CHLORIDE IN CONCRETE ADMIXTURES

(An Arizona Method)

SCOPE

1. (a) This test method describes a procedure for determining the chloride content of products used as admixtures in Portland cement concrete. The method employs a standard addition technique using a chloride electrode.

(b) This test method involves hazardous material, operations, and equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

(c) See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

APPARATUS

2. Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus shall consist of the following:

(a) 300 mL tall-form beaker (Pyrex #1060 or equivalent), calibrated to indicate 100 mL volume.

(b) An analytical balance capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M231, except the readability and sensitivity of any balance utilized shall be at least 0.001 gram.

(c) Suitable magnetic stirrer and stirring bar.

(d) Chloride electrode system (Orion Ionplus #9617 combination chloride electrode or equivalent) suitable for use in the 300 mL beaker.

(e) Specific ion meter (Orion EA 940 or equivalent) suitable for use with the chloride electrode system.

(f) 3 mL disposable syringe with cap (Beckton Dickinson # 301112 or equivalent).

(g) 1.000 mL pipette or pipetter accurate to ± 0.001 mL.

REAGENTS

3. (a) Chloride standard solution, 1.000 mg/mL (1000 PPM), (Fisher Scientific #LC13000-1, or equivalent, or made by transferring 1.648 gram dried primary standard sodium chloride into a 1 liter volumetric flask and diluting to 1000 mL with demineralized water).

(b) Nitric acid, 8% (made by transferring 80 mL of concentrated nitric acid into a 1 liter volumetric flask containing approximately 500 mL demineralized water and diluting to 1000 mL with demineralized water. **Caution must be exercised in preparing and using this solution! It should be properly labelled and treated as a hazardous material.**

PROCEDURE

4. (a) Weigh 1.000 ± 0.010 gram sample of admixture into a beaker. (If liquid, weigh by difference using a syringe). Record the sample weight to the nearest 0.001 gram as "A."

(b) Transfer 25 mL nitric acid, 8% into the beaker and swirl to mix with the sample.

(c) Dilute to 100 mL with demineralized water.

(d) Place a stirring bar into the beaker, place the beaker onto the magnetic stirrer, insert the electrode(s) into the solution, and initiate stirring. Stir at a constant moderate rate such that the vortex created by stirring does not expose the tip(s) of the electrode(s) to the air. The rate of stirring and the temperature (25.0 ± 1.0 °C) shall be held constant throughout the procedure.

(e) After the reading has stabilized, record it to the nearest millivolt, as "E_1."

(f) Pipette 1.000 ± 0.001 mL chloride standard solution into the beaker.

(g) After the reading has stabilized, record it to the nearest millivolt, as "E_2."

(h) Calculate $\Delta E = E_1 - E_2$.

(i) If ΔE is less than 18 mV, repeat steps (a) through (h) with a smaller size sample.

(j) Prepare a reagent blank solution by transferring 25 mL of Nitric Acid, 8% into a 300 mL beaker and diluting to 100 mL with demineralized water. Repeat steps (d) through (h).

CALCULATIONS AND REPORT

5. (a) Calculate chloride concentration, "C_o" in the sample reading solution and "C_b" in the reagent blank reading solution, and record each to the nearest 0.010 mg/mL, as follows:

$$C_o \text{ or } C_b = \frac{1}{(101)\,10^{(\Delta E/S)} - 100}$$

Where: S = Electrode slope at 25 ± 1.0 °C as determined in accordance with manufacturer's recommendations. (The slope should equal approximately 59 millivolts for a properly functioning electrode.)

(b) Calculate chloride concentration in the admixture in parts per million, "C", and report to the nearest 10 PPM as follows:

$$C = \frac{10^5 (C_o - C_b)}{A}$$