluble. When the strongly acid solution containing any suspended sulphides is cool, it is saturated with hydrogen sulphide. The solution may contain cadmium, bismuth, antimony, stannous and stannic tin and small quantities of lead. The residue may contain copper, arsenic and mercuric sulphides, and should be washed once with cold concentrated hydrochloric acid.

The separation of copper from cadmium, and arsenic from antimony and tin, simplifies the subsequent analysis.

GROUP 2A: COPPER, ARSENIC AND MERCURY

The sulphides are dissolved in the minimum amount of aqua regia, and any small particles of sulphur are removed. The solution is then diluted and made alkaline with ammonium hydroxide. When this solution is treated with strong magnesium chloride solution, the arsenic is precipitated as crystalline magnesium ammonium arsenate. The solution and precipitate are separated.

CONFIRMATORY TESTS—

Arsenic—The white precipitate is dissolved in a small quantity of dilute nitric acid and the solution is boiled with ammonium molybdate reagent. A bright yellow precipitate should form.

Copper—A portion of the solution is treated with an excess of acetic acid and potassium ferrocyanide solution. If cupric ions are present, a red precipitate is formed.

Mercury—Another portion of the solution is acidified with dilute hydrochloric acid and treated with a few drops of either stannous chloride solution or potassium iodide solution. In the presence of mercuric ions the stannous chloride produces a white or grey precipitate, and the potassium iodide a scarlet precipitate soluble in excess of reagent.

GROUP 2B: CADMIUM, BISMUTH, ANTIMONY, TIN AND LEAD

The acid solution is evaporated to small volume, one or two drops of dilute sulphuric acid are added, the solution is allowed to stand for a few minutes and it is then diluted slightly. Any white precipitate present (lead sulphate) is removed. A very small portion of the solution is tested for stannous tin by either of the tests described below. If tin is found to be present, it is oxidised by dropwise addition of bromine water until the colour of the bromine just persists, and the excess of bromine is removed by boiling the solution (see Note 1). The clear solution is then treated with ammonium hydroxide and saturated with hydrogen sulphide.

Bismuth and cadmium sulphides are precipitated and removed; antimony and tin remain in solution as thio salts. The precipitate containing bismuth and cadmium sulphides is dissolved in the minimum quantity of hydrochloric acid, the solution is boiled to remove hydrogen sulphide and an excess of ammonium hydroxide is added. Bismuth hydroxide is precipitated; cadmium remains in solution.

CONFIRMATORY TESTS—

Lead—The white precipitate is dissolved in ammonium acetate solution and the solution is treated with potassium chromate solution, when a yellow precipitate should form.

Bismuth—The white precipitate is dissolved in the minimum quantity of dilute hydrochloric acid and the solution is diluted considerably, when a white precipitate should form owing to hydrolysis. Alternatively, with sodium stannite, bismuth salts are reduced to black metallic bismuth.

Cadmium—The ammoniacal solution is treated with hydrogen sulphide, when the presence of cadmium is indicated by formation of a yellow precipitate.

Tin and antimony—The solution of thio salts is evaporated almost to dryness, and the precipitated sulphides are dissolved in the minimum quantity of hot dilute hydrochloric acid. The solution is diluted slightly and treated with a small piece of magnesium. The gases evolved are allowed to pass through a spot of 0·5 N iodine solution on a filter-paper, which turns bright orange in the presence of stibine owing to the formation of the coloured iodide (see Notes 2 and 3). After the magnesium has dissolved, the solution is tested for stannous tin with mercuric chloride solution, which produces a white silky precipitate, or with a speck of solid cacotheline, when a violet colour is produced.

Alternative tests for antimony—1. If a bright platinum wire with a piece of zinc wool wound around the bottom is dipped into an acid solution containing antimony, a brown-black stain is formed on the platinum.

2. An acid solution containing antimony produces a purple precipitate when it is treated with a few drops of rhodamine B reagent and a few crystals of solid sodium nitrite.

NOTES

1. The tests described permit stannous and stannic tin to be distinguished, but do not allow stannic tin to be detected in the presence of stannous tin.

As stannous sulphide is insoluble in colourless ammonium sulphide solution, it seemed possible to separate stannic tin, but difficulties were encountered.

An acid solution of stannous chloride was treated with ammonium hydroxide, and the solution containing the suspended white precipitate was saturated with hydrogen sulphide. This solution was found to contain stannic tin, presumably formed by atmospheric oxidation.

Addition of ammonium hydroxide to an acid solution containing bismuth and stannous tin produces a black precipitate containing metallic bismuth. Appreciable quantities of stannic tin are found in the solution.

2. Iodine solution was found to be most suitable for testing for stibine. Traces of arsenic sulphide present as a sol in the hydrochloric acid solution produce sufficient arsine to blacken silver nitrate solution, which cannot therefore be used. Small quantities of arsine do not interfere with the antimony test described, although large quantities bleach the iodine completely.

3. For example, 1 ml of a solution containing 5×10^{-6} g-ions of each of the elements cadmium, arsenic, antimony and tin was analysed on a semimicro scale. It was necessary to precipitate the sulphides in the presence of ammonium chloride and to allow the solution to stand, so that all the cadmium was precipitated; 0·2 *N* iodine solution was used in the antimony test. The tests described gave positive results.

REFERENCES

1. Holness, H., and Trewick, R. F. G., *Analyst*, 1950, **75**, 276.
2. See, for example, Cornog, J., *J. Chem. Educ.*, 1938, **15**, 420, and El-Badry, H., McDonnell, F. R. M., and Wilson, C. L., *Anal. Chim. Acta*, 1950, **4**, 440.

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June 15th, 1954

USE OF 8-HYDROXYQUINOLINE FOR THE DETERMINATION OF ZINC IN SOLUTIONS CONTAINING COPPER

THE usual method for the determination of zinc in solutions containing copper entails the separation of the copper as cuprous thiocyanate. This method is tedious and time consuming. Evans[1] avoided the effects of copper by converting it into a soluble cyanide complex and then precipitating the zinc as sulphide. Ducret[2] observed that copper does not interfere in the determination of zinc with salicyldioxime. Rây *et al*[3,4] recommended the conversion of copper to its complex with thiourea, after which the zinc could be determined with quinaldinic acid or its sodium salt. This method is satisfactory, but the reagent is costly and is not always available. We find that it can be replaced by the cheaper reagent 8-hydroxyquinoline and the volumetric determination of copper can form part of the procedure.

METHOD

REAGENTS—

8-*Hydroxyquinoline in ethanol*, 2 *per cent. solution.*

Acetic acid, dilute—Dilute 5 to 8 ml of glacial acetic acid to 150 ml with water.

PROCEDURE—

Treat the solution containing copper and zinc with an excess of potassium iodide and determine the copper by titrating the iodine with standard sodium thiosulphate solution. Then add 4 to 6 g of thiourea until the precipitate of cuprous iodide dissolves to give a colourless solution. Acidify the solution with dilute acetic acid and add about 5 g of sodium acetate. Heat the solution to about 60° C and slowly add the alcoholic solution of 8-hydroxyquinoline until it is present in slight excess. The solution should be carefully stirred during the addition. Continue heating for 2 to 3 minutes, when the precipitate of the zinc - 8-hydroxyquinoline complex becomes granular. Separate the precipitate by filtration on a tared sintered-glass crucible, wash it with warm water, dry it at 130° to 140° C and weigh it. Alternatively, dissolve the precipitate in 2 N hydrochloric acid, with slight warming if necessary, and dilute to a known volume. Titrate aliquots of this solution with a standard potassium bromate solution, methyl red being used as indicator, or add a known excess of potassium bromate solution to aliquots and determine the unused bromate.

RESULTS

Results for the determination of zinc in the presence of copper by precipitation as the complex with 8-hydroxyquinoline and the volumetric bromate method were as follows—

Copper taken, g	0·0395	0·0790	0·0395	0·0790	0·0395	0·0790	0·0395	0·0790
Zinc taken, g	0·0654	0·0654	0·0981	0·0981	0·1308	0·1308	0·1635	0·1635
Zinc found by pyrophosphate method, g	—	—	—	—	0·1316	0·1318	0·1646	0·1635
Zinc found by oxinate method, g	0·0653	0·0661	0·0984	0·0985	0·1316	0·1310	0·1638	0·1630

In the pyrophosphate method the copper was precipitated and separated as cuprous thiocyanate. The method also gives good results if the zinc - 8-hydroxyquinoline complex is determined gravimetrically.

