1.0 TITLE

Particle Size Analysis by Wet and Dry Sieve for Soils, Including Coarse Fragments

2.0 LOCATION

Soils Prep Lab and Soils Digestion Lab

3.0 SCOPE & APPLICATION

This method is applicable to soils, spoils or tailings. Particle size distribution has a profound effect on the success of reclaiming mined or other drastically disturbed land just as it does on managing any agronomic area. Critical factors in revegetation and management of disturbed or degraded sites that are affected by particle size are moisture, temperature, and air relations, as well as physical impedance of roots, chemical reactivity, and erosion.

In most soils, spoils or tailing material, particles are classified as primary or secondary. Primary particles are individual sands, silts, and clays that exist separately with no forces of attraction binding two or more of the particles together. Secondary particles form when two or more primary particles are bound or attracted together by physical or chemical forces.

The relative amounts of primary sand, silt and clay particles usually are referred to as soil texture (SOPSO019). If the percentages of sand, silt and clay are known, then the soil texture can be classified into several categories using the USDA textural triangle (SOPSO019).

For additional grain size requirements multiple sieve sizes are used. Dry sieving can be done for those analyses $\geq$ 105um sieve. Wet sieve is required for anything < 105um sieve.

4.0 SUMMARY & METHOD MODIFCAITONS

When sieving for analytical purposes, wet or dry, a total weight of the dried, “as received” sample is recorded on an Excel particle size template. The sample is shaken through the required sieves and the sample fractions retained on the screens are individually gathered, weighed back dry and recorded. The percent passing through each screen is calculated on the Excel particle size template. The percent passing values are then recorded on the LIMS workgroup to be reported.

Sample > 2000um sieve are considered Coarse Fragments. These larger fragments contribute relatively few nutrients when considered on a short-term basis; however, they do influence physical properties of the soil or spoil, thus affecting plant growth.

Soils and spoils < 2000um are the greatest contributors for soil mediums. There is not an optimum texture for all purposes and all plants because requirements vary greatly, but a plant growth medium with enough sand to allow for aeration and looseness to permit plant root growth and development and enough clay for adequate nutrient and water- holding capacity would be ideal.

These particle sizes are used in the success of reclaiming mined or drastically disturbed lands.

GRAIN SIZE	USDA SOIL CLASSIFICATION
Greater than 10 inches	Stone
3 inches to 10 inches	Cobble
2000um to 3 inches	Gravel
½ inch to 3inches	Coarse Gravel
2000um to ½ inch	Fine Gravel
53um to 2000um	Sand
1000um to 2000um	Very Coarse Sand
500um to 1000um	Coarse Sand
250um to 500um	Medium Sand
105um to 250um	Fine Sand
53um	Very Fine Sand
<53um to 2 um	Silt
< 2 um	Clay

5.0 REFERENCES

5.1. Reclaiming Mine Soils and Overburden in the Western United States Analytical Parameters and Procedures, pp. 59-63, 1987.

5.2. Particle Size Analysis - Hydrometer Method, 15-5, Methods of Soil Analyses, ASA No.9, 2nd Edition, 1986 Annual Book of ASTM Standards.

5.3. ASA No. 9, Part 1, 2nd Edition, Section 15.

5.4. Soil Survey Manual, Agriculture Handbook No. 18, pp. 205 – 216.

5.5. Ninmo, J.R., and Perkins, K.S, 2002, Dry-Aggregate size distribution, in Dane, J.H., and Topp, G.C., eds, Methods of soil analysis, Part 4—Physical methods: Madison, Wisconsin, Soil Science Society of America, sec 2.6.2.1.a, p. 320-321.

6.0 SAMPLE COLLECTION, HANDLING & PRESERVATION

6.1 Samples are dried at < 40°C and stored in the soils dry storage area.

6.2 Hold Times:

Parameter Description	Matrix	Hold Time Starts - Ends With
SIEVE	Solid	Hold times have not been established for this procedure.

