

APPENDIX C.2

PROCEDURES FOR LABORATORY ANALYSIS OF SURFACE/BULK DUST  
LOADING SAMPLES



## Appendix C.2

### Procedures For Laboratory Analysis Of Surface/Bulk Dust Loading Samples

This appendix discusses procedures recommended for the analysis of samples collected from paved and unpaved surfaces and from bulk storage piles. (AP-42 Appendix C.1, "Procedures For Sampling Surface/Bulk Dust Loading", presents procedures for the collection of these samples.) These recommended procedures are based on a review of American Society For Testing And Materials (ASTM) methods, such as C-136 (sieve analysis) or D-2216 (moisture content). The recommendations follow ASTM standards where practical, and where not, an effort has been made to develop procedures consistent with the intent of the pertinent ASTM standards.

#### C.2.1 Sample Splitting

##### Objective -

The collection procedures presented in Appendix C.1 can result in samples that need to be reduced in size before laboratory analysis. Samples are often unwieldy, and field splitting is advisable before transporting the samples.

The size of the laboratory sample is important. Too small a sample will not be representative, and too much sample will be unnecessary as well as unwieldy. Ideally, one would like to analyze the entire gross sample in batches, but that is not practical. While all ASTM standards acknowledge this impracticality, they disagree on the exact optimum size, as indicated by the range of recommended samples, extending from 0.05 to 27 kilograms (kg) (0.1 to 60 pounds [lb]).

Splitting a sample may be necessary before a proper analysis. The principle in sizing a laboratory sample for silt analysis is to have sufficient coarse and fine portions both to be representative of the material and to allow sufficient mass on each sieve to assure accurate weighing. A laboratory sample of 400 to 1,600 grams (g) is recommended because of the capacity of normally available scales (1.6 to 2.6 kg). A larger sample than this may produce "screen blinding" for the 20 centimeter (cm) (8 inch [in.]) diameter screens normally available for silt analysis. Screen blinding can also occur with small samples of finer texture. Finally, the sample mass should be such that it can be spread out in a reasonably sized drying pan to a depth of < 2.5 cm (1 in.).

Two methods are recommended for sample splitting: riffles, and coning and quartering. Both procedures are described below.

##### Procedures -

Figure C.2-1 shows 2 riffles for sample division. Riffle slot widths should be at least 3 times the size of the largest aggregate in the material being divided. The following quote from ASTM Standard Method D2013-72 describes the use of the riffle.

Divide the gross sample by using a riffle. Riffles properly used will reduce sample variability but cannot eliminate it. Riffles are shown in Figure C.2-1. Pass the material through the riffle from a feed scoop, feed bucket, or riffle pan having a lip or opening the full length of the riffle. When using any of the above containers to feed the riffle, spread the material evenly in the container, raise the container, and hold it with its front edge resting on top of the feed chute, then slowly tilt it so that the material flows in a uniform stream through the hopper straight down over the center of the riffle into all the slots, thence into the riffle pans, one-half of the sample being collected in a pan.



Under no circumstances shovel the sample into the riffle, or dribble into the riffle from a small-mouthed container. Do not allow the material to build up in or above the riffle slots. If it does not flow freely through the slots, shake or vibrate the riffle to facilitate even flow.<sup>1</sup>

Coning and quartering is a simple procedure useful with all powdered materials and with sample sizes ranging from a few grams to several hundred pounds.<sup>2</sup> Oversized material, defined as > 0.6 millimeters (mm) (3/8 in.) in diameter, should be removed before quartering and be weighed in a "tared" container (one for which its empty weight is known).

Preferably, perform the coning and quartering operation on a floor covered with clean 10 mil plastic. Take care that the material is not contaminated by anything on the floor or that any portion is not lost through cracks or holes. Samples likely affected by moisture or drying must be handled rapidly, preferably in a controlled atmosphere, and sealed in a container to prevent further changes during transportation and storage.

The procedure for coning and quartering is illustrated in Figure C.2-2. The following procedure should be used:

1. Mix the material and shovel it into a neat cone.
2. Flatten the cone by pressing the top without further mixing.
3. Divide the flat circular pile into equal quarters by cutting or scraping out 2 diameters at right angles.
4. Discard 2 opposite quarters.
5. Thoroughly mix the 2 remaining quarters, shovel them into a cone, and repeat the quartering and discarding procedures until the sample is reduced to 0.4 to 1.8 kg (1 to 4 lb).

### C.2.2 Moisture Analysis

Paved road samples generally are not to be oven dried because vacuum filter bags are used to collect the samples. After a sample has been recovered by dissection of the bag, it is combined with any broom swept material for silt analysis. All other sample types are oven dried to determine moisture content before sieving.

Procedure -

1. Heat the oven to approximately 110°C (230°F). Record oven temperature. (See Figure C.2-3.)
2. Record the make, capacity, and smallest division of the scale.
3. Weigh the empty laboratory sample containers which will be placed in the oven to determine their tare weight. Weigh any lidded containers with the lids. Record the tare weight(s). Check zero before each weighing.
4. Weigh the laboratory sample(s) in the container(s). For materials with high moisture content, assure that any standing moisture is included in the laboratory sample container. Record the combined weight(s). Check zero before each weighing.