REFERENCES

1. Evans, B. S., *Analyst*, 1946, **71**, 460.
2. Ducret, L., *Bull. Soc. Chim. France*, 1946, **13**, 392.
3. Rây, P., and Dutt, N. K., *Z. anal. Chem.*, 1939, **15**, 265.
4. Rây, P., and Sarkar, T. C., *Mikrochemie*, 1939, **27**, 64.

DEPARTMENT OF CHEMISTRY
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EAST PAKISTAN

S. Z. HAIDER
M. H. KHUNDKAR

June 6*th*, 1954

THE ELIMINATION OF THE BLANK VALUE IN THE UNTERZAUCHER METHOD FOR THE MICRO-DETERMINATION OF OXYGEN

ALTHOUGH there should not be a blank value in Unterzaucher's method[1,2] for the micro-determination of oxygen, persistent blank values have been reported by many analysts.[3]

Until recently the same difficulty has been experienced in this laboratory with an apparatus similar to that described by Chambers,[4] who reported blank values equivalent to 0·04 to 0·05 ml of 0·01 N sodium thiosulphate. In attempts to eliminate this blank value all rubber connections were replaced by ground-glass joints and experiments were performed with silica combustion tubes from different sources, various preparations of carbon-black and iodine pentoxide being used, but in tests with 180 ml of nitrogen used at a rate of 9 ml per minute, these high blanks could not be reduced. The nitrogen used in these and in all subsequent experiments was commercial "oxygen-free" nitrogen (containing approximately 10 p.p.m. of oxygen), further purified by passage over reduced copper oxide at 500° C.

On the assumption that this persistent blank value was due to the incomplete removal of air adsorbed on the carbon-black and on the inner surface of the combustion tube, a modified method of conditioning the apparatus was recently tested. A silica combustion tube (Thermal Syndicate tubing), packed in the usual manner with 4 cm of silica chips, 14 cm of pelleted carbon (from Arthur H. Thomas Company, Philadelphia) and 1 cm of silica wool was gently tapped until a clear channel was formed above the carbon pellets. The exit end of the tube was then closed by a ground-glass cap and the section containing the carbon was heated for about two hours at 1120° C, the tube being continuously evacuated by a Hyvac pump. The unpacked portion of the tube was heated in the same manner for about 1 hour and finally the carbon was again heated for 2 hours. After the admission of nitrogen and careful re-adjustment of the carbon filling to its normal position, the combustion tube was connected to the rest of the apparatus and a stream of nitrogen was passed through the complete assembly for 24 hours. During this period the furnace temperature was maintained at 1120° C and the anhydro-iodic acid,[2] HI_3O_8, was heated to 118° C. When 180 ml of nitrogen at a rate of 9 ml per minute were passed through the apparatus after this treatment, there was no measurable blank observed in the 30 to 40 tests performed during the working life of the combustion tube. In more than 40 tests on a second tube conditioned in the same manner, there was no blank.

Although the technique described in this Note has not yet been applied to more than two combustion tubes, the procedure may be of value to other analysts.

REFERENCES

1. Unterzaucher, J., *Ber.*, 1940, **73**, 391.
2. ——, *Analyst*, 1952, **77**, 584.
3. Report of Discussion at the Meeting of the American Chemical Society, Chicago, September, 1950, *Anal. Chem.*, 1951, **23**, 530.
4. Chambers, W. T., *Rubb. Chem. Technol.*, 1950, **23**, 727.

IMPERIAL CHEMICAL INDUSTRIES RESEARCH DEPARTMENT
ALKALI DIVISION
NORTHWICH
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A. F. COLSON
June 9th, 1954

THE REMOVAL OF DISSOLVED CARBON DIOXIDE IN THE VOLUMETRIC DETERMINATION OF BORON

MACRO amounts of boron are usually determined volumetrically as boric acid by potentiometric titration with sodium hydroxide solution betwen pH 5·7 and 8·6, or by visual titration, using suitable indicators, after the boric acid has been complexed with mannitol. Dissolved carbon dioxide interferes with the titration and must be removed by boiling the acid solution. This operation is tedious and must be performed carefully to prevent loss of boric acid by volatilisation in the steam.

It may not be generally known that carbon dioxide can be effectively removed from such solutions if the pH value is adjusted to approximately 3 and a vigorous stream of compressed air (2·5 to 3 litres per minute) is bubbled through the solution for 10 to 15 minutes before the titration. This operation is most conveniently carried out in a covered 600-ml squat beaker; no loss of boric acid has been observed. The compressed air should be filtered, but no special provisions to remove carbon dioxide from it are necessary. (A lower pH value requires excessive amounts of sodium hydroxide solution to bring the solution to pH 5·7, whilst at a higher pH value there is incomplete removal of carbon dioxide.)

This is shown by the results in Table I for distilled water and for solutions of 1 g of sodium carbonate acidified with hydrochloric acid, both solutions being free from boric acid. The pH value of each solution was adjusted to the value shown in the Table, carbon dioxide was removed by bubbling and the pH was then re-adjusted to 5·7. Then 15 g of mannitol were added (as would be required in a titration of boric acid) and the determinations were completed by titration to pH 8·6. The values recorded in the last column of Table I are those required to shift the pH from 5·7 to 8·6.

TABLE I

TYPICAL RESULTS

Sample	0·1 *N* sodium hydroxide, ml
400 ml of distilled water	2·35
400 ml of boiled distilled water	0·12
400 ml of distilled water adjusted to pH 3 and bubbled for 10 minutes	0·11, 0·08
400 ml of de-ionised water	0·06, 0·08
1 g of Na_2CO_3 in 400 ml, acidified, adjusted to pH 5 and bubbled for 10 minutes	>20
1 g of Na_2CO_3 in 400 ml, acidified, adjusted to pH 3 and bubbled for 5 minutes	1·97
1 g of Na_2CO_3 in 400 ml, acidified, adjusted to pH 3 and bubbled for 10 minutes	0·17, 0·14, 0·17
1 g of Na_2CO_3 in 400 ml, acidified, adjusted to pH 3 and bubbled for 15 minutes	0·16
1 g of Na_2CO_3 in 400 ml, acidified, adjusted to pH 2 and bubbled for 10 minutes	0·15

RESEARCH LABORATORIES
THE BRITISH ALUMINIUM CO. LTD.
CHALFONT PARK
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H. JACKSON
R. E. BAILEY
June 8th, 1954

A MODIFIED METHOD FOR THE DECOMPOSITION OF CHROMITE

DURING an investigation on the composition of Indian chromites, various methods for the decomposition of the mineral were tried. Methods based on acid treatment of chromite were found to be unsuitable for its complete analysis. The mineral can be decomposed completely by fusion with sodium peroxide[1] and sintering with sodium peroxide,[2] and these are considered the best methods for attacking chrome ores.[3]

Stevens,[4] when studying the composition of chromites from the western hemisphere, fused the ore with a mixture of sodium carbonate and potassium nitrate.[5]

This method was also used by the author in the initial stages of the work mentioned above, as it afforded means, at the beginning of the analysis, for separating chromium, vanadium, aluminium and silica (if any) from iron, calcium, magnesium, manganese, titanium, and so on. But it was observed that this method was laborious and as many as four fusions, each followed by extraction of the fused mass, were sometimes necessary before a 0·5000-g sample was completely attacked. There was also an appreciable attack on the platinum crucible, 5 to 10 mg of platinum being dissolved in the fusion of one sample. Sodium carbonate,[6] although it does not attack the platinum crucible, acts but slowly on the mineral.

In order to overcome the difficulties in the methods mentioned above, fusion of the chromite with sodium carbonate in an atmosphere of oxygen was tried. The results have been satisfactory and a $\frac{1}{2}$-g sample can be completely decomposed in about 2 hours by a single fusion. The amount of platinum dissolved from the crucible is so small that its separation before precipitation of iron is not essential. The procedure is suitable for the complete analysis of chromite, as there is no contamination from the crucible. A clean separation of chromium, vanadium, aluminium and silica from iron, calcium, magnesium, manganese and titanium is possible at the beginning of the analysis and this is an additional advantage over methods based on digestion with acid or fusion with bisulphate.

Fusion with sodium carbonate in an atmosphere of oxygen has also been tried for extracting vanadium from vanadiferous iron ores, instead of fusion with sodium carbonate - potassium nitrate mixture.[7] This also yields satisfactory results, obviating any attack on the platinum crucible. It is suggested that fusion with sodium carbonate in an atmosphere of oxygen could be used with advantage in place of fusion with sodium carbonate - potassium nitrate mixture for decomposing many other minerals and ores requiring an oxidising flux.

