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**Testing methods for perfluorooctane-
sulfonate (PFOS) and perfluorooctanoate
(PFOA) in industrial water and wastewater**

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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 National Institute of Advanced Industrial Science and Technology (AIST)/ Japan Environmental Management Association for Industry (JEMAI) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

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Testing methods for perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) in industrial water and wastewater

Introduction

This Japanese Industrial Standard has been prepared based on the first edition of ISO 25101 published in 2009 with some modifications of the technical contents made so as to be applicable to industrial water and wastewater.

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 the explanations is given in Annex JD. Annex JA to Annex JC contain matters that are not given in the corresponding International Standard.

1 Scope

This Standard specifies the methods for determination of the linear-chain isomers of 1, 1, 2, 2, 3, 3, 4, 4, 5, 5, 6, 6, 7, 7, 8, 8, 8-heptadecafluorooctanesulfonate (perfluorooctanesulfonate PFOS) and 2, 2, 3, 3, 4, 4, 5, 5, 6, 6, 7, 7, 8, 8, 8-pentadecafluorooctanoate (perfluorooctanoate, PFOA) in industrial water and wastewater by using the solid-phase column and high performance liquid chromatograph tandem mass spectrometer (LC/MS/MS meter) (see table 1).

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

ISO 25101 : 2009 *Water quality — Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) — Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry* (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**.

Table 1 PFOS and PFOA

	Formula	Abbreviation	CAS ^{a)} -No
Perfluorooctanesulfonate	CF ₃ (CF ₂) ₇ SO ₃ H	PFOS	1763-23-1
Perfluorooctanoate	CF ₃ (CF ₂) ₆ COOH	PFOA	335-67-1
NOTE 1 Perfluorooctanesulfonate and perfluorooctanoate mean the linear-chain type, however, these designations can include branched type. In this Standard, the analyte is the linear-chain type.			
NOTE 2 PFOA includes the acids and its salts.			
Note ^{a)} CAS : Chemical Abstract Service			

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 0136 *General rules for high performance liquid chromatography / mass spectrometry*

JIS K 0211 *Technical terms for analytical chemistry (General part)*

JIS K 0215 *Technical terms for analytical chemistry (Analytical instrument part)*

JIS K 0557 *Water used for industrial water and wastewater analysis*

JIS K 1107 *Nitrogen*

JIS K 8039 *Acetonitrile for pesticide residue and polychlorinated biphenyl analysis (Reagent)*

JIS K 8359 *Ammonium acetate (Reagent)*

JIS K 8891 *Methanol (Reagent)*

JIS R 3503 *Glass apparatus for chemical analysis*

JIS R 3505 *Volumetric glassware*

JIS Z 0701 *Silicagel desiccants for packaging*

3 Common matters

3.1 General

The general matters of chemical analysis shall be in accordance with JIS K 0050.

3.2 Terms and definitions

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

3.3 High performance liquid chromatograph tandem mass spectrometry (LC/MS/MS method)

General matters of the high performance liquid chromatograph tandem mass spectrometry shall be in accordance with JIS K 0136.

3.4 Water

Water to be used in this Standard shall be of A1 to A4 in accordance with JIS K 0557.

3.5 Reagents

The reagents used shall be as follows.

- a) The reagents used shall be in accordance with the relevant Japanese Industrial Standards (hereafter referred to as “JIS”), and shall not interfere with the test. If the reagent has no relevant JIS specification, that which does not interfere with the test shall be used.
- b) Reagents used for preparation of reference solutions shall be traceable to the National Measurement Standard (Article 134, the Measurement Act). If these are hard to obtain, commercially available reagents of which the purity and uncertainty are known shall be used.
- c) Care shall be exercised against the indoor contamination, the inhalation by or adherence to the human body of reagents, wastewater, etc. The operation of concentration and volatilization of solvents shall be carried out at a place provided with a proper exhaust equipment such as inside the draft. Handling of the reagents shall be with sufficient care, and in accordance with the related laws and regulations.

3.6 Glassware

The general matters of glassware shall be as follows.

- a) The glassware to be used, unless otherwise specified, shall be in accordance with JIS R 3503 and JIS R 3505. If the procedure includes heating operation, borosilicate glass – 1 specified in JIS R 3503 shall be used.
- b) The desiccant to be used for desiccators, unless otherwise specified, shall be the Type A, Class 1 silica gel desiccant for packaging specified in JIS Z 0701.
- c) The glassware shall be washed with water specified in 5.2 a), then with methanol specified in JIS K 8891, and left until the methanol has volatilized prior to use.

