CCS Z16

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 11893-1989

Water quality--Determination of total phosphorus--Ammonium molybdate spectrophotometric method

水质 总磷的测定 钼酸铵分光光度法

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1 Subject content and scope of application

GB/T 11893-1989 is the Chinese national standard on water quality--determination of total phosphorus--ammonium molybdate spectrophotometric method. 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 3 November 1989 by the State Bureau of Technical Supervision, and has been in force since 1 July 1990. Classification: ICS (ICS) , CCS Z16. 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 standard specifies the method of using potassium persulfate (or nitric acid- perchloric acid) as the oxidant to digest the unfiltered water sample and using ammonium molybdate to determine the total phosphorus with spectrophotometry. Total phosphorus includes dissolved, particulate, organic and inorganic phosphorus. This standard applies to surface water, sewage, and industrial wastewater. Take 25 mL sample; the minimum detectable

concentration of this standard is

0.01 mg/L, and the upper limit of determination is

0.6 mg/L. Under acidic conditions, arsenic, chromium, and sulfur interfere with the determination.

2 Principles

Under neutral conditions, the sample is digested with potassium persulfate (or nitric acid-perchloric acid), and all the phosphorus contained is oxidized to orthophosphate. In an acidic medium, orthophosphate reacts with ammonium molybdate to form phosphomolybdic heteropoly acid in the presence of antimony salt, which is immediately reduced by ascorbic acid to form a blue complex.

3 Reagents

Unless otherwise specified, the reagents used in this standard shall be analytical reagents and distilled water (or water of equivalent purity) that meet national or professional standards.

3.2 Nitric acid (HNO3) with a density of

3.3 Perchloric acid (HClO4) shall be a guaranteed reagent, and the density shall be

3.4 Sulfuric acid (H2SO4). 1+1.

3.5 Sulfuric acid, about c (H2SO4)=1 mol/L. Add 27 mL of sulfuric acid (3.1) to 973 mL of water.

3.11 Turbidity-chromaticity compensation solution. Mix two volumes of sulfuric acid (3.4) and one volume of ascorbic acid solution (3.9). Prepare it on the day of use.

3.12 Phosphorus standard stock solution. Weigh 0.2197±

0.001 g of potassium dihydrogen phosphate (KH2PO4) that was dried at 110 °C for 2 h and cooled in a desiccator; dissolve it in water and transfer to a 1000 mL volumetric flask; add about 800 mL of water, add 5 mL of sulfuric acid (3.4), dilute with water to the mark, and mix well.

1.00 mL of this standard solution contains 50.0 μg of phosphorus. This solution can be stored in glass bottles for at least six months.

3.14 Phenolphthalein, 10 g/L solution. Dissolve

0.5 g of phenolphthalein in 50 mL of 95% ethanol.

4 Instruments

Common laboratory equipment and the following instruments.

4.1 Medical portable steam sterilizer or general pressure cooker (1.1~

1.4 kg/cm2). 4.2 50 mL graduated tube with a ground-mouth stopper.

5 Sampling and samples

5.1 Take 500 mL of water sample, and add 1 mL of sulfuric acid (3.1) to adjust the pH value of the sample to be lower than or equal to 1; or do not add any reagents and store it in a cold place.

5.2 Preparation of samples. Take 25 mL of the sample (5.1) and put it in a stoppered graduated tube (4.2). When taking the sample, carefully shake it well to obtain a sample, in which both the dissolved part and the suspended part are representative.

6 Analysis steps

6.1 Blank sample Carry out a blank test according to the provisions of (6.2), replace the sample with water, and add the same volume of reagents as that during the determination.

6.2.1 Digestion

6.2.2 Color reaction Add 1 mL of ascorbic acid solution (3.9) to each part of the digestion solution and mix well; after 30 s, add 2 mL of molybdate solution (3.10) and mix well.

6.2.3 Spectrophotometry After the sample is standing at room temperature for 15 min, use a cuvette with an optical path of 30 mm to measure the absorbance at a wavelength of 700 nm with water as a reference. After deducting the absorbance of the blank test, check the phosphorus content from the working curve (6.2.4).

6.2.4 Drawing of a working curve Take 7 stoppered graduated tubes (4.2) and add

0.0 mL,

0.50 mL,

15.0 mL of phosphate standard solution (3.14) respectively. Add water to 25 mL. Then carry out the determination according to the determination steps of (6.2). Use water as a reference to measure the absorbance. After deducting the absorbance of the blank test, draw a working curve with the corresponding phosphorus content.

7 Representation of results

The total phosphorus content is expressed in C (mg/L) and calculated according to the following formula.

8 Precision and accuracy

8.1 Thirteen laboratories determine (with the digestion method of 6.2.1.1) unified samples with a phosphorus content of

8.2 Six laboratories determine (with the digestion method of 6.2.1.2) unified samples with a phosphorus content of

8.2.1 Repeatability The relative standard deviation within a laboratory shall be 1.4%.