# CHAPTER 5 MINERAL DUSTS - GRAVIMETRIC METHOD



<b>Table of Contents</b>				
I.	Introd	oduction		
II.	Definitions		5 - 1	
III.	Threshold Limit Values (TLVs®)			
	A.	Respirable Dust	5 - 2	
	B.	Additive Respirable Exposure Formula	5 - 4	
	C.	Nuisance Dusts	5 - 4	
IV.	Respirable and Total Dust Sampling Equipment			
	A.	Filter-Cassette	5 - 5	
	B.	Sampling Pump	5 - 6	
	C.	Respirable Dust Sampling Train	5 - 7	
	D.	Total Dust Sampling Train	5 - 9	
V.	Respirable and Total Dust Sampling Procedures			
	A.	Pre-survey Calibration of Sampling Pump	5 - 11	
	B.	Control Filter Cassette	5 - 11	
	C.	Assemble the Sampling Train	5 - 12	
	D.	Attach the Sampling Train to the Miner	5 - 13	
	E.	Instructions to the Miner	5 - 13	
	F.	Collect the Sample	5 - 14	
VI.	Post-Inspection Procedures			
	A.	Review Health Field Notes	5 - 16	
	B.	Post-survey Calibration of Sampling Pump	5 - 16	
	C.	Cyclone Cleaning and Assembly	5 - 16	
	D.	Transport the Samples for Analyses	5 - 17	
	E.	Compliance Determination	5 - 17	
	F.	Report Writing	5 - 17	
Figure	5-1.	Filter Cassettes, Lots, and Storage Bags	5 - 6	
Figure		Respirable Dust Cyclone and Holder Assembly	5 - 7	
Figure	5-3.	Respirable Dust Sampling Train	5 - 8	
Figure		Total Dust Sampling Train	5 - 9	
Figure	5-4a	Total Dust Cassette Holder Option	5 - 10	

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# Chapter 5 MINERAL DUSTS - GRAVIMETRIC METHOD

#### I. Introduction

In most cases, exposure to mineral dust is determined by the gravimetric method, using a sampling pump and filter cassette (pre-weighed) to determine dust concentrations in the mine atmosphere. Gravimetric sampling is used to determine personal exposure (breathing zone) and can also be used to screen for other mineral dusts that may require particle counting to determine compliance, such as the six silicates specified in Chapter 6 (non-asbestiform talc, perlite, natural graphite, mica, soapstone, and amorphous silica or diatomaceous earth).

#### II. Definitions

**Cyclone** - also called "cyclone separator" or "particle sizer" – is a dust collecting device which separates particles by size and is used as part of the respirable dust sampling train. As air enters the intake hole, or "vortex," respirable dust swirls in a conical device toward a filter cassette where it is trapped. The larger dust particles fall to the bottom and are collected in the "grit pot."

**Filter Lot** - all of the filter cassettes with the same pre-weight date. A list of the filter lots is provided by MSHA Technical Support and is periodically updated. The list contains a range of filter numbers, by date, for each filter lot. Unused filters may be used for another sampling shift if they have not been individually opened and are in the same filter lot. Double-check the filter list to confirm the lot.

**Gravimetric Method** - a sampling procedure that measures the weight of the dust collected on the filter media to determine the concentration of the contaminant in the miner's breathing zone. Most mineral dusts are sampled gravimetrically.

**Respirable Dust** - particles suspended in the air with a size of less than or equal to 10 microns in diameter. Although these dusts are invisible, they can be highly toxic because their small size allows them to penetrate to the deepest areas of the lungs and cause pneumoconiosis. Some examples of respirable dusts are silica, coal, and metal ore dusts.

Respirable dust is identified as that fraction of dust that passes a size selector (MSHA uses a 10mm nylon cyclone) and has the following characteristics:

Aerodynamic Diameter	
microns (unit density sphere)	% Passing Selector
$\leq 2.0$	90
2.5	75
3.5	50
5.0	25
10.0	0

**Total Dust** - airborne particles that are <u>not</u> selectively collected with regard to their size. Larger particles tend to be deposited within the nose and upper airways of the respiratory tract, whereas smaller particles may be drawn deeper into the respiratory tract and may be deposited in the airways and airspaces of the lung. These dusts may cause toxic effects when inhaled in large quantities.

# III. Threshold Limit Values (TLVs®)

#### A. Respirable Dust

In metal and nonmetal mines, *crystalline silica* (which is sometimes called *free silica*) is one of two substances for which MSHA has a respirable dust standard. The other substance is coal dust and is typically encountered at MNM operations utilizing coal-fired kilns. There are three forms of crystalline silica: *quartz*, *cristobalite*, *and tridymite*.

