XRD DETERMINATION OF RESPIRABLE CRYSTALLINE SILICA IN ENVIRONMENTAL SAMPLES

MP. Gómez[(1)], E. Zumaquero[(1)], A. Escrig[(1)], E. Monfort[(1)]
A. Salomoni[(2)], F. Cremonini[(2)]

[(1)]Instituto de Tecnología Cerámica (ITC). Asociación de Investigación de las Industrias Cerámicas (AICE)
Universitat Jaume I. Castellón. Spain.
[(2)]Centro Ceramico di Bologna. Bologna. Italy.

1. INTRODUCTION

In the traditional ceramic industries, quartz is used as a raw material in most compositions, either as a natural clay constituent or added as a separate raw material, where it plays a key role in all manufacturing process stages (control of plasticity, green mechanical strength, firing shrinkage, etc.). It has therefore been common practice for years to evaluate occupational exposure to respirable crystalline silica (RCS) in certain workplaces in this type of industry.

However, the classification in 1997 by the International Agency for Research on Cancer (IARC) of some forms of RCS as "carcinogen category 1" has led to review of certain aspects related to occupational protection in regard to free silica. Thus, in 2003, the European Commission addressed harmonisation of the exposure limits to free silica, proposing a value of 0.05 mg/m^3 as daily exposure limit, which is 50% of the value currently in force in Spain. This proposed reduction of the RCS Exposure Limits makes it necessary to use increasingly sensitive analysis methods.

In view of the above, a study has been undertaken to compare the results obtained by two specialised laboratories in the study of ceramic materials, where different methods are used for the determination of RCS, focusing particularly on the determination of the detection limits and quantification, as well as analysis, of samples taken in industrial environments.

This poster presents the results of two interlaboratory exercises devoted to the determination of RCS by X-ray diffraction (XRD) (a widespread analytical technique), carried out by the Instituto de Tecnología Cerámica (ITC) and Centro Ceramico di Bologna (CCB). Calibration curves were made with two different methods.

2. MATERIALS AND METHOD

At ITC the calibration curves were constructed according to the method described in standard NIOSH 7500, however, using cellulose filters, and the certified reference material BCR-066 Quartz. On the other hand, CCB prepared the filters by aeolian deposition, and used a reference quartz (NW12).

In both cases, RCS was determined by XRD without any pre-treatment of the filters before the analytical determination. The measurement conditions are detailed in Table 1 and the calibration curves are shown in Figures 1 and 2.

LABORATORY	ANODE	STEP SIZE	STEP TIME	DETECTOR
ITC	Cu	0.015º (2σ)	5 s	Position-sensitive solid state
CCB	Cu	0.030º (2σ)	15s	Scintillation

Table 1. XRD measurement conditions

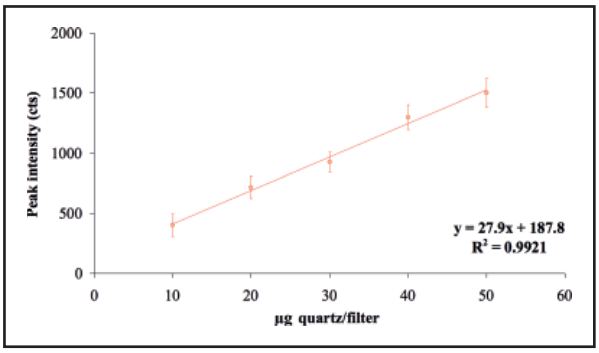

Figure 1. CCB calibration curve

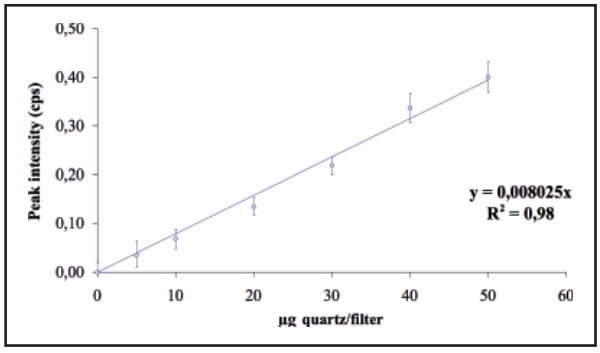

Figure 2. ITC calibration curve

Two series of samples were used in the study: one series was obtained by personal sampling in industrial environments and the other consisted of synthetic samples prepared with standard quartz. The results obtained at both laboratories were compared and analysed.

3. RESULTS AND DISCUSSION

The detection limits and uncertainties associated with the determination of RCS in environmental samples on filters were determined and discussed in the study. Good correlation was observed between the results obtained at both laboratories, as Figure 3 shows. These results will be discussed in greater detail in the poster presentation.

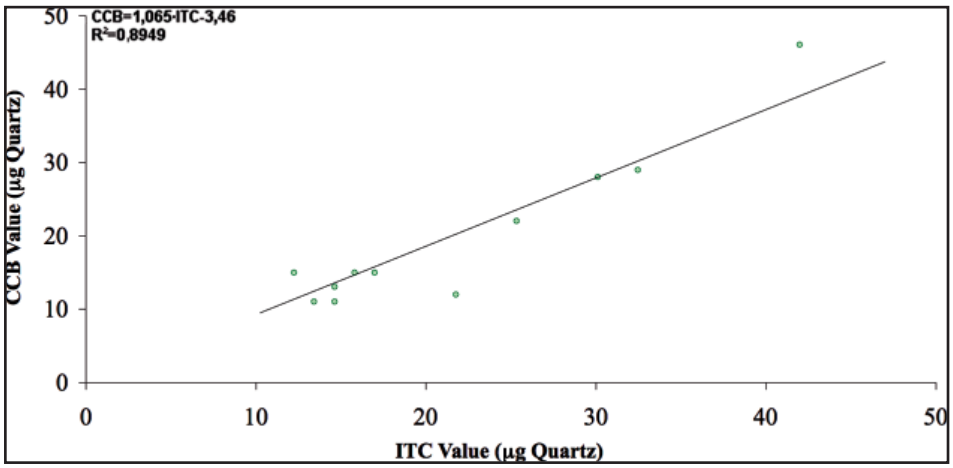

Figure 3. Intercomparison between the ITC and CCB laboratories

4. ACKNOWLEDGEMENTS

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