Application Note

Very Rapid Analysis of Supply Water, Sludge and Animal Food

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1 Introduction

ICP-OES is a multi-element technique that allows for the fast analysis of more than 75 elements on the Periodic Table in a variety of matrices. This Application Note examines the analysis of six water and animal food samples.

2 Principle

2.1 Technique used

The elemental analysis of the water and animal food samples were 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

The separate samples were dissolved using different concentrations and volumes of acids depending on the sample type. The weights of the samples and the volume and concentrations of acid used are given in Table 1 below.

Table 1: Sample preparation

	Weight	Acid
Chicken food	2.01415 g	250 mL of 5% HCl
Mineral vitamin complement	1.0093 g	500 mL of 2% HCl
Diet food	3.5955 g	100 mL of 5% HCl

4 Instrument specification

The work was undertaken on a ULTIMA and is also applicable in a ULTIMA 2 ICP spectrometer. The specifications of this instrument are listed below.

Table 2: Specification of spectrometer

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

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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 the table below.

Table 4: Operating conditions

Parameters	Specifications
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	2.8 bars (41 psi)
Sample uptake	1 mL/min
Type of nebulizer	Parallel flow
Type of spray chamber	Cyclonic
Argon humidifier	No
Injector tube diameter	3.0 mm

Because the calibration was undertaken in water and the samples have various matrices, a high generator power is used to minimize the matrix effects.

6 Wavelength selection and analytical conditions

The method used is the semi-quantitative method for fast and accurate analysis of 35 elements in less than 2 minutes. The calibration is made with two points, 0 and 5 mg/L for most elements. These standards are prepared in deionized water.

The most sensitive lines for each of the elements were used for analysis and, in some cases, we used two lines because of known interferences.

Table 5: Analytical conditions

Element	Slits (µm)	Analysis mode	Integration time (sec)
All elements	20 x 15	Gauss	0.2 - 0.5

Table 6: Wavelengths and background corrections used

Element	Wavelength (nm)	Background correction (nm)	Integration time (sec)
Ag	328.07	- 0.0379	0.1
Al	308.21	+ 0.0434	0.1
Al	396.15	- 0.0397/ + 0.0399	0.1
As	189.04	- 0.0246	0.3
B	249.77	- 0.0422	0.1
Ba	455.40	- 0.0381	0.1
Be	313.04	- 0.0423	0.1
Bi	223.06	+ 0.0417	0.3
Ca	317.93	- 0.058	0.1
Cd	228.80	+ 0.0501	0.1
Co	228.62	- 0.0398	0.1
Cr	267.72	- 0.0252	0.1
Cu	324.75	+ 0.0335	0.1
Fe	259.94	- 0.0255	0.1
Hg	194.16	- 0.0348/ + 0.0355	0.2
K	766.49	- 0.0458	0.1
Li	670.78	- 0.067	0.1
Mg	279.81	+ 0.0329	0.1
Mn	257.61	- 0.0387	0.1
Mo	202.03	- 0.0423	0.1
Na	589.89	- 0.0424	0.1
Ni	221.65	- 0.0337	0.1
P	178.23	- 0.0231	0.1
Pb	220.35	- 0.0259	0.3
S	181.98	+ 0.0164	0.1
Sb	206.83	- 0.0299	0.3
Se	196.03	- 0.0198	0.3
Si	251.61	- 0.0252	0.1
Sn	189.99	- 0.0221	0.1
Sr	407.77	+ 0.0641	0.1
Ti	337.28	- 0.045	0.1
Tl	190.86	- 0.035	0.3
V	292.40	- 0.024	0.1
V	311.07	+ 0.0439	0.1
W	207.91	- 0.0256	0.1
Zn	213.86	+ 0.041	0.1
Zr	343.82	- 0.0519	0.1

7 Results

In each of the samples 35 elements were analyzed although in the results presented here only the elements that could be compared with certified samples are shown. As the calibration curve was obtained between 0 and 5 ppm, the samples were diluted by 10 when required. Both the diluted and undiluted results are presented.

