# TOTAL CHLORIDE CONTENT IN CEMENT, MORTAR AND CONCRETE

## 1.0 SCOPE

1.1 This method describes the procedure for the determination of the total chloride content of dry hydrated Portland cement, mortar or concrete. The method is limited to materials that do not contain sulfide, but the extraction procedure may be used for all such materials.

1.2 Total chloride content is determined by the potentiometric titration of chloride with silver nitrate.

## 2.0 APPLICABLE DOCUMENTS

2.1 ASTM C114 Standard test methods for chemical analysis of hydraulic cement, section 19, chloride.

2.2 H. A. Berman, Research Chemist, FHWA, DOT Wash. D.C., "Determination of Chloride in Hardened Portland Cement Paste, Mortar and Concrete." (This paper is based on Report FHWA-RD-72-12)

## 3.0 APPARATUS

3.1 Chloride Ion Selective Electrode with appropriate reference electrode.
Note: Carefully follow the instruction manual for set up, storage and maintenance of the electrodes as described by the manufacturer.

3.2 Potentiometer with millivolt scale readable to 1 mV or better.

3.3 Buret with 0.1 m$\ell$ divisions.

3.4 Magnetic stirrer and teflon stirring bars.

3.5 # 41 Filter Paper.

## 4.0 REAGENTS

4.1 Concentrated $HNO_3$ (sp.gr. 1.42, Nitric Acid)

4.2 Standard 0.01 Normality NaCl (Sodium Chloride) solution; oven dry reagent grade NaCl at 105◦C for 1 hour, air cool, weigh out 0.5844 g, dissolve in distilled water and transfer to a 1 litre volumetric flask. Fill up to the mark with distilled water and mix.

4.3 Standard 0.01 Normality $AgNO_3$ (Silver Nitrate) solution; weigh 1.7000 g of reagent grade Silver Nitrate into a 250 m$\ell$ beaker. Add about 50 m$\ell$ distilled water to dissolve the Silver Nitrate, stir with glass rod until crystals dissolve. Add the mixture to a 1 litre volumetric flask, wash contents of beaker with

distilled water into 1 litre flask. Fill flask to mark, stopper and mix to make homogenous.

To standardize the Silver Nitrate, titrate against 20 mℓ of 0.01 N NaCl prepared in 4.2 using millivoltmeter as described in procedure and calculate using the following formula:

$$\mathbf{N_1V_1 = N_2V_2}$$

$N_1$ - Normality of Sodium Chloride
$V_1$ - Volume of Sodium Chloride Titrated
$N_2$ - Normality of Silver Nitrate
$V_2$ - Volume of Silver Nitrate needed to reach end point of Titration

$$\mathbf{N_2 = 0.2 / V_2}$$

for 20 mℓ 0.01 N NaCl .

4.4 Fill Buret with $AgNO_3$ (Silver Nitrate).

## 5.0 PROCEDURE

5.1 Weigh duplicate 1 gram representative samples of powdered material under test into tared 250 mℓ beakers. Record exact weights to nearest 0.0001 g.

5.2 Add 10 mℓ of distilled water, swirling to bring powder into suspension.

5.3 Add 3 mℓ of 70% $HNO_3$ (Nitric Acid). Use a glass stir rod to mix and break up any lumps to form a slurry.

5.4 Heat the slurry rapidly to boiling. Do not allow to boil for more than a few seconds. Remove from heat.

5.5 Filter slurry into a 250 mℓ beaker using filter paper. Wash residue from beaker and stir rod through filter using hot distilled water. Wash contents of filter paper thoroughly with hot distilled water letting it drain. Repeat washing until a volume of about 75 mℓ is obtained. Allow filtrate to air cool to room temperature.

5.6 Fill the electrode(s) with the solutions recommended by the manufacturer. Connect electrodes to the millivoltmeter and determine the approximate reading of the equivalence point by immersing the electrode(s) in a beaker of distilled water. Allow to stabilize and record. Remove the beaker and wipe electrodes with absorbent paper.

5.7 Rinse with distilled water a magnetic stirring bar and add to the sample prepared in 5.5. Place the beaker on the magnetic stirrer. Immerse the electrodes into the solution taking care that the stirring bar does not strike the electrodes; begin gently stirring.

5.8 Begin titration, recording the volume of standard $AgNO_3$ (Silver Nitrate) verses the millivolt readings to bring the readings to about 40 mV below the equivalence point. Continue the titration with smaller increments (0.2 mℓ). As

you reach the equivalence point, reduce increments to 0.1 mℓ, recording all mV readings after each addition. As the titration proceeds, equal additions of $AgNO_3$ will cause larger changes in the mV readings. When the titration passes the equivalence point the change per increment will then decrease. Continue titration long enough to establish that the meter readings are progressively decreasing. The endpoint of the titration is reached when the maximum difference in the mV readings occurs for equal volumes of $AgNO_3$. This can usually be determined without plotting a curve and occurs at the approximate equivalence point of the electrode(s) in distilled water. (In practice, with AT&U equipment, distilled water reads somewhere between 230 to 250 mV).

## 6.0 CALCULATIONS AND REPORT

6.1 Calculate and report the percent chloride to the nearest 0.001 % as follows:

**Cl, % = (3.5453 * V * N) / W**

Where:

V = millilitres of 0.01 N $AgNO_3$ solution used for titration of the sample (equivalence point).

N = exact normality of 0.01 N $AgNO_3$ solution.

W = weight of sample, grams.

6.2 The results of two properly conducted tests by the same operator on the same sample shall not differ by more than 0.007 percent chloride.