METHOD

Grind the chrome ore to 200-mesh B.S.S. to facilitate separation of the chromite by flotation with a dense liquid. Mix 0·5000 g of the chromite with 4 to 5 g of anhydrous sodium carbonate, place the mixture in a platinum crucible and cover it with 1 g of anhydrous sodium carbonate. Heat the crucible with a Meker burner until the melt is tranquil. Pass a stream of oxygen through a bent platinum tube that impinges on the surface of the melt. Swirl the crucible occasionally to facilitate mixing of the mineral grains with the alkali. Completion of the reaction is indicated by the absence of gas bubbles in the melt. Then cool the crucible, extract the melt with water and filter the slurry. Chromium, aluminium, vanadium and silica are in the filtrate. The residue, containing iron, calcium, magnesium, manganese, titanium, and so on, is completely soluble in dilute hydrochloric acid. If hydrogen sulphide is passed through this solution, no platinum sulphide is precipitated.

A platinum Rose crucible could be used instead of the bent platinum tube.

This work is published by permission of the Director, Geological Survey of India.

REFERENCES

1. Sanjiv, U., and Rao, M. R. A., *Curr. Sci.*, 1947, **16**, 88.
2. Rafter, T. A., *Analyst*, 1950, **75**, 485.
3. Bryant, F. J., and Hardwick, P. J., *Ibid.*, 1950, **75**, 12.
4. Stevens, R. E., *Amer. Min.*, 1944, **29**, 1.
5. Hillebrand, W. F., Lundell, G. E. F., Bright, H. A., and Hoffman, J. I., "Applied Inorganic Analysis," John Wiley & Sons, Inc., New York, 1953, p. 525.
6. Treadwell, F. P., and Hall, W. T., "Analytical Chemistry," Volume II, John Wiley & Sons, Inc., New York, 1948, p. 424.
7. Schoeller, W. R., and Powell, A. R., "The Analysis of Minerals and Ores of the Rarer Elements," Charles Griffin & Co., Ltd., London, 1940, p. 141.

CHEMICAL LABORATORY
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June 8th, 1954

A POSSIBLE SOURCE OF ERROR IN THE DETERMINATION OF TRACE METALS, PARTICULARLY LEAD

IN this laboratory it has been the usual custom to measure out certain reagents by means of graduated pipettes, in particular ammonium citrate solution used in the determination of lead in foodstuffs with dithizone. It has recently been found that the enamel used for filling the graduation marks on one make of graduated pipette now contains lead, and it is extracted by ammonium citrate solution in sufficient quantities to contaminate the "lead-free" solution with lead. If this had not been immediately noticed, it could have led to some peculiar results.

The enamel is stated to be "resistant to practically all chemical action other than that which attacks the glass itself," but this statement is untrue. As a precaution, the enamel has been removed by appropriate solvents from all graduated pipettes used. Incidentally, the makers of the pipettes have admitted that the enamel contains lead, but stated that this was not so until recently. It must be remembered that the graduations and inscriptions on bulb pipettes, burettes, calibrated flasks, and so on, do not usually come into contact with the solutions for which they are used. With graduated pipettes, however, the lower graduation marks are always immersed in the liquid being measured while the pipette is being filled, and pipettes may even be left in the bottle of reagent for some time.

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June 8th, 1954

Apparatus

A MODIFIED HAND-OPERATED HIGH-PRESSURE HYDROGEN SULPHIDE GENERATOR

Two hydrogen sulphide generators have been described.[1,2] The following description is of an apparatus that is simpler to construct and easier to dismantle, clean and re-assemble than the earlier versions.

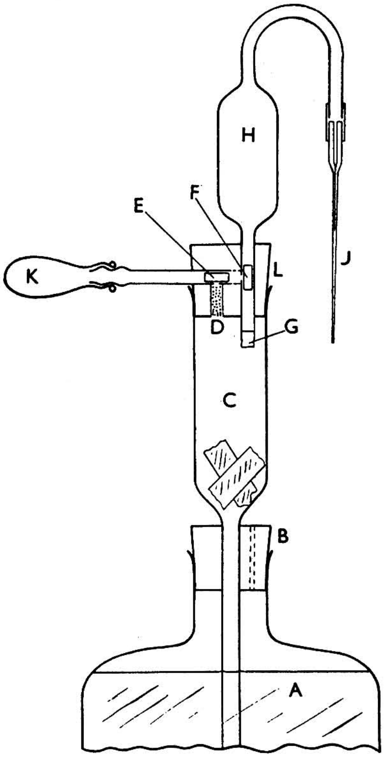

Fig. 1. Hydrogen sulphide generator

The apparatus is shown in Fig. 1. Diluted hydrochloric acid (1 + 1) is contained in a 1-litre bottle, A. The funnel, C, is held by a grooved cork, B, the open end of C being closed by a specially bored rubber bung, L, which supports the gas reservoir, H, and the pumping unit. The gas reservoir is constructed from a 25-ml pipette and has an internal valve, F. It is closed at one end by a piece of boring from a rubber bung, G, and the capillary jet, J, is attached to the other end. The pumping device is a straight piece of open-ended glass tubing attached to a strong rubber teat, K, and containing an internal valve, E, similar to valve F; the open end leads directly to valve F. Valve E is connected to the funnel, C, through a hole, D, which is packed with cotton-wool to retain spray.

The valves E and F are made by covering a pin-hole in the wall of the tube with a rectangular piece of fine rubber membrane, which is rolled up and pushed into position with a glass rod. The rubber should extend almost completely round the interior of the tubing so that its elasticity holds it firmly in position.

In operation, compression of K forces gas through F, and relaxation of K causes F to close and E to open, so that the gas from C is drawn into K and acid is sucked into the funnel. By compressing K three or four times per minute, a pressure is produced inside H, and a continuous flow of gas issues from J.

REFERENCES

1. Stock, J. T., and Heath, P., *Analyst*, 1951, **76**, 496.
2. Stock, J. T., and Fill, M. A., *Mikrochim. Acta*, 1953, Part 1–2, 89.

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June 15th, 1954

Ministry of Food

LIST OF CURRENT STATUTORY INSTRUMENTS AND STATUTORY RULES AND ORDERS RELATING TO FOOD

This List of Current Statutory Instruments and Statutory Rules and Orders, Sectional List No. 33, *has been revised to July* 31*st*, 1954, *and can be obtained from H.M. Stationery Office at cost of postage. See Analyst*, 1954, **79**, 111.

British Standards Institution

NEW SPECIFICATIONS*

B.S. 2533:1954. Chlorobenzene. Price 2s.
B.S. 2535:1954. Dibutyl Sebacate. Price 2s. 6d.
B.S. 2536:1954. Di-2-ethylhexyl Sebacate. Price 2s.

AMENDMENT SLIP*

A PRINTED slip bearing amendments to a British Standard has been issued by the Institution, as follows—

PD1970—Amendment No. 3 (September, 1954) to B.S. 684:1950. Methods of Analysis of Oils and Fats.

Book Reviews

BENTLEY'S TEXT-BOOK OF PHARMACEUTICS. Sixth Edition by HAROLD DAVIS, B.Sc., Ph.D., F.P.S., F.R.I.C., with the collaboration of M. W. PARTRIDGE, B.Pharm., B.Sc., Ph.D., F.P.S., and C. L. SARGENT, F.P.S. Pp. xiv + 1078. London: Baillière, Tindall & Cox Ltd. 1954. Price 42s.

A. O. Bentley's "Pharmaceutics" was first published in 1926, and through successive editions it has become ever more firmly established as an essential textbook for students of pharmacy. This sixth revision brings the work into line with the British Pharmacopoeia, 1953, and also conforms with the changes recently made in the syllabus of the qualifying examination for registration as a pharmacist.

The book starts with a history of the British Pharmacopoeia and the International Pharmacopoeia, and then goes on, in Part II, to a discussion of Physicochemical Principles, presenting that part of physical chemistry required by students qualifying in pharmacy. Dr. M. W. Partridge is responsible for these ten chapters, which include expositions on hydrogen-ion concentration, colloidal systems, emulsions, enzymes, osmotic pressure, chromatography and other themes; the last-named two chapters appealed to the reviewer as being very clear outline accounts of difficult subjects. Mr. C. L. Sargent took over the revision of Part III, Pharmaceutical Processes and Plant, and gives an account of the general processes used in manufacturing pharmacy. Full and useful illustrated descriptions are accorded to this important aspect of pharmaceutics. One notices, in the chapter on distillation, a section on the commercial preparation of mineral-free water by use of ion-exchange resins.

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* Obtainable from the British Standards Institution, Sales Department, 2, Park Street, London, W.1.