7.0 APPARATUS & SUPPLIES

7.1. Balance, sensitivity 0.1 g. Verify calibration daily or before use (see SOPAD013).
7.2. Drying oven constant at < 40°C
7.3. Drying oven constant at 105°C
7.4. Plastic drying pans and plastic wrap
7.2. Mortar and Pestle
7.5. Sieves, plastic should be used if any sub sample will be used for metal analysis.
7.6. Sieve shaker.
7.7. Sieve bottom plate with hose for wet sieve.
7.8. Opened edge sieve cover for wet sieve.
7.9. DI water with faucet sprayer
7.10. 150mL beakers or larger if needed
7.11. Zip lock baggies
7.12. Tarp

8.0 REAGENTS & STANDARDS

8.1 Sodium metaphosphate (50g/Liter), Dispersing agent. Shelf-life is one month from date of creation, and the sodium metaphosphate is stored in the base cabinet in the main soils digestion lab.

9.0 SAFETY

9.1 HAZARDS

All procedures within ACZ pose some safety hazards that may be avoided with attention to detail.

9.2 SAFETY TECHNIQUE

9.2.1 Safety glasses are required and the use of gloves and lab coat is strongly recommended. Shorts and open-toed shoes are not allowed in the lab.

9.2.2 Use care when pouring and pipeting reagents. Always add acid to water. Use the proper method when washing glassware.

9.2.3 Do not eat or use tobacco products in the lab.

9.2.4 Wipe up ALL spills immediately. Implement the Emergency Response Plan if necessary.

9.2.5 Do not wear gloves or lab coat outside of the laboratory. Remove gloves before using a computer, telephone, etc.

9.2.6 Do not conduct “experiments” unrelated to the analysis.

9.3 PROTECTIVE EQUIPMENT

9.3.1 Use a fume hood when there is a potential for strong fumes.

9.3.2 A fire extinguisher is located in soils prep and extraction labs and soils drying room.

9.3.3 An emergency shower and eye wash station is located in the soils prep lab.

10.0 INTERFERENCES

10.1. It is extremely important to get a representative sample size which takes into consideration the sub sample volume and total weight compared to sub sample weight. If the particle size of the sub sample is too large or too small, then the analytical data representing the whole will be incorrect See table 11.1.

10.2. Silt and clay adhere to larger rock fragment and are easily confused with rock fragments. For dry sieving gently use the mortal and pestle to break away any particle size differentiation. Be careful not to break apart rock fragments. For wet sieving soak the sub sample in sodium metaphosphate to disperse the silt and clay from larger particle sizes.

11.0 PROCEDURES

11.1. **Dry sieving** (only for ≥ 105um sieve; also client specific)

11.1.1 Create a LIMS workgroup in soils analysis, Department 25, SA-G-SIEVE-ANALYSIS.

11.1.2 Obtain a "Sieve Fraction" form (FRMSO009) for each of the samples to be analyzed.

11.1.3 Label each form with the appropriate LIMS number and highlight the sieve sizes that will be analyzed. You may need to manually change a sieve size if that particular size is not on the form.

11.1.4 Gather the samples to be analyzed. The samples must be air dried at <40°C before particle size analysis can be performed (See SOPSO001). Make sure the entire sample can be dried before doing so.

11.1.5 If the sample needs to be analyzed by other methods, make an appropriate sub sample split at this time. If the sample is of large quantity, place the sample on a tarp and roll the opposite corners back and forth to mix (see SOPAD047). Separate the sample into 4 sections and take the opposite sections for the sample. Repeat this with the split sample if the sample is still too large. The larger the screen size the larger the sample volume should be.

Table 11.1

Nominal diameter of largest particle	Approximate minimal mass for dry wt. of sample
2000um	100g
3/8 inch	200g
¾ inch	1000g
1 ½ inch	8000g
3 inch	60,000g

11.1.6 Weigh the entire dried "as received" sub sample to be used for the particle size analysis (see Table 11.1) and record the weight in the "dry sample weight" box on the form.

11.1.7 Organize the appropriate screens in order so the largest size sieve is on top. For Coarse Fragment analysis the sieves can be stacked on top of the sieve 2000um with the sieve bottom plate underneath to be done all at once. **If metal analysis is to be done on the**

sample or a sub-sample, stainless steel and/or plastic sieves must be used. Make sure the sieves are clean of residue by using the compressed air and/or acetone.

11.1.8 Carefully use a mortar and pestle to allow the break up of particles to pass each screen. Do not break up rock fragments with the mortar and pestle. See §13.0 for calculations

11.1.9 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. Or use the sieve shaker located in the soils prep lab for as long as needed. Do not turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until no more than 1 mass % of the residue on a sieve passes that sieve during 1 minute of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

11.1.10 For sieve sizes <2000 and >105, mortar and pestle each sample retained on each sieve to ensure proper particle size distribution. Do not break down rock fragments or manipulate sample through the sieve. Repeat §11.1.9.

