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GB/T 14637-2021

Replacing GB/T 14637-2007

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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1 Scope

GB/T 14637-2021 is the Chinese national standard on determination of copper,iron and zinc in industrial circulating cooling water and scale - atomic absorption spectrometric method, 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 20 August 2021 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 March 2022. 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 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

8.2.3 to prepare the scale sample test solution to prepare a dissolved sample blank solution.

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.