



**INDIANA DEPARTMENT OF TRANSPORTATION
DIVISION OF MATERIALS AND TESTS**

**DETERMINING SULFATE CONTENT IN SOILS
BY COLORIMETRIC METHOD
ITM No. 510-25**

1.0 SCOPE.

- 1.1** This test method measures the soluble sulfate concentration in subgrade soils by using turbidimetric methods.
- 1.2** This ITM may involve chemical handling during operations, and may not address all of the safety problems associated with the use of the test method. The user of the ITM is responsible for establishing appropriate safety and health practices and determining the applicability of regulatory limitations prior to use.

Latex gloves are required to be used when handling the sulfate reagent. The reagent is toxic, and care is required to be taken to avoid ingestion or contact with the skin or eyes.

2.0 REFERENCES.

2.1 AASHTO Standards.

R 58 Dry Preparation of Disturbed Soil and Soil-Aggregate Samples for Test
M 231 Weighing Devices Used in the Testing of Materials

3.0 TERMINOLOGY.

- 3.1 Terms and Abbreviations.** Definitions for terms and abbreviations will be in accordance with the Department's Standard Specifications, Section 101 and the following:
- 3.2 Filtrate.** Soil/water slurry that has passed through a filter
- 3.3 Parts Per Million (ppm).** A measure of concentration
- 3.4 Turbidimetry.** The measure of the loss in intensity of a light beam as the beam passes through a solution. The terms "colorimetry" and "spectrophotometry" are also used.

- 4.0 SIGNIFICANCE AND USE.** This ITM shall be used to determine the water-soluble sulfate ion content in soil. The results are used to determine if chemical modification or stabilization of the soil is appropriate.

5.0 APPARATUS.

- 5.1 Balance, Class G 2, in accordance with AASHTO M 231
- 5.2 Beakers, 600 ml
- 5.3 Buchner funnel
- 5.4 Colorimeter and accessories
- 5.5 Dry, lint-free tissue
- 5.6 Filter paper, fine porosity, 2-5 micron
- 5.7 Graduated Cylinder, 25 ml
- 5.8 Latex gloves
- 5.9 Mortar and pestle
- 5.10 Oven, capable of maintaining a temperature of $140 \pm 9^{\circ}\text{F}$
- 5.11 Pans, brush, and spatula
- 5.12 Pipettes, 2 ml disposable
- 5.13 Pulverizing apparatus
- 5.14 Sample cell (vial), glass, 10 ml
- 5.15 Side arm flask
- 5.16 Sieves, No.4 and No.40, conforming to the requirements of AASHTO M 92
- 5.17 Sulfate reagent (Powdered pillow)
- 5.18 Vacuum
- 5.19 Wash bottle, 500ml, for distilled or de-ionized water
- 5.20 Wide-mouth rectangular or round high-density polyethylene (HDPE) bottles, 500ml

6.0 MATERIAL.

- 6.1 Distilled or de-ionized water

7.0 SAMPLE PREPARATION.

- 7.1 The sample may be air dried, or oven dried at a temperature not exceeding 140°F.
- 7.2 After the sample is dried, the sample is crushed, ground, and split to obtain an approximately 500g sample that is passing pass the No.4 sieve.
- 7.3 The sample is pulverized to obtain a sample that is passing the No. 40 sieve.
- 7.4 The sample is split to obtain three approximately 20g samples and each sample is weighed to the nearest 0.1g.

8.0 PROCEDURE.

8.1 Soil Slurry.

- 8.1.1 Place one sample of soil into the HDPE bottle and record the weight of the soil slurry to the nearest 0.1 g.
- 8.1.2 Add approximately 400 ml of distilled or de-ionized water to the sample. The ratio of the water to soil should be approximately 20:1.
- 8.1.3 Cap the bottle and shake the soil slurry for 1 minute.
- 8.1.4 Soak the sample for a minimum of 12 hours.
- 8.1.5 Label the samples A, B, and C. Each sample will have two vials. One for calibration and one for testing. These should be labeled A₁, A₂.

8.2 Filtration.

- 8.2.1 Place a filter paper into the Buchner funnel and slightly moisten the filter paper with distilled or de-ionized water.
- 8.2.2 Place the stem of the funnel through the rubber stopper and affix the stopper/funnel assembly onto the top opening of the side arm flask.
- 8.2.3 Shake the soil slurry sample A for 1 minute before the filtration.
- 8.2.4 Start the vacuum, pour the soil slurry sample into the funnel, and collect the filtrate in the side arm flask. Do not rinse material into the Buchner funnel or add any water. A filtration of 100-150 ml of the soil slurry is adequate. The side of the flask and the Buchner funnel must be cleaned with distilled or de-ionized water before reuse.

