Analysis of particulate contamination in personal dust monitor sampling

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ABSTRACT: The National Institute for Occupational Safety and Health (NIOSH) has continued study of the Personal Dust Monitor (PDM) developed by Rupprecht & Patashnick Co. Inc. through a CDC contract and currently being commercialized by Thermo Fisher Scientific. This report examines the effect of oversize particles, cyclone inversion, and cleaning procedures. Clean and exposed filters were examined using light microscopy and particulates counted before and after various instrument manipulations. The data presented show that the dry cleaning procedures do not cause significant (greater than limit of detection) sample contamination by respirable or oversize particulates and that the cyclone is resistant to particulate carry over during inversion.

1 Introduction

The personal dust monitor (PDM) is a continuous measuring dust sampler incorporated into a miners cap lamp. The PDM represents a new way to determine personal respirable coal mine dust exposure in a continuous way. The timely data and electronic records produced will provide significantly more information to miners than currently possible using conventional dust samplers. This new technology will inevitably raise questions regarding the meaning and interpretation of the data. The EPA has gone though similar processes in converting filter type air pollution measurements to continuous monitoring systems measurements. The original data remains, but new ways to understand the impact of the additional information had to be developed. This understanding comes from use of the instruments. Published data on the PDM confirm that it measures personal dust exposure of miners plus many other parameters. Our understanding and interpretation of these findings is on the rapidly rising portion of the learning curve. This report addresses questions regarding the appearance and significance of large particles on PDM filters, the handling of filters, instrument cleaning and their impacts on measurement accuracy.

2 Oversize Particulates

The 1995 NIOSH criteria document contains two definitions of relevance to respirable dust. The first is the definition of respirable coal mine dust being: "That portion of airborne dust in coal mines that is capable of entering the gas-exchange regions of the lungs if inhaled; by convention, a particle-size-selective fraction of the total airborne dust; includes particles with aerodynamic diameters less than *approximately* (emphasis added) 10 µm." The document also defines the respirable convention to be: "The target sampling curve for instruments approximating the respirable fraction." The definition continues with the theoretical mathematical equations.

MSHA, by convention, defines oversize particulate as being greater than 10µm in diameter. When more than 30 such particles are found by microscopic examination of approximately 2.5 mm^2 of filter surface area of a 37 mm sampling filter, then the sample is voided due to the presence of oversize. The value of 30 oversize particles originated from a calculation that it would take 9615 particles of 20 µm diameter coal at a density of 1.3 $gram/cm^3$ to change the mass on the filter by 0.1 mg.

2.1 Sources of Oversize Particles on Filters

The presumed sources of oversize particles in the current sampler were from improperly assembled samplers, dumping of cyclones,[1] or other mishandling of the sample. These sources are minimized in the PDM since the sampling train undergoes minimal disassembly for cleaning purposes and the BGI cyclone design (BGI, Inc., Waltham, MA) used in the PDM is resistant to dumping. Mishandling of filters is minimized as a source of oversize particles since the filter never is exposed to the atmosphere outside of the PDM during the time at which the filter mass is determined by the electronics of the instrument. If the filter area of the sampler is ever opened during sampling, an error is displayed on the screen and a record of the event occurs in the data file.

There are then three ways in which oversize particles can be deposited onto the PDM filters during sampling: First, the actual penetration through the BGI cyclone; second, the release of previously collected and agglomerated respirable dust in the instrument that was not removed during cleaning of the section between the

[1] Dumping is defined as the act of inverting the cyclone such that oversize particles captured in the cyclone grit pot reenter the sampling air stream and become deposited on the filter.

cyclone outlet and the filter; and third, the remote possibility of a cyclone dump.

2.2 Penetration Through a Cyclone

Figure 1 shows the theoretical lung penetration curves of the Mining Research Establishment (MRE) sampler, the former American Conference of Governmental Industrial Hygienists (ACGIH) Dorr-Oliver cyclone having at a 2 lpm flow rate, and the International Standards Organization (ISO) BGI cyclone flowing at 2.2 lpm. MRE samplers use gravity to separate respirable dust from an air sample and have a sharp cut off at about 7 µm in aerodynamic diameter. Cyclone samplers do not have a sharply defined cut off but instead have an asymptotic decay in the upper size range. Table 1 shows the fraction of particles by aerodynamic size that will be classified as respirable mass by the cyclones. This means that a dust sample, properly classified by cyclone, will have a small but finite number of particles greater than 10 µm in diameter.

