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SHORT REPORT



A novel sampling cassette for field-based analysis of respirable crystalline silica

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ABSTRACT

Field-based methods for the analysis of respirable crystalline silica are now possible with the availability of portable instrumentation. Such methods also require the use of cassettes that facilitate direct-on-filter analysis of field samples. Conventional sampling cassettes can be modified such that they are amenable to direct-on-filter analysis while remaining compatible with common respirable dust samplers. The required modifications are described herein, and one version of such an analysis-ready cassette is described and evaluated in comparison to more traditional cassette designs. The novel cassette was found to result in a slightly higher mass of collected respirable material (for the same sampling duration), though this is likely due to the conductive material of the cassettes, which prevents particle wall losses in comparison to the more commonly used styrene cassette material. Both types of cassettes demonstrated comparable predictability in terms of respirable crystalline silica in a sample.

KEYWORDS

Exposure monitoring;
personal sampling; quartz

Introduction

In mining as well as in many other industries, airborne respirable crystalline silica (RCS) may be monitored to ensure that workers are not exposed to unacceptably high concentrations. When inhaled in large quantities, RCS can result in an array of irreversible and debilitating health outcomes, including silicosis and lung cancer (NIOSH 2002; Steenland 2005; Leung et al. 2012; Steenland and Ward 2014; Cohen et al. 2016). Historically, exposure monitoring has required samples to be sent to an external laboratory for analysis, and results are not available for at least several days, in which time workers may continue to be unknowingly exposed to high concentrations of RCS. In contrast, recent advances in portable instrumentation have enabled field-based monitoring strategies, wherein exposure levels can be evaluated during or at the end of a worker's shift. Previous publications have outlined one field-based methodology using transmission Fourier transform infrared spectrometry (FTIR) for direct-on-filter (DOF) analysis of RCS in respirable dust samples (Miller et al. 2012; Miller et al. 2013; Cauda et al. 2016; Miller et al. 2017; Cauda et al. 2018; Hart et al. 2018). A DOF X-ray diffraction method for RCS analysis is already in use internationally for occupational samples (HSE 2005) and

other DOF methods have been described in various publications, though each of these methods is still designed to be used in the laboratory rather than directly at the worksite (Toffolo and Lockington 1981; Schwerha et al. 2002; Kauffer et al. 2005).

While the availability of suitable analytical instrumentation is perhaps the most vital component to enable field-based analysis, another essential component of this method is a sampling cassette which allows the sample filter to be analyzed without needing to remove it from the cassette. Allowing the filter to remain in the cassette protects the sample from damage or loss of material, as well as improves the efficiency of the analytical process. Conventional sampling cassettes are not directly compatible with the described FTIR method, but small modifications to the design can create a novel sampling cassette that is.

Cassettes designed for compatibility with DOF FTIR analysis should fulfill the following criteria in order to meet the specifications of the DOF while not compromising the sample.

1. Cyclone compatibility. The sampling cassette must be compatible with respirable dust samplers currently in use, such as the 10-mm nylon

