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**Testing methods for water quality by flow
analysis—Part 4: Orthophosphate and
total phosphorus**

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal for establishment of Japanese Industrial Standard submitted by Japan Environmental Measurement and Chemical Analysis (JEMCA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

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JIS K 0170 series consists of the following 9 parts under the general title

“Testing methods for water quality by flow analysis”:

Part 1: Ammonium nitrogen

Part 2: Nitrite nitrogen and nitrate nitrogen

Part 3: Total nitrogen

Part 4: Orthophosphate and total phosphorus

Part 5: Phenol index

Part 6: Fluoride

Part 7: Chromium (VI)

Part 8: Anionic surfactants

Part 9: Cyanide compounds

Testing methods for water quality by flow analysis—

Part 4: Orthophosphate and total phosphorus

Introduction

This Japanese Industrial Standard has been prepared based on the first editions of ISO 15681-1 and ISO 15681-2 published in 2003, with some of the technical contents modified so as to make them applicable to apparatuses, etc. used in Japan.

The portions with continuous sidelines or dotted underlines are the matters in which the contents of the corresponding International Standard have been modified. A list of modifications with explanations is given in Annex JA.

1 Scope

This Standard specifies the method for determination of orthophosphate contained in industrial water, industrial wastewater, etc. by using the flow analysis by molybdenum blue absorption spectrophotometry and the method for determination of total phosphorus by the flow analysis including the pretreatment in which phosphorus compounds in the sample are oxidized to orthophosphate by the heat oxidation-decomposition using peroxodisulfate. The method is also applicable to surface water, ground water, seepage water, etc.

It does not apply to samples with much suspended matter.

NOTE: The International Standards corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 15681-1:2003 *Water quality—Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA)—Part 1: Method by flow injection analysis (FIA)*

ISO 15681-2:2003 *Water quality—Determination of orthophosphate and total phosphorus contents by flow analysis (FIA and CFA)—Part 2: Method by continuous flow analysis (CFA)* (Overall evaluation: MOD)

The symbols which denote the degree of correspondence in the contents between the relevant International Standard and JIS are IDT (identical), MOD (modified), and NEQ (not equivalent) according to ISO/IEC Guide 21-1.

WARNING The reagents shall be handled with utmost attention, ensuring compliance with the relevant laws, ordinances, regulations and the like.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS K 0050	<i>General rules for chemical analysis</i>
JIS K 0094	<i>Sampling methods for industrial water and industrial wastewater</i>
JIS K 0101	<i>Testing methods for industrial water</i>
JIS K 0102	<i>Testing methods for industrial wastewater</i>
JIS K 0115	<i>General rules for molecular absorptiometric analysis</i>
JIS K 0126	<i>General rules for flow analysis</i>
JIS K 0211	<i>Technical terms for analytical chemistry (General part)</i>
JIS K 0215	<i>Technical terms for analytical chemistry (Analytical instrument part)</i>
JIS K 0557	<i>Water used for industrial water and wastewater analysis</i>
JIS K 8150	<i>Sodium chloride (Reagent)</i>
JIS K 8253	<i>Potassium peroxodisulfate (Reagent)</i>
JIS K 8533	<i>Bis [(+)-tartrato] diantimonate (III) dipotassium trihydrate (Reagent)</i>
JIS K 8576	<i>Sodium hydroxide (Reagent)</i>
JIS K 8905	<i>Hexaammonium heptamolybdate tetrahydrate (Reagent)</i>
JIS K 8951	<i>Sulfuric acid (Reagent)</i>
JIS K 9007	<i>Potassium dihydrogen phosphate (Reagent)</i>
JIS K 9502	<i>L(+)-Ascorbic acid (Reagent)</i>
JIS R 3503	<i>Glass apparatus for chemical analysis</i>
JIS R 3505	<i>Volumetric glassware</i>

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in JIS K 0101, JIS K 0102, JIS K 0115, JIS K 0126, JIS K 0211 and JIS K 0215 and the following apply.

