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Determination of copper, iron and zinc in industrial circulating cooling water and scale - Atomic absorption spectrometric method

工业循环冷却水及水垢中铜、铁、锌的测定 原子吸收光谱法

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Determination of copper, iron and zinc in industrial circulating cooling water and scale - Atomic absorption spectrometric method

1 Scope

This document specifies the determination method of copper, iron and zinc content in industrial circulating cooling water and scale in boiler water system or circulating water system - atomic absorption spectrometry.

This document is applicable to the determination of copper content of 0.1mg/L~20mg/L, iron content of 0.1mg/L~20mg/L and zinc content of 0.1mg/L~20mg/L in industrial circulating cooling water; the determination of copper content ≥ 0.005%, iron content ≥ 0.01%, zinc content ≥ 0.005% in scale.

This document is also applicable to the determination of copper, iron and zinc in boiler water and the determination of copper, iron and zinc in scale in other industrial water, raw water and water systems, as well as the determination of zinc content in phosphorus-zinc pre-film solution for industrial circulating cooling water.

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 4470, *Analytical spectroscopic methods. Flame emission, atomic absorption and atomic fluorescence. Vocabulary*

GB/T 6682-2008, *Water for analytical laboratory use - Specification and test methods*

DL/T 1151.2, *Analytical methods of scale and corrosion products in power plants. Part 2: Sampling and dissolving method for scale and corrosion products*

HG/T 3530, *Industrial circulating cooling water. Sampling and production for sludge and corrosion products*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 4470 apply.

4 Principle

The specimen is atomized and sprayed into the air-acetylene flame. Copper, iron, and zinc are pyrolyzed into ground state atoms. Take copper resonance line 324.7nm, iron resonance line 248.3nm, and zinc resonance line 213.9nm as analysis lines to measure the absorbance of copper, iron, and zinc atoms.

5 Reagents or materials

WARNING -- Acetylene gas used in this document is flammable and explosive when mixed with air. Pay attention to safety. Prevent leakage. Operate strictly according to the specifications. Strong acids are corrosive, so be careful when using them. When splashed on body, rinse with plenty of water. Avoid inhalation or contact with skin.

Reagents used in this document, unless otherwise specified, are only analytically pure reagents. See GB 6819 for the requirements of acetylene gas used in the test.

5.1 Water: GB/T 6682-2008, grade two.

5.2 Hydrochloric acid.

5.3 Nitric acid.

5.4 Perchloric acid.

5.5 Nitric acid solution: 1+1.

5.6 Nitric acid solution: 1+99.

5.7 Silver nitrate solution: 10g/L.

5.8 Copper, iron, zinc standard stock solution: 1000mg/L. Commercially available or prepare according to Annex A.

5.9 Copper, iron, zinc standard solution I: 50mg/L. Accurately pipette 5.00mL of standard stock solutions of copper, iron and zinc respectively. Put each into a 100mL volumetric flask. Use water to dilute to the scale. Shake well. The solution is valid for 1 month.

5.10 Zinc standard solution II: 5mg/L. Accurately pipette 5.00mL of zinc standard

a) Weigh about 0.2g~0.3g of the prepared scale sample, accurate to 0.1mg. Place it in a 250mL glass beaker.

b) Add a small amount of water to the beaker to fully wet the scale sample. Slowly add 15mL of hydrochloric acid and 5mL of nitric acid. Cover with a watch glass. Shake well. Slowly heat and boil on a hot plate or an adjustable electric stove for 20min. If there is still a brown or brown residue, add 10mL of hydrochloric acid. Boil until the solution is clear.

c) Remove the beaker. Add 10mL of perchloric acid when the beaker is slightly cooled. Reheat until it starts to emit thick white smoke. Move the watch glass slightly. Continue to heat slowly for 15min~20min. Never evaporate the solution to dryness.

d) Remove the beaker. Add 10mL of nitric acid solution (5.6) while the breaker is hot. Stir well to dissolve the salt on the wall of the beaker.

e) If the scale sample is completely dissolved, transfer the solution to a 100mL volumetric flask. Use a straw to absorb the nitric acid solution (5.6) to wash the inner wall of the beaker (not less than 3 times). Collect the washing solution together in a 100mL volumetric flask. Add nitric acid solution (5.6) to dilute to the scale. Shake well. It shall be the scale sample test solution. If the dissolved solution contains white suspended matter, use medium-speed quantitative filter paper to filter. Use nitric acid solution (5.6) (about 10mL each time) to wash the beaker wall and the sediment and filter paper attached to the wall several times (not less than 5 times). The filtrate and washings are collected together in a volumetric flask. Add nitric acid solution (5.6) to dilute to the scale. Shake well. It shall be the scale sample test solution.

