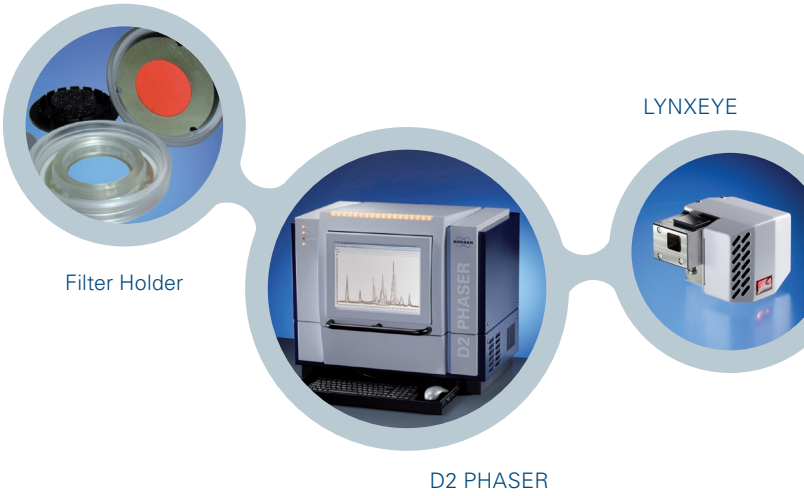
Bruker AXS



Application Report XRD 8

D2 PHASER Desktop XRD: Silica Dust Analysis

The D2 PHASER is a portable desktop XRD instrument for research and quality control. It is easy to operate and independent of external media such as cooling circuits. Thanks to the LYNXEYE detector it is the fastest desktop XRD system on the market.

This report demonstrates its use for monitoring occupational exposure to respirable silica.

Tab. 1: Experimental settings.

D2 PHASER, LYNXEYE detector
Cu radiation (30 kV, 10 mA), Ni filter
Continuous scan from 26.1 to 27.1° 2Theta Step width 0.01° Counting time: 1) > 30 µg: 1 sec per step 2) < 30 µg: 5 sec per step
1) Total scan time 5 min. 2) Total scan time 25 min.
4° Soller collimators, 1.0 mm divergence slit, anti-scatter screen
LYNXEYE detector opening 5° 2Theta

Lung cancer and other health issues are known to be associated with occupational exposure to crystalline silica, SiO_2. This is a typical component of soil and rocks. Clear exposure/response relations were reported for e.g. miners, diatomaceous earth and construction workers, granite, pottery, refractory bricks, or foundry workers. Occupational exposure to respirable silica is a preventable health hazard and therefore, the concentrations are monitored.

X-ray powder diffraction is capable of distinguishing polymorphs of crystalline silica (quartz, cristobalite, tridymite). Furthermore, XRD may account for the interference with other minerals that may additionally be present at the workplace. Sampling of the airborne particles on filters and their investigation is regulated by several national norms like NIOSH 7500, OSHA ID-142, MSHA P-2, and others. The concentration of an unknown silica phase is determined from a calibration, which needs to be established from reference samples using e.g. the DIFFRAC.DQUANT software.

Filter papers with different amounts of quartz deposited were measured applying the D2 PHASER and a special sample holder for filters. Experimental details are summarized in Table 1. Figure 1 shows several diffraction scans of

the 100% quartz peak. The different intensities are directly related to the concentration of the deposited quartz dust. The net intensities of the different specimens show a clear linear correlation with the concentrations (see inset).

The calibration fully complies with the NIOSH norm. The curve has zero offset of 2.7 µg (±5 µg permitted in NIOSH 7500). The limit of detection (LOD) in this example is about 8 µg. It can further be reduced by increasing the measurement time. A counting time of 5 sec per step for example, increases the total scan time to about 25 min but reduces the LOD below 5 µg. The precision is better 1 % relative for concentrations exceeding 100 µg and better 10 % relative between 10 and 100 µg.

To conclude, our cost-effective desktop XRD system D2 PHASER allows for fast, precise and norm compliant analyses of airborne respirable silica particles on filters.

