Application Note XRD 621

Respirable Silica with the D8 ENDEAVOR

- Quantification of α-Quartz Filters

X-ray diffraction (XRD) is used by industrial hygienists to monitor hazardous airborne respirable crystalline silica particles in the workplace. The D8 ENDEAVOR x-ray diffractometer is ideal for this application, featuring state-of-the-art components and fully automated measurement strategies to allow quantification of crystalline silica to very low quantities in minutes.

Summary

- α-quartz dust on filter media were prepared by ashing and measured using the D8 ENDEAVOR
- DIFFRAC.DQUANT was used to build the calibration curve and determine the amount of α-Quartz
- Measurement time of 1 min per filter produced a linear calibration curve down to 5 µg

Millions of workers are exposed to harmful respirable crystalline silica every year. Lung exposure to crystalline silica can cause multiple diseases including silicosis, COPD, and lung cancer. As a measure to further protect workers, OSHA recently reduced the workday permissible exposure limit (PEL) and action level to 50 mg/m^3 and 25 mg/m^3, respectively. Workspaces from many different industries will need to be monitored and recertified to ensure compliance with this new standard. The amount of crystalline silica in a volume of air can be quantified following the analytical method described in NIOSH 7500 in which x-ray diffraction is used to detect and quantify different phases of crystalline silica that are considered hazardous.

Introduction

Crystalline silica (SiO_2) exists as several different naturally occurring polymorphs including quartz, cristobalite, and tridymite. These minerals are harmless as large macroscopic crystals, but long term exposure to microscopic dust-sized particles can cause silicosis and increased risk of lung cancer.

Respirable crystalline silica is a potential health hazard in many industries including mining, construction, demolition, foundries, and manufacturing. Government regulatory agencies have established Permissible Exposure Limits (PEL) for workers during a single shift. In the United States, OSHA has determined the PEL for crystalline silica to be 50 µg averaged over an 8 hour day.

While other analytical techniques can determine the total silica (amorphous and crystalline) present, only XRD can positively identify and quantify the different polymorphs.

Filter Preparation

This study focuses on quartz, the most common hazardous crystalline SiO2 phase. The method presented here could also be applied to cristobalite and tridymite.

Respirable α-quartz on filter media (NIST SRM 2950a were processed using the ashing method described in NIOSH 7500 and mounted in specimen holders specifically designed to accommodate filters.)

Figure 1. Filter sample holder.

Generator	**40 kV, 40 mA**
Divergence Slit	**V10***
Soller Slits	**4°**
Step Size	**0.02°**
Time/Step	**0.2 seconds**
Beta Filter	**None**
Total Time	**64 seconds**

Table 1. Primary quartz peak measurement conditions. *V10 divergence slit maintains a constant irradiated area on the filter

