Transport Roads & Maritime Services

NSW GOVERNMENT

Test method T309

Cement content and mix proportions of hardened concrete

OCTOBER 2012

Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
		Reformatted and Revision Summary Added	D.Dash	May 1999
		Date on Test Method Revised to Agree with Date on Revision Summary	D.Dash	Feb 2001
Ed 2/ Rev 0	All	Reformatted RMS template	J Friedrich	October 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T309 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T309

Cement content and mix proportions of hardened concrete

1. Scope

This test method sets out the procedure for the determination of the cement content of hardened Portland cement concrete containing nominal 20 mm sized aggregate. A method of determining the proportions of sand and coarse aggregate is also given thus providing a means for assessing the original mix proportions.

The method is derived from the Australian Standard 1012 Part 15.

Note: The method outlined is intended primarily for use where the aggregates do not release soluble lime under the conditions of test and where the concrete has not been chemically attacked by external agencies.

2. Sampling

(a) Take every precaution to ensure that the sample is truly representative of the material under test. The test samples normally take the form of concrete test cylinders, concrete cubes or sections cut from existing work.

(b) When the sample is a concrete cylinder tested for compressive strength, collect all the crushed material from the broken cylinder and select sufficient pieces to make up a mass of 3 to 5 kg.

(c) When a concrete test cylinder is to be tested without prior compression testing, split the cylinder by the Brazilian Shear Test Method and take one half of the cylinder as the test sample.

(d) Concrete cores of diameter below 150 mm are taken complete as test sample.

(e) Crush or break the material constituting the test sample into pieces about 20 mm in size.

Separation of Sand and Cement from the Aggregate

3. Apparatus

(a) Muffle furnace capable of maintaining a temperature of $600^{\circ}C \pm 20^{\circ}C$

(b) Stainless steel tray of a size to fit into the muffle furnace and large enough to hold the sample during calcination

(c) Tongs strong enough to handle the tray when loaded with the sample

(d) 4.75 mm Australian Standard Sieve

(e) Mortar with a rubber pestle

4. Procedure

(a) Place the test sample in the stainless steel tray and place in the muffle furnace

(b) Raise the temperature to $600^{\circ}C$ and maintain at this temperature for a period of four hours. (Should it be necessary to use a different temperature or time the details of temperature and time used shall be reported)

(c) Carefully break up the pieces by hand or in a mortar using a rubber pestle to remove the sand and cement from aggregate. Use a knife, spatula or brush to clean the aggregate taking care to collect all the sand and cement

(d) Separate the sand and cement from the coarse aggregate by screening on 4.75 mm sieve

(e) Place the coarse aggregate in a tin on a paint shaker and shake for 10 to 15 minutes to remove adhering cement/sand. Brush the aggregate with a small paint brush to remove dust, if necessary

scraping with a knife to remove any last traces of adhering cement/sand. Continue cleaning the coarse aggregate until all cement and sand particles have been passed through the sieve

(f) Determine the mass of the material retained on the 4.75 mm sieve and the material passing the sieve and record the masses.

Cement Content

5. Apparatus

(a) General laboratory glassware including a burette, pipettes

(b) Hot plate

(c) Ball mill

(d) Water bath

(e) Pestle and mortar

(f) A thermostatically controlled oven with good air circulation capable of maintaining a temperature within the range $105 \pm 3^{\circ}C$

(g) Analytical balance

(h) 300 μm AS sieve

6. Reagents

(a) EDTA (0.02 M). Dissolve 6.445 g of diaminoethanetetra-acetic acid disodium salt and dilute to 1 litre. Standardise against a standard calcium chloride solution using Eriochrome Black T as an indicator

(b) Standard Calcium Chloride Solution. Place 2 g of $CaCl_2$ weighed to the nearest 0.1 mg in a 400 mL beaker and cover with a watch-glass

Add 100 mL of 5.5 M Hydrochloric Acid, warm to expel CO_2 and dilute to 1000 mL in a volumetric flask

1 mL of $CaCl_2$ solution is equivalent to 0.00112 g CaO.

