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* Ask for our booklet on "Titration in non-aqueous media"

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Summaries of Papers In this Issue

Beam-shaped Electrothermal Graphite Tube Furnace for Atomic-absorption Spectrophotometry

Commercial graphite tubes (Massmann type) generally have a cylindrical shape and only a portion of the internal volume is available for absorption, owing to the geometry of the focused light beam. The aim of this work was to find the best geometry of the atomisation chamber to exploit the absorbing power of the optical system.

Starting from the geometry of the light beam of an atomic-absorption spectrophotometer (Pye Unicam SP1900), the size and geometry of the tube have been defined. The temperature distribution along the axis of the tube, the diffusion of the atomic vapour from the centre to the ends of the tube and the residence time of the atoms within the tube were studied to determine their influence on the analytical sensitivity.

As theoretical calculations demonstrated that the improved tube should give an appreciable enhancement of the sensitivity, a graphite tube was constructed according to this new geometry. The experimental results obtained with different elements confirmed the expected improvement in the analytical performance.

Keywords: *Atomic-absorption spectrophotometry*; *electrothermal atomiser*; *beam-shaped graphite tube*

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Analyst, 1978, **103**, 1089–1099.

Interference of Calcium on Barium as a Means of Assessing Atomic-absorption Spectrophotometers

The well documented interference of calcium on the atomic-absorption determination of barium has been studied by using a number of different commercially available instruments. The widely differing results obtained on varying the calcium to barium ratios using these instruments are thought to indicate that the interference is essentially an instrumental artefact, and should be largely avoidable.

Keywords: *Atomic-absorption spectrophotometry*; *instrumentation*; *barium determination*; *calcium interference*

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Analyst, 1978, **103**, 1100–1103.

Direct Determination of Ammonium-nitrogen by Flame Emission Spectrometry in a Hydrogen - Nitrogen Diffusion Flame

A method for the determination of trace amounts of ammonium ion in aqueous solution has been developed that utilises the chemiluminescent emission at 336.0 nm of the NH species produced when ammonia gas generated from alkaline sample solutions is introduced into a hydrogen - nitrogen diffusion flame.

The method has been applied successfully to the determination of the exchangeable ammonium-nitrogen content of soils. A practical detection limit of 0.2 μg ml^{-1} has been obtained for a 5-ml sample.

Keywords: *Ammonium-nitrogen determination*; *NH emission spectrometry*; *hydrogen - nitrogen diffusion flame*; *soil analysis*

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Analyst, 1978, **103**, 1104–1115.

ANALYSIS 79

Atomation in Industrial and Clinical Chemistry

The next Analysis conference will be held at The City University, London on 16th–18th July, 1979.

This conference will focus attention on the cross-fertilization of ideas and concepts of automation between workers in clinical, industrial and academic environments.

Automation implies a system approach that can be applied at all levels of the analytical procedure and when applied in a complete sense has in addition to a technical and scientific impact an influence on managerial, organisational and economic considerations. The conference will focus attention on all these aspects within the general framework of the following sessions:

- Education
- New Instrumentation
- Costing and Management
- Applications
- Standardisation

The programme committee comprising Dr. Peter Stockwell, Dr. Fred Mitchell, Derrick Porter and Fred Fearn, have invited an international team of authoritative speakers to provide the keynote lectures and to chair the various sessions. Whilst the majority of speakers have been invited, there will be sessions for short, submitted papers. Authors wishing to present papers in these areas should submit abstracts for consideration by the programme committee before 30th November 1978.

For further details on Analysis 79 contact Beverly Humphrey, Scientific Symposia Ltd., 33/35 Bowling Green Lane, London EC1R 0DA. Tel: 01 837 1212.

Spectrophotometric Determination of 6,7-Dihydroxycoumarin and Its Methoxy Derivatives

A spectrophotomctric method for the determination of the single components in a mixture of 6,7-dihydroxycoumarin and its methoxy derivatives is described. The method is based on the determination of 6,7-dihydroxycoumarin after complexation with molybdophosphoric acid in buffered solution and of 6-hydroxy-7-methoxy-, 7-hydroxy-6-methoxy- and 6,7-dimethoxycoumarin by treatment with sodium methylate solution followed by difference absorbance measurements. This procedure has also been applied successfully to the study of the methylation reaction of 6,7-dihydroxycoumarin.

