SULFATE IN SOILS

(An Arizona Method)

1. SCOPE

1.1 This test method describes a procedure for determining sulfate content in soil using a turbidimeter. The sulfate content determined is defined in terms of the method and may be called water soluable sulfate.

1.2 This test method involves hazardous material, operations, and equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.3 See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

1.4 The extraction procedure, Subsections 4.1 through 4.4, is the same as is used in Arizona Test Method 736, “Chloride In Soils”.

2. APPARATUS

2.1 Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus for this test procedure shall consist of the following:

2.1.1 Turbidimeter - with at least ± 2% accuracy to 1000 NTU (nephelometric turbidity units) and at least ± 5% accuracy to 4000 NTU, with sample cuvettes.

2.1.2 Balances or scales:

2.1.2.1 One balance or scale capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M 231, except the readability and sensitivity shall be at least 0.1 gram.

2.1.2.2 An analytical balance capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M231, except the readability and sensitivity shall be at least 0.1 milligram.

2.1.3 Beaker(s) - three, 200 mL capacity each.

2.1.4 Erlenmeyer flask - 500 mL capacity with stopper.

2.1.5 Volumetric flask - 1000 mL capacity, accurate to 0.3 mL.

2.1.6 Reagent dispensing bottle - 500 mL capacity, capable of repeatedly dispensing 5 mL of reagent.

2.1.7 Reagent storage bottles - three, one-liter capacity, each with cap.

2.1.8 Dropping bottle - 60 mL, with dispensing tip or dropper.

2.1.9 Centrifuge tube - 50 mL, with cap.

3. REAGENTS

3.1 Conditioning Reagent.

3.1.1 Into a reagent storage bottle, add 300 mL demineralized water, 30 mL Reagent Grade Concentrated Hydrochloric Acid, 100 mL Reagent Grade Isopropyl Alcohol, 75 grams Reagent Grade Sodium Chloride, and 50 mL Reagent Grade Glycerol. Mix well. Transfer to a reagent dispensing bottle as needed.

3.2 Sulfate Standard Solution, 0.100 mg/mL (100 ppm).

3.2.1 Into a 1000 mL volumetric flask, add 147.9 mg Reagent Grade Anhydrous Sodium Sulfate. Fill the flask to the 1000 mL mark with demineralized water. Mix well. Transfer to a reagent storage bottle.

3.3 Barium Chloride Reagent Solution, 1%.

3.3.1 Into a 1000 mL volumetric flask, add 10 grams of Reagent Grade Barium Chloride Powder, Anhydrous. Fill the flask to the 1000 mL mark with demineralized water. Mix well. Transfer to a reagent dispensing bottle as needed, and the remainder to a reagent storage bottle.

3.4 Nitric Acid, 20%.

3.4.1 Measure 10 mL concentrated nitric acid into a suitable size beaker containing approximately 40 mL demineralized water and mix well. Transfer to a dropping bottle.

CAUTION: Exercise extreme caution in preparing and using the Nitric Acid solution. It must be properly labeled and treated as a hazardous material.

3.5 Demineralized water.

4. PROCEDURE

4.1 Weigh 100.0 ± 0.1 grams of soil passing a No. 10 sieve into a 500 mL Erlenmeyer flask.

4.2 Weigh 300.0 ± 0.1 grams demineralized water into the flask.

4.3 Stopper the flask, shake vigorously, and let the mixture stand undisturbed for one hour.

4.4 Carefully, with minimal disturbance of the sediment, decant 50 mL of the extract solution into a centrifuge tube.

4.4.1 If the decanted extract solution is clear, proceed to Subsection 4.5.

4.4.2 If the decanted extract solution is not clear, place the tube into the centrifuge and centrifuge at a minimum of 5000 RPM for 10 minutes. If the solution is still not clear, add 2 drops of 20% Nitric Acid solution to the tube and centrifuge again. Repeat until the centrifugate is clear.

4.5 Transfer 20.0 mL of the clear extract solution (or centrifugate) into a 200 mL beaker. This is the sample aliquot, “ALSAM.”

4.5.1 Dispense 5 mL of Conditioning Reagent and 5 mL of Barium Chloride Reagent Solution into the beaker, and dilute to 100 mL. This is the “Sample Solution.”

4.6 Transfer 20.0 mL of Sulfate Standard Solution into a second 200 mL beaker. This is the standard aliquot “ALSTD.”

4.6.1 Dispense 5 mL of Conditioning Reagent and 5 mL of Barium Chloride Reagent Solution into this beaker, and dilute to 100 mL. This is the "Standard Solution."

4.7 Dispense 5 mL of Conditioning Reagent and 5 mL of Barium Chloride Reagent Solution into a third 200 mL beaker, and dilute to 100 mL. This is the "Reagent Blank Solution."

4.8 Stir the three solutions and let them stand undisturbed for at least 15 minutes.

4.9 Rinse a sample cuvette with demineralized water and then with freshly stirred Reagent Blank Solution. Immediately transfer enough Reagent Blank Solution to fill the cuvette to the mark.

4.9.1 Place the cuvette into the turbidimeter and read the turbidity to the nearest 0.1 NTU. The turbidity of the Reagent Blank Solution is recorded as "TBBNK".

4.10 Rinse a sample cuvette with demineralized water and then with freshly stirred Sample Solution. Immediately transfer enough Sample Solution to fill the cuvette to the mark.

4.10.1 Place the cuvette into the turbidimeter and read the turbidity to the nearest 0.1 NTU. Record the turbidity of the Sample Solution as "TBSAM."

4.11 Rinse a sample cuvette with demineralized water and then with freshly stirred Standard Solution. Immediately transfer enough Standard Solution to fill the cuvette to the mark.

4.11.1 Place the cuvette into the turbidimeter and read the turbidity to the nearest 0.1 NTU. Record the turbidity of the Standard Solution as "TBSTD."

4.12 Compare the values of "TBSAM" and "TBSTD." If "TBSAM" is larger than "TBSTD", repeat Subsections 4.5 and 4.5.1 using a suitably smaller sample aliquot. Record this volume, to the nearest 0.1 mL, as "ALSAM." Repeat Subsections 4.10 and 4.10.1.

5. CALCULATION AND REPORT

5.1 Calculate sulfate content in the soil in parts per million, "S", and report to the nearest 10 ppm as follows:

$$S = 300 \times \left[\frac{ALSTD \times TBSAM}{ALSAM \times TBSTD} \right]$$

Where:

ALSTD = 20.0 mL (Size of the standard aliquot).
TBSTD = Turbidity corresponding to ALSTD.
ALSAM = Size of the sample aliquot, mL.
TBSAM = Turbidity corresponding to ALSAM.

Note: If the turbidity of the Reagent Blank Solution “TBBNK” is 0.40 or higher, for greater accuracy, the value of “TBBNK” shall be subtracted from “TBSAM” and from “TBSTD” before calculating S above.