

droplet size distributions for solutions containing varied concentrations of Sodium Dodecyl Sulfate are presented for direct comparison, and the mechanism by which droplets acquire a net excess charge will be discussed. In addition, the direct comparison of the tertiary aerosol size distributions with atomic ion emission contour maps from the ICP affords us an indication of relative contributions of plasma operating conditions versus sample introduction conditions that lead to the chemical matrix effect. Also, the results of laser light scattering experiments designed to deliberately create aerosols that are solely comprised of droplets with net positive or net negative charge will be presented..

(521) DETECTION OF NONMETALS IN AQUEOUS SOLUTIONS BY A HIGH-POWER HELIUM MICROWAVE-INDUCED PLASMA

OKAMOTO YUKIO, Toyo University, Faculty of Eng., Dept. Electrical and Electronics, 2100, Kujirai, Kawagoe, Saitama, Japan
High-power (~ 1 kW, 2.45 GHz) annular-shaped microwave- induced helium, argon and nitrogen plasmas (He, Ar and N₂-MIPs) at atmospheric pressure were produced and the characteristics were compared. The plasmas were generated by an Okamoto cavity (Anal. Sci. 7 (1991) 283). No external cooling was used to stabilize the plasmas. The discharge tube (tangential-flow torch; 8 mm i.d., 10 mm o.d.) consists of two concentric tubes, as well as two gas flow for plasma support with a spiral trajectory in order to confine the plasma. An aqueous solution was injected into the plasma by an ultrasonic system with desolvation and condensation. With solution nebulization, the typical plasma gas flow rates for He, Ar and N₂ were 10.5 l/min, 1.2 l/min. And 10.5 l/min at power of 600 W, 250 W and 1000 W, respectively.

The electron excitation temperature derived from a Boltzmann plot of eight Fe I lines for He, Ar and N₂ were on the order of 4900 K, 5500 K and 5500 K, respectively. The electron density determined by Stark- broadened profile of the least-squares fit to the H I emission line (Balmer, 486.1 nm) for He, Ar and N₂ were 2.1, 5.0 and 0.3 x 10¹⁴/cm³, respectively. The background spectrum for the He-MIP was much simpler than that for Ar and N₂-MIPs.

Introduction of aqueous chloride as NaCl and bromide as KBr produced intense ion emission lines of Cl II(479.5, 481.0 and 482.0 nm) and Br II(475.0, 478.6 and 481.7 nm) at the center of the He-MIP. The upper-state energy of the Cl II and Br II lines are 16.0 and 14.2 eV, respectively. Due to their high excitation energies, these ions lines were not observed in the Ar and N₂-MIP. The detection limits of 0.1 ppm for Cl II(479.5 nm) and 0.2 ppm for Br II(470.5 nm) were obtained in the He-MIP. The He-MIP was better suited for the determination of nonmetals in the aqueous solutions.

(522) DETERMINATION OF IODINE BY INDUCTIVELY COUPLED PLASMA SECTOR FIELD MASS SPECTROMETRY (ICP/SFMS)

BERTRAND LANGLOIS, Jean-Luc Dautheribes, Jean-Michel Mermet, C.E.A. Valrho DCC/DRRV/SEMP, BP 181 - Bat 166, Bagnols sur Ceze Cedex, France

Iodine is a challenging element to determine because of memory effects and possible influence of the chemical form. Inductively coupled plasma sector field mass spectrometry (ICP-SFMS) was used to determine iodine as iodide (I⁻) in a nitric media with added ascorbic acid as a reducing agent. Several nebulizers and spray chambers were tested to improve sensitivity and to decrease memory effects. At first, we studied effects of ascorbic acid concentration on stability of iodide and on iodine noise level.