11.1.11 Print labels to the soils printer and attach to a zip lock bag. With a sharpie label corresponding zip lock bags for each of the sieve sizes required for analysis with LIMS #, <screen size, and date.

11.1.12 Record DRY in the "method" column of FRMSO009 and the appropriate sieve size row on the form.

11.1.13 Weigh a zip lock bag or a tared drying pan and record the weight for the "tare weight" on the form.

11.1.14 Place the sample that is retained on the specified sieve in the zip lock bag or zeroed drying pan. Weigh the sample retained and record the weight in the "sieved sample + tare wt" column on the form. Clean each sieve and the sieve bottom plate with compressed air and/or acetone after each sample to remove any residual material.

11.1.15 Data for each sample is individually entered into the spreadsheet FRMSO009 in LabWeb. Check all entries for transcription errors and then print the data. Do not save changes to the form. Repeat this step for each sample.

11.1.16 Transcribe the calculated Material Passing % data from the spreadsheet FRMSO009 to the LIMS workgroup sheet. Make sure the correct sieve size is reported to LIMS.

11.1.17 Enter the "Material Passing %" data into the LIMS database.

11.1.18 AREV data in LIMS and turn into department supervisor for SREV.

11.2. **Wet Sieving** (mandatory for <105um sieve, but can be used for all sieve sizes; also client specific)

11.2.1 Create a LIMS workgroup in soils analysis, Department 25, SA-G-SIEVE-ANALYSIS.

11.2.2 Obtain a "Sieve Fraction" form (FRMSO009) for each of the samples to be analyzed.

11.2.3 Label each form with the appropriate LIMS number and high light the sieve sizes that will be analyzed. You may need to manually change a sieve size if that particular size is not on the form.

11.2.4 Gather the samples to be analyzed. The samples must be air dried at <40°C before particle size analysis can be performed (See SOPSO001) Make sure the entire sample can be dried before doing so.

11.2.5 If the sample needs to be analyzed by other methods, make an appropriate sub sample split at this time. If the sample is of large quantity, place the sample on a tarp and roll the opposite corners back and forth to mix (see SOPAD047**)**. Separate the sample into 4 sections and take the opposite sections for the sample. Repeat this with the split sample if the sample is still too large. The larger the screen size the larger the sample volume should be.

11.2.6 Record WET in the "method" column and the appropriate sieve size row on the form FRMSO009.

11.2.7 Label a 150mL beaker for each of the sieve sizes to be analyzed. A larger beaker may be needed for larger particle sized materials or for larger quantities of a particle size. Each beaker must be labeled with the LIMS # and the appropriate sieve size. Place these beakers in order on a cart.

11.2.8 Weigh each beaker and record its weight in the "tare weight" column on the form.

11.2.9 In a labeled plastic cup or clean tared drying pan, weigh the entire dried and as received sub sample to be used for the particle size analysis (see Table 18.2). Record the weight in the "dry sample weight" box on the form.

11.2.10 Cover each sample with Sodium Metaphosphate solution to aid in soil dispersement. Stir the sample to allow for the solution to be mixed with the sample.

11.2.11 Let the samples sit in the dispersing reagent until rock material looks clean.

11.2.12 Set the shaker next to the sink and attach the sprayer to the DI water faucet. Replace the DI water hose with the longer hose hanging on the wall.

11.2.13 Place the wet sieve bottom plate on the shaker with the hose in the sink.

11.2.14 Gather and stack the appropriate sieves and stack them in order so that the largest sieve size is on top. Make sure the sieves are clean of residue by using the compressed air and/or acetone.

11.2.15 Place the opened edge sieve cover on the top of the sieve stack and use a bungee cord to secure the sieve stack while shaking. Turn on the shaker to make sure the sieves stay in place while shaking.

11.2.16 Pour the soaked sample on the top sieve and rinse the cup out with the DI water sprayer.

11.2.17 Turn on the shaker.

11.2.18 Spray water on the sample in the sieves until the water coming out of the bottom is clear. You may have to pick up and crumble or mash any lumps that have not soaked through

using your thumb and fingers, be sure to rinse any material that is stuck to your fingers back on to the sieve. Do not force any material through the sieve openings.