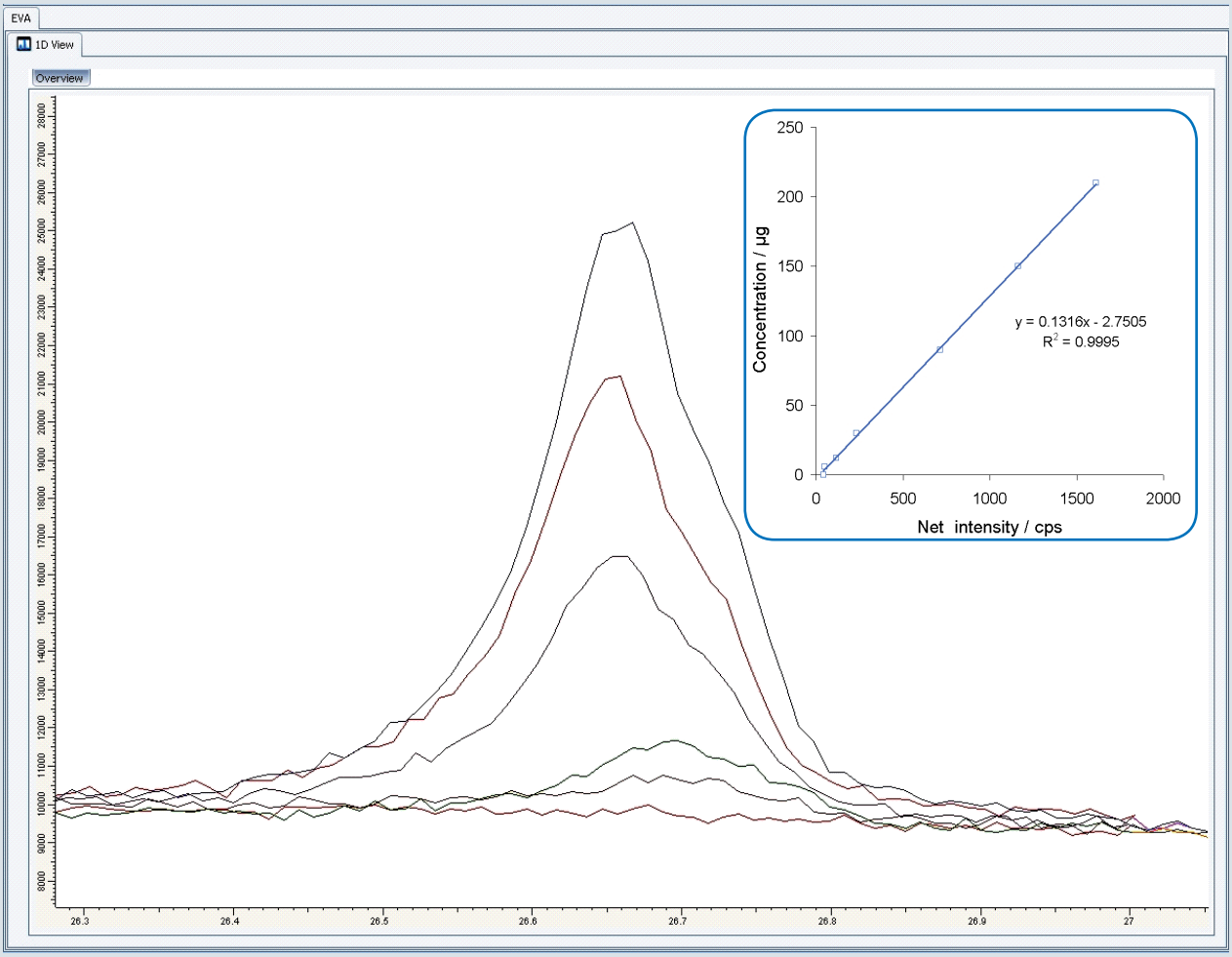


Fig. 1: Diffraction signals of a blank, 12, 30, 90, 150, and 210 µg quartz on filters at the position of the strongest quartz peak all measured with 1 sec per step. The inset shows the calibration curve of the net intensities determined using DIFFRAC.EVA vs. the known concentrations.

All configurations and specifications are subject to change without notice. Order No. DOC-R88-EXS008.
© 2009 Bruker AXS. Printed in Germany.

Bruker AXS GmbH
Karlsruhe, Germany
Phone +49 (721) 595-2888
Fax +49 (721) 595-4587
info@bruker-axs.de
www.bruker-axs.de

Bruker AXS Inc.
Madison, WI, USA
Phone +1 (800) 234-XRAY
Phone +1 (608) 276-3000
Fax +1 (608) 276-3006
info@bruker-axs.com
www.bruker-axs.com