8.2.3 Dissolve scale samples by microwave digestion. Do as follows:

a) Weigh 0.2g~0.3g of the prepared scale sample, accurate to 0.1mg. Place in the digestion inner tank of microwave digestion instrument. Moisten with a few drops of water. Slowly add 6mL of hydrochloric acid and 2mL of nitric acid. Shake the digestion inner tank. Let the gas run out (if a large amount of gas is generated, pre-digest the inner tank on the electric heating plate at 100°C for 20min, and then add the same proportion of digestion solution to about 10mL). Cover the tank tightly and put it in the outer tank. Put the digestion tank into the microwave digester. Digest the scale sample according to the instrument instructions.

b) After the digestion is complete, transfer the solution in the tank to a 250mL glass beaker. Wash the digestion tank and lid with a small amount of nitric acid solution (5.6) and pour it into a beaker. Add 10mL of perchloric acid. Place beaker on hot plate or adjustable hotplate. Slowly heat until white smoke is emitted for 15min~20min. Never evaporate the solution to dryness. Remove the beaker from hot plate or adjustable hotplate. Add 10mL of nitric acid solution (5.6) while the breaker is hot. Stir well to dissolve the salt on the wall of the beaker.

c) If the scale sample is completely dissolved, transfer the solution to a 100mL volumetric flask. Use a straw to absorb the nitric acid solution (5.6) to wash the inner wall of the beaker (not less than 3 times). Collect the washing solution together in a 100mL volumetric flask. Add nitric acid solution (5.6) to dilute to the scale. Shake well. It shall be the scale sample test solution. If the dissolved solution contains white suspended matter, filter it with medium-speed quantitative filter paper. Use nitric acid solution (5.6) (about 10mL each time) to wash the beaker wall and the sediment and filter paper attached to the wall several times (not less than 5 times). The filtrate and washings are collected together in a volumetric flask. Add nitric acid solution (5.6) to dilute to the scale. Shake well. It shall be the scale sample test solution.

8.2.4 Except that the scale sample is not added, follow the same operation as in 8.2.2 or 8.2.3 to prepare the scale sample test solution to prepare a dissolved sample blank solution.

9 Determination steps

9.1 Determination of copper content

9.1.1 Drawing of copper calibration curve

Accurately pipette 0.00mL (blank), 0.50mL, 1.00mL, 1.50mL, 2.00mL of copper standard solution I. Respectively place in 50mL volumetric flasks. Use nitric acid solution (5.6) to dilute to the scale. Shake well. The copper content of this series of calibration solutions is 0.00 mg/L, 0.50 mg/L, 1.00mg/L, 1.50mg/L, 2.00mg/L. Under the best working conditions of the instrument, at a wavelength of 324.7nm, zero with reagent blank. Measure its absorbance. Take the measured absorbance as the ordinate, and the corresponding copper content (mg/L) as the abscissa, to draw a calibration curve or calculate a regression equation. The linear correlation coefficient of the calibration curve shall be greater than 0.999, otherwise it shall be redrawn.

9.1.2 Determination of water samples

Accurately pipette an appropriate amount of specimen solution (8.1). Place in a 50mL volumetric flask. Use nitric acid solution (5.6) to dilute to the scale. Shake well. According to the same instrument conditions in the preparation of the calibration curve, adjust to zero with reagent blank. Measure its absorbance. Find out the corresponding copper content from the calibration curve. If the copper content is lower than the lower limit of detection or exceeds the range of the calibration curve, the dilution factor can be adjusted and measured again.

9.1.3 Determination of scale samples

Accurately pipette appropriate amount of scale sample test solution (8.2.2 or 8.2.3) and dissolved sample blank solution (8.2.4). Place in a 50mL volumetric flask. Use nitric acid solution (5.6) to dilute to the scale. Shake well. According to the same instrument conditions in the preparation of the calibration curve, zero with reagent blank. Measure the absorbance of the dissolved sample blank solution and the scaled sample solution after diluting to constant volume, respectively. Find out the corresponding copper content from the calibration curve. If the copper content is lower than the lower limit of detection or exceeds the range of the calibration curve, the dilution factor can be adjusted and measured again.

