Plenary and keynote lectures

Inorganic mass spectrometry of solid samples

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Within the field of instrumental inorganic trace analysis the use of mass spectrometry is enjoying at present a resurgence of interest. Existing and sometimes long established methods such as spark source mass spectrometry and thermal ionization mass spectrometry are finding new applications. Also, rapid development of new methods are appearing, and become quickly commercialized as market potential is realised, such as inductively coupled plasma mass spectrometry and glow discharge mass spectrometry. This new wave of interest and the diversification of the methodology are driven by materials science which requires characterization of purity with increased sensitivity and accuracy for an expanding range of elemental impurities, in a rapidly expanding range of technologically important materials. Controlled impurity doping, segregation effects at interfaces, grain boundary migration and recrystallisation effects, lead to needs for application in increasingly difficult analytical problems in which localised analysis at the specimen's surface, within gradients, even the three-dimensional distribution of impurities is required.

The lecture will concentrate on common characteristics of a selected number of the now available methods: spark source mass spectrometry, laser mass spectrometry, secondary ion mass spectrometry and dito ion imaging and glow discharge mass spectrometry. The common characteristic features of all these methods will be emphasised especially as far as they relate to the ion source. Indeed, the ion source in which energy is deposited into the sample to atomise and ionise a representative part of it in a vacuum environment for extraction and analysis in the mass spectrometer, overwhelmingly determines both the type and the instrumental complexity of the mass spectrometric equipment and the characteristics of mass spectra. These, in turn are responsible for the case of calibration and the general quality of the analytical data that can be obtained.

A few examples of state of the art performance of the several types of mass spectrometric techniques as now routinely available will be selected from the author's laboratory experience.

Analytical procedures for environmental and biological monitoring

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Health prevention of persons exposed to chemical substances is achieved by limitation and supervision of the concentrations a) of substances in the air (Environmental Monitoring, EM)

b) of substances and metabolites in body fluids (Biological Monitoring, BM).

Both strategies belong to the sphere of responsibility of the analytical chemist. EM and BM show requirements which are different in some characteristic facts among them:

ЕМ

The assessment of exposure is difficult because the concentrations strongly depend on the location and time of measurement. The analytical determination of ppm and ppb concentrations in the air in general do not make problems nowadays. Unsolved, however, is the problem of the quality control and the determination of certain species of substances.

BM

It offers a better means of the assessment of individual exposure. The complexity of the biological matrix, its low availability, and the small concentration of substances are still a great problem for analytical chemistry. Nowadays $\mu g/l$ -concentrations of metals, organic solvents etc. can routinely be determined in biological matrices. Efficiency of analytical chemistry, however, must further be improved. Certain substances, for instance, PAH's certain metals like Pt, Ni, Co cannot be determined in the required range of concentrations. Huge efforts have to be done to determine the substances at the location of their effects for instance bound to DNA, Hb or certain proteins.

The possibilities and limitations of instrumental analysis in the field of environmental and occupational health are discussed using characteristic examples. The problems of analytical reliability and the comparability of results are discussed.

Coupling techniques in LC/MS and SFC/MS

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Over the past years, continuous interest in coupling liquid chromatography (LC) and supercritical fluid chromatography (SFC) to mass spectrometry (MS) has led to several recent developments that will be commented.

Several years ago, open-tubular columns were predicted to be difficult or even impossible to use for HPLC, and this has been unfortunately proven to be true in practice, thus the obvious reduction of the eluent volume for LC/MS coupling has not been taken. The situation is not so pessimistic for narrow bore packed columns (<1 mm i.d.), however, optimization of some LC/MS interfaces, e.g. the thermospray, requires relatively high solvent flow rates as delivered by conventional HPLC instrumentation.