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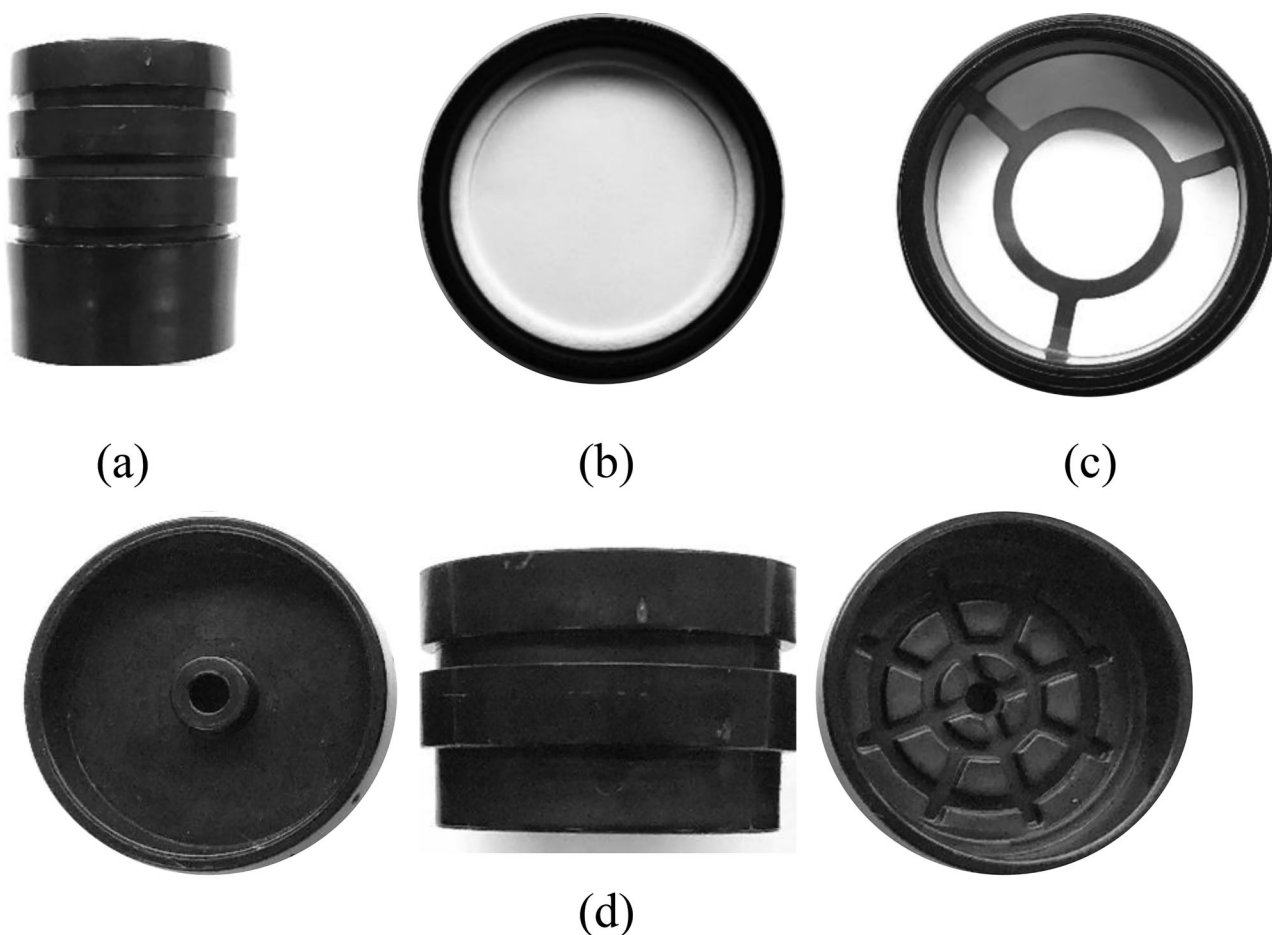


Figure 1. The novel cassette as it appears when fully assembled (a); the cassette top and first spacer are removed to show the cellulose spacer on top of the PVC filter (b); the second spacer shown from below to show the stainless-steel support which sits below the PVC filter (c); and the top and bottom of the cassette are removed for FTIR analysis, while the filter (along with cellulose spacer and stainless-steel support) remains secured between the two spacers (d).