- 8.2.5 Repeat the filtration procedure for ~~the remaining two~~ soil slurry samples for all samples used for the test, collecting the filtrate in separate flasks.

8.3 Measurement.

- 8.3.1 Using a clean, uncontaminated disposable pipette, measure 10ml of filtrate ~~A~~ and transfer the filtrate into the vial. This vial will be considered the "blank" sample that will be used for calibration. ~~The samples used as the "blanks" should be A₁, B₁, and C₁. Always start with A₁.~~
- 8.3.2 Cap the vial and wipe the outside of the vial to remove anything that will obstruct a light beam from passing through the vial and filtrate.
- 8.3.3 Press the POWER key to turn the meter on.
- 8.3.4 Place ~~A₁, the blank sample vial~~ into the sample chamber/holder aligned with the Δ housing mark.
- 8.3.5 Cover ~~A₁, the blank sample~~ with the instrument cap.
- 8.3.6 Press the ZERO/SCROLL key. The display will show "---" then "0" which will confirm the zero calibration.
- 8.3.7 Add the sulfate reagent powder (SulfVer 4 Powder Pillow) to the second sample vial with 10 ml filtrate. ~~The second sample vial will be A₂.~~ After adding the reagent, a white turbidity may develop if there is sulfate present in the sample.
- 8.3.8 Cap ~~A₂ the vial~~ and invert the vial several times to mix the sample.
- 8.3.9 Wait 5 minutes for the reagent to ~~completely~~ dissolve ~~completely~~ and disperse throughout ~~A₂ the sample~~.
- 8.3.10 Wipe the outside of ~~A₂ the vial~~ and place ~~A₂ the sample~~ into the sample chamber/holder aligned with the Δ housing mark.
- 8.3.11 Cover the sample cell with the instrument cover.
- 8.3.12 Press the READ/ENTER key. The display will show "---" followed by the results in mg/L sulfate.
- 8.3.13 ~~Take a minimum of three readings for each sample and average the results.~~ Repeat measurement 8.3 for all samples. Average the results of ~~A₂, B₂, and C₂.~~

9.0 CALCULATIONS.

- 9.1 Calculate the sulfate content in ppm by multiplying the average of the three Colorimeter tests by the Dilution Ratio from Table 1.

| COLORIMETER METHOD | | | |
|-------------------------------|-----------------------|----------------|----------------------------|
| Filtrate/De-ionized Water, ml | Soil/De-Ionized Water | Dilution Ratio | Sulfate Content Range, ppm |
| 10/0 | 1:20 | 20 | 40-1400 |
| 10/10 | 1:40 | 40 | 80-2800 |
| 10/20 | 1:60 | 60 | 120-4200 |
| 10/30 | 1:80 | 80 | 160-5600 |
| 10/40 | 1:100 | 100 | 200-7000 |
| 10/50 | 1:120 | 120 | 240-8400 |
| 10/60 | 1:140 | 140 | 280-9800 |
| 10/70 | 1:160 | 160 | 320-11200 |
| 10/80 | 1:180 | 180 | 360-12600 |
| 10/90 | 1:200 | 200 | 400-14000 |

Table 1. DILUTION RATIO FOR SULFATE TESTING

Example:

Average of three Colorimeter results = 50 mg/L

Dilution Ratio = 20 (From Dilution Ratio Table)

Sulfate Content (ppm) = 50 x 20 = 1000 ppm

- 9.2 The sulfate test at this dilution ratio will only read concentrations of 2-70 mg/l (40-1400 ppm). To obtain higher sulfate concentration use Table 1. If the concentration is higher than 1400 ppm, there will be a blinking number on the colorimeter screen indicating the result is out of the measuring range limit. When this occurs, the sample is diluted with more de-ionized water as follows:

9.2.1 Measure 5 ml of the filtrate into a 10 ml graduated cylinder and add 5ml of distilled or de-ionized water to the sample. The new solution is now at a 1:40 dilution ratio, which corresponds to a maximum reading range of 2800 ppm as indicated in Table 1.

9.2.2 Place the new diluted solution in a vial, repeat Sections 8.3.7 through 8.3.12 for the required measuring range, and determine the sulfate content.

- 10.0 **REPORT.** Report the concentration of sulfate in parts per million (ppm)