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Technical Brief

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Charles Lorberau

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Investigation of the Determination of Respirable Quartz on Filter Media Using Fourier Transform Infrared Spectrophotometry

Charles Lorberau
National Institute for Occupational Safety and Health
Cincinnati, Ohio

Introduction

As the inhalation of dusts containing alpha-quartz and other crystalline silica polymorphs poses a significant risk for the development of respiratory disease, limits for safe exposure have been defined by a number of groups concerned with occupational safety and health. For example, the National Institute for Occupational Safety and Health (NIOSH) has defined an 8-hour time-weighted average (TWA) of 0.05 mg/m^3 as the Recommended Exposure Limit (REL) for respirable quartz.[1]

Current NIOSH sampling and analysis methods[2,3] for the determination of quartz require collection of the respirable fraction of the dust on filters with subsequent ashing and either redeposition onto silver filters for analysis by X-ray Powder Diffractometry (XRPD) or pressing a potassium bromide pellet of the ash for analysis by dispersive infrared spectrophotometry. These steps may result in losses of material or help to introduce error in the analysis.

Sampling and analysis methods in which the collection filter is analyzed without additional sample preparation might mitigate losses and provide for a more efficient procedure. Efforts to develop such a direct-on-filter method employing XRPD, while producing results within 25 percent of the reference value at the 95 percent confidence level, showed that the samples had to be referred to standards which were prepared by deposition of a quartz aerosol.[4] It would be preferable to employ a method in which calibration standards could be prepared by filtration of a quartz suspension rather than the above mentioned procedure which is awkward and requires specialized equipment. Transmission infrared spectrophotometry affords this possibility, particularly when combined with the opportunity provided by Fourier Transform Infrared (FT-IR) Spectrophotometry for enhanced sensitivity and accurate spectral subtraction.

Experimental

Filter samples containing quartz, either alone or in the presence of broadband interferences (coal dust) or structured interferences (cristobalite or kaolinite), were prepared by filtering aliquots of the appropriate suspension onto filter media. (Initial studies were performed to indicate which commercially available filters were appropriate.) Calibration samples, using Standard Reference Material (SRM) 1878[5] as the reference material, were prepared over the range of 20 to 1000 μg.

Spectra were collected on a Nicolet 60-SX Fourier Transform Infrared Spectrophotometer (Nicolet Analytical Instruments, Madison, WI) at two wavenumber resolution using a KBr beamsplitter and triglycine sulfate (TGS) detector. A mirror velocity of 0.813 cm/sec with full aperture was employed in the data collection.

The absorbances were estimated by a number of approaches: 1) peak height after subtraction of the estimated filter contribution, 2) peak height after spectral subtraction of the filter contribution, and 3) integrated absorbance after spectral subtraction of the filter contribution.

The influence of particle size on analysis was investigated by analyzing samples of sized fractions of SRM 1878, obtained by collecting aerosolized reference material with an Andersen Mark II impactor (Andersen Sampler Inc., Atlanta, GA). The absorbance per unit mass served as a guide to the effect of this parameter.

Results and Discussion

The PVC-copolymer filters DM-450 and DM-800 (Gelman Sciences, Ann Arbor, MI) proved to have the best analytical performance (lowest absolute absorbance and lowest relative standard deviation of absorbance) of the 15 commercially available 25-mm-diameter filters examined in the study. Filter absorbance variations, both within-filter and between-filter, were determined to be 1.3–2.4 percent at the wavenumbers optimal for quartz analysis, 798 cm^{-1} and 779 cm^{-1}. This places a limit on the performance of a direct-on-filter IR method.

The optimum procedure for the determination of absorbance of a sample due to the analyte was found to be subtraction of the absorbance of a representative filter based on weight, followed by the determination of the resultant baseline-corrected peak height at either 798 cm^{-1} or 779 cm^{-1}. Calibration curves by such a procedure were found to be linear to 1 mg and limits of detection, defined as three times the standard deviation of a blank, were found to be 2.9 μg (at 798 cm^{-1}) and 5.3 μg (at 779 cm^{-1}).

A slight curvature in the coal dust's spectrum in the quartz analytical bandwidth, which could not be corrected, resulted in a bias for samples contain-

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ing this broadband absorber. The bias, when 1 mg of coal dust was present, resulted in quartz being underestimated by 6–9 percent.

For samples containing structured interferences, correction of the interferences was examined by both spectral subtraction of the spectrum of the interference and by use of correction curves to estimate the contribution due to the interference at the band of analytical interest. For these interferences, correction curves were prepared of baseline-corrected absorbance at the quartz 798 cm^{-1} band versus baseline-corrected absorbance at a unique interference absorbance band. These were found linear for the bands 913 cm^{-1} (kaolinite) and 621 cm^{-1} (cristobalite) over the range 40–200 μg.

Both spectral subtraction and estimation from correction curves were found to overcorrect for the amount of interference and resulted in a negative bias in the quantitation of quartz. Despite knowledge of the identity and level of interferent, quartz values were underestimated, by almost 20 percent for some samples, at both high (200 μg) and low (50 μg) loadings.

As noted by other researchers, the absorbance of quartz is particle size dependent.[6] Differences in the absorbance per unit weight were as high as 30 percent for some size fractions. Further, the ratio of the baseline-corrected intensities of the 798 cm^{-1}, 779 cm^{-1} doublet were found to vary with particle size. This ratio also was found to have less variability than the integrated absorbance of the doublet and may prove to be useful to correct for particle size mismatch between samples and standards.

Conclusions and Recommendations

The use of FT-IR for the direct-on-filter analysis of quartz does not offer any substantial improvement in analytical performance over existing dispersive IR methods. This approach is vulnerable to interferences, corrections for the presence of which have not been found to be totally successful.

The analytical performance of direct-on-filter IR methods are limited by variations in the absorbance of the filter substrate, which restrict the LOD to about 3 μg of quartz. Any inhomogeneity of the filter deposit from collection of the aerosol would elevate the limit of detection. The dependence of absorbance on quartz particle size observed may pose further difficulty for quartz IR determinations, especially for samples whose particle size distribution substantially varies from that of the reference material used for calibration.

Details of this research can be found in the NIOSH project final report, "Investigation of the Determination of Respirable Quartz on Filter Media using Fourier Transform Infrared Spectrophotometry," which is available from the National Technical Information Service (NTIS), Springfield, VA 22161, as document PB 90130105.

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Mr. Lorberau is with the Inorganic Methods Development Section, Methods Research Branch, Division of Physical Sciences and Engineering, National Institute for Occupational Safety and Health, 4676 Columbia Pkwy., Cincinnati, OH 45226.