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## Test Procedure for

# DETERMINING SULFATE CONTENT IN SOILS — COLORIMETRIC METHOD



## TxDOT Designation: Tex-145-E

**Effective Date: February 2005**

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### 1. SCOPE

- 1.1 This method determines the soluble sulfate content of soil by using turbidimetric techniques.
  - 1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.
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### 2. DEFINITIONS

- 2.1 *Turbidimetric Technique*—The process of measuring the cloudiness of a liquid and translating it into concentration. A reagent (sulfate test tablet) is combined with a sample, which causes cloudiness or turbidity in the sample.
  - 2.2 *PPM*—Parts per million; measure of concentration.
  - 2.3 *Filtrate*—(Soil/water) material that has passed through a filter.
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### 3. APPARATUS

- 3.1 *Colorimeter and the following accessories:*
    - 10 mL glass sample vial
    - Sulfate test tablets
    - White plastic rod.
  - 3.2 *9 V battery.*
  - 3.3 *Auger sampler.*
  - 3.4 *Core sampler, 2 in. diameter × 4 ft. long.*
  - 3.5 *Balance, Class G2 in accordance with Tex-901-K, minimum capacity of 3500 g.*
  - 3.6 *Sieves, U.S. Standard No. 4 (4.75 mm) and No. 40 (425 μm).*
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- 3.7            *Mortar and pestle.*
  - 3.8            *Crusher.*
  - 3.9            *Oven, capable of maintaining a temperature of  $60 \pm 5^{\circ}\text{C}$  ( $140 \pm 9^{\circ}\text{F}$ ).*
  - 3.10          *Beakers (glass or plastic), 250 mL.*
  - 3.11          *Beakers (glass or plastic), 600 mL.*
  - 3.12          *Graduated cylinder TD (ex) (glass or plastic), 10 mL.*
  - 3.13          *Graduated cylinder (glass or plastic), 100 mL.*
  - 3.14          *Wide-mouth round high-density poly ethylene (HDPE) bottles, 8 oz. (250 mL).*
  - 3.15          *Wide-mouth round HDPE bottles, 16 oz. (500 mL).*
  - 3.16          *Funnel (glass or plastic), 80 mm diameter with short wide stems.*
  - 3.17          *Filter paper (fine porosity), 15 cm diameter.*
  - 3.18          *2 mL disposable pipettes.*
  - 3.19          *Wash bottle, 16 oz. (500 mL), for distilled or deionized water.*
  - 3.20          *Dessicator.*
  - 3.21          *Latex gloves.*
  - 3.22          *Tweezers.*
  - 3.23          *Pans, brush, and spatula.*
  - 3.24          *Sample splitter.*
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#### **4. MATERIAL**

- 4.1            *Distilled or deionized water.*
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### **PART I—FIELD DETERMINATION OF SULFATES**

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#### **5. SCOPE**

- 5.1            The following procedure describes preparing soil samples for determining sulfate content in the field.

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## 6. PROCEDURES

### 6.1 *Preparing Sample:*

- 6.1.1 Sample soil within the depth of proposed stabilization with a hand auger at the sampling frequency required by the guide schedule.
- 6.1.2 Obtain a 300 g representative sample.
- 6.1.3 Air-dry the sample to constant weight.
- 6.1.4 Pulverize the 300 g to pass the No. 40 (425  $\mu$ m) sieve.
- 6.1.5 Split the sample and obtain 10 g of representative sample. Do replicates for more accuracy. This means split the material to obtain three samples of 20 g each and run on each sample the colorimetric test.
- 6.1.6 Weigh 10 g to the nearest 0.1 g.

