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METHOD OF TESTING SOILS, CONCRETE PATCHING MATERIALS AND WATERS FOR SULFATE CONTENT

A. SCOPE

This method describes test procedures for determination of the soluble sulfate content of soils, concrete patching materials and waters. Concrete patching materials can be rapid setting concrete, mortar, bonding agent or material, fast setting grout, filler material or shotcrete. These results are used in determining the corrosive nature of the environment for concrete structures, as well as for other purposes. This test method is divided into the following parts:

1. Sulfate content of waters
2. Sulfate content of soils
3. Sulfate content of concrete patching materials

B. REFERENCES

California Test 201 — Soil and Aggregate Sample Preparation
ASTM C 109 — Compressive Strength of Hydraulic Cement Mortars
ASTM E 11 — Woven Wire Test Sieve Cloth and Test Sieves

C. PROCEDURE

PART 1. SULFATE CONTENT OF WATERS

1A. SCOPE

This method is used to determine the sulfate content of waters.

1B. REAGENTS AND MATERIALS

Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

1. Preparing Eluent – Dissolve 21.0 g of Sodium Bicarbonate, NaHCO_3 in deionized water and dilute to 500 mL volume. Dissolve 26.5 g of Sodium Carbonate, Na_2CO_3 in deionized water and dilute to 500 mL volume. Transfer each solution to a separate polypropylene bottle. Prepare eluent solution by adding 2 mL of NaHCO_3 , 7 mL of Na_2CO_3 solutions, and dilute to 1000 mL with deionized water.
2. Standard Sulfate solution, 1000 ppm: Dissolve 1.8145 g of K_2SO_4 in deionized water and dilute to 1000 mL.
3. Standard Chloride solution, 1000 ppm: Dissolve 1.6485 g of NaCl (dried at 140°C) in deionized water and dilute to 1000 mL.

4. Volumetric/automatic pipettes.
5. Volumetric flasks: 100 – 1000 mL
6. Erlenmeyer flasks: 500 mL and stopper #10
7. Poly vial: 5 mL vials and caps for use in autosampler
8. Acrodisc CR 0.5 - 1.0 inch syringe filter with 0.45 micron PTFE membrane
9. Disposable 10 mL syringes

1C. ANALYTICAL INSTRUMENT AND EQUIPMENT

Ion Chromatograph (IC) – The Metrohm model 761 Compact has been found to be satisfactory for this method. Any comparable instrument can be used as well.

1D. TEST PROCEDURE

1. Calibration Curve:

Prepare a mixture of sulfate and chloride standards at concentrations of 5, 10, 20, 50, 100, and 200 ppm. In six 100 mL volumetric flasks, pipette, 0.5, 1, 2, 5, 10, and 20 mL each sulfate and chloride solution at 1000 ppm (Part 1, Section B.2 and 3), dilute to 100 mL with deionized water. Transfer each standard into a polypropylene bottle. Pipette 5 mL of each standard into a sample vial to run on the IC.

NOTE: The IC is set up to analyze sulfate and chloride from one run.

2. Blank: Pipette 5 mL of deionized water into a sample vial and cap. Blank is run at the beginning of every run to determine possible contamination.
3. Water samples: Pipette 5 mL into a sample vial and cap. Samples should be free of particles; otherwise, syringe filters should be used to filter each sample.
4. Prepare a program on the IC by running a blank, calibration curve standards, check standard, samples, and shutdown mode at the end of the run. Samples with high concentrations of sulfates require dilution.

1E. CALCULATION

$$C_a = (S_a - S_0)/m$$

Where:

C_a = is the amount of the analyte in the sample

S_a = signal response of analyte in the sample

S_0 = intercept of the calibration line

m = slope of the calibration line

PART 2. SULFATE CONTENT OF SOILS

2A. SCOPE

This method describes the test procedure for determination of the water-soluble sulfate content of soils.

2B. REAGENTS AND MATERIALS

Refer to Part 1, Section B for these items.

2C. EQUIPMENT

Refer to Part 1, Section C for analytical instrument and equipment.

2D. TEST PROCEDURE

1. Prepare the soil sample in accordance with California Test 201. Split a sample from material passing a No. 8 sieve.
2. Prepare calibration curve as in Part 1, Section (D.1).
3. Weigh 100 g of soil and place it in a 500 mL Erlenmeyer flask. Add 300 mL of deionized water, place a stopper on the flask, and shake vigorously for 15 minutes. Centrifuge the sample, then filter the sample or let the sample settle overnight.
4. Pipette 5 mL of sample into a sample vial and cap. All samples must be filtered using syringe filters to ensure they are free of particulates.
5. Prepare a run program for IC with a blank, calibration curve standards, check standard, samples, and shutdown mode at the end of the program. Dilution factor of three is entered to compensate for the 3 to 1 extraction of soil.
6. It will be necessary to make dilutions on samples with sulfate content higher than the range of the calibration curve.
7. Check the reliability of the calibration curve by running a check standard for every 10 unknown samples.

2E. CALCULATION

Calculation of the sulfates in soil samples is the same as in Part 1, Section E.

PART 3. SULFATE CONTENT OF CONCRETE PATCHING MATERIALS

3A. SCOPE

This method describes the test procedure for determination of the water-soluble sulfate content of concrete patching materials.

3B. REAGENTS AND MATERIALS

Refer to Part 1, Section B for these items.

3C. EQUIPMENT

1. Refer to Part 1, Section C for analytical instrument and equipment.
2. Specimen molds described in ASTM C 109
3. U.S. Standard No. 50 sieve
4. Either a mortar and pestle or a power driven mechanical device such as a Jaw Crusher or a pulverizing apparatus.

3D. TEST PROCEDURE

1. Prepare and cast a 2 inch cube of patching material as recommended by the manufacturer's data sheet. Let cure 7 days at ambient conditions. Crush and pulverize the cured block, split a 100 g sample from the pulverized material passing a No. 50 sieve.
2. Proceed with testing as in Part 2, Section D, part 2.

2E. CALCULATION

Calculation of the sulfates in patching materials is the same as in Part 1, Section E.

D. HEALTH AND SAFETY

It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Prior to handling, testing or disposing of any materials, testers must be knowledgeable about safe laboratory practices, hazards and exposure, chemical procurement and storage, and personal protective apparel and equipment.

Caltrans Laboratory Safety Manual is available on the Caltrans website.

End of Text
(California Test 417 contains 4 pages)