SalierGeotechnical Limited

DETERMINATION OF CHLORIDE CONTENT OF CONCRETE

Lab Test Reference 315
British Standard Reference
BS1881 : Part 124 Test 10.2

Principal Apparatus

i) A thermostatically controlled drying oven capable of maintaining a temperature of 105oC +/-5oC
Lab Inventory No. xxx
ii) Analytical balance capable of weighing up to 5kg accurate to 5g
Lab Inventory No. xxx
iii) BS410 Test sieves 5mm, 2.36mm, 150 mircon, 600 micron
iv) Mortar and Pestle
v) 500ml conical flask (Stopped)
vi) Hotplate
vii) Desiccator
viii) Buchner funnel, flask and filter papers

Reagents

Reagents of analytical quality and only distilled water shall be used.

1. Nitric Acid: relative density 1.4

2. Silver Nitrate Standard solution 0.1 moles/1. Dry powdered silver nitrate at 150oC for 2 hr. (The drying oven can be re-set to operate at this temperature). Cool in a desiccator and dissolve and 16.989g in water and dilute to 1 litre. Store the solution in an opaque glass bottle and protect from prolonged exposure to light.

3. Thiocyanate Standard Solution, approx 0.1 moles/1. Dissolve 7.6g of Ammonium Thiocyanate on 9.7g of Potassium Thiocyanate in water and dilute to 1 litre. Standardise against the silver nitrate solution using iron III indicator, once a week or each time a determination of chloride is carried out (see note 1).

4. Iron III indicator Solution. To 50g of ammonium ferric sulphate, add 60ml of water and warm to dissolve. Add 10ml of nitric acid, cool, and store in a bottle.

5. 3, 5, 5 – trimethylhexanol (nonyl alchol)

1.0 Preliminaries

1.1 The chemistry laboratory shall be used to perform this test and clear working areas of bench must be prepared before testing proceed

1.2 Ensure the sample number and test schedule correspond.

1.3 Obtain the appropriate test work sheet from drawer.

1.4 Check the calibration status of the equipment and sieves.

1.5 Check the condition of the sieves before testing commences.

2. Standard Test Method

2.1 Preparation of Test Portion

Produce an analytical sample by using the same method of preparation as described in paras 2.1 to 2.4 on page 194.

2.2 Analytical Procedure

The method of determination used is known as the Volhard method of titration where a known volume of excess silver nitrate solution of known concentration is used to completely precipitate the chloride ions as the very insoluble Silver chloride. The solution must be acidic to methyl red or some of the chloride will fail to precipitate giving a lower result. The excess silver ions are then back titrated using a solution of Potassium Thiocyante of the same concentration as the Silver Nitrate, using Iron (III) ions as the indicator.

Cl - (aq) + Ag = (aq)   Ag Cl (s)

excess and known quantity

Ag + (aq) + SCN - (aq)   Ag SCN (s)

unknown   known vol & conc

At End Points   3 SCN (aq) + Fe3+ (aq)   Fe (SCN)3

blood red colours

2.3 Weigh into a stoppered 500ml conical flask. 5+ 0.005g of the analytical sample and record as Mc. Disperse with 50ml of water and add 10ml of Nitric Acid. (This should be done in the fume cupboard to allow dispersal of the corrosive and irritating vapour).

2.4 Allow any effervescence to subside and then add 50ml of hot water, boil for 4 to 5 minutes and keep warm for a further 10 to 15 minutes.

2.5 If the supernatant liquid is turbid, then set up the Buchner funnel, flask and Whatman No. 40 filter paper and wet with distilled water. Ensure that no creases occur and the paper is centrally placed in the funnel. Attach to the vaccum pump and switch on.

2.6 Cautiously pour the contents of the flask slowly into the centre of the funnel ensuring the fluid in the funnel never completely covers the paper. This should prevent the pores of the paper from becoming blocked and fine silicaceous suspension which will greatly arrest the rate of filtering.

2.7 Wash out the contents of the beaker completely with the hot water. Transfer the filtrate from the filter flask to an other conical flask, again using hot water to ensure complete transfer of the slightly yellow solution.

2.8 Cool to room temperature and add a measured excess of the silver nitrate solution. Record as V_5 (ml).

2.9 Add 2 to 3 ml of 3, 5, 5 - trimethylhexanol, stopper the flask and shake vigorously to coagulate the precipitate (Note 2). Alternatively, use the magnetic stirrer on the hotplate.

2.10 Add 1 ml of iron III indicator solution and titrate with the thiocyanate solution to the first permanent red colour. Record the volume required as V6 (ml). (See Note 3).

3. Calculations

3.1 Calculate the chloride iron content J as a percentage of the cement for the equation

$$J = \frac{(V_5\ V6\ m)}{0.1} \quad x \quad \frac{0.3545}{M_c} \quad x \quad \frac{100}{C_1}$$

where M_c = mass of sample (in g)

V_5 = Volume of 0.1M silver nitrate solution added (ml)

V_6 = Volume of Thiocyanate solution used (ml)

m = Molarity of the Thiocyanate solution (in mol/l)

C_1 = cement content of sample used (%)

4. Reporting

4.1 The chloride iron content J is expressed as a percentage of the cement to the nearest 0.10% (m/m).

NOTES

Standardisation of Silver Nitrate

1. Measure out accurately 10ml of Potassium Chloride 0.1 M using a clean burette and add a few drops of alkaline dichromate indicator and a few spatulas of Calcium Carbonate. Stir the mixture magnetically and measure the first permanent signs of a red percipitate.

D, the correction factor = VOLUME (Ag+) -aq

VOLUME (SCN-) aq

This ratio should be as close to one as possible.

2. If the solution fails to cloud, the chloride ion content is too low to be detected by this method and be reported so.

3. Towards the end of titration, a short time will elapse before the colour fades to white. If the tinge remains after 2 minutes, then the titration is deemed to be complete.