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Replacement of filters for respirable quartz measurement in coal mine dust by infrared spectroscopy

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**Keywords:** respirable silica, quartz, nylon filter, polypropylene filter, polyvinyl chloride filter, coal mine dust

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## Abstract

The objective of the present study is to compare and characterize nylon, polypropylene (PP), and polyvinyl chloride (PVC) membrane filters that might be used to replace the vinyl/acrylic copolymer (DM-450) filter currently used in the Mine Safety and Health Administration (MSHA) P-7 method (Quartz Analytical Method) and the National Institute for Occupational Safety and Health (NIOSH) Manual of Analytical Methods 7603 method (QUARTZ in coal mine dust, by IR re-deposition). This effort is necessary because the DM-450 filters are no longer commercially available. For example, the MSHA Pittsburgh laboratory alone analyses annually approximately 15,000 samples according to the MSHA P-7 method that requires DM-450 filters. There is an impending shortage of DM-450 filters. Membrane filters suitable for on-filter analysis should have high infrared (IR) transmittance in the spectral region 600-1000 cm<sup>-1</sup>. Nylon (47 mm, 0.45 µm pore size), PP (47 mm, 0.45 µm pore size) and PVC (47 mm, 5 µm pore size) filters meet this specification. Limits of detection and limits of quantification were determined from Fourier transform infrared spectroscopy (FTIR) measurements of blank filters. The average measured quartz mass and coefficient of variation were determined from test filters spiked with respirable  $\alpha$ -quartz following MSHA P-7 and NIOSH 7603 methods. Quartz was also quantified in samples of respirable coal dust on each test filter type using the MSHA and NIOSH analysis methods. The results indicate that PP and PVC filters may replace the DM-450 filters for quartz measurement in coal dust by FTIR. PVC filters of 5 µm pore size was seemed to be suitable replacement although their ability to retain small particulates should be checked by filter experiment.

## Introduction

About 2.2 million workers are exposed to respirable crystalline silica (RCS) in the U.S. and they are at risk of developing a diversity of pulmonary diseases such as lung cancer, pulmonary tuberculosis, silicosis, and other respiratory diseases.<sup>(1)</sup> Therefore, it is essential to be able to collect and analyze samples of air to determine RCS concentrations for comparisons with exposure limit values and to determine the effectiveness of engineering controls. According to the Mine Safety and Health Administration (MSHA) and National Institute for Occupational Safety and Health (NIOSH) methods, the airborne respirable fraction of a workplace aerosol is to be collected using a size-selective cyclone coupled to a cassette (see 30 CFR Part 74). Both the MSHA P-7 method (Quartz Analytical Method)<sup>(2)</sup> and NIOSH Manual of Analytical Methods (NIOSH) 7603 (QUARTZ in coal mine dust, by IR re-deposition)<sup>(3)</sup> require the sample filters to be ashed and the residue redeposited, either onto a 9 or 10 mm diameter area of a vinyl/acrylic co-polymer (DM-450) filter, respectively. NIOSH previously reported that the DM-450 filter showed lowest absorbance in the infrared (IR) wavelengths of interest and the lowest relative standard deviation of absorbance among 15 different filters.<sup>(4)</sup>

Approximately 15,000 samples are annually analyzed by the MSHA Pittsburgh laboratory staff in order to enforce the Respirable Coal Mine Dust standard when quartz is present in the dust.<sup>(5)</sup> However, the required DM-450 filter is no longer commercially available. The objective of the present study is to characterize potentially suitable alternative filters for quartz measurement in coal dust according to methods using Fourier Transform Infrared (FTIR) spectrometry.

**METHOD****Filters**

A previous study examined more than 20 commercially available membrane filters including mixed cellulose ester (MCE), cellulose triacetate, cellulose nitrate, polycarbonate, polypropylene (PP), polyamide (nylon), polyvinyl chloride (PVC), PVC/acrylic copolymer (DM), acrylic polymer, polysulfone, polyvinylidene difluoride, polytetrafluoroethylene and silver filters. The study determined that nylon, PP, and PVC filters are suitable for direct-on-filter IR analysis since these materials have no strong IR peaks that overlap the characteristic peaks of quartz.<sup>(6)</sup> A NIOSH report<sup>(4)</sup> investigated 15 different filters to select suitable filters for direct-on-filter FTIR analysis and selected two varieties of PVC and DM-450 filters because they presented a background of less than 0.5 absorbance units in the spectral range to be used for quartz quantitation. The same criterion was adopted for comparisons reported here. The four different filters that were tested in this study are listed in Table 1.

