

APD2000 PRO

Silica Dust Quantification: Calibration Curves



Introduction

Silica is one of the most common minerals in the earth crust and it can be found in three crystalline forms (polymorphs): quartz, cristobalite and tridymite.

Nowadays, it is well known that crystalline silica dust inhalation can cause both silicosis and lung cancer, being a health hazard especially for mining, construction and foundry workers.

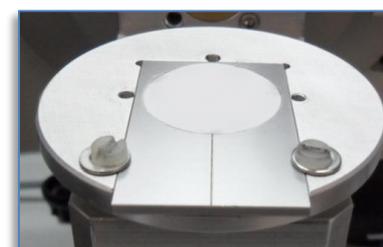
In order to prevent occurrence of pathologies, several national and international norms regulate the occupational exposure to silica dust¹, by defining procedures for sampling airborne particles on filters and their concentration estimation.

X-Ray Diffraction (XRD) is a non-destructive technique particularly effective for quantification of silica dust concentration on filters: in fact, on the one hand each polymorph can be investigated and quantified and on the other hand potential occurrence of interfering crystalline mineral phases is detected.

Quantification of unknown silica dust on filters is achieved by means of calibration curves, determined by using reference samples with a known silica concentration.

Following the UNICHIM 2398 method, our partner lab (Analitica S.A.S.²) deposited several known amounts of quartz on silver filters, which were mounted on an APD2000 diffractometer, equipped with a dedicated sample holder and spinner.

Calibration curve was then built for quartz (101) peak by a dedicated software.



Summary

Crystalline silica dust inhalation can cause lung cancer: X-Ray Diffraction is an effective technique to monitor its concentration in workplaces where the related health hazard is high. In order to quantify the concentration of silica dust, calibration curves with reference samples were determined by using APD 2000 PRO, equipped with spinner, monochromator and a dedicated sample holder for filters.

¹ NIOSH 7500, OSHA ID 143, MDHS 14/3, UNICHIM 2398

² <http://www.analitica-lab.it/lang1/>

Product Specifications

APD 2000 PRO is a Theta / 2Theta powder diffractometer equipped with a large choice of accessories, which make it versatile to cope with various requirements of an academic or industrial laboratory for pharmaceutical, mineral and environmental analysis.



Sample Preparation

Samples were prepared by our partner lab Analitica S.A.S., following UNICHIM 2398 method.

Different amounts of quartz (BCR066³) were deposited on silver filters with a diameter of 25 mm and porosity of 0.8 μm by means of a dust cloud chamber. Weighing was performed with a microbalance (1 μg resolution).

Silver filters were chosen as an aid to correct the matrix absorption effect and to reduce the background.

Goniometer radius [mm]:	175	Secondary monochromator:	Graphite
X-Ray Source:	Cu LFF	Detector:	Point scintillation detector
Power [kV; mA]:	40;40	Step[°]:	0.02
Divergent slit[°]:	1	Time/step [s]:	8
Anti-scatter slit[°]:	1	Measurement range[°]:	26.1÷27.1
Soller slit (x 2) [°]:	2.3	Time/measure [m]:	7.3
Receiving slit [mm]:	0.2	Number of samples:	8
Spinner [rpm]:	90	Mass range [μg]:	3÷114

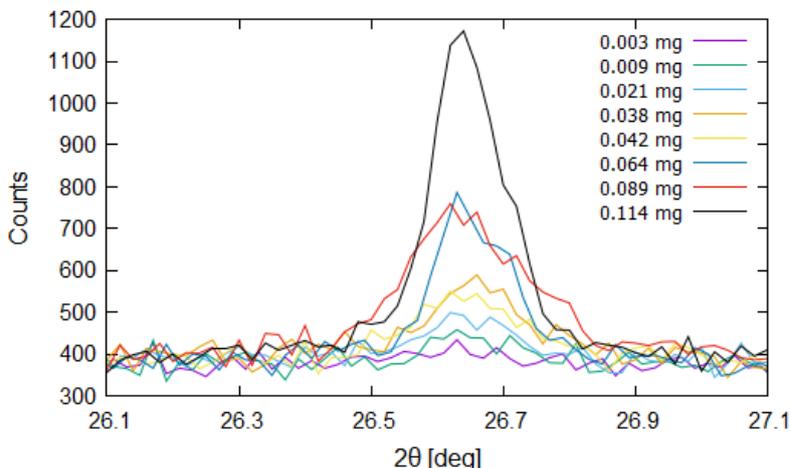
³ https://ec.europa.eu/jrc/sites/default/files/rm/BCR-066_report.pdf

Results

The quartz (101) peak is reported on the right for different silica dust mass values on filters.

Peak area is calculated by integration after background removal, according to method described above; each filter was measured 3 times and then the average area value was computed.

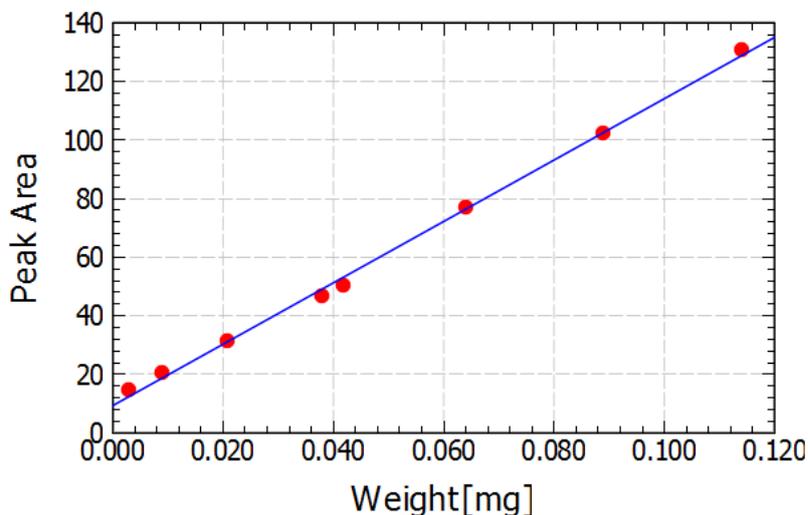
Calibration curve is reported below with estimated Limit Of Detection (LOD) (95% confidence interval).



$$y = 1048.74x + 9.28$$

$$R^2 = 0.998$$

(LOD)= 0.004 mg (4 μg)



Conclusions

The calibration curve for crystalline silica dust (quartz) was determined by analyzing known quartz amounts on Ag filters with APD2000 PRO diffractometer, equipped with spinner and secondary monochromator.

The resulting Limit Of Detection with these settings is 4 μg, which can be further reduced by proper adjustment of measurement parameters.

About GNR SRL

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GNR can rely on a well-established team of highly qualified researchers and technicians, supported by the cooperation with leading University departments, which ensures a constantly updated technological growth.

GNR is present on the main international markets through an efficient and motivated technical and commercial network, able to provide outstanding support for any customer requirements.