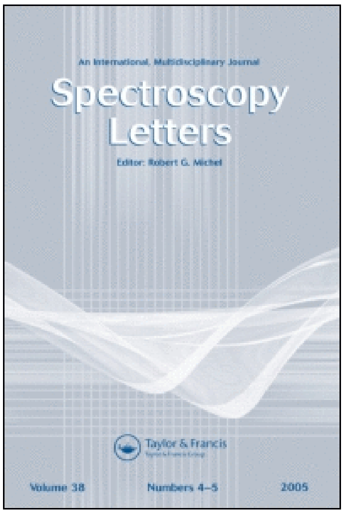

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Use of Organic Solvents as Additives in Inductively Coupled Argon Plasma with Atomic Emission Spectrometric Detection

Maria das Graças A. Korn[a]; Elisabeth de Oliveira[b]
[a] Institute de Química, Universidade Federal da Bahia, Salvador, Brazil [b] Instituto de Química, Universidade de São Paulo, SP, Brazil

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USE OF ORGANIC SOLVENTS AS ADDITIVES IN INDUCTIVELY COUPLED ARGON PLASMA WITH ATOMIC EMISSION SPECTROMETRIC DETECTION

Keywords: Inductively coupled plasma atomic emission spectrometry; Additives; Aqueous/organic medium; Physical properties

Maria das Graças A. Korn[a,*], Elisabeth de Oliveira[b]

[a]Instituto de Química, Universidade Federal da Bahia, CEP 40170-280, Salvador, Bahia, Brazil. [b]Instituto de Química, Universidade de São Paulo, CP 26077, CEP 05599-970, SP, Brazil.

ABSTRACT

The effect of nitric acid/organic aerosols on the emission intensity of metals from the first row transition series, plus Cd and Pb was investigated. The addition of organic solvents to reference solutions diminished the aerosol mean drop size, by reducing the surface tension of the solutions. Correlation between signal intensity and BEC with the main physical characteristics (surface tension and viscosity) of the nitric acid/acetic acid and nitric acid/alcohol solutions was studied. Analytical curves (0 - 2.0 μg ml^{-1}) were evaluated using a matrix matching procedure, and detection limits indicated an improvement (ca 40%) in analytical performance as well as in linearity, sensitivity and precision.

INTRODUCTION

Inductively coupled plasma atomic emission spectrometry (ICP-AES) has been widely recognized as a suitable technique for the determination of trace elements, the particular advantages being the multi-element capability, large dynamic range and effective background correction. However, several problems have been indicated by researchers, e.g., spectral interferences caused by matrix components, nebulizer blockage owing to the high solids content of solutions, or analyte emission enhancement[1,2]. In addition, ICP-AES suffers from a significant drawback because detection sensitivity is sometimes inadequate to comply with requirements for the determination of trace elements in several types of samples. As a means of addressing this problem, solvent extraction has proved to be an effective means of both increasing sensitivity and removing matrix interferences.

The introduction of an organic solution is known to affect plasma stability and excitation conditions, but to what extent depends largely on the properties of the organic solution and on the ICP equipment used. Many workers have reported on the effects of organic solvents in elemental determination employing ICP-AES in the last years[3-10]. A detailed investigation of the nebulization of organic solvents in atomic spectrometry has been reported by Boorn et al.[4]. In that paper, the relative rates of evaporation from aerosol droplets were shown to be critical in determining the transport efficiencies of organic solvents more volatile than water. On comparison of the transport efficiency of an organic solvent of low volatility, such as nitrobenzene, to water, the difference in surface tension was considered to be the dominant effect. In a study by Boorn and Browner[5] the quantitative effects of 30 common organic solvents on analytical signals obtained from a medium power (1.75 kW) argon ICP were studied. The tolerance of an ICP discharge for organic solvents was discussed in terms of the "limiting aspiration rate". A reasonable correlation was found between limiting aspiration rates and evaporation factors for a number of solvents. In general the ICP has decreasing stability as evaporation rates of solvents increase. This indicates that solvent vapor loading is the major factor influencing plasma stability with organic solvent introduction. Maessen and co-workers[11-13] and Weir and Blades[14,15] have described several studies on the mechanisms of organic solvent/plasma interactions. A survey of the literature on the use of organic samples and solutions in ICP-AES reveals that little has been published on the effects of mixed aqueous organic solvents[16-23]. Olesik and Moore[16] showed that both atom and ion emission Sr signals were depressed when samples contained 0.1 - 2.0% organic solvent. McCrindle and Rademeyer[20-22] and Razic and co-workers[23] investigated the effect of an ethanol-loaded sample solution on plasma spectrometric determinations and observed an increase in emission intensity[20] and improved detection limits for various metals[22]. Korn and Oliveira[24] reported a trace mercury determination in 20% acetic acid solution carried out using a matrix-matched calibration standard, and significant improvement could be achieved in terms of linearity and sensitivity.

