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(JEMCA/JSA)

**Testing methods for water quality by flow
analysis—Part 2: Nitrite nitrogen and
nitrate nitrogen**

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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 2: Nitrite nitrogen and nitrate nitrogen

Introduction

This Japanese Industrial Standard has been prepared based on the first edition of ISO 13395 published in 1996 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 of determination for the nitrite nitrogen and nitrate nitrogen contained in industrial water, industrial wastewater, etc. by using the flow analysis according to absorption spectrophotometry. 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 Standard corresponding to this Standard and the symbol of degree of correspondence are as follows:

ISO 13395:1996 *Water quality—Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analysis (CFA and FIA) and spectrometric detection* (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 *General rules for chemical analysis*

JIS K 0094 *Sampling methods for industrial water and industrial wastewater*

JIS K 0101 *Testing methods for industrial water*

JIS K 0102 *Testing methods for industrial wastewater*

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 8019	<i>Sodium nitrite (Reagent)</i>
JIS K 8102	<i>Ethanol (95) (Reagent)</i>
JIS K 8107	<i>Disodium dihydrogen ethylenediamine tetraacetate dihydrate (Reagent)</i>
JIS K 8116	<i>Ammonium chloride (Reagent)</i>
JIS K 8180	<i>Hydrochloric acid (Reagent)</i>
JIS K 8197	<i>N-1-Naphthylethylenediamine dihydrochloride (Reagent)</i>
JIS K 8548	<i>Potassium nitrate (Reagent)</i>
JIS K 8576	<i>Sodium hydroxide (Reagent)</i>
JIS K 8951	<i>Sulfuric acid (Reagent)</i>
JIS K 8983	<i>Copper (II) sulfate pentahydrate (Reagent)</i>
JIS K 9005	<i>Phosphoric acid (Reagent)</i>
JIS K 9066	<i>Sulfanilamide (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 apply.

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 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) 3) of JIS K 0102 and tested as early as possible.

6 Measurement of nitrite nitrogen

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. In the continuous flow analysis (hereafter referred to as the "CFA"), the sample and buffer solution are continuously mixed in a capillary. This flow is mixed and made to react with the flow of colouring reagent solution containing sulfanilamide and *N*-1-naphthylethylenediamine dihydrochloride. The absorbance of the red azo compound generated as a result of diazo coupling reaction is measured at a wavelength in the vicinity of 540 nm to determine nitrite ion.

Range of determination N: 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

Suspended matter, if present in the sample, may lead to clogging of the transport tubes and will interfere with the photometric measurement. They shall be removed by membrane filtration or dialysis.

The coexistence of surfactant and high polymer organic matter will interfere with determination. They shall be removed by dialysis or activated carbon treatment.

The colouring of sample causes a positive error. It shall be corrected by measuring the absorbance of sample.

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
FIA method of phosphoric acid-naphthylethylenediamine colouring	6.3.2.1	6.3.2.2	8	8.6	9	10
FIA method of hydrochloric acid-naphthylethylenediamine colouring	6.3.3.1	6.3.3.2				
CFA method of phosphoric acid-naphthylethylenediamine colouring	6.3.4.1	6.3.4.2				
CFA method of hydrochloric acid-naphthylethylenediamine colouring	6.3.5.1	6.3.5.2				

6.3.2 FIA method of phosphoric acid-naphthylethylenediamine colouring

6.3.2.1 Preparation of reagents and reagent solutions

6.3.2.1.1 Reagents

The reagents shall be as follows.

- Sulfanilamide**, specified in JIS K 9066.
- N-1-Naphthylethylenediamine dihydrochloride**, specified in JIS K 8197.
- Phosphoric acid**, specified in JIS K 9005.
- Sodium nitrite**, specified in JIS K 8019.

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) **Carrier solution** Water is used.
- b) **Sulfanilamide • *N*-1-naphthylethylenediamine solution** Dissolve 5 g of sulfanilamide and 0.5 g of *N*-1-naphthylethylenediamine dihydrochloride in water. Add to this solution 50 ml of phosphoric acid, and add water to make 500 ml. Preserve this solution in a coloured glass bottle in a cool and dark place.

NOTE: Sulfanilamide solution and *N*-1-naphthylethylenediamine dihydrochloride solution may be separately prepared and their flow channel separated.