**Calibration curves**

Calibration curves for respirable  $\alpha$ -quartz (Standard Reference Material (SRM) 1878a, National Institute of Standards and Technology (NIST)) and kaolinite (Thermo Fisher Scientific, Pittsburgh, PA, USA) were prepared according to MSHA P-7 and NIOSH 7603, where a suspension of 1878a and a suspension of kaolinite in isopropyl alcohol were deposited on the test filters using vacuum filtration. The SRM 1878a provides an accurate calibration for samples from occupational environments.<sup>(7)</sup> Media blanks are subtracted from the sample spectra in both MSHA P-7 and NIOSH 7603 procedures, but the preparation of filters as media blanks differs

slightly between methods. In both methods a filter media blank serves as the spectroscopic background for FTIR analysis, thus the result of background subtraction would ideally be an absorbance spectrum of zero at all wavelengths. The MSHA P-7 method requires only one filter for a single batch. This filter is prepared by filtering isopropyl alcohol through it. This single filter is scanned at the beginning of a series of sample measurements and its spectrum used as the background measurement for all of the samples in a series. On the other hand, in NIOSH 7603 each single sample filter has a matching background filter. The pair are made by cutting two 25-mm diameter filters from one 47-mm filter. The two pieces are stacked during sample filtration so that the bottom piece receives the same treatment except for particle collection and is used as the media blank, while the top piece collects all of the sample particles. This approach presumes that any variability is minimal between adjacent locations of a 47 mm diameter filter.

In the experiments reported here, a circular 25-mm diameter glass fiber filter (SKC Inc. Eighty Four, PA) was placed on a fritted base and one or two 25-mm diameter test filters (depending on whether following MSHA or NIOSH method, respectively) were placed on the top of the glass fiber filter. After placing the filter stack using a filtration apparatus with a funnel, isopropyl alcohol was added to the funnel prior to adding sample from the suspension. Seven different masses of respirable  $\alpha$ -quartz, ranging from 20 to 700  $\mu\text{g}$  (as per the MSHA method) and ranging from 10 to 250  $\mu\text{g}$  (as per the NIOSH method) using different volumes from a standard suspension were deposited on examples of each filter type. After deposition the filters were placed in a petri dish to air dry at ambient temperature before the FTIR analysis. Absorbance spectra of sample filters were recorded with an FTIR (Thermo Fisher Scientific, Nicolet 6700, Waltham, MA, USA) spectrometer with the Omnic software package (version 8.1.11, Thermo

Fisher Scientific). Absorbance spectra of the quartz and kaolinite samples were analyzed by two different methods: 1) the MSHA P-7 method was used to measure the absorbance peak area with a baseline between 815 and 770  $\text{cm}^{-1}$  for quartz and also to measure the absorbance peak area with a baseline drawn between 930 and 900  $\text{cm}^{-1}$  for kaolinite, and 2) the NIOSH 7603 method was used to measure the peak height of the absorbance band peak at 800  $\text{cm}^{-1}$  with a baseline between 820 and 670  $\text{cm}^{-1}$  for quartz and peak height of the absorbance band peak at 915  $\text{cm}^{-1}$  with a baseline between 960 and 860  $\text{cm}^{-1}$  for kaolinite. Calibration curves of respirable  $\alpha$ -quartz were constructed from the known masses (from aliquot volume of suspension in isopropyl alcohol) of respirable  $\alpha$ -quartz and FTIR absorbance at 800  $\text{cm}^{-1}$ .

#### **Limit of detection (LOD) and Limit of Quantitation (LOQ)**

Twelve of each type of filter were prepared following MSHA P-7 and NIOSH 7603 to determine the LOD and LOQ of the methods. In this case the twelve filters were all blanks; i.e. the aliquot of isopropyl alcohol that ran through filters contained no quartz or kaolinite. Each filter was analyzed by FTIR and the net absorbance at 800  $\text{cm}^{-1}$  was determined. The quartz mass equivalent to the background absorption was calculated using the quartz calibration curve. From measurements of the 12 blank filters the standard deviation of quartz mass was calculated. The LOD and LOQ were determined by multiplying the standard deviation of effective quartz mass by 3 and 10, respectively.

