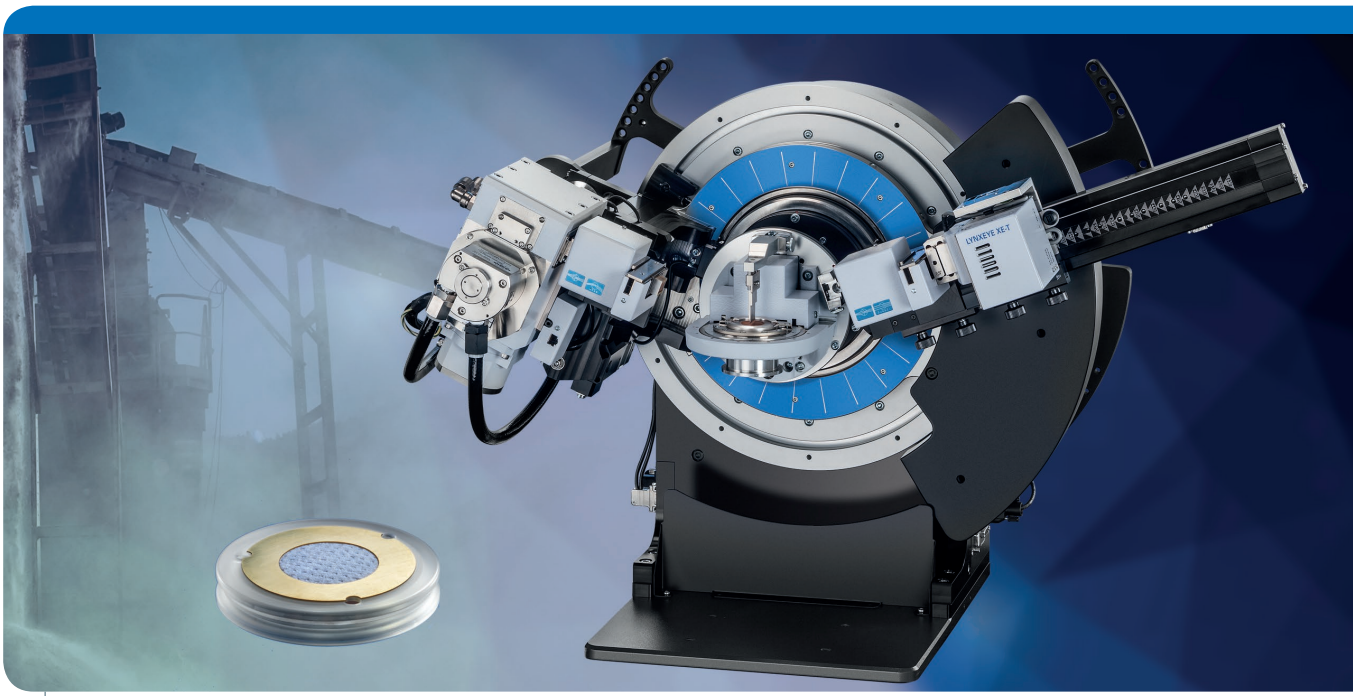

Application Report XRD 32

D8 DISCOVER Plus

● Lower Limits of Detection (LOD) and Quantification (LOQ) for Respirable Silica

The D8 DISCOVER Plus equipped with the vertical ATLAS goniometer and the high-efficiency turbo X-ray source (TXS-HE) is a powerful system for powder diffraction. Users benefit from the 6 kW rotating anode technologie enabling very low limits of detection and quantification in addition to rapid data collection.

Powder diffraction in Bragg-Brentano geometry benefits from the line focus of the rotating anode. It ensures optimal illumination of the sample with a high-intensity X-ray beam, exceeding intensities known from sealed tubes up to 5 times.

It's easy to understand that a more intense primary beam provides more reflected X-rays, therefore users take advantage from:

- significantly reduced measurement time while maintaining the same statistical relevance
- increased productivity by higher sample turnaround driven by a shorter measurement time
- lower limits of detection (LOD) and quantification (LOQ) due to higher statistical relevance at measurement times comparable to lower powered X-ray sources.

In X-ray powder diffraction the peak of the compound to be determined needs to be separated from the background of a blank sample (LOD). The

Limit of Quantification (LOQ) defines the lowest value that can safely be quantified. It exhibits a larger confidence interval than the LOD. Furthermore, it should consider the slope of the calibration curve. After IUPAC suggestions, the detection and the quantification limits may be estimated from the noise in the background or blank data (b) and from the slope of a straight calibration curve (p) as LOD = 6b/p and LOQ = 9b/p, respectively.