Part IV is devoted to Dispensing and contains a wealth of knowledge on prescriptions, mixtures, emulsions, pill (and tablet) coatings, plasters and many other types of dispensed commodities. There is also a chapter on incompatibles, and all this information is likely at any time to be invaluable to analysts who may be confronted with the task of reporting upon a dispensed preparation. Eight chapters, extending in all to almost 200 pages, on Pharmaceutical Microbiology constitute Part V and form eloquent testimony to the modern transformation in the science of therapeutics. Beginning with an introductory chapter on the biology of bacteria and moulds, the subject is developed in all its ramifications and embraces illuminating accounts of antibiotics (including manufacturing details with flow sheets), sterilisation of medicaments, tests for sterility, the dispensing of sterile medicaments, vaccines and sera and preservation of pharmaceutical products. Every modern advancement seems to be included and, by way of a single illustration, one notices under the description of the Rideal - Walker test that mention is made of the fact that amendments to B.S. No. 541 were published in 1943, 1951 *and* 1953.

The subject of Pharmaceutics includes the processing, characterisation and testing of surgical dressings, sutures and ligatures, and much detailed information on all these topics is to be found in Part VI. The subject matter of Part VII is devoted to the official Pharmaceutical Preparations and contains authoritative information about injections, emulsions, ointments and many other preparations of the British Pharmacopoeia, 1953, including blood products and plasma substitutes. One wonders why the excellent illustrated account of tablet manufacture was not included in Part III, where it seems more appropriately to belong. The last chapter in the book, written by Mr. Sargent, is devoted to a discussion of medical gases and the equipment for their administration. There follows an appendix containing dose tables of all the official drugs and preparations. The magnitude of the work may be judged from the index, which occupies over 13 pages of small type.

Although it is only claimed that this is a students' textbook, Dr. Davis and his collaborators have, in fact, produced a most valuable documented work of reference presenting a solid background of knowledge essential to all analysts concerned with the examination of medicinal substances.

N. L. ALLPORT

THORPE'S DICTIONARY OF APPLIED CHEMISTRY. Fourth Edition. Volume XI. SOIL, ORGANIC CONSTITUENTS OF—ZYMURGY. Edited by M. A. WHITELEY, O.B.E., D.Sc., F.R.I.C. Pp. x + 1145. London and New York: Longmans, Green & Co. Ltd. 1954. Price 140s.

The first volume of this monumental work appeared in June, 1937, and contained 703 pages. This present volume is the eleventh and last of the text itself, but it will be followed shortly by a final Index Volume, which will provide a consolidated index to the whole of the Fourth Edition.

The whole work, therefore, covers a seventeen-year span of chemical knowledge at a period of its most rapid development, and it is inevitable that the first volumes must start to become out of date long before the last are issued. The only solution to this difficulty appears to be a policy similar to that of the painting of the Forth Bridge, namely, to begin revision again at the beginning. It is to be hoped that something of this nature will be possible.

All the features of previous volumes commended by former reviewers are to be found in the present work. The standards of thoroughness, authority, accuracy and, above all, readability are all well maintained, and "Thorpe" is still one of the few books on chemistry that one opens to find a particular piece of information—and reads on and on. The publishers, too, have done their work well; the efficient binding of an 1145-page book so as to withstand the inevitable heavy handling that a dictionary must receive is in itself a major undertaking.

Some of the more important monographs included in this volume are those on stereochemistry, sulphur dyestuffs (44 pages), sulphuric acid (46 pages), terpenes (55 pages), valency, vitamins (13 pages), waxes, and zinc (61 pages). For these, as elsewhere in the work, there are adequate references to the literature. There is also a very useful chapter on Synthetic Drugs, which, incidentally, one might reasonably expect to find under the heading of "Drugs," especially as there is no other entry under "Synthetic." However, this rather unexpected arrangement has permitted the editors to include in this latest volume an up-to-date article on a subject about which much less was known when Volume IV, in which "Drugs" would otherwise have occurred, was written in 1940.

Incidentally, the sulphonamides do not occur in their alphabetical position, although they are dealt with fully under Synthetic Drugs. Presumably anomalies of this kind, of which there are a few, are bound to arise in such a work. The final Index Volume should, however, remove any difficulty that might arise in locating just what is wanted.

As usual chemical analysis is well represented. There are long self-contained articles on sugar analysis and water analysis, and each of the more important entries, such as sulphur, tannin, tea and whisky, has its own analytical sub-section.

All who have been concerned with this work may justifiably be proud of it. To possess it is to command a wealth of authoritative and concise information on every aspect of applied chemistry

JULIUS GRANT

CHROMATOGRAPHY. A REVIEW OF PRINCIPLES AND APPLICATIONS. By E. LEDERER and M. LEDERER. Pp. xviii + 460. Amsterdam and New York: Elsevier Publishing Co. Ltd.; London: Cleaver-Hume Press Ltd. 1953. Price 60s.

The publication of this substantial volume focuses attention once again on an important and rapidly growing branch of chemistry of particular importance to the analyst. Several books dealing with specific aspects of chromatography have already appeared during the year, but the present work is different in attempting to provide a comprehensive treatment of all aspects of the subject. The very wide scope of the book is indicated by the heading of the five divisions into which the work is divided: (1) Adsorption Chromatography, (2) Ion-exchange Chromatography, (3) Partition Chromatography, (4) Chromatography of Organic Substances, (5) Chromatography of Inorganic Substances. Each division includes several chapters. For example, division (4) contains 21 chapters, many of them being necessarily short. Details such as R_F values, solvents and tests used for the separation of groups of various substances are summarised in 123 tables. The general arrangement, however, is clear and orderly, which makes it a simple matter to find out what has been done in a particular field. Another point that stresses the comprehensive nature of the work is an excellent list of references, 1879 of them, with an additional list of special references for each chapter in an addendum. These references include papers up to the end of 1952, and even some in 1953.

It must be emphasised that, as stated in the title, the book is a review, so that the reader who is concerned with a particular practical problem will often find himself faced with a number of alternative methods of approach without clear guidance as to which he is likely to find the most useful. He will also often find it necessary to refer to the original papers for practical details. However, the field covered is so large that to have included a more detailed discussion of the various applications would have necessitated an ambitious work of several volumes.

Chemists wishing to make use of the chromatographic technique will find this book an invaluable source of reference.

F. H. BURSTALL

THE INFRA-RED SPECTRA OF COMPLEX MOLECULES. By L. J. BELLAMY, B.Sc., Ph.D. Pp. xviii + 323. London: Methuen & Co. Ltd.; New York: John Wiley & Sons Inc. 1954. Price 35s.

Infra-red spectroscopy is being used to an ever-increasing extent for elucidating the structure of complex organic compounds. From a study of the position and intensity of the absorption bands in the infra-red spectrum of an organic compound, it is frequently possible to identify the functional chemical bonds and atomic groupings in the compound. In order to do this, the analyst must have a knowledge of the empirical correlations that have been made between absorption-band frequencies and organic functional groups; he must also be able to appreciate the factors that affect the reliability of these correlations. This information, which has been largely acquired during the last decade, is widely dispersed through the technical literature and so far has not been readily accessible. The available correlation charts are usually of limited application and give no critical evaluation of the assignments.

In this timely book, Dr. Bellamy lists absorption-frequency correlations for most organic and certain inorganic groups and gives a critical account of the original observations on which the assignments are based; he indicates the classes of compounds that have been studied and discusses the factors influencing the intensity and frequency of their absorption bands. Each chapter is devoted to a single type of chemical bond or atomic grouping and is introduced by a summary of the appropriate correlations; it contains a table summarising the absorption-frequency ranges, the correlations being then discussed in detail and the chapter being completed with a most useful bibliography that covers the relevant literature up to the end of 1952, with a few 1953 references. The infra-red spectra of thirty selected compounds are reproduced in the book and are used as examples to demonstrate the various correlations.

Dr. Bellamy has wisely restricted his book to the empirical interpretation of infra-red spectra and has not attempted to cover practical aspects of spectroscopy, such as quantitative analysis and instrumentation. Nevertheless, it is to be regretted that he has not emphasised the importance of sample preparation and the desirability of recording, whenever possible, the spectra of solutions as well as those of solid samples. The spectra of dilute solutions of hydroxyl- and carbonyl-containing compounds in non-polar solvents are less complicated by inter-molecular hydrogen-bonding and are to that extent easier to interpret. Moreover, polymorphic forms of a compound give identical spectra in solution, but different ones in the solid state. These points are sometimes overlooked by chemists attempting to interpret infra-red spectra.

The book contains several proof-reading errors. In correlation chart No. 1 (page 5), "$>CH\equiv CR$" should read "$CH\equiv CR$" and "$C\equiv O$," "$C\equiv C$." Again, on page 117, line 10, "1762" should be "1702." However, these errors are small and do not detract from the use of the book. There is no doubt that an organic chemist will find it of immense value for assessing the contribution that infra-red spectroscopy can make to his particular problem, but every infra-red spectroscopist who has to interpret the spectra of complex organic molecules will find this book essential for his day-to-day activities and will wish to thank Dr. Bellamy for the care that he has taken in its preparation.

