



Analysis of Dust Samples

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Keywords: environment

Elements: Cd, Cr, Cu, K, Mo, Na, Ni, Pb, Sb, Sn, Zn, Zr

1 Introduction

This Application Note examines the analysis of dust samples using ICP-OES. There were a number of elements of interest, including the alkalis. ICP-OES is a multi-element technique that allows for the fast analysis of more than 75 elements on the Periodic table.

2 Principle

2.1 Technique used

The elemental analysis of dust 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

$$LOD = k * BEC * RSD_0$$

The limits of detection are calculated using the following formula:

With:

LOD limit of detection,

k: equals to 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 calibration provides the BEC. The RSD₀ is evaluated by running the blank ten times.

3 Sample preparation

Two types of sample preparation were examined. The first method was used for the analysis of Cd, Cr, Cu, Mo, Ni, Pb, Sb, Sn, Zn, and Zr and the second for Na and K.

The composition of the prepared samples was:

Sample 1:

- 2 g/L of dust salt
- 0.2g of fusion salt (H₃BO₃ + K₂CO₃)
- 100mL of 65% HNO₃

Sample 2:

- 0.4 g/L of dust salt
- 50 mL of 37% HCl



4 Instrument specification

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

Table 1: 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
1st order resolution	0.005 nm
2nd order resolution	0.010 nm

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

Parameters	Specifications	
	Sample 1	Sample 2
Generator power	1300 W	1100 W
Plasma gas flowrate	12 L/min	12 L/min
Auxiliary gas flowrate	Not used	Not used
Sheath gas flowrate	0.2 L/min	0.3 L/min
Nebulizer gas flowrate	0.85 L/min	0.85 L/min
Nebulizer flowrate	3.2 bars (47 psi)	3.2 bars (47 psi)
Sample uptake	1 mL/min	1 mL/min
Type of nebulizer	Parallel flow	Parallel flow
Type of spray chamber	Cyclonic	Cyclonic
Argon humidifier	No	No
Injector tube diameter	3.0 mm	3.0 mm

Because the matrix of sample 2 had lower dissolved solids, a lower generator power was used.

6 Wavelength selection and analytical conditions

The most sensitive line for each element was used. In some cases two lines were employed because of known interferences.

Table 4: Analytical conditions

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

Wavelengths and background correction points are shown below in table 5.

Table 5: Wavelengths used for analysis

Element	Wavelength (nm)	Integration time (s)
Cd	228.802	0.5
Cr	267.716	0.5
Cu	324.754	0.5
K	766.490	0.5
Mo	202.030	0.5
Na	589.592	0.5
Ni	221.647	0.5
Pb	220.353	0.5
Sb	206.833	0.5
Sn	189.989	0.5
Zn	213.856	0.2
Zr	349.621	0.5

7 Results

7.1 Calibration

Four calibration standards were prepared using the fusion salt and the acid to matrix match the standards to the samples.



For analysis of sample 1 (all concentrations are in mg/L).

Table 6: Standard Concentrations

Element	Std 0	Std 1	Std 2	Std 3
Cd	0	2		
Cr	0	2	20	
Cu	0	2	20	
Mo	0	2		
Ni	0	2		
Pb	0		20	500
Sb	0	2		
Sn	0	2		
Zn	0		20	500
Zr	0	2		

Element	Blank	Std 1	Std 2	Std 3
K	0		20	
Na	0	2	20	

7.2 Limits of detection

The limits of detection are calculated using the formula in 2.3.

