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DETERMINATION OF CHLORIDE CONTENT IN ORGANIC ADDITIVES FOR PORTLAND CEMENT CONCRETE

A. 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.

The procedures for this test method are divided into the following parts:

1. Manual Titration
2. Automatic Titration

B. REFERENCES

ASTM Designation: E 200

C. PROCEDURE

PART 1. MANUAL TITRATION

1A. 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.

1B. 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 in. 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.

1C. CALCULATION

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

where: A = Volume of $AgNO_3$, mL, from plot

N = Normality of $AgNO_3$

PART 2. AUTOMATIC TITRATION

2A. 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 1.698 g of $AgNO_3$ in deionized water and dilute to 1000 mL.

2. Automatic titrator with a chloride specific ion electrode for endpoint determination.

2B. TEST PROCEDURE

Place 10.0 g of the sample in a 400 mL beaker.

1. Add 200 mL of deionized water.

2. Add 20 mL of concentrated HNO_3.

3. Titrate the sample with silver nitrate solution to the nearest 0.05 mL.

4. Make a blank determination as in steps 1-3 above except do not weigh a sample into the beaker.

2C. CALCULATION

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

where: A = Volume of $AgNO_3$, mL

B = Volume of the blank, mL

N = Normality of $AgNO_3$

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 at:

http://www.dot.ca.gov/hq/esc/ctms/pdf/lab_safety_manual.pdf

Users of this method do so at their own risk.

End of Text
(California Test 415 contains 3 pages)