3.1 total phosphorus

the expression based on the concentration of phosphorous of orthophosphate among phosphorous compounds determined by molybdenum blue absorption spectrophotometry after pretreatment according to the procedure in clause 7 in which the organic matter, etc. in the sample is oxidation-decomposed

4 Common matters

4.1 General

The general matters common to the chemical analyses shall be in accordance with JIS K 0050. The general requirements relating to the flow analysis shall be in accordance with JIS K 0126.

4.2 Water

The water used in this Standard shall be the water of A3 or A4 specified in JIS K 0557.

4.3 Reagents

The reagents to be used shall be those specified in Japanese Industrial Standards and shall not be such that they impair the measurement. In the case of using reagents not specified in Japanese Industrial Standards, those reagents shall not be such that they impair the measurement. The reagent blank shall be periodically checked.

NOTE: The concentration indicated in parentheses at the rear of the designation of a reagent solution, except when it is a reference solution, is an approximate concentration.

4.4 Glassware

The glassware to be used, unless otherwise specified, shall be in accordance with JIS R 3503 and JIS R 3505.

5 Sample

5.1 Sampling

The sampling, sample container, water sampler and sampling operation shall be in accordance with JIS K 0094. For the sample container, a polyethylene bottle with ground-in stopper and a glass bottle with ground-in stopper shall be used.

A polypropylene bottle with ground-in stopper, a polystyrene bottle with ground-in stopper or a polycarbonate bottle with ground-in stopper may be used.

5.2 Handling of sample

The test shall be carried out immediately after sampling. When this is not practicable, the sample shall be preserved according to 3.3 b) 8) of JIS K 0102 and tested as soon as possible.

6 Measurement of orthophosphate

6.1 Principle

In the flow injection analysis (hereafter referred to as the "FIA"), the sample is injected in the carrier solution flowing continuously in a capillary, and the sulfuric acid-ammonium molybdate solution containing potassium tartrate antimonate (III) solution and ascorbic acid solution flowing continuously in a capillary are mixed with it in the capillary. The heteropoly compound produced by the reaction of orthophosphate in the sample with molybdic acid is reduced by ascorbic acid, and the absorbance of resultant molybdenum blue is measured at a wavelength in the vicinity of 880 nm to determine orthophosphate.

In the continuous flow analysis (hereafter referred to as the "CFA"), air is injected into the continuous flow of sample in a capillary at constant intervals to segment the flow. The sulfuric acid-ammonium molybdate solution containing potassium tartrate antimonate (III) solution and ascorbic acid solution flowing continuously in the capil-

lary are mixed with the said flow of sample in the capillary, and the absorbance of resultant molybdenum blue in the capillary is measured at a wavelength in the vicinity of 880 nm to determine orthophosphate.

Range of determination P: 0.01 mg/L to 1 mg/L

NOTE: Though it differs depending on the apparatus and measuring conditions, repeatability of this method, expressed by relative standard deviation (%), is not more than 10 %.

6.2 Interferences

Arsenate ion, nitrite ion and oxidizing substance will interfere with the determination. For further details regarding interferences, see JIS K 0102.

6.3 Classification of measuring methods and reagents and apparatus

6.3.1 Classification of measuring methods

Classification of measuring methods shall be in accordance with table 1.

Table 1 Classification of measuring methods

Classification of measuring method	Clause					
	Reagent and preparation of reagent solution	Apparatus	Measuring operation	Calculation of concentration	Expression of results	Test report
Three-flow channel type FIA method of molybdenum blue colouring	6.3.2.1	6.3.2.2	6.4	6.5	6.6	8
<u>Two-flow channel type FIA method of molybdenum blue colouring</u>	<u>6.3.3.1</u>	<u>6.3.3.2</u>				
CFA method of molybdenum blue colouring	6.3.4.1	6.3.4.2				

6.3.2 Three-flow channel type FIA method of molybdenum blue colouring

6.3.2.1 Preparation of reagents and reagent solutions

6.3.2.1.1 Reagents

The reagents shall be as follows.

