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Method for determination of tetra-through octachlorodibenzo-*p*-dioxins, tetra-through octachlorodibenzofurans and dioxin-like polychlorinatedbiphenyls in industrial water and waste water

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In the event of any doubts arising as to the contents,
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Foreword

This Japanese Industrial Standard has been revised by the Minister of Economy, Trade and Industry, through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Act. This edition replaces the previous edition (**JIS K 0312**:2008), which has been technically revised.

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Method for determination of tetra-through octachlorodibenzo-*p*-dioxins, tetra-through octachlorodibenzofurans and dioxin-like polychlorinated biphenyls in industrial water and waste water

Introduction

This Japanese Industrial Standard was established in 1999 as “Method for determination of tetra-through octachlorodibenzo-*p*-dioxins, tetra-through octachlorodibenzofurans and coplanar polychlorobiphenyls in industrial water and waste water”, and has gone through three revisions including this one. This Standard has been prepared based on **ISO 18073**:2004, Edition 1, and **ISO 17858**:2007, Edition 1, with some modifications of the technical contents.

The vertical lines on both sides and dotted underlines indicate changes from the corresponding International Standards. A list of modifications with the explanations is given in Annex JE. Annex JA to Annex JD are unique to **JIS** and not given in the corresponding International Standards.

1 Scope

This Standard specifies a method for the determination of tetra-through octachlorodibenzo-*p*-dioxins, tetra-through octachlorodibenzofurans and dioxin-like PCBs in industrial water and waste water using a gas chromatograph/mass spectrometer (hereafter referred to as GC-MS). The GC-MS employed in this Standard shall be the double-focusing mass spectrometer (hereafter referred to as MS) where the capillary column of a gas chromatograph (hereafter referred to as GC) is used and its resolution is 10 000 or more.

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

ISO 18073:2004 *Water quality—Determination of tetra- to octa-chlorinated dioxins and furans—Method using isotope dilution HRGC/HRMS*

ISO 17858:2007 *Water quality—Determination of dioxin-like polychlorinated biphenyls—Method using gas chromatography/mass spectrometry* (overall evaluation: MOD)

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

WARNING Since dioxins are very toxic, inhalation, accidental ingestion, direct contact to skin, etc. shall be avoided as much as possible, and ventilation of pretreatment room and analysis room and control of waste liquid and waste shall be sufficiently carried out. Since the health of the measurer may be damaged by inhalation or accidental ingestion

of other drugs, solvents, etc., the handling shall be carried out as carefully as possible, and attention shall be paid to sufficient ventilation in the laboratory.

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 0094 *Sampling methods for industrial water and industrial wastewater*
- JIS K 0114 *General rules for gas chromatography*
- JIS K 0123 *General rules for gas 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 0216 *Technical terms for analytical chemistry (Environmental part)*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 1107 *Nitrogen*
- JIS K 8040 *Acetone for pesticide residue and polychlorinated biphenyl tests (Reagent)*
- JIS K 8117 *Dichloromethane for pesticide residue and polychlorinated biphenyl analysis (Reagent)*
- JIS K 8180 *Hydrochloric acid (Reagent)*
- JIS K 8550 *Silver nitrate (Reagent)*
- JIS K 8574 *Potassium hydroxide (Reagent)*
- JIS K 8637 *Sodium thiosulfate pentahydrate (Reagent)*
- JIS K 8680 *Toluene (Reagent)*
- JIS K 8825 *Hexane for pesticide residue and polychlorinated biphenyl tests (Reagent)*
- JIS K 8891 *Methanol (Reagent)*
- JIS K 8951 *Sulfuric acid (Reagent)*
- JIS K 8987 *Sodium sulfate (Reagent)*
- JIS K 9502 *L(+)-Ascorbic acid (Reagent)*
- JIS K 9702 *Dimethyl sulfoxide (Reagent)*
- JIS K 9703 *2,2,4-Trimethylpentane (Reagent)*
- JIS R 3503 *Glass apparatus for chemical analysis*
- JIS R 3505 *Volumetric glassware*
- JIS Z 8401 *Rounding of numbers*

3 Terms, definitions and abbreviations

3.1 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 0094**, **JIS K 0114**, **JIS K 0123**, **JIS K 0211**, **JIS K 0215** and **JIS K 0216**, and the following apply.

3.1.1

dioxins

generic term of tetra-through octachlorodibenzo-*p*-dioxins (hereafter referred to as PCDDs), tetra-through octachlorodibenzofurans (hereafter referred to as PCDFs) and dioxin-like PCBs (hereafter referred to as DL-PCBs)

NOTE This definition differs from that specified in **JIS K 0216:2014**.

