

## Power interruption experiments on ICP and MIP

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eliminating the analyte ions upon which sensitive measurements depend. We have been studying the plasma and interface regions both optically and mass spectroscopically, under a variety of conditions, with and without the shielding of the torch, to investigate when and how polyatomic ions arise. A special interface has been constructed, with a fibre optic viewing port coupled to a scanning high resolution spectrometer, to allow sensitive measurements of what happens in the interface region. These studies have suggested that a variety of mechanisms are involved in the formation of polyatomic ions. The results of our studies will be presented and critically evaluated.

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(342) Tu PM 15:40

**COMPARISON OF MATRIX EFFECTS IN QUADRUPOLE AND HIGH RESOLUTION ICP MASS SPECTROMETERS**

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Since early publications on a quadrupole ICP mass spectrometer (ICP-QMS), many researchers have investigated spectroscopic and non-spectroscopic interferences. Though a high resolution ICP mass spectrometer (HR-ICP-MS) can solve most analytical problems due to spectroscopic interferences, few studies on the formation mechanism of polyatomic ions and, in addition, non-spectroscopic interferences (matrix effect) have been reported. The matrix effect observed in ICP-QMS is closely related to a space charge effect occurring in the region between a skimmer cone and ion optics. In HR-ICP-MS, however, the coulomb repulsion can be negligible compared with kinetic energies, which ion species obtain from the acceleration by the high voltage (6-8kV) after the skimmer, therefore analyte signals in the presence of matrix elements may show different behaviors from those in ICP-QMS. In this study, effects of acceleration voltage on physical properties of plasma jets after the sampling and skimmer cones and on behaviors of analyte signals in the presence of matrix elements were investigated in order to clarify the matrix effect peculiar to HR-ICP-MS. The matrix effect was compared with that in ICP-QMS. While the electron number density decreased after the sampling cone but increased after the skimmer with increasing the acceleration voltage, the signals for polyatomic ions were increased with increasing the acceleration voltage. These results suggest the increased electron number density after the skimmer cone may promote collision induced reactions of neutral Ar with ions. When 10<sup>-7</sup> M analytes with 10<sup>-3</sup> M matrix elements were introduced into ICP-QMS and HR-ICP-MS, a signal suppression was observed in ICP-QMS, as expected, but a slight signal enhancement was observed in HR-ICP-MS. Furthermore, the rate of the signal enhancement in HR-ICP-MS varied with the acceleration voltage applied to the interface region. This may be also related to the enhanced electron number density caused by the ionization of matrix elements after the skimmer.

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(343) Tu PM 16:00

**TOWARD IMPROVING DETECTION LIMITS IN AN INDUCTIVELY COUPLED PLASMA ON-AXIS TIME OF FLIGHT MASS SPECTROMETER**

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Recently, time of flight (TOF) mass analyzers have emerged in the field of elemental mass spectrometry as an attractive alternative to scanning mass filters (quadrupole and sector-based). TOF systems

can generate complete mass spectra at repetition rates greater than 15 kHz. Additionally, the newly developed on-axis TOF mass spectrometer (ion source and TOF drift region are coaxial) offers better ion transmission and reduced mass bias than the traditional orthogonal or right angle extraction instruments. In this study the limits of detection of the inductively coupled plasma on-axis time of flight mass spectrometer (ICP-OA-TOFMS) are explored. Research has focused on the characterization of ion transmission from the atmospheric pressure ICP source to the high-vacuum environment of the mass spectrometer. Sources of ion loss in the mass spectrometer are thought to include skimming of the supersonic expansion (10--2), transmission of the primary ion beam through the ion optics (10-1), duty factor (10-1) and transmission of the flight tube (10-1). Reducing ion losses through these regions should result in an increase in ion signals and lower detection limits. Novel electrode arrangements including ion funnels, multipole ion guides and electrostatic extraction optics have been explored for ion collection and focusing in the post-skimmer supersonic expansion. Additionally, ion beam transmission in the ion-optic train was studied to determine the locations of the most significant loss.

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**PRODUCTION OF A MICROWAVE-INDUCED HELIUM PLASMA AT ATMOSPHERIC PRESSURE AND ITS ANALYTICAL FEATURES**

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A high-power (~ 1 kW cw, 2.45 GHz) annular-shaped microwave-induced helium plasma (He-MIP) was developed and preliminary characteristics are presented. The He-MIP was better suited for the determination of halogens. The plasma was generated at atmospheric pressure by an Okamoto cavity (Anal. Sci., 7 (1991) 283) which was modified to focus the energy.

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(345) Tu PM 16:40

**POWER INTERRUPTION EXPERIMENTS ON ICP AND MIP**

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The power interruption (PI) technique is a powerful method for getting insight in the presence and strength of excitation mechanisms in plasmas. It is to be expected that especially for plasmas used in the field of spectrochemical analysis this insight might lead to a better understanding by which guidelines for the application optimization can be obtained. In this study we investigate the application of PI on the ICP and a microwave plasma, the so-called TIA (torch a injection axiale). The basic idea behind the PI technique is to interrupt the first link in the power balance  $EM \rightarrow \{e\} \rightarrow \{h\} \rightarrow env$ . Which expresses how the electrons {e} are heated by the EM field and cooled by collisions with heavy particles {h}, which on their turn pass their energy to the environment by means of heat conduction or convection. Due to this chain of reactions the steady state temperature of the electrons must be larger than that of the heavy particles: i.e.  $T_e > T_h$ . If the first link, the heating of {e}, is interrupted various processes will take place. The primary process is the sudden decrease of  $T_e$  which is followed by recombination and the transport of an interrupted convection flow. Most of these responses to PI can be followed by time resolved optical emission spectroscopy. The