Table 7: Limits of detection

Element	Wavelength (nm)	BEC (mg/L)	RSD Blank (%)	LD ($\mu\text{g/L}$)
Cd	228.802	0.0204	0.23	0.10
Cr	267.716	0.037	0.32	0.40
Cu	324.754	0.052	0.29	0.45
Mo	202.030	0.0158	0.45	0.20
Ni	221.647	0.0206	0.59	0.40
Pb	220.353	0.2359	0.45	3.20
Sb	206.833	0.221	0.54	3.60
Sn	189.989	0.0799	0.63	1.50
Zn	213.856	0.0532	0.44	0.70
Zr	349.621	0.0616	0.28	0.50

Element	Wavelength (nm)	BEC (mg/L)	RSD Blank (%)	LD ($\mu\text{g/L}$)
K	766.490	0.7982	0.60	14.0
Na	589.592	0.364	0.50	5.50

The limits of detection depend on the level of contaminants. For example, either the standards were contaminated with Cu from the deionized water, acid or fusion salt. The contaminated level is estimated to be 0.362 mg/L.

7.3 Analytical results

Table 8: Results

Dust			
	Concentration (mg/L)	RSD (%) on 3 replicates	Expected Concentrations
Cd	2.65	1.1	< 2
Cr	2.97	0.5	3 < Cr < 10
Cu	6.33	1.5	3 < Cu < 10
Mo	0.0797	3.3	< 2*
Ni	0.569	0.8	< 2
Pb	129.3	0.1	40 < Pb < 500
Sb	0.369	1.5	< 2
Sn	1.75	0.95	< 2
Zn	459.8	0.6	40 < Zn < 500
Zr	0.0182	2.2	< 2

Dust			
	Concentration (mg/L)	RSD (%) on 3 replicates	Expected Concentrations
K	12.50	2.2	3 < K < 15
Na	19.50	1.3	3 < Na < 15

We believe that Cd contamination may have occurred. Na is out of the range due to contaminants added during the preparation of the sample.



7.4 Stability test

The sample was measured ten times.

Table 9.1: Results of stability test

	Cd	Cr	Cu	Mo	Ni
	2.5823	2.9108	6.2633	0.0758	0.565
	2.4913	2.9168	6.0706	0.0744	0.5669
	2.7126	2.9013	6.338	0.0797	0.5714
	2.6169	2.9689	6.4002	0.0762	0.5495
	2.7059	2.9818	6.5185	0.0796	0.5682
	2.6771	2.9587	6.3231	0.0824	0.5755
	2.6452	2.9545	6.3928	0.0771	0.5566
	2.6143	2.9861	6.4329	0.0765	0.5728
	2.6385	3.0375	6.2025	0.0774	0.5702
	2.6896	3.0646	6.3647	0.0724	0.5642
RSD (%)	2.5	1.8	2.0	3.7	1.4
Mean value	2.64	2.97	6.33	0.077	0.57

Table 9.2: Results of stability test

	Pb	Sb	Sn	Zn	Zr
	125.87	0.3517	1.6545	437.757	0.0185
	126.97	0.3268	1.6557	453.57	0.0187
	128.37	0.3422	1.7203	456.3194	0.0182
	127.6	0.3821	1.7406	459.1573	0.0179
	132.74	0.3656	1.7027	462.0504	0.0178
	132.49	0.3935	1.8268	456.86	0.0181
	132.39	0.3709	1.7156	460.36	0.0186
	129.65	0.3629	1.7305	439.05	0.0183
	128.3	0.3743	1.748	449.92	0.0177
	129.87	0.371	1.7638	450.28	0.0188
RSD (%)	1.9	5.3	2.9	1.9	2.1
Mean value	129.43	0.36	1.73	452.53	0.018

Table 9.3: Results of stability test

	K	Na
	13.1726	20.1005
	12.9057	20.5959
	13.1669	19.3722
	12.5343	19.7364
	13.116	18.9523
	12.4106	19.5085
	12.486	19.0537
	13.1377	19.526
	11.8138	19.1854
	12.7337	19.2672
	11.9276	19.4772
	12.5840	19.2672
RSD (%)	3.7	2.4
Mean value	12.67	19.50

8 Summary

This application report shows that the ICP-OES is a technique suitable for the fast analysis of dust samples for a variety of elements, including alkali elements. The high content of fusion salt doesn't disturb the plasma due to the high power available. Even with the difficult matrix, the limits of detection are excellent.



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