3.7 Working curve

In preparation of working curve, reference solution shall be prepared in 4 to 6 stepped up concentrations within the determination range so that the amount of target analyte at the time of introduction into the high performance liquid chromatograph tandem mass spectrometer is equal to the amount in the determination range.

NOTE 1 The designation of test target compound used is in accordance with the nomenclature of compound determined by the Chemical Society of Japan [based on the organic chemistry nomenclature of the International Union of Pure and Applied Chemistry (IUPAC)] and abbreviations, PFOS and PFOA, are used. The designation of reagents is based on the inorganic chemistry nomenclature and organic chemistry nomenclature of IUPAC and is basically conformed to the compound nomenclature determined by the Chemical Society of Japan and the designation of JIS reagents.

NOTE 2 The concentration of a reference solution is expressed by the mass in 1 ml ($\mu\text{g/ml}$ or ng/ml).

NOTE 3 The concentration of mixed solution of liquid reagent A and liquid re-

agent B is expressed by $A - B(a + b)$, which indicates that A and B are mixed in the rate of a ml and b ml.

NOTE 4 The concentration indicated in parentheses at the rear of the designation of a reference solution is an accurate concentration.

4 Sample

4.1 Sampling

4.1.1 Sample container

Sample container used shall be a 100 ml to 1 000 ml bottle with polypropylene screw cap. For the polypropylene screw cap, use of a packing of tetrafluoroethylene resin, etc. shall be avoided to prevent contamination.

The sample containers shall consist of materials which do not contaminate the sample or adsorb the target analyte during use or storage. The container shall be washed with water in 5.2 a), then with methanol in 5.2 b) and left to stand until methanol has evaporated. Use of all types of fluororesin, elastomer and tetrafluoroethylene resin materials shall be avoided since they are associated with the risk of contamination. Use of glassware shall be avoided due to potential analyte loss due to adsorption. Further, since all of the sampled volume will be used, the sampling containers of the required volume shall be prepared.

4.1.2 Sampling operation

The sampling operation shall be as follows.

- a) **Sampling of surface water** It shall be in accordance with 4.1.1 or 4.1.2 of JIS K 0094.

Examine the water for contamination from fluorocarbon resin and rubber and ensure freedom from influence of such contamination prior to use.

- b) **Sampling of water at each depth** It shall be in accordance with 4.1.4 of JIS K 0094.

Examine the water for contamination from fluorocarbon resin and rubber and ensure freedom from influence of such contamination prior to use.

- c) **Sampling from piping device** It shall be in accordance with 4.3 of JIS K 0094.

Examine the water for contamination from fluorocarbon resin and rubber and ensure freedom from influence of such contamination prior to use.

4.2 Handling of sample

The handling of sample shall be as follows.

- a) Carry out the test immediately after sampling. If this is not practicable, store the sample in a dark place at 0 °C to 10 °C, and test it as quickly as possible.
- b) For measurement of the sample volume, the whole volume of which is to be used, either subtract the mass of the sample container from the mass of the container containing the sample, or put a mark on the container at the water surface level at

the time of sampling and then after completion of the test, fill water up to the mark and take the volume of this water as the volume of the sample.

5 Pretreatment of sample

5.1 Summary

The summary of pretreatment of sample shall be as follows.

- a) For the pretreatment of sample, add ^{13}C -labelled PFOS and ^{13}C -labelled PFOA as the internal standard substance, perform adsorption of the target analyte by passing water through a solid-phase column by the pressure reduction method ¹⁾ and clean it with a cleaning solution. Then, elute the target analyte with eluting solvent and make a specified amount by concentrating.
- b) If suspended matter in the sample interferes with passing water at a constant rate and thus the pretreatment of the sample is difficult, separation and extraction of the suspended matter shall be performed in accordance with Annex JB for pretreatment.
- c) Commercially available reagents and instruments often vary in quality. Therefore, check the blank test results, recovery and repeatability of the analyte for each new lot of reagents or instruments and for each series of pretreatment.

Note ¹⁾ The pressure reduction method is a method in which sample is supplied by making the bottom part under reduced pressure. The apparatus is composed of a suction piping part, a vacuum pump or an aspirator.

5.2 Reagents

The reagents used shall be as follows.

