GB

NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 13.060.50;71.040.40

CCS G 76

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)

锅炉用水和冷却水分析方法 痕量铜、铁、钠、钙、镁含量的测定 电感耦合等离子体质谱（ICP-MS）法

(ISO 17294-2:2016, Water quality -- Application of inductively coupled plasma mass spectrometry (ICP-MS) -- Part 2: Determination of selected elements including uranium isotopes, NEQ)

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

Issued by: State Administration for Market Regulation;
Standardization Administration of PRC.

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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)

Warning -- The strong acids used in this document are corrosive, avoid inhalation or contact with skin. If being splashed, rinse immediately with plenty of water and seek medical attention immediately in severe cases.

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. ^{6}Li, ^{45}Sc, and ^{74}Ge 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

10.1 Isobaric interference: The elements with different atomic numbers and the same atomic mass have the same mass-to-charge ratio, which cannot be effectively resolved by a mass spectrometer. It may cause serious interference. It can be corrected by selecting the appropriate isotope of the element to be measured, or by using the calibration formula.

10.2 Interference of the more abundant isotopes on adjacent elements: The more abundant isotopes will produce tailing peaks, which will affect the measurement of adjacent mass peaks. The resolution of the mass spectrometer can be adjusted to reduce this interference.

10.3 Polyatomic (molecular) ion interference: The interference caused by a polyatomic ion consisting of two or three atoms and having the same mass-to-charge ratio as an element to be measured. The interference of polyatomic (molecular) ions is largely affected by the operating conditions of the instrument, which can be reduced by adjusting the instrument parameters.

10.4 Physical interference: It includes the interference caused by the difference in viscosity, surface tension, and total dissolved solids between the test sample and the standard solution. Physical interferences can be corrected with internal standard substances.

10.5 Matrix suppression (ionization interference): An increase in the concentration of easily ionizable elements will increase the number of electrons and cause a transition in plasma equilibrium; usually, it will reduce the analytical signal; the situation is called matrix suppression. Matrix interferences can be corrected with the internal standard method.

10.6 Memory interference: When samples or standards with large differences in concentration are analyzed continuously, the element to be measured in the sample is easy to deposit and to stay on the vacuum interface, spray chamber, and atomizer, which will cause memory interference. The increase in the time for rinsing the instrument between the tests of different samples will help to avoid such interferences.

11 Safety matters

11.1 An exhaust device shall be installed above the burner of the instrument.

11.2 Regularly check the pipeline to prevent gas leakage and strictly abide by the relevant operating procedures.

11.3 Argon and helium are inert gases, when using them, pay attention to ventilation to

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