### 6.2 *Measuring Sulfate Content:*

- 6.2.1 Place the 10 g soil sample in an 8 oz. (250 mL) HDPE bottle.
- 6.2.2 Add 200 mL of distilled or deionized water. This provides an initial dilution ratio of 1:20.
- 6.2.3 Vigorously shake the bottle of soil/water solution by hand for 1 min. Wait for 12 hr.
- 6.2.4 After a minimum of 12 hr. has elapsed, place one filter paper along the inside of the funnel and place the funnel into an 8 oz. (250 mL) beaker to capture the filtrate.
- 6.2.5 Shake the bottle of soil/water solution again for 1 min. and pour the solution into the funnel. Allow all the solution to filter and drain into the beaker.
- 6.2.6 Using a clean, 2 mL disposable pipette, fill the 10 mL graduated cylinder with the filtrate. Place the 10 mL filtrate into a sample vial. Wipe the outside of the vial clean with a Kimwipe® or equivalent delicate task wipe to remove dirt, fingerprints, or anything else that will obstruct a light beam from passing through the glass and filtrate.  
**Note 1**—Insure the test vial is clean and free from any residue from previous testing.
- 6.2.7 Switch the colorimeter unit to “ON.”
- 6.2.8 Press the *MODE* key until “SUL” method is displayed.
- 6.2.9 Place the sample vial into the sample chamber with the  $\Delta$  vial mark aligned with the  $\Delta$  housing mark.
- 6.2.10 Press the *ZERO/TEST* key. The method symbol flashes for approximately 3 sec. and confirms zero calibration.

- 6.2.11 After zero calibration, remove the vial from the sample chamber.
- 6.2.12 Add one sulfate test tablet to the vial without touching the tablet with hands and crush immediately with the white plastic rod provided.  
*CAUTION:* Use latex gloves and tweezers when handling the sulfate test tablet. The tablets are toxic and care should be taken to avoid contact with the skin, eye, or ingestion.
- 6.2.13 Tamp the sulfate test tablet with the rod in a random motion until it is completely dissolved and dispersed throughout the sample vial.
- 6.2.14 Cap the vial, wipe the outside of the glass clean again, and immediately place the sample in the chamber with the  $\Delta$  marks aligned.
- 6.2.15 Press the *ZERO/TEST* key. The method symbol flashes for approximately 3 seconds and the result will appear in the display.
- 6.2.16 Take a minimum of three readings per sample and average the results.  
**Note 2**—If an error message is displayed, go to Section 6.2.19.
- 6.2.17 Multiply the average result times the dilution.  
  
(Example:  $100 \text{ (average reading)} \times 20 \text{ (1:20 dilution)} = 2000 \text{ ppm.}$ )  
  
**Note 3**—This test will only read concentrations from 5-200 mg/l (100-4000 ppm) for a 1:20 dilution.
- 6.2.18 Report the concentration in parts per million (ppm).
- 6.2.19 If the following error messages are displayed, report the following:
- 6.2.19.1 “– *Err message*: The result is below the measuring range limit.”  
■ Report the sulfate concentration as <100 ppm.
- 6.2.19.2 “÷ *Err message*: The result exceeds the measuring range.”  
■ This requires diluting the sample with more deionized water to bring the reading within the measurable range, as stated below:
- Measure out 5 mL of the filtrate into a 10 mL graduated cylinder and add exactly 5 mL of distilled or deionized water using a clean pipette.
  - The new solution is now at 1:40 dilution ratio, which corresponds to a maximum reading range of 8,000 ppm. Place the new diluted solution in a sample vial and repeat Sections 6.2.8–6.2.18.
  - If a “÷ *Err message*” is displayed again, this means the material has more than 8,000 ppm sulfate content. Report the sulfate concentration as > 8,000ppm.

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## 7. TEST REPORT

- 7.1 Report average sulfate concentration in ppm to the nearest whole number.

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## PART II—LABORATORY TEST

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## 8. SCOPE

- 8.1 The following procedure describes preparing soil samples for determining sulfate content in the laboratory.

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## 9. PROCEDURES

### 9.1 *Preparing Sample:*

- 9.1.1 Dry the entire Shelby tube or bagged sample in a  $60 \pm 5^{\circ}\text{C}$  ( $140 \pm 9^{\circ}\text{F}$ ) oven and allow to cool to  $25 \pm 3^{\circ}\text{C}$  ( $77 \pm 5^{\circ}\text{F}$ ) in a desiccator to constant weight.
- 9.1.2 After the sample is dried, crush, grind, and split to obtain approximately a 1500 g (3.3 lb.) representative sample to pass the 4.75 mm (No. 4) sieve.
- 9.1.3 Pulverize the 1500g sample to pass the 425  $\mu\text{m}$  (No. 40) sieve.
- 9.1.4 Split the sample to obtain three 40 g representative sample and weigh to the nearest 0.1 g.