7.1 Calibration

Table 7: Standards calibration

Name of the standard	Element	Solutions from SPEX	Concen -tration
STDLOW	All elements of interest		0 ppm
STDQC19	As, Be, Ca, Cd, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Ni, Pb, Sb, Se, Sr, Ti, Tl, V, Zn, Zr	QC21-100 PLZR2-2Y	5 ppm
STDQC7	K, Si, Al, B, Ba, Na, Ag	QC7A-100	5 ppm except Si 2.5 and 50 ppm
STDPSW	P, S, W	PLP9-2Y PLS9-2Y PLW9-2Y	5 ppm
STDHg, 10% HNO3	Hg	PLHG4-2Y	1 ppm
STDSn, 10% HCl	Sn	PLSN5-2Y	1 ppm

7.2 Waters

Table 8: Water results

	WATERS				
	19161		24160		
Element	Analysis AAS (µg/L)	Reference (mg/L)	Analysis (mg/L)	Reference ICP-OES (µg/L)	
Al	1.51	1.42 ± 0.41	155.2	132.9	140
As			85.2	84	79
Cd	3.30	3.66 ± 0.44	4.9	5.0	5.1
Cr	2.85	2.93 ± 0.74	24.9*	11.7	10.4
Cu	1.11	1.14 ± 0.12	18.1	< 40	
Fe	5.11	5.25 ± 0.81			
Hg			16.5	46.7	44
Mn	3.23	3.32 ± 0.28			
Mo			53.4	52.0	54
Ni	0.50	0.55 ± 0.12	12.7	10.5	11.0
Pb			107.7	98.8	93
Zn	1.15	1.16 ± 0.09	121.6	108.8	108

*High concentration due to an interference on the left part of the peak.

7.3 Animal feed fodder

Table 9: Results for animal feed

PROVENDER 5918 (2.0577 g in 250mL HCl 5%)			
Element	Sample - not diluted	Sample - 10 fold dilution	Reference
Ca	1.91 g/kg	2.2	2.1 ± 0.5
Cu	8.5 mg/kg		3.9 ± 1.7
Mg	1.40 g/kg	1.58	1.7 ± 0.2
P	1.54 g/kg	1.51	1.7 ± 0.4
Zn	45.8 mg/kg		51.4 ± 7.6

Cu and Zn were not determined in the diluted solution, because the concentration was already low in the original solution.

7.4 Chicken feed

Table 10: Results for chicken feed

Chicken food 5920 (2.0415 g in 250 mL HCl 5%)			
Element	Sample - not diluted	Sample - 10 fold dilution	Reference
Ca	0.84%	1.05%	0.91 ± 0.09
Cu	17.7 mg/kg		14 ± 4
Fe	162 mg/kg		
K	0.68%	0.81%	0.75 ± 0.07
Mg	0.15%	0.16%	0.16 ± 0.02
Mn	85.3 mg/kg		84 ± 8
Na	0.14%	0.16%	0.14 ± 0.02
P	0.56%	0.59%	0.59 ± 0.05

7.5 Diet product and mineral vitamin complement

Table 11: Results for diet product and mineral vitamin complement

	Mineral vitamin complement 2198a (1.0093 g in 500 mL of 2%HCl)		Diet product 401 (3.8955 g in 100 mL of 5%HCl)	
Element	Analysis	Reference	Analysis	Reference
As	0.3 mg/kg (< LD)	4.1 ± 3.7		
Ca	20.84%	23.29 ± 2.33	63 mg/kg	47 ± 5
Cd	0.20 mg/kg	0.23 ± 0.23		
Co	99.03 mg/kg	105.9 ± 37.9		
Cu	3060 mg/kg	3029 ± 389	0.50 mg/kg	0.33 ± 0.20
Fe	9428 mg/kg	12745 ± 1878	2.92 mg/kg	2.9 ± 0.3
K	0.08%	0.12 ± 0.09	391 mg/kg	441 ± 44

Table 11: continued

	Mineral vitamin complement 2198a (1.0093 g in 500 mL of 2%HCl)		Diet product 401 (3.8955 g in 100 mL of 5%HCl)	
Element	Analysis	Reference	Analysis	Reference
Mg	1.02%	1.10 ± 0.11	87.7 mg/kg	93 ± 9
Mn	8812 mg/kg	9636 ± 812	2.02 mg/kg	2.3 ± 0.5
Na	0.26%	0.26 ± 0.09	364 mg/kg	401 ± 40
P			308 mg/kg	314 ± 31
Pb	9.76 mg/kg	5.0 ± 4.9		
S	1.93%	2.0 ± 1.3		
Zn	29294 mg/kg	35891 ± 5892	2.65 mg/kg	2.7 ± 0.3

8 Summary

This application report shows that the ICP-AES is a technique well adapted for the fast analysis of various food samples. The accuracy can be improved by adjusting the analytical conditions to specific sample types. For example, the range of calibration can be adjusted or the calibration standards matrix matched to the samples. As an alternative to the quantitative analysis, the Win-IMAGE is available which offers the whole spectrum acquisition within 2 minutes. Whole spectrum acquisition gives the capacity to make a semi-quantitative analysis, retrospective analysis and easily analyze multiple wavelengths for improved accuracy.

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