In this work the effect of aqueous-organic solvent solutions on the emission intensity of metals of the first transition series plus Cd and Pb was investigated. The possibility of using organic solvents as additives in order to improve the analytical performance of the spectrometer was also studied.

EXPERIMENTAL

Apparatus

The ICP used was a Spectro Analytical Instruments Spectroflame Sequential Spectrometer. Instrumentation and operation conditions used are listed in Table 1. Samples were nebulized with the use of a peristaltic pump and three replicate measurements were made.

Surface tension was measured by the ring method using a Du Nouy tensiometer[25], and viscosity was determined with an Ostwald viscosimeter [26].

Reagents

All reagents employed were of analytical grade. Working standards of 1000 μg ml^{-1} were prepared by dissolving salts of metallic ions in nitric acid. The standards used were prepared by appropriate dilutions of the working standards with 1% nitric acid and organic solvents.

Element Transitions

Elements, wavelength and total excitation potentials (with ionization potentials added for ion lines) are listed in Table 2. Both low and high excitation potential lines were chosen specifically to gauge the effects of the aerosol characteristics of the organic solvents on these transitions[27, 28].

RESULTS AND DISCUSSION

Variations in emission intensity due to the presence of small quantities of organic solvents in 1% nitric acid solutions of V, Mn, Fe, Co, Ni, Cr, Cu, Zn, Pb depends on the variation of the transport conditions of the solutions to the plasma. Formation of an aerosol with smaller mean droplet diameter than for 1% nitric acid solutions occurs, leading to a more efficient analyte mass transport and consequently to changes in the excitation characteristics. Five parameters, namely observation height (10, 12 e 14 mm above the load coil), nebulizer pressure (26, 29 and 32 psi) solvent solutions (nitric acid, nitric acid-carboxylic acid, nitric acid-alcohol,

TABLE 1

Instrumental and Operating Conditions

Spectrometer	Monochromator diffraction grating in Paschen-rounge mounting,
R.f. generator	Free-Running, 27.12 MHz. Operating power 1.30 kW
Nebulizer	Glass, Meinhard type TR-30-3A
Ar flow rate	Outer 12.0 l min^{-1}; intermediate 1.0 l min^{-1}
Nebulizer pressure	32 psi
Observation height	12 mm above load coil
Integration time	5 s
Solution uptake rate	1.5 ml min^{-1}

TABLE 2

Elements, Wavelengths and Excitation Potentials

Element	Ionic Line (II)	$Pot_{ion.+exc.}$ (eV)	Atomic Line(I)	$Pot_{.exc.}$ (eV)
Cd	214.438	14.7	228.802*	5.41
Ni	221.647	14.3	232.003*	5.30
Co	231.162	13.5	345.350*	4.02
Fe	239.562	13.0	259.940*	4.77
Mn	257.610	12.2	239.482*	4.43
Cr	267.716	12.9	425.435*	2.91
Pb	220.353*	14.7	283.418	4.40
V	312.528	11.0	437.924*	3.13
Cu	224.700*	15.9	324.754	3.82
Zn	206.191*	15.4	334.856	7.78

Observations: (I) atomic line; (II) ionic line.