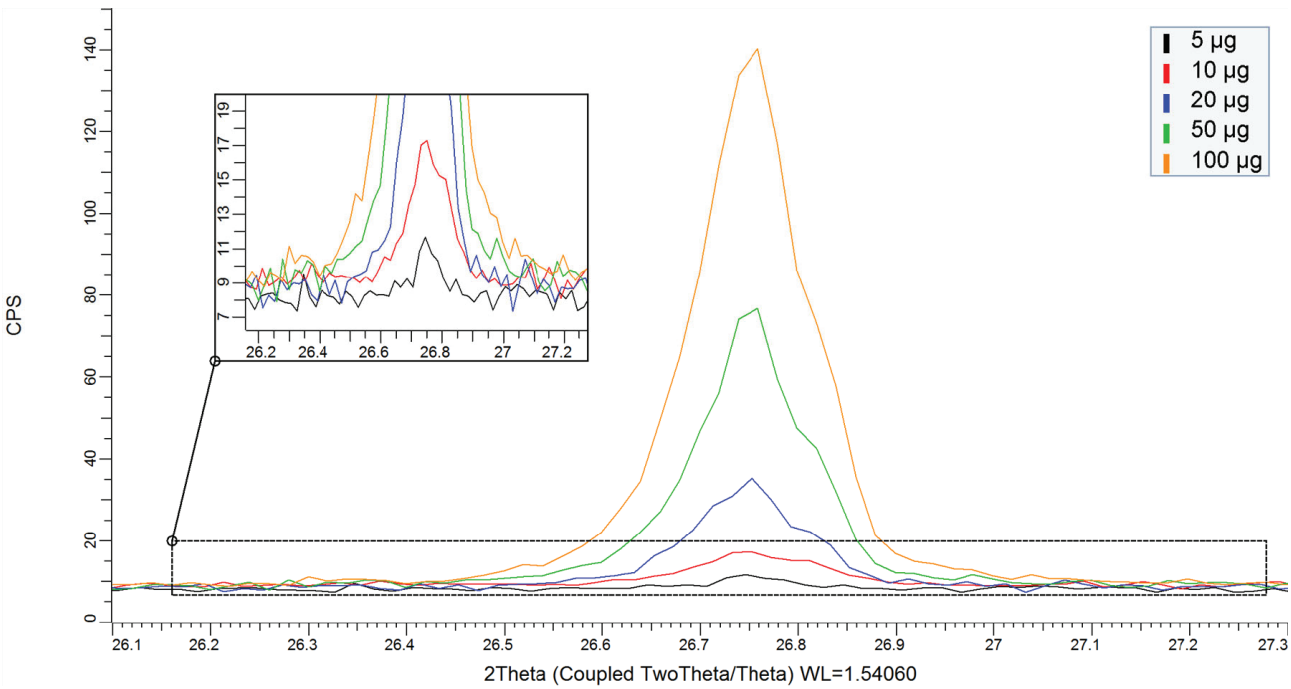
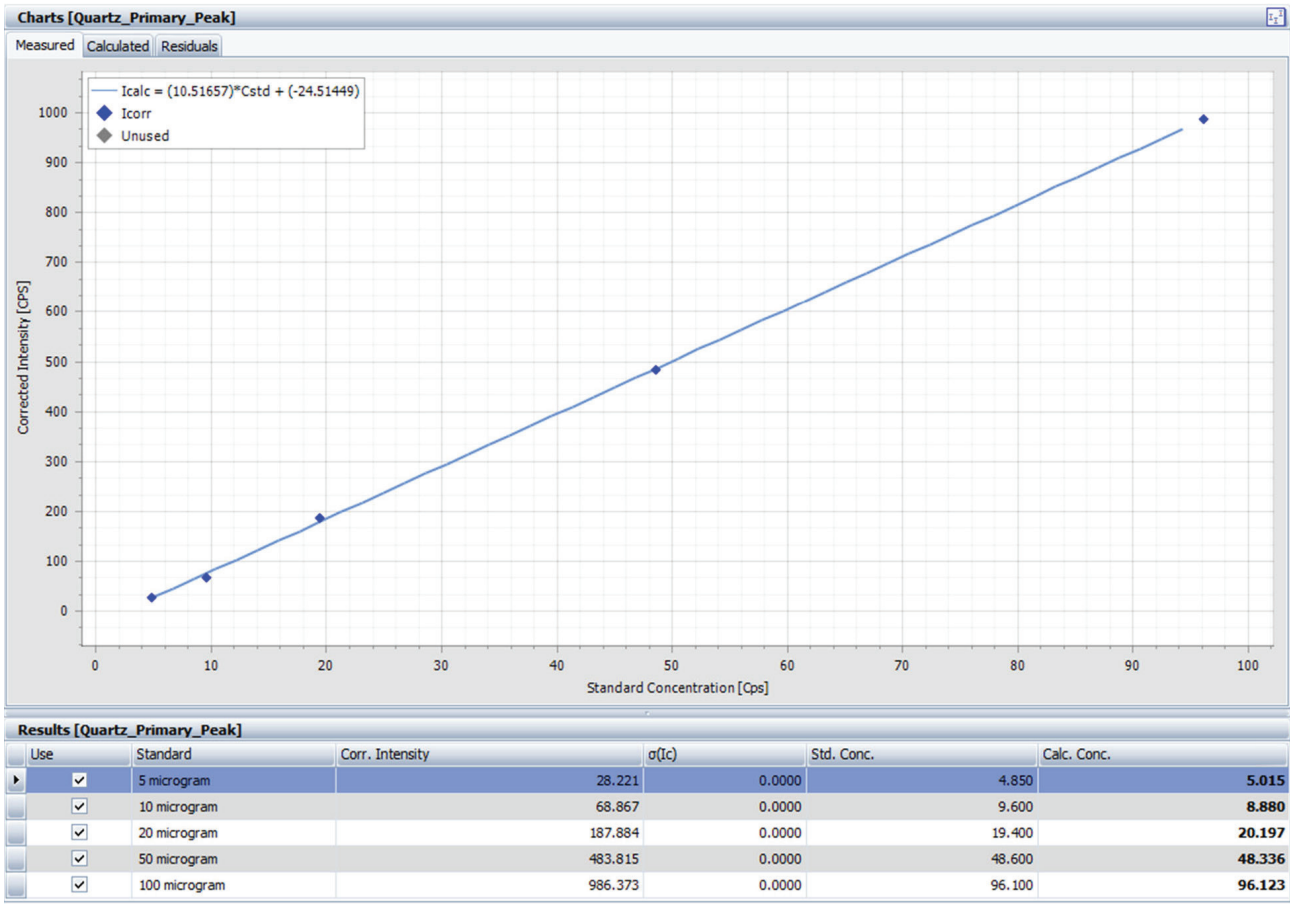


Figure 2. Primary diffraction peak for α-quartz. Measurement time of 1 minute can accurately detect and quantify masses down to 5 µg.



