NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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Determination of calcium and magnesium in industrial circulating cooling water and scale - Atomic absorption spectrometric method

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1 Scope

This document specifies the determination method of the content of calcium and magnesium in industrial circulating cooling water and the content of calcium and magnesium in the scale of the boiler water system or circulating water system - atomic absorption spectrometry.

This document is applicable to the determination of calcium content of 0.5 mg/L~75 mg/L and magnesium content of 0.1 mg/L to 50 mg/L in industrial circulating cooling water; it is applicable to the determination of calcium content >=0.005% and magnesium content >=0.005% in scale.

This document is also applicable to the determination of calcium and magnesium content in the scale of various other industrial water, raw water, and water systems, as well as calcium content in phosphorus-zinc pre-film solution for industrial circulating refrigeration.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document;

for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 602 Chemical reagent -- Preparations of standard solutions for impurity

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

Terms and definitions defined in GB/T 4470 are applicable to this document.

4 Principles

The sample is sprayed into air-acetylene flame or nitrous oxide-acetylene flame by atomization, calcium and magnesium are pyrolyzed into ground state atoms; take calcium resonance line 422.7 nm and magnesium resonance line 285.2 nm as analytical lines to measure their absorbances, respectively. When using the air-acetylene flame to measure calcium and magnesium, adding strontium chloride or lanthanum chloride can inhibit the interference of water treatment chemicals and various coexisting elements in water (see Appendix A). When using the nitrous oxide-acetylene flame to measure calcium and magnesium, add cesium chloride to suppress interference.

5 Reagents or materials

Warning: The acetylene gas specified in this document is flammable and explosive when mixed with air; nitrous oxide is harmful to the human body; pay attention to safety in use, prevent leakage, and operate in strict accordance with the specifications. Strong acids are caustic, so pay attention when using them; handle them with care; if being splashed by them, rinse with plenty of water; avoid inhalation or skin contact.

Unless otherwise specified, the reagents used in this document are analytical grade only.

See GB 6819 for the requirements for acetylene gas used in the test. The standard solution for impurity used in the test shall be prepared in accordance with the provisions of GB/T 602 unless other provisions are specified.

5.7 Nitric acid solution: 1+99.

min~10 min before measurement can be performed.

8 Preparation of samples

8.1 Collection of water sample and preparation of sample solutions Collect a certain amount of water sample from the flowing water to be tested;

immediately, add the hydrochloric acid solution (5.5) to acidify the water sample to pH ~1 [generally, add 2.0 mL of hydrochloric acid solution (5.5) per 100 mL of water sample] to prepare a sample solution, and record the volume of the collected water sample and the volume of the added hydrochloric acid solution (5.5). The collected and acidified water sample shall be clear and transparent, otherwise, it shall be filtered with medium-speed quantitative filter paper. The sample solution is sealed and stored in the sampling container and can be placed stably for 14 days.

8.2 Preparation of scale sample and sample solutions

8.2.1 Collect and prepare scale sample according to the requirements of HG/T 3530 or DL/T 1151.2, and then dissolve the scale sample according to the steps of 8.2.2 or 8.2.3.

to prepare the scale sample solution.

8.2.2 Use the electric heating method to dissolve the scale sample, and operate according to the following requirements:

a) Weigh about 0.2 g~0.3 g of the prepared scale sample, and the weight shall be accurate to 0.1 mg; place it in a 250 mL glass beaker.

b) Add a small amount of water to the beaker to fully wet the scale sample; slowly add 15 mL of hydrochloric acid and 5 mL of nitric acid, cover with a watch glass, and shake well; slowly heat and boil the solution on an electric hot plate or adjustable electric furnace for 20 min. If there is still brown or tan residue, add

10 mL of hydrochloric acid and boil until the solution is clear.

c) Remove the beaker, add 10 mL of perchloric acid after the solution cools slightly, then heat until thick white smoke begins to emit; remove the watch glass slightly, and continue to slowly heat for 15 min~20 min; must not evaporate the solution to dryness.

d) Remove the beaker, add 10 mL of the nitric acid solution while it is still hot, and stir well to dissolve the salts on the wall of the beaker.

e) If the scale sample is completely dissolved, transfer the solution to a 100 mL volumetric flask; use a pipette to absorb the nitric acid solution to wash the inner wall of the beaker (no less than 3 times), and collect the washing solution in the 100 mL volumetric flask; add nitric acid solution to dilute to the Scale, and shake well; it is the scale sample solution. If

the dissolved solution contains white suspended solids, filter it with medium-speed quantitative filter paper; wash the beaker wall, the precipitation attached to the wall, and filter paper with the nitric acid solution (about 10 mL each time) several times; wash them no less than 5 times; collect the filtrate and the washing solution in a volumetric flask, add nitric acid solution to dilute to the Scale, and shake well; it is the scale sample solution.

8.2.3 Use microwave digestion to dissolve scale sample, and operate according to the following requirements:

a) Weigh 0.2 g~0.3 g of the prepared scale sample, and the weight shall be accurate to 0.1 mg; put it into the digestion inner tank of the microwave digestion apparatus, add a few drops of water to wet it, and slowly add 6 mL hydrochloric acid and 2 mL nitric acid; shake the digestion inner tank, and let the gas run out (if a large amount of gas is generated, the inner tank can be pre-digested at 100 °C for 20 min on the electric hot plate, and then add the same proportion of digestion solution to about 10 mL); cover the tank tightly and put it into the vessel. Put the digestion vessel into the microwave digestion apparatus, and digest the scale sample according to the instrument instructions.

b) After the digestion is completed, transfer the solution in the tank to a 250 mL glass beaker, wash the digestion tank and the lid with a small amount of nitric acid solution, and pour the washing solution into the beaker together; add 10 mL perchloric acid; place the beaker on an electric hot plate or adjustable electric furnace, and slowly heat until white smoke emits for 15 min~20 min; do not evaporate the solution to dryness; remove the beaker from the electric hot plate or adjustable electric furnace, add 10 mL of the nitric acid solution while it is still hot, and stir well to dissolve the salts on the wall of the beaker.

c) If the scale sample is completely dissolved, transfer the solution to a 100 mL volumetric flask, use a pipette to suck the nitric acid solution to wash the inner wall of the beaker (no less than 3 times), and collect the washing solution in the 100 mL volumetric flask; add nitric acid solution to dilute to the Scale, and shake well; it is the scale sample solution. If the dissolved solution contains white suspended solids, filter it with medium-speed quantitative filter paper; wash the beaker wall, the precipitate attached to the wall, and the filter paper with the nitric acid solution (about 10 mL each time) several times; wash them no less than 5 times; collect the filtrate and the washing solution in the volumetric flask, add nitric acid solution to dilute to the Scale, and shake well; it is the scale sample solution.

8.2.4 Except for not adding the scale sample, follow the same operation as preparation of the scale sample solution in 8.2.2 or 8.2.3 to prepare the blank solution.

zero-setting with the reagent blank, and respectively measure the absorbances of the blank solution and the scale sample solution after dilution; then, obtain the corresponding calcium content (mg/L) from the calcium calibration curve. If the calcium content is lower than the detection limit or exceeds the range of the calibration curve, the dilution factor can be adjusted and then re-measured.