Application Note

Analysis of Quartz Filters

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1 Introduction

Quartz filters are used to monitor air contamination; they are often placed in work areas where potential specific contamination can occur. The analysis of impurities in quartz filters was undertaken after digestion with HF and HNO_3. The ULTIMA was used because of its high sensitivity.

2 Principle

2.1 Technique used

The elemental analysis of solutions 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

Samples were digested as follows:

One quartz filter was placed in a 25 mL volumetric flask with 4 mL of 40% Hydrofluoric acid (HF) and 2 mL of 69% Nitric acid (HNO_3).

Any heating should be restricted to 55°C to avoid the loss of Si. However, if Si is not required, heating to higher temperatures will ensure all Si is lost and reduces the dissolved solids content of the solutions.

4 Instrument specification

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

Table 1: Specification of spectrometer

Parameters	Specifications
Mounting	Czerny-Turner
Focal length	1 m
Number of grating	2
Grating 1 number of grooves	4 320 gr/mm
Grating 2 number of grooves	2 400 gr/mm
Thermoregulation	Yes
Variable resolution	Yes
Nitrogen purge	Yes

Table 2: Specification of RF Generator

Parameters	Specifications
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	By computer
Control of pump flow	By computer
Cooling	Air

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4 Operating conditions

The operating conditions are listed in Table 3 below.

Table 3: Operating conditions

Parameter	Condition
RF Generator power	1100 W
Plasma gas flowrate	13 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer flowrate	2.8 bars
Sample uptake	1 mL/min
Type of nebulizer	Tangential
Type of spray chamber	Cyclonic in inert material
Argon humidifier	No
Injector tube diameter	3.0 mm

5 Standards

The standards were prepared in the same acid mixture as the samples. The concentrations of the standards, in mg/L, are given in the following table:

Table 4: Standard concentration

	Standard1	Standard 2	Standard 3	Standard 4
Be	0	0.005	0.020	0.050
Cd	0	0.5	1	2
Cr	0	1	8	16
Ni	0	2	16	32
Pb	0	0.5	2	4

6 Wavelength selection and analytical conditions

Mode of calculation: Mode maximum, ideal for traces. Five points measured, one used for calculation.
Entrance slit: 20 µm
Exit slit: 15 µm
Integration time: 4 s and 2.5 s for background correction.

7 Accuracy

Table 5 gives the concentration in µg/l obtained on three samples compared to results expected.

Table 5: Results for accuracy

Element	Sample 1 Obtained/Expected		Sample2 Obtained/Expected		Sample 3 Obtained/Expected	
Be	0.39	0.40	44.5	48	0.61	0.60
Cd	11.9	12	157	160	1972	2000
Cr	121	120	1857	2000	182.5	200
Ni	192	240	23732	24000	1885	2000
Pb	42.8	40	3593	3600	362	360

8 Conclusion

For the analysis of low levels, it is important to get accurate "blank" values for environmental air samples. For environmental air sampling every batch of silica filters must be analyzed. The results show the methodology used and the instrumentation perform this task very well.

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