J. E. Page

Practical Methods in Biochemistry. By F. C. Koch and M. E. Hanke. Sixth Edition. Pp. x + 537. London: Baillière, Tindall & Cox, Ltd. 1953. Price 38s. 6d.

No reviewer could fail to be impressed by the great amount of labour and care that have gone into the compilation of this volume, but a medical student using it as a guide to his practical studies in biochemistry might feel rather differently about it. Only a professional teacher perhaps is competent to pass judgment on its educational merits, but to a layman in these matters it would seem that its wide range and extremely detailed treatment would make it difficult for the student to perceive the basic principles underlying the methods described and to assess the relative importance of the various precautions that have to be taken, for example, in an analysis or a biochemical preparation. True, as the authors point out in the introduction, it is even more necessary in biochemistry than in analytical or organic chemistry that the details of a method should be rigidly adhered to if accurate and reproducible results are to be attained, and in a general handbook of biochemical methods the principle of giving the fullest details can be accepted as axiomatic; but surely, in a manual intended for medical students, it is at least arguable that some simplification should be made even to the extent of sacrificing accuracy, because the purpose of such a book is to teach the principles of analysis and to explain the limitations and significance of standard methods, and not to turn medical students into skilled laboratory technicians. If, however, it is accepted that the medical student benefits by having to perform, with successful results, 319 experiments ranging from the simple colour tests for carbohydrates through a miscellaneous variety of biochemical preparations, and experiments on hydrogen-ion activity, on the properties of enzymes and on gas analysis, to microbiological assays and chemical tests for hormones, then this is indeed an admirable guide. For the training of a graduate wishing to specialise in his fourth year in biochemistry and particularly in clinical biochemistry, nothing better could be imagined, and the book should prove equally useful to the general analyst who has to undertake occasional biochemical tests and has no specialised knowledge of this field.

The authors have obviously been at great pains to include all that they considered essential to achieve satisfactory results and have evidently introduced modifications of their own to some of the published methods when they considered these to be inadequate. Interspersed with the experimental details are many useful practical hints, for example, on the cleaning of mercury for manometric work, on the maintenance of glass electrodes and on the design of a novel glass electrode for micro-analysis. The preparation of the many standard solutions and reagents required is described in an appendix.

Not even the most difficult of biochemical techniques have deterred the authors, and the chapter on "Manometric methods with the Van Slyke - Neill Apparatus" is a most valuable account of the uses to which this versatile apparatus can be put. Microbiological assays represent perhaps the most difficult field in which to give students practical instruction, but from a rapid reading of the chapter dealing with this subject it would seem that all the essential details for success have been covered. One might, however, query the wisdom of including in a students' textbook the estimation of the cobalamins by means of *Lactobacillus leichmannii*. The authors themselves state that "the microbiological estimation of cobalamins is generally considered to be the most difficult of microbiological analyses, with frequent inconsistencies, failure of checking among replicates, and occasional unexplained lack of growth." If anything, this is an under-statement, and the use of some other organism for the assay of vitamin B_{12} will eliminate what otherwise will often be a frustating and not very profitable experience.

Many new experiments have been added to this sixth edition of a book first published in 1934. They include exercises in paper chromatography and the use of ion-exchange resins, the estimation of succinic dehydrogenase by means of a simple form of Warburg apparatus and the estimation of saponification numbers with diethylene glycol as solvent, as well as a new chapter in which have been gathered all the biochemical preparations previously scattered throughout the book, together with some new ones.

The book is well printed and contains few misprints, and a great deal of space has been saved by printing explanatory notes in smaller type. It has a good index, which will facilitate its use as a book of reference, and the price is reasonable considering its size and the large amount of information it contains.

F. A. ROBINSON

Publications Received

FERTILIZER EXPERIMENTS AT THE VAALHARTZ AGRICULTURAL RESEARCH STATION (1946–50). Science Bulletin No. 338, Chemistry Series No. 193. By D. G. WESSELS, M.Sc., and T. P. PRETORIUS, M.Sc. Pp. 76. Pretoria, South Africa: Department of Agriculture. 1954. Price 1s.

AGRONOMY EXPERIMENTS AT THE VAALHARTZ AGRICULTURAL RESEARCH STATION (1946–50). Science Bulletin No. 350, Agricultural Education and Research Series No. 11. By W. VAN DER MERWE, B.Sc., D. G. WESSELS, M.Sc., and T. P. PRETORIUS, M.Sc. Pp. 70. Pretoria, South Africa: Department of Agriculture. 1954. Price 1s.

THE VITAMINS: CHEMISTRY, PHYSIOLOGY, PATHOLOGY. Volume II. Edited by W. H. SEBRELL, JUN., and R. S. HARRIS. Pp. xiv + 766. New York: Academic Press Inc.; London: Academic Books Ltd. 1954. Price $16.50; 132s.

CHEMICAL THERMODYNAMICS. By I. PRIGOGINE and R. DEFAY. Translated by D. H. EVERETT. Pp. xxxii + 543. London and New York: Longmans, Green & Co. Ltd. 1954. Price 63s.

SEMI-MICRO QUANTITATIVE ORGANIC ANALYSIS. By R. BELCHER, B.Sc., Ph.D., F.R.I.C., F.Inst.F., and A. L. GODBERT, M.Sc., Ph.D. Second Edition. Pp. x + 222. London and New York: Longmans, Green & Co. Ltd. 1954. Price 21s.

THE ELEMENTS OF CHROMATOGRAPHY. By T. I. WILLIAMS, M.A., B.Sc., D.Phil., F.R.I.C. Pp. xii + 90. London and Glasgow: Blackie & Son Ltd. 1954. Price 9s. 6d.

QUALITATIVE SCHNELLANALYSE. By G. CHARLOT, D. BEZIER, R. GAUGUIN, J. M. ODEKERKEN and A. SCHLEICHER. Pp. vi + 82. Berlin: Walter de Gruyter & Co. 1954. Price DM7.80.

TECHNIQUES IN CLINICAL CHEMISTRY. A Handbook for Medical Laboratory Technicians. By F. N. BULLOCK, F.I.M.L.T., F.R.M.S., F.C.S. Pp. x + 171. Bristol: John Wright & Sons Ltd. 1954. Price 16s. 6d.

TECHNIQUE OF ORGANIC CHEMISTRY. Volume VI. MICRO AND SEMIMICRO METHODS. By N. C. CHERONIS. Pp. xxiv + 628. New York and London: Interscience Publishers Inc. 1954. Price $12.00; 96s.

NEW INSTRUMENTAL METHODS IN ELECTROCHEMISTRY. THEORY, INSTRUMENTATION, AND APPLICATIONS TO ANALYTICAL AND PHYSICAL CHEMISTRY. By P. DELAHAY. Pp. xviii + 437. New York and London: Interscience Publishers Inc. 1954. Price $11.50; 92s.

REPORT OF THE ANALYTICAL METHODS COMMITTEE

THE Report of the Lead Panel of the Metallic Impurities in Foodstuffs Sub-Committee, "The Determination of Lead in Foodstuffs," reprinted from *The Analyst*, July 1954, **79**, 397–402, is now available from the Secretary, The Society for Analytical Chemistry, 7–8, Idol Lane, London, E.C.3; price to members 1s. 6d., or to non-members 2s. 6d. Reports of the Analytical Methods Committee are only obtainable from the Secretary (not through Trade Agents) and remittances must accompany orders.

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ANALYTICAL ASSISTANT with experience in forensic work and analysis of drugs required for a Research Station near Cambridge. Salary £400-£500 p.a. Write Box No. 3880, THE ANALYST, 47, Gresham Street, London, E.C.2.

ASSISTANT required for permanent post in Central London Laboratory engaged on smoke filtration research. Qualifications: Inter. B.Sc.; experience organic analysis; initiative and accustomed to working without close supervision. Salary £400-£500 p.a., according to experience. Send full particulars to Box No. 3881, THE ANALYST, 47, Gresham Street, London, E.C.2.

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APPLICATIONS are invited for appointment as micro-analyst in the Department of Organic Chemistry at a salary up to £700 a year according to qualifications. Previous experience in micro-analysis would be an advantage, but applicants prepared to undergo training would be considered. Applications (three copies) stating date of birth, qualifications and experience, together with the names of three referees, should reach the Registrar, The University, Leeds, 2 (from whom further particulars may be obtained), not later than 22nd December, 1954.