- Quartz is the most common form of crystalline silica and it is found in almost every type of metal and nonmetal ore. It is important to sample for the miners' respirable silica-bearing dust exposures.
- Cristobalite and tridymite are forms of silica derived from superheating quartz. They have a different crystalline structure than quartz and are generally accepted as the most hazardous forms of crystalline silica. They are found naturally in volcanic rock formations or in some refractories and fire brick used to insulate some cement kilns, lime kilns or dryers. Cristobalite is also found in bentonite and calcined diatomaceous earth. Note that routine respirable samples are not analyzed for cristobalite and tridymite unless specifically requested.

The TLVs<sup>®</sup> for respirable silica-bearing dust is dependent upon the amount (%) of quartz (Q), cristobalite (C), and/or tridymite (T) present in the dust.

<u>wt. of silica (Q, C, or T) in milligrams (mg)</u> X 100 = % silica (Q, C, or T) total wt. of dust on filter in milligrams (mg)

Note: The percentage of each form of silica must be calculated separately and rounded to one decimal place.

For example, if the weight of quartz is 0.90 milligrams:

# 1. The $TLV^{\otimes}$ formula for respirable quartz is:

$$\frac{10 \text{ mg/m}^3}{\text{%quartz} + 2} = \text{TLV}^{\text{®}} \text{ in mg/m}^3$$

For example, if the quartz content of the sample is 18.0 percent (%):

$$\frac{10 \text{ mg/m}^3}{18.0 \text{ quartz} + 2} = \frac{10 \text{ mg/m}^3}{20.0} = 0.50 \text{ mg/m}^3 \text{ TLV}^{\text{(8)}} \text{ for quartz}$$

# 2. The TLV® formula for respirable cristobalite is:

$$\frac{5 \text{ mg/m}^3}{(\%\text{C}) + 2} = \text{TLV}^{\text{(B)}} \text{ in mg/m}^3$$

For example, if the cristobalite content of the sample is 18.0 percent (%):

$$\frac{5 \text{ mg/m}^3}{18.0 + 2} = \frac{5 \text{ mg/m}^3}{20.0} = 0.25 \text{ mg/m}^3 \text{ TLV}^{\text{®}} \text{ for cristobalite}$$

# 3. The $TLV^{\otimes}$ formula for respirable tridymite is:

$$\frac{5 \text{ mg/m}^3}{(\%\text{T}) + 2} = \text{TLV}^{\textcircled{\$}} \text{ in mg/m}^3$$

For example, if the tridymite content of the sample is 8.0 percent (%):

$$\frac{5 \text{ mg/m}^3}{8.0 + 2} = \frac{5 \text{ mg/m}^3}{10.0} = 0.50 \text{ mg/m}^3 \text{ TLV}^{\text{(B)}} \text{ for tridymite}$$

#### B. Additive Respirable Exposure Formula

Since the three forms of silica -- quartz, cristobalite, and tridymite -- affect the respiratory system similarly, combined exposures to these contaminants are *additive*. The formula for the mixture of quartz, cristobalite, and tridymite is:

$$\frac{10 \text{ mg/m}^3}{(\%\text{Q}) + 2(\%\text{C}) + 2(\%\text{T}) + 2} = \text{TLV}^{\circledR} \text{ in mg/m}^3 \text{ for mixture}$$

where: Q = Quartz, C = Cristobalite, and T = Tridymite.

For example, if the sample contained 3.0 percent (%) quartz, 4.0 % cristobalite, and no (0%) tridymite:

$$\frac{10 \text{ mg/m}^3}{3.0 + 2(4.0) + 2(0) + 2} = \frac{10 \text{ mg/m}^3}{13} = 0.77 \text{ mg/m}^3 \text{ TLV}^{\$} \text{ for the mixture.}$$

#### C. Nuisance Dusts

Total dust sampling should be considered at mines where the sampling history indicates that the dust contains less than one percent quartz and no asbestos. The dust must have a specific enforceable TLV<sup>®</sup> or be considered a "nuisance dust." A TLV<sup>®</sup> of 10 mg/m³ is applicable for the Nuisance Particulates listed in Appendix E of the *Threshold Limit Values for Chemical Substances in Workroom Air Adopted by the ACGIH for 1973*. These "nuisance" or "listed" dusts are also named on the request for laboratory analysis form (MSHA Form 4000-29) under the heading \*\*Appendix E, 1973 ACGIH TLV Material List.

Note: Silica analysis is not performed on total dust samples.