* Lines used to study ion/atom ratio

$Pot_{ion.+exc.}$ = ionization + excitation potential ionic lines

Pot_{exc} = excitation potential atomic lines[27,28].

nitric acid-acetone and nitric acid-ether) and concentration of the elements (0, 0.1, 0.5, 1.0 and 2.0 μg ml^{-1}) were optimized using a univariate model in SBR (signal/background ratio) and multivariate data analysis[29].

Effect of the Solvent Nature

The univariate model helps to choose the type and the concentration of the organic solvent mixed with nitric acid. For alcohol, the SBR always increased in the presence of 2% methanol, ethanol, propanol and butanol plus 1% nitric acid, at a given power of 1.3 kW, observation height o f 12 mm above the load coil and two nebulizer pressures (26 and 29 psi). For a higher nebulizer pressure (32 psi), the elements Cu, Pb and Zn showed different behavior, due to the emission of atomic lines and not ionic lines as for the other elements, which presented a different response to the effect of aerosol droplet size and high argon nebulizer pressure, probably due lower residence times of the solution in the plasma and cooling of the plasma. For further studies butanol was chosen because of the enhancement factor obtained for most of the elements studied as shown in figure 1, increase in the raw intensity of the analyte lines and decrease in the intensity of the blank solution. The effect of the variation of butanol concentration can be seen in figure 2 at one plasma condition. For Cd, Ni, Co and Fe, both analyte (Ia) and blank (Ib) intensity increase with higher butanol concentration in order to decrease SBR. For others elements were observed that analyte intensity increase and blank intensity decrease resulting in a higher enhancement factor values when butanol concentration is increased. The emission intensities of analyte lines with different ionization and excitation potential are affected in different way. Emission lines due to high ionization+excitation potential are more subjects to matrix interferences[1]. The solution 1% nitric acid-3% butanol was chosen as the best for

