AQUEOUS EXTRACTION OF ORTHOPHOSPHATE, NITRATE, CHLORIDE AND SULFATE FROM SOILS AND SEDIMENTS

1. SCOPE AND APPLICATION

1.1. This extraction technique is intended to leach water-soluble (or 'available') orthophosphate, nitrate, chloride, and sulfate anions from soil and sediment samples.

1.2. The resulting aqueous extract is analyzed as a water sample using the appropriate analytical method:

1.2.1. Orthophosphate - See DEP SOP NU-070.

1.2.2. Sulfate, chloride and nitrate - See DEP SOP NU-083.

2. SUMMARY OF THE METHOD

A homogeneous sub-sample of the soil or sediment is mixed thoroughly with deionized (DI) water using an automated shaker and allowed to settle. The liquid extract is then decanted and analyzed.

3. EQUIPMENT AND SUPPLIES

3.1. Platform shaker

3.2. Nalgene disposable wide mouth 250 mL bottles with cap

3.3. 100 mL graduated cylinder (Class A) or bottle-top dispenser

3.4. 50 mL capped plastic centrifuge tubes

4. REAGENTS AND STANDARDS

Note: Archive the preparation of the standards in the Standard Preparation Tracker in the Laboratory Information Management System (LIMS). Unique ID numbers for each standard are generated in the module and documented in the appropriate analysis standards sheet submitted with each analysis folder or in the prep worksheet created in LIMS. A Certificate of Analysis for each purchased standard is maintained in the binder kept in C307. All containers holding standards must be labeled with their unique ID and expiration date. See the appropriate analysis SOP for specific information about reagents and standards.

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Note: Before preparing standards, or running samples, please read the safety notes in the safety/hazardous waste section of this SOP and in the appropriate analysis SOP, and the MSDS for the chemicals used in the analysis.

4.1. DI water

4.2. For spike solutions etc. see relevant analytical SOP (1.2.).

4.3. Ottawa Sand Standard (20-30 mesh) - For preparing extraction blanks and laboratory fortified blanks (LFBs).

5. SAMPLE HANDLING AND PRESERVATION

Extracted samples are stored refrigerated. The holding times for the samples are the same as for the equivalent aqueous samples, 48 hours from the time of sampling for orthophosphate and nitrate analysis, and 28 days for sulfate and chloride analysis. Treat all samples as potentially hazardous.

6. SAMPLE PREPARATION AND HANDLING

6.1. Create a new extraction prep worksheet which can be found under the prep ID "EXT-AQ" in the Prep Worksheet Manager module of LIMS. Complete the upper part of the sheet, which contains some general information including: worksheet ID, analyst's name, SOP (NU-044), spiking solution serial number and units, start and completion date and time of the extraction, shaker equipment ID, balance ID, prep comments, and review information.

Update the table below the general information section. Add spike codes and amounts for spiked sample IDs, serial numbers for the standard solutions used, sample wet weights, and sample comments (if needed). Make sure test IDs and other auto-filled information such as standard IDs, matrix, % solid, and reprep info for samples are correct and updated. Check that sample dry weight calculations are correct. Prep batch IDs are assigned in the worksheet. Ask a supervisor to review the worksheet.

6.2. Sample homogenization (for soils and sediments) - If the sample cannot be thoroughly mixed within its container without spilling, empty the entire sample into a stainless steel tray. Remove any rocks, shells, twigs, leaves, etc. from the samples and mix thoroughly to achieve homogeneity. A mortar and pestle may be used to break up any clumps. Return the sample to the jar.

6.3. Weigh out approximately 5.0 g of the sediment or soil sample into a pre-labeled 250 mL container. This weight may be varied depending on the expected concentration of the analyte. Record the exact weight of the sample on the extraction sheet.