3.1.2

isomer

each compound whose number of chlorines substituent is the same, but their positions are different

3.1.3

homologue

a group of compounds whose number of chlorines substituent is the same, but their positions are different

3.1.4

isomers substituted by chlorine at 2,3,7,8-positions

total 17 types of isomers substituted by chlorine at 2,3,7,8-positions, being composed of 7 types of PCDDs, and 10 types of PCDFs, as given in Table 1

Table 1 Isomers substituted by chlorine at 2,3,7,8-positions

	Isomers substituted by chlorine at 2,3,7,8-positions	Abbreviation
PCDDs	2,3,7,8-Tetrachlorodibenzo- <i>p</i> -dioxin	2,3,7,8-TeCDD
	1,2,3,7,8-Pentachlorodibenzo- <i>p</i> -dioxin	1,2,3,7,8-PeCDD
	1,2,3,4,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	1,2,3,4,7,8-HxCDD
	1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	1,2,3,6,7,8-HxCDD
	1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin	1,2,3,7,8,9-HxCDD
	1,2,3,4,6,7,8-Heptachlorodibenzo- <i>p</i> -dioxin	1,2,3,4,6,7,8-HpCDD
	1,2,3,4,6,7,8,9-Octachlorodibenzo- <i>p</i> -dioxin	1,2,3,4,6,7,8,9-OCDD
PCDFs	2,3,7,8-Tetrachlorodibenzofuran	2,3,7,8-TeCDF
	1,2,3,7,8-Pentachlorodibenzofuran	1,2,3,7,8-PeCDF
	2,3,4,7,8-Pentachlorodibenzofuran	2,3,4,7,8-PeCDF
	1,2,3,4,7,8-Hexachlorodibenzofuran	1,2,3,4,7,8-HxCDF
	1,2,3,6,7,8-Hexachlorodibenzofuran	1,2,3,6,7,8-HxCDF
	1,2,3,7,8,9-Hexachlorodibenzofuran	1,2,3,7,8,9-HxCDF
	2,3,4,6,7,8-Hexachlorodibenzofuran	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-Heptachlorodibenzofuran	1,2,3,4,6,7,8-HpCDF
	1,2,3,4,7,8,9-Heptachlorodibenzofuran	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,6,7,8,9-Octachlorodibenzofuran	1,2,3,4,6,7,8,9-OCDF

3.1.5

DL-PCBs

of PCBs, compounds where ortho positions (2,2',6 and 6') are not substituted by chlorine (hereafter referred to as non-ortho compounds) and compounds having one substituted chlorine at the ortho position (hereafter referred to as mono-ortho compounds), which are given in Table 2

Table 2 DL-PCBs

	DL-PCBs	Abbreviation
Non-ortho compounds	3,3',4,4'-Tetrachlorobiphenyl	3,3',4,4'-TeCB (#77)
	3,4,4',5-Tetrachlorobiphenyl	3,4,4',5-TeCB (#81)
	3,3',4,4',5-Pentachlorobiphenyl	3,3',4,4',5-PeCB (#126)
	3,3',4,4',5,5'-Hexachlorobiphenyl	3,3',4,4',5,5'-HxCB (#169)
Mono-ortho compounds	2,3,3',4,4'-Pentachlorobiphenyl	2,3,3',4,4'-PeCB (#105)
	2,3,4,4',5-Pentachlorobiphenyl	2,3,4,4',5-PeCB (#114)
	2,3',4,4',5-Pentachlorobiphenyl	2,3',4,4',5-PeCB (#118)
	2',3,4,4',5-Pentachlorobiphenyl	2',3,4,4',5-PeCB (#123)
	2,3,3',4,4',5-Hexachlorobiphenyl	2,3,3',4,4',5-HxCB (#156)
	2,3,3',4,4',5'-Hexachlorobiphenyl	2,3,3',4,4',5'-HxCB (#157)
	2,3',4,4',5,5'-Hexachlorobiphenyl	2,3',4,4',5,5'-HxCB (#167)
	2,3,3',4,4',5,5'-Heptachlorobiphenyl	2,3,3',4,4',5,5'-HpCB (#189)
NOTE Figures in parentheses indicate IUPAC No.		

3.2 Abbreviations

Abbreviations shall be as given in Table 3.

Table 3 Abbreviations

Abbreviation	Name
TeCDD	tetrachlorodibenzo- <i>p</i> -dioxin
PeCDD	pentachlorodibenzo- <i>p</i> -dioxin
HxCDD	hexachlorodibenzo- <i>p</i> -dioxin
HpCDD	heptachlorodibenzo- <i>p</i> -dioxin
OCDD	octachlorodibenzo- <i>p</i> -dioxin
TeCDF	tetrachlorodibenzofuran
PeCDF	pentachlorodibenzofuran
HxCDF	hexachlorodibenzofuran
HpCDF	heptachlorodibenzofuran
OCDF	octachlorodibenzofuran
TeCB	tetrachlorobiphenyl
PeCB	pentachlorobiphenyl
HxCB	hexachlorobiphenyl
HpCB	heptachlorobiphenyl
DeCB	decachlorobiphenyl
DMSO	dimethyl sulfoxide
IUPAC	International Union of Pure and Applied Chemistry
NOTE The abbreviation of chemical compound may be expressed with "s" which means multiple.	

4 Outline of determination method

After the dioxins in industrial water and waste water are extracted, they shall be cleaned up, then identified and determined using a GC-MS. The flow diagram of the analysis is shown in Figure 1.