

## Poster Sessions

The classification, in 1997, by the International Agency for Research on Cancer (IARC) of certain forms of respirable crystalline silica (RCS) [1] as a category 1 carcinogen has led to the revision of a number of aspects relating to occupational protection against free silica. Thus, in 2003, the European Commission addressed the harmonisation of RCS exposure limits, proposing a value of 0.05 mg/m<sup>3</sup> as daily occupational exposure limit, which is 50% of the value currently in force in Spain. The reduction of the RCS occupational exposure limits makes it necessary to use increasingly sensitive analysis methodologies.

In this study, different analysis methodologies [2] were reviewed and a new methodology is proposed, using CIP 10-R personal samplers [3] and polyurethane foams, which allow the captured amount of powder to be increased with a flow rate up to 10 L/min, thus improving the detection and quantification limits.

The new methodology was validated in accordance with quality standard UNE-EN ISO/IEC 17025.

The proposed new methodology, using a CIP 10-R sampler, provides acceptable recovery and repeatability values. The methodology enables the detection limits attained by current methodologies (cyclone-type personal samplers with a 2 L/min flow rate) to be reduced to one fifth

The certified reference materials used to determine the traceability of the method were standard powders BCR-66 and SRM 1878a, and the standard powder deposited on filters SRM 2551-2557.

[1] The measurement of the exposure of workers to respirable crystalline silica (RCS) and the work of the international standard organisation (ISO). Working group TC146/SC2/WG7 Silica. P.R. Stacey IOHA **2005** [2] W.J. Miles, *American Industrial Hygiene Association Journal*, **1999**, *60* (3), 396-402. [3] T. Lee, S. Won Kim, W. P. Chisholm, J. Slaven, M. Harper. *Ann. Occup. Hyg.*, **2010**, *54* (6), 697-709.

**Keywords: respirable crystalline silica (RCS), CIP 10-R, analytical methodology**

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#### An study of the evolution of the NSA fertilizers with humidity

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NSA fertilizers are formed mainly by the 2AN·AS and 3AN·AS double salts (AN: ammonium nitrate; AS: ammonium sulphate) [1], mixed in a determined range of percentages. But it is known that the 3AN·AS salt is meta-stable and evolves into the 2AN·AS salt [2]. Even though it is known that the evolution is catalyzed by the presence of humidity in the environment, this effect was not studied in deep.

In this work we analyze the evolution of the 3AN·AS salt in different humidity conditions in the presence of free AS salt. We analyze also the evolution of mixtures of AS and AN, in different proportions and in different humidities conditions. This work is an extension of a previous work, started sometime ago [3].

Results show the influence of the humidity conditions, the presence of free AS, the time of the experiment, and the initial amount of both double salts in the samples.

Analyses were made using XRPD data collected in an Agilent Nova diffractometer, with a CCD area detectors, and Rietveld fit. Acknowledgements: Spanish MICINN (MAT2006-01997, MAT2010-15094 and CSD2006-015, Consolider Ingenio 2010, "Factoría de Cristalización") financial support and FEDER funding

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[1] J.M. Montejó-Bernardo, S. García-Granda, A. Fernández-González. *Acta Cryst. B.* **2010**, *66*, 358-365. [2] T. Y. Ling, C. K. Chan. *Environ. Sci. Technol.* **2007**, *41*, 8077-8083. [3] J.M. Montejó-Bernardo, S. García-Granda. *Book of abstract of the 2<sup>nd</sup> MISCA 2010*, 126. Oviedo (Asturias, Spain).

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#### Crystalline structure identification by TEM with Microdiffraction, PED and CBED

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Microdiffraction and Convergent Beam Electron Diffraction (CBED) are two techniques used in TEM to identify respectively the space and the point group of a crystal. One of the interests is to work with a small beam to overcome problems regularly met with the selected area diffraction such as the curvatures, faults and thicknesses variations.

Microdiffraction requires a slightly convergent beam with a nanometers probe size and this approach enables the Ewald sphere to probe several strata of the reciprocal lattice. Thus, when the sample is perfectly oriented in a zone axis, Microdiffraction patterns give access to ideal symmetries (position and intensity of diffracted beams), the gaps and the differences of periodicity between the Zero and the First Order Laue Zone. From this method, the space group of a crystalline sample is determined by identifying the Bravais lattice, the presence of glide mirrors and helicoidals axes depicted on some mains patterns [1].

Nevertheless, electron diffraction patterns are highly affected by dynamical effects due to the strong interaction of electrons with matter. Precession Electron Diffraction (PED) is a recent technique perfectly adapted to obtain more kinematical diffraction patterns by using a rocking beam illumination. Diffraction patterns obtained with a de-scanned precession of the incident beam contain as well more reflections and this allows to identify surely and easily the Bravais lattice and the glide mirrors [2].

CBED used with the Buxton or Tanaka (multibeam) methods allows to appreciate the symmetries inside the diffracted and incident disks of a slightly away from a perfect zone axis pattern. By comparing it with simulated patterns, the identification of the point group is then possible [3].

Both techniques were used to identify the space group P6<sub>3</sub>/mmc and the point group mmm of Ti<sub>3</sub>SiC<sub>2</sub> and TiSi<sub>2</sub> respectively, two crystalline phases found by microanalysis (EELS, EDX and STEM HAADF). On one hand, CBED was very useful to find the point group mmm in working with only one zone axis. On the other hand, the space group Fddd has been determined in the same zone axis by Microdiffraction and PED.



Fig.1: Microdiffraction

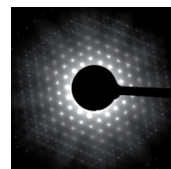


Fig.2: Precession



Fig.3: CBED Multibeam