The influence of the chemical form was also studied by comparing mineral iodine (NaI) and organic iodine (CH₃I). Different behaviors in terms of signal and time response were

observed. The low volatilization temperature of iodomethane (315,5 K) seems to be the main explanation for the higher CH₃I signal. Volatilization occurs during the nebulization of a 2% nitric acid solution containing iodomethane. In contrast to sodium iodide, it was found that CH₃I is injected into the plasma as a vapor and not as small droplets of an aerosol. As a result, transport efficiency of iodomethane between the nebulizer and the torch is significantly higher than the 3-5 % efficiency usually obtained for most of the elements with a conventional sample introduction system. Furthermore, responses obtained with different species containing iodine (potassium iodate, 2-iodoethanol and iodoacetic acid) were found to be similar. All these species have a boiling point higher than 373 K.

Finally, because of the easy volatilization of iodomethane, care must be taken in order not to loose iodine during sample preparation and during analysis. A special introduction system has been designed in order to confine the head space of sample during the time of analysis. A specific protocol was also used for preparation of iodine standards.

In order to overcome these limitations, a direct sample injection system (DIHEN) will be used. On one hand, we expect to minimize the difference of response observed between CH₃I and I⁻ in terms of sensitivity. On the other hand, memory effects caused by spray chambers should decrease drastically. Moreover, it is possible to add a guard electrode to the conventional torch assembly. The influence of the electrode on sensitivity was studied.

(523) OPEN VESSEL MICROWAVE EXTRACTION OF METALS IN BIOLOGICAL AND BOTANICAL MATERIALS

M. CRISTINA LOMBARDI, Leticia Costa, Joaquim Nobrega, Ana Rita Nogueira*, University Federal of Sao Carlos/Dept. of Chemistry, P.O. Box 676, Sao Carlos, SP, Brazil

The complete sample decomposition is not always necessary when measurements are carried out using optical techniques. Depending on both technique employed and sample matrix characteristics, an extraction procedure can be completely suitable for quantitative determinations. To implement this approach is essential to perform a critical evaluation of the behavior of each element, of the extractor solution, and of the temperature and time of extraction. The temperature can be controlled adopting microwave-assisted procedures that guarantee fast heating and repeatability. In this work an open-vessel microwave system (Star 6, CEM) was employed to investigate the efficiency of the extraction procedures. The extraction of Al, Ca, Cu, Fe, Mg, Mn, and Zn was evaluated in biological (bovine liver, SRM 1577b, NIST) and botanical (apple leaves, SRM 1515, NIST; trace elements in spinach leaves, SRM 1570a, NIST; and commercial teas) materials. The extraction procedures were investigated using 1 or 10% v/v hydrochloric or nitric acid solutions. An alkaline solution containing a mixture of water-soluble tertiary amines (CFA-C, Spectrasol) was also tested. All procedures were carried out at temperatures in the range 80-105 °C during time intervals shorter than 10 min. The analytes were determined by flame and graphite furnace atomic absorption spectrophotometry (Intralab, model Gemini AA12/1475 and Varian model 800, respectively), and by inductively coupled plasma atomic emission spectrometry (AtomScan 25, TJA). Copper, Mn, and Zn were quantitatively extracted from biological and botanical materials with all solutions employed. The complete extraction of these elements from biological materials was easier than from botanical materials and it was also observed that acid solutions were more efficient than the CFA-C mixture for botanical samples. Calcium and Mg were quantitatively extracted in some tea samples and it was found that Mg is weaker bound to the matrix than Ca. Aluminum and Fe were poorly extracted in all samples. The



© Lyle Wilson

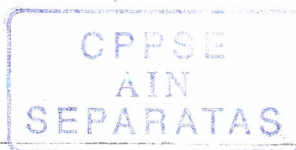
**The 26th Annual Conference of the
Federation of Analytical Chemistry and
Spectroscopy Societies (FACSS)**

45th International Conference on
Analytical Sciences and Spectroscopy (ICASS)

**Final Program
Book of Abstracts**

October 24-29, 1999

**Vancouver Convention and Exhibition Centre
<http://FACSS.org/info.html>**



PROCI-1999.00022
LOM
1999
SP-1999.00022