OHD L-49
METHOD OF TEST FOR
DETERMINING SOLUBLE SULFATE CONTENT IN SOIL

1. **SCOPE.** This method measures the soluble sulfate content of soil by using turbidimetric methods. The results are used to determine whether chemical stabilization (with lime, fly ash, cement kiln dust, etc.) of the tested soil is appropriate. The acceptable range of sulfate contents and modified construction methods are not covered here.

   A. Portions of this method are also applicable to measuring soluble sulfates in water used in construction. Since this method is sensitive to the pH of the test solution, this method is not applicable to measuring soluble sulfates in stabilized soil, or in chemical stabilizers.

   B. The methods and apparatus described here will measure soluble sulfate contents between 200 and 8000 ppm (0.02 and 0.08 %), which is appropriate for the purposes listed above. The methods may be modified to extend the range of the test.

   C. Soluble sulfates are frequently present as gypsum. Soils containing gypsum typically yield erratic moisture contents, and may require special treatment for routine lab testing.

2. **DEFINITIONS:**

   A. Turbidimetry - the measurement of the loss in intensity of a light beam as it passes through a solution. The terms “colorimetry” and “spectrophotometry” are also used.

   B. ppm - parts per million; a measure of concentration**.**

   C. Supernatant - The liquid remaining on top after denser materials have settled.

   D. Filtrate - soil/water solution that has passed through a filter.

3. **APPARATUS.**

   A. Colorimeter and the following accessories
      1. 10-ml glass sample vial
      2. Sulfate test tablets
      3. White plastic tamping rod

   NOTE: The model used by the Geotechnical Lab is the Orion AQUAFast II colorimeter, manufactured by the Thermo Electron Corporation.

   B. Balance or scale capable of weighing the sample to 0.01 g
   C. Wide-mouth round High-Density Poly Ethylene (HDPE) bottles, 250-ml
   D. Funnel (glass or plastic), 80-mm dia. with short wide stems
   E. Filter paper (fine porosity), 15-cm dia.
   F. Pipette, 10-ml (glass or plastic)
   G. Latex gloves
   H. Dry, lint-free tissues (Kimwipes)

I. Miscellaneous - Laboratory glassware (beakers, graduated cylinders, stirring rods), spoon or small scoop
J. Distilled or deionized water
K. Sodium sulfate (Na_2SO_4), about 0.05 g per test

4. SAMPLE. A test specimen will typically be obtained from inplace samples, borrow pit samples, or pedological samples.

NOTE: Sulfate-rich soils may be very localized, and detecting them may require more sampling than is usually done.

A. A specimen shall be obtained from the entire air-dried sample (including coarse material) by splitting or quartering. Specimen size shall be in accordance with the table below, or one-half of the submitted sample, whichever is smaller. Care should be taken to insure that the gradation of the test specimen is representative of the entire sample.

Diameter of Largest Particle, mm (in.)	Minimum Sample Mass, kg
2.00 (#10)	0.3
9.5 ($^3/_8$)	1
25.0 (1)	2
50 (2)	4
75 (3)	5

B. The test specimen shall be crushed to pass a #10-sieve. All practical methods may be used to break or crush particles to the required size. Hard particles retained on the #10-sieve after crushing may be discarded.

C. The material passing the #10-sieve shall be split or quartered to obtain a specimen of about 30 g. The specimen shall be oven-dried at 230 ± 9°F (110 ± 9°C) to a constant mass.

5. PROCEDURE.

A. Soil slurry. Weigh approximately 5 g of soil into the HDPE bottle, record the actual mass of soil to 0.01 g. Add approximately 200 g of distilled or deionized water, record the actual mass of water added to 0.01 g. The ratio of water to soil should be approximately 40:1. Cap the bottle and shake, either:
   1. by hand, for 15 seconds every 5 minutes, for 15 minutes, or
   2. with a mechanical shaker, for 15 minutes.

The slurry shall be left to soak for at least 16 hours. The slurry shall be shaken immediately before filtration.

B. Filtration. Place a filter paper cone inside the funnel and place the small end of the funnel into a 250-ml beaker to capture the filtrate. Wet the cone with distilled or deionized water, and allow the excess to drain. Pour out any water in the beaker.

NOTE: Form the filter paper cone by folding a filter paper disk into a quarter, and opening so that one side has three thicknesses and the other has one.

For each test specimen, pour the supernatant into the funnel. Allow the liquid to filter and drain into the beaker. Fill the filter paper cone no more than ½ full at any time. Do not rinse material into the funnel, or otherwise add any water. It is not necessary to filter all of the supernatant; 30 ml is adequate.

