Application Note

Analysis of Glass Samples

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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 very suitable for the determination of trace and major elements in the same sample or matrix. This Application Note presents data on the analysis of 9 elements in glass samples.

2 Principle

2.1 Technique used

The elemental analysis of these samples 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.

3 Sample preparation

1 g of glass was dissolved in 20 mL of 20% HF. Heat was applied to dissolve the glass and evaporate to a lower volume. The sample was diluted with 10 mL of 50 % HCl and then with deionized water up to a volume of 100 mL. 0.1 g of H_3BO_3 was added to improve the solubility of fluoride forming elements.
In this sample preparation process, the Si matrix is lost as volatile SiF_6, which reduces the matrix and dissolved solids.

4. Instrument specification

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

Table 1: 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 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

An internal standard (Y II at 371 nm) was used to improve the Ca and Na results. A separate monochromator was employed to allow for simultaneous measurement of the internal standard with the measurement of the analyte.

5 Operating conditions

The operating conditions are listed in Table 3 below.

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Table 3: Operating conditions

Parameter	Condition
RF Generator power	1000 W
Plasma gas flowrate	12 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.3 L/min
Nebulizer gas flowrate	0.6 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.

Table 4: Analytical conditions

Element	Slits (µm)	Analysis mode	Integration time (sec)
All elements	20 x 15	Direct peaking	1

The use of the internal standard for the determination of Ca and Na enhanced the accuracy for both elements.

Table 5: Analytical conditions

Elements	Wavelength (nm)	Background Correction (nm)
Al	308.215	
Ba	233.527	
Ca	422.673	
Fe	259.940	+ 0.063
K	766.490	
Mg	280.270	
Mn	257.610	+ 0.041
Na	589.592	- 0.128
S	182.568	- 0.020
Ti	337.280	

7 Discussion

7.1 Semi-quantitative analysis

The semi-quantitative method allows the determination of 36 elements in less than three minutes. Background correction was used for each element to compensate for matrix or acidity differences. The table gives the results of the semi-quantitative analysis compared to those expected.

Table 6: Semi-quantitative analysis

Element	Expected Concentration (mg/l)	Obtained Concentration (mg/l)
Al	16.4	15.0
B		0.57
Ba		0.17
Ca	119.3	111.0
Fe	3.35	3.05
K	7.88	7.84
Mg	62.6	54.2
Mn		0.15
Na	277.5	251.0
S	4.4	4.9
Sr		0.04
Ti	2.03	1.91
Zn		0.13
Zr		0.06

As an alternative, an option called Win-IMAGE is available, which offers the whole spectrum acquisition within 2 minutes. Whole spectrum acquisition gives the capability to undertake a semi-quantitative analysis, perform a retrospective analysis and analyze multiple wavelengths of an element to improve accuracy.

7.2 Calibration curve

Calibration curves were made with the standards in Table 7 (concentrations are in %).

Table 7: Standard concentrations

	SO_3	BaO	MnO	Fe_2O_3	MgO	Al_2O_3	TiO_2	CaO	Na_2O	K_2O
1	0.436	0.665	0.0023	0.026	2.62	2.22	0.146	6.95	14.04	
2		1.90	0.52	2.25	1.62	2.70		10.91	11.11	0.015
3	0.041	3.41	0.093	1.32	0.56	3.84		13.89	7.2	0.011
4	0.065		0.183	0.104	3.08	0.98	0.014	9.21		1.48
5	0.28					3.30	0.075	8.70	18.90	0.70
6		0.05		0.067	4.07	1.35	0.036		13.56	0.35

7.3 Results

After calibration, sample A was measured continually for 40 min with the results for concentration and %RSD shown in Table 8. Then sample 3 was measured three times (results given in Table 9). The next day Sample 3 was measured again with no re-slope of the calibration curve (results shown in Table 10).

Table 8: Mean concentrations (in %) analyses of Sample A over 40 minutes

Elements	Theoretical Concentration	Measured Concentration	RSD over 40 min
Al	0.45	0.386	1.65
Ba		0.10	0.32
Ca	5.04	5.059	0.55
Fe	0.213	0.22	0.90
K	0.03	0.045	12.7
Mg	4.22	4.2	0.28
Mn	2	2.00	0.53
Na	17.11	17.34	0.24
S	0.024	0.020	20.5
Ti	0.017	0.021	1.52

Table 9: Mean concentration (in %) standard 3

Elements	Theoretical Concentration	Measured Concentration
Al	3.84	3.898
Ba	3.41	3.405
Ca	13.89	13.96
Fe	1.32	1.307
K	0.011	0.0105
Mg	0.56	0.592
Mn	0.093	0.085
Na	7.2	7.13
S	0.041	0.0457
Ti		0.0148

Table 10: Concentration (in %) next day of standard 3

Elements	Theoritical concentration	Without re-slope	With re-slope
Al	3.84	3.995	3.88
Ba	3.41	3.245	3.32
Ca	13.89	13.65	13.85
Fe	1.32	1.284	1.29
K	0.011	0.0058	0.009
Mg	0.56	0.578	0.574
Mn	0.093	0.082	0.079
Na	7.2	6.98	7.17
S	0.041	0.042	0.0393
Ti		0.014	0.014

8 Summary

This application report shows that the ICP-AES is an appropriate technique for the analysis of glass samples due to the very large dynamic range. The high reproducibility of the ULTIMA was shown and enables a high throughput with less time needed for re-slope of the calibration curves. The accuracy for major elements was obtained using the internal standard.

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