Title: Preparation of Soil and Other Solid Samples

Approvals (Signature/Date):

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RL-PRP-003

REVISION 4

Facility Distribution No. _____ **Distributed To:_____**

1. SCOPE AND APPLICATION

1.1. This procedure describes the steps taken to prepare a soil, sediment or related matrix sample for radiochemical analysis. The goal is to provide a homogeneous starting material, free of extraneous matter from which portions representative of the entire sample may be taken for analysis. Samples are then acid digested or dissolved to bring any radionuclides from environmental contamination into solution.

1.2. Refer to Policy P-R-01 for method detection limit information.

2. SUMMARY OF METHOD

2.1. The objectives of this procedure are twofold. The first is to detail a procedure by which a homogeneous solid sample is produced, from which representative aliquots are taken for digesting or dissolution.

2.2. The entire sample is dried and milled, and an aliquot is sieved to remove extraneous material.

2.3. The second objective is to detail the procedure for the removal of radionuclides of interest by digesting an aliquot of the sample. The alkaline earth metals are separated from the digestate, leaving the other elements and actinide metals in solution. Large amounts of organic matter may first be removed by dry ashing at a high temperature. The soil is digested with nitric acid and hydrogen peroxide, while constantly stirring on a hot plate. This digestion method, while not ensuring complete dissolution of the sample, does solubilize the environmental contamination adsorbed onto surfaces of the matrix. The radionuclides in the digestate are then converted to chloride salts.

2.4. The chloride salts are dissolved in concentrated nitric acid. The alkaline earth elements (Ca, Sr, etc.) are separated by precipitation with fuming nitric acid for strontium analysis. The supernate may be evaporated and taken for actinide element analysis.

3. DEFINITIONS

3.1. Homogenized – A substance having uniform composition.

4. INTERFERENCES

4.1. None.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Minimum personal protective equipment consists of safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes.

5.1 Specific Safety Concerns or Requirements

- Close safety gate on ball mill before starting. Gate should remain closed until rollers have stopped.
- If an employee is required to work in the room with the ball mill in operation for more than two hours during a day, ANSI approved hearing protection must be used. A sign is posted on the door of this room to indicate the hearing protection requirement.
- When pulverizing samples, wait until pulverizer has stopped before removing sample. Caution: The pulverizer dish is heavy. Use proper lifting techniques when moving the dish.
- If an employee is required to work in the room with the pulverizer in operation for more than five hours during a day, ANSI approved hearing protection must be used. A sign is posted on the door of this room to indicate the hearing protection requirement.
- Remove samples from muffle furnace using Zetex® gloves and tongs.
- 16M HNO_3 and 30% H_2O_2, when mixed together, will liberate large quantities of oxygen. If these are mixed together and the container is sealed, pressure may build up causing a pressure explosion. Use extreme caution when wet-ashing samples.
- This method uses 90% Nitric (fuming) acid. Use extreme caution when handling this reagent. Rubber or heavy plastic gloves should be used.

5.2 Primary Materials Used

The following is a list of the materials used in this method that have a serious or significant hazard rating. **NOTE:** This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Balance - top loading, 2 kg capacity minimum, readable to 0.1 g.

6.2. Beakers - Pyrex glass and Teflon, sizes appropriate for sample.

6.3. Brush - soft bristle, for sieves and sieve pans.

6.4. Centrifuge.

6.5. Centrifuge tubes - 90 mL, polypropylene, with optional caps.

6.6. Containers - plastic jar or metal can approximately 4 L or other volume appropriate for the sample size, wide mouth, with appropriate lid.

6.7. Drying pan or container.

6.8. Filter - glass fiber, 1.2 micrometer pore size.

6.9. Flasks - Side-arm, size appropriate for filtrate.

6.10. Funnels - Buchner, various sizes; powder, large, separatory, size appropriate for sample, or disposable.

6.11. Furnace - muffle, capable of maintaining 500°C.

6.12. Gloves - Zetex®.

6.13. Grinding cylinders - 1¼ inch diameter by 1¼ inch height or metal grinding balls. One grinding ball is equivalent to 2 grinding cylinders.

