Differential-pulse Voltammetric Determination of Phosphate as Molybdovanadophosphate at a Glassy Carbon Electrode and Assessment of Eluents for the Flow Injection Voltammetric Determination of Phosphate, Silicate, Arsenate and Germanate

The redox behaviour of molybdovanadophosphate at a glassy carbon electrode is described and a procedure is given for the voltammetric determination of phosphate as molybdovanadophosphate at a glassy carbon electrode in a static system. Procedures are also given for the voltammetric flow injection determination of phosphate, silicate, arsenate and germanate by the injection of heteropolyacids pre-formed in various aqueous, aqueous acetone and aqueous ethanolic reagents into eluents consisting of the reagent blank. This procedure effectively eliminates the background signal of the blank and allows precise determinations to be made. Plots of electrode potential against the current obtained are given.

Silicate and phosphate can be determined at 10^{-7} and 10^{-6} M levels, respectively. Arsenate has only been determined at the 10^{-5} M level, and the precise determination of germanate is difficult owing to adsorption at the glassy carbon electrode.

Keywords: *Orthophosphate, silicate, arsenic and germanium determination*; *voltammetry*; *flow injection analysis*

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Analyst, 1981, **106**, 1288–1295.

Use of Ascorbic and Thioglycollic Acids to Eliminate Interference from Iron in the Aluminon Method for Determining Aluminium

The use of ascorbic and thioglycollic acids as inhibitors for the interference of iron in the aluminon method of Hsu have been examined. The use of ascorbic acid, as proposed by Jayman and Sivasubramaniam, has been found to change iron interference from positive to negative causing aluminium to be underestimated. However, the addition of 0.2 ml of a 1% *V*/*V* solution of thioglycollic acid to solutions containing aluminium in amounts ranging from 10 to 50 μg has been proved to suppress the interference from up to 900 μg of iron.

Keywords: *Aluminium determination*; *aluminon method*; *ascorbic acid*; *iron interference*; *thioglycollic acid*

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Analyst, 1981, **106**, 1296–1301.

Effect of Temperature on the Structural Rearrangements of Polyesters (LAC-series) when Used as Liquid Stationary Phases in Gas - Liquid Chromatography

This study deals with the effect of temperature on the gas-chromatographic behaviour of thiols, alkanes, branched-chain alkanes and cyclic alkanes on polyester (LAC-446, LAC-745, LAC-772, LAC-860, LAC-886, LAC-841 and LAC-935) liquid stationary phases. Increases were observed in specific retention volumes as well as improvements in the separations at certain temperatures. The critical temperatures were different for the various liquid stationary phases; these were 110 °C for LAC-446 and LAC-886 and 120 °C for LAC-860 and LAC-772. Differential-thermal analysis of two of the liquid stationary phases indicated a change in the physical properties of the polymers during heating.

Keywords: *Gas - liquid chromatography*; *liquid stationary phases*; *effect of temperature*; *LAC-series of polyesters*; *structural rearrangements*

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Analyst, 1981, **106**, 1302–1308.

Rapid Automated Enzymatic Method for the Determination of Alcohol in Blood and Beverages Using Flow Injection Analysis

An enzymatic method for the determination of alcohol using flow injection analysis is described. Samples are suitably diluted and introduced directly into the system. Blood alcohol is analysed by the stop-flow technique at a rate of 70–80 samples per hour and the results are compared with those from headspace gas chromatography. Alcohol in several beverages is analysed by the continuous-flow technique at a rate of up to 120 samples per hour. In both instances the result is available less than 30 s after injection.

Keywords: *Flow injection analysis*; *alcohol determination*; *enzymatic method*; *whole blood*; *stop-flow technique*

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Analyst, 1981, **106**, 1309–1317.

A Copper(I) Iodide Paper for the Detection and Determination of the Concentration of Mercury Vapour in the Workplace Atmosphere

The development and preparation of a test paper for mercury in air is described. A measured volume of air is drawn through a silica gel-loaded filter-paper coated with a mixture of copper(I) iodide and sodium carboxymethylcellulose, and in the presence of mercury a pink stain is produced. The paper has been validated in the laboratory and a factory for the reliable determination of mercury vapour in air at concentrations up to 100 $\mu g\ m^{-3}$.

Keywords: *Mercury vapour*; *copper(I) iodide test paper*; *workplace atmosphere*; *air*

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Analyst, 1981, **106**, 1318–1325.

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Open-cell Polyurethane Foam as a Sorbent in the Extraction of Iodine-131

Open-cell polyurethane foams have proved to be effective as sorbents for inorganic and organic species and as supporting materials for various hydrophobic organic phases. They possess outstanding sorption, mass-transfer and hydrodynamic properties, which enable them to be used at relatively high flow-rates of aqueous solutions during column operation. Iodine-131 can be extracted from samples of water and milk using resilient open-cell polyurethane foams in static and pulsed column beds, when in the form of a cylindrical packing impregnated with a tri-alkylamine containing dissolved inactive iodine. Pulsed column beds, which have some advantages over static column beds, can be conveniently automated. This may lead to the development of a new type of environmental monitoring device for radioactive iodine.

Keywords: *Iodine-131 extraction*; *polyurethane foam*; *water*; *milk*; *sorbent*

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Analyst, 1981, **106**, 1326–1333.

Reactive Ion-exchange Precipitation Procedure for the Determination of Trace Amounts of Oxalate

The feasibility of using reactive ion-exchange for the determination of oxalate in aqueous solution has been demonstrated. The procedure consists of pH adjustment of the analyte solution, concentration of oxalate on a lead(II)-loaded cation exchange mini-column in the form of lead oxalate precipitate, reactive elution of oxalate by sulphuric acid dissolution and analysis of the concentrated eluate by permanganate titration. Almost quantitative yields of oxalate are obtained for concentrations typically found in human urine. The procedure represents a simple and reliable technique to concentrate oxalate for determination by classical methods.

Keywords: *Oxalate trace determination*; *reactive ion exchange*; *precipitation*; *titration*

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Analyst, 1981, **106**, 1334–1337.

Sample Fusion at Low Temperature for the Potentiometric Determination of Fluorine in Silicate Materials

Short Paper

Keywords: *Fluorine determination*; *silicates*; *alkaline fusion*; *fluoride ion-selective electrode*

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Analyst, 1981, **106**, 1338–1341.

Determination of Fluoride Ion in Bovine Milk Using a Fluoride Ion-selective Electrode

Short Paper

Keywords: *Fluoride determination*; *milk*; *ion-selective electrode*

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Analyst, 1981, **106**, 1341–1344.

Use of a Carbon Fibre Indicator Electrode for the Potentiometric Titration of Glycine in Glacial Acetic Acid

Short Paper

Keywords: *Glycine determination*; *potentiometric titration*; *carbon fibre indicator electrode*

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Analyst, 1981, **106**, 1344–1347.

Reagent for the Spectrophotometric Determination of Iron(II) in Alkaline Solution

Short Paper

Keywords: *Iron(II) determination*; *pyridine-2-hydrazide reagent*; *spectrophotometry*

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Analyst, 1981, **106**, 1348–1351.