### 9.2 *Measuring Sulfate Content:*

- 9.2.1 Weigh 20 g to the nearest 0.1 g of oven-dried soil and place the soil in a 16 oz. (500 mL) HDPE bottle.
- 9.2.2 Add 400 mL of distilled or deionized water. This provides an initial dilution ratio of 1:20.
- 9.2.3 Vigorously shake the bottle of soil/water solution by hand for 1 min. Wait for 12 hr.
- 9.2.4 After a minimum of 12 hr. has elapsed, place one filter paper along the inside of the funnel and place the funnel into a 600 mL beaker to capture the filtrate.
- 9.2.5 Agitate the sample again for 1 min. and pour the soil/water solution into the funnel. Allow all the solution to filter and drain into the beaker. Ensure the funnel is covered during the filtration process to ensure soil and other substance does not contaminate the solution.
- 9.2.6 Using a clean, uncontaminated pipette, measure out 10 mL of the filtrate into a 10 mL graduated cylinder. Transfer the 10 mL filtrate from cylinder into a sample vial. Wipe the outside of the vial clean with a Kimwipe® or task wipe to remove dirt, fingerprints, or anything else that will obstruct a light beam from passing through the glass and filtrate.

**Note 4**—Insure the test vial is clean and free from any residue from previous testing.

- 9.2.7 Switch the colorimeter unit to “ON.”
- 9.2.8 Press the *MODE* key until “SUL” method is displayed.
- 9.2.9 Place the sample vial into the sample chamber with the  $\Delta$  vial mark aligned with the  $\Delta$  housing mark.
- 9.2.10 Press the *ZERO/TEST* key. The method symbol flashes for approximately 3 sec. and confirms zero calibration.
- 9.2.11 After zero calibration, remove the vial from the sample chamber.
- 9.2.12 Add one sulfate test tablet to the vial without touching the tablet with hands and crush immediately with the white plastic rod provided.
- CAUTION:* Use latex gloves and tweezers when handling the sulfate test tablet. The tablets are toxic and care should be taken to avoid contact with the skin, eye, or ingestion.
- 9.2.13 Tamp the sulfate test tablet with the rod in a random motion until it is completely dissolved and dispersed throughout the sample vial.
- 9.2.14 Cap the vial, wipe the outside of the glass clean again, and immediately place the sample in the chamber with the  $\Delta$  marks aligned.
- 9.2.15 Press the *ZERO/TEST* key. The method symbol flashes for approximately 3 sec. and the result will appear in the display. Take a minimum of three readings per sample and average the results.
- 9.2.16 If an error message is displayed, go to Section 9.2.19.
- 9.2.17 Multiply the average result times the dilution.
- (Example:  $100 \text{ (average reading)} \times 20 \text{ (1:20 dilution)} = 2000 \text{ ppm.}$ )
- Note 5**—This test will only read concentrations from 5-200 mg/l (100-4000 ppm)
- 9.2.18 Report the concentration in parts per million (ppm).
- 9.2.19 If the following error messages are displayed, take the corresponding actions:
- 9.2.19.1 “- *Err message:* The result is below the measuring range limit.”
- Report the sulfate concentration as < 100 ppm.
- 9.2.19.2 “÷ *Err message:* The result exceeds the measuring range.”
- Dilute the sample with more deionized water to bring the reading within the measuring range, as stated below:
    - Measure out 5 mL of the filtrate into a 10 mL graduated cylinder and add exactly 5 mL of distilled or deionized water using a clean pipette.

- The new solution is now at 1:40 dilution ratio, which corresponds to a maximum reading range of 8,000 ppm. Place the new diluted solution in a sample vial and repeat Sections 9.2.8–9.2.18.
- If “÷ Err message” is displayed again, this means the material has more than 8,000 ppm sulfate content and additional dilutions will be required until an absolute number is obtained. Repeat Sections 9.2.8 through 9.2.15 and Sections 9.2.17 and 9.2.18.

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## **10. TEST REPORT**

10.1 Report average sulfate concentration in ppm to the nearest whole number.