#### **Precision of measurements of quartz samples of known mass**

Five different masses (3, 4, 5, 8 and 10 of quartz based on the concentration of respirable  $\alpha$ -quartz in isopropyl alcohol at 5  $\mu\text{g ml}^{-1}$ ) were loaded onto examples of each type of filter. The

absorbance at  $800\text{ cm}^{-1}$  for each was measured 12 times and the coefficient of variation (CV) was calculated to determine the precision of the FTIR measurement. Recovery of quartz mass was calculated from the average mass determined from FTIR measurements and the known mass deposited by the aliquot of respirable  $\alpha$ -quartz suspension in isopropyl alcohol.

### **Filter penetration**

There is a possibility of sample penetration wherein quartz particles could pass through the sample filter during the re-deposition and be lost to analysis. Penetration of quartz particles for each type of filter was investigated by adding a filter during the re-deposition process of the NIOSH 7603. Three 25-mm nearly circular filters were cut from a single 47-mm filter. Seven 47-mm diameter examples of each type filter were cut into three pieces and the three superimposed during re-deposition such that the last piece served as a background for the FTIR and the first two pieces were analyzed for quartz content. In this case, any significant quantity of quartz that passed through the first filter would be caught on the second. Seven different masses of the quartz ranging from 10 to 250  $\mu\text{g}$  were loaded for each filter type.

### **Pore size measurement (Bubble point test)**

Pore size for each filter was determined by a standard test method for pore size characteristics of membrane filters by bubble point and mean flow pore test (F316-03, American Society for Testing and Materials (ASTM) International).<sup>(9)</sup> The bubble point test determines the minimum air pressure required to force liquid from the largest wetted pore of a filter. The measured pore size rates the filter's ability to serve as a particle barrier. To measure pore sizes a bubble point apparatus was built containing a relief valve, a digital pressure gauge (Entech Instrument Inc.,

Simi Valley, CA) and a 47 mm filter holder with fine metal grating. The initial atmospheric pressure value was measured and deducted from the final pressure required for the formation of bubbles on the liquid (isopropyl alcohol). The pore size of each type of filter was calculated by equation (1) using the surface tension value for isopropyl alcohol of 21 dynes/cm (CRC Handbook of Chemistry and Physics).<sup>(10)</sup>

$$d = \frac{C\gamma}{p} \quad (1),$$

where d is the limiting diameter ( $\mu\text{m}$ ),  $\gamma$  is surface tension (mN/m or dynes/cm), p is pressure in pounds per square inch and C is a constant equal to 0.415. The bubble point pressure was measured three times for five different examples of each of the types of filters.

### **Coal dust sampling**

Pittsburgh coal dust (from the Pennsylvania State University's coal collection via Mr. Jon Volkwein of the Office of Mine Safety and Health Research, NIOSH, Pittsburgh, PA, USA) which had been ground to a fine powder was aerosolized using a fluidized bed aerosol generator (Model 3400, TSI Inc., Shoreview, MN, USA). The generated aerosol was introduced into a calm air chamber through a Kr-85 aerosol neutralizer (Model 3012A, TSI Inc.). Information regarding the particle size-distribution and testing set up have been presented previously.<sup>(10-11)</sup> The respirable size fraction of airborne coal dust were collected using 10-mm nylon cyclones (Sensidyne, Clearwater, FL, USA) loaded with PVC filters (GLA-5000, SKC Inc.). Personal air sampling pumps (PCXR4 and PCXR8, SKC Inc.) were connected to the 10-mm nylon cyclones. The flow rates ( $1.7 \text{ l} \cdot \text{min}^{-1}$ ) through the samplers were calibrated using a Gilibrator-2 calibration device (800271, Sensidyne), using a calibration jar (225-112, SKC Inc.). The flow rates were

calibrated before and after sampling to ensure the flow rate did not change by more than  $\pm 5\%$ .

Three different levels of coal dust mass (to provide approximately 0.5, 1 and 1.5 mg per sample) were tested with at least two repetitions for each level. The PVC filters were equilibrated at least 72 hours in a weighing room at constant relative humidity and temperature. Then the filters were passed through an electrostatic bar (Mettler-Toledo, Columbus, Ohio) before weighing to dissipate static charge. Weighing was performed with a micro balance (XP6U, readability 0.1  $\mu\text{g}$ , Mettler-Toledo) for PVC filters. The coefficients of variation (CV) of multiple weighings of PVC blank filters ranged from 0.03 to 0.06 %.