The classification of a dust as a "nuisance" or "listed" dust is dependent on the commodity generating the dust exposure, and not by the analysis requested or

whether the sample is of a total or respirable particle size. For example, if a respirable dust sample is collected at a limestone handling facility and a quartz analysis is requested, the sample should be identified as a listed dust on Form 4000-29 ("Yes" on Line 18 of the respective sample data column).

#### IV. Respirable and Total Dust Sampling Equipment

#### A. Filter Cassette

Use only the pre-weighed filter cassettes purchased from the contract manufacturer. These cassettes consist of a 37-mm diameter polyvinyl chloride membrane filter with a 5-micron ( $\mu$ ) pore size. The cassettes are individually sealed in a small plastic bag by the manufacturer (refer to Figure 5-1).

The individually sealed filter cassettes are provided in *lots*. Each lot is preweighed on the same date. This sealed packaging method makes any tampering with the unused cassettes readily apparent. In order for a sample to be taken, the individually sealed plastic bag containing the sampling filter cassette must first be opened. This sealed plastic bag should only be opened when it is time to mount the cassette on the sampling train. Likewise, the sealed plastic bag containing the control sample cassette does not need to be opened. The control sample cassette must be from the same lot as the sample cassettes.

Dust filters are not shipped with an expiration date. The sample will be voided by the lab if the control is determined out of range at the time of analysis.

NOTE: Excess unused filters may be sent to the MSHA Laboratory, used for calibration purposes, or for demonstration purposes. Contact your District Office for guidance if needed.



Figure 5-1. Filter Cassettes, Lots, and Storage Bags

#### B. Sampling Pump

- 1. **Respirable Dust** The flow rate for the sampling pump must be 1.7 liters per minute (Lpm), plus or minus a field tolerance of 5 percent, which is a range of 1.615 Lpm to 1.785 Lpm. Sampling at this flow rate ensures that the 10-mm cyclone properly separates respirable from non-respirable particles.
- **2. Total Dust** The recommended flow rate is 1.7 Lpm. However, flow rates between 1.0 Lpm and 2.0 Lpm are acceptable.

# C. Respirable Dust Sampling Train

The "sampling train" includes all parts of the sampling setup: steel lapel holder, filter cassette, 10-mm cyclone assembly, tubing, and sampling pump (refer to Figures 5-2 and 5-3).



Figure 5- 2. Respirable Dust Cyclone and Holder Assembly



Figure 5-3. Respirable Dust Sampling Train

## **D.** Total Dust Sampling Train

Total dust sampling uses the same type of filter cassette required for respirable dust sampling. Assemble the apparatus with O-rings placed on the top and bottom of the cassette to seat the cassette snugly in the cassette holder (refer to Figure 5-4), or use the top of the metal cyclone holder seated into the cassette holder, as shown in Figure 5-4a. **Note:** A cyclone is <u>not</u> used.



**Figure 5- 4. Total Dust Sampling Train** 



Figure 5- 4a. Total Dust Cassette Holder Option

#### V. Respirable and Total Dust Sampling Procedures

**Full-shift personal** samples <u>must</u> be collected during the miner's entire workshift, regardless of the number of hours worked. [Short-term sampling is <u>not</u> acceptable for mineral dust contaminants.]

#### A. Pre-Survey Calibration of Sampling Pump

Prepare and calibrate the sampling pump using the procedures in Chapter 4.

#### **B.** Control Filter Cassette

A control filter cassette is used to adjust post-weights for variability associated with temperature and humidity variations. Prepare one *control* filter during the sampling period for each day of sampling. The control filter(s) must be from the same filter lot as the sample filter cassettes (*i.e.*, the same pre-weight date and stored under same environmental conditions). Do <u>not</u> remove the caps or end plugs from the control filter cassette. When sample filters from different lots are used, a control filter from each lot must be used.

- 1. Remove the control filter cassette from its storage bag. Do <u>not</u> remove the inlet or outlet plugs from the cassette.
- 2. Expose the control filter to the same time, temperature, and handling conditions as the ones that are used for sampling; *i.e.*, carry the control filter(s) in a shirt or coverall pocket while making rounds to check on the samples; keep the control filter(s) with the exposure samples after sampling; and treat the control filter(s) the same as the exposed filters when in the office or laboratory.
- 3. At the end of the shift, place a white sample seal label (MSHA Form 4000-30) on the filter cassette. Date, sign, and mark "CONTROL" on the sample seal. Record all control filter information in the Health Field Notes.