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Sample weighing procedure:

- Check the maintenance book of the balance to confirm that the balance calibration was checked on the day you intend to use it. If it was not done, perform balance calibration checks (see DEP SOP CM-011 for detailed instructions). Record the balance ID in the prep worksheet.
- Place a pre-labeled 250 mL container onto an analytical balance and tare the balance so that it reads 0.0000 g. Using a clean stainless steel spatula, carefully transfer enough homogenized sample to the weighing boat to yield 5.0 g +/- 0.01 g of material. The QC sample (duplicates and/or spikes) weights should be within 0.005 g of each other. Record the weight under "sample wet weight" in the prep worksheet, and check the % solid information and calculated sample dry weight for each sample.

6.4. Using a 100 mL graduated cylinder or bottle-top dispenser, add 100 mL of DI water to the sediment. If the sample is a spike or LFB, also add the correct amount of spiking solution to the sample (see 7.3.). Record on the extraction preparation worksheet the serial number and amount of each spiking solution added.

6.5. Shake the mixture on the mechanical platform shaker for 30 minutes at 150-200 rpm.

6.6. Allow the sediment to settle overnight in the refrigerator.

6.7. Decant 50 mL of the liquid extract into a pre-labeled plastic 50 mL centrifuge tube. Discard the remainder of the extract. For chloride and sulfate, wait at least 4 days after decanting to analyze samples to allow for further extraction.

6.8. For all analyses, the start day/time is when the samples are placed on the shaker. The finish day/time is when the last sample has been decanted. The preparation day/time in LIMS will be the same as the start day/time. Store the extracts in the refrigerator until analysis. Samples/extracts must not be stored in refrigerators containing standards.

6.9. Make sure that the prep worksheet in LIMS has been reviewed, then complete the worksheet; this changes sample status from “W” to “V”.

6.10. When analyzing the extracts, pre-filter the aliquot to be analyzed using a polypropylene syringe filter with a 0.45 µm nominal pore size.

7. QUALITY CONTROL SAMPLES

An extraction batch is defined as a set of 20 samples or less. Each extraction batch must be prepared with its own set of QC samples. Randomly choose a sample from each batch to be

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used as the quality control sample. See relevant analytical SOP or supervisor for QC acceptance limits (1.2.).

7.1. Extraction Blank - One blank per batch, prepared and filtered in the same way as a sample, using Ottawa sand as the matrix.

7.2. Sample Duplicate (Precision) - The precision may be determined from a sample duplicate or a spike duplicate. Generally, because the concentrations of the analytes are not known in advance, both sample and spike duplicates are prepared for each batch, using the same sample for the sample duplicates and to prepare the matrix spikes (7.3.1.).

7.3. Spikes (Accuracy/Precision):

7.3.1. Sample Matrix Spikes - Use the same spiking solutions and spike concentrations that are used for water sample analysis. The amount of spiking solution added is ten times the amount added for water samples, because the final volume of the extract is 100 mL, compared to 10 mL for a water sample.

7.3.2. LFB - One LFB per batch is prepared in the same way as a matrix spike, using Ottawa sand as the matrix.

8. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

8.1 Review the Laboratory Health and Safety Plan (accessible on the intranet at https://www.fldepnet.org/system/files/Division-of-Environmental-Assessment-and-Restoration/governing-documents/Health%20%20Safety%20Plan_0.docx).

8.2. Safety glasses, gloves and lab coats should be worn at all times whilst handling samples and reagents.

8.3. Read the MSDS information for safe reagent and sample handling.

8.4. Excess sample should be disposed of in the appropriate waste container.

9. REFERENCES/DEFINITIONS/VALIDATION DATA

For definitions see section 3 of the FDEP Quality Manual. All abbreviations used throughout the document are defined at least once in the document usually at the first time of use.

9.1. DEP SOP NU-070, Analysis of Ortho Phosphate using the Bran+Luebbe Segmented Flow Analyzer.

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9.2. DEP SOP NU-083, Determination of Sulfate, Nitrate, Chloride, and Bromide using the Metrohm Ion Chromatography System.

9.3. DEP SOP CM-011, Gravimetric Analysis: Analytical Weight and Balance Calibration

9.4. Quality Manual, Bureau of Laboratories, FDEP.

9.5. Laboratory Health and Safety Plan.

Appendix of Significant Changes

05/11/2015 Information about creating and completing prep worksheets in the Prep Worksheet Manager module in LIMS was added to Section 6.

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