



Analysis of Several Elements in a Copper Matrix

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1 Introduction

ICP-AES is a multi-element technique that allows the analysis of nearly all the elements of the periodic table. It is particularly suitable for the determination of trace elements in high salt content solutions. Here we present as an example the analysis of 42 elements in a 10 g/L Copper matrix.

2 Principle

2.1 Technique used

The elemental analysis of solutions was undertaken by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). The sample is nebulized then transferred to an argon plasma. It is decomposed, atomized and ionized whereby the atoms and ions are excited. We measure the intensity of the light emitted when the atoms or ions return to lower levels of energy. Each element emits light at characteristic wavelengths and these lines can be used for quantitative analysis after a calibration.

2.2 Wavelength choice

The choice of the wavelength in a given matrix can be made using the "profile" function, or by using Win-IMAGE, which is rapid semi-quantitative analysis mode using multiple wavelengths. The principle is the same in either case: record the scans of analytes at low concentration, and of the matrix. By superimposing the spectra, we see possible interferences.

2.3 Limit of detection estimation

The limits of detection are calculated using the following formula:

$$\text{LOD} = k \times \text{BEC} \times \text{RSD}_0$$

With:

LOD = limits of detection,
k= 3 for the normal 3-sigma values,
BEC = Background equivalent concentration,
RSD₀ = relative standard deviation of the blank.

To calculate the LOD, a calibration curve is constructed using two points, 0 ppm and 5 ppm, or some concentration where the calibration is linear; this gives the BEC. The RSD₀ is evaluated by running the blank ten times.

3 Sample preparation

Three calibration standards were prepared:

- The blank prepared with a 10,000 mg/L copper solution from Spex Certiprep.
- A standard made using Spex Certiprep's QC 21, QC 7 and six other elements: Te, In, Bi, Ga, Y and Zr prepared with single element solutions of 1000 mg/L, also from Spex Certiprep. The standards concentration was 5 ppm.
- The third standard at 5 ppm was prepared, by dilution with the copper matrix, of single element solutions of 1,000 µg/L made from Nb, Ta, Ge and Au. These solutions are available from Spex Certiprep.

4 Instrument specification

The work was done on a ULTIMA. The specifications of this instrument are listed in Tables 1 and 2.

**Table 1: Specification of spectrometer**

Parameters	Specifications
Mounting	Czerny Turner
Focal length	1 m
Thermoregulation	Yes
Nitrogen purge	Yes
Grating number of grooves	2400 gr/mm
1st order resolution	0.010 nm
2nd order resolution	0.005 nm
Order	2nd order

Table 2: Specification of RF Generator

Parameters	Specifications
Type of generator	Solid state
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	By computer
Control of pump flow	By computer
Cooling	Air

5 Operating conditions

The operating conditions are listed in Table 3 below.

Table 3: Operating conditions

Parameter	Condition
RF Generator power	1200 W
Plasma gas flowrate	12 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer gas flowrate	0.6 L/min
Nebulizer flowrate	3.5 bars (52 psi)
Sample uptake	1 mL/min
Type of nebulizer	Concentric
Type of spray chamber	Cyclonic
Argon humidifier	Yes
Injector tube diameter	3.0 mm

6 Wavelength selection and analytical conditions

The line with the highest sensitivity was used for each of the elements, as there were no interfer-

ence problems. The conditions were the same for all elements, except for the alkalis.

Table 4: Analysis conditions

Element	Slits μm	Analysis Mode	Integration Time (sec)
All elements	20 x 15	Direct Peaking	8
Alkali elements	20 x 15	Gaussian	1

The use of the argon humidifier, the large internal diameter (ID) of the injector tube enabled trouble free analysis, even with high dissolved salts. The larger ID injector tube also ensures a minimization of interferences. Due to the high dissolved salts, an initial conditioning of the spray chamber is advised for maximum stability. It is imperative to use matched standards or standard addition due to the viscosity of the solutions.

7 Discussion

7.1 Calibration curve

Calibration curve was made with two points: 0 and 5 mg/L.

7.2 Results

The results are shown in Table 5 below.

8 Summary

Dilution of a sample is undesirable if the best detection limits are desired. The results in this Application Note show that HORIBA Scientific spectrometers give excellent detection limits and are capable of undertaking an analysis of difficult samples, such as those with high dissolved solids.



Table 5: Results

Element	Wavelength (nm)	BEC (ppm)	LOD ($\mu\text{g/L}$)	LOD (ppm in solid)
P	178.220	133	4.94	0.49
S	181.978	0.481	24.15	2.42
Hg	184.890	0.064	4.19	0.42
Sn	189.989	0.086	3.20	0.32
Tl	190.864	0.282	12.54	1.25
As	193.699	0.167	8.37	0.84
Se	196.090	0.291	9.15	0.92
Mo	202.030	0.043	1.94	0.19
Zn	206.200	0.046	1.86	0.19
W	207.911	1.030	25.20	2.52
Ge	209.426	0.322	23.10	2.31
Cd	214.438	0.039	0.79	0.08
Sb	217.581	0.529	12.15	1.22
Bi	223.061	4.300	96.90	9.69
Ta	226.230	0.631	23.25	2.33
Co	228.610	0.047	1.67	0.17
In	230.606	0.391	10.34	1.03
Ni	232.003	0.091	2.28	0.23
Te	238.578	0.813	34.80	3.48
Au	242.795	0.024	0.92	0.09
B	249.773	0.018	0.60	0.06
Si	251.611	0.112	3.05	0.30
Mn	257.610	0.004	0.21	0.02
Fe	259.940	0.023	1.03	0.10
Cr	267.715	0.024	0.83	0.08
Mg	279.553	0.002	0.08	0.01
Pb	283.306	0.443	22.65	2.27
V	292.402	0.038	2.58	0.26
Ga	294.364	0.258	13.01	1.30
Bi	306.772	1.660	178.50	7.85
Nb	309.418	0.362	51.30	5.13
Be	313.042	0.006	0.16	0.02
Ti	334.941	0.164	11.22	1.12
Ag	338.289	0.114	2.00	0.20
Zr	343.823	0.019	0.31	0.03
Y	371.030	0.004	0.87	0.09
Ca	393.366	0.009	0.30	0.03
Al	396.152	0.107	1.80	0.18
Sr	407.771	0.001	0.02	0.00
Ba	455.400	0.002	0.04	0.00
Na	589.592	0.080	2.24	0.22
Li	670.784	0.019	0.38	0.04
K	766.490	0.122	2.33	0.23

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