- c) **Nitrite nitrogen reference solution (N: 100 mg/L)** Dry sodium nitrite at 105 °C to 110 °C for about 4 h and leave it to cool in a desiccator. Obtain the purity of the sodium nitrite according to JIS K 8019, take sodium nitrite corresponding to 0.4926 g to 100 % NaNO₂, dissolve it in water, transfer it to a 1 000 ml volumetric flask and make up to the mark with water. Preserve this reference solution in a cool and dark place.
- d) **Nitrite nitrogen reference solution (N: 20 mg/L)** Take correctly 20 ml of nitrite nitrogen reference solution (N 100 mg/L) in a 100 ml volumetric flask and make up to the mark with water. Prepare this solution freshly before use.
- e) **Nitrite nitrogen reference solution (N: 1 mg/L)** Take correctly 1 ml of nitrite nitrogen reference solution (N: 100 mg/L) in a 100 ml volumetric flask and make up to the mark with water. Prepare this solution freshly before use.

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 introduction device may be used.

- c) **Reaction part**, consisting of fluoro-resin e.g. polytetrafluoroethylene tube having an internal diameter of 0.5 mm to 0.8 mm and tube joints of inert plastic and with minimum dead volumes.
- d) **Detection part** A spectrophotometric detector capable of measuring at a wavelength of 520 nm to 560 nm shall be used.
- e) **Recording part**, capable of recording the signals from the detector.

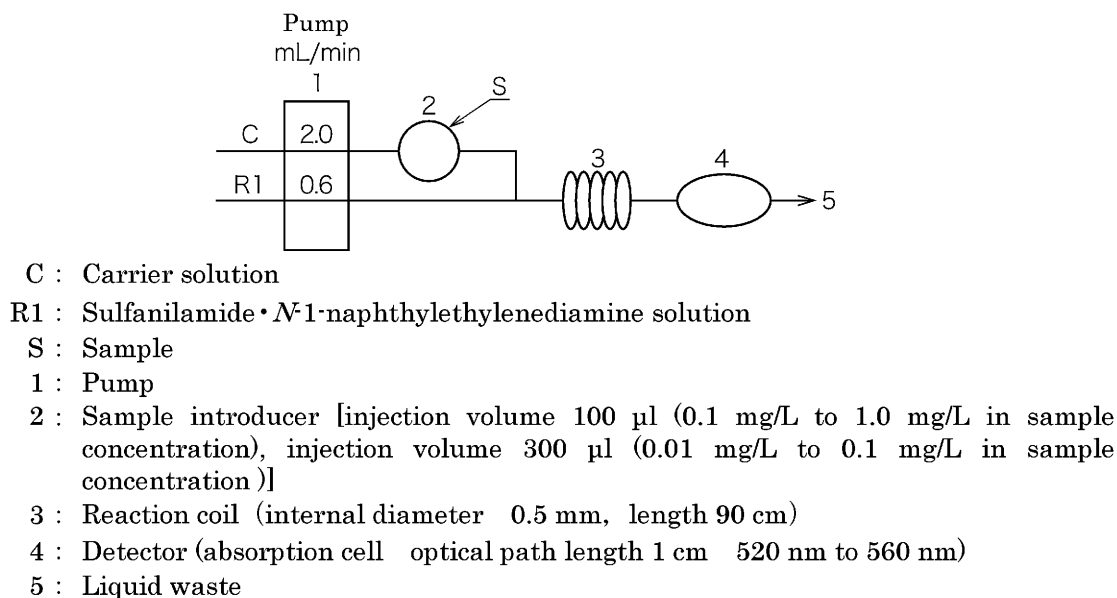


Figure 1 Example of a system for FIA method of hydrochloric acid-naphthylethylenediamine colouring

6.3.3 FIA method of hydrochloric acid-naphthylethylenediamine colouring

6.3.3.1 Preparation of reagents and reagent solutions

6.3.3.1.1 Reagents

Reagents shall be as follows.

- Sulfanilamide**, in accordance with 6.3.2.1.1 a).
- N*-1-Naphthylethylenediamine dihydrochloride**, in accordance with 6.3.2.1.1 b).
- Hydrochloric acid**, specified in JIS K 8180.
- Sodium nitrite**, in accordance with 6.3.2.1.1 d).

6.3.3.1.2 Preparation of reagent solutions

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

- Carrier solution** Water is used.
- Sulfanilamide solution** Dissolve 20 g of sulfanilamide in mixed solution of 100 ml of hydrochloric acid and approximately 800 ml of water, and add water to make 1 000 ml. Preserve this solution in a cool and dark place.
- N*-1-Naphthylethylenediamine dihydrochloride solution** Dissolve 2 g of *N*-1-naphthylethylenediamine dihydrochloride in mixed solution of 100 ml of hydrochloric acid and approximately 800 ml of water, and add water to make 1 000 ml. Preserve this solution in a cool and dark place.
- Sulfanilamide • *N*-1-naphthylethylenediamine solution** Mix 50 ml of sulfanilamide solution and 50 ml of *N*-1-naphthylethylenediamine dihydrochloride solu-