HOMOGENIZATION AND PULVERIZATION OF SOLID SAMPLES (Specific to the Retsch PM100 Planetary Ball Mill)

1. SCOPE AND APPLICATION

This method details the operating instructions for the Retsch planetary motion centrifugal ball mill (model PM200) and the specific procedures required to produce a homogenized and pulverized sub-sample suitable for preparation and analysis of environmental samples. Homogenization is important for the preparation and analysis of solid samples as most solid samples are heterogeneous by nature. This procedure is designed to generate a product with all its constituents as evenly distributed throughout the contents as is possible.

2. SUMMARY OF THE METHOD

A representative subsample is pulverized by means of a planetary motion centrifugal ball mill. Separate aliquots of the pulverized subsample are to be used for solid sample analysis.

3. APPARATUS AND EQUIPMENT

3.1. Calibrated balance capable of measuring up to 300 g.

3.2. Stainless steel spatulas.

3.3. Porcelain crucibles.

3.4. Glass pestle.

3.5. Polypropylene graduated 50 mL digestion tubes and caps, Environmental Express or equivalent.

3.6. Retsch PM200 planetary motion ball mill.

3.7. Tungsten carbide (or other suitable materials) grinding balls (dimensions can vary).

3.8. Tungsten carbide (or other suitable materials) cup with lid.

3.9. 15 mL and 50 mL polypropylene centrifuge tubes and caps, Corning or equivalent.

4. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Solid samples for metals analyses do not require preservation. The laboratory has established a six-month holding time for solid samples. Solid samples for metals analysis are stored in either refrigerator # 1 or # 8 in the receiving room.

5. SEDIMENT GRINDING & HOMOGENIZATION PROCESS:

5.1. Obtain custody of samples requiring homogenization from receiving room.

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5.2. In general, all samples logged in for test IDs S-ICP-TO, S-ICPMS-TO, S-TKN, S-TP, and S-TOC are milled following this procedure if the percent solid at 105 °C (see DEP SOP MT-055) is 20 % or greater. If the sample is less than 20 % solid, proceed to step 5.14 for test IDs S-ICP-TO, S-ICPMS-TO, and S-TOC. No further preparation necessary for test IDs S-TKN and S-TP.

5.3. Place the wet sample in grinding cup to fill ~75 %. Wet sample grinding produces a finer grain size after homogenization (~1 µg).

5.4. Place 30 grinding balls in each of the two grinding cups.

5.5. Place the cap on each cup making sure that the O-ring is in the groove in the cap.

Note: Ensure the two grinding cups with sample and lid are the same gross weight to avoid unpleasant vibrations while milling.

5.6. Open the lid on the grinder and place the sealed cups onto the turntable.

5.7. Place the legs of the 'spider' (the clamp to hold the cup in place) in the third set of notches and pull up on the red locking mechanism while tightening the bolt. The red locking mechanism should make a locking sound when the sample cup is secure.

Note: Locking sleeves which do not move downwards solely by spring power cannot properly prevent the threaded spindle from working loose. The grinding bowls could be hurled out.

5.8. Close the lid on the ball mill. The machine should lock the lid automatically.

5.9. Start grinding program # 1. The parameters are set to grind for 60 seconds at 500 RPM. If the sample does not appear to be sufficiently ground, then grind for an additional 30 seconds and continue grinding in 30 second increments until sufficiently ground. Note any additional grinding on the prep sheet.

5.10. Remove the sample cup upon completion of the grinding program. CAUTION: the sample cup may be HOT.

5.10.1. Lift up on red locking mechanism while loosening bolt. Remove the 'spider' and place to the side.

5.10.2. Remove the sample cup from the turntable.

5.11. Separate the sample from the grinding balls (by passing the sample through a no. 18 sieve or by using a stainless-steel spatula or transfer pipette) and collect the ground sample into a ceramic bowl labeled with a corresponding sample ID. Wash labware (sieve, spatula, pipette, etc.) according to DEP SOP MT-007 between each sample.

5.12. For samples requiring S-TKN and/or S-TP, collect a subsample of the ball-milled soil sample and place in a centrifuge tube labeled with the corresponding nutrient sample ID.