6.14. Heat lamp.

6.15. Hot plates - stirring, magnetic.

6.16. Ice bath.

6.17. Ball Mill - mechanical rotation device for containers.

6.18. Oven - drying, forced-air, capable of maintaining 105°C.

6.19. Pen - felt tip, permanent black ink.

6.20. Pipet - transfer, glass, disposable, with rubber bulb.

6.21. Pulverizer - concentric ring.

6.22. Pump - mechanical vacuum, or aspirator.

6.23. Shaker - wrist action (if separatory funnel is used).

6.24. Sieves – various sizes, U.S. Standard, stainless steel or brass, 8" diameter, 2" height above cloth.

6.25. Sieve pans and lid to fit sieves.

6.26. Stir bars - Teflon-coated magnetic.

6.27. Stirring rods - glass and Teflon, length appropriate for beaker or tube.

6.28. Tape - plastic.

6.29. Tech Pen/ Pencil.

6.30. Timer (may be part of the shaker).

6.31. Tongs.

6.32. Tubing - vacuum, ¼" inside diameter.

6.33. Vacuum manifold for filtration.

6.34. Vortex.

6.35. Watch glass - Pyrex, appropriate size and Teflon, appropriate size.

6.36. Zetex® cloth.

7. REAGENTS AND STANDARDS

7.1. Reagents are prepared from analytical reagent grade chemicals unless otherwise specified below. Reagent water, which must have an electrical resistivity of 1 megohm-cm or greater when obtained, is used throughout. Reagent water is obtained from the Nano-pure system. Label all reagents as outlined in procedure RL-RPL-001.

NOTE: Consult the Material Safety Data Sheets for the properties of these reagents and how to work with them.

7.2. 12M Hydrochloric acid (12M HCl) - concentrated. **WARNING**: Corrosive.

7.3. 8M Hydrochloric acid (8M HCl)- Add 670 mL of concentrated HCl to approximately 200 mL of reagent water. Dilute to 1 L with reagent water and mix well. **CAUTION**: Corrosive.

7.4. 30% Hydrogen peroxide (30% H_2O_2). **WARNING**: Corrosive and Oxidizer.

7.5. 90% Nitric acid (90% HNO_3) - Fuming. **WARNING**: Corrosive and Oxidizer.

7.6. 16M Nitric acid (16M HNO_3) - Concentrated. **WARNING**: Corrosive and Oxidizer.

7.7. 8M Nitric acid (8M HNO_3) - Add 500 mL 16M HNO_3 to approximately 400 mL reagent water. Dilute to 1L with reagent water and mix well. **WARNING**: Corrosive and Oxidizer.

7.8. 2M Nitric acid (2M HNO_3) - Add 125 mL of 16M HNO_3 to 800 mL of reagent water. Dilute to 1 L with reagent water and mix well.

8. SAMPLE COLLECTION, PRESERVATIVES, SHIPMENT AND STORAGE

The sample may be collected in glass or plastic containers using no preservatives. Storage of the sample prior to analysis should not exceed six months.

9. QUALITY CONTROL

9.1. All quality control data shall be maintained and available for easy reference.

9.2. Yield monitors (carriers and tracers) and QC spikes are prepared with a pre-set mass and/or activity and distributed appropriately in coded vials for use during sample analysis. Consult the latest version of the client specific Quality Assurance Summary (QAS) for the appropriate yield monitors, spikes, carriers, and/or tracers to use.

9.3. Consult the QAS for client specific information regarding QC frequency.

9.4. Refer to SOP RL-DR-001 for sample and QC data acceptance criteria and corrective action.

10. CALIBRATION

10.1. None.

11. PROCEDURE

NOTE: If any parameter is found to be out of limits, consult supervision. Also, a nonconformance will be issued to the Quality Assurance Group.

NOTE: One time procedural variations are allowed if deemed necessary by the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size or other parameters. Any variation in procedure shall require approval by supervision and immediate notification of the Quality Assurance Group. If contractually required, the client shall be notified prior to any procedure changes. A Nonconformance Memo shall be completed and forwarded to the Quality Assurance Group within one day of the supervisor's approval. The Nonconformance Memo will be filed in the project file.

11.1. Label an appropriate number of containers with the work order number. Tare the containers and record the tare weight on the Soil Drying Worksheet. Transfer the corresponding sample into the appropriate container. Weigh each container and record the weight on the Soil Drying Worksheet.

11.2. **NOTE**: If tritium and/or carbon-14 analyses are required, the tritium and/or carbon-14 aliquot must be removed prior to drying the sample.