MOISTURE ANALYSIS

Date: _____	By: _____
Sample No: _____	Oven Temperature: _____
Material: _____	Date In: _____ Date Out: _____
	Time In: _____ Time Out: _____
Split Sample Balance: _____	Drying Time: _____
Make _____	
Capacity _____	Sample Weight (after drying)
Smallest division _____	Pan + Sample: _____
	Pan: _____
Total Sample Weight: _____	Dry Sample: _____
(Excl. Container)	
Number of Splits: _____	MOISTURE CONTENT:
	(A) Wet Sample Wt. _____
Split Sample Weight (before drying)	(B) Dry Sample Wt. _____
Pan + Sample: _____	(C) Difference Wt. _____
Pan: _____	$\frac{C \times 100}{A}$
Wet Sample: _____	A = _____ % Moisture

Figure C.2-3. Example moisture analysis form.

5. Place sample in oven and dry overnight. Materials composed of hydrated minerals or organic material such as coal and certain soils should be dried for only 1.5 hours.
6. Remove sample container from oven and (a) weigh immediately if uncovered, being careful of the hot container; or (b) place a tight-fitting lid on the container and let it cool before weighing. Record the combined sample and container weight(s). Check zero before weighing.
7. Calculate the moisture, as the initial weight of the sample and container, minus the oven-dried weight of the sample and container, divided by the initial weight of the sample alone. Record the value.
8. Calculate the sample weight to be used in the silt analysis, as the oven-dried weight of the sample and container, minus the weight of the container. Record the value.

C.2.3 Silt Analysis

Objective -

Several open dust emission factors have been found to be correlated with the silt content (< 200 mesh) of the material being disturbed. The basic procedure for silt content determination is mechanical, dry sieving. For sources other than paved roads, the same sample which was oven-dried to determine moisture content is then mechanically sieved.

For paved road samples, the broom-swept particles and the vacuum-swept dust are individually weighed on a beam balance. The broom-swept particles are weighed in a container, and the vacuum-swept dust is weighed in the bag of the vacuum, which was tared before sample collection. After

weighing the sample to calculate total surface dust loading on the traveled lanes, combine the broom-swept particles and the vacuumed dust. Such a composite sample is usually small and may not require splitting in preparation for sieving.

Procedure -

1. Select the appropriate 20-cm (8-in.) diameter, 5-cm (2-in.) deep sieve sizes. Recommended U. S. Standard Series sizes are 3/8 in., No. 4, No. 40, No. 100, No. 140, No. 200, and a pan. Comparable Tyler Series sizes can also be used. The No. 20 and the No. 200 are mandatory. The others can be varied if the recommended sieves are not available, or if buildup on 1 particulate sieve during sieving indicates that an intermediate sieve should be inserted.
2. Obtain a mechanical sieving device, such as a vibratory shaker or a Roto-Tap<sup>®</sup> without the tapping function.
3. Clean the sieves with compressed air and/or a soft brush. Any material lodged in the sieve openings or adhering to the sides of the sieve should be removed, without handling the screen roughly, if possible.
4. Obtain a scale (capacity of at least 1600 grams [g] or 3.5 lb) and record make, capacity, smallest division, date of last calibration, and accuracy. (See Figure C.2-4.)
5. Weigh the sieves and pan to determine tare weights. Check the zero before every weighing. Record the weights.
6. After nesting the sieves in decreasing order of size, and with pan at the bottom, dump dried laboratory sample (preferably immediately after moisture analysis) into the top sieve. The sample should weigh between ~ 400 and 1600 g (~ 0.9 and 3.5 lb). This amount will vary for finely textured materials, and 100 to 300 g may be sufficient when 90% of the sample passes a No. 8 (2.36 mm) sieve. Brush any fine material adhering to the sides of the container into the top sieve and cover the top sieve with a special lid normally purchased with the pan.
7. Place nested sieves into the mechanical sieving device and sieve for 10 minutes (min). Remove pan containing minus No. 200 and weigh. Repeat the sieving at 10-min intervals until the difference between 2 successive pan sample weighings (with the pan tare weight subtracted) is less than 3.0%. Do not sieve longer than 40 min.
8. Weigh each sieve and its contents and record the weight. Check the zero before every weighing.
9. Collect the laboratory sample. Place the sample in a separate container if further analysis is expected.
10. Calculate the percent of mass less than the 200 mesh screen (75 micrometers [ $\mu\text{m}$ ]). This is the silt content.

### SILT ANALYSIS

Date: \_\_\_\_\_

By: \_\_\_\_\_

Sample No: \_\_\_\_\_

Sample Weight (after drying)

Material: \_\_\_\_\_

Pan + Sample: \_\_\_\_\_

Pan: \_\_\_\_\_

Split Sample Balance: \_\_\_\_\_

Dry Sample: \_\_\_\_\_

Capacity: \_\_\_\_\_

Make \_\_\_\_\_

Final Weight: \_\_\_\_\_

Smallest Division \_\_\_\_\_

Net Weight <200 Mesh

% Silt = Total Net Weight x 100 = \_\_%

### SIEVING

Time: Start:	Weight (Pan Only)
Initial (Tare):	
10 min:	
20 min:	
30 min:	
40 min:	

Screen	Tare Weight (Screen)	Final Weight (Screen + Sample)	Net Weight (Sample)	%
3/8 in.				
4 mesh				
10 mesh				
20 mesh				
40 mesh				
100 mesh				
140 mesh				
200 mesh				
Pan				

Figure C.2-4. Example silt analysis form.



## References For Appendix C.2

1. "Standard Method Of Preparing Coal Samples For Analysis", *Annual Book Of ASTM Standards, 1977*, D2013-72, American Society For Testing And Materials, Philadelphia, PA, 1977.
2. L. Silverman, *et al.*, *Particle Size Analysis In Industrial Hygiene*, Academic Press, New York, 1971.