strength of the technique as applied to the ICP is that there are different time scales for the various processes so that they can be unraveled from each other. However, for smaller plasmas like the TIA in which due to the presence of large gradients, fluxes and Te-values are large as well, the decay of the electron gas can be so rapid that the time constant of recombination and diffusion approaches the values of electron cooling. The following topics will be dealt with: The direct monitoring during the PI of the electron gas in an ICP by means of Thomson scattering. The determination of the electron decay time as a function of radial position. The classification of the responses of the various excited levels in relation with their production/destruction balances, such as the electron-ruled Boltzmann balance of excitation/deexcitation, the Saha balance of ionization/recombination and balances for which not the electrons but heavy particles are important like charge and excitation transfer. We will also discuss the responses of the continuum radiation and molecular spectra to PI. Especially the comparison of the results as found for the TIA with those of the ICP provide essential insights. The most important differences can be traced back to the simple fact that the TIA is much more narrow than the ICP.

**LC-MS ANALYSIS OF DRUGS: INSTRUMENTATION  
AND APPLICATIONS II**

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**HPLC-CHEMICAL REACTION INTERFACE MASS  
SPECTROMETRY (HPLC/CRIMS) AND ITS  
APPLICATION IN DRUG DISPOSITION STUDIES**  
F. ABRAMSON

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**THE ROLE OF MASS SPECTROMETRY IN AN  
INTEGRATED DRUG DISCOVERY METABOLISM  
PROGRAM**  
J. YERGEY

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**ENANTIOSELECTIVE DETERMINATION OF METHYL  
PHENIDATE (RITALIN™) IN HUMAN PLASMA AT 100  
PG/ML SENSITIVITY BY LC-MS-MS**  
C. BUGGE

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**PHARMACOKINETIC ANALYSIS OF STAVUDINE IN  
LATE DEVELOPMENT STUDIES FOLLOWING 96-WELL  
SOLID PHASE EXTRACTION AND LC-MS/MS**  
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**HIGH THROUGHPUT LC/MS IN SUPPORT OF DIRECTED  
ORGANIC SYNTHESIS**  
K. MATUSZAK

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**NEW PARADIGMS FOR HIGH THROUGHPUT ANALYSIS  
AND PURIFICATION OF COMBINATORIAL LIBRARIES:  
DEVELOPMENTS IN AUTOMATED PARALLEL HPLC/MS**  
D. KASSEL

**ADVANCES IN EXTRACTION METHODOLOGIES II**

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**SURFACTANTS IN SUPERCRITICAL FLUID  
TECHNOLOGY**

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Matt Yates University of Texas, Department of Chemical  
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The effects of various surfactants on the interfacial tension between water or organics and carbon dioxide are reported along with measurements of colloid formation and stability. Fundamental thermodynamic properties including the solvation of the surfactant tail by carbon dioxide, the adsorption of the surfactant at the interface, the balance between the interactions of the surfactant with each phase, and the interactions between droplets are examined and used to interpret the phase behavior, droplet size and stability in microemulsions, emulsions and latexes. We have formed water-in-CO<sub>2</sub> and CO<sub>2</sub>-in-water emulsions that are stable for hours. These emulsions may be used for cleaning, extraction, and phase transfer reactions. The stability of silica particles suspensions stabilized by surfactants in CO<sub>2</sub> has been determined as a function of CO<sub>2</sub> density.

(353) Tu PM 14:20

**ACCELERATED SOLVENT EXTRACTION OF  
HYDROCARBONS, FATS, AND POLYMERS**

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Accelerated solvent extraction (ASE™) uses pressurized organic solvents at elevated temperatures to improve extraction speed and efficiency. For many matrices and for a variety of solutes, ASE has proven to be equivalent or superior to Soxhlet and reflux extraction techniques. Underground storage tanks containing hydrocarbon-based fuels are found worldwide. Many of these underground storage tanks are known to have leaks, allowing gasoline, diesel or other fuels to contaminate the surrounding soil. Thus, the ability to determine the level of hydrocarbon contamination in soils is an important analytical tool. Soxhlet and reflux extraction methods are time-consuming and require large volumes of solvents. Accelerated solvent extraction methods typically range from 10 to 25 minutes in duration and require from 15 to 50 mL of extraction solvent, depending upon the sample size. The extraction of hydrocarbon contaminants such as BTEX, diesel, and TPH in soils can be completed in less than 20 minutes with less than 30 mL of solvent using ASE. Accelerated solvent extraction has been successfully applied to the extraction of fat from food matrices. Various types of cheese, powdered infant formula, and both raw and prepared meat are efficiently extracted with ASE. Several traditional methods that require hours to complete can be replaced with an ASE method that can be completed in minutes. One method for meat samples requires only 12 minutes and 25 mL of solvent. The percent fat values from the ASE extractions compare well to the percent fat values obtained from the traditional methods. ASE has also been successful in the extraction of antioxidant additives from polyolefins, plasticizers from poly(vinyl chloride) (PVC), and aromatic processing oils from rubber. For example, the extraction of antioxidants such as Irganox 1010 and Irgafos 168 from polypropylene and polyethylene can be