



Analysis of 10 g/l Pb

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1 Introduction

Samples containing 10 g/L of Pb were presented to this laboratory for trace determination. Concentrations of the analytes were expected to be less than 100 ppm. This Application Note presents a selection of the best wavelengths for each analyte in this matrix as well as an estimation of the detection limits.

2 Principle

2.1 Technique used

The elemental analysis of these samples was undertaken by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). 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 Limits 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,

RSD_0 = 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 RSD_0 is evaluated by running the blank ten times.

3 Sample preparation

10 g of pure Pb was dissolved with 40 mL of 50% HNO_3 and diluted to 1000 mL with 1 % HNO_3 .

4. Instrument specification

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

Table 2: Specification of spectrometer

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

Table 3: 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	1050 W
Plasma gas flowrate	12 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.15 L/min
Nebulizer gas flowrate	0.8 L/min
Nebulizer flowrate	3 bars (45 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

For each element, the line with the highest sensitivity was used for analysis, because there were no problems with interferences. For all the elements the conditions were the same except for alkaline elements.

Table 4: Analytical conditions

Element	Slits (μm)	Analysis mode	Integration time (sec)
Minor elements	20 x 15	Direct peaking	8
Major elements	20 x 80	Gaussian	0.5

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 advisable for maximum stability. It is imperative to use matched standards or standards additions due to the viscosity of the solutions.

7 Limits of detection

The limits of detection have been calculated using the formula in paragraph 2.3.

Table 5: Limits of detection

Element	Wavelength (nm)	BEC (mg/L)	LOD (mg/kg)
Ag	328.068	0.042	0.10
Al	167.020	0.04	0.35
Al	396.520	0.12	0.36
As	189.042	0.26	0.78
Au	267.795	0.029	0.087
Ba	455.403	0.0024	0.007
Bi	223.061	0.22	0.30
Cd	228.802	0.014	0.04
Co	228.616	0.02	0.03
Cu	324.754	0.032	0.11
Fe	259.940	0.048	0.14
Hg	184.890	0.053	0.16
Hg	194.227	0.26	0.78
K	766.490	0.039	0.12
Ni	221.647	0.066	0.20
Pt	214.423	0.08	0.24
Pt	224.552	0.32	0.96
Pt	265.945	0.10	0.30
Pt	306.471	0.17	0.51
Sb	206.833	0.24	0.72
Se	196.090	0.553	1.6
Te	214.275	0.198	0.6
Te	238.578	0.75	2.25
Tl	190.864	2.45	7
Tl	276.787	0.53	2
Tl	351.924	1.2	4
Sr	407.771	0.0015	0.005
Zn	202.548	0.050	0.15

8 Summary

To achieve the lowest detection limits, dilution is undesirable. The results show the JY spectrometers are able to perform an excellent analysis, even with high dissolved solids. This enables the analysis to be performed to the best detection limits possible.



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