#### **Quartz measurement in coal dust**

After collection of coal dust in the calm air chamber, the pre-weighed filters with coal dust were weighed again. Then the filters were placed in a 50 ml beaker and low-temperature ashed in a Plasma Prep II (Model 11005; SPI Inc., West Chester, PA, USA) reactor for 3 hours. The residue from the ashed filters was gently suspended in 10 ml isopropanol and sonicated for 5 minutes and then re-deposited on each filter as described by the MSHA P-7 and NIOSH 7603 methods. Quartz masses were determined by the MSHA P-7 and NIOSH 7603 methods. In order to determine the instrumental precision (Coefficients of variance (CV)) of the MSHA P-7 and NIOSH 7603 methods, the quartz mass from each filter was measured 12 times.

## **RESULTS**

#### **Calibration curves, LODs and LOQs**

The slopes of calibration curves forced through zero along with regression coefficient ( $R^2$ ), LODs and LOQs of respirable  $\alpha$ -quartz for each type of filter as determined by MSHA P-7 and NIOSH

7603 methods are shown in Table 2. The LOD's measured here varied from 0.59 to 1.5  $\mu\text{g}$ . FTIR spectra between 1000 and 660  $\text{cm}^{-1}$  from 12 different blank filters of each type filter are shown in Figure 1. Each sample was measured 12 times to determine the LOD and the sample was removed from sample holder and put back for the next measurement. This ensures that the measured precision reflects real world filter handling. The background absorbances of the blank filters in spectral regions adjacent to analytical wavelength for quartz (800  $\text{cm}^{-1}$ ) and kaolinite (915  $\text{cm}^{-1}$ ) (dotted lines) of each filter type were small, a few hundredths of an absorbance unit, and were relatively constant over the spectral region.

#### **Precision measurement of known quartz mass samples**

Percentage of recovery of quartz and coefficients of variance (%) in quartz spiked samples are shown in Table 3. The CV decreases as the quartz mass is increased in most of the filters.

#### **Filter penetration**

No evidence of penetration of the quartz particles through the filters was found based on IR analysis of the second filters. This is somewhat surprising for the 5  $\mu\text{m}$  PVC filters but is supported by the recoveries of the low mass standards on the primary filter; i.e. there was no detectable quartz signature on the second filters.

#### **Bubble Point Test**

Pore size determination by the Bubble Point test with isopropyl alcohol yielded a pore size of  $0.847 \pm 0.004 \mu\text{m}$  for the nylon filter,  $0.755 \pm 0.002 \mu\text{m}$  for PP filter and  $5.08 \pm 0.102$  for the PVC filter.

**Quartz measurement in coal dust**

A total of 36 samples (12 samples for each filter) were collected and the masses of respirable coal dust on them ranged between 0.35 and 1.6 mg. Quartz masses determined by FTIR analysis ranged between 16 - 107  $\mu$ g. A linear regression comparison of quartz mass as determined by MSHA P-7 and NIOSH 7603 showed a near unity relationship for all filter types, a slope of regression of 1.04 and regression coefficient ( $R^2$ ) 0.978.

The CVs for measurements using all the filter types combined were found to be in the range of 0.4-10 %. The CVs from three types of filters were significantly different from each other for both MSHA P-7 and NIOSH 7603 methods (Mann-Whitney Rank Sum Test; SigmaPlot for Windows Ver. 12.5, Systat Software Inc.). The nylon filters showed the largest CVs in coal dust samples followed by PP and PVC filters. Quartz masses from the samples were divided by net mass of coal dust and multiplied by 100 for the calculation of quart content (%). The average quartz contents as determined on nylon, PP and PVC filters with MSHA P-7 were 5.6, 5.5 and 6.6 %, respectively. The average quartz contents as determined on nylon, PP and PVC filters with NIOSH 7603 were 6.1, 6.3 and 6.8 %, respectively. Quartz content between filters and methods did not show a significant difference as determined by a Mann-Whitney Rank Sum Test.

**DISCUSSION**

The FTIR spectral results from the blank filters in the present study are comparable to what was found in a previous study in which PVC and DM-450 filters showed similar spectra (no IR peaks that would grossly interfere with measurement of 800 and 915  $\text{cm}^{-1}$ ).<sup>(6)</sup> The nylon filter did not

provide even particle deposition for coal dust on filter in all re-deposition arrangements, i.e. when one or two filters were superimposed during the re-deposition procedure (NIOSH 7603). The samples with nylon filters used for calibration curves may also have had uneven deposition of 1878a on the filter but if so it was not detectable by visual inspection. Examples of uneven and even particle deposition of ashed coal dust on (a) a nylon filter, (b) a PP filter and (c) a PVC filter are shown in Figure 2. The PP filters showed even particle depositions except for one sample with the NIOSH 7603 method and no uneven particle deposition was found with the MSHA P-7 method. In general, uneven particle deposition is to be avoided because it may create random variability between standards and samples with regard to the optical properties of the filter and sample. In fact, the CVs of measurements of various masses of spiked quartz dust deposits on nylon filters were significantly larger than those of measurements on filters of other materials where the deposits were visibly more uniform. But characteristics other than non-uniform deposition on the nylon filters may contribute to their larger CVs.