- a) **Water** The water of A4 (or A3) in 3.4 shall be used for preparation, operation and blank test of reagents. Use water immediately after purification. Subject the water to the procedure given in Annex JA prior to use in order to confirm that no peak is found at the position corresponding to the retention time of the target analyte and that it has no influence on the test. If required, perform purification in accordance with NOTE 6 to clause 4 of JIS K 0557. Alternatively, water of A4 or A3 may be treated with activated carbon or the like.
- b) **Methanol** The methanol specified in JIS K 8891 shall be used. Prior to use, carry out the operation in 5.4 c) on 10 ml of methanol, and then perform the operation in 6.4 d) on the resultant solution in order to confirm that no peak is found at the position corresponding to the retention time of the target analyte. After the seal is broken, store in a place free from contamination.
- c) **^{13}C -labelled PFOS internal standard stock solution**, of 50 $\mu\text{g/ml}$ concentration.
- d) **^{13}C -labelled PFOS internal standard solution (10 $\mu\text{g/ml}$)** Take 1 ml of ^{13}C -labelled PFOS internal standard stock solution (50 $\mu\text{g/ml}$), transfer it into a 5 ml volumetric flask already containing 1 ml to 2 ml of methanol and add methanol up to the mark. Store it, if required, in a dark place at 5 °C or lower.

- e) **¹³C-labelled PFOS internal standard solution (0.1 µg/ml)** Take 1 ml of ¹³C-labelled PFOS internal standard solution (10 µg/ml), transfer it into a 100 ml volumetric flask already containing 2 ml to 5 ml of methanol and add methanol up to the mark. Store it, if required, in a dark place at 5 °C or lower.
- f) **¹³C-labelled PFOA internal standard stock solution**, of 50 µg/ml concentration.
- g) **¹³C-labelled PFOA internal standard solution (10 µg/ml)** Take 1 ml of ¹³C-labelled PFOA internal standard stock solution (50 µg/ml), transfer it into a 5 ml volumetric flask already containing 1 ml to 2 ml of methanol and add methanol up to the mark. Store it, if required, in a dark place at 5 °C or lower.
- h) **¹³C-labelled PFOA internal standard solution (0.1 µg/ml)** Take 1 ml of ¹³C-labelled PFOA internal standard solution (10 µg/ml), transfer it into a 100 ml volumetric flask already containing 2 ml to 5 ml of methanol and add methanol up to the mark. Store it, if required, in a dark place at 5 °C or lower.
- i) **Nitrogen**, of Grade 2 high purity in accordance with JIS K 1107.

NOTE : The internal standard stock solution obtained on the market may be used.

5.3 Implement

The implements shall be as follows.

- a) **Graduated test tube with polypropylene screw cap**, of 10 ml to 20 ml capacity, and graduated in 0.5 ml and 1 ml increments.
- b) **Solid-phase column**, filled with about 50 mg to 1 000 mg of absorbent. It shall not contain fluorocarbon resin except tetrafluoroethylene resin, and shall be applicable to the sample load up to 1 000 ml. Condition by passing a conditioning solution prior to use.

Determine the flow rate of the sample, the type and required amount of conditioning solution, cleaning solution and eluting solvent prior to use.

NOTE : Examples of solid-phase adsorbent, conditioning solution, cleaning solution and eluting solvent are shown in Annex A.

5.4 Operation

The operation shall be as follows.

- a) Passing of sample in the solid-phase column shall be performed under the condition that the column is completely filled with water. Shake the sample taken in 4.1.2 to mix well, and to the whole amount thereof, add the ¹³C-labelled PFOS internal standard solution (0.1 µg/ml) and ¹³C-labelled PFOA internal standard solution (0.1 µg/ml) (for example, 10 µl of each), then let this sample run through the solid-phase column at a flow rate of 3 ml/min to 6 ml/min by the pressure reduction method.

If a large amount of suspended matter is contained in the sample, carry out the operation in Annex JB.

- b) After the inside of the solid-phase column is cleaned by letting the cleaning solu-

tion flow slowly from the upper end of the solid-phase column, remove the solution in the solid-phase column by centrifuging it at the centrifugal force of 15 000 m/s² (1 500 g) for 2 min or by depressurizing or pressurizing. Then, clean the inside wall of the sample container in which the sample was contained with about 2 ml to 5 ml of eluting solvent, let the washings flow continuously slowly from the upper end of the solid-phase column and receive it in the graduated test tube with polypropylene screw cap. Repeat this operation two or three times to elute the target analyte.

The flow rate of elution shall be about one drop per second.

NOTE 1 Cleaning the inside wall of the sample container in which the sample was contained with eluting solvent such as methanol will improve the reduction of recovery due to adsorption to the inside wall of the sample container.

- c) Blow nitrogen gently into the eluate while heating the eluate at 40 °C or lower until it has concentrated to about 1 ml and take this as the measuring solution.

When the determination is not carried out immediately, store this concentrated solution at a dark place at 5 °C or under.