- Potassium dihydrogen phosphate, for pH reference solution use, specified in JIS K 9007.
- Hexaammonium heptamolybdate tetrahydrate, specified in JIS K 8905.
- Bis [(+)-tartrato] diantimonate (III) dipotassium trihydrate, specified in JIS K 8533.
- Sulfuric acid, specified in JIS K 8951.

- e) **L (+)-Ascorbic acid**, specified in JIS K 9502.
- f) **Sodium dodecyl sulfate**

6.3.2.1.2 Preparation of reagent solutions

The reagent solutions shall be prepared as follows. The prepared solution shall be degassed as necessary.

- a) **Ammonium molybdate solution** Add 40 g of hexaammonium heptamolybdate tetrahydrate in approximately 800 ml of water to dissolve, then add water to make 1 000 ml.
- b) **Potassium tartrate antimonate (III) solution** Add 3.0 g of bis[(+)-tartrato] dian-timonate (III) dipotassium trihydrate in approximately 800 ml of water to dissolve, then add water to make 1 000 ml.
- c) **Sulfuric acid (0.63 mol/L)-ammonium molybdate solution** Take approximately 500 ml of water in a beaker, and while stirring, add gradually 35 ml of sulfuric acid. After cooling, add 213 ml of ammonium molybdate solution and 72 ml of potassium tartrate antimonate (III) solution and add water to make the total volume 1 000 ml.
- d) **Ascorbic acid solution (60 g/L)** Add 6.0 g of L(+)-ascorbic acid in approximately 80 ml of water to dissolve, then add 0.1 g of sodium dodecyl sulfate and add water to make the total volume 100 ml. Prepare this solution freshly before use.
- e) **Carrier solution** Water is used.
- f) **Orthophosphate reference solution (P: 50 mg/L)** Heat potassium dihydrogen phosphate at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 2 h and leave it to cool in a desiccator. After dissolving 220 mg of this in water, transfer it into a 1 000 ml volumetric flask and make up to the mark with water. Preserve this solution in a cool and dark place.
- g) **Orthophosphate reference solution (P: 10 mg/L)** Take 20 ml of orthophosphate reference solution (P: 50 mg/L) in a 100 ml volumetric flask and make up to the mark with water. Prepare this solution freshly before use.
- h) **Phosphate reference solution (P: 1.0 mg/L)** Take 2 ml of orthophosphate reference solution (P: 50 mg/L) in a 100 ml volumetric flask and make up to the mark with water. Prepare this solution freshly before use.

NOTE: As an example of orthophosphate solution products having traceability, those bearing the mark of JCSS (Japan Calibration Service System) are traceable to the Measurement Law.

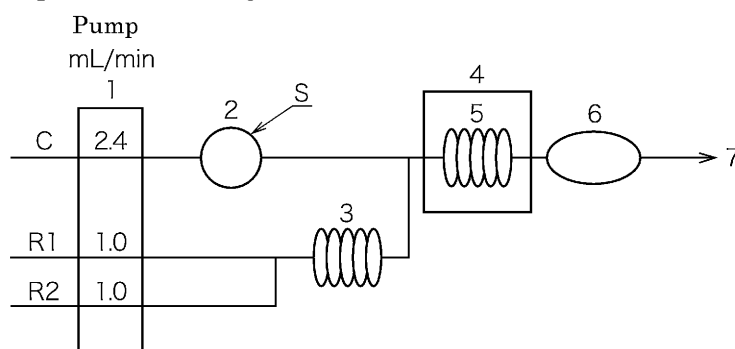
6.3.2.2 Apparatus

In general, the system comprises the following components (see figure 1).