The selection of the pore sizes of the filters was based on matching as closely as possible the pore size of the DM-450 filter, but no PVC filter was available near that pore size. Since no particle penetration in the re-deposition procedure was found for the PVC filters, it was determined that larger pore sizes of nylon and PP filters might possibly be useable for quartz measurement in coal dust with FTIR. Larger pore sizes of the nylon filter (0.8, 1.2, 5.0  $\mu\text{m}$  pore size) and PP filter (10  $\mu\text{m}$  pore size) are commercially available. However, nylon filters with 5.0  $\mu\text{m}$  pore size and PP filters with 10  $\mu\text{m}$  pore size showed significant particle penetration and they were not evaluated further.

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The estimated LOD reported in a NIOSH document<sup>(4)</sup> was 2.9 µg measured with 20 blank DM-450 filters and the instrumental LOD was 0.4 µg measured with 10 repetitive measurements of a single DM-450 filter. The LODs reported this study are comparable to those reported in a previous study i.e., 0.6 to 2 µg depending on the filters and analysis methods (Table 2).

## CONCLUSION

Three different filters including nylon, polypropylene and polyvinyl chloride filters were characterized for possible use of quartz measurement in coal dust with FTIR analysis in accordance with MSHA P-7 and NIOSH 7603. Quartz measurements made with polypropylene (pore size 0.45 µm) and polyvinyl chloride (pore size 5.0 µm) filters were similar to those made with the vinyl/acrylic co-polymer (DM-450) filter currently used in standard methods for analysis of quartz by FTIR. Therefore they may be suitable substitutes. The nylon filter did not perform adequately as shown by the larger variation. The ability of 5 µm pore size PVC filters to retain fine particulate is an unexpected finding and should be confirmed in filter experiment. However, PP filters are suitable replacement with appropriate pore size.