11.2.19 Turn off the shaker

11.2.20 Clean the counter that will be used to transfer the sub samples from the sieve to the beaker.

11.2.21 Remove the top sieve and rinse the sub sample retained on the sieve into the appropriate beaker. You may need to carefully turn the sieve upside down on the clean counter and manually push the material up and out of the screen for the medium size mesh openings. Gather any material that has fallen on the counter and place it in the beaker with the rest of the sample. For smaller particle sizes use a squeegee to gather material on the counter.

11.2.22 Clean the counter between each sieve transfers.

11.2.23 Repeat for each of the sieve sizes. You may need to do additional rinsing on the very fine mesh sizes to ensure complete particle size distribution. Make sure each sieve is cleaned of all material between samples.

11.2.24 Place all the beakers in order in the drying oven and dry overnight at 105°C.

11.2.25 When the samples are dry, remove them from the oven and allow them to reach room temperature.

11.2.26 Weigh back the beakers and record the weights in the "sieved sample + tare wt" column on the form. Save the sub samples until the data is entered and reviewed.

11.2.27 Data for each sample is individually entered into the spreadsheet FRMSO009 in Qualtrax. Check all entries for transcription errors and then print the data. Do not save changes to the form. Repeat this step for each sample.

11.2.28 Transcribe the Material Passing % data from the spreadsheet FRMSO009 to the LIMS workgroup sheet. Make sure the correct sieve size is reported to LIMS.

11.2.29 Enter the "Material Passing %" data into the LIMS database.

11.2.30 AREV data in LIMS and turn into department supervisor for SREV.

12.0 QUALITY CONTROL

12.1 Analyze one sample duplicate (DUP) for each workgroup of 20 or less client samples. Passing duplicates (RPD < 20) are difficult to achieve with large particle sizes and minimal sample volume. Make sure the initial sub samples are properly prepared. If initial splits are properly prepared, then duplicate failure is an indicator of sample non-homogeneity, and the samples may be qualified with an "RC" flag.

12.2. The analysis is acceptable if the following QC limits are met:

- Duplicate RPD $\leq$ 20 %

Data is qualified at SREV if the RPD > 20 %.

13.0 DATA CALCULATIONS, DATA REPORTING & ARCHIVING

13.1 If a large sample is used for the screen sizes >2000um and then an additional sub sample <2000um is taken for the remainder of the sieve analysis, you must calculate the true sub sample size <2000um from the original total sample weight used.

13.1.1 In the "Enter Dry Sample Weight (g):" box record the entire whole sample to be screened

Example: 397.71g sample to be screened through 4750um and 2000um.

Retained on the 4750um = 0.50g
Retained on the 2000um = 1.20 g

(Retained g / total g) x 100 = % retained

(0.50g/397.71g) x 100 = 0.1% retained
(1.20g / 397.71g) x 100 = 0.3% retained

Enter the data directly in the "Sieved Sample + Tare Wt(g)" column to obtain the % material retained and the % material passing each sieve (0.50g retained on 4750um and 1.20g retained on 2000um to calculate 0.4% retained and 99.6% passing)

**This step must be done and saved so the additional sub sample will be calculated on the total sample weight.

13.1.2 Weigh the sub sample to be used for sieving <2000um.

Example: 40g sub sample to be screened through 2000um. The 40 g sub sample must be calculated to reflect the total sample size (397.71g) as follows:

(40g / 99.6) x 100 = 40.16g

The "dry sample weight" will be recorded as 40.2g

13.2 % Coarse Fragments = wt (g) >2000um / Total Sample wt. x 100

13.3 Relative Percent Difference (RPD) is calculated as follows:

$$RPD = \frac{\left|X_1 - X_2\right|}{\left(\frac{X_1 + X_2}{2}\right)} \times 100$$

Where: X_1 = Material passing % of the sample
X_2 = Material passing % of the sample duplicate

13.4 MDL is 0.1% and PQL is 0.5%.

14.0 METHOD PERFORMANCE/DETECTION LIMITS

14.1 An MDL study is not needed for this method.

14.2 A Demonstration of Capability (DOC) study is not applicable to this method. Initial Method Training Form (FRMQA004) is required.

