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Summaries of Papers in this Issue

High-resolution Carbon-13 Solid-state Nuclear Magnetic Resonance Spectroscopy

The status of high-resolution carbon-13 solid-state nuclear magnetic resonance (NMR) spectroscopy with respect to quantitative organic analysis is discussed. As the techniques for obtaining NMR spectra from solids are somewhat different from those used in liquid-state NMR, the experimental concepts are outlined first. Spectra that illustrate a few of the topics that have been studied in the author's laboratory are then presented. Finally, the problem of quantitateness (or indeed non-quantitateness) of such carbon-13 solid-state NMR spectra is examined.

Keywords: Solid-state carbon-13 nuclear magnetic resonance spectroscopy

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Analyst, 1982, **107**, 241–252.

Determination of Lead and Antimony in Urine by Atomic-absorption Spectroscopy with Electrothermal Atomisation

Two methods have been developed for determining antimony and lead in urine. In the first method the urine is wet oxidised and the metals are extracted as chelates into an organic phase. This is then analysed by atomic-absorption spectroscopy after carbon-rod atomisation. Nitric acid must be eliminated from the wet-oxidation mixture because it causes incomplete recovery of the antimony.

In the second method the lead and antimony are extracted directly from the urine and are again determined by atomic-absorption spectroscopy with carbon-rod atomisation. This is the preferred method for the routine analysis of normal urine samples. The wet-oxidation method is used for samples that contain abnormal amounts of coproporphyrins, chelating agents or other organic materials. Both methods gave comparable results in the range 0–200 $\mu\text{g l}^{-1}$ of lead and antimony.

Keywords: Antimony determination; lead determination; urine analysis; atomic-absorption spectroscopy; electrothermal atomisation

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Analyst, 1982, **107**, 253–259.

Micro-determination of Tin in Organotin Compounds by Flame-emission and Atomic-absorption Spectrophotometry

A general method is described for the micro-determination of tin in organotin compounds, using either flame-emission or atomic-absorption spectrophotometry. Air - acetylene, dinitrogen oxide - acetylene and air - hydrogen flames have been compared: either of the air flames may be used, but the last mentioned is to be preferred as it is less noisy. The effect of different instrumental and chemical parameters on the emission and absorption of tin has been investigated. A number of organotin compounds have been analysed by both methods as well as by an alternative extraction - spectrophotometric method. The proposed method, in which samples are dissolved in a mixed solvent of water - hydrochloric acid - ethanol - butan-2-one and are aspirated into a near stoichiometric air - hydrogen flame in an atomic-absorption spectrophotometer, is simple, accurate, rapid and equally useful for organic and for inorganic compounds. Metallic tin is used as the standard.

Keywords: Tin determination; organotin compounds; atomic-absorption spectrophotometry; flame-emission spectrophotometry; micro-analysis

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Analyst, 1982, **107**, 260-268.

Determination of Mercury in Coal by Non-oxidative Pyrolysis and Cold Vapour Atomic-fluorescence Spectrometry

Mercury was liberated from coal by non-oxidative pyrolysis at 800 °C with a nitrogen purge and collected in acidified potassium permanganate solution. Subsequent determination was by cold vapour atomic-fluorescence spectrometry using an argon-sheathed windowless cell. The mercury vapour was generated continuously by pumping tin(II) chloride and sample to a novel reduction cell. This continuous flow system offered excellent precision, with a 2% relative standard deviation at 0.5 ng ml⁻¹ and a detection limit (2σ) of 0.043 ng ml⁻¹. The accuracy and precision were assessed by performing replicate analyses on a number of coals, including NBS SRM 1632a bituminous coal, for which a mean value of 134 ng g⁻¹ and a standard deviation of 3.1 ng g⁻¹ were obtained. The excellent sensitivity, precision and extended linear working range of the method (5 ng g⁻¹ to 25 μg g⁻¹) are noted.

Keywords: Mercury determination; coal; cold vapour atomic-fluorescence spectrometry; non-oxidative pyrolysis; continuous flow system

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Analyst, 1982, **107**, 269-275.