Dry the sample by placing it into an oven set at approximately 105°C, for at least 8 hours or until dry.

NOTE: Consult supervision for analyte and client specific drying temperature and time.

CAUTION: Do not dry the aliquot to be used for **Tc-99** analysis above 95°C.

11.3. After drying, cool and weigh the sample and container. Record the dried weight on the Soil Drying Worksheet. If percent moisture is requested, enter the appropriate information on the Percent Moisture Form and calculate (see Figure 1).

NOTE: Consult supervision prior to proceeding regarding specific client contractual requirements for sieving and/or ball milling. Record client specific information on the analytical work sheet or in the sample preparation tab in RadCalc and/or the Drying Worksheet.

11.4. Ball Milling

NOTE: If an employee is required to work in the room with the ball mill in operation for more than two hours during the day, ANSI approved hearing protection must be used. A sign shall be posted on the door of this room to indicate the hearing protection requirement.

NOTE: Samples may have been dried in the ball mill container. Prior to the addition of the grinding cylinders, ensure that the sample is not a solid mass. If a solid mass is present, break up the sample and transfer to a clean container prior to adding the grinding cylinders.

11.4.1. Add the following to a container of appropriate size: one large, porcelain grinding cylinder per 200g or fraction of sample weight, plus one additional cylinder

NOTE: One metal grinding ball is equivalent to 2 grinding cylinders. If metal grinding balls are used, do not use more than 12 balls in a large can or the integrity of the can may be compromised.

11.4.2. Close the container tightly and label the lid of the container with the work order number.

11.4.3. Place the container on its side on a set of ball mill rollers. Start the mill. Allow samples to roll for at least 1 hour.

NOTE: Consult supervision for client-specific time requirements.

11.4.4. Stop the mill and remove the container.

11.4.5. If the sample requires pulverizing, continue to section 11.5. If pulverizing is not required, proceed to Sieving, section 11.6.

11.5. Pulverizing

NOTE: If an employee is required to work in the room with the pulverizer in operation for more than five hours during the day, ANSI approved hearing protection must be used. A sign shall be posted on the door of this room to indicate the hearing protection requirement.

11.5.1. If the sample has been ball milled, remove the cylinders and brush off any excess sample into the container.

11.5.2. Place the sample in the pulverizing bowl located inside the pulverizer. Pulverize for the appropriate time.

NOTE: If the entire sample does not fit in the pulverizer at one time, recombine all pulverized fractions into the original milling container and roll for an additional 60 minutes before sieving.

11.6. Sieving

11.6.1. **NOTE:** If the aliquot or initial sample mass is appreciably over 1.0 kg, it is advisable to sieve the sample in more than one portion.

NOTE: Consult the QAS or supervision for client-specific sieve size requirements. Record the client specific information on the analytical worksheet and/or in the Sample Preparation logbook.

Set a clean, dry No. 10 mesh (2 mm) and/or a No. 120 mesh (125 μm) stainless steel or brass sieve, or the particular sieves specified by the client, on top of a clean receiving pan.

NOTE: For samples requiring alpha/ beta analysis, use a 120 sieve. For samples requiring only gamma analysis, use a 10 sieve. For samples requiring any other analysis, use an 80 sieve.

11.6.2. Transfer sample into the sieve stack, using appropriate step below:

11.6.1.1. Open the pulverizer and remove the bowl from the pulverizer. Transfer an appropriate aliquot of the homogenized sample onto the sieve. Tap or shake the pulverizing bowl over the sieve to remove any adhering sample.

11.6.1.2. If the sample has not been pulverized, open the container and remove the cylinders and brush off any excess sample into the container. Transfer an appropriate aliquot of the homogenized sample or the entire sample onto the sieve.

11.6.3. Shake the sieve assembly for about one minute.

11.6.4. Open the assembly and transfer the sample in the receiving pan to an appropriately labeled sample container.

11.6.5. Return the residue retained on the No. 10 mesh sieve to the original sample container.

11.6.6. If additional pulverizing is required upon completion of sieving, repeat section 11.5.

NOTE: Certain clients require that each sample be divided for duplicate analyses at TestAmerica Richland and another laboratory. If the client-specific QAS or supervision requires the sample to be split at this point, go to Attachment A for instructions.