NOTE 2 In the operation of blowing nitrogen, care shall be taken so as not to scatter the concentrated solution. The flow rate of nitrogen should be controlled to the degree where the movement on the surface of concentrated solution is barely visible. And care shall also be taken that when exsiccated, the target analyte can be scattered by blowing nitrogen.

- d) Carry out the operations of a) to c) on the same amount of water as the sample, and take it as the solution for blank test. Carry out the blank test for each series of pretreatment operation. Carry it out at least once in every 10 samples
- e) Carry out the operations of a) to c) on the actual sample to which the reference solution has been added and take it as the solution for addition recovery test. For the evaluation of influence by the variation of the matrix and the sensitivity of measuring instrument, when there is a difference in the property of sample and expected concentration, carry out the addition recovery test for each series of operation.

6 High performance liquid chromatograph tandem mass spectrometry

6.1 Summary

The summary of high performance liquid chromatograph tandem mass spectrometry shall be as follows.

- a) Introduce a specified amount of measuring solution into the high performance liquid chromatograph tandem mass spectrometry, ionize PFOS and PFOA by using the negative mode of the electronic spray-ionization method (ESI). Measure PFOS and PFOA by using the selective reaction detection method (SRM) and then determine the linear-chain type PFOS and PFOA according to the working curve method.

- b) Optimize the elution condition of the high performance liquid chromatograph to the separation column to be used. Contamination from instruments shall be at most not more than one-tenth the expected concentrations in real samples.
- c) Interferences from instruments are significant for commercially available high performance liquid chromatograph tandem mass spectrometer because many parts are made of fluororesin, elastomer, tetrafluoroethylene resin, fluorine series polymer oil or the like which are all associated with the risk of contamination. Therefore, it is necessary to check for possible contamination from individual parts, and where required, replace them with parts made of materials such as stainless steel and polyetheretherketone (PEEK) which have been confirmed to be free from contamination.
- d) Parts such as HPLC vial, tubing, solvent filter, valve seal and degasser, etc. should be checked for contamination.

Determination range PFOS : 0.02 pg to 100 pg, PFOA : 0.1 pg to 100 pg (differs depending on the apparatus and measuring conditions)

NOTE 1 Substances producing generated ion in the same retention time as the target analyte and the matrix interference can cause reduction of sensitivity, lowering of S/N ratio, or the like, and therefore the influence of such interferences should be removed to the utmost level.

NOTE 2 In the case of clean samples, the influence of matrix interference is small. However, with those samples containing high concentration and complicated matrix such as waste water sample, measurement errors originating from matrix effect can be easily produced due to ion suppression or ion enhancement and care should be taken in handling of these.

6.2 Reagents

The reagents used shall be as follows.

- a) **Water**, in accordance with 5.2 a).
- b) **PFOS standard stock solution**, of 50 µg/ml concentration.
- c) **PFOS standard solution (1 µg/ml)** Take 1 ml of PFOS standard stock solution (50 µg/ml), transfer it into a 50 ml volumetric flask already containing 2 ml to 5 ml of methanol and add methanol up to the mark. Store it, if required, in a dark place at 5 °C or lower.
- d) **PFOS standard solution (10 ng/ml)** Take 1 ml of PFOS standard solution (1 µg/ml), transfer it into a 100 ml volumetric flask already containing 2 ml to 5 ml of methanol and add methanol up to the mark. Store it, if required, in a dark place at 5 °C or lower.
- e) **PFOA standard stock solution**, of 50 µg/ml concentration.
- f) **PFOA standard solution (1 µg/ml)** Take 1 ml of PFOA standard solution (50 µg/ml), transfer it into a 50 ml volumetric flask already containing 2 ml to 5 ml of methanol and add methanol up to the mark. Store it, if required, in a dark place at 5 °C

or lower.

- g) **PFOA standard solution (10 ng/ml)** Take 1 ml of PFOA standard solution (1 µg/ml), transfer it into a 100 ml volumetric flask already containing 2 ml to 5 ml of methanol and add methanol up to the mark. Store it, if required, in a dark place at 5 °C or lower.
- h) **¹³C-labelled PFOS internal standard solution (0.1 µg/ml)**, in accordance with 5.2 e).
- i) **¹³C-labelled PFOA internal standard solution (0.1 µg/ml)**, in accordance with 5.2 h).
- j) **Methanol**, in accordance with 5.2 b).
- k) **Acetonitrile**, specified in JIS K 8039. Prior to use, perform the procedure in 5.4 c) on 100 ml of acetonitrile, and further subject it to the procedure in 6.4 d) in order to confirm that no peak is found at the position corresponding to the retention time of the target analyte. After the seal is broken, store it at a place free from contamination.
- l) **Ammonium acetate**, specified in JIS K 8359.