**Optical Emission Spectrometry with an Inductively Coupled
Radiofrequency Argon Plasma Source and Direct Sample
Introduction from a Graphite Rod**

Preliminary studies of analytical performance are described for an instrumental assembly in which microlitre volumes of liquid samples are applied to a graphite rod, desolvated and the rod is inserted axially directly into a continuously operating low-power inductively coupled argon plasma. Simplex optimisation of operating parameters has been undertaken for manganese to determine the most favourable signal to background intensity ratios and detection limits are reported for manganese, cadmium, cobalt, copper, iron, nickel and lead.

Keywords: Optical emission spectrometry; inductively coupled plasma; direct sample introduction; graphite rod

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Analyst, 1982, **107**, 276–281.

**2-Thiobarbituric Acid as a Reagent for the Determination of
Bismuth(III) by Normal and Derivative Spectrophotometry**

A spectrophotometric study of the reaction of 2-thiobarbituric acid (TBA; 4,6-dihydroxy-2-mercaptopyrimidine) with bismuth(III) is presented. Bismuth(III) forms a coloured complex with an absorbance maximum at 416 nm with TBA in acid solution (pH 1.2–1.7). The maximum absorbance value is achieved within about 25 min. Beer's law is obeyed for a bismuth concentration of up to 10.2 $\mu\text{g ml}^{-1}$. The molar absorptivity of the complex is $2.4 \times 10^4 \text{ l mol}^{-1} \text{ cm}^{-1}$ at 416 nm and the sensitivity of the reaction is, according to Sandell's calculation, $8.7 \times 10^{-3} \mu\text{g cm}^{-2}$ per 0.001 absorbance unit. Cobalt(II), nickel(II), magnesium(II), tin(II), lead(II), zinc(II), barium(II), manganese(II) and aluminium(III) do not interfere in the determination. The same procedure can also be used for the spectrophotometric determination of copper(II), which seriously interferes in the bismuth determination. A satisfactory method for the determination of bismuth in the presence of copper by derivative spectrophotometry is, however, reported.

Keywords: Bismuth(III) determination; 2-thiobarbituric acid; derivative spectrophotometry

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Analyst, 1982, **107**, 282–287.

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Ion-selective Polymeric-membrane Electrodes with Immobilised Ion-exchange Sites. Part II. Selectivity and Application Studies on a Calcium Electrode

A further study is reported of a calcium ion-selective electrode where the sensor is a dialkylphosphate group which is covalently bound to poly(styrene-*b*-butadiene). Selectivity in the order $\text{Ca}^{2+} > \text{Ba}^{2+} > \text{Sr}^{2+} > \text{Mg}^{2+} > \text{M}^{+}$ was found. Interference was encountered from transition metal cations but no irreversible poisoning was observed. The response to surfactants was found to be less than that exhibited by established electrodes. The performance of the covalently bound sensor electrode for the monitoring of coking-plant effluent has been compared with other analytical techniques. This electrode appears to offer advantages of robustness, reliability and improved lifetime over a commercial liquid membrane electrode and a PVC membrane electrode for this application.

Keywords: Ion-selective electrode; calcium analysis; polymeric membrane; immobilised ion-exchange groups; effluent monitoring

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Analyst, 1982, **107**, 288–294.

Determination of Obscuration. Part I. Evaluation of the Dry Extract Method

The obscuration values obtained using the AOAC dry residue method were compared with theoretical values and the limits of applicability of the AOAC factor were calculated. It is recommended that obscuration be expressed as the difference in density rather than in strength and that a corresponding density factor be used in place of the strength factor when applying the dry extract method. The scope of invariability of the density factor is significantly larger than that of the common strength factor.

Keywords: Obscuration; ethanol; alcoholic strength determination

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Analyst, 1982, **107**, 295–300.

Gas-chromatographic Determination of 1-(2-Chloroethyl)-3-(*trans*-4-methylcyclohexyl)-1-nitrosourea (Methyl-CCNU). Part I. Cyclisation to a Substituted Aminooxazoline

Methods for the gas-chromatographic determination of methyl-CCNU employing cyclisation to the substituted aminooxazoline have been explored. These procedures were found to be unsatisfactory for the determination of the drug in body fluids.

Keywords: Methyl-CCNU determination; aminooxazoline; gas chromatography

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Analyst, 1982, **107**, 301–306.