11.6.7. Transfer weighed aliquots of the sample into clean, labeled beakers (the weights vary depending on analysis). If applicable, transfer weighed aliquots of matrix material to the blank and LCS sample beakers.

NOTE: The sample may be transferred to a plastic jar for aliquoting at a later date.

Record all aliquot weights. Proceed to step 11.7 for acid digestion or to the appropriate procedure for dissolution.

11.7. Soil and Sediment Acid Digestion

11.7.1. Add the specified yield monitors for the analyses requested to all beakers. Rinse the vials three times with about 5 mL of 8M HNO_3, (use 2M HNO_3 when transferring strontium carrier and spikes) and add the rinses to the beaker to assure quantitative transfer of the yield monitor solutions. Record the vial labels.

11.7.2. Add spikes from the vials to the appropriate beakers. Transfer the vial contents as described in the previous step. Record the vial labels.

11.7.3. Evaporate the samples to dryness if muffling is required.

CAUTION: Do not muffle if **Pb-210** analysis has been requested instead proceed to step 11.7.5(The melting point of lead is 327^0 C.). Do not muffle the aliquot to be used for **Tc-99** analysis.

NOTE: Ensure the muffle furnace is set no higher than 500°C. When the muffle is greater than 500°C the glass beaker could melt.

11.7.4. **NOTE**: If samples do not appear to contain significant organic matter, muffling may not be required. Consult supervision.

When muffling is required (see supervision for instructions), use a TECH PEN or HI-TEMP PENCIL to label the sample beaker with the work order number. Place the beaker with the sample in an unheated muffle furnace. Set the temperature of the furnace to approximately 500°C and turn on the furnace. Muffle the sample at this temperature for at least 6 hours.

11.7.5. Remove the beaker from the muffle furnace using Zetex® gloves and tongs, and let the beaker cool on a Zetex® pad.

11.7.6. Add about 50 mL of 8M HNO_3 to appropriately labeled blank, LCS, and cooled sample beakers (the volume of 8M HNO_3 required may be less depending on the sample size and the analysis requested).

11.7.7. Add a large magnetic stir bar to each labeled beaker and place the beaker on a magnetic stirring hot plate in a fume hood. Place a watch glass on the beaker.

11.7.8. Adjust the temperature so that the solution just boils. Allow the sample to reflux for about 45 minutes with constant stirring. Wash down the sides of the beaker periodically with small amounts of 8M HNO_3.

11.7.9. After heating, remove the sample from the hot plate and allow the soil to settle. Decant most of the solution into a clean, labeled beaker.

11.7.10. Add about 50 mL of 2M HNO_3 to the residue, and rinse the beaker walls with 2M HNO_3 (the volume of 2M HNO_3 required may be less depending on the sample size or analysis requested). Replace the watch glass on the beaker. Reflux for about 45 minutes with constant stirring. Remove the sample from the hot plate and allow the soil to settle.

11.7.11. **NOTE**: Make sure vacuum is applied to the Buchner funnel prior to decanting the samples.

Transfer the solution from step 11.7.9 into a Buchner filter funnel fitted with a moistened glass fiber filter. Collect the sample digestate under vacuum in a side-arm flask. Rinse the beaker with a small amount of 2M HNO_3 and add the rinsate to the funnel/filter.

NOTE: A disposable filter may be used.

NOTE: Samples may be centrifuged instead of filtered. If the sample is centrifuged, rinse the beaker with small amounts of reagent water, adding the rinses to the centrifuge tube. Centrifuge for approximately 10 minutes. Decant the supernate to a clean labeled beaker. Rinse the sample residue with a small amount of 2M HNO_3. Centrifuge for approximately 10 minutes and decant to the appropriate beaker.

11.7.12. Decant the 2M HNO_3 solution containing the soil residue from step 11.7.10 through the same Buchner funnel and filter paper with vacuum on. Rinse the beaker with 2M HNO_3 and add to the funnel. Continue to apply vacuum until the soil residue is free of visible liquid and liquid ceases to drip from the funnel. Rinse the filtered residue with up to 25 mL reagent water.

11.7.13. Transfer the filtered digestate from the side-arm flask back into the beaker from step 11.7.9. Rinse the flask with small volumes of 2M HNO_3 and add the rinsate to the beaker. For Tc-99 samples procedure to the appropriate separations procedure at this point.