Keywords: 6,7-*Dihydroxycoumarin*; 6-*hydroxy*-7-*methoxycoumarin*; 7-*hydroxy*-6-*methoxycoumarin*; 6,7-*dimethoxycoumarin*; *spectrophotometry*

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Analyst, 1978, **103**, 1116–1120.

Spectrophotometric Determination of Methylmercury in Fish Tissue with Dithizone Using a Dual-wavelength Procedure

A dithizone spectrophotometric procedure is described for the measurement of trace concentrations of methylmercury salts. The application of a simple equation using absorbance measurements taken at two wavelengths cancels out small differences in excess of dithizone arising between blank and sample, thus ensuring good precision in the range 0.1–4.0 μg ml^{-1}.

The developed method is used in combination with the Westöö extraction procedure to determine methylmercury concentrations in fish tissue. A crab meat sample contained less than 0.04 μg g^{-1}, and values for eight tuna fish ranged from 0.08 to 0.41 μg g^{-1}.

Keywords: *Methylmercury determination*; *fish tissue analysis*; *dual-wavelength spectrophotometry*; *dithizone*

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Analyst, 1978, **103**, 1121–1126.

Determination of a Non-volatile *N*-Nitrosamine on a Food Matrix

A method devised for the determination of *N*-nitrososarcosine, in which the *N*-nitrosamine in solution is denitrosated with hydrogen bromide to form volatile products that are rapidly removed and determined in a chemiluminescence analyser, has been applied successfully to the same compound on powdered corn flakes. Differentiation of *N*-nitrososarcosine and a number of other *N*-nitrosamines and *N*-nitrosamides from inorganic nitrite was achieved by decomposing the nitrite with acetic acid prior to the denitrosation of the *N*-nitroso compounds. In the presence of a secondary amine receptor limited nitrosation can occur during the process of differentiation but this can be prevented through the use of ascorbyl palmitate. In differentiating between large amounts of nitrite and much lower levels of *N*-nitrososarcosine on corn flakes, using a chemiluminescence analyser, the duration of the response from the nitrite can be shortened by freeze-drying the food matrix in the presence of ascorbic acid. The spectrophotometric determination of *N*-nitrososarcosine as nitrosyl bromide released into solution by the action of hydrogen bromide was hindered by the presence of powdered corn flakes.

Keywords: N-*Nitroso compound determination*; N-*Nitrososarcosine determination*; *food analysis*; *chemiluminescence analyser*

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Analyst, 1978, **103**, 1127–1133.

Sulphonated Alizarin Fluorine Blue (AFBS)

Part IV. A Critical Comparison of the Use of AFBS Against Alizarin Fluorine Blue (AFB) and the Fluoride Electrode for the Determination of Low Fluoride Concentrations; Interferences with the AFBS Method and Their Removal

The AFBS and AFB positive absorptiometric methods for the determination of fluoride in water were evaluated with respect to sensitivity, range, reproducibility, rate of complex formation and stability of the colours formed. The spectrophotometric methods were also compared statistically with the use of the fluoride electrode over the same working range. The AFBS method is shown to be slightly superior to the parent AFB method and more reproducible than the electrode except at very low concentrations. The interference of 24 common ions in the AFBS method was examined. Most interfering cations are removed by ion exchange; aluminium and iron require alternative treatment. The important anionic interferences of phosphate and sulphate are readily avoided. The interference study led to the identification of a 1 : 1 : 1 lanthanum - AFBS - mercury ternary mixed-cation complex.

Keywords: *Sulphonated alizarin fluorine blue*; *lanthanum(III) complexes*; *fluoride determination*; *fluoride electrode*; *ternary bimetallic complexes*

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Analyst, 1978, **103**, 1134–1147.