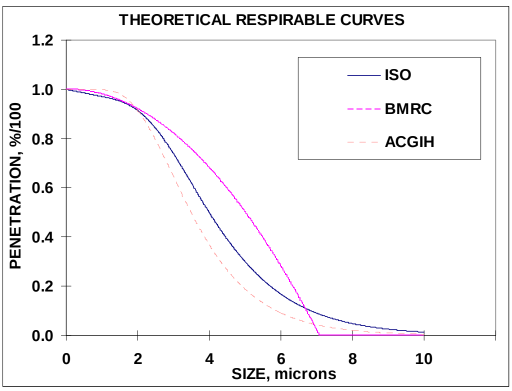

Figure 1. Classification criteria

The other consideration when attempting to determine the presence of oversize particles on a sample filter is the fact that optical microscopic examination of a filter can determine only the equivalent circular diameter of a particle. Particle aerodynamic diameter is used to measure the actual way in which the dust moves in air and becomes deposited in the lung. The aerodynamic diameter is defined as a particle having the same movement in air as a spherical unit-density particle. Low density particles may in fact have very large equivalent circular diameters yet still be classified as respirable. Measuring equivalent circular diameter of a particle on a filter cannot accurately determine if a particle is respirable or not without measurement of that specific particle's density and shape factor.

2.3 Release of Agglomerated Respirable Dust

Another way in which oversize particles may be deposited on the filter during sampling is if cyclone-classified respirable particles deposit and agglomerate in the

Table 1. Calculated fraction of particles of a particular size that may be deposited on sample filters.

Aerodynamic diameter µm	Percent of ISO classified as respirable %	Percent of ACGIH classified as respirable %	Percent of MRE classified as respirable
3.5	62	50	75
4	50	37	68
10	1.3	0.48	0
12	0.39	0.19	0
14	0.12	0.032	0
16	0.04	0.01	0
18	0.01	0.003	0
20	0.005	0.001	0

instrument's internal plumbing between the outlet of the cyclone and the filter, are not removed by the current cleaning procedures, are not removed during the 30 minute -- non-sampling -- instrument warm-up period, and become dislodged during sampling. RI 9669 discussed the loss of particulate in this portion of the instrument to be a part of the instruments overall 6.6% negative bias. It was not determined how much of this loss was cleaned from the instrument during each cleaning with compressed air between each day of sampling. The compressed air cleaning method in the laboratory used a laboratory dry and oil free compressed air sources or in the field used cans of compressed air. The cyclone grit pot and tapered element filter holder were removed from the instrument and compressed air was sprayed into all exposed openings. The grit pot was also cleaned by spraying with compressed air.

On properly cleaned units, there remains a thin film of coal mine dust on the internal surfaces, so the possibility exists for some portion of that dust to end up on a subsequent sample, either as agglomerated oversize or as respirable dust. Experiments by Hinds (1982) show that particle attachment to surfaces is proportional to the particle diameter (d), while removal forces are proportional to d^3. This means that as particle size decreases, the force to remove those particles increases as the cube root. For practical purposes, particles less than10µm in size are not likely to be removed by common forces such a vibration or sampling air flows. However, if these particles accumulate to the point where a layer of 0.1 to 10 mm forms, then they can more readily be dislodged as agglomerates. Dust films observed on internal sample lines in properly cleaned units did not approach this deposit thickness. The NIOSH compressed air cleaning method is presumed to remove those thicker deposits that could be readily dislodged during sampling.

2.4 Measurement of Particulates Released by Motion Forces

The question of whether the act of wearing the PDM could generate sufficient forces to release oversize or respirable dust from a previously collected sample was also studied. An apparatus was constructed to continuously bend and flex the inlet tubing to the instrument while simultaneously raising and dropping one end of an upright PDM every 13 seconds for a period of eight hours. The height of the drop was about two inches onto the laboratory floor. The PDMs used for this test were returned from a field project where they each had been used for about 144 hours over a four week period. The PDMs were maintained by mine personnel and cleaned according to the latest NIOSH cleaning procedures. In the laboratory, groups of three PDMs at a time were tested due to weight limitations of the test apparatus.