- cyclone and the aluminum cyclone. The characteristics of different types of samplers have been discussed elsewhere (Lidén and Kenny 1993; Maynard 1993; Sass-Kortsak et al. 1993; Harper et al. 1998; Bartley et al. 1994; Lee et al. 2010; Lee et al. 2012; Stacey et al. 2013; Cauda et al. 2014; Stacey et al. 2014).
2. Conductivity. The cassette should be composed of a conductive (or static dissipative) material to minimize wall losses, thus maximizing the proportion of the collected respirable dust sample that is deposited on the filter. Implications of the conductivity of cassette material have been discussed elsewhere (Baron 1998; NIOSH 2016).
 3. Clear path to sample filter. The infrared beam must be able to penetrate the sample without interference from the cassette or support materials.
 - a. An opening with a minimum diameter of 8 mm at the center of the filter must be available for the infrared beam to pass through, either through sufficiently large inlet/outlet openings or through removable components of the cassette. This allows the FTIR beam to encounter the sample without being obstructed by the material of the cassette.
 - b. Similarly, the backing or support material for the filter must also have a centered opening 8 mm or larger in diameter or must be composed of a material transparent to mid-wave infrared (IR) radiation. To avoid the need to handle a dust-laden sample filter, this criterion should be achieved without removing the support material before analysis.
 4. Stability. The filter must remain secured and immobile in the cassette during sampling, during removal from the respirable sampler, and during analysis in the FTIR instrument. If components of the cassette must be removed to fulfill *criterion (3a)*, the act of opening the cassette should not cause the sample to move, become dislodged, or be otherwise disturbed. Reassembly should be

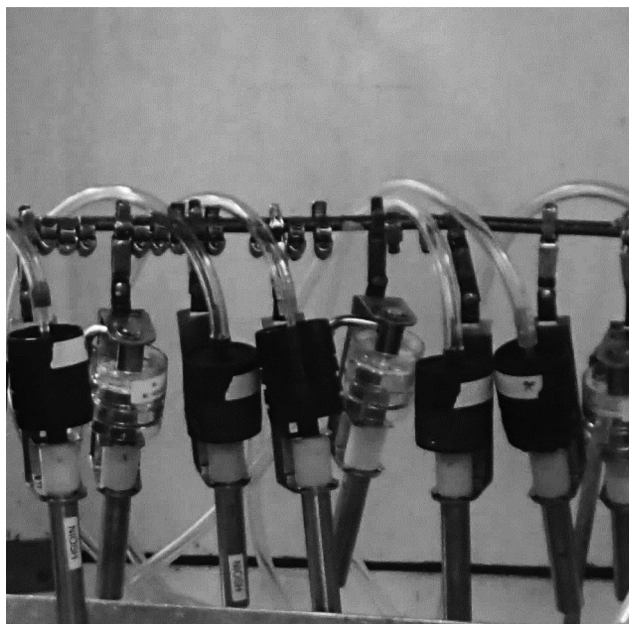


Figure 2. A portion of the circular scaffold within the Marple chamber is shown. The three cassette types are arranged in the order Novel-1, Reference, and Novel-2 around the circular scaffold.

possible in order to transport the cassette for additional analysis, if desired.

5. Consistency of sample. The combination of cassette and support material results in a consistent, predictable deposition of respirable dust (and by extension, RCS) on the filter, preferably independent of mass loading. The significance of respirable dust deposition is discussed in greater detail by Miller et al. (2013).

The commonly used three-piece cassette does not have a sufficiently large opening for the IR beam to pass through to reach the sample; removing the middle portion of the cassette would leave the filter unsecured. Additionally, the cellulose backing pad used to support the filter is not transparent to IR radiation. The three-piece cassette is frequently composed of styrene, which is not conductive or static dissipative.

Through a Cooperative Research and Development Agreement, Zefon International and the Pittsburgh Mining Research Division of the National Institute for Occupational Safety and Health (NIOSH) designed a modified version of the three-piece, 37-mm cassette (fulfilling *criterion 1* above). The four-piece novel cassette is composed of conductive polypropylene (*criterion 2*) and is pictured in [Figure 1](#). Removal of the top and bottom piece of the cassette allows the FTIR instrument full access to the sample (*criterion 3a*),

while the filter remains secured between two spacers and a cellulose ring between the first spacer and the top of the filter adds extra stability to prevent filter movement (*criterion 4*). In place of the conventional cellulose filter backing, a stainless-steel ring supports the filter while keeping the center portion unobstructed (*criterion 3b*); this type of support is already used in the Coal Mine Dust Sampling Cassette (part number 803462-2PK, Zefon International, Ocala, FL). The novel cassette components are shown in [Figure 1](#). The remainder of this article evaluates the novel cassette with respect to *criterion 5*, by comparing the consistency of the respirable dust mass collected using the novel cassette and using the styrene three-piece cassette.

