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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 43872-2024

**Test methods for determining the immobilization ratio of
chloride in cement**

水泥氯离子固化率检测方法

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3 Terms and definitions

Free chloride is the chloride left loose in the pore solution of hardened cement paste. Bound chloride is the chloride that has combined with the hydration products of the cement and no longer moves freely in that pore solution. Total chloride content is the sum of the two.

The chloride immobilization ratio is the percentage of the total chloride content that the hardened paste holds as bound chloride.

4 Basic requirements for the test

The paste mixer follows JC/T 729 and the mould follows JC/T 726. The mould is made up of dividers, end plates, a base, a clamping device and locating pins, can be taken apart, and casts three cubes of 20 mm by 20 mm by 20 mm. The curing box follows JC/T 959 and the drying oven GB/T 30435.

Two balances are needed: an analytical balance with a range of at least 200 g and a division of not more than 0.0001 g, and an electronic balance with a range of at least 1000 g and a division of not more than 0.1 g. The 0.15 mm and 0.50 mm test sieves follow GB/T 6003.1; filter paper and the suction filtration set follow GB/T 176; the bottles are polyethylene or polyvinyl chloride.

The casting room is held at 20 +/- 2 degrees Celsius with a relative humidity of not less than 50 per cent, and that humidity is recorded at least once a day during working hours. Test water is of grade three or better to GB/T 6682.

Four reagents are made up. For the filtration method, sodium chloride solution at 0.5 mol per litre: guaranteed grade sodium chloride is dried at 105 +/- 5 degrees Celsius to constant mass, 29.2500 g +/- 0.0001 g is weighed, dissolved in a 200 mL beaker and made up to

1000 mL. Saturated calcium hydroxide solution: not less than analytical grade calcium hydroxide, 1.65 g $\pm$ 0.01 g per litre, dissolved and made up to 1000 mL. For the equilibrium method, sodium chloride solution at the same concentration but dissolved with 150 mL of the saturated calcium hydroxide solution and made up to 1000 mL with more of it. Silver nitrate titrant at 0.01 mol per litre: 1.6987 g $\pm$ 0.0001 g of silver nitrate, dried two hours at 105 $\pm$ 5 degrees Celsius, dissolved and made up to 1000 mL, kept in a brown bottle away from light.

5 Filtration method, the reference method

Cement paste is cast, cured, then crushed and steeped in sodium chloride solution; free chloride and total chloride are both measured on the steeped material and the immobilization ratio follows from the difference.

For casting, 300 g $\pm$ 1 g of cement is weighed with 120 mL $\pm$ 1 mL, or 120 g $\pm$ 1 g, of water, giving a water to cement ratio of 0.40. The paste is mixed to GB/T 1346 and poured evenly into the mould; one end of the mould is lifted about 30 mm and dropped ten times, then the other end is lifted 30 mm and dropped ten times, and the surface is struck off. The specimens are cured in water under the standard conditions of GB/T 17671 for 28 days, counted from the moment water met cement.

The cured specimen is crushed and the 0.15 mm to 0.50 mm fraction is sieved out and dried at 60 $\pm$ 1 degrees Celsius to constant mass. A portion of 40.0 g $\pm$ 0.5 g goes into a 500 mL beaker with 400 mL of the sodium chloride solution; the beaker is sealed with cling film and left to stand seven days in the laboratory. Fast filter paper or a sintered glass funnel is rinsed with water first, then the sample is filtered under suction, 50 mL of water in total being used to wash it through.

The filtered material is dried again at 60 $\pm$ 1 degrees Celsius to constant mass, ground in an agate mortar until all of it passes the 0.15 mm sieve, reduced to 10 g following GB/T 176, mixed and sealed in a clean dry bottle.

Total chloride is determined to GB/T 176 on 0.5000 g $\pm$ 0.0001 g of that material, and free chloride to JGJ/T 322 on a further 0.5000 g $\pm$ 0.0001 g, the silver nitrate titrant being at 0.01 mol per litre. Each determination is run three times and the mean taken; if one of the three lies more than 10 per cent either side of the mean it is dropped and the mean of the rest is taken, and if two of the three lie outside that band the determination is repeated.

The immobilization ratio is the difference between total and free chloride divided by the total chloride and multiplied by one hundred, reported to one decimal place, with both chloride contents in milligrams per gram.

6 Equilibrium method, the alternative method

Here the crushed paste is sealed in a sodium chloride solution until the solution and the

pore liquid come to equilibrium, and the bound chloride is worked out from the change in the chloride concentration of the solution.

Casting and curing follow the filtration method. The specimen is crushed, the 0.15 mm to 0.50 mm fraction sieved out, 40.0 g $\pm$ 0.5 g of it dried at 60 $\pm$ 1 degrees Celsius to constant mass. Exactly 10.0 g goes into a dry plastic bottle with 100 mL of the sodium chloride solution made up in saturated calcium hydroxide, pipetted in, and the bottle is sealed and left to stand seven days at 20 $\pm$ 2 degrees Celsius. Then 10 mL of the clear upper liquid is pipetted into a 250 mL conical flask, 90 mL of water is added and mixed, and that is the test solution.

The material left in the bottle is filtered as in the filtration method, dried at 60 $\pm$ 1 degrees Celsius to constant mass, ground until all of it passes the 0.15 mm sieve, mixed and sealed in a clean dry bottle.

A 20 mL portion of the test solution is titrated to JGJ/T 322 with silver nitrate at 0.01 mol per litre. The chloride concentration of the test solution follows from the titrant concentration times the volume of titrant used, divided by the volume of test solution taken, and is reported to three decimal places.

The bound chloride then follows from the volume of the soaking liquid, its initial chloride concentration, the chloride concentration found in the test solution and the net mass of the test portion, with 35.45 as the relative atomic mass of chlorine. The printed equation came through the source file damaged and is not reproduced here; the quantities it uses are the bound chloride in milligrams per gram, the volume of soaking liquid in millilitres, the initial and final chloride concentrations in moles per litre, and the mass of the test portion in grams. The result is reported to three decimal places.

Free chloride is determined to JGJ/T 322 on 0.5000 g $\pm$ 0.0001 g of the material left in the bottle. The rule of three determinations, with one outlier dropped and two outliers forcing a repeat, applies here as well. The immobilization ratio is the bound chloride divided by the sum of bound and free chloride, multiplied by one hundred and reported to one decimal place.

7 Repeatability and reproducibility

Results obtained in one laboratory, on the same equipment and by the same operator within a short time, are to agree within a relative error of 10 per cent.

Results obtained on the same sample by different laboratories, different operators, at different times and on different equipment are to agree within a relative error of 15 per cent.