



LETTER TO THE EDITOR

Editorial Note. Letters to the Editor are peer reviewed to ensure that the arguments are reasonable and clearly expressed. However, letters may express a particular opinion rather than a balanced interpretation. Authors of papers commented on are invited to reply, but neither the journal nor peer reviewers should be assumed to support the arguments made.

Analysis of Crystalline Silica in Bulk Materials

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Dear Sir,

We are writing concerning results presented in *Annals of Occupational Hygiene* as part of a manuscript by [Radnoff and Kutz \(2014\)](#). The manuscript presents the results of seven analyses without associated uncertainty or validated Limit of Quantitation (LOQ) for bulk crystalline silica content with values reported <1% down to <0.1%. In the Methods section, they state the samples were 'analysed according to NIOSH Method 7500 for the presence of quartz silica down to 0.1% w/w. This method includes protocols for analysing bulk or settled dust samples'. The NIOSH 7500 method ([NIOSH, 2003](#)) is designed to quantify respirable samples collected on a filter and does not include specific procedures for analyzing bulk or settled dust samples beyond using bulk samples to identify interferences in the air samples. The user of the method

is directed from Paragraph 4a: Interference check. Prepare area dust sample or settled dust bulk sample for XRD analysis ... to Paragraph 11: Obtain a qualitative X-ray diffraction scan of the area air sample (or bulk settled dust) to determine the presence of free silica polymorphs. The subsequent quantitative section (Paragraph 12) refers only to the air sample filter analysis. No evaluation data or LOQ estimate are presented in NIOSH 7500 to support bulk analysis and it is unlikely that the method could be used to measure down to or <0.1% in any case.

[Verma et al. \(2002\)](#) evaluated an infrared method for bulk analysis between 1 and 75% and concluded that although it could be used to determine down to 1% in routine analyses, the method was ineffective <1% silica. A more recent evaluation of an X-ray diffraction method ([Martin et al., 2012](#)) suggests that it

may be very difficult to go much <1%, even with the additional use of the Rietveld refinement in X-ray diffraction (LOQ = 0.76%). We are unaware of any method that has been published for the determination of crystalline silica in bulk materials that can measure down to 0.1%. If the laboratory used by Radnoff and Kutz has been able to modify NIOSH 7500 to achieve this goal, we would welcome publication of the details of the modification and methods validation as this would be of value to the occupational health community.

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REFERENCES

- Martin J, Beuparant M, Lesage J *et al.* (2012) Development of a quantification method for quartz in various bulk materials by X-ray diffraction and the Rietveld method. *Powder Diffr*; **27**: 12–9.
- National Institute for Occupational Safety and Health (NIOSH). (2003) Method 7500: silica, crystalline by XRD, Issue 4, 15 March 2003. NIOSH Manual of Analytical Methods, 4th ed. Publication No. 94–113. Available at <http://www.cdc.gov/niosh/docs/2003-154/pdfs/7500.pdf>. Accessed 10 January 2014.
- Radnoff DL, Kutz MK. (2014) Exposure to crystalline silica in abrasive blasting operations where silica and non-silica abrasives are used. *Ann Occup Hyg*; **58**: 19–27.
- Verma DK, Johnson DM, Des Tombe K. (2002) A method for determining crystalline silica in bulk samples by Fourier transform infrared spectrophotometry. *Ann Occup Hyg*; **46**: 609–15.

REPLY

Analysis of Silica in Bulk Materials Response

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Dear Editor,

We are pleased to respond to the letter to the editor from Harper and Key-Schwartz (2014) regarding our recent article (Radnoff and Kutz, 2014).

In 1999, Miles identified that, of the methods available to analyze crystalline silica in bulk samples, the use of acid digestion followed by analysis using X-ray diffraction employing a method such as NIOSH 7500 (NIOSH 2003a) showed promise as a technique for determining crystalline silica concentrations at the lower thresholds required by product legislation (0.1% by weight; Miles, 1999). Previous to this,

other researchers indicated that estimation of crystalline silica (quartz) content to 0.1% by weight is possible with careful sample preparation and instrument optimization (Carter *et al.*, 1987; Salter and Riley, 1994). In our study, an American Industrial Hygiene Association-accredited laboratory analyzed the bulk abrasive samples using NIOSH 7500 methodology; however, a modified approach to sample preparation to increase sensitivity was used.

When analyzing for crystalline silica content, concentration is not measured directly by the laboratory. Rather, the mass of crystalline silica present in the