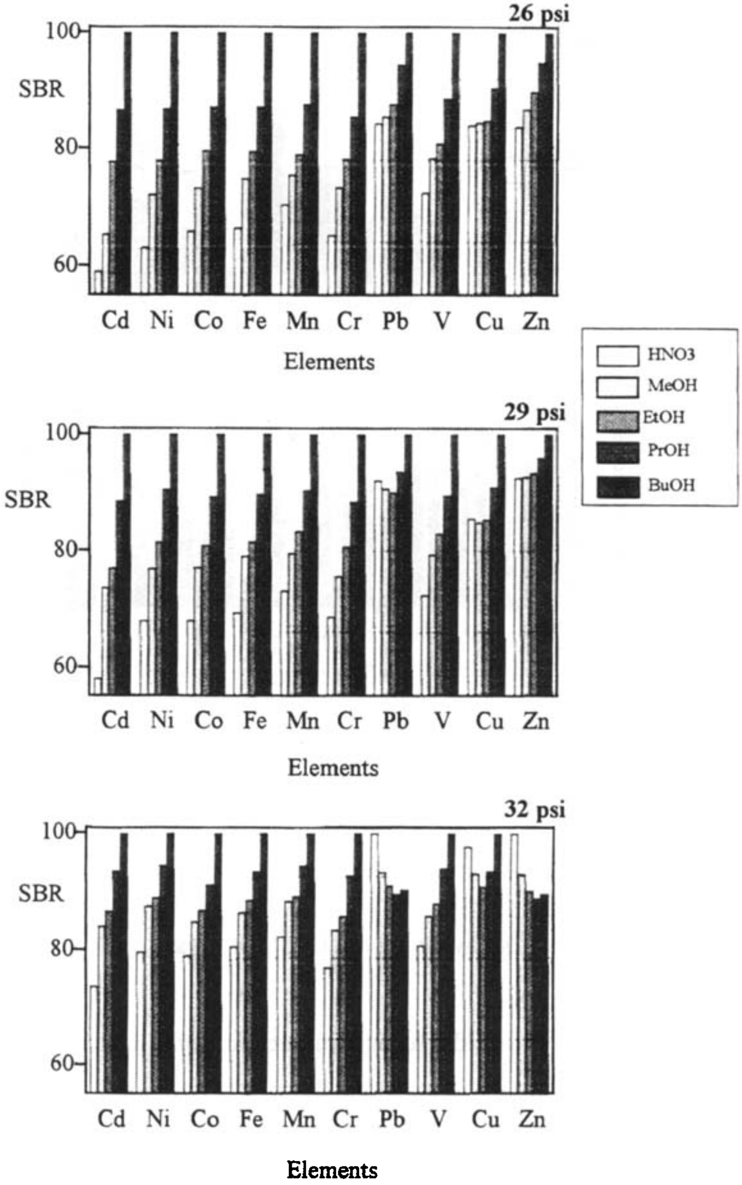

FIG. 1 - Variation of the signal-to-background ratio for aqueous-organic solutions 1% nitric acid, 1% nitric acid-2% methanol, 1% nitric acid-2% ethanol, 1% nitric acid-2% propanol and 1% nitric acid-2% butanol; nebulizer pressure 26, 29 and 32 psi; observation height 12 *mm*; r.f. power 1.30 kW .

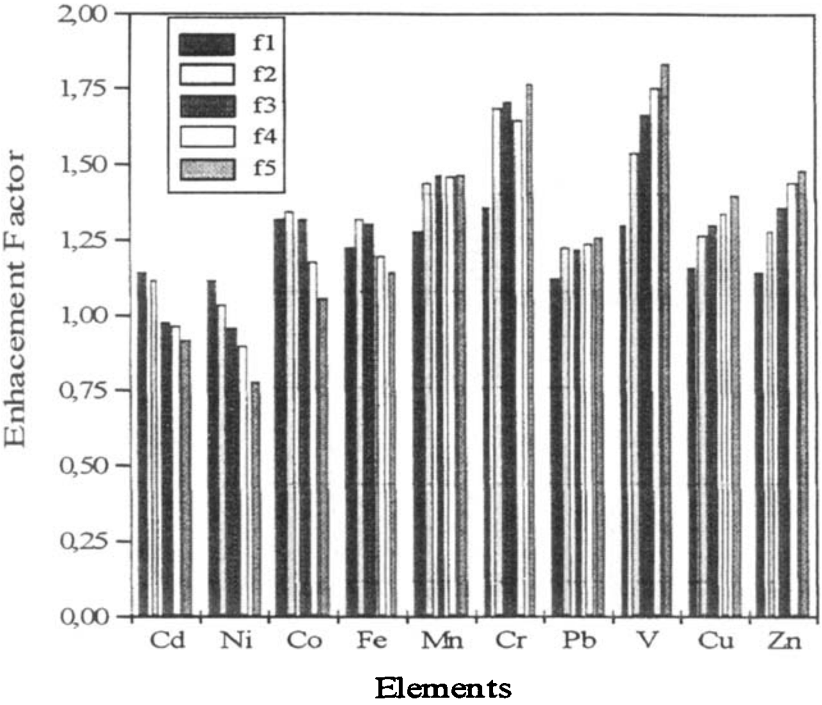

FIG. 2 - Variation of the enhancement factor or the elements (5 μg ml^{-1}), for different butanol concentrations (f1=1%; f2=2%; f3=3%; f4=4% and f5=5%); observation height 12 mm; nebulizer pressure 29 psi .

simultaneous studies, showing a 40±25 % gain in the ration $(SBR_{(BuOH)})/(SBR_{(HNO3)})$.

For the carboxylic acids, formic and acetic acids were studied. For all elements the results were similar to those of the alcohol. The largest SBR corresponds to 20% acetic acid -1% nitric acid as can be seen in figure 3. For concentrations above 20% of acetic acid the plasma was no longer stable, extinguishing at 40%.

The results presented in fig. 1-3 were the mean of three independent determination and the relative standard deviation obtained were within 0.5% for Mn and 2% for Pb.