New PDM filters were used in this experiment. These filters were examined by an optical microscope using transmitted light at a magnification of 32X. A minimum of 30 optical fields were observed and particles greater than 20µm in size were counted as oversize particles. Filters counted in this way were called certified. Filters were then run in the PDM as described below and were post counted according to same procedures by the same analyst. Figure 2 shows some examples of filter pre and post counts. For each test, a high efficiency particle air (HEPA) filter was placed on the PDM inlet. The PDM was started and programmed to run for 8 hours. Optically certified filters were in place when the units were turned on and run during instruments normal 30 minute warm-up. At 25 minutes after the start of the warm-up, filters were replaced with a second optically counted filter for the subsequent sampling period. The PDM self zeros at the end of the warm-up period and no mass that may have been deposited during warm-up is recorded.

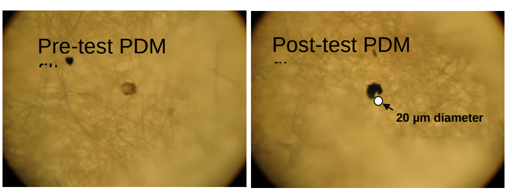

Figure 2. Typical pre and post count PDM filters with examples of oversize particulates observed.

After the second certified sample filters were installed, the PDM was placed on the apparatus and the motion and bumping action was begun. At the end of the testing the filters were removed and post counted and the PDM data file was downloaded. Table 2 contains the results of pre and post optical counts of the number of oversize particles on the filters. The companion-PDM reported-mass value (MASSO TOTAL) was also recorded. The mass data indicated that two samples were close to the 0.022 mg limit of detection of the PDM. Unfortunately, two of PDM's failed to record mass in this testing, but the optical data for this test was still valid.

The conclusion from the data in Table 2 was that no statistically significant difference in oversize particle pre and post counts for the 8-hour sampling period was observed. There was also no quantifiable mass recorded by the PDM. The small mass gain observed is most likely a positive filter bias previously observed and reported Page et al. (2007).

2.5 Anecdotal Testing

At MSHA's request, NIOSH examined one of their PDM field units, using the same procedures as above. The recent history of the unit involved at least 96 hours of testing in various District 7 mines, some of which operated diesel equipment. The warm-up filter contained no significant difference in particulates between pre and post test counts.

The mass recorded during the sampling period of this test was 0.0084 mg, however, the optical counts of particulates greater than 20µm in size were significantly more. The pre-test count was nine particles in 34 fields versus a post-test count of 240 particles in 34 fields. Upon closer examination using variable focusing of the post-test particles, it was seen that the particulates were low density agglomerates that looked very similar to diesel particulate structures (Figure 3). Also of note was that the particulates were all about 40 to 55µm in size. Researchers conclude that the observed particles were large (greater than 40 µm), low density agglomerates that were likely released by the flexing of the inlet tube and that they were of sufficiently low density to penetrate the cyclone, but not to contribute in a substantial way to the mass on the filter. This is an anecdotal result and not much confidence is placed in the result or conclusion until it can be reliably repeated and the density of the particulates determined.

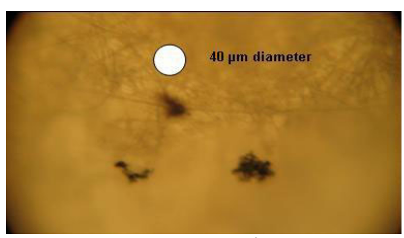

Figure 3. Example type of particulate released from MSHA unit #116 during motion testing.

Table 2. Results of PDM filters sampling HEPA filtered air and subject to 8-hours of continuous movement and bumping.

			Pre-test		Post-test		
TEST	**PDM #**	**Filter #**	**Fields**	**Oversize particles**	**Fields**	**Oversize Particles**	**MASSO TOTAL**
1	105	PT1AG	36	3	34	4	-0.011
1	112	PT1AH	38	6	36	4	-0.007
1	113	[1]PT1AI	36	2	36	4	-
2	110	PT1AJ	34	18	36	16	-0.004
2	126	PT1AK	34	2	36	4	0.006
2	127	PT1AL	32	15	36	18	0.027
3	115	[2]PT1AM	32	2	38	10	0.003
3	132	PT1AN	36	5	34	6	0.028
3	133	[3]PT1AO	32	9	38	9	-
						Average	0.006

NOTE
[1] Damaged filter when removing from TE after the test. Flat line data
[2] PDM 115 stopped operating after 6 hours and 43 minutes mass recorded to that point.
[3]PDM133 data periodically reported "TE not detected" from 11:42AM on.

