NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB/T 41330-2022

Analysis of water used in boiler and cooling system - Determination of trace copper, iron, sodium, calcium and magnesium - Inductively coupled plasma mass spectrometry (ICP-MS)

Issued on: March 9, 2022 **Implemented on: October 1, 2022**

Issued by: SAMR; SAC

Table of Contents

1 Scope

This document describes the determination of trace copper, iron, sodium, calcium, and magnesium content in water used in boiler and cooling systems by inductively coupled plasma mass spectrometry.

This document is applicable to the determination of trace copper, iron, sodium, calcium, and magnesium content in water used in boiler and cooling systems. Measurement range: copper content 0.1 μg/L~1000 μg/L, iron content 5 μg/L~1000 μg/L, sodium content 10 μg/L~1000 μg/L, calcium content 10 μg/L~1000 μg/L, magnesium content 1 μg/L~ 1000 μg/L; when the content exceeds 1000 μg/L, it shall be diluted and then measured.

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 6041 General rules for mass spectrometric analysis

GB/T 6907 Analysis of water used in boiler and cooling system -- Sampling method of water

GB/T 33087-2016 Ultra pure water for instrumental analysis specification and test methods commercially available standard solutions.

5.9 Copper standard stock solution II: 100 mg/L.

5.10 Iron standard stock solution I: 1000 mg/L. Weigh 1.000 g of metallic iron, and the weight shall be accurate to 0.2 mg. Add it to 100 mL hydrochloric acid solution, and heat the solution until the metallic iron all dissolves; cool the solution and transfer it to a 1000 mL volumetric flask; dilute it with water to the mark. Or use commercially available standard solutions.

5.11 Iron standard stock solution II: 100 mg/L.

5.12 Sodium standard stock solution I: 1000 mg/L. Weigh 2.542 g standard sodium chloride that has been pre-burned to a constant amount at 500 °C~600 °C, and the weight shall be accurate to 0.2 mg; dissolve it in water and transfer to a 1000 mL volumetric flask; dilute with water to the mark, and shake well. Or use commercially available standard solutions.

5.13 Sodium standard stock solution II: 100 mg/L.

5.14 Calcium standard stock solution I: 1000 mg/L. Weigh 2.500 g of high-grade pure calcium carbonate that has been pre-dried at 105 °C~110 °C to a constant amount, and the weight shall be accurate to 0.2 mg. Place it in a 100 mL beaker, add 50 mL water and 10 mL hydrochloric acid, then dissolve it and transfer to a 1000 mL volumetric flask; dilute it with water to the mark, and shake well. Or use commercially available standard solutions.

5.15 Calcium standard stock solution II: 100 mg/L.

5.16 Magnesium standard stock solution I: 1000 mg/L. Weigh 1.660 g of premium pure magnesium oxide that has been pre-burned at 800 °C+/-50 °C to a constant amount, and the weight shall be accurate to 0.2 mg. Place it in a 100 mL beaker, add a small amount of water to wet the sample, and add 25 mL of hydrochloric acid; then, dissolve it and transfer it to a 1000 mL volumetric flask; dilute it to the mark with water, and shake well. Or use commercially available standard solutions.

5.17 Magnesium standard stock solution II: 100 mg/L.

5.18 Mixed standard stock solution: The certified mixed standard solution can be purchased; or use the standard stock solution I or II of each element to prepare the mixed standard stock by group according to the mutual interference between elements, the properties of the standard solution and the content of the element to be measured.

Then, store the solutions in sealed polyethylene or polypropylene bottles.

5.19 Mixed standard solution: 10 mg/L. Pipette 10.00 mL copper, iron, sodium, calcium, and

magnesium standard stock solution II, put it in a 100 mL volumetric flask, and dilute to the mark with a nitric acid solution (1+99); shake well. The solution shall be prepared just before being used.