15.0 DOCUMENTATION

15.1 Record the appropriate information for all reagents in the soils department Standard/Reagent Log.

15.2 Make sure the following information is included on the Workgroup:

- Analysis date, start time and completion time.
- Analytical method used.
- Analyst's initials.
- Calculated QC data results.
- Any remarks about analysis or samples.
- Data review/checklist form.
- Standard/reagent prep sheet, if applicable.
- *ANY OTHER PERTINENT INFORMATION*

16.0 WASTE MANAGEMENT/POLLUTION PREVENTION

16.1 Refer to ACZ's Waste Management Plan for appropriate disposal details for this method.

17.0 DEFINITIONS

17.1 Sample Duplicate (DUP)—Two aliquots of a sample are analyzed in the same workgroup, under identical circumstances. Analysis of a duplicate sample indicates the precision associated with the laboratory procedure.

17.2 Relative Percent Difference (RPD)--The difference between two replicates divided by the average of those two replicates, and the final value multiplied times 100.

18.0 TABLES & DIAGRAMS

18.1 Table 11.1: Screen size & sample volume guidlines

18.2 Table 18.2: What size sample is needed

Nominal diameter of largest particle	Approximate minimal mass for dry wt. of sample
2000um	100g
3/8 inch	200g
¾ inch	1000g
1 ½ inch	8000g

3 inch	60,000g

18.3 Table 18.3: Particle sizes used in the success of reclaiming mined or drastically disturbed lands.

GRAIN SIZE	USDA SOIL CLASSIFICATION
Greater than 10 inches	Stone
3 inches to 10 inches	Cobble
2000um to 3 inches	Gravel
½ inch to 3inches	Coarse Gravel
2000um to ½ inch	Fine Gravel
53um to 2000um	Sand
1000um to 2000um	Very Coarse Sand
500um to 1000um	Coarse Sand
250um to 500um	Medium Sand
105um to 250um	Fine Sand
53um	Very Fine Sand
<53um to 2 um	Silt
< 2 um	Clay

18.4 Table 18.4: Available Sieve Sizes

LIMS Product	Inches	Millimeters	Micrometers	Mesh	USDA Classification
Sieve76200	3	76.2	76,200		Cobbles
Sieve63000	2 ½	63	63,000		Coarse Gravel
Sieve45300	1 ¾	45	45,300		
Sieve31500	1 ¼	31.5	31,500		
Sieve22400	7/8	22.4	22,400		
Sieve16000	5/8	16.0	16,000		
Sieve12700	½	12.7	12,700		Fine Gravel
Sieve11200	7/16	11.2	11,200		
Sieve9500	3/8	9.5	9,500		
Sieve8000	5/16	8.0	8,000		
Sieve5660		5.66	5,660	3.5	
Sieve4750		4.75	4,750	4	
Sieve4000		4.0	4,000	5	
Sieve2800		2.8	2,830	7	
Sieve2000		2.0	2,000	10	Very Coarse Sand
Sieve1400		1.4	1,400	14	
Sieve1000		1.0	1,000	18	Coarse Sand
Sieve850		.85	850	20	
Sieve707		.707	707	25	
Sieve500		.5	500	35	Medium Sand
Sieve250		.25	250	60	
Sieve177		.18	177	80	
Sieve125		.125	125	120	
Sieve105		.105	105	150	Fine Sand
Sieve74		.074	74	200	
Sieve63		.063	63	230	
Sieve53		.053	53	270	Very Fine Sand
Sieve45		.045	45	325	Very Fine Sand (USGS)
Texture	No sieve	0.053 to 0.002			Silt

Texture	No sieve	<0.002			Clay

19.0 CORRECTIVE ACTION

19.1 For QC samples that do not meet the method acceptance criteria, refer to §12.2. Refer to CAR344 memo for additional information regarding non-conformances. A copy is available from the QA/QC Officer.

19.2 For any SOP/method deviation the analyst must fill out section 1 of a minor corrective action report (FRMQA001). The department supervisor must fill out section 2. A project manager may need to fill out section 3, if applicable. The QA/QC Officer will close a minor corrective action. Attach a copy of the minor corrective action report to all workgroups affected.

19.3 For any system failure a major corrective must be opened and the problem investigated. A department supervisor or the QA/QC Officer can open a major corrective action. The corrective action will be assigned a unique tracking number by the QA/QC Officer and will be closed by the QA/QC Officer once the failure has been resolved. Use FRMQA001.