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DIVISION OF ENGINEERING SERVICES
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DETERMINATION OF CHLORIDE CONTENT IN ORGANIC ADDITIVES FOR PORTLAND CEMENT CONCRETE

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read **SAFETY AND HEALTH** in Section D 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.

SCOPE

This method includes two potentiometric procedures used to determine the chloride content of organic compounds used as additives in portland cement concrete. These compounds are usually ligno-sulfonate derivatives and are received in the form of liquids or dry powders.

This test method is divided into the following parts:

1. Manual Titration
2. Automatic Titration

PART 1: MANUAL TITRATION

A. APPARATUS

1. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society. Prepare and store solutions in accordance with ASTM Designation: E 200.
2. Standard 0.1 N silver nitrate titrant. Dissolve 17.0 g of AgNO_3 in deionized water and dilute to 1000 mL.
3. A pH meter, with a mV-scale, low drain, sensitive to 1 mV.

4. Chloride specific ion electrode and a double junction reference electrode. Refer to the literature accompanying electrodes for the proper filling solutions, maintenance and operating procedures.
5. 50-mL graduated buret with 0.05 mL graduations.
6. Magnetic stirring bar and stirring plate.

B. TEST PROCEDURE

1. Weigh 10.0 g of the sample into a 400-mL beaker.
2. Add 200 mL of deionized water.
3. Carefully introduce a magnetic stirring bar and commence stirring.
4. Add 20 mL of concentrated nitric acid.
5. Immerse the electrodes into the sample. Make certain that the level of the outer solution of the double junction reference electrode is at least 1 inch above the level of the sample solution in the beaker.
6. Stir at a moderately fast rate, but do not allow the vortex to go below the ends of the electrodes.

7. Turn the pH meter to the on position and allow it to stabilize, if necessary.
8. Titrate the sample with 0.1 N silver nitrate. Record the potential [E] in millivolts after each volumetric addition. As the end point is approached, the change in the potential will significantly increase, at which time, add the silver nitrate in smaller equal volume increments (e.g., 4 drops).
9. Determine the end point by plotting the curve of potential [E] against volume, mL. Find the point of inflection and determine the volume at this point, A.

C. CALCULATION

$$\% C1 = \frac{A \times N \times 3.55}{\text{Mass of sample, g}}$$

where: A = Volume of AgNO₃, mL, from plot
N = Normality of AgNO₃

PART 2: AUTOMATIC TITRATION

A. APPARATUS

Use the same reagents and materials as in Part 1, in addition to those listed below.

1. Standard 0.01 N silver nitrate titrant. Dissolve 2.395 g of AgNO₃ in deionized water and dilute to 1000 mL.
2. Automatic titrator with a chloride specific ion electrode for endpoint determination.

B. TEST PROCEDURE

1. Place 10.0 g of the sample in a 400-mL beaker.
2. Add 200 mL of deionized water.
3. Add 20 mL of concentrated HNO₃.
4. Titrate the sample with silver nitrate solution to the nearest 0.05 mL.

5. Make a blank determination as in steps 1-4 above except do not weigh a sample into the beaker.

C. CALCULATION

$$\% C1 = \frac{(A - B) \times N \times 3.55}{\text{Mass of sample, g}}$$

where: A = Volume of AgNO₃, mL
B = Volume of the blank, mL
N = Normality of AgNO₃

D. 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 the user of 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:

ASTM Designation: E 200
Caltrans Laboratory Safety Manual

End of Test
(California Test 415 contains 2 pages)