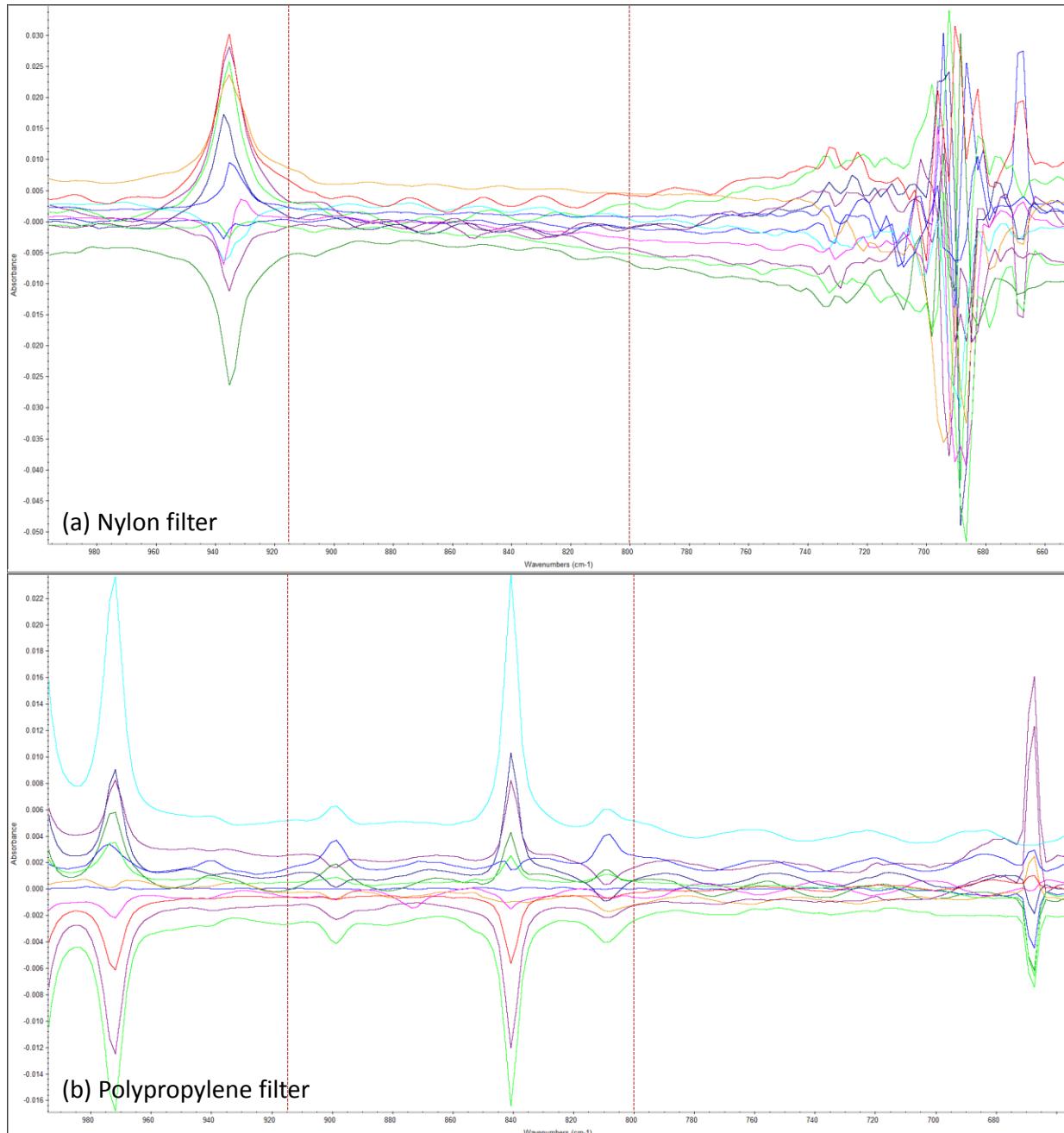
## DISCLAIMER

The findings and conclusion in this report are those of the author(s) and do not necessarily represent the official position of the Center of Disease Control and Prevention/ the Agency for Toxic Substances and Disease Registry.

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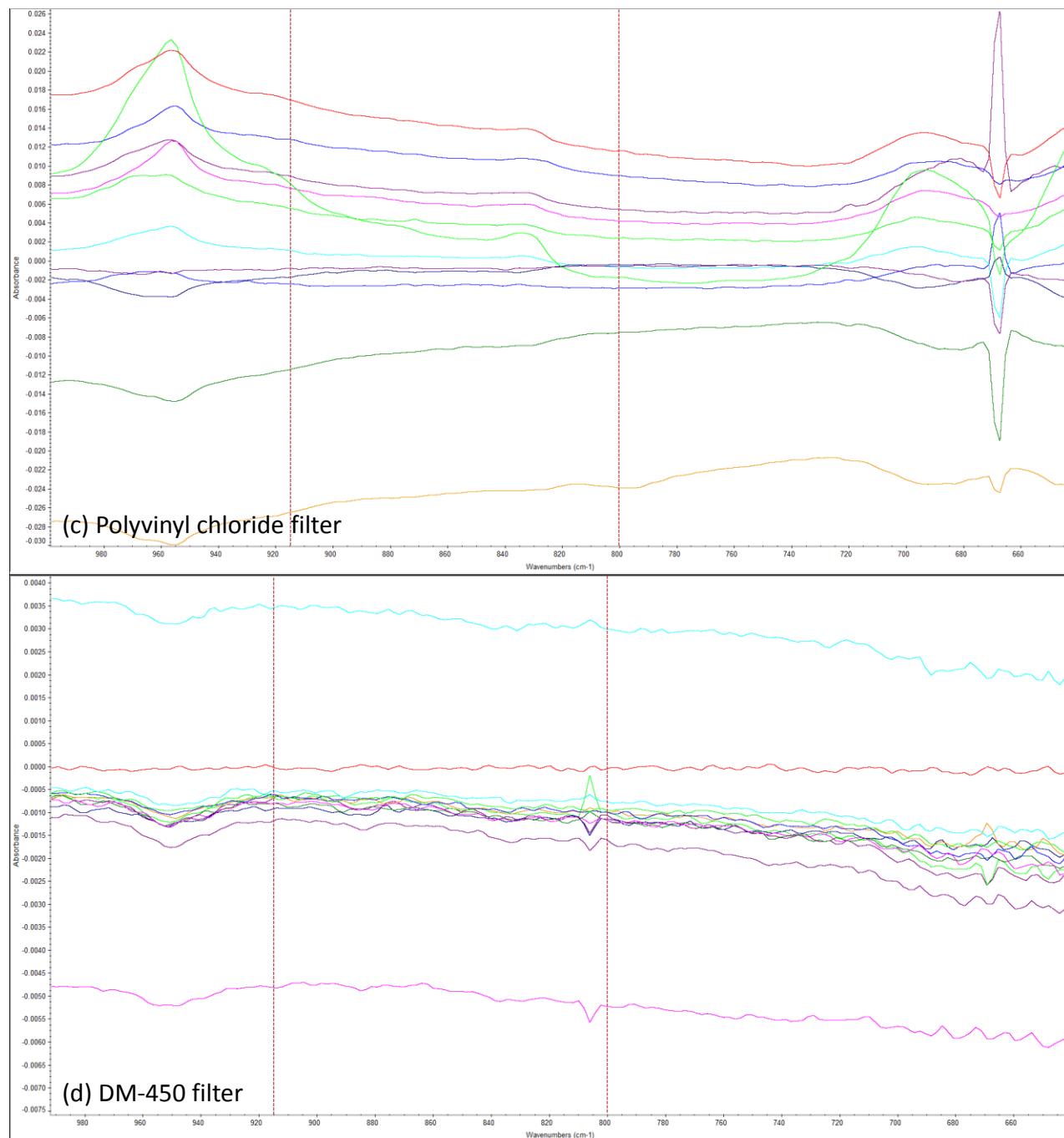