Methods

Respirable dust samples were collected on 37-mm, 5- μm pore PVC filters (SKC Inc., Eighty Four, PA) with 10-mm nylon Dorr Oliver cyclones at a flow rate of 1.7 lpm (via critical orifice). In each test, 15 sample filters were housed in the novel sampling cassette described above (*Novel-1*) (individual components from Zefon International, Ocala, FL), 15 in the commonly used three-piece styrene cassette (*Reference*) with cellulose support pad (SKC Inc., Eighty Four, PA), and 15 in an unrelated prototype cassette (*Novel-2*; results not included here). Samples were arranged on a circular scaffold within a calm-air aerosol chamber with inside diameter of approximately 1.2 m (described in greater detail by Marple and Rubow 1983), with the 3 cassette types alternating around the scaffold (*Novel-1, Reference, Novel-2, Novel-1, etc.*). See [Figure 2](#).

Dust was aerosolized by a fluidized bed aerosol generator (model 3400 A, TSI, Shoreview, MN) and passed through a Kr-85 aerosol neutralizer (Model 3012 A, TSI, Shoreview, MN) before being introduced through the top of the chamber. A stable dust concentration within the chamber was maintained through each test, monitored by a TEOM (model 1400 A, Thermo Scientific, Waltham, MA). Over the course of two sampling sessions, five samples per cassette type were collected for each of six target loadings (500 μg , 1,000 μg , and 2,000 μg in the first session, and 750 μg , 1,200 μg , and 2,200 μg in the second session). Sampling pumps were shut off when a target loading was reached, with the remaining samplers continuing until all three targets had been reached. The test was repeated for three different dust samples (Sample 1 from a sand and gravel mine, approximately 6% RCS;

Table 1. Respirable dust mass collected by the *Novel-1* cassette ($n = 86$ samples) relative to the *Reference* cassette ($n = 90$ samples).

Target loading (μg)	Collected respirable dust mass average ratio (<i>Novel-1</i> to <i>Reference</i> cassette)		
	Dust 1	Dust 2	Dust 3
500	1.061	1.008	1.043
750	1.024	1.066	1.034
1,000	1.027	1.057	1.052
1,200	1.040	1.046	1.053
2,000	1.039	1.046	1.036
2,200	1.015	1.041	1.090
overall	1.034	1.044	1.051

Sample 2 from a coal mine, approximately 5% RCS; and Sample 3 from a copper mine, approximately 16% RCS; RCS content was previously determined by X-ray diffraction analysis of respirable samples). A total of four *Reference* cassette samples (one Dust 1 1,000- μg loading, two Dust 2 500- μg loadings, and one Dust 2 2,000- μg loading) were determined to be invalid and excluded from analysis due to sampler failure.

All filter samples were pre- and post-weighed in a temperature- and humidity-controlled weighing room using a model XP2U microbalance (Mettler Toledo, Columbus, OH). The sample set included approximately 5% lab blanks to verify accurate measurement from the microbalance. Analysis of blanks indicated an average gain of 1 μg with a standard deviation (SD) of 2 μg ; sample data were not blank corrected.

Following gravimetric analysis, samples were analyzed on a portable transmission infrared spectroscopy instrument (ALPHA model, Bruker Optics, Billerica, MA); each sample spectrum consisted of 16 co-added scans (total analysis time of approximately 20 seconds) collected at a resolution of 4 cm^{-1} using Blackman-Harris apodization. A blank PVC filter (from the same lot as the sample filters) was used as the background (reference spectrum). Although the *Novel-1* cassette enables infrared analysis directly in the cassette, the traditional three-piece styrene cassette does not. Each sample (from both types of cassette) was removed from its cassette and placed in a filter holder in order to ensure that all samples were handled in the same manner. The filter holder was inserted in the instrument so that the instrument's IR beam passed through the center portion of each sample. Sample spectra were baseline corrected and then integrated between 816–767 cm^{-1} ; this region (*800 area*) of the spectrum is commonly used in IR methods of quantifying RCS (quartz) (Miller et al. 2012; Miller et al. 2013; MSHA 2013; NIOSH 2017a, 2017b), and the integrated area is proportional to the mass of RCS (quartz) present in the sample.