ANALYSTS required in Welwyn Garden City for the control analysis of synthetic vitamins and drugs at all stages from raw material to pharmaceutical product. Previous experience of industrial analysis essential. Commencing salary according to qualifications, age and experience. Five-day week. Pension Fund. Apply in writing to Secretary, Roche Products, Limited, 15, Manchester Square, London, W.1.

JUNIOR ANALYST male/female REQUIRED FOR ESSENTIAL OILS/AROMATIC CHEMICALS, S.E. LONDON. Progressive and permanent post with good remuneration for applicant capable of working accurately and with minimum supervision. Write to Box No. 3882, THE ANALYST, 47, Gresham Street, London, E.C.2.

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EAST MALLING RESEARCH STATION. An Analytical Chemist is required in the Plant Protective Chemistry Section. Work will consist mainly of analysis of spray materials and residues. Candidates should hold a degree in chemistry or equivalent qualification; experience in analysis of compounded materials would be an asset. The post will be in the Experimental Officer class on Ministry of Agriculture salary scales. Further particulars and application form from the Secretary, East Malling Research Station, near Maidstone, Kent, by whom applications should be received not later than 31st December, 1954.

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CHEMIST required in the Department of Insecticides and Fungicides for general analytical work and assisting in developing analytical methods for determining insecticides in soil and in plant materials. Experience of analytical work in any field would be an advantage. The appointment will be in the Experimental Officer Class with starting salary for graduates between £390 and £690 per annum, according to age and experience. Scales for women are somewhat lower. Superannuation scheme.

Applications, quoting reference 105, with names of two referees, to be addressed to the Secretary.

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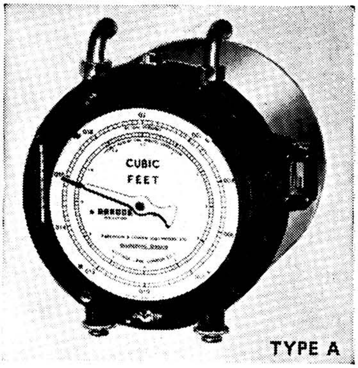
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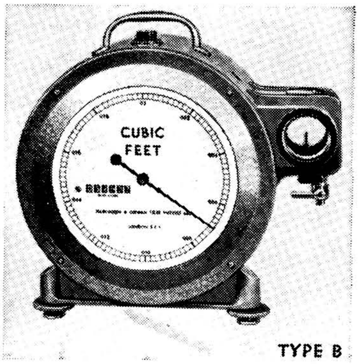
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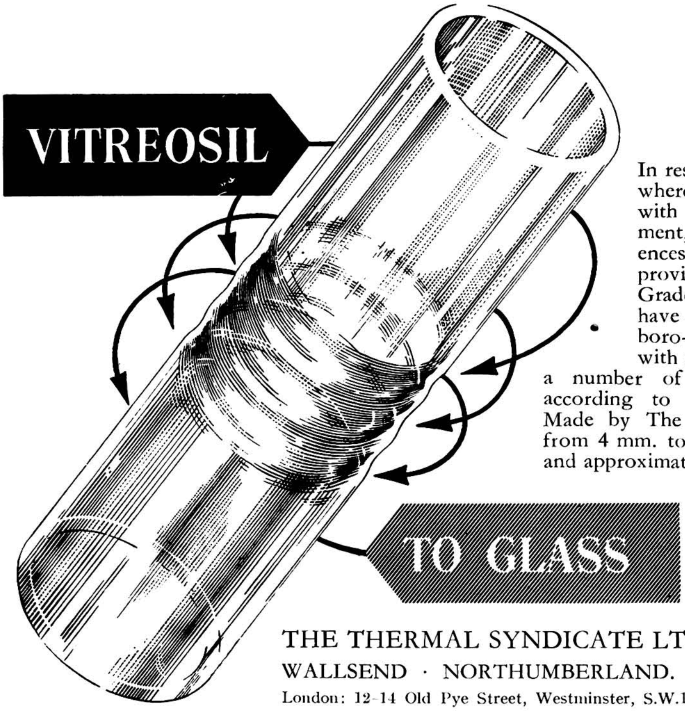

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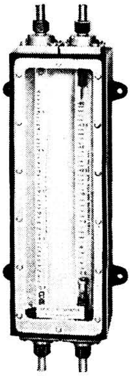

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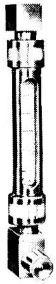

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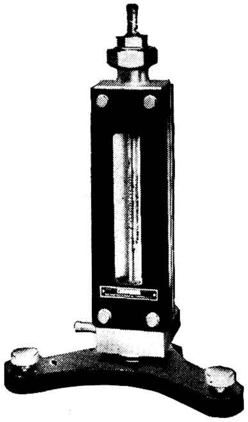

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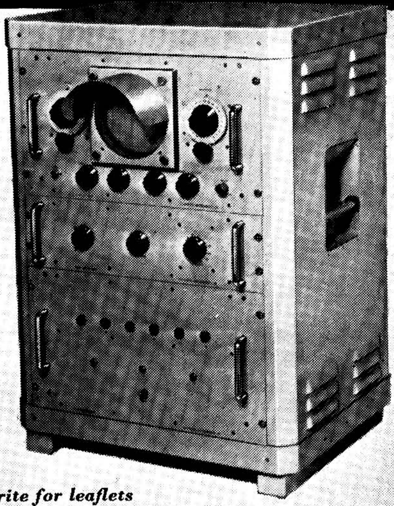

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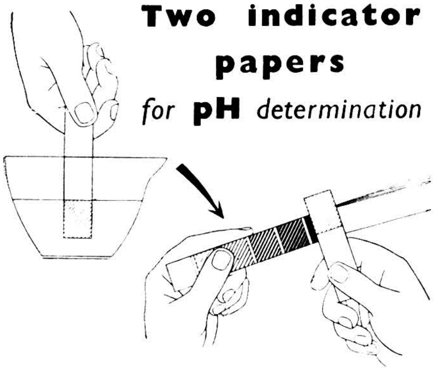

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1,2,3,4 Di-O-isopropylidene galacto-pyranose	15 - D
4,6, O-Ethylidene-α-D-glucopyranose	8 - G
L-Fucose	50 - G
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D-Galactose	15 - H
Gluconic delta lactone (+61·7)	10 - D
α-D-Glucosamine hydrochloride	17 - D
Glucosaminic acid	40 - G
β-Glucose	27 - D
α-D-Glucose penta-acetate	26 - H
β-D-Glucose penta-acetate	36 - H
D-Glucose-1-phosphate, dipotassium salt dihydrate	18 - G
Glucuronolactone	48 - D
DL-Glyceraldehyde	35 - G
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I-Inositol (inosite) inactive	45 - H
Inulin (bacteriological grade)	40 - H
2-Keto-D-gluconic acid, Ca salt	5 - G
Lactobiono-*delta*-lactone	23 - D
D-Laevulose (fructose)	15 - H
Lead Tetra acetate (85–95%)	25 - H
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D-Lyxose	50 - G
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D-Mannitol (mannite)	24 - K
D-Mannose (+14·25)	15 - D
Melezitose	35 - D
α-D-Melibiose	43 - D
β-D-Melibiose dihydrate (+129·3)	45 - D
Methyl-4,6-O-benzylidine-2,3-di-(O-tosyl)-D gluco-pyranoside	30 - D
α-Methyl-D-glucopyranoside	23 - H
α-Methyl-D-mannoside	12 - D
Naphthoresorcinol	12 - G
Ninhydrin (triketo-hydrindene-hydrate)	35 - D
o-Nitrophenyl-β-D-galactopyranoside	90 - D
D-Raffinose, pentahydrate	27 - H
L-Rhamnose (+8·5) (isodulcite)	9 - D
D-Ribonolactone (D-ribonic acid-γ-lactone)	45 - D
D-Ribose	60 - D
Sodium borohydride	35 - D
D-Sorbitol (cryst) (99%)	19 - K
L-Sorbose	12 - H
Sucrose octa-acetate	49 - K
β-1,2,3,4 Tetra-O-acetyl-6-O-triphenylmethyl glucopyranose	10 - G
α-α-D-Trehalose	11 - G
2,3,5-Triphenyl tetrazolium chloride	24 - D
Turanose	60 - G
Xanthosine (xanthine-9-ribofuranoside)	20 - G
D-Xylose	44 - H
L-Xylose	16 - G

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Volume 79. No. 945, Pages 725-792 December, 1954

THE ANALYST

THE JOURNAL OF THE SOCIETY FOR ANALYTICAL CHEMISTRY

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VOL. 79

1954

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Errata

The following errata are listed in the 1954 Index of *The Analyst*. They are repeated below in a manner such that they may be cut out and stuck over the incorrect matter.