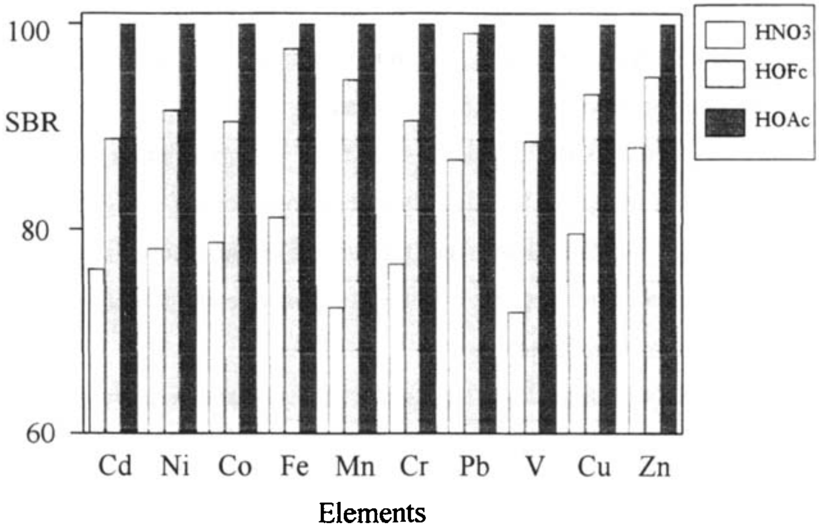

FIG. 3 - Variation of the signal-to-background ratio for aqueous-organic solutions 1% nitric acid, 1% nitric acid-20% formic acid and nitric acid-20% acetic acid ; nebulizer pressure 29 psi; observation height 12 mm; r.f. power 1.30 kW.

The tolerance of the plasma for acetone and ether was 3% and 2% respectively, due to the high proportion of the solvents in the aerosol carried to the plasma, cooling and extinguishing it. The gain obtained for the SBR was 20±8 % in relation to the 1% nitric acid solution, showing no analytical interest.

Influence of the Physical Properties

The emission intensity depends on two major factors: analyte mass transport rate to the plasma and the degree of dissociation, ionization and excitation of the species in the plasma. The analyte mass transport rate depends on the efficiency of the nebulizer and the physical properties of the solvent. The increase in the transport efficiency to the combined nebulizer, spray chamber and torch leads to enhancement of emission intensity and increase in solvent load to the plasma for the aqueous- organic solutions compared with nitric acid solutions.

Table 3 shows the physical properties of each solution employed. From the results it appears that density and viscosity change very little from one solution to another. However, the surface tension shows a significant decrease with the increase of the organic component content in all aqueous-solvent solutions investigated.

In order to obtain high transport efficiency and a decrease in the interferences it is very important that the primary aerosol be as fine as possible, for a given set of gas and liquid flow-rates. Among the physical properties of the solution, surface tension is probably the most important for the formation of the primary aerosol. Owing to the high surface tension of water, many workers have suggested the addition of reagents to the aqueous solutions to decrease the surface tension and hence to improve the transport and/or atomization efficiency[30,31]. A study published by Boorn and Browner[4] showed that the differences in nebulization efficiency are primarily due to surface tension effects when aqueous and organic solvents are compared. The correlation between surface tension and emission intensity of the elements studied in 1% nitric acid, 1% nitric acid -20% acetic acid and 1% nitric acid - 3% butanol are presented in fig. 4. A linear inverse relationship was found between the measured signal intensity for Cd, Co, Cr, Cu, Fe, Mn, Ni and V and surface tension of the solutions and a direct correlation for Pb and Zn, the elements for which atomic lines were used, which differ in behavior from ionic lines, as seen before.