Figure 1. FTIR spectra from 12 different blanks of (a) nylon, (b) polypropylene, (c) polyvinyl chloride and (d) DM-450 filters in wavenumber region of  $1000\text{-}660\text{ cm}^{-1}$ .

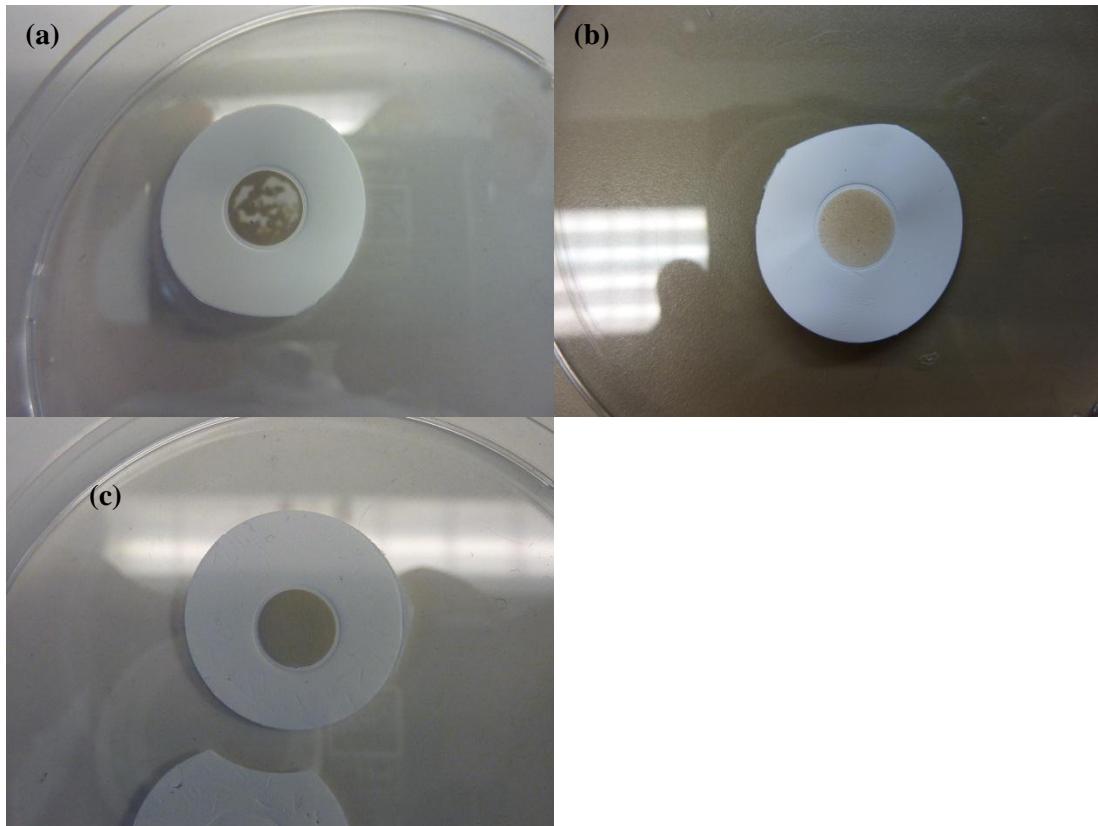


Figure 2. Example pictures of (a) uneven particle deposition on nylon filter deposited using NIOSH 7603, (b) even particle deposition on a polypropylene filter deposited using MSHA P-7 method and (c) even particle deposition on a PVC filter deposited using NIOSH 7603

