DEPARTMENT OF TRANSPORTATION
DIVISION OF ENGINEERING SERVICES
Transportation Laboratory
5900 Folsom Boulevard
Sacramento, California 95819-4612

TEST METHOD FOR THE CHEMICAL ANALYSIS OF PORTLAND CEMENT, PORTLAND CEMENT CONCRETE, FLY ASH, POZZOLAN, AND BLENDED CEMENT

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH"** in Part 4 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 test method is divided into the following parts:

1. Chemical Analysis of Portland Cement
2. Chemical Analysis of Fly Ash, Pozzolan and Blended Cement
3. Determination of Chlorides in Hardened Portland Cement Concrete
4. Safety and Health

PART 1. CHEMICAL ANALYSIS OF PORTLAND CEMENT

The procedures used in the chemical analysis of portland cement are described in this group of tests. For routine samples, the x-ray fluorescence method is used. For referee samples, or samples where there is a question as to the results, the reference test methods described in ASTM Designation: C 114 shall be used.

A. APPARATUS

1. X-Ray fluorescence spectrometer (XRF) meeting the requirements in ASTM Designation: D 4326.
2. Fluxing device with platinum-gold alloy crucibles as described in ASTM Designation: D 4326.
3. Fusion flux used is a 50 % mixture of lithium tetraborate and lithium metaborate. Lithium bromide solution is supplied by the flux device manufacturer.
4. An oscillating hot plate.
5. Simultaneous Carbon and Sulfur Determinator. Eltra Model CS-800 Induction Furnace has been found satisfactory.
6. Muffle furnace, capable of maintaining 750°C.
7. Various standard cements certified by the National Institute of Standards and Technology (NIST).

B. TEST PROCEDURE

Before testing, pass samples through a U.S. No. 20 sieve in order to mix the sample, break up lumps and remove foreign materials. Discard hardened lumps that do not break up on sieving or brushing. Store the cement in airtight moisture proof containers to prevent aeration or absorption of moisture prior to test.

1. This portion of the method will determine the oxides of silica, aluminum, iron, calcium, magnesium,

sodium, potassium and sulfur. Test in accordance with ASTM Designation: D 4326 except as noted below.

a. 4Select a series of at least seven NIST standard cements that will bracket the expected concentrations of the elements in the unknown samples. Prepare standards in the same way as for samples. It is recommended that new standard fused disks be made every 6 months.

b. Weigh 1.00 g of cement as received into the platinum alloy crucible and 6 g of fluxing material. Add 3 drops of lithium bromide solution as recommended by the manufacturer to prevent the fused disks from adhering to the crucibles.

c. Fuse the samples following the manufacturer's recommendations. Analyze fused disks in the XRF.

2. Determine insoluble residue, carbon dioxide and loss on ignition in accordance with ASTM Designation: C114.

3. An alternative test procedure for sulfur trioxide is to analyze with the carbon sulfur determinator. Follow the manufacturer's recommendations for the testing of cement.

PART 2. CHEMICAL ANALYSIS OF FLY ASH, POZZOLAN AND BLENDED CEMENT

A. TEST PROCEDURE

1. Determine the oxides of silica, aluminum, iron, calcium, magnesium, total sodium and total potassium in the same manner as for cement in Part1 of this test except as indicated below.

a. Heat the sample prior to fusing as indicated for material previously ashed in ASTM Designation: D 4326.

b. Weigh 0.50 g of the ashed flyash sample and 6.5 g of fluxing material into the crucible. Add 400 µL of liquid lithium bromide to the sample before fusing.

2. Determine loss on ignition, available sodium oxide, available potassium oxide and available alkali in accordance with ASTM Designation: C 311.

3. Determine sulfur trioxide with the carbon sulfur determinator. Follow the manufacturer's recommendations for the testing of cementitious products.

PART 2. DETERMINATION OF CHLORIDES IN HARDENED CEMENT CONCRETE

This part describes a procedure for determining chlorides in hardened portland cement concrete. This method includes two potentiometric titration procedures, one manual and the other with an automatic titrator.

A. APPARATUS

1. Chloride specific ion electrode and a double junction reference electrode.

2. Standard solutions and reagents: 0.10 N silver nitrate, concentrated nitric acid, dilute nitric acid (1:99), 0.05 N ammonium thiocyanate. Make and store solutions in accordance with ASTM Designation: E 200.

4. Ferric iron indicator, 5 g of ferric ammonium sulfate dissolved in 50 mL of 1 N nitric acid.

5. Automatic titrator that will be able to sense an endpoint with the attached chloride specific ion electrode and the reference electrode.

6. 50-mL graduated buret with 0.05 mL graduations.

7. An oscillating hot plate.

8. Filtering apparatus consisting of a Buchner funnel, filter flask and a vacuum attachment.

B. TEST PROCEDURE

1. Pulverize hardened portland cement concrete and pass through a U.S. No. 8 sieve.

2. Weigh 2.0 to 5.0 g of sample, to the nearest 0.1 g, into a 400-mL breaker.

3. Add 100 mL of boiling water and place on the oscillating hot plate.

4. Add a slight excess of 0.1 N silver nitrate with a volumetric pipette (5 mL is an approximate amount of silver nitrate for concrete that contains no more than 0.15 % chloride). Record the amount added.

5. Boil the sample for two minutes, remove it from the heat and cool slightly.

6. Slowly add 20 mL of concentrated nitric acid. Effervescence will often occur.

7. Heat and break up any lumps of undissolved cement with the flattened end of a stirring rod, cover the sample and boil it for 2 min.

8. Filter through a medium speed filter paper in a Buchner funnel with suction. The filtrate should be clear.

9. Wash the residue three times with 1:99 nitric acid. Discard the residue and filter paper.

10. Quantitatively transfer the filtrate to a 400-mL beaker and cool it to room temperature.

11. Carry three standards through the test procedure with the same amount of acid and silver nitrate but without cement.

12. Adjust all standards and samples to approximately equal volume with de-ionized water.

13. Titration of Samples:

    a. Manual titration using ferric ion indicator. With concrete that is low in iron, add a few drops of ferric iron indicator. Titrate with 0.05 N ammonium thiocyanate that has been standardized against the silver nitrate standard to the first permanent red color. Use a buret and record all readings to the nearest 0.01 mL.

    b. Automatic titration with the chloride specific ion electrode. Following the manufacturer's instructions, titrate the sample with 0.05 N ammonium thiocyanate that has been standardized against a silver nitrate standard solution. Record the amount of ammonium thiocyanate needed to titrate the silver in the sample to the nearest 0.1 mL.

14. Calculate chloride by the following formula:

$$\text{Chloride, \%} = \frac{\left[\left(A \times N\right) - \left(B \times M\right)\right] \times \left(F\right) 100}{\text{sample mass, } g}$$

Chloride (ppm) = % x 10 000

Where:
A = mL of $AgNO_3$
N = N of $AgNO_3$
B = mL of NH_4SCN
M = N of NH_4SCN
F = chloride conversion factor, 0.03546

PART 4. 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.

Prior to handling testing or disposing of any waste materials, testers are required to read the Caltrans Laboratory Safety Manual.

These guidelines pertain to requirements for general safety principles, standard operating procedures, protective apparel, disposal of materials and how to handle spills, accidents,

emergencies, etc. Testers are mandated to always observe good hygiene practices. Wash hands after handling samples and before eating, drinking or smoking. Users of this method do so at their own risk.

REFERENCES:
ASTM Designations: C 114, C311, D 4326 and E 200; Caltrans Lab Safety Manual

End of Text
(California Test 404 contains 4 pages)