



## Analysis of Quartz Filters

Agnès Cosnier  
Jobin Yvon S.A.S., Horiba Group  
Longjumeau, France

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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<sub>3</sub>. The JY 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 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.

### 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<sub>3</sub>).

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 JY 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



## 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**

	Standard 1	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  $\mu\text{m}$

**Exit slit:** 15  $\mu\text{m}$

**Integration time:** 4 s and 2.5 s for background correction.

## 7 Accuracy

Table 5 gives the concentration in  $\mu\text{g/l}$  obtained on three samples compared to results expected.

**Table 5: Results for accuracy**

Element	Sample 1		Sample 2		Sample 3	
	Obtained	Expected	Obtained	Expected	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.



In the USA:  
Jobin Yvon Inc.  
3880 Park Avenue  
Edison, NJ 08820  
Tel: 1-732-494-8660  
Fax: 1-732-494-8796  
E-mail:  
emission@jyhoriba.com

In France:  
Jobin Yvon S.A.S.  
16-18, rue du Canal  
91165 Longjumeau Cedex  
Tel: (33) 1/64 54 13 00  
Fax: (33) 1/69 09 90 88

Germany: (49) 89/46 23 17-0  
Italy: (39) 2/57 60 56 90  
U.K.: (44) 20/82 04 81 42

In Japan:  
Horiba Ltd.  
2 Miyano Higashi, Kisshoin  
Minami-ku, Kyoto 601-8510  
TEL: (81) 75 313 8121  
FAX: (81) 75 321 5725  
www.horiba.com

China: (86) 10/6836 6542  
Spain: (34) 91/724 16 57  
Other Countries: Contact JY S.A.S.

1-877-JYHORIBA

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