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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 15453-2018

Replacing GB/T 15453-2008

Determination of chloride in water for industrial circulating cooling system and boiler

工业循环冷却水和锅炉用水中氯离子的测定

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1 Scope

GB/T 15453-2018 is the Chinese national standard on determination of chloride in water for industrial circulating cooling system and boiler, in the field of chemical technology. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 7 June 2018 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 January 2019. Classification: ICS 71.040.40, CCS G76. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Standard specifies the method to determine chloride content in water for industrial circulating cooling system and boiler. The molar method and potentiometric titration method in this Standard are applicable to the determination of chloride content in natural water, circulating cooling water, softened water and boiler water. The determination range of molar method is 3mg/L~150mg/L. When it exceeds 150mg/L, the sampling volume can be appropriately reduced. Determine after dilution. The determination range of potentiometric titration method is 5mg/L~1000mg/L. Coprecipitation and enrichment spectrophotometry method is suitable for the determination of chloride in demineralized water and boiler feed water. The determination range is 10μg/L~100μg/L.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 601, Chemical reagent - Preparations of standard volumetric solutions

GB/T 602, Chemical reagent - Preparations of stock standard solutions

GB/T 603, Chemical reagent - Preparations of reagent solutions for use in test methods

GB/T 6682, Water for analytical laboratory use - Specification and test methods

3 General

WARNING -- The strong acids used in this Standard are corrosive. Avoid inhalation or contact with skin during use. If splashed on the human body, rinse immediately with plenty of water. In severe cases, seek medical attention immediately. The reagents and water used in this Standard, unless otherwise specified, shall be analytically pure reagents and grade three water that meets the requirements of GB/T 6682. The standard titration solution, impurity standard solution, preparation and product required in the test shall be prepared in accordance with the provisions of GB/T 601, GB/T 602 and GB/T 603, unless other requirements are specified.

4 Molar method

4.1 Principle Use potassium chromate as indicator. In the pH range of 5.0~9.5, use silver nitrate standard titration solution to titrate. Silver nitrate reacts with chloride to form silver chloride white precipitate. When there is excess silver nitrate, it reacts with potassium chromate indicator to form brick red silver chromate precipitation. It indicates that the reaction has reached the end point. The reaction formulas are:

4.2 Reagents or materials

4.2.1 Nitric acid solution: 1+300.

4.2.2 Sulfuric acid solution: c(1/2H2SO4) is about 0.1mol/L.

4.2.3 Silver nitrate standard titration solution: c(AgNO3) is about 0.02mol/L. Prepare according to GB/T 601. It can also be prepared according to Annex A.

4.2.4 Potassium chromate indicator solution: 50g/L.

4.2.5 Phenolphthalein indicator solution: 10g/L ethanol solution.

4.3 Test steps Use a pipette to measure 50mL or 100mL of water sample into a 250mL

conical flask. Add two drops of phenolphthalein indicator solution. If the water sample turns red, use nitric acid solution or sulfuric acid solution to adjust the pH of the water sample. Make the red just colorless. Add 1.0mL of potassium chromate indicator solution. Under the condition of white background, use silver nitrate standard titration solution to titrate until the brick red color just appears. Conduct the blank test at the same time.

4.4 Result calculation (White) (Brick red) titration solution to titrate till the potential jump point appears. The chloride ion content can be calculated from the volume of the silver nitrate standard titration solution consumed by the jump point.

5.2 Reagents or materials

5.2.1 Nitric acid solution: 1+300.

5.2.2 Sulfuric acid solution: c(1/2H2SO4) is about 0.1mol/L.

5.2.3 Silver nitrate standard titration solution: c(AgNO3) is about 0.05mol/L.

5.2.4 Methyl orange indicator solution: 1g/L.

5.3 Instruments and equipment

5.3.1 Automatic potentiometric titrator: Equipped with silver/silver chloride reference electrode, silver electrode or composite silver electrode.

5.4 Test steps Use a pipette to take an appropriate volume of water sample into a measuring cup. For water samples with pH >7 or water samples containing antiscalants, adjust the pH of the water sample to 3~4 (it may add two drops of methyl orange indicator solution. Use nitric solution or sulfuric solution to adjust the pH of water sample. Make the solution turn from yellow to orange-red). Place the measuring cup on the titration stand. Insert the electrodes. Switch on the potentiometric titrator and stirrer. Use silver nitrate standard titration solution to titrate. Stop titration when a complete jump curve appears.

5.5 Result calculation The chloride content is calculated in mass concentration rho2. The value is expressed in milligrams per liter (mg/L), calculated according to formula (2): Where, V1 - The value of the volume of the standard titration solution of silver nitrate consumed when the specimen is titrated to the potential jump point, in milliliters (mL); c - The exact value of the actual concentration of the silver nitrate standard titration solution, in moles per liter (mol/L); M - The value of the molar mass of chlorine, in grams per mole (g/mol) (M = 35.45);

6.2.6 Ferric nitrate-perchloric acid solution: Weigh 12.0g of ferric nitrate [Fe(NO3)3·9H2O]. Use 43mL of perchloric acid and appropriate amount of water to dissolve. Then use water to dilute to 1000mL.

6.2.7 Chloride standard stock solution: 0.1mg/mL.

6.2.8 Chloride standard solution: 10μg/mL. Pipette 10mL of chloride standard stock solution to a 100mL volumetric flask. Use water to dilute to the scale.

6.3 Instruments and equipment

6.3.1 Spectrophotometer: Equipped with a 30mm absorption cell.

6.3.2 Centrifuge: The rotating speed can reach 5000r/min. It is equipped with a 250mL polyethylene centrifuge tube.

6.3.3 All glassware, polyethylene centrifuge tubes, sampling bottles shall be immersed in nitric acid solution. Rinse with water before use.

6.4.1 Plotting of calibration curve

6.4.1.1 Use a pipette to respectively measure 0.00mL (blank), 0.20mL, 0.40mL, 0.60mL, 1.00mL, 1.50mL, and 2.00mL of chloride standard solution. Pour into 250mL polyethylene centrifuge tubes. Use water to dilute to about 200mL. The corresponding chloride ion masses are 0.0μg, 2.0μg, 4.0μg, 6.0μg, 10.0μg, 15.0μg and 20.0μg, respectively.

6.4.1.2 Add 4mL of lead nitrate solution to the centrifuge tube. Shake well. Add 4mL of disodium hydrogen phosphate-potassium dihydrogen phosphate mixed solution. Shake well. Let stand for 5min.

6.4.1.3 Place the centrifuge tube in the centrifuge tube holder. Centrifuge at 5000r/min for 5min. Pour off the centrifuge. Leave the precipitate in the centrifuge tube. Add 10.00mL of ferric nitrate-perchloric acid solution to the centrifuge tube. Make the precipitate dissolved completely.

6.4.1.4 Add 1.00mL of mercury thiocyanate-methanol solution. After 5min of color development, use a 30mm absorption cell at a wavelength of 460nm. Use blank as a reference. Measure its absorbance.

6.4.1.5 Take the measured absorbance as the ordinate and the corresponding chloride ion mass as the abscissa to plot a calibration curve or calculate a regression equation.