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Application of Furnace Atomic Non-Thermal Excitation Spectrometry for the Determination of Non-Metals

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This absence of spectral interference in AFS is due to several factors. (1) The fluorescence spectrum is relatively simple, being confined principally to the resonance lines used in the excitation source. (2) Changes in DC signals (i.e. background shifts) are discriminated against by the use of AC electronics in AFS. (3) Before spectral interference can occur in AFS the absorption profile of the interfering element must overlap with the emission profile of the source. (4) Interference will not occur unless the population of the interfering element in the correct energy level (usually the ground state) is significantly high. (5) Interference will not happen unless the quantum efficiency is significantly high at the wavelength concerned.

Data were presented showing linear dynamic ranges of some five to six orders of magnitude for the ASIA system, evidence of relative freedom from chemical matrix effects and ionization interference and detection limits in the p.p.b. region. It was also demonstrated, by curves of growth, that the ICP is a line source.

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Various atomic spectrometry techniques are used for the determination of elemental concentrations in a wide range of samples. Although procedures for the determination of metallic impurities are well established, the development of successful methods for the measurement of non-metal elements at trace levels has proved more difficult. In particular, better methods are required for the determination of B, Cl, P, S, Si, etc., in industrial and clinical samples.

There is currently considerable interest in the development of new types of excitation source for the determination of metals and non-metals by atomic emission spectrometry. Most of the studies involve high-energy helium plasmas which are capable of generating atomic and ionic emission from elements at the level of 1 $\mu g\ g^{-1}$ or 1 $\mu g\ l^{-1}$. One of the main problems encountered in the use of analytical helium plasmas is the disruption of the discharge when the sample material is vaporized. This problem has been addressed in furnace atomic non-thermal excitation spectrometry (FANES) by combining the systems used to atomise and excite the sample components. In FANES, a hollow cathode discharge is generated inside an electrothermal atomizer tube operated at a pressure of 10–20 Torr (*ca.* 1.3–2.7 kPa) (Littlejohn *et al.* 1983; Falk *et al.* 1984). The graphite tube acts as the hollow cathode as well as the vaporizing device. The sample is deposited as a solution (typically 20 μl) into the graphite tube at atmospheric pressure. The solvent is evaporated and the residue charred by electrothermal heating to remove some of the matrix. The atomizer is then evacuated and filled with helium to a pressure of 10–20 Torr, for the establishment of a discharge. Once the plasma is formed, the graphite tube is heated rapidly to temperatures in excess of 2000 °C to vaporize and atomize the sample. Excitation of analyte atoms in the discharge gas is caused mainly by electron collisions and when helium is used as the plasma gas, the excitation limit of the discharge is 21 eV. It is therefore possible to excite both non-metals and metallic impurities in the sample.

The application of FANES to the determination of non-metals has been investigated. Thirty-one atom and ion emission lines of B, Br, Cl, F, I and P have been identified in the range 200–900 nm and detection limit values measured for each wavelength. The best values were in the range 0.4–2 $\mu g\ ml^{-1}$ for the halogens, and 0.01–0.02 $\mu g\ ml^{-1}$ for B and P, respectively. In general, improved sensitivity is achieved when the atomizer is operated at higher pressures and higher discharge currents.

The usefulness of the FANES technique in industrial analysis has been illustrated by the determination of P and Si in antifreeze, B in a fire-resistant fabric, and Br, Co, Fe and Mn in an acetic-acid-based liquor used in terephthalic acid production.

References

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