Respirable Silica

Silica is a typical component of soil and rocks but is also contained in foundry materials or concrete. Clear exposure/response relations were reported for e.g. miners, diatomaceous earth and construction workers, granite, pottery, refractory bricks, or foundry workers. Lung cancer and other health issues are known to be associated with occupational exposure to breathable crystalline silica, SiO_2. This makes silica dust, or free silica, the 2nd largest cause of occupational cancer after asbestos.

Occupational exposure to respirable silica is a preventable health hazard. Exposure limits were issued by several national authorities and therefore, concentrations of silica are to be monitored. While at present the NIOSH norm requests a detection limit of 20 µg, continuous efforts aim at pushing the detection limit towards much lower concentrations. Table 1 details some experiment conditions of the D8 DISCOVER Plus used in this study for the measurement of silica loaded filters.

The DIFFRAC.DQUANT software was used to establish norm compliant drift-corrected calibrations, evaluate unknowns and to report the results directly to the laboratory LIMS system or to the internal instrument database. This software supports absorption correction as requested by NIOSH 7500 and also segment calibrations. Figure 1 illustrates the noise level (about ±5 cps) and peak intensities whereas the calibration curve is shown in Figure 2. With the slope 26.7 cps/µg of the calibration a detection limit LOD = 1 µg and a quantification limit LOQ = 2 µg are obtained. Achieving similar results requires about ten times longer measurements with conventional sealed tube instruments (Rodesney et al. How low can you go? Denver X-ray conference, Big Sky, Montana, 2017).

These extremely low values proof the D8 DISCOVER Plus with TXS-HE to be an extremely powerful and sensitive tool for quantitative phase analysis at lowest concentration levels.

Geometry	Bragg-Brentano, 560 mm
Generator	45 kV, 120 mA
Detector	LYNXEYE XE
Divergence Slit	1.5 mm
Soller Slit	4.0°
Scan Range	19 – 40° 2Theta
Step Size	0.02°
Time/Step	0.5 seconds

Table 1: Experiment conditions of the rotating anode D8 DISCOVER Plus diffractometer.

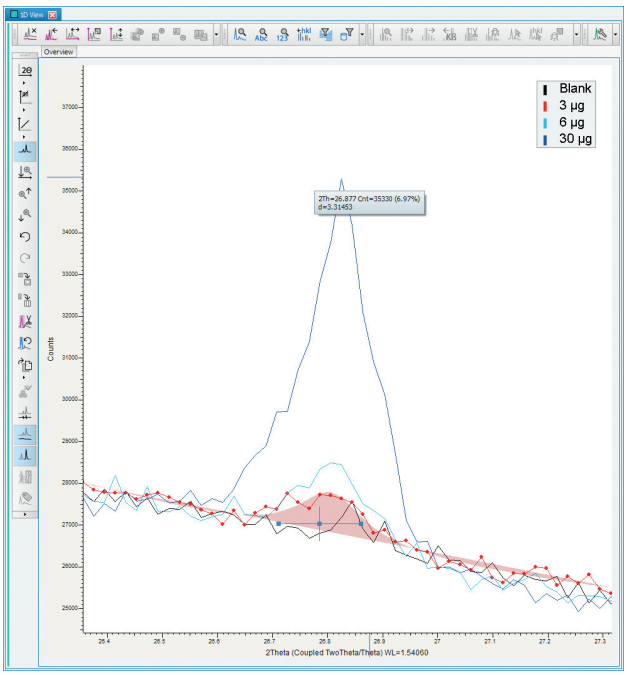

Figure 1: DIFFRAC.EVA chart highlighting the strongest quartz peak of a 3 µg sample (red), together with data from a blank (black) and two higher loadings. The background variance is about ±5 cps.

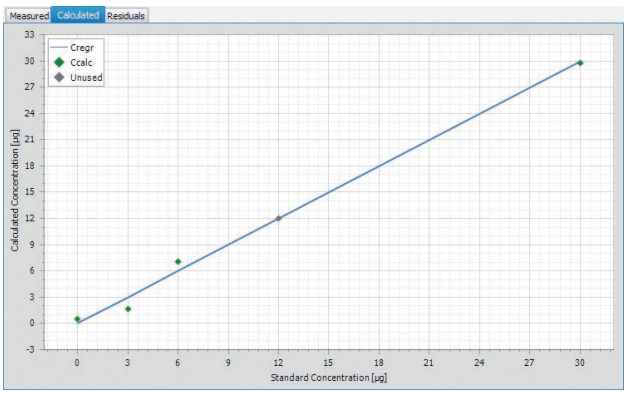

Figure 2: DIFFRAC.DQUANT calibration curve for quartz.

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