Application note | 41521

X-ray diffraction

Analysis of silica in air filters using ARL X'TRA Companion X-ray Diffractometer

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Introduction

The analysis of silica in air filters is a critical aspect of occupational health and safety, aimed at protecting workers from the harmful effects of inhaling crystalline silica particles, which can lead to diseases such as silicosis and lung cancer. The NIOSH 7500 method (NIOSH, 2003) provides a detailed and standardized procedure for the determination of crystalline silica in air samples using X-ray diffraction (XRD). This method involves the collection of air samples on filters, which are then analyzed to identify and quantify the crystalline silica phases, including quartz, cristobalite, and tridymite.

XRD is chosen for its high specificity and sensitivity in distinguishing different crystalline forms of silica, making it a reliable analytical technique. The method outlined in NIOSH 7500 requires meticulous sample preparation, including the proper collection and handling of air filters, calibration with known standards, and strict adherence to analytical parameters to ensure accurate and reproducible results. Following the NIOSH 7500 norm and equivalent standards helps ensure the reliable monitoring of workplace air quality, thereby safeguarding workers' health. The use of XRD in analyzing silica in air filters is an essential component of industrial hygiene and environmental monitoring programs, facilitating the detection and control of hazardous silica exposure in occupational settings.

Instrument and software

The Thermo Scientific™ ARL™ X'TRA Companion X-ray Diffractometer (c.f. Figure 1) is a simple, easy-to-use benchtop XRD instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion XRD uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available on demand. Thanks to the innovative solid state pixel detector (55 x 55 µm pitch), the ARL X'TRA Companion XRD provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS (laboratory information management system).

Figure 1. ARL X'TRA Companion X-ray diffraction system.

Experiment

PVC air filters spiked with 5, 10, 20, 30, 50, 100 µg quartz (SRM NIST 2950 respirable quartz) were measured for 10 minutes. The samples were loaded in dedicated air filter sample cups and measurements between 26.1 and 27.3°2θ (Quartz 111 reflection) were performed in reflection mode using Cu Kα (1.541874 Å) radiation with sample spinning. (c.f. Figure 2). A single peak fit in Profex software [1] was carried out to build a linear regression using the integral intensities. Afterwards, 11 measurements of a blank filter sample were performed and using the same fit and the linear equation, the residual quartz content was calculated. The limit of detection (LoD) is calculated as 3*σ residuals.

Results and discussion

Performing a linear regression (Figure 3) results in a R^2 = 0.99 and therefore very good linearity. The standard error of estimate (SEE) is calculated as 3.8 µg. From blank sample measurements, a LoD of 1.8µg is calculated, which easily complies with the requirements given in the NIOSH 7500 norm.

Your benefits

The ARL X'TRA Companion XRD provides data perfectly suited to quantify respirable quartz in air filters. Utilizing a calibration curve in a **one-click refinement** delivers results within **minutes**, ensuring **ease-of-use** for operators and **reducing training requirements**. XRD analysis of air filter samples complies with the **NIOSH 7500** standard.

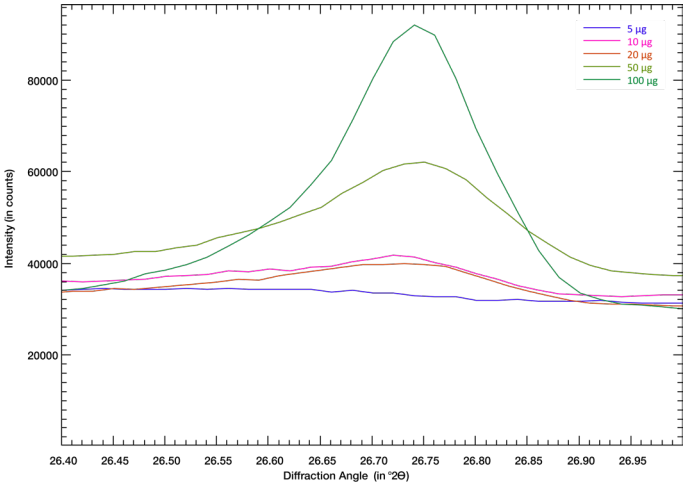

Figure 2. XRD patterns of 5, 10, 20, 50, 100 µg NIST 2950 Quartz samples

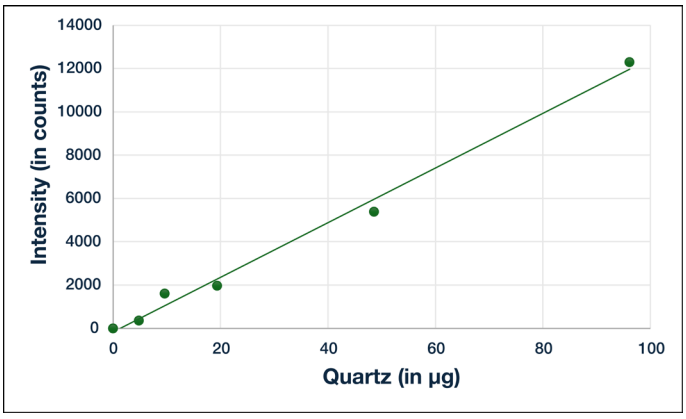

Figure 3. Linear correlation between the reference and values determined by XRD of 0, 5, 10, 20, 50, 100 µg Quartz samples.

Reference

1. N. Döbelin, R. Kleeberg, *J. Appl. Crystallogr.* **2015**, 48, 1573-1580.

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