NOTE: It is desirable for the filtrate to be essentially clear, but it frequently may be cloudy or colored. A clearer filtrate can be obtained by adding 2-3 drops of concentrated nitric acid to the liquid before filtering. Addition of acid to the filtrate will also clear it, but less effectively.

If the filtrate cannot be tested the same day, cover the beaker to minimize evaporation.

C. Measurement.

1. Using a clean, uncontaminated pipette, fill a sample vial to the 10-ml mark with the filtrate solution. Cap, and wipe the outside of the vial clean with a Kimwipe to remove dirt, fingerprints, or anything else that will obstruct a light beam from passing through the glass and filtrate. Check for bubbles in the area below the fill mark, tap gently to free bubbles.
2. Switch the colorimeter unit to "ON".
3. Press the MODE key until the "SUL" method is displayed.
4. Place the sample vial into the sample chamber with the ▽ mark aligned with the △ housing mark.
5. Press the "ZERO/TEST" key. The method symbol flashes for approximately three seconds and confirms zero calibration.
6. After zero calibration, remove the vial from the sample chamber.
7. Add one sulfate test tablet to the vial and crush immediately with the white plastic rod provided. Take care that the entire tablet is used.

   CAUTION: Use latex gloves or tweezers when handling the sulfate test tablet. The tablets are toxic and care should be taken to avoid ingestion or contact with skin or eyes.

8. Tamp the sulfate tablet with the rod in a random motion until it is completely dissolved and dispersed throughout the sample vial.
9. Cap the vial, wipe the outside of the glass clean again, and immediately place the sample in the chamber with the marks aligned.
10. Press the "ZERO/TEST" key. The method symbol flashes for approximately three seconds and the result will appear in the display.
11. Take a minimum of three readings per specimen and average the results.
12. If the following error messages are displayed, take the corresponding actions:

- Err	The result is below the measuring range limit; record the colorimeter reading as <5 ppm.
+Err	The result is above the measuring range limit; record the colorimeter reading as >200 ppm.

D. Cleanup and disposal of waste. After each test, rinse all used glassware with distilled or deionized water to minimize contamination between samples.

The used chemicals and samples are toxic, and should not be poured down a drain, or in trash. The sample vial containing the filtrate and dissolved chemicals should be emptied into a beaker or other container, and about 0.05 g of sodium sulfate per test added to the waste material to consume the unreacted chemicals. The resulting mixture is safe to pour down the drain.

6. CALCULATIONS.

A. Dilution ratio:

$$D = \frac{W_w}{W_s}$$

where	D	dilution ratio of soil slurry
	W_w	mass of water added to slurry
	W_s	mass of oven-dried soil in slurry

B. Sulfate concentration in soil:

$$C = RD$$

where	C	sulfate concentration, ppm
	R	colorimeter reading, ppm
	D	dilution ratio

NOTE: If the final colorimeter reading was out-of-range, use R=5 or 200, as appropriate, calculate the concentration, and report the fractional concentration as less than or greater than the calculated value as appropriate. For example, if the colorimeter reading was <5 ppm, and the dilution ratio is 40, the sulfate concentration should be reported as <200 ppm.

APPENDIX A. EXTENSION OF THE METHOD TO HIGHER CONCENTRATIONS

The standard method will measure soil sulfate concentrations between 200 and 8000 ppm, with a precision of no more than 40 ppm. If the concentration is higher than 8000 ppm, most practitioners do not recommend stabilization with calcium-based stabilizers. Therefore, it is usually adequate to report that the concentration is >8000 ppm. If an actual number is desired, the method above can be modified as follows.

A-1. **PROCEDURE**.

A. Prepare and test the sample as above, through section "C" of the procedure.

B. If the colorimeter reading is below or within the measuring range limit, proceed as above; no further testing is required. If the colorimeter reading is above the measuring range limit, proceed as follows:

1. Pipette 10 ml of filtrate into a clean beaker.
2. Add 40 ml of distilled or deionized water to the beaker, stir to mix.
3. Measure the sulfate content of the diluted solution as in "C" above.
4. If the colorimeter reading is still out of range, repeat steps 1-3, using the diluted solution instead of the filtrate, until the reading is within range.

A-2. **CALCULATIONS**.

1. Dilution ratio:

$$D_f = D_i \frac{\left(V_f + V_w\right)}{V_f}$$

where D_f dilution ratio of diluted filtrate
D_i initial dilution ratio
V_f volume of undiluted filtrate, ml
V_w volume of water added, ml

2. If more than one dilution is required, repeat this calculation for each successive dilution.

3. Calculate the soil sulfate concentration as in the standard method, using the final dilution ratio.