Table 1. Information on filters tested in the present study

Filter type	Diameter (mm)	Pore size (μm)	Manufacturer	Reference number
Nylon	47	0.45	Millipore	HNWP04700
Polypropylene	47	0.45	Sterlitech	PP04547100
Polyvinyl chloride (GLA-5000)	47	5.00	Zefon	FPVC547
Vinyl/acrylic co-polymer (DM-450)	47	0.45	PALL Life Sciences	T10634

Table 2. Slopes of calibration curves, method limit of detections and limit of quantifications for each filter type.

Method	Filter type				
	Nylon	Polypropylene	PVC	DM-450	
Slope of calibration curve ( $R^2$ )*	MSHA	0.0538 (0.999)	0.0505 (0.999)	0.0492 (0.985)	0.0546 (0.996)
	NIOSH	0.0025 (0.998)	0.0023 (0.999)	0.0021 (0.945)	0.0021 (0.999)
LOD ( $\mu\text{g}$ )	MSHA	1.8	0.72	0.52	0.99
	NIOSH	1.5	1.2	1.5	0.59
LOQ ( $\mu\text{g}$ )	MSHA	6.0	2.4	1.7	3.3
	NIOSH	4.9	4.0	4.9	2.0

$R^2$  is the correlation coefficient.

Table 3. Recovery and coefficient of variance in spiked samples of respirable  $\alpha$ -quartz suspension in isopropyl alcohol.

Method	Filter type	Calculated quartz mass by volume of the suspension ( $\mu\text{g}$ )				
		3	4	5	8	10
		Quartz recovery (%) and CV (%)				
MSHA P-7	Nylon	103/4.9	94/4.3	97/6.0	106/2.7*	100/1.9*
	PP	100/6.9	105/2.0*	105/2.0*	100/1.6*	106/1.6*
	PVC	110/3.6	95/2.5*	113/2.6*	105/1.8*	104/0.7
	DM-450	104/1.6*	98/6.7	100/5.0	96/1.9*	98/1.0*
NIOSH 7603	Nylon	67/12	84/11	100/6.9	101/3.5	101/4.7
	PP	84/4.8	84/2.9	112/4.7	106/4.3	104/7.0
	PVC	114/4.5	116/3.8	112/4.2	118/1.8*	109/2.1*
	DM-450	119/6.9	106/3.4	94/3.1	96/2.8*	98/2.1*

\*All 12 measurements were within 5% of the mean quartz mass.

Table 4. Quartz mass in coal dust samples and coefficient of variance.

Sample	Quartz mass in coal dust (µg)		CV (%) <sup>**</sup>	
	MSHA P-7	NIOSH 7603	MSHA P-7	NIOSH 7603
Nylon1*	56	63	5.9	6.9
Nylon2	90	989	2.0	3.1
Nylon3	100	107	7.3	3.2
Nylon4*	40	45	6.3	6.8
Nylon5*	66	72	2.0	4.5
Nylon6*	46	51	10	7.4
Nylon7*	47	49	5.9	6.2
Nylon8*	63	71	3.4	3.3
Nylon9*	29	31	3.8	4.3
Nylon10*	16	21	5.7	4.5
Nylon11*	54	58	6.5	3.3
Nylon12*	24	26	5.6	3.8
PP1	79	89	2.1	3.2
PP2	101	107	3.9	4.6
PP3	92	94	2.1	3.3
PP4	92	105	1.4	2.5
PP5	55	65	1.0	2.0
PP6	36	42	0.55	1.2
PP7	64	75	3.7	4.5
PP8	56	71	7.6	3.0
PP9	19	26	3.1	3.4
PP10	30	32	0.7	0.87
PP11	47	55	2.7	1.4
PP12*	45	49	8.0	6.9
PVC1	71	73	0.34	0.67
PVC2	90	92	0.56	0.65
PVC3	96	94	0.28	0.43
PVC4	52	54	1.8	2.00
PVC5	65	70	0.85	0.99
PVC6	84	90	1.07	1.2
PVC7	71	74	0.39	0.48
PVC8	39	39	1.65	2.3
PVC9	34	33	0.51	0.76
PVC10	39	39	1.65	1.5
PVC11	41	48	1.53	1.6
PVC12	39	38	1.23	1.3

\* sample showed uneven particle distribution

\*\* Coefficients of variance from 12 different FTIR measurement for each sample