3 Cyclone Inversion

Guidelines for using the current sampler with the Dorr-Oliver cyclone have recommended that the cyclone, with close-coupled filter, not be rotated more than 120 degrees from vertical. This is to prevent the possibility of oversize particles already separated from the air and retained in the cyclone grit pot from falling back into the cyclone body and being deposited onto the filter. In reality, the centrifugal forces generated inside of the cyclone are much greater than the force of gravity and should prevent gravity from moving oversize particles from the grit pot onto the filter.

The likelihood of cyclone dumping being a source of oversize in the current sampler is remote. Nevertheless, it is possible and the guidelines for not tipping the current cyclone are appropriate. A dumping event with the PDM is even more unlikely because the filter is remote and not close coupled to the cyclone and no direct path exists between the grit pot and the filter. Furthermore, the BGI cyclone grit pot is larger and contains a larger lip to prevent dumping. Finally, a significant dump of a cyclone would result in a rapid, step-change in the mass loading data file which could be readily detected in the data file.

An experiment was conducted where 1 gram of monodisperse 20 µm glass spheres were placed into the grit pots of PDM and Coal Mine Dust Personal Sampling Unit (CMPDSU) Dorr-Oliver cyclones. The density of the spheres was 2.5 g/cm^3 which results an equivalent aerodynamic diameter of 32 µm. The PDM, with an optically certified filter, was turned on and programmed to run for one hour. During sampling, the PDM was inverted along its longitudinal axis for 20 complete rotations in each direction. It was then inverted on its transverse axis for 20 complete rotations in each direction. At the end of the sample period the filters were optically examined for oversize particles. The same test was conducted for the CMPDSU with its sampling pump running.

Results in Table 3 demonstrate that the PDM filters had some level of oversize contamination on the filters prior to the test, probably as a result of the way in which the filters are packaged. Post test counts, however, were not statistically different ($p = 0.124$) from the pretest counts. The MSA filters used in the CMPDSU were very clean and showed no oversize particles in either pre or post counts. In no cases were 20 µm spherical particles detected.

4 Observations of Oversize Particulate on Personal Samples Taken with A Prototype PDM

Tests with six prototype PDMs by Volkwein et al. (2003) involved underground sampling with side-by-side gravimetric filter samples. Inlet shape, length of conductive tubing, and BGI-4CP cyclones, identical to the PDM configuration, were used to collect gravimetric filter samples in parallel with the PDM. Results showed no difference in mass collection between the two systems. Filters from the PDM, companion gravimetric filters, and blank filters from this testing were preserved and stored at NIOSH.

Table 3. Filter counts of dumping experiments with glass spheres

PDM FILTERS					
PRE-DUMP FILTER DATA			POST-DUMP FILTER DATA		
	microscope			microscope	
Filter	Fields	particles	Filter	fields	Particles
PDM filter #1	37	16	PDM filter #1	36	12
filter #2	36	4	filter #2	36	9
filter #3	32	6	filter #3	36	5
filter #4	31	0	filter #4	38	3
filter #5	32	10	filter #5	36	15
SUM	168	36		182	44

CMDPSU FILTERS					
PRE-DUMP FILTER DATA			POST-DUMP FILTER DATA		
	microscope			microscope	
Filter	Fields	particles	Filter	fields	particles
MSA			MSA		
#2345	62	0	#2345	76	0
#3656	42	0	#3656	74	0
#3657	54	0	#3657	72	0
#3658	55	0	#3658	70	0
#3659	55	0	#3659	74	0
	268	0		366	0

4.1 Prototype Filter Examination

Filters from the prototype work were retrieved from storage and sent to the MSHA technical support laboratory for microscopic analysis of oversize particles similar to their standard methods. Optical counting techniques were used for the 37 mm gravimetric and 13 mm PDM filters such that an equivalent surface area proportional to each filter diameter was used to count oversize particulates on the filters. Both blank control filters and filters worn by miners and NIOSH researchers were examined. Similar cleaning procedures were used for the prototype PDM testing. The prototype PDMs had about 200 hours of use with routine cleaning prior to this examination.