11.7.14. Place the beakers on a hot plate and evaporate to **just dryness**. **DO NOT BAKE**. When salts start appearing, cover the sample with a watch glass and lower the temperature setting of the hot plate to minimize losses due to spattering. Wet ash the samples with small amounts of concentrated HNO_3 and 30% H_2O_2 at least three times, more if necessary. Evaporate to **just dryness**.

11.7.15. Strontium only requests may proceed to the appropriate strontium procedure. The strontium sequential analysis continues at Section 11.8. Proceed to the appropriate analytical procedure for isotopic analyses.

11.8. Fuming Nitric Acid Separation

NOTE: This section is a continuation of the Strontium sequential analysis.

11.8.1. **WARNING**: Fuming nitric acid is used in the next several steps. It is hazardous, especially when brought in contact with organic materials including skin. Exercise extreme care. Rubber or heavy plastic gloves should be used. Immediately rinse any area contacted with fuming nitric acid.

Suspend, and if possible dissolve, the sample residue in a minimum amount of concentrated HNO_3 while stirring. Heat briefly if needed. If necessary, use a stir rod to loosen all solids sticking to the beaker and break up all large pieces.

11.8.2. Transfer the sample to a labeled 90 mL plastic centrifuge tube, rinsing the beaker and rod (if used) with a minimum amount of concentrated HNO_3. Larger samples may require two or more centrifuge tubes. Vortex the remaining solids to ensure that any large pieces are broken up. Add at least 50 mL of fuming nitric acid to each tube.

NOTE: It is recommended that the centrifuge tubes be capped. Capping the tubes before centrifuging can minimize the fumes.

11.8.3. Cool in an ice bath for 20-30 minutes. Remove from the ice bath and wipe the outside of the tube dry. Centrifuge until the supernate is clear (at least 10 minutes). Save the supernate for plutonium and/or other actinide analyses, if requested, and transfer to a labeled beaker.

11.8.4. If the precipitate is less than 20 mL per tube, proceed to 11.8.6. To precipitates greater than 20 mL per tube, vortex to break up the precipitate and add about 20-30 mL of fuming nitric acid. Thoroughly mix the precipitate with the acid. Centrifuge, and decant the supernate into the beaker with the supernate from step 11.8.3. The final precipitate from this step or step 11.8.3 will be used for strontium analysis, if requested.

11.8.5. Evaporate the supernate to dryness when plutonium or other actinides are requested. The warm hot plate may be covered with Zetex® to prevent spattering.

11.8.6. Proceed to the appropriate analytical procedure with each fraction and analytical worksheet(s).

12. DATA ANALYSIS AND CALCULATIONS

NOTE: Consult the client specific QAS or supervision to determine the appropriate calculation to be used.

12.1. Percent Moisture (Wet Sample Weight):

$$\text{Percent Moisture} = \left(\frac{M_i - M_d}{M_i - M_p} \right) 100$$

where:

M_i = the combined weight of the initial sample and the pan weight in grams.

M_d = the combined weight of the dried sample and the pan weight in grams.

M_p = the pan weight in grams.

12.2. Percent Moisture (Dry Sample Weight):

$$\text{Percent Moisture} = \left(\frac{M_i - M_d}{M_d - M_p} \right) 100$$

where:

M_i = the combined weight of the initial sample and the pan weight in grams.

M_d = the combined weight of the dried sample and the pan weight in grams.

M_p = the pan weight in grams.

13. METHOD PERFORMANCE

13.1. The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use.

13.2. Method Demonstration of Capability documentation is maintained in the quality files.

14. POLLUTION PREVENTION

14.1. Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents to be disposed.

15. WASTE MANAGEMENT

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

The following waste stream is produced when this method is carried out.

- Aqueous acidic waste pH<2. The waste is collected in an appropriate container and transferred into an Acid Waste container color coded with Yellow tape and a Yellow cap.

16. REFERENCES

16.1. Chieco, N.A., D.C. Bogen, and E.O. Knutson. eds. 1990 EML Procedures Manual, Pu-02a, HASL-300, 27th Edition, Volume 1. Environmental Measurements Laboratory, US Department of Energy, New York, New York.