Results [Quartz_Primary_Peak]

Use	Standard	Corr. Intensity	σ(Ic)	Std. Conc.	Calc. Conc.
☑	5 microgram	28.221	0.0000	4.850	**5.015**
☑	10 microgram	68.867	0.0000	9.600	**8.880**
☑	20 microgram	187.884	0.0000	19.400	**20.197**
☑	50 microgram	483.815	0.0000	48.600	**48.336**
☑	100 microgram	986.373	0.0000	96.100	**96.123**

Figure 3. Calibration curve of net intensities determined using DIFFRAC.DQUANT. R2=0.9998

Measurement and Analysis

For analyzing very low masses of quartz, the Bragg-Brentano parafocusing geometry is ideal to maximize intensity and achieve good peak resolution.

All filter samples were measured on a D8 ENDEAVOR equipped with 66-position sample handler to automate data collection. A LYNXEYE XE-T detector was used to achieve superior signal-to-noise ratio, and uniquely offers energy resolution equivalent to a graphite monochromator (recommended by NIOSH). A primary variable divergence slits maintains a constant beam footprint on the filter, illuminating the entire deposition area throughout the duration of the scan. The experimental setup exceeds the requirements called for in regulatory XRD procedures. Experimental conditions are reported in table 1.

DIFFRAC.DQUANT software is then used to build a calibration curve from the standard reference samples (Figure 3) and automate the analysis of subsequent unknown specimens. DIFFRAC.DQUANT is NIOSH compliant, capable of tracking tube drift and correcting for absorption. The calculated concentrations (Figure 3) derived from the calibration curve are within the NIST certified values for the filters.

Conclusions

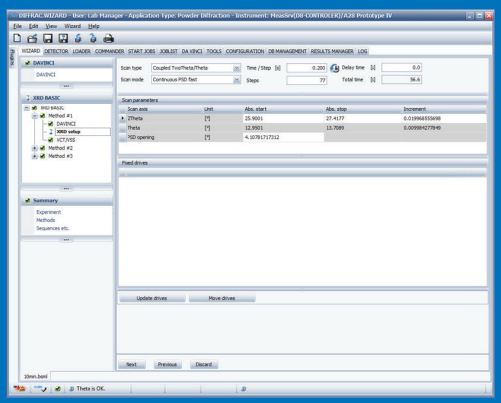
Respirable silica dust is a health hazard in many industrial workplaces. Monitoring the amount of airborne silica plays an important role in protecting millions of workers around the world and ensuring compliance with OSHA regulations.

The D8 ENDEAVOR with LYNXEYE XE-T and DIFFRAC.DQUANT is the most complete, efficient, and automated solution for rapidly and accurately measuring silica dust on filter media. This turn-key solution not only meets current OSHA standards, but a standard 10x less, future-proofing against reduced PELs.

DIFFRAC.SUITE Workflow for Respirable Silica Analysis

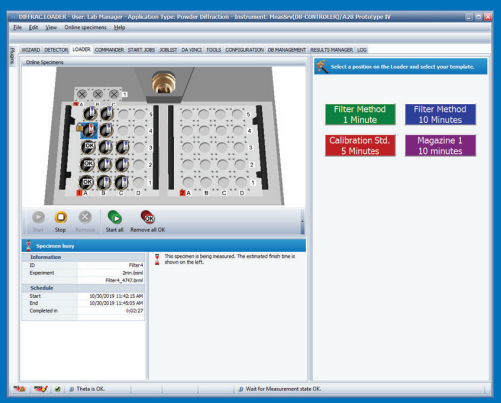
PLAN in DIFFRAC.WIZARD

- Set up multiple measurements for silica analyte peaks
- Large beam footprint to illuminate entire filter
- Spin sample to improve statistics



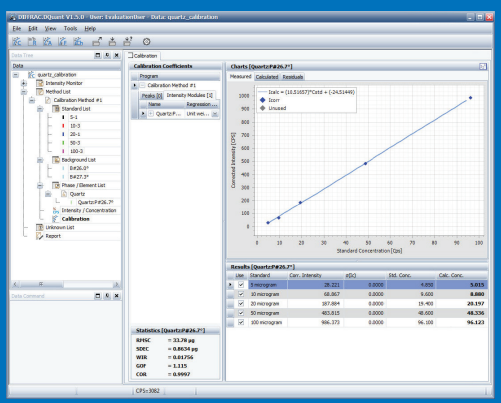
MEASURE in DIFFRAC.MEASUREMENT

- Launch pre-defined experiment methods using the Loader interface
- Automate data collection and analysis of large sample sets
- Access results in the integrated Results Manager



ANALYZE in DIFFRAC.DQUANT

- Manage calibration curve, track and correct drift
- Automate analysis of unknowns
- Generate reports



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