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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 6913-2023

Analysis of water used in boiler and cooling system - Determination of phosphate

锅炉用水和冷却水分析方法 磷酸盐的测定

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Foreword

This document was issued on 17 March 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 October 2023.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 6913-2023 covers the measurement of phosphate in water used in boilers and cooling systems, and it separates three things a laboratory may be asked for: orthophosphate, total inorganic phosphate and total phosphate. Two methods are given. Clause 4 is the ammonium molybdate spectrophotometric method, whose summary describes orthophosphate reacting in an acidic medium in the presence of an antimony salt to give a coloured product read photometrically. Clause 5 is the continuous flow version of the same chemistry, for a laboratory running samples in series on an analyser. Clause 6 fixes what the test report carries. Appendix A, normative, collects precautions to observe during the test and opens with filtration of the water sample; Appendix B, informative, deals with judging and removing interference from coexisting ions; Appendix C, normative, sets the performance check for the continuous flow analyser system. Phosphate is dosed into boiler water deliberately, to keep hardness from setting as scale on heat transfer surfaces, so its concentration is a control parameter rather than a contaminant, and a figure biased by turbidity or by an interfering ion misdirects the dosing instead of simply being wrong. For power and industrial plant chemistry laboratories and water treatment service companies.

This document describes methods for the determination of orthophosphate, total inorganic phosphate, and total phosphate content in water used in boiler and cooling system: ammonium molybdate spectrophotometric method and continuous flow-ammonium molybdate spectrophotometric method.

This document applies to the determination of orthophosphate, total inorganic phosphate, and total phosphate content in water used in boiler and cooling system. The measurement range of the ammonium molybdate spectrophotometric method is 0.05 mg/L ~ 50 mg/L (based on PO43-); the measurement range of the continuous flow-ammonium molybdate spectrophotometric method is 0.1 mg/L ~ 50 mg/L (based on PO43-).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

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

3 Terms and definitions

No terms and definitions need to be defined in this document.

4 Ammonium molybdate spectrophotometric method

4.1 Determination of orthophosphate content

4.1.1 Method summary

In the presence of acidic media and antimonic salts, orthophosphate reacts with ammonium molybdate solution to produce yellow phosphomolybdenum heteropoly acid. Use ascorbic acid to reduce it to phosphomolybdenum blue, and measure it by the spectrophotometric method at a wavelength of 710 nm.

The reaction formula is:

4.1.2 Reagents or materials

Unless otherwise specified, only use analytical reagents or above.

4.1.2.1 Water: GB/T 6682-2008, grade 3.

4.1.2.2 Ascorbic acid solution: 100 g/L. Store it in brown bottles at 4 °C, valid for 14 days.

4.1.2.3 Ammonium molybdate solution: 26 g/L. Weigh 13 g of ammonium molybdate and 0.35 g of potassium antimony tartrate (KSbOC4H4O6.1/2H2O); dissolve in 200 mL of water; add 230 mL of sulfuric acid solution (1+1); mix well; cool and use water to dilute to 500 mL; shake well. Store it in brown bottles, valid for 2 months.

4.1.2.4 Phosphate standard stock solution (based on PO43-): 500 mg/L. Weigh 0.716 5 g of potassium dihydrogen phosphate that has been dried to a constant weight at 100 °C ~ 105 °C, accurate to 0.2 mg; dissolve in about 500 mL of water; transfer to a 1 000 mL volumetric flask; use water to dilute to the mark; shake well.

4.1.2.5 Phosphate standard solution (based on PO43-): 20 mg/L. Pipette 20.00 mL of phosphate standard stock solution into a 500 mL volumetric flask; use water to dilute to the mark; shake well. Formulate this solution when needed.

4.1.3 Instruments and apparatuses

Spectrophotometer: Equipped with an absorption cell with a thickness of 1 cm.

4.1.4 Test procedure

4.1.4.1 Preparation of sample

Filter the water sample collected on site through medium-speed filter paper and collect into clean and dry glass bottles for subsequent analysis. Operate things to note during the test according to Appendix A. See Appendix B for the interference judgment and elimination of coexisting ions in the sample.

4.3.2.3 Potassium persulfate solution: 40 g/L. Store in brown bottles, valid for 14 days.

4.3.2.4 Other reagents: Same as 4.1.2.

4.3.3 Instruments and apparatuses

Spectrophotometer: Equipped with an absorption cell with a thickness of 1 cm.

4.3.4 Test procedure

Transfer an appropriate volume of sample (4.1.4.1) to a 100 mL Erlenmeyer flask by referring to Table 1; add water to approximately 40 mL. Add 1.0 mL of sulfuric acid solution and 5.0 mL of potassium persulfate solution; place it on an electric furnace to maintain a slight boil for about 30 minutes for digestion. When boiling, add water regularly to keep the solution volume at 25 mL ~ 30 mL. After cooling, use sodium hydroxide solution to adjust the pH to 3 ~ 10 and transfer to a 50 mL volumetric flask. Add 2.0 mL of ammonium molybdate solution and 1.0 mL of ascorbic acid solution; use water to dilute to the mark; shake well; leave at room temperature for 10 minutes. Use a spectrophotometer to measure the absorbance at a wavelength of 710 nm with a 1 cm absorption cell and a blank as a reference.

Note: Other digestion methods, such as microwave digestion, can also be used.

4.3.5 Result calculation

4.3.5.1 The total phosphate (based on PO43-) content rho3 is expressed in terms of mass concentration, in milligrams per liter (mg/L), and is calculated according to Formula (3):

Where:

m - the mass of PO43- found from the calibration curve (4.1.4.2) or calculated from the regression equation, in micrograms (ug);

V - the volume of the sample transferred, in milliliters (mL).

Express the calculation result to two digits after the decimal point.

4.3.5.2 The organophosphate (based on PO43-) content rho4 is expressed in terms of mass

concentration, in milligrams per liter (mg/L), and is calculated according to Formula (4):

Adjust the instrument according to the instrument instruction manual to achieve optimal usage conditions. After starting up, first replace the reagent with water to check the tightness of the entire analysis flow path and the smoothness of the liquid flow. After the baseline stabilizes (about 20 minutes), the system begins to take the reagent package.