9.2 Determination of iron content

9.2.1 Preparation of iron calibration curve

Accurately pipette 0.00mL (blank), 1.00mL, 2.00mL, 4.00mL, 6.00mL of iron standard solution I. Respectively place in 50mL volumetric flasks. Use nitric acid solution (5.6) to dilute to the scale. Shake well. The iron content of this standard series is 0.00 mg/L, 1.00 mg/L, 2.00 mg/L, 4.00 mg/L, 6.00 mg/L, respectively. Under the best working conditions of the instrument, at a wavelength of 248.3nm, zero with reagent blank. Measure its absorbance. Take the measured absorbance as the ordinate, and the corresponding iron content (mg/L) as the abscissa, to draw a calibration curve or calculate a regression equation. The linear correlation coefficient of the calibration curve shall be greater than 0.999, otherwise it shall be redrawn.

9.2.2 Determination of water samples

Accurately pipette an appropriate amount of specimen solution (8.1). Put in a 50mL volumetric flask. Use nitric acid solution (5.6) to dilute to the scale. Shake well. According to the same instrument conditions in the preparation of the calibration curve, the reagent blank is used for zero adjustment. Measure its absorbance. Find out the corresponding iron content from the calibration curve. If the iron content is lower than the lower limit of detection or exceeds the range of the calibration curve, the dilution factor can be adjusted and re-measured.

9.2.3 Determination of scale samples

Respectively and accurately pipette appropriate amount of scale sample test solution (8.2.2 or 8.2.3) and dissolved sample blank solution (8.2.4). Place in 50mL volumetric flasks (choose the appropriate dilution factor according to the scale source and scale type. Generally, the scale sample mainly composed of calcium and magnesium scale is diluted by about 10 times to 100 times. The scale sample mainly composed of iron oxide scale is diluted by about 100 times to 500 times. Dissolved sample blank solution and scale test solution use the same dilution factor). Use nitric acid solution (5.6) to dilute to the scale. Shake well. According to the same instrument conditions in the preparation of the calibration curve, the reagent blank is used for zero adjustment. Measure the absorbance of the dissolved sample blank solution and the scaled sample solution after diluting to constant volume, respectively. Find out the corresponding iron content from the calibration curve. If the iron content is lower than the lower limit of detection or exceeds the range of the calibration curve, the dilution factor can be adjusted and re-measured.

9.3 Determination of zinc content

9.3.1 Drawing of zinc calibration curve

Accurately pipette 0.00mL (blank), 2.00mL, 4.00mL, 6.00mL, 8.00mL of zinc standard solution II. Place in 50mL volumetric flasks, respectively. Use nitric acid solution (5.6) to dilute to the scale. The zinc content of this standard series is 0.00mg/L, 0.20mg/L, 0.40mg/L, 0.60mg/L, 0.80mg/L. Under the optimal conditions of the instrument, at a wavelength of 213.9nm, zero with reagent blank. Measure its absorbance. Take the measured absorbance as the ordinate, and the corresponding zinc content (mg/L) as the abscissa, to draw a calibration curve or calculate a regression equation. The linear correlation coefficient of the calibration curve shall be greater than 0.999, otherwise it shall be redrawn.

9.3.2 Determination of water samples

Accurately pipette an appropriate amount of sample solution (8.1). Put it into a 50mL volumetric flask. Use nitric acid solution (5.6) to dilute to the scale. Shake well. According to the same instrument conditions in the preparation of the calibration curve, adjust the reagent blank to zero. Measure its absorbance. Find out the corresponding zinc content from the calibration curve. If the zinc content is lower than the lower limit of detection or exceeds the range of the calibration curve, the dilution factor can be adjusted and re-measured.

9.3.3 Determination of scale samples

Respectively and accurately pipette appropriate amount of scale sample solution (8.2.2 or 8.2.3) and dissolved sample blank solution (8.2.4). Place in 50mL volumetric flasks. Use nitric acid solution (5.6) to dilute to the scale. Shake well. According to the same instrument conditions in the preparation of the calibration curve, the reagent blank is used for zero adjustment. Measure the absorbance of the dissolved sample blank solution and the scale sample solution after diluting to constant volume, respectively. Find out the corresponding zinc content from the calibration curve. If the zinc content is lower than the lower limit of detection or exceeds the range of the calibration curve, the dilution factor can be adjusted and re-measured.

10 Result calculation

10.1 Calculation of copper, iron and zinc content in water samples

The content of copper, iron and zinc is calculated by the mass concentration ρ, in milligrams per liter (mg/L), calculated according to formula (1):

$$\rho = \rho_1 f \frac{V_1}{V} \qquad \cdots\cdots (1)$$

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