#### C. Assemble the Sampling Train

#### 1. Respirable Dust

- a. Remove the sample filter cassette from its individually-sealed plastic bag. If the bag has been opened or the cassette is damaged, do not use it.
- **b.** Record the cassette number on the Health Field Notes form.
- **c.** Remove the plugs from the cassette and place them in a clean, convenient location.
- **d.** Make sure the number of the cassette is facing up, on the pump side of the assembly. When properly assembled, the cyclone will "lock" into the steel lapel holder and the cyclone inlet will face forward.
- **e.** Tighten the set screw on the lapel holder so that all fittings are tight and the cassette is secured.
- f. Connect the tubing to the metal outlet tube of the lapel holder holding the cyclone and filter cassette and attach the other end of the tubing to the sampling pump inlet.
- **2. Total Dust** (Note: Quartz analysis is <u>not</u> performed on total dust samples.)
  - **a.** Remove the sample filter cassette from the plastic storage bag. If the bag has been opened or the cassette is damaged, do not use it.
  - **b.** Record the cassette number on the Health Field Notes form.
  - **c.** Remove the plugs from the cassette and place them in a clean, convenient location.
  - **d.** Attach the cassette outlet (the side where the cassette number is visible) to one end of the tubing with a nylon or plastic coupler. The other end of the tubing is connected to the sampling pump inlet. Note: A cyclone is <u>not</u> used.

#### D. Attach the Sampling Train to the Miner

- 1. Attach the sampling pump and sampling train to the miner. Clip, pin, or tape the tubing to the miner's clothing so that it does not interfere with the miner's job performance and does not present a safety hazard (such as, tubing catching on moving machine parts or railings). If the miner is not wearing a shirt or belt, the inspector should provide a belt or vest to facilitate sampling. These may be purchased as needed through the District Office.
- 2. Attach the sampler to the miner's clothing within the breathing zone. For respirable dust sampling, attach the sampling train so that the cyclone's grit pot is on the bottom of the assembly and the numbered side of the cassette is facing up. Be certain that the cyclone inlet is facing away from the body of the miner.

#### E. Instructions to the Miner

- 1. Explain to the miner what you are doing, what the sampling device does, and the reason for the sampling (i.e., the hazard). If available, issue a Miner Health Hazard Information Sheet or Card.
- 2. Instruct the miner not to remove the sampling pump or sampling train at any time or cover the sampler or cyclone inlet with a coat or anything else. If the miner must leave the mine property or work area during the shift, the inspector should turn the sampling pump off and remove the sampling train. Sampling should resume once the miner returns.
- 3. Instruct the miner not to bump, drop, abuse, or tamper with the sampling pump or sampling train.
- 4. Emphasize the need for the miner to continue to work in a routine manner and report to you any unusual occurrences or problems encountered during the sampling period.
- 5. Inform the miner when and where the sampler will be removed and that you will be checking the equipment throughout the shift.
- **6.** When conducting respirable sampling, instruct the miner not to invert the cyclone holder. If this occurs, it should be reported immediately to the inspector.

#### F. Collect the Sample

- **1.** Start the sampling pump and replace the security cover.
- **2.** Record the following information in the Health Field Notes (refer to Chapter 21, Section V):
  - Time the sampling pump was started;
  - Pump and filter cassette identification numbers;
  - Miner's name, job title, and work location(s);
  - Shift hours per day and days per week worked;
  - Any respirator worn or expected to be worn (brand, model, type of filters); and
  - Whether an acceptable respiratory protection program exists (see Chapter 16 for criteria for evaluating respiratory protection program).
- 3. During each full-shift sample, the inspector must observe the miner being sampled as frequently as is necessary to determine that a representative sample is being conducted of the normal activities. Check the sampling pump and sampling train to make sure that the sampling pump is operating properly, and to make sure the tubing and connections are not leaking. Do not adjust the flow rate at any time while sampling. Record what tasks the miner has performed in the time between subsequent checks so that the completed Health Field Notes will describe the miner's full work shift. This requirement does not necessarily preclude the inspector from doing other inspection work while conducting the sampling.
- 4. Evaluate dust conditions. Dusty work environments may require changing filter cassettes to keep them from becoming overloaded. If dust conditions are excessive during respirable and total dust sampling, the filter should be changed. Consecutive samples, using shorter sampling periods, should be collected for the full shift. See Chapter 2 Section IV, for discussion of collecting consecutive samples.