5.20 Internal-standard standard stock solution: 10 mg/L or 1 mg/L, commercially available. 6Li, 45Sc, and 74Ge should be selected as internal standard elements. See

Appendix A for the selection of the internal standard corresponding to the mass number

of each element to be measured.

5.21 Internal-standard standard solution: According to the requirements of the instrument manual, dilute the internal standard stock solution with a nitric acid solution (1+99) to an appropriate concentration.

5.22 Mass spectrometer tuning solution: 1 μg/L or 10 μg/L; other concentrations recommended by the instrument manual can also be used. Standard solutions containing elements such as lithium (Li), yttrium (Y), beryllium (Be), magnesium (Mg), cobalt (Co), indium (In), thallium (Tl), lead (Pb), and bismuth (Bi) should be selected and used as the mass spectrometer tuning solutions.

5.23 Argon gas: The purity is not less than 99.999%.

5.24 Helium: The purity is not less than 99.999%.

5.25 Microporous filter membrane: cellulose acetate filter membrane with a pore size of 0.45 μm.

6 Instruments and equipment

6.1 Inductively coupled plasma mass spectrometer (ICP-MS) should be equipped with a collision/reaction cell.

6.2 Digestion equipment: a temperature-controlled electric hot plate or a microwave digestion apparatus or a graphite digestion apparatus.

6.3 Sampling bottle: The material shall be high-density polypropylene, high-density polyethylene, or fluorinated polyethylene propylene (FEP). The material of the bottle body and bottle cap shall not contain or leach any measured elements.

6.4 Beaker: The material shall be polytetrafluoroethylene, 100 mL or 250 mL.

6.5 Volumetric flask: The material shall be polypropylene, meltable polytetrafluoroethylene (PFA), or quartz.

7 Test steps

7.1 General

7.4.1 According to the content range of the element to be measured in the sample, select the appropriate calibration solution series.

7.4.2 Pipette an appropriate amount of mixed standard solution, place it in 8 volumetric flasks with a volume of 100 mL, and dilute it to the mark with a nitric acid solution (1+99); shake well to obtain the low concentration series calibration solutions, with the concentrations of 0.0 μg/L, 5.0 μg/L, 10.0 μg/L, 20.0 μg/L, 40.0 μg/L, 60.0 μg/L, 80.0 μg/L, and 100.0 μg/L.

7.4.3 Pipette an appropriate amount of mixed standard solution, place it in 8 volumetric flasks with a volume of 100 mL, and dilute it to the mark with a nitric acid solution (1+99); shake well, and obtain the high concentration series calibration solution, with the concentrations of 0 μg/L, 50 μg/L, 100 μg/L, 200 μg/L. L, 400 μg/L, 600 μg/L, 800 μg/L, and 1000 μg/L.

7.4.4 The internal-standard standard solution can be added directly to the calibration solution, or it can be added automatically by a peristaltic pump before the sample is nebulized.

7.4.5 The concentration range of the calibration solution can be adjusted according to the actual concentration of the element to be measured in the sample.

7.5 Determination

7.5.1 Instrument preparation After igniting the plasma, set the optimal working conditions according to the instrument manual. Use the mass spectrometer tuning solution to adjust the indicators of the instrument, such as the sensitivity, the oxide, the double charge, and the resolution;

after the requirements are met, inject the sample and determine it.

7.5.2 Plotting the calibration curve After the instrument meets the requirements, measure the calibration solution in turn.

Taking the ratio of the element signal to be measured to the internal-standard signal as the ordinate, and the mass concentration (μg/L) of the corresponding element as the abscissa, draw the calibration curve and calculate the regression equation; the correlation coefficient shall not be less than 0.995.

7.5.3 Sample determination The test conditions are the same as 7.5.1. Before the measurement of each sample, rinse the system with a nitric acid solution until the signal is stable. When the sample is measured, it shall be added with an internal standard solution, and the amount is the same as the calibration Standard curve. Each sample shall be measured twice in parallel, and take the arithmetic mean as the measurement result. If the content of the element to

10 Interference and cancellation