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METHOD OF TESTING SOILS AND WATERS FOR SULFATE CONTENT

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Section H of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

A. SCOPE

This method describes test procedures for determination of sulfate content of soils and waters. These results are used in determining the corrosive nature of the environment for concrete structures, as well as for other purposes.

In this test method the sulfate ion is precipitated with barium chloride, in an acidic medium, to barium sulfate crystals of uniform size. The barium sulfate present in suspension is determined by a measurement of its turbidity and comparison with a known standardization curve.

Color or suspended materials in large quantities may interfere with this test method. If the amount of color or suspended materials are small in comparison to the sulfate concentration, interference can be corrected by running blanks from which the barium chloride is missing. Large amounts of organic materials and silica in excess of 500 mg/L may inhibit satisfactory barium sulfate precipitation.

B. APPARATUS

1. A Photometer shall be operative at 420 mm, or nephelometer. The Hach

turbidimeter, Model 2100A, has been found to be satisfactory for this method.

2. A magnetic stirrer, with teflon covered magnetic stirring bars, is required.
3. A stopwatch shall be needed if the magnetic stirrer is not equipped with an accurate timer.
4. A measuring spoon shall have a capacity of 0.2 to 0.3 mL.

C. REAGENTS

1. Conditioning Reagent.

In 300 mL of distilled or deionized water, dissolve 30 mL of concentrated HCl, 100 mL of 95 % ethyl or isopropyl alcohol and 75 g of sodium chloride. Add 50 mL of glycerol and mix the contents well.

2. Barium Chloride:

BaCl₂ · 2H₂O, crystals, passing the 850- μ m sieve and retained on the 600- μ m sieve

3. Standard Sulfate Solution:

1.00 mL = 100 μg SO_4 . Dissolve 147.9 mg of anhydrous Na_2SO_4 in distilled or deionized water and dilute to 1000 mL.

D. PROCEDURE

1. Calibration Curve:

Prepare sulfate standards from 0 to 3.0 mg of SO_4 . In three 100-mL volumetric flasks, pipet 10.00, 20.00, and 30.00 mL of standard sulfate solution (Section C.3) and dilute to 100 mL with distilled water, mix thoroughly. These dilutions will give standards from 0 to 3.0 mg of SO_4 in 1.0 mg increments.

Treat each sulfate standard in the following manner to obtain a calibration curve. Transfer standard to 250-mL Erlenmeyer flask, add 5 mL of conditioning reagent, swirl, and pour some of the solution into the absorption cell to take a blank reading. Pour the solution from the absorption cell back into the 250-mL Erlenmeyer flask and add a teflon covered stirring bar and place on magnetic stirrer. Adjust the stirrer to the maximum speed at which the solution can be stirred without splashing. Add a measuring spoonful of barium chloride and start timing. Stir for exactly 1 min.

Immediately after the stirring period, pour some of the solution into the absorption cell of the photometer and measure the maximum turbidity reading. Maximum turbidity usually occurs within 2 min. Record the highest turbidity obtained during the 2-min period. Check reliability of the calibration curve by running a standard for every three or four unknown samples.

2. Measurement of Sulfates in Samples

- a. For water samples, proceed directly to "c".
- b. For soil samples, proceed as follows:

- (1) Weigh 100 g of soil and place it in a 500-mL Erlenmeyer flask.
- (2) Add 300 mL of water. Place a stopper on the flask and shake the flask vigorously for 15 min. Centrifuge the sample, filter the sample, or let the sample settle overnight.
- (3) Pipet approximately 20 mL of the water layer into a 100-mL volumetric flask. Dilute this sample to 100 mL, and proceed as below for water samples.
 - c. Color or turbidity blank: Run a blank for each sample using same procedure as for the standards, but omitting the barium chloride.
 - d. Measurement of barium chloride turbidity of a sample: Measure 100 mL of sample into a 250-mL Erlenmeyer flask. Follow exactly precipitation and measurement procedure used with standards. Correct reading for original color and turbidity by subtracting blank reading from the reading obtained for the sample. If corrected readings exceed that obtained for the 3.0 mg of SO_4 standard, repeat the color or turbidity blank and measurement of barium chloride turbidity of the sample using a suitably smaller aliquot of the sample diluted to 100 mL.

NOTES

1. Large amounts of colored or suspended matter may interfere with the turbidity readings which may give incorrect sulfate readings.
2. Check calibration by running a standard with each group of three or four samples.
3. The magnetic stirrer should be at same speed for all standards and samples.

E. CALCULATION

1. For water samples:

$\text{mg/L of SO}_4 = (\text{mg of SO}_4 \times 1,000) / \text{mL of the sample.}$

2. For soil samples:

$\text{mg/kg of SO}_4 = (\text{mg of SO}_4 \times 3000) / \text{mL of the sample}$

F. REPORTING RESULTS

Record all procedures and data obtained in a bound numbered laboratory book, and record on appropriate forms as required.

G. ALTERNATE PROCEDURES

Other methods which may be used are "Standard Methods", 16th Edition, Part 426A and B, "Gravimetric Method"; and the ASTM Designation: D 516, "Sulfate Ion in Water".

H. SAFETY AND HEALTH

This method may involve hazardous materials, operations and equipment. This method does not purport to address all the safety problems associated with its use. It is the responsibility of whomever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Prior to handling, testing or disposing of any of waste materials, testers are required to read: Part A (Section 5.0), Part B (Sections: 5.0, 6.0, 10.0 and 12.0) and Part C (Section 1.0) of Caltrans Laboratory Safety Manual. These sections pertain to requirements for general safety principles, standard operating procedures, protective apparel, disposal of materials and how to handle spills, accidents, emergencies, etc. Users of this method do so at their own risk.

REFERENCES:

"Standard Methods for the Examination of Water and Wastewater", 16th Edition, 1985, APHA-AWWA-WPCF.

U.S. Environmental Protection Agency Manual, "Methods for Chemical Analysis of Water and Waste", 1979 Edition.

"1983 Annual ASTM Standards", Volume 11.01, "Water (1)". ASTM Designation: D 516

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