

X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD for Monitoring Silica Dust Exposure

Application Report 42

Lung cancer and other health problems are known to be associated with occupational exposure to respirable crystalline silica, SiO_2 . This makes silica dust, or free silica, the second leading cause of occupational cancer after asbestos. Silica is a typical component of soil and rocks, but is also found in foundry materials or concrete. Clear exposure-response relationships have been reported, for example, for miners, diatomaceous earth and construction workers, granite, pottery, refractory brick or foundry workers.

Occupational exposure to respirable silica is a preventable health hazard. Exposure limits have been established by several national authorities and therefore silica concentrations should be monitored. The procedure for sampling airborne particles on filters and testing them with X-rays is governed by national standards such as NIOSH 7500, OSHA ID-142, MSHA P-2, and others.

The D6 PHASER can distinguish between the polymorphs of crystalline silica (quartz, cristobalite and tridymite) and can be calibrated to provide the concentration using appropriate standards. Detection and quantification limits are pushed to the limit by using up to 1,200 W of X-ray power in combination with high flux Soller collimators and Dynamic Beam Optimization. This results in the highest sensitivity of the measurement method combined with a short analysis time (Figure 1).

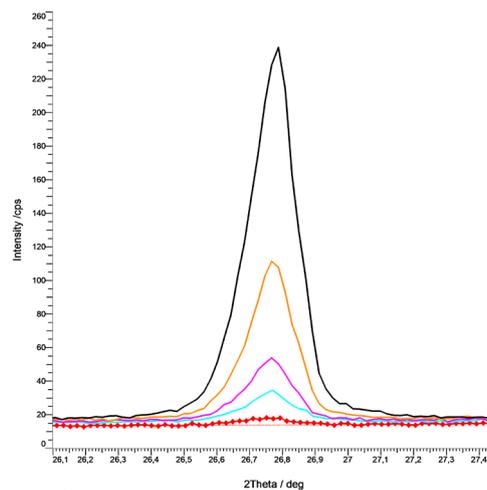


Figure 1

The X-ray intensity changes with the silica concentration. Here 100, 50, 20, 10 and 5 μg samples of a NIST standard (measured at 1.5 min per quartz peak scan) are shown. Five μg (red curve with symbols) can already be separated from the background (dotted line). NIOSH 7500 requests a detection limit of 20 μg .



Figure 2
D6 PHASER equipped for Dynamic Beam Optimization (DBO) with Variable Divergence Slit (VDS), Motorized Air Scatter Screen (MASS) and LYNXEYE XE-T detector.

The DIFFRAC.DQUANT software is used to perform standard-compliant drift-corrected calibrations, evaluate unknowns, and report results directly to the laboratory's LIMS system or internal instrument database. It fully supports absorbance correction as required by NIOSH 7500 or segment calibrations as required by MSHA.

The intensities were determined using the peak areas of the analytical profile refinement in DQUANT v2. The $K\alpha$ 1 and 2 doublet was included in the simultaneous refinement of a split pseudo-Voigt function (to account for peak asymmetry) and a linear background was used. Outlier samples in the calibration were eliminated by multiple preparations of the same standards. Absorption correction was not applied in this example as it affects higher concentrations, while the purpose of this study is to establish the limit of quantification (LOQ) that can be achieved with a D6 PHASER.

The resulting calibration (Figure 3) fully meets the requirements of the NIOSH standard. The curve has a zero offset of 0.4 μg (± 5 μg is allowed). The limit of quantification (LOQ) in this example is approximately 5 μg . Theoretically, it could be further reduced by increasing the measurement time. However, given the extremely small amount of material deposited on the filter, sampling errors are likely to be higher than the counting statistics would indicate.

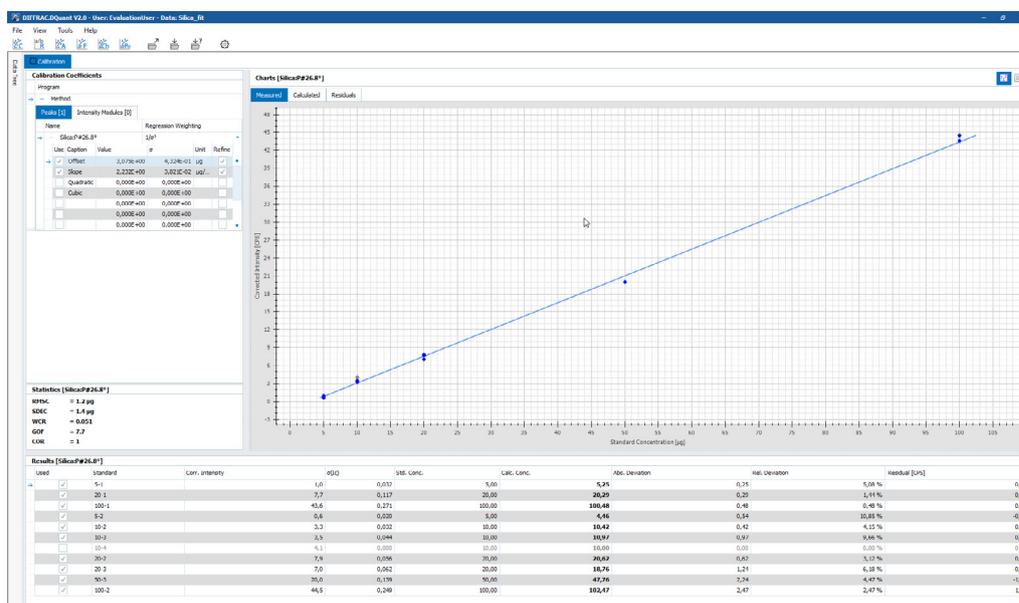


Figure 3
The calibration in DIFFRAC.DQUANT shows high linearity. The limit of quantification estimated according to DIN and IUPAC norms is about 5 μg .

Data were collected with a D6 PHASER 1,2kW, Cu radiation, no K-beta filter, 4° high X-ray flux Soller collimators, primary variable divergence slit (constant illumination mode, 15 mm wide), motorized air scatter screen. The silica peak was measured with scan range 25.741 to 27.433° 2Theta, total scan time 96 sec with the LYNXEYE XE-T detector in high resolution mode.

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