Table 2. Predictive equations from the calibration set of samples, where x is *800 area* (arbitrary units) and y is gravimetric mass (in mg).

	<i>Reference</i>	<i>Novel-1</i>
Dust 1	$y = 4.953x - 0.086$ ($R^2 = 0.968$)	$y = 4.155x - 0.040$ ($R^2 = 0.994$)
Dust 2	$y = 2.530x + 0.070$ ($R^2 = 0.975$)	$y = 2.012x + 0.043$ ($R^2 = 0.949$)
Dust 3	$y = 1.691x + 0.035$ ($R^2 = 0.988$)	$y = 1.521x - 0.056$ ($R^2 = 0.988$)

Within each sample set (dust and cassette type), samples were ranked in order from lowest gravimetric mass to highest. Samples with odd rank were used as the calibration set, and samples with even rank were reserved as a test set. Predictive equations were determined for the calibration set of samples by regression of *800 area* (x -variable) and gravimetric mass (y -variable). Equations were then used to predict gravimetric mass in the test set of samples, based on the *800 area* response. Predicted gravimetric mass was compared to measured gravimetric mass via linear regression.

Results

For each loading-cassette combination, the average mass of collected respirable dust was determined. *Novel-1* results were compared to *Reference* results as a ratio, where perfect equivalency of the two cassette types is indicated by a ratio of one. Results are reported in Table 1 for Dusts 1, 2, and 3.

Predictive equations in the form of Equation 1 (determined for the two cassette types and for each dust using the calibration set of samples) are reported in Table 2.

$$\text{gravimetric mass (mg)} = m \cdot (800 \text{ area}) + b \quad (1)$$

Sample loadings of the calibration set ranged from 0.449–2.360 mg (for *Reference* cassette samples) and 0.461–2.430 mg (for *Novel-1* cassette samples).

These equations represent the relationship between *800 area* measurement (proportional to RCS mass) and overall respirable dust mass but are not directly comparable between the two cassette types (due to distinct patterns of dust deposition within the two cassettes as a result of the different backing supports) or between the three different dusts (due to different content of both RCS and other types of minerals that can also impact this region of the FTIR spectrum (Ojima 2003, NIOSH 2017b)).

Figure 3 shows measured gravimetric mass (x -axis) compared to predicted gravimetric mass (y -axis) for the test set of samples. Sample loadings of the test set ranged from 0.464–2.359 mg (for *Reference* cassette samples) and 0.491–2.457 mg (for *Novel-1* cassette samples). For both regressions shown, the significance

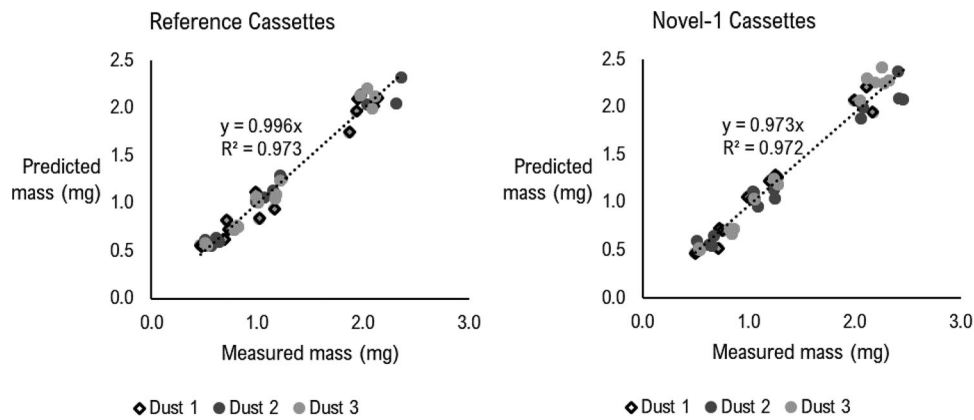


Figure 3. Measured and predicted gravimetric mass (mg) is compared for *Reference* cassettes ($n = 41$, left) and for the *Novel-1* cassettes ($n = 45$; right).

of the intercept was tested and found to be non-significant. As in Table 1, perfect equivalency of the predicted and measured value is indicated by a ratio (slope) of one.