16.2. Dolezal, J., P. Povondra, and Z. Sulcek, Decomposition Techniques in Inorganic Analysis, American Elsevier Publishing Co., New York (1968).

16.3. Brewer, S. W., Office of Water Resources Research, U.S. Department of Commerce, Report PB-220949 (1973).

16.4. Shuster, C.N. and B.H. Pringle, Proc. Nat. Shellfisheries Assoc., 59 91 (1969).

16.5. Jones, A.S.G., Marine Geol., 14, 1 (1973).

16.6. Knauer, G.A. and J.H. Martin, Limnol. Oceanogr. 18 (1973) 597.

16.7. American Society of Testing and Materials. "Standard Method for Soil Sample Preparation for the Determination of Radionuclides", C 999-83, in Annual Book of ASTM Standards, v. 12.01 ASTM, Philadelphia.

16.8. American Society of Testing and Materials. "Determination of Water (Moisture) Content of Soil and Rock", D2216-92, in Annual Book of ASTM Standards, v. 12.01 ASTM, Philadelphia.

16.9. RL-QAM-001, TestAmerica Richland Laboratory Quality Assurance Manual, latest revision.

16.10. Associated SOPs

16.10.1. RL-ALP-001 – Actinides by Extraction Chromatography

16.10.2. RL-ALP-005 – Isotopic Thorium in Environmental Matrices

16.10.3. RL-DR-001 – Review of Environmental and Bioassay Data

16.10.4. RL-PRP-010 – Urine and Water Sample Preparation by Calcium Phosphate Precipitation

16.10.5. RL-RPL-001 – Reagent and Non-Radioactive Standards Labeling

17. MISCELLANEOUS

17.1. Responsibilities

Analyst: Implements SOP as written.

Counting Room: Performs review on raw instrument data.

Technical Data Reviewer: Performs final data review.

Project Manager: Confirms final review and prepares data for reporting to client.

QA Manager: Performs product quality assessments as defined in the Quality Assurance policies.

17.2. Records Management/Documentation

17.2.1. All records generated by this analysis will be filed and kept in accordance with STL QA policies for records management and maintenance

18. REVISION HISTORY

- Revision 1, 5/27/2010
  - Updated text to remove prior corporate references.
  - Updated text to point user to Attachment A when samples are to be split.
- Revision 2, 7/26/2011
  - Section 11.5.2 removed description of bowl
  - Section 11.6.1 added note
- Revision 3, 8/20/2012
  - Section 11.4.1 was updated.
  - References to logbook in regards to the soil drying log were changed to log sheet.
- Revision 4, 05/20/2013
  - Added '& Other Solids' to title
  - Replaced 'Lab ID' with 'work order number' throughout.
  - Removed notes following step 11.7.14.