4.2 Prototype Results

Results of the optical examination of the prototype test filters for particulates greater than 20 µm are in Table 4. None of the gravimetric filters met the MSHA excessive oversize criteria of 30 particles in 10 fields. The PDM filters showed fewer oversize particles than the gravimetric samples. This could occur because the dust deposit on the smaller PDM filter must be thicker -- for an equivalent mass-- presenting the possibility that oversize may have been hidden. If this was true, one would expect to see a proportion of 5:32 (proportional to the counting areas of 10:64 for PDM vs 37 mm filters) of the number seen in the gravimetric filters. When data are normalized for the thickness of deposit, only sample M4 showed slightly more oversize on the PDM filter than the gravimetric. These filters were not expected to be used for this examination and no particulate counts were done on the filters prior to sampling. However, control filters show little contamination with oversize particulates and researchers have some confidence that the particles observed were a result of the sampling or post sampling handling process. The results in Table 4 indicate that no oversize particle contamination from the use and cleaning of the prototype PDM after 200 hours of use occurred.

5 Significance of Cross-Sample Contamination

Putting some perspective into the significance of filter contamination, filters were prepared with dust masses less than the minimum detection limit and microscopic photographs were taken of the filter deposits. To collect a deposit below the limit of detection, a known and steady dust concentration was created in the Marple chamber. Three PDM inlets were placed in the chamber while sampling with HEPA filters over the inlets. Using the concentration measured by a tapered element oscillating microbalance (Model 1400, TSI, Inc. Shoreview, MN) in the Marple chamber, the time required to deposit nominally 0.010, 0.020 and 0.030 µg of respirable coal dust onto the filters was calculated. The PDM HEPA inlet filters were removed and the PDMs sampled the atmosphere for the calculated time intervals before the HEPA filters were replaced.

Figure 4 shows the overall appearance and Figure 5 shows the microscopic appearance of the blank and exposed filters at various low level mass loadings. Comparing these microscopic images to the previous photographs of oversize and post cleaning filter results, we can conclude that a substantial amount of respirable dust is required to reach the 0.022 mg limit of PDM detection. This level of contamination was not observed in any of this testing.

6 Conclusion

The PDM represents a new way to determine personal respirable coal mine dust exposure. Many sources of error have been eliminated by the PDM warm-up procedure, automatically obtaining the tare weight of the filter, and measuring the final end-of-shift mass, all performed without human intervention. The current design of the PDM precludes comprehensive cleaning using detergent and ultrasonic techniques, commonly used with the CMPDSU. Alternative dry cleaning using compressed air has been developed and used in the NIOSH studies. Results presented in previously by Volkwein et al. 2006 demonstrated that the units remained accurate after an average of 437 hours of testing using these dry cleaning procedures. The data presented above further indicate that the dry cleaning procedures do not cause significant (greater than limit of detection)

Table 4. Prototype PDM and companion gravimetric filter oversize particle counts.

Sample	PDM filter No	Oversize Particles >20 µm Count	Companion gravimetric filter No	Oversize Particles >20 µm Count
Control	77	1	93	0
Control	119	0	156	0
M1	113	0	148	10
M4	115	4	156	9
M1	71	1	83	7
Ca	75	4	87	29
Sh	76	0	90	14
xx	99	0	121	11
Control	98	2 (f)	130	0
Control	112	0	147	0
P-1	85	0	103	5
P-2	96	1	116	26
P-3	111	3	143	21

(f) = fibers

PDM filters - 10 fields of 0 25mm²

PVC filters - 64 fields of 0 25mm²

Figure 4. Appearance of full PDM filter
After sampling low mass loadings of coal dust.

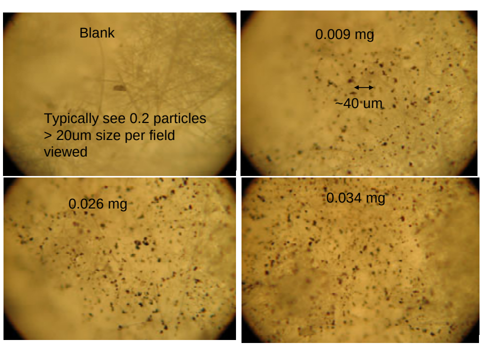

Figure 5. Microscopic appearance of showing the numbers of dust particles per area required to achieve the indicated mass.

sample contamination by respirable or oversize particulates and do not cause significant (greater than limit of detection) response to the final end-of-shift personal dust exposure of a miner.

Acknowledgements

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