6.3 Implement and apparatus

The implement and apparatus used shall be as follows.

- a) **Graduated test tube with polypropylene screw cap**, in accordance with 5.3 a).
- b) **Microsyringe**, of 10 µl to 20 µl capacity.
- c) **High performance liquid chromatograph tandem mass spectrometer**, consisting of the following :
 - 1) **Separation column**, made of stainless steel, 2 mm to 6 mm in inside diameter, and 50 mm to 300 mm in length. A guard column may be used for preventing the performance degradation of the separation column.
 - 2) **Filler** Silica gel or synthetic polymer, etc. which is chemically bonded with octadecyl radical (ODS) or that having the separative power at least equal thereto, with particle diameter of about 1 µm to 5 µm. Example of column to be used is shown in Annex B.
 - 3) **Eluent**, suitable for the apparatus and separation column, for example, combination of the organic solvent such as methanol and acetonitrile, and ammonium acetate solution (2 mmol/L). An example of analytical condition is shown in Annex C.
 - 4) **Eluent condition**, suitable for the separation column. An example of analytical condition is shown in Annex C.
 - 5) **Ionization**, of the electrospray ionization method. Negative mode ionization (negative ESI) shall be possible.
 - 6) **Detector**, capable of carrying out the selective reaction detection method (SRM).

NOTE : These conditions differ depending on the analytical apparatus and measuring conditions. Example of the instrumental analytical conditions for separation of the linear-chain isomers and side-chain

isomers of PFOS and PFOA is shown in Annex C. The organic fluorine compounds of different number of carbon chains can be measured by this method. An example of the measuring condition of this method is shown in Annex JC.

6.4 Operation

The operation shall be as follows.

- Set the mass-to-charge ratio (m/z) of the precursor ion and product ion of PFOS, ^{13}C -labelled PFOS, PFOA, ^{13}C -labelled PFOA to the high performance liquid chromatograph tandem mass spectrometer prior to measurement. Table 2 shows examples of precursor ion and product ion.
- Take 1 ml of PFOS standard solution (10 ng/ml) and PFOA standard solution (10 ng/ml) in a graduated test tube with polypropylene screw cap respectively and add ^{13}C -labelled PFOS internal standard solution (0.1 $\mu\text{g/ml}$) and ^{13}C -labelled PFOA internal standard solution (0.1 $\mu\text{g/ml}$)(for example, 10 μl of each) using a microsyringe. Blow nitrogen gently into this solution while heating it at 40 °C or lower until it has concentrated to about 1 ml.
- Introduce an appropriate amount (for example, 10 μl) of the solution prepared in b) into a high performance liquid chromatograph tandem mass spectrometer, record the chromatogram according to the selective reaction detection method (SRM) and confirm the position of peak corresponding to the retention time of the detected PFOS, ^{13}C -labelled PFOS, PFOA and ^{13}C -labelled PFOA.

Table 2 Example of selected ion of target analyte and internal standard substance

No	Name of compound	Abbreviation	Mass-to-charge ratio (m/z)		
			Precursor ion $M_1^{\text{a)}$	Product ion 1 $M_2^{\text{a)}$	Product ion 2 $M_3^{\text{a)}$
1	Perfluorooctane sulfonic acid	PFOS	499	80	99
2	Perfluorooctanoic acid	PFOA	413	369	169
3	$^{13}\text{C}_4$ -labelled perfluorooctane sulfonic acid ^{b)}	$^{13}\text{C}_4$ -PFOS	503	80	99
4	$^{13}\text{C}_8$ -labelled perfluorooctane sulfonic acid ^{b)}	$^{13}\text{C}_8$ -PFOS	507	80	99
5	$^{13}\text{C}_4$ -labelled perfluorooctanoic acid ^{b)}	$^{13}\text{C}_4$ -PFOA	417	372	169
6	$^{13}\text{C}_8$ -labelled perfluorooctanoic acid ^{b)}	$^{13}\text{C}_8$ -PFOA	421	376	172
NOTE : In the target analyte and internal standard, the linear-chain type is the target analyte.					
Notes ^{a)} M_2 is used as a quantitative ion and M_3 is used as a confirmatory ion. M_1 is the precursor ion of each compound.					
^{b)} Internal standard substance.					

- Introduce the measuring solution obtained in 5.4 c) [of the same amount as the standard solution (for example, 10 μl)] into a high performance liquid chromatograph tandem mass spectrometer, record the chromatogram by carrying out the operation similar to c), and after confirming the coincidence with the retention time