- **5.** Throughout the shift, record all other pertinent information in the Health Field Notes:
  - Times that the sampling train was checked and condition of sampling equipment. If the sampling pump needs to be changed out, stopped, or restarted for any reason, record the times involved;
  - If consecutive samples were taken, record the number of any additional cassettes used during the sampling period;
  - Activity of miner, equipment operating in the area, and approximate time spent at each activity;
  - General description of the controls in use and whether or not they seem adequate;
  - Potential sources of exposure, a general description of these sources, number of persons affected, and possible additional control measures;
  - Any other samples taken and the results, if available (e.g. noise, detector tubes, organic vapor badges); and
  - Environmental conditions (such as wind conditions, temperature, humidity, etc.).
- **6.** Collect the sampling train from the miner.
  - **a.** <u>Turn off</u> the sampling pump. Turn off the pump **before** removing the sampling train or pump. Record the time the pump was turned off.
  - **b.** <u>Carefully</u> remove the sampling train:
    - Respirable dust sampling keep the cyclone <u>upright</u> to prevent the non-respirable dust in the grit pot from falling back through the cyclone onto the filter. Remove the sample filter cassette from the lapel holder and replace the cassette plugs. Place a white sample seal (MSHA Form 4000-30) on the sample filter cassette. Date and sign the sample filter cassette seal.

Total dust sampling - uncouple the sample filter cassette
from the sampling train and replace the cassette plugs.
 Place a seal on the sample filter cassette. Date and sign the
sample filter cassette seal.

#### **VI.** Post-Inspection Procedures

#### A. Review Health Field Notes

Check that all the necessary information is included in the Health Field Notes.

#### B. Post-Survey Calibration of Sampling Pump

Check the sampling pump calibration using the procedures in Chapter 4.

#### C. Cyclone Cleaning and Assembly (respirable dust sampling only)

- 1. Unscrew the *grit pot* from the cyclone. Empty the grit pot by turning it upside down and tapping it gently on a solid surface.
- 2. Gently wash cyclone, grit pot, *coupler* (or collar) and the *vortex finder* (cyclone cap) by agitating in warm soapy water or, preferably, wash in an ultrasonic bath. <u>Do not insert anything</u> which can score or scratch the inner lining walls of the cyclone (e.g., pipe cleaner).
- **3.** Rinse thoroughly in clean water, shake off excess water, and set aside to dry before reassembly.
- 4. Inspect the cyclone and filter holder parts for damage or excessive wear, for a loose coupler or vortex finder, and for scoring or rifling (which can trap respirable particles, preventing them from reaching the filter). Pay special attention to the top internal circumference of the cyclone when inspecting for scoring and rifling. Replace damaged or defective parts.
- **5.** Reassemble the cyclone, vortex finder (cyclone cap), and grit pot.

#### D. Transport the Samples for Analysis

- 1. Complete the Request for Laboratory Analysis (MSHA Form 4000-29) for the samples taken (refer to Chapter 21, Section VII). In Item No. 15 on the form, designate "R" for respirable samples, "T" for total dust samples or "CB" for blank cassette / control filter cassette. In Item No. 16, fill in the analysis desired, for example, "quartz," or "total weight." Samples and controls from different lots, used on the same day, should be listed on separate Request for Laboratory Analysis (RLA) forms. If consecutive filters were used for an individual, list the individual's filters on a separate RLA form. Be sure to provide all the information the laboratory will need to fill out the Personal Exposure Data Summary (PEDS) or Area Sample Data Summary (ASDS).
- 2. Ship the Request for Laboratory Analysis form(s) with the samples to the MSHA Laboratory. Filter cassettes and controls can be shipped together in the same container if space allows. Never split cassettes from the same inspection and mail them separately.

#### **E.** Compliance Determination

The laboratory will complete the analysis and return an Analytical Report that will include the information that was formerly calculated using the Lotus Health Calculation spreadsheet application. The Analytical Report will include the sample weight, concentration, TLV<sup>®</sup> or STEL, error factor, and calculated enforcement TLV<sup>®</sup> (TLV<sup>®</sup> times error factor). A PEDS or ASDS will also be generated as appropriate.

## F. Report Writing

- 1. Inspection reports should include a copy of the Request for Laboratory Analysis, Health Field Notes, the Laboratory-provided Analytical Report, completed Personal Exposure Data Summary (PEDS), or ASDS calculation worksheets, citation/orders, and any other supplemental information collected during the inspection.
- 2. The laboratory submits electronic PEDs and analytical reports for samples analyzed to the inspector, supervisor, and district health specialist via Email. These must be copied by the inspector and completed with the Action Code and Citation number when applicable.

3. When manually completing the PEDS or the ASDS (refer to Chapter 21, Sections VIII & IX), be sure that the concentration and exposure limit units of measurement are the same as those listed for the contaminant code (refer to Chapter 3), and that the Action Code and Citation number have been included when applicable.