- a) **Liquid-sending part** A low pulsation pump shall be used.
- b) **Sample introduction part** A six-way selector valve shall be usually used. An appropriate injection volume shall be selected. If required, an automated sample in-

roduction device may be used.

- c) **Reaction part**, consisting of fluororesin, e.g. polytetrafluoroethylene tube having an internal diameter of 0.5 mm to 0.8 mm, tube joints of inert plastic and with minimum dead volumes and a thermostat capable of heating and maintaining at a specified temperature.
- d) **Detection part** A spectrophotometric detector capable of measuring at a wavelength in the vicinity of 880 nm.
- e) **Recording part**, capable of recording the signals from the detector.
- f) **Thermostat**, capable of heating to 60 °C.



C : Carrier solution

R1 : Sulfuric acid (0.63 mol/L)-ammonium molybdate solution

R2 : Ascorbic acid solution (60 g/L)

S : Sample

1 : Pump

2 : Sample introducer [injection volume 640 μ L (orthophosphate concentration 0.01 mg/L to 0.10 mg/L), injection volume 100 μ L (orthophosphate concentration 0.10 mg/L to 1.00 mg/L)]

3 : Reaction coil (internal diameter 0.7 mm, length 120 cm)

4 : Thermostat (60 °C)

5 : Reaction coil (internal diameter 0.7 mm, length 150 cm)

6 : Detector wavelength 880 nm

7 : Waste liquid

Figure 1 Example of a system for three-flow channel type FIA method of molybdenum blue colouring

6.3.3 Two-flow channel type FIA method of molybdenum blue colouring

6.3.3.1 Preparation of reagents and reagent solutions

6.3.3.1.1 Reagents

Reagents shall be as follows.

- a) **Potassium dihydrogen phosphate**, in accordance with 6.3.2.1.1 a).
- b) **Hexaammonium heptamolybdate tetrahydrate**, in accordance with 6.3.2.1.1 b).
- c) **Bis [(+)-tartrato] diantimonate (III) dipotassium trihydrate**, in accordance with 6.3.2.1.1 c).

- d) **Sulfuric acid**, in accordance with 6.3.2.1.1 d).
- e) **L (+)-Ascorbic acid**, in accordance with 6.3.2.1.1 e).
- f) **Sodium dodecyl sulfate**, in accordance with 6.3.2.1.1 f).

6.3.3.1.2 Preparation of reagent solutions

The reagent solutions shall be prepared as follows. The prepared solution shall be degassed as necessary.

- a) **Sulfuric acid (1.21 mol/L)-ammonium molybdate solution** Take approximately 800 ml of water in a beaker, and while stirring, add gradually 67 ml of sulfuric acid. Add to this 5.48 g of hexaammonium heptamolybdate tetrahydrate and 0.25 g of bis [(+)-tartrato] diantimonate (III) dipotassium trihydrate and dissolve, and add water to make 1 000 ml.
- b) **Ascorbic acid solution (3 g/L)** Dissolve 3.0 g of L(+)-ascorbic acid and 1 g of sodium dodecyl sulfate in 1 000 ml of water. Prepare this solution freshly before use.
- c) **Colouring reagent solution** Mix sulfuric acid (1.21 mol/L)-ammonium molybdate solution and ascorbic acid solution (3 g/L) in equal amount. Prepare this solution freshly before use.
- d) **Carrier solution**, in accordance with 6.3.2.1.2 e).
- e) **Orthophosphate reference solution (P: 50 mg/L)**, in accordance with 6.3.2.1.2 f).
- f) **Orthophosphate reference solution (P: 10 mg/L)**, in accordance with 6.3.2.1.2 g).
- g) **Orthophosphate reference solution (P: 1.0 mg/L)**, in accordance with 6.3.2.1.2 h).

6.3.3.2 Apparatus

In general, the system comprises the components shown in 6.3.2.2, except that the thermostat shall be as follows (see figure 2).

- a) **Thermostat**, capable of heating to temperature around 70 °C.