



### C. ANALYTICAL INSTRUMENT AND EQUIPMENT

Ion Chromatograph (IC) – The Dionex DX 120 model has been found to be satisfactory for this method. Any comparable instrument can be used as well.

### D. 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 Ion Chromatograph.

**Note:** The Ion Chromatograph 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 Ion Chromatograph 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.

### E. CALCULATION

$$A_s = \text{Function}(R_s) * V_c/V_s * D_s$$

Where

$A_s$ : is the amount of the analyte in the sample

Function: is a form of the current calibration curve function

$R_s$ : is the response of the analyte in the sample

$V_c$ : is the volume of standard injected

$V_s$ : is the volume of sample injected from the method or schedule

$D_s$ : is the dilution factor from the method or schedule for the sample

## PART 2. SULFATE CONTENT OF SOILS

### A. SCOPE

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

### B. REAGENTS AND MATERIALS

Refer to Part 1, Section B for these items.

### C. EQUIPMENT

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

### D. TEST PROCEDURE

1. Prepare calibration curve as in Part 1, Section (D.1).
1. 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.

2. 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.
3. Prepare a run program for the Ion Chromatograph 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.
4. It will be necessary to make dilutions on samples with sulfate content higher than the range of the calibration curve.
5. Check the reliability of the calibration curve by running a check standard for every 10 unknown samples.

#### **E. CALCULATION**

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

#### **F. REPORTING RESULTS**

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

#### **G. ALTERNATE PROCEDURES**

Other methods that may be used are "Standard Methods", 17<sup>th</sup> Edition, 1989, "Gravimetric Method", and ASTM Designation: D 516, "Sulfate Ion in Water."

### **PART 3. SAFETY AND HEALTH**

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 the Caltrans Laboratory Safety Manual. These sections pertain to

requirements for general safety principals, standard operation 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, 17<sup>th</sup> Edition, 1989, APHA-AWWA-WPCF**  
**U.S. Environmental Protection Agency Manual (EPA), "Methods for Chemical Analysis of Water and Waste"**  
**ASTM Designation: D516**

**End of Text**  
**(California Test 417 contains 3 pages)**