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Akbar Montaser^a

^a Department of Chemistry, Arya-Mehr University of Technology, Tehran, Iran

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INDUCTIVELY COUPLED PLASMAS AS EXCITATION SOURCES
FOR ATOMIC FLUORESCENCE SPECTROMETRY

Akbar Montaser,
Department of Chemistry
Arya-Mehr University of Technology, Tehran, Iran

ABSTRACT

The inductively coupled Ar plasma (ICP) is introduced as a multielement excitation source for flame atomic fluorescence spectrometry. The ICP-excited and the EDL-excited atomic fluorescence detection limits are compared for 20 elements.

INTRODUCTION

The real and potential advantages of the inductively coupled plasma (ICP) for optical emission^{1,2}, atomic absorption (AAS)^{3,4} and atomic fluorescence (AFS)^{5,6} spec-

trometry have been reviewed recently by Barnes⁷. Using an air-acetylene flame, Greenfield et.al⁴ employed an ICP as an excitation source for the AAS determination of Ca, Mg, and Cu. It was found that there was no significant line broadening due to the high plasma temperature and the half-line widths obtained were comparable with those of the hollow-cathode lamps. In this communication an ICP is introduced as an excitation source for AFS. The ICP possesses potential advantages (intensity, multielement capability) compared to excitation sources such as dye lasers^{8,9}, thermostated microwave excited electrodeless discharge lamp (EDL)^{5,10}, pulsed hollow cathode lamp^{11,12}, and continuum sources¹³. Pulsed or CW tunable dye lasers are the most intense but the most expensive sources and can not be considered as a viable excitation source at the present time. For most elements the design and construction of the next most intense source, the EDL can presently be considered as much art as science. Pulsed hollow cathode lamps and continuum sources lack sufficient intensity. In those laboratories that the ICP experimental facilities are available, the ICP may be considered as an alternative AFS excitation source which possesses great intensity and a high excitation capability for a large number of elements.

EXPERIMENTAL

The components of the ICP-Flame AFS system are described in Table 1. A 10 mm high, 4 mm wide aperture was mounted in front of plasma system to select the desired emitting region. The aperture was followed by a chopper and a cylindrical quartz lens system which subsequently focused the plasma radiation onto a rectangular separated air-acetylene flame. A spherical quartz lens focused the fluorescence radiation onto the 10 mm high entrance slit of the monochromator. Stock solutions of 10,000 $\mu\text{g/ml}$ of elements were prepared from their reagent grade salts using deionized water. All analyte and blank solutions contained 1% nitric acid.

RESULTS AND DISCUSSION

The operating parameters for the ICP excitation source were selected based on Scott et.al.^{14,15} investigations on the line profile of Ca, Sr, and Ar. These authors used a Fabry-Perot interferometer to measure the profiles of spectral lines over wide concentration ranges of the analyte and at different observation heights in the plasma. It was found that for the operating power of 1000 W, self-absorption and self-reversal were absent at

TABLE 1
Experimental System

Component	Description and Supplier
Spectrometer	Model EUE-703-70 AA-AE-AF Spectrophotometer used in conjunction with Model EU-700-32 Controller (GCA/McPherson Instrument Acton, Mass, USA)
ICP Excitation System	Model HFP-2500 D RF generator with a rated power output of 2500 W, 27.12 MHz, Model APCS-1 auto power control, Model AMN-2500E auto matching network, Model PT-2500 plasma torch stand, Model T1.5 torch assembly, Model TN-1 right-angle pneumatic type Teflon nebulizer, and Model SC-2 dual tube aerosol chamber (Plasma Therm, INC., Kresson, N.J., USA)
EDL Excitation System	Model MPG-4-119 microwave generator with a rated output of 120 W, 2450 MHz (Kiva Instrument Co., Rockville, MD., USA) used in conjunction with an A-antenna (Model 2254-5002G1, Sorensen Co., Manchester, N.H., USA)

an observation height of 15-20 mm when the analyte concentration was about 1000 $\mu\text{g/ml}$. Higher concentration or observation height caused these phenomena.

The ICP operating parameters are shown in Table 2. The bottom of the rectangular aperture was placed at the observation height of 10 mm. Therefore, the plasma radiation received by the flame contained the emitting channel located at 10 to 20 mm above the load coil. The plasma forward power was also increased to 1600 W to allow efficient simultaneous excitation of up to 20 ele-

TABLE 2
Experimental Parameters

Generator Forward Power.	1600 W
Generator Reflected Power.	about 4 W
Plasma Gas Flow Rate.	15 L./min of Ar.
Nebulizer Carrier Gas Flow Rate.	1.0 L./min of Ar.
Sample Nebulization Rate	2.0 ml/min
Observation Height	10-20 mm above the load coil
Entrance and Exit Slit Height	10 mm
Entrance and Exit Slit Width	0.3 mm for Cu, Mg and Be 0.5 mm for Ag, Co, Mn, Sn and Tl 1.0 mm for Cd, Pb, Zn, Ca, Ni, Sb, Fe, Se, Sr, Cr, Te, and Hg
Photomultiplier Tube Voltage.	1000 V
Atomic Fluorescence Integration Time.	3 Sec.
Analyte Concentration for ICP	1000 $\mu\text{g/ml}$

ments (1000 $\mu\text{g/ml}$ each) and to eliminate possible self absorption. The selection of 20 elements was based on the observation that prolong aspiration of 20,000 $\mu\text{g/ml}$

TABLE 3
Comparison of the ICP-Excited and EDL-Excited Flame
Atomic Fluorescence Detection Limits

Element	Wavelength, nm	Detection Limits ($\mu\text{g/ml}$)	
		ICP-Excited AFS	EDL-Excited AFS
Ag	328.1	0.008	0.0008
Cd	228.8	0.006	0.001
Cu	324.8	0.008	0.002
Mg	285.2	0.004	0.0007
Pb	405.7	0.04	0.03
Zn	213.8	0.006	0.005
Ca	422.7	0.02	0.02
Co	240.7	0.02	0.01
Mn	279.4	0.008	0.006
Ni	232.0	0.05	0.05
Sb	231.1	0.8	0.05
Sn	303.4	1.0	0.5
Tl	377.6	0.09	0.01
Fe	248.3	0.08	0.02
Be	234.9	0.01	0.01
Se	196.0	0.4	0.1
Sc	460.7	0.02	0.02
Cr	357.9	0.03	0.009
Te	214.3	0.1	0.06
Hg	253.7	0.06	0.02

solution caused deposit formation on the aerosol injection orifice.

Table 3 shows a comparison of the ICP-excited and EDL-excited atomic fluorescence detection limits for 20 elements using the same flame experimental set-up. The thermostated EDL's^{5,10} were operated at their optimum microwave power. The detection limit was defined as the concentration required to give a signal-to-root mean square noise ratio of 2. It can be seen that the EDL-excited AFS detection limits are generally superior to the ICP-excited system by a factor of 2 to 10. Therefore, when the ICP experimental facilities are available in a laboratory, the ICP potential as an intense multi-element excitation source for AFS should be considered.

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