Ionic to Atomic Line Intensity Ratio

The ionic to atomic line intensity ratio is a classic way to measure the atomization and ionization efficiency in an ICP[33]. From the concept

TABLE 3

Physical Properties of Solutions

Solution	Surface Tension $(10^{-3}$dyn.cm$^{-1})$	Density[32] (g cm^{-1})	Viscosity (g m^{-1}s^{-1})
1% Nitric acid	73.3±0.2	0.90	0.87±0.02
1% Formic acid	72.0±0.3	0.92	0.90±0.03
5% Formic acid	68.2±0.1	0.95	0.96±0.01
10% Formic acid	64.0±0.2	1.01	1.12±0.05
20% Formic acid	59.5±0.2	1.3	1.21±0.04
1% Acetic acid	70.5±0.4	0.93	0.92±0.03
5% Acetic acid	62.6±0.2	0.98	1.03±0.04
10% Acetic acid	56.4±0.4	1.02	1.17±0.03
Acetic acid 20%	48.0±0.1	1.04	1.36±0.02
Acetic acid 30%	46.3±0.3	1.05	1.52±0.03
Methanol 2%	63.0±0.2	0.85	0.88±0.01
Ethanol 2%	66.0±0.5	0.87	0.92±0.02
Propanol 2%	59.6±0.4	0.91	0.94±0.01
Butanol 2%	49.0±0.2	0.94	0.970.02
Butanol 3%	42.6±0.2	0.98	1.00±0.05
Butanol 5%	35.9±0.3	1.01	1.05±0.03
Acetone 3%	54.1±0.5	0.93	0.99±0.02
Ether 2%	46.9±0.4	0.92	1.00±0.02

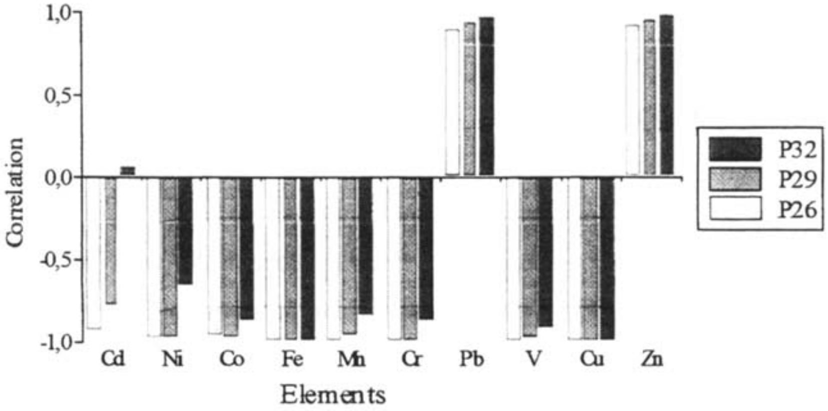

FIG. 4 - Correlation between the net intensity of the elements and the surface tension for 1% nitric acid, 1% nitric acid-20% acetic acid and 1% nitric acid-3% butanol media, for three nebulizer pressures, observation height 12 mm and r.f. power 1.30 kW.

of soft and hard lines[34], the ionic line intensity is highly sensitive to a change in excitation and ionization conditions in contrast to the atomic line intensity. Variation in the atomic line intensity will reflect mostly the change in the aerosol formation and transport efficiency.

The M II/ M I ratio for the elements studied increased in the order: 1% nitric acid < 1% nitric acid-20% acetic acid < 1% nitric acid- 3% butanol solutions. Some results are presented in table 4. One may conclude that the introduction of aqueous-organic solutions into the plasma the vaporization and atomization process must be more complete and energy must be more efficiently transported from the plasma toroid into the aerosol channel.

Sensitivity and Detection Limits

The calculated values, from analytical curves, of the sensitivity and the detection limits are shown in Table 5, indicating an improvement in analytical performance of the ICP-AES used, as well as linearity and sensitivity for all elements in aqueous- organic media.

The precision was evaluated calculating the relative standard deviations (RSDs). The same solutions were measured repeatedly (n=10) and the RSDs values was better for aqueous-organic media (Table 6).

CONCLUSIONS

In this work we studied the effects of nitric acid/organic aerosols on the emission intensity of first row transition series metals, plus cadmium and lead using ICP-AES. Various parameters were changed and the results showed sensitivity enhancement compared with the use of nitric acid alone.

