ICP ATOMIC EMISSION SPECTROSCOPY
APPLICATION NOTE 17

Application Note

# Analysis of Metals in 30 g/l Barium Nitrate

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## 1 Introduction

A sample with 30 g/L barium nitrate was presented to this laboratory for trace determination. As there was only one sample to be analyzed, which had a complex matrix, the standard addition method was used for analysis.

## 2 Principle

### *2.1 Technique used*

The elemental analysis of a 30 g/L Barium Nitrate solution 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 Limits of detection estimation*

The limits of detection are calculated using the following formula:

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

With:
LOD = limits of detection,
k= 3 for the normal 3-sigma values,
BEC = Background equivalent concentration,
$\mathrm{RSD}_0$ = 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 $\mathrm{RSD}_0$ is evaluated by running the blank ten times.

## 3 Sample preparation

3.0068 g of sample was weighed and dissolved with 5 mL 69 % $HNO_3$ and diluted to 100 mL using deionized water.

Two spiked samples were prepared for the standard addition method.
Addition 1: 0.1 mL of standard "A" with 25 mL of $Ba(HNO_3)_2$.
Addition 2: 0.2 mL of standard "A" with 25 mL of $Ba(HNO_3)_2$.

Standard "A" contained:
2 g/L of S,.
0.25 g/L of Ca, Fe, Na.

The concentrations of the spiked samples are listed in the Table 1.

**Table 1: Standard Addition concentration**

| Element | Wavelength | Concentration of<br>Unknown sample | | |
|---|---|---|---|---|
| | | | Addition1 | Addition2 |
| Ca | 315.885 | X | X + 1 | X + 2 |
| Fe | 259.940 | X | X + 1 | X + 2 |
| Na | 589.592 | X | X + 1 | X + 2 |
| Sr | 407.771 | X | X +8 | X + 16 |

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## 4. Instrument specification

The work was done on a ULTIMA.The specifications of this instrument are listed below in Table 2 and 3.

**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 4 below.

**Table 4: 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 | 0.8 L/min |
| Nebulizer flowrate | 3 bars (45 psi) |
| Sample uptake | 1 mL/min |
| Type of nebulizer | Parallel |
| Type of spray chamber | Cyclonic |
| Argon humidifier | No |
| 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. The analysis conditions were the same for all elements except the alkali elements.

**Table 5: Analytical conditions**

| Element | Slits (µm) | Analysis mode | Integration time (sec) |
|---|---|---|---|
| All elements | 20 x 15 | Direct peaking | 8 |
| Na | 20 x 80 | Gaussian | 0.5 |

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

## 7 Discussion

### *7.1 Limits of Detection*

The limits of detection were calculated using the formula in paragraph 2.3. They were calculated in µg/kg in the solid sample.

**Table 6: Limits of detection**

| Elements | LOD (µg/kg) |
|---|---|
| Ca | 47 |
| Fe | 3.7 |
| Na | 121 |
| Sr | 29 |

### *7.2 Determination of the unknown sample*

Using the two spiked samples and the unknown, a calibration curve was constructed. The concentrations of the traces in the unknown are given by the intercept of their calibration curves. The results for unknown are given in the table below.

**Table 7: Results**

| Element | Concentration in mg/l | Concentration in mg/kg | Expected Value in mg/kg |
|---|---|---|---|
| Ca | 0.83 | 27.6 | < 100 |
| Fe | 0.09 | 3 | < 50 |
| Na | 0.352 | 11.7 | < 100 |
| Sr | 6.7 | 222.7 | < 500 |

## 8 Summary

To achieve the lowest detection limits, dilution is undesirable. The results show the HORIBA Scientific 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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