Vol. 78, 1953: March, p. 181.

Replace the 4th line from the foot of the page by—

Serum sodium $= 48 \cdot 88\ (x - y)$ mg per 100 ml,

Vol. 79, 1954: February, p. 110.

Replace the lettering of the key at the top of Fig. 1 by—

Extracting Liquid

Extracted Liquid

Vol. 79, 1954: April, p. 221.

Replace the 2nd, 3rd and 4th lines of "*Ammonium hydroxide - ammonium chloride buffer solution* (*pH* 10)" in list of reagents by—

ammonium chloride in 570 ml of ammonium hydroxide, sp.gr. 0·880, and make up to 1 litre with water. Adjust the pH to 10 to 10·1 with either hydrochloric acid, sp.gr. 1·18, or ammonium hydroxide, sp.gr. 0·880, as required, using a suitable pH meter.

Vol. 79, 1954: May, p. 273.

Replace the 2nd line below Table I by—

solvents than either the bromide or iodide, and they substantiate Irving and Rossotti's[1]

Vol. 79, 1954: September, p. 532.

Replace the 7th line by—

timorous to talk without other written text to a friendly audience for **10** to **30** minutes—

Replace the 24th line by—

on the lamp side of the slide carrier and at the *top right-hand* corner as he looks towards the

Vol. 79, 1954: October, p. 617.

Replace the 9th line of text by—

Russell Eggitt and Ward[2]) and if a sensitive colour test is used, this may lead to a convenient

Replace the 16th line of the text by—

of the 2:2′-dipyridyl complex.

Vol. 79, 1954: October, p. 622.

Replace the 7th line by—

sufficient 2:2′-dipyridyl was present for 2400 μg, although only 20 to 70 μg of tocopherol

Vol. 79, 1954: December, p. 731.

Replace the 5th line of the synopsis by—

procedure are equivalent to 0·05 p.p.m. or less of lead. Although the blanks

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

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Méthodes et Réactions de l'Analyse Organique. Vol. III. Réactions Colorées et Fluorescences. Pesez and Poirier. (Publication received), 464.
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Organische Fällungsmittel in der quantitativen Analyse. Prodinger. 3rd Edn. (Publication received), 116.
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Qualitative Inorganic ——. New Physico-Chemical Approach. Charlot. Trans. by Murray. (Publication received), 528; (Review), 723.
Qualitative Schnellanalyse. Charlot, Bézier, Gauguin, Odekerken and Schleicher. (Publication received), 792.
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Spot Tests. Vol. I. Inorganic Applications. Feigl. Trans. by Oesper. 4th Edn. (Publication received), 312; (Review), 463; Vol. II. Organic Applications. (Publication received), 528; (Review), 722.
Standard Methods of Clinical Chemistry. Vol. I. Reiner. (Publication received), 60; (Review), 309.
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Journal of —— of the U.S.S.R. in English Translation. Vol. VII, No. 1, January–February, 1952. (Publication received), 60; (Review), 461.
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Volumetric Glassware, Scientific Aspects of Design and Accuracy. National Physical Laboratory. (Publication received), 660.
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Applied Chemistry: Reports on Progress of ——. Society of Chemical Industry. (Publication received), 660.
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Biochemical Analysis: Methods of ——. Glick. Vol. I. (Publication received), 392; (Review), 658.

Preparations. Snell. Vol. 3. (Publication received), 246; (Review), 659.

Biochemistry: General ——. Fruton and Simmonds. (Review), 112.

Practical Methods in ——. Koch and Hanke. 6th Edn. (Publication received), 464; (Review), 791.

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Bonds: Strengths of Chemical ——. Cottrell. (Publication received), 660.

Book reviews:

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A.S.T.M. Methods for Emission Spectrochemical Analysis, 527.

Balston and Talbot. Guide to Filter Paper and Cellulose Powder Chromatography, 58.

Barnett. Silage Fermentation, 590.

Bellamy. Infra-red Spectra of Complex Molecules, 790.

Bowen and Wokes. Fluorescence of Solutions, 59.

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Chatt. Economic Crops. Vol. III. Cocoa, 181.

Davis and MacDonald. Richmond's Dairy Chemistry. 5th Edn., 59.

Davis, Partridge and Sargent. Bentley's Text-Book of Pharmaceutics. 6th Edn., 788.

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Flett and Gardner. Maleic Anhydride Derivatives. Reactions of the Double Bond, 57.

Book reviews—*continued.*

Food Law Institute. Federal Food, Drug and Cosmetic Law. Administrative Reports, 1907–49. 309.

Fruton and Simmonds. General Biochemistry, 112.

Gaddum. Pharmacology. 4th Edn., 526.

Gautier. Mises au Point de Chimie Analytique Pure et Appliquée et d'Analyse Bromatologique, 392.

Glick. Methods of Biochemical Analysis. Vol. I, 658.

Hillebrand and Lundell. Applied Inorganic Analysis with Special Reference to Analysis of Metals, Minerals and Rocks. 2nd Edn., 113.

Ingold. Structure and Mechanism in Organic Chemistry, 659.

Institution of Water Engineers. Approved Methods for Physical and Chemical Examination of Water. 2nd Edn., 525.

Koch and Hanke. Practical Methods in Biochemistry. 6th Edn., 791.

Kolthoff and Sandell. Textbook of Quantitative Inorganic Analysis. 3rd Edn., 182.

Lederer and Lederer. Chromatography, 790.

Lingane. Electroanalytical Chemistry, 311.

Pigott. Ferrous Analysis. Modern Practice and Theory. 2nd Edn., 57.

Pollard and McOmie. Chromatographic Methods of Inorganic Analysis, with Special Reference to Paper Chromatography, 246.

Radley. Starch and its Derivatives. 3rd Edn., Vol. I, 57; Vol. II, 591.

Radley and Grant. Fluorescence Analysis in Ultra-Violet Light. 4th Edn., 591.

Reiner. Standard Methods of Clinical Chemistry. Vol. I, 309.

Samuelson. Ion Exchangers in Analytical Chemistry, 183.

Scribner and Meggers. Index to Literature on Spectrochemical Analysis. Part III, 1946–50. 660.

Siggia. Quantitative Organic Analysis via Functional Groups. 2nd Edn., 722.

Snell. Biochemical Preparations. Vol. III, 659.

Surrey. Name Reactions in Organic Chemistry, 723.

Swanson and Fuyat. Standard X-ray Diffraction Powder Patterns. Vol. II, 312.

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Venkataraman. Chemistry of Synthetic Dyes. Vol. II, 461.

Vickery. Chemistry of Lanthanons, 310.

Wallis. Practical Pharmacognosy. 6th Edn., 245.

Whiteley. Thorpe's Dictionary of Applied Chemistry. 4th Edn. Vol. XI, 789.

Wolstenholme and Cameron. Ciba Foundation Colloquia on Endocrinology. Vols. II and V, 58.

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Journal of Analytical Chemistry of U.S.S.R. in English Translation. Vol. VII, No. 1, Jan.–Feb., 1952. 461.

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Cheesemaking: Standardisation of Milk for ——. Neethling. (Publication received), 60.
Chemical(s) Papers: Current ——. Chemical Society. 1954, No. 1. (Publication received), 184.
Toxic —— in Agriculture: Residues in Food. Report of the Working Party. (Publication received), 246.
Chemical Society: Annual Reports on Progress of Chemistry for 1953. (Publication received), 592.
Current Chemical Papers. 1954, No. 1. (Publication received), 184.
Chemistry: Annual Reports on Progress of —— for 1953. Chemical Society. (Publication received), 592.
Experimental Inorganic ——. Guide to Laboratory Practice. Dodd and Robinson. (Publication received), 116.
Industrial and Manufacturing ——. Part II. Inorganic. Vols. I and II. Martin. 6th Edn. by Francis. (Publication received), 464.
Intermediate Practical —— Course. Turton. (Publication received), 464.
Name Reactions in Organic ——. Surrey. (Publication received), 464; (Review), 723.
of Carbon Compounds. Vol. II, Part B. Alicyclic Compounds. Rodd. (Publication received), 312.
Organic ——. Fuson and Snyder. 2nd Edn. (Publication received), 528.
Outline of ——. Summary for First Year Students. Manson. Trans. and revised by Turner. (Publication received), 464.
Practical Physical ——. Findlay. 8th Edn. by Kitchener. (Publication received), 464.
Reports on Progress of Applied ——. Society of Chemical Industry. (Publication received), 660.
Strengths of Chemical Bonds. Cottrell. (Publication received), 660.
Structure and Mechanism in Organic ——. Ingold. (Review), 659.
Technique of Organic ——. Vol. VI. Cheronis. (Publication received), 792.
Techniques of Clinical ——. Bullock. (Publication received), 792.
Thorpe's Dictionary of Applied ——. 4th Edn. Vol. XI. (Publication received), 528; (Review), 789.
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Dictionary: French–English —— for Chemists. Patterson. 2nd Edn. (Publication received), 592.

