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Method for determination of tetra-through octachlorodibenzo-*p*-dioxins, tetra-through octachlorodibenzofurans and dioxin-like polychlorinatedbiphenyls in stationary source emissions

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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 0311**: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 stationary source emissions

## Introduction

This Japanese Industrial Standard was established in 1999, and has gone through three revisions including this one. After the previous revision was made in 2008, organic solvents used for extraction have been designated as specified chemical substances in the Industrial Safety and Health Act. In step with this, this Standard has been revised in order to ensure the safety of analytical workers.

No corresponding International Standard has been established at this point.

## 1 Scope

This Standard specifies a method for the determination of tetra-through octachlorodibenzo-*p*-dioxins, tetra-through octachlorodibenzofurans and dioxin-like PCBs in flue gas which are generated by combustion and chemical reactions, and discharged to a flue, stack or duct (hereafter referred to as duct) in stationary source emissions 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.

**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 0095 *Methods for sampling of flue gas*

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 0301 *Methods for determination of oxygen in flue gas*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 0901 *Form, size and performance testing methods of filtration media for collecting airborne particulate matters*
- JIS K 1107 *Nitrogen*
- JIS K 8040 *Acetone for pesticide residue and polychlorinated biphenyl tests (Reagent)*
- JIS K 8099 *Diethylene glycol (Reagent)*
- JIS K 8105 *Ethylene glycol (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 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 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*
- JIS Z 8808 *Methods of measuring dust concentration in flue gas*

### 3 Terms, definitions and abbreviations

#### 3.1 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 0095**, **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

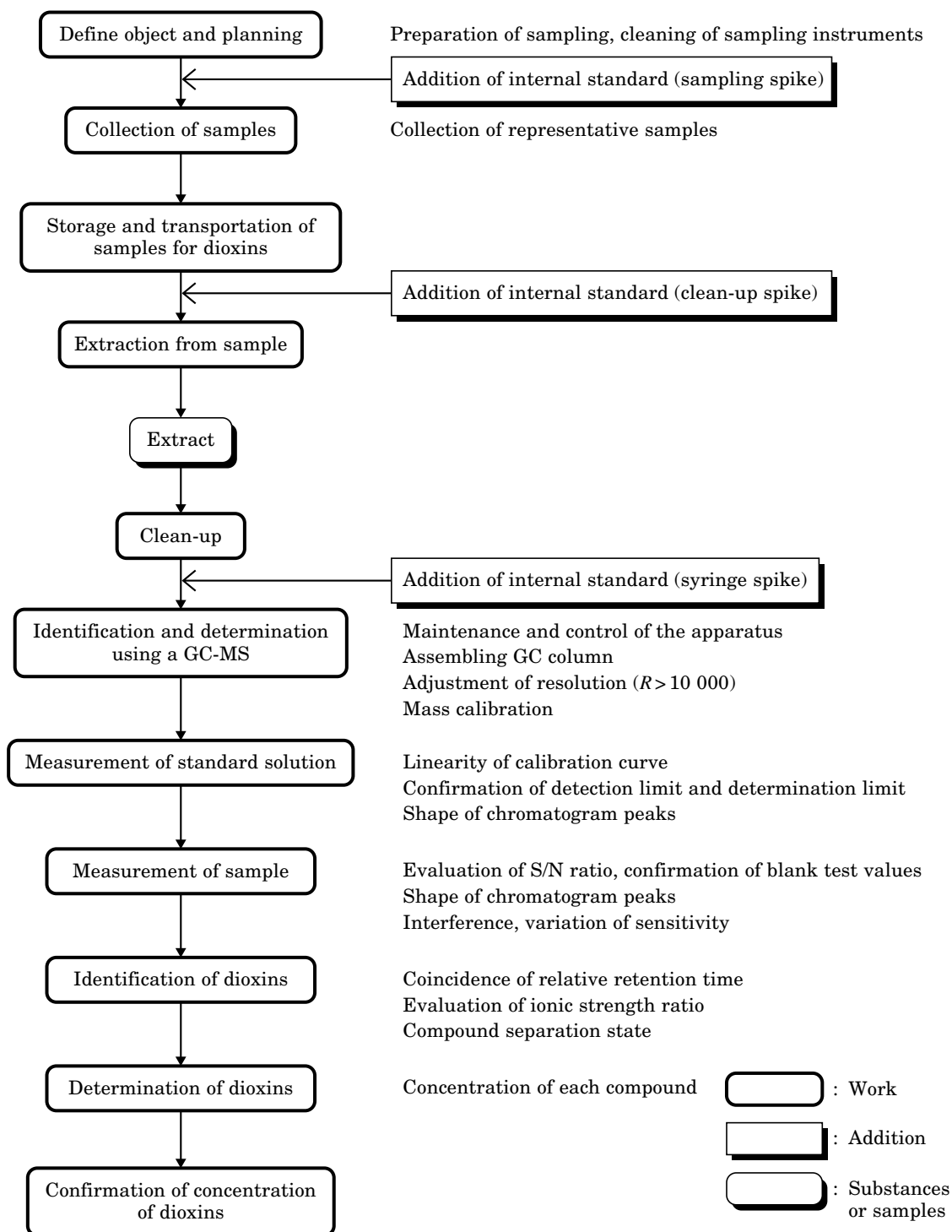
Abbreviations shall be as given in Table 3.

**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

The dioxins in flue gas are collected by “filtration collection” using e.g. cylindrical filter paper, “absorption collection” using an absorbing bottle (impinger) or “adsorption collection” using an adsorption column. After extracting dioxins from collecting part, they are cleaned up, then identified and determined using a GC-MS. The flow diagram of the analysis is shown in Figure 1.



**Figure 1** Flow diagram of the determination of dioxins in flue gas