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INDUSTRIAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

JC/T 420-91

**Chemical analysis method of
chlorine in cement raw materials**

水泥原料中氯的化学分析方法

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Chemical analysis method of chlorine in cement raw materials

1 Subject content and scope of application

This Standard specifies an analytical method for the determination of chlorine in cement raw materials by phosphoric acid distillation-mercury salt titration.

This Standard applies to the determination of chlorine in cement raw materials.

2 Method summary

At a temperature gradient of 170 ~ 280 °C, use a specified distillation device, decompose the sample with phosphoric acid and hydrogen peroxide, use purified air as carrier, and separate chlorine by distillation. Use 0.1 mol/L nitric acid as absorption solution, and distill for 5 to 8 min (depending on the content), then add ethanol that is equivalent to 75 % (V/V) volume to the distillate. At a pH of about 3.5, using phenylazoformic acid 2-phenylhydrazide as indicator, perform titration with mercury nitrate standard solution.

3 Reagents

3.1 The water used for chemical analysis refers to deionized water. The reagents used shall be analytical or guarantee reagents. The reagents used for calibration shall be reference reagents. When in doubt about deionized water and the reagents used, it shall be inspected.

3.2 Phosphoric acid [85 % (*m/m*) H₃PO₄].

3.3 Ethanol [95 % (V/V) C₂H₅OH].

3.4 Nitrogen peroxide [30 % (*m/m*) H₂O₂].

3.5 0.5 mol/L sodium hydroxide (NaOH) solution: DISSOLVE 2 g of sodium hydroxide in 100 mL of water.

3.6 0.5 mol/L nitric acid solution: TAKE 3 mL of nitric acid [65 % (*m/m*)] and DILUTE to 100 mL with water.

3.7 Chlorine standard solution: Accurately WEIGH 0.3297 g of spectrally pure

sodium chloride that has been dried at 105 ~ 110 °C for 2 h, DISSOLVE in a small amount of water, then TRANSFER into a 1 L volumetric flask, DILUTE to the mark with water, and SHAKE well. 1 mL of this solution contains 0.2 mg of chlorine.

PIPETTE 200 mL of the above solution into a 1 L volumetric flask, DILUTE to the mark with water, and SHAKE well. 1 mL of this solution contains 0.04 mg of chlorine.

3.8 Bromophenol blue indicator [0.1 % (m/V)] ethanol solution: DISSOLVE 0.1 g of bromophenol blue in 100 mL of 20 % ethanol.

3.9 phenylazoformic acid 2-phenylhydrazide [1 % (m/V)] ethanol solution: DISSOLVE 1 g of phenylazoformic acid 2-phenylhydrazide in 100 mL of 95 % ethanol.

3.10 0.001 mol/L mercury nitrate [Hg(NO₃)₂] standard solution: WEIGH 0.34 g of mercury nitrate [Hg(NO₃)₂ · $\frac{1}{2}$ H₂O], DISSOLVE in 10 mL of 0.5 mol/L nitric acid, TRANSFER into a 1 L volumetric flask, DILUTE to the mark with water, and SHAKE well.

3.11 0.005 mol/L mercury nitrate [Hg(NO₃)₂] standard solution: WEIGH 1.67 g of mercury nitrate [Hg(NO₃)₂ · $\frac{1}{2}$ H₂O], DISSOLVE in 10 mL of 0.5 mol/L nitric acid, TRANSFER into a 1 L volumetric flask, DILUTE to the mark with water, and SHAKE well.

Mercury nitrate standard solution calibration method: USE a micro-burette to accurately add 0.20 mg (or 1.40 mg) of chlorine standard solution to a 50 mL Erlenmeyer flask; ADD 20 mL of ethanol (95 %) and a few drops of 0.5 mol/L sodium hydroxide until the solution turns blue; ADD dropwise 0.5 mol/L nitric acid until the solution just turns yellow, and then ADD 1 drop in excess (pH is about 3.5); ADD 10 drops of phenylazoformic acid 2-phenylhydrazide indicator; USE 0.001 mol/L (or 0.005 mol/L) mercury nitrate standard solution to titrate until cherry red appears. At the same time, a blank test is performed.

Titer of mercury nitrate standard solution for chlorine, which is calculated according to formula (1):

$$T_{Cl^-} = \frac{0.20}{V_1 - V_0} \quad \text{or} \quad T_{Cl^-} = \frac{1.40}{V_1 - V_0} \quad \dots\dots\dots (1)$$

where:

T_{Cl^-} - the milligrams of chlorine equivalent to per milliliter of mercury nitrate

6 Analysis steps

6.1 Before the test, the distillation device shall be connected according to the figure above and the components shall be inspected.

6.2 The sample quantity can be determined according to the chlorine content. The weighing shall be accurate to 0.0002 g.

When the chlorine content is less than 0.2 %, weigh 0.3 ~ 0.5 g; when the chlorine content is 0.2 % ~ 1 %, weigh 0.3 ~ 0.1 g. Carbonate samples should be 0.3 g.

6.3 PLACE the weighed sample in a dried quartz distillation tube. Do not allow the sample to adhere to the tube wall.

6.4 ADD approximately 2 mL of water and 5 drops of 0.5 mol/L nitric acid to a 50 mL Erlenmeyer flask; PLACE the Erlenmeyer flask in the suction bottle; STOP the rubber stopper.

6.5 PUT the holder on the quartz distillation tube; ADD 5 drops of hydrogen peroxide (30 %) solution and 5 mL of phosphoric acid (85 %) to the distillation tube. After most of the carbon dioxide gas generated by the decomposition of the sample escapes, immediately PUT the ground stopper and PLACE it in a heating furnace with a temperature gradient of 170 ~ 280 °C. Quickly CONNECT the inlet and outlet parts of the distillation tube with silicone rubber tubes (connect the outlet tube first, then the inlet tube). PUT ON the furnace lid.

6.6 START the suction pump; ADJUST the airflow speed to 230 ± 50 mL/min. After 5 minutes of distillation, TURN OFF the suction pump, REMOVE the connecting tube, REMOVE the distillation tube and PLACE it in the test tube holder.

6.7 PURGE the thin glass tube connected to the suction bottle and its lower end with 95 % ethanol in the conical flask (approximately 12 mL of ethanol). REMOVE the Erlenmeyer flask from the suction bottle; ADD 2 drops of 0.1 % bromophenol blue indicator and a few drops of 0.5 mol/L sodium hydroxide to the solution until it turns blue; then NEUTRALIZE with 0.5 mol/L nitric acid until the solution just turns yellow, and ADD 1 drop in excess; ADD 10 drops of 1 % phenylazoformic acid 2-phenylhydrazide indicator; TITRATE with 0.001 mol/L mercury nitrate standard solution until cherry red appears.

6.8 When the chlorine content is 0.2 % ~ 1 %, the distillation time shall be 8 min, and the titration shall be performed with 0.005 mol/L mercury nitrate standard solution.

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