The variation of emission intensities when aqueous-organic solutions are introduced into the plasma is due not only to the change of transport

TABLE 4

Ionic to Atomic Line Intensity Ratio for Different Media. Nebulizer Pressure 29 psi; Observation Height 12 mm; r.f. Power 1.30 kW.

Element	ion/atom ratio		
	HNO_3	HOAc	BuOH
Cd	1.20±0.05	1.42±0.03	1.53±0.04
Ni	2.37±0.04	2.62±0.03	2.85±0.03
Co	6.05±0.15	6.45±0.09	7.13±0.10
Fe	0.76±0.01	0.86±0.03	0.95±0.01
Mn	17.4±0.25	19.3±0.20	23.3±0.30
Cr	8.52±0.04	10.7±0.10	12.4±0.15
Pb	3.24±0.08	3.42±0.06	3.45±0.07
V	5.11±0.10	6.17±0.09	7.15±0.08
Zn	9.64±0.12	9.72±0.11	9.75±0.15

TABLE 5

Analytical Parameters for the Elements. 1% Nitric Acid, 1% Nitric Acid-20% Acetic Acid and 1% Nitric acid- 3% Butanol. Nebulizer Pressure 29 psi. Observation Height 12 mm.

Element	parameters	1% nitric acid	nitric-acetic	nitric-butanol
Cd	Sens. .±s.	2.7±0.2	3.8±0.4	4.6±0.6
	D. L	0.0057	0.0037	0.0034
Ni	Sens. ±s	1.3±0.2	1.8±0.1	1.9±0.3
	D. L.	0.0231	0.0154	0.0150
Co	Sens. ±s	2.3±0.1	3.3±0.3	3.5±0.2
	D. L.	0.0362	0.0224	0.0219
Fe	Sens. ±s	4.0±0.4	5.5±0.2	6.1±0.3
	D. L.	0.0238	0.0146	0.0134
Mn	Sens. ±s	38±3	52±5	56±4
	D. L.	0.0038	0.0025	0.0022
Cr	Sens. ±s	3.4±0.3	4.7±0.3	5.2±0.4
	D. L.	0.0207	0.0122	0.0115
Pb	Sens. ±s	0.21±0.05	0.20±0.04	0.19±0.04
	D. L.	0.0127	0.0120	0.0123
V	Sens. ±s	6.3±0.8	8.9±0.6	9.7±0.5
	D. L.	0.0036	0.0030	0.0031
Cu	Sens. ±s	21±3	29±2	31±2
	D. L.	0.0339	0.0196	0.0182
Zn	Sens. ±s	0.34±0.06	0.33±0.05	0.31±0.04
	D.L.	0.0424	0.0410	0.0405

Sens. = sensitivity ($x10^3$) and D. L. = Detection Limits ($\mu g\ ml^{-1}$)

TABLE 6

Relative Standard Deviations (RSDs) for the Elements. 1% Nitric Acid, 1% Nitric Acid- 20% Acetic Acid and 1% Nitric Acid- 3% Butanol. Nebulizer Pressure 29 psi. Elements Concentration 0.5 μg mL^{-1}. Observation Height 12 mm.

Element	RSD (%)		
	HNO_3	HOAc	BuOH
Cd	1.10	0.80	0.85
Ni	1.05	0.92	0.93
Co	0.95	0.81	0.83
Fe	0.76	0.70	0.71
Mn	0.55	0.49	0.47
Cr	0.66	0.45	0.48
Pb	1.63	1.20	1.15
V	0.63	0.57	0.53
Zn	1.75	1.52	1.53

efficiency related to the surface tension of the solutions, but also to the different conditions in the excitation of the species present in the plasma source. Atomic and ionic lines present different behavior with the variation some of the parameters studied.

The overall analytical performance both of the 1% nitric acid-20% acetic acid and 1% nitric acid-3% butanol media indicates that they may be more successfully used when lower detection limits are desired.

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