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## **METHOD OF TESTING SOILS AND WATERS FOR SULFATE CONTENT**

### **A. SCOPE**

This method describes test procedures for determination of the 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. This test method is divided into the following parts:

1. Sulfate Content of Waters
2. Sulfate Content of Soils

### **B. 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,  $\text{NaHCO}_3$  in deionized water and dilute to 500 mL volume. Dissolve 26.5 g of Sodium Carbonate,  $\text{Na}_2\text{CO}_3$  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  $\text{NaHCO}_3$ , 7 mL of  $\text{Na}_2\text{CO}_3$  solutions, and dilute to 1000 mL with deionized water.
2. Standard Sulfate solution, 1000 ppm: Dissolve 1.8145 g of  $\text{K}_2\text{SO}_4$  in deionized water and dilute to 1000 mL.
3. Standard Chloride solution, 1000 ppm: Dissolve 1.6485 g of  $\text{NaCl}$  (dried at  $140^\circ\text{C}$ ) 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 in. 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 of each sulfate and chloride solution at 1000 ppm (Part 1B, Number 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:

$C_a$  = 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 1B for these items.

## **2C. EQUIPMENT**

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

## **2D. TEST PROCEDURE**

1. Prepare calibration curve as in Part 1, Section 1D.
2. 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 min. Centrifuge the sample, then filter the sample or let the sample settle overnight.
3. 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.
4. Prepare a run program for IC with a blank, calibration curve standard. 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.
5. It will be necessary to make dilutions on samples with sulfate content higher than the range of the calibration curve.
6. 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 1E.

## **C. 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.

Users of this method do so at their own risk.

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**(California Test 417 contains 3 pages)**