Alternatively the EDTA may be standardised against a standard cement sample

(c) Triethanolamine (20%). Dissolve 100 mL in 400 mL water

(d) Hydrochloric Acid (10 M)

(e) Potassium Hydroxide Solution (8M). Dissolve 44.8g AR KOH in 60 mL distilled water, cool, make up to 100 mL in a volumetric flask and store in a plastic bottle.

CAUTION: Potassium Hydroxide Solution is very alkaline and corrosive and can cause severe burns. Avoid contact with eyes, skin and clothing. If spilt wash off immediately with water. The solid generates much heat when added to water. Safety glasses must be worn.

(f) Potassium Cyanide

(g) Patton and Reeder Indicator. 0.5 g of Indicator is ground together with 50 g KNO_3. Use 0.2 g for titration

7. Preparation of Samples

(a) Mix the material passing the 4.75 mm sieve thoroughly and quarter down carefully taking care not to lose any fines to a sample of 500 g.

(b) Place the sample in a ball mill and reduce to a fine powder passing the 300 μm sieve.

(c) Recover the material carefully from the ball mill. Remove any free iron from the sample as far as possible with a magnet, mix carefully and separate out a 20 g sample by coning and channelling. Remove any further particles of iron from the reduced sample.

8. Procedure

(a) Dry the sample in an oven at 105°C - 110°C for one hour, cool, weigh out accurately 0.5 g and place in a 250 mL beaker.

(b) Add 10 mL of cold water and disperse by swirling gently. Add 10 mL of 10 M hydrochloric acid cautiously and when effervescence has ceased swirl gently once more. Break up any lumps with a glass rod.

(c) Evaporate the solution to dryness on a water bath. Bake on a hot plate, with a "speedivap" watch-glass as a cover on the beaker for a period of 1 to 1.5 hours.

(d) Cool the beaker and contents and add 10 mL of hydrochloric acid, cover with a watch-glass and boil for about 30 seconds. Crush any lumps with a glass rod.

(e) Cool the beaker, dilute, filter and wash the contents into a 250 mL wide mouth Erlenmeyer flask.

(f) Add 10 mL of 20% triethanolamine followed by 4 mL of 8 M potassium hydroxide solution to the first flask and mix.

(g) Allow the flask to stand for three to five minutes, with occasional swirling. Add about 30 mg of potassium cyanide and swirl the flask until dissolved.

CAUTION: Potassium Cyanide is very poisonous by ingestion. Avoid contact with skin and as a precaution wash hands after completing titrations. Avoid contact of the solution with acids as HCN gas (extremely poisonous) is liberated. Make the solution alkaline pH >> 12, add calcium hypochlorite (dry chlorine) and stand for 24 hours before discarding.

(h) Add 0.2 g of Patton and Reeder indicator and titrate with standard EDTA to a pure blue end point.

(i) Refill the burette and put into the second flask 10 mL of triethanolamine followed by an amount of titrant equal to the amount required less 1 mL. Add 4 mL of 8 M potassium hydroxide, 30 mg of potassium cyanide and one measure (0.2 g) of indicator.

(j) Continue the titration as with the first sample. Record the volume of EDTA used.

(k) Carry out a blank titration replacing the sample with distilled water and using all the other chemicals specified above.

9. Calculations

One millilitre of 0.02 M EDTA is equivalent to 1.1216 mg of calcium oxide (CaO).

(a) Calculate the amount of calcium oxide present in the sample of sand/cement as a percentage of the mass of sand/cement mixture taken for the analysis.

(b) Calculate the percentage of cement in the sand/cement mixture by the formula:

$$Percentage\ cement = \frac{Calcium\ oxide\ percent}{0.64}$$

(c) Calculate the percentage of sand present by difference.

(d) Calculate and report the relative amounts of cement, sand and aggregate in the original sample taken and the mix proportions by mass as follows:

$$\frac{Mass\ of\ cement}{Mass\ of\ cement} : \frac{Mass\ of\ sand}{Mass\ of\ cement} : \frac{Mass\ of\ aggregate}{Mass\ of\ cement}$$

(e) Calculate and report the cement content of the hardened concrete as follows:

$$\%\ Cement = \frac{Mc \times 100}{M_C + M_S + M_a}$$

Where

M_s = *Mass of sand*

M_a = *Mass of aggregate*

M_c = *Mass of cement*