Discussion

Compared to filters housed in the commonly used *Reference* cassette, filters housed in the *Novel-1* cassette collected slightly more respirable dust on average (3.4–5.1%). This could be attributed to the difference in electrical conductivity of the two cassette types: the styrene material of the *Reference* cassette is insulating and thus is expected to result in greater wall losses, while the conductive material of the *Novel-1* cassette decreases the likelihood of dust particles depositing on the cassette walls (NIOSH 2016). The use of Kr-85 neutralizer for dust entering the aerosol chamber likely mitigated the effect of *Reference* cassette wall losses (compared to *Novel-1*). Laboratory methods for the analysis of respirable dust and RCS may address wall losses by wiping cassette walls to re-capture particles lost to wall deposition. In field-based analysis circumstances, particularly where dust collection occurs without the aid of charge neutralization, it would be more advantageous to use a conductive cassette material for DOF analysis in order to maximize the amount of respirable material that is ultimately deposited on the filter.

The strong relationship (Table 2) between FTIR *800 area* (measured only at the center portion of the filter) and gravimetric mass (measured for the entire filter) across all samples suggest a very consistent deposition of respirable dust on the filter and a proportional relationship between the FTIR measurement at the center of the sample filter and the quantity of respirable dust present on the entire sample filter. As

a result, the measured *800 area* of a sample can be used to predict the gravimetric mass (when the predictive equation for the specific dust and sampler combination is known).

A general predictive equation (i.e., a calibration curve) could be determined using samples of pure RCS (quartz), where *800 area* corresponds directly to the mass of quartz present. For this study, each of the dust samples contained minerals in addition to quartz, some of which may also contribute (either positively or negatively) to the measured *800 area*, and therefore, the predictive equations of Table 2 apply only to the specific dusts evaluated. The equations were used to predict total respirable dust mass rather than RCS mass. Figure 3 demonstrates that the *Reference* cassettes result in a nearly perfect 1:1 (95% confidence interval 0.973–1.102 for the regression coefficient) relationship between measured and predicted values, whereas the *Novel-1* cassettes result in a slight underestimate of mass (compared to the measured value) (95% confidence interval 0.951–0.999 for the regression coefficient), although the overlap in the two confidence intervals suggests that the difference is not statistically significant. For the purposes of field-based analysis, the *Novel-1* cassette would nonetheless provide a reliable estimate of dust mass, despite the slight underestimation. If this cassette type were to be used for regulatory compliance purposes, the accuracy of that estimate would likely need to be evaluated in greater detail first.

Conclusions

This study evaluated a novel cassette designed to facilitate field-based, direct-on-filter analysis of RCS using transmission FTIR spectroscopy. The novel cassette is similar in design to the commonly used three-piece styrene cassette, but an additional spacer allows

the top and bottom portions of the cassette to be removed after sampling to allow unobstructed analysis using an FTIR instrument. The filter is supported by a stainless-steel ring rather than a cellulose backing pad and is further stabilized by a cellulose spacer ring on top of the filter. The novel cassette is composed of a conductive polypropylene material rather than styrene. While the conductive material results in a net gain in respirable dust deposited on the sample filter (up to approximately 5% relative to the styrene cassette), the novel cassette appears to result in samples with considerable comparability to the commonly used cassette, and the consistent deposition of respirable dust material on the sample filter is compatible with a direct-on-filter method of analysis.

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Disclaimer

The findings and conclusions in this paper are those of the authors and do not necessarily represent the official position of the National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention. Mention of any company or product does not constitute endorsement by NIOSH.

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