5.13. Calculate the percent solid content of the sample at 60 °C in the same manner as described in DEP SOP MT-055 (for determinations at 105 °C). Enter the data in

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the MT-60C-PS form located at \\floridadep\data\DEAR\Labs\Lab5\Chemistry\InorganicPrep\METALS\% SOLID LOG BOOK\PS60% & GRINDING-2025

5.14. The dried sample **MUST** be broken up with a glass pestle while using the ceramic drying dish as a mortar, or sample can be sandwiched between two disposable weighing dishes and broken up with a hammer to break up any organic material (i.e. leaves, grasses, sticks, etc.) or large chunks that will not pass through a no. 18 sieve. The sample sandwiched between two disposable weighing dishes should be placed in a zip-top plastic bag, such as Ziploc, to prevent the loss of dust while breaking down the dried sample.

5.15. Transfer the dried, broken up sample to a 50 mL polypropylene tube with cap for test IDs S-ICP-TO and S-ICPMS-TO and to a 15 mL centrifuge tube with corresponding nutrient sample ID for test S-TOC.

5.16. Label the sample tubes appropriately.

5.17. Generate a prep worksheet to change the S-TOC status from 'W' to 'V' using the Prep Worksheet Manager module in the LIMS. The Preparation ID that should be used is 'SOP-MT-064'. The Prep Start Date/Time should be the start day/time of the 60 °C percent solid procedure and the Prep Completion Date/Time should be the end of the 60 °C percent solid procedure.

6. CLEANING AND MAINTENANCE

Refer to the PM-200 operating instructions for details on maintenance.

6.1. Rinse the grinding cup with DI water and scrub with a brush to remove sample deposits. Rinse the inside of the cup and rim with 20 % aqua regia. Clean the grinding cup between each sample.

6.2. To clean the grinding balls, place them in a No. 18 sieve. Place the sieve into a tray filled with soapy water so that the grinding balls are submerged. Use a brush to clean the grinding balls. Use cleaned and dried grinding balls for each sample.

6.3. Check the roller of the locking trunnion for easy running and oil. If necessary, use mineral oil to lubricate.

6.4. Clean the magnets on the locking trunnion.

6.5. Check the threaded spindle and locking sleeve for easy running. A drop of mineral oil may be necessary for smooth operation.

6.6. The thickness of section D1 on the three 'spider feet' should be checked for wear. The thickness should not be less than 7.5 mm. Notify supervisor if the thickness of D1 is less than 7.5 mm. The 'spider' will need to be replaced.

6.7. Check the height of section H1 of the latching brackets. They should not be less than 17 mm. Notify supervisor if height is less than 17 mm.

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7. SAFETY/HAZARDOUS WASTE MANAGEMENT

7.1. Only use grinding bowls of type "C". This, in conjunction with the grinding bowl fixture, is a safety component.

7.2. Never allow threaded spindle (spider clamp) to lie loose in the grinding bowl fixture without the grinding bowls clamped in.

7.3. Always ensure that the red sleeve has latched in properly. Otherwise the grinding bowl is not securely clamped.

7.4. Review the Laboratory Health and Safety Plan.

7.5. Always wear proper protection in the laboratory. All samples are handled as if they contain hazardous amounts of all analytes. Gloves, eye protection, and lab coats should be worn when handling any sample.

7.6. Avoid the particulate matter produced from grinding samples. A wet grinding of solid samples is preferred to limit the production of aerosolized particles. If a sample is already dry, consider adding water to it prior to grinding and drying the sample after grinding in the ball mill (notify supervisor before adding water to any samples). The weight of the sample before and after adding the water must be recorded if water is added as a grinding aid. Otherwise, if a dry sample must be ground, only open the grinding cup in a hood to limit exposure to any aerosolized particles.

