

## Quantitative analysis of quartz and cristobalite by X-Ray Diffraction in ambient samples

Sandra Amore, María Mabel Puelles, Rodrigo Alvarez and Mónica Borinsky

INTI-QUIMICA Instituto Nacional de Tecnología Industrial-Centro de Química  
Avenida General Paz 5445, B1650WAB San Martín, Buenos Aires, Argentina  
samore@inti.gob.ar;puelles@inti.gob.ar

### Introduction

Actually, one of the most important problems in workplace health is respirable crystalline silica (quartz and cristobalite) dust exposure of workers, because they may produce silicosis.

The Occupational Safety and Health Administration (OSHA) has established Permissible Exposure Limit (PEL): 0.1 mg/m<sup>3</sup> for quartz and 0.05 mg/m<sup>3</sup> for cristobalite.

This actual regulation impose the necessity of choose a suitable quantification method capable of determine low concentrations of both forms of crystalline silica from samples collected in workplaces atmospheres.

### Experimental

• **Equipment:** Phillips PW1730/10, X-Ray diffractometer. Copper target tube, settled at 40 kV and 40 mA.

• **Sampling:** Samples are collected with a pump in a PVC filter using a Cyclone, with allow to capture less than 10 µm diameter dust, during four to eight hours (flow rate: 1.7 l/min).

• **Standard Reference Material (SRM):** Respirable quartz (NIST 1878<sup>®</sup>) and respirable cristobalite (NIST 1879<sup>®</sup>).

• **Standard preparation:** A series of stock standards is prepared weighting the SRM and suspending them in 2-propanol. The working standards are prepared taking aliquots of the stock standards and transferring them in silver membranes filters using a vacuum filtering apparatus. The deposition is fixed to the membrane and **checked its uniform (es gramaticalmente correcto?)**

Stocks (mg/ml)	Aliquots (ml)	Working standards (mg)
200	5, 10, 15	1.000, 2.000, 3.000
50	2, 5, 10	100, 250, 500
10	1, 2, 5	10*, 20*, 50

\*These standards are omitted for cristobalite analysis.

• **Sample preparation:** The PVC filter with the respirable air collected is dissolved in THF. The dust suspended is quantitatively transferred to a silver membrane.

• **Working analytical parameters:**

Quartz	Peak (°2θ)
Primary	26.85
Secondary	21.05
Tertiary	50.23
Quaternary	60.10
Silver Primary	38.31

Cristobalite	Peak (°2θ)
Primary	21.98
Secondary	36.03
Tertiary	31.46
Silver Primary	38.09

### Conclusion

This method shows several advantages:

- \* It is reliable for selective quantification of both crystalline silica species, which is impossible with other techniques.
- \* It is more specific and sensitive than traditional techniques
- \* Sample preparation and analysis are simple and quick.
- \* It is a non destructive method.
- \* The interferences can be minimized measuring alternative diffraction angles of the analyte.

• **Analytical procedure:** The standards are measured and the area under the peak is used to construct the calibration curve for each peak taking into account background correction. Relative Intensity vs. Relative Concentration is calculated using the silver of the filter as internal standard:

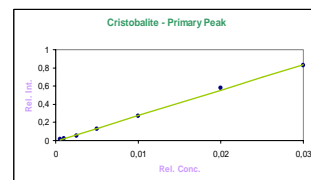
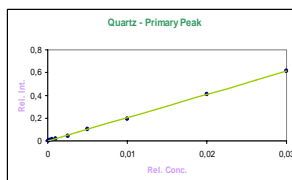
$$\text{Relative Intensity} = \frac{\text{Analyte Intensity}}{\text{Ag Intensity}}$$

$$\text{Relative Concentration} = \frac{\text{Analyte Concentration}}{\text{Ag Concentration}}$$

The samples are scanned for interferences inspection. The samples are measured and the micrograms amount of quartz and cristobalite are calculated for each line using the calibration curve.

### Results

The calibration curves obtained present good correlation coefficients. The curves of Quartz primary peak and Cristobalite primary peak are plotted as examples:



### Calculations:

The PEL is determinated as follows:

$$\% \text{ Respirable Analyte} = \frac{\text{weight of analyte (mg)} \times 100}{\text{total air sample weight (mg)}}$$

$$\text{PEL for Respirable dust containing quartz (mg/m}^3\text{)} = \frac{10 \text{ mg/m}^3}{2 + \% \text{ Respirable Quartz}}$$

$$\text{PEL for Respirable dust containing cristobalite (mg/m}^3\text{)} = \frac{1/2 \times 10 \text{ mg/m}^3}{2 + \% \text{ Resp. Cristob.}}$$

The results are usually reported as mg/m<sup>3</sup>:

$$\text{Analyte concentration} = \frac{\text{total analyte weight (mg)}}{\text{total air volume (m}^3\text{)}}$$

This value can be compared with the regulated limit.