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Optics: Advanced Treatise on Physical Chemistry. Vol. IV. Physico-Chemical ——. Partington. (Publication received), 60.
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Pharmaceutics: Bentley's Text-book of ——. Davis, Partridge and Sargent. 6th Edn. (Publication received), 592; (Review), 788.
Pharmacognosy: Practical ——. Wallis. 6th Edn. (Publication received), 116; (Review), 245.
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Physical Chemistry: Advanced Treatise on ——. Vol. IV. Physico-Chemical Optics. Partington. (Publication received), 60.
Chemistry: Practical ——. Findlay. 8th Edn. by Kitchener. (Publication received), 464.
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  British Standards Institution. Annual Report, 1953–54. 724.
  British Standards Institution. Yearbook, 1954. 592.
  Bullock. Techniques in Clinical Chemistry, 792.
  Chadelle and Vandael. Chimie Analytique. Appliqué à la Métallurgie. 3rd Edn., 60.
  Charlot. Qualitative Inorganic Analysis. New Physico-Chemical Approach, 528.
  Charlot, Bézier, Gauguin, Odekerken and Schleicher. Qualitative Schnellanalyse, 792.
  Chatt. Economic Crops. Vol. III. Cocoa, 60.
  Chemical Society. Annual Reports on Progress of Chemistry for 1953. 592.
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  Cheronis. Technique of Organic Chemistry. Vol. VI, 792.
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  Davis, Partridge and Sargent. Bentley's Textbook of Pharmaceutics. 6th Edn., 592.
  Delahay. New Instrumental Methods in Electrochemistry, 792.
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  Fuson and Snyder. Organic Chemistry. 2nd Edn., 528.
  Gaddum. Pharmacology. 4th Edn., 184.
  Gautier. Mises au Point de Chimie Analytique Pure et Appliquée et d'Analyse Bromatologique. 2nd Series, 724.
  Glick. Methods of Biochemical Analysis. Vol. I, 392.
  Goldman and Jacobs. Chemical Methods in Industrial Hygiene, 116.
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Harley and Wiberley. Instrumental Analysis, 592.
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Honig, Jacobs, Lewin, Minrath and Murphy. Van Nostrand Chemist's Dictionary, 184.
Institution of Water Engineers. Approved Methods for Physical and Chemical Examination of Water. 2nd Edn., 312.
Ivanovszky. Wachs-Enzyklopädie. Vol. I, 2nd Edn., 592.
Johnstone. Minerals for Chemical and Allied Industries, 528.
Jordan. Vapour Pressure of Organic Compounds, 464.
Koch and Hanke. Practical Methods in Biochemistry. 6th Edn., 464.
Lederer and Lederer. Chromatography. Review of Principles and Applications, 184.
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Manson. Outline of Chemistry, 464.
Martin. Industrial and Manufacturing Chemistry. Part II. Inorganic. Vols. I and II. 6th Edn., 464.
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National Physical Laboratory. Balances, Weights and Precise Laboratory Weighing, 660.
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Partington. Advanced Treatise on Physical Chemistry. Vol. IV. Physico-Chemical Optics, 60.
Patterson. French–English Dictionary for Chemists. 2nd Edn., 592.
Pesez and Poirier. Méthodes et Réactions de l'Analyse Organique. Vol. III. Réactions Colorées et Fluorescences, 464.
Pharmaceutical Society of Great Britain. Calendar, 1953–1954. 60.
Price. Organic Syntheses, Vol. 33. 60.
Prigogine and Defay. Chemical Thermodynamics, 792.
Prodinger. Organische Fällungsmittel in der quantitativen Analyse. 3rd Edn., 116.
Radley. Starch and its Derivatives. Vol. II. 3rd Edn., 60.
Radley and Grant. Fluorescence Analysis in Ultra-Violet Light. 4th Edn., 312.
Reiner. Standard Methods of Clinical Chemistry. Vol. I, 60.
Rodd. Chemistry of Carbon Compounds. Vol. II, Part B. Alicyclic Compounds, 312.
Scribner and Meggers. Index to Literature on Spectrochemical Analysis. Part III, 1946–50. 592.
Sebrell and Harris. Vitamins: Chemistry, Physiology, Pathology. Vol. I, 464; Vol. II, 792.
Sell. Significance of Properties of Petroleum Products, 660.
Siggia. Quantitative Organic Analysis via Functional Groups. 2nd Edn., 392.

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Society of Chemical Industry. Reports on Progress of Applied Chemistry, 1953. 660.
Sumner. Clayton's Theory of Emulsions and their Technical Treatment. 5th Edn., 464.
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Tin Research Institute. Properties of Tin, 464.
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Wallis. Practical Pharmacognosy. 6th Edn., 116.
Weihrich, R. Chemische Analyse in der Stahlindustrie. 4th Edn., 312.
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Williams. Elements of Chromatography, 792.
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Assay and Detection of Pyrogens, 592.
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General Survey of British Pharmacopoeia 1953. 592.
Journal of Analytical Chemistry of the U.S.S.R. in English Translation. Vol. VII, No. 1, 60.
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Russia: Journal of Analytical Chemistry of U.S.S.R. in English Translation. Vol. VII, No. 1, January–February, 1952. (Publication received), 60; (Review), 461.
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Spectrochemical Analysis: Index to Literature on ——. Part III, 1946–50. Scribner and Meggers. (Publication received), 592; (Review), 660.
  Analysis: Methods for Emission ——. A.S.T.M. (Publication received), 184.

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- **Standards:** Food —— (Margarine) Order, 1954. 391.
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- **Starch** and its Derivatives. Vol. I. Radley. 3rd Edn. (Review), 57; Vol. II. (Publication received, 60; (Review), 591.
- **Statistical Analysis** in Chemistry and Chemical Industry. Bennett and Franklin. (Publication received), 724.
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  - Die chemische Analyse in der Stahlindustrie. Weihrich. 4th Edn. by Winkel. (Publication received), 312.
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U.S. National Bureau of Standards: Recommendations for Disposal of Carbon-14 Wastes. (Publication received), 392.
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Vitamins: Chemistry, Physiology, Pathology. Vol. I. Sebrell and Harris. (Publication received), 464; Vol. II, 792.
Volumetric glassware: Reports on metric units of volume and standard temperature of ——. B.S. 501, 554:1952. Amendment slip, 180.

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ERRATA:

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p. 181, 4th line from foot of page. *For* "Serum sodium = 48·88 $(x - y)$ mg per ml," *read* "Serum sodium = 48·88 $(x - y)$ mg per 100 ml."

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p. 110, key at top of Fig. 1. *For* "Extracted liquid" *read* "Extracting liquid" *and vice versa.*

p. 221, 2nd line of *Ammonium hydroxide - ammonium chloride buffer solution* (*p*H 10) in list of reagents. *After* "sp.gr. 0·880" *add* "and make up to 1 litre with water."

p. 273, 2nd line below Table I. *For* "Irving and Rossotti's[6]" *read* "Irving and Rossotti's[1]."

p. 277, 2nd line below Table VI. *For* "Belcher" *read* "Belcher, Nutten and Stephen." 2nd line below Fig. 3. *After* "Belcher" *add* "*et al.*[6]"

p. 291, 3rd line from foot of page. *For* "m.p. 225° C" *read* "m.p. 255° C."

p. 346, the first 4 lines under Table I were inserted in error and should be deleted.

p. 347, the heading at the top of the page should read "METHOD FOR THE DETERMINATION OF TANTALUM."

p. 348, *for* "METHOD FOR THE DETERMINATION OF TANTALUM" *read* "METHOD FOR THE DETERMINATION OF NIOBIUM."

p. 459, table I, column 7 ("Pyrethrin I"), 5th line (sample E). *For* "0·01 (0·01)" *read* "0·34 (0·01)."

p. 532, 7th line. *For* "temerarious" *read* "timorous." 24th line. *For* "screen side" *read* "lamp side"; *for "left-hand" read "right-hand."*

p. 617, 9th line of text. *For* "Russel" *read* "Russell." 16th line of text. *Delete* "with tocopherol."

p. 622, 7th line. *For* "to oxidise" *read* "for."

p. 731, 5th line of Synopsis. *For* "0·005 p.p.m." *read* "0·05 p.p.m."

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