7.7. Leftover ground wet or dry sample can be disposed of in the trash can.

8. REFERENCES

8.1. Operation manual for the Retsch PM200 Planetary Ball Mill Grinder.

8.2. DEP SOP MT-055, Determination of Percent Dry Solids.

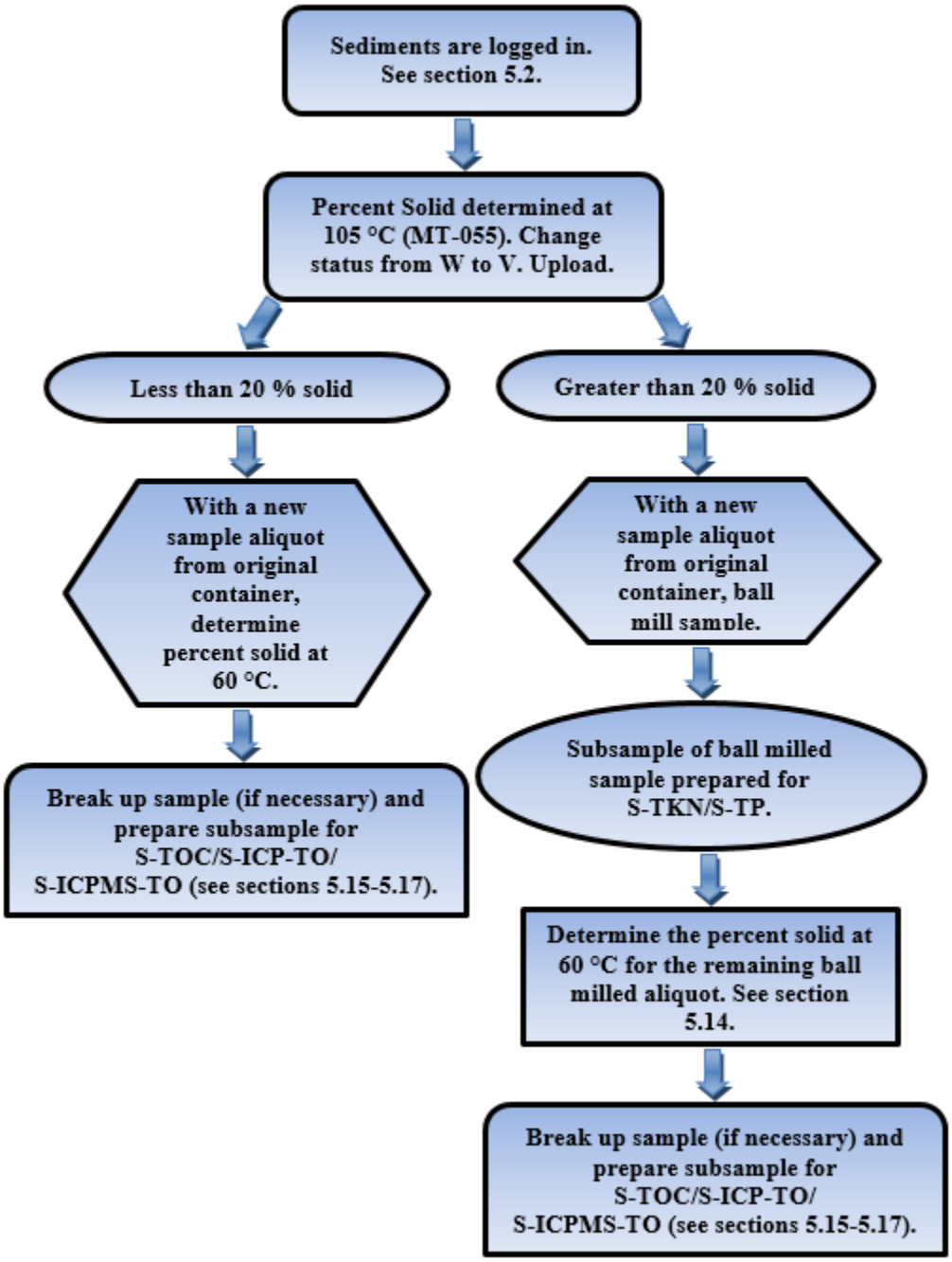
8.3. Laboratory Quality Manual.

8.4. Laboratory Health and Safety Plan.

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Appendix A

Procedure for Ball Milling Ambient and Other Sediments



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Appendix B

Appendix of Significant Change**s**

11/25/2009	Updated subsample collection procedures and sample preparation procedures in Section 5. Removed preservation requirement of keeping samples below 4 °C in section 4.
11/04/2010	Section 6.4 was updated to be consistent with other metals group SOPs.
10/12/2015	Section 5 edited for clarity and simplicity starting at section 5.15. Flowchart added as Appendix A.
10/25/2016	Minor edits; checked for 508 compliance.
11/27/2018	SOP updated to apply to the Laboratory's new PM200 ball mill.

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