18.1. Procedural Flow Chart

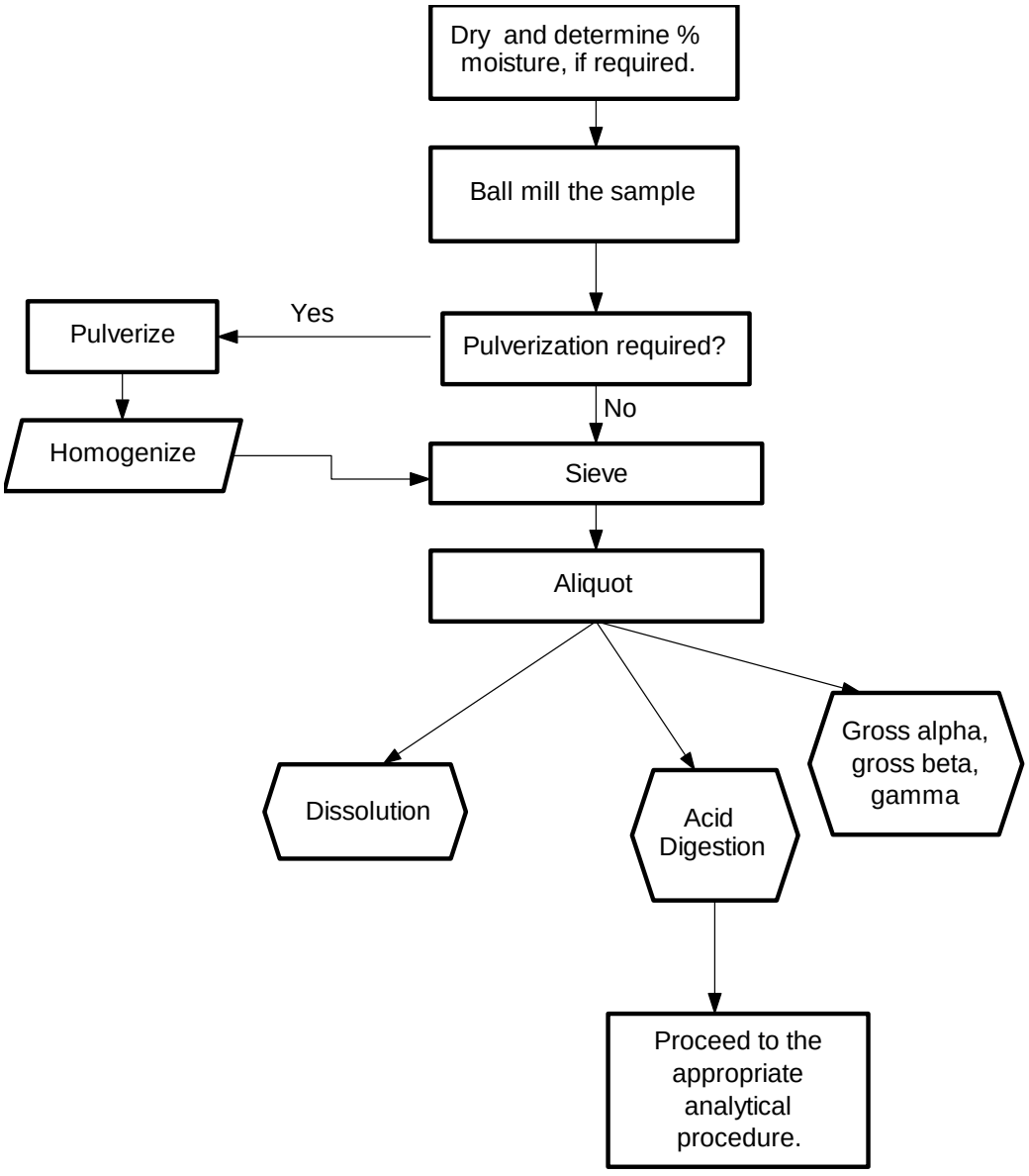

Figure 1: Percent Moisture Determination

Percent Moisture

Batch No.: #######

Matrix: Sediment

Client:,

Analyst:

Sample Identifier	Weight of Empty Container (A)	Container + Fraction (B)	Dry Sample + Container (C)	Moisture% (Wet Wt)= (B-C)/(B-A) *100	Moisture% (Dry Wt)= (B-C)/C-A) *100	Soilids%= 1-(B-C)/(B-A)
HLFV9-1AC	173.0	226.7	216.5	19.0	23.4	81.0
HLFWJ-1AC	172.5	228.1	217.1	19.8	24.7	80.2
HLFWM-	173.0	218.4	212.6	12.8	14.6	87.2
HLMLA-1AC	172.2	224.2	21.8	20.0	25.0	80.0
HLMLK-1AC	172.7	238.5	224.7	21.1	26.8	78.9
HLMLM-1AC	172.2	204.3	198.8	17.1	20.7	82.9
HLMLP-1AC	172.3	223.9	217.5	12.4	14.2	87.6
HLMLQ-1AC	171.8	229.7	214.0	27.1	37.2	72.9
HLMLT-1AC	171.8	243.0	230.5	17.6	21.3	82.4

Comments

ATTACHMENT A

FMC-Idaho Client Specific Instruction

1. Soil and rock samples submitted for analysis will be dried, ground, pulverized and sieved as described in steps 11.1 through 11.6.6 of this procedure. If the sample must be split for analysis at another facility, proceed to step 2.
2. Split the samples using "Alternate Shoveling". Take a series of scoops selected randomly from the entire sample, depositing the alternate scoops in two piles containing an equal number of scoops.

   NOTE: The minimum number of scoops should be nine for each pile. Small scoop sizes should be used to achieve lower grouping and segregation error. Repeat if necessary until enough sample is divided for additional analysis.
3. Package the portion of sample that is to be shipped to another facility according to client-specific instructions. Proceed to step 11.6.7 to continue analysis.