JIS

JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 0312 : 2005

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

ICS 13.060.25; 13.060.30

Reference number : **JIS K 0312 : 2005 (E)**

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS K 0312** : 1999 is replaced with this **JIS K 0312** : 2005.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

Date of Establishment: 1999-09-20

Date of Revision: 2005-06-20

Date of Public Notice in Official Gazette: 2005-06-20

Investigated by: Japanese Industrial Standards Committee

Standards Board

Technical Committee on Environment and Recycling Policy

JIS K 0312:2005, First English edition published in 2005-12

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

In the event of any doubts arising as to the contents, the original JIS is to be the final authority.

Printed in Japan

NH/AT

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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

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

The minimum limits of detection of GC/MS in this Standard, though it is liable to vary in accordance with an apparatus or analytical conditions, are 0.1 pg for tetrachlorinated compounds and pentachlorinated compounds, 0.2 pg for hexachlorinated compounds and heptachlorinated compounds, 0.5 pg for octachlorinated compounds and 0.2 pg or less for dioxin-like PCBs.

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 chromatographic analysis*

JIS K 0123 *General rules for analytical methods in 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 0557 *Water used for industrial water and wastewater analysis*

JIS K 1107 *High purity nitrogen*

JIS K 8040 *Acetone for pesticide residue and polychlorinated biphenyl analysis*

JIS K 8117 *Dichloromethane for pesticide residue and polychlorinated biphenyl analysis*

JIS K 8180 *Hydrochloric acid*

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JIS K 8574 *Potassium hydroxide*

JIS K 8637 *Sodium thiosulfate pentahydrate*

JIS K 8680 *Toluene*

JIS K 8825 *Hexane for pesticide residue and polychlorinated biphenyl analysis*

JIS K 8891 *Methanol*

JIS K 8951 *Sulfuric acid*

JIS K 8987 *Sodium sulfate*

JIS K 9702 *Dimethyl sulfoxide*

JIS K 9703 *2,2,4-Trimethylpentane*

JIS R 3503 *Glass apparatus for chemical analysis*

JIS R 3505 *Volumetric glassware*

JIS Z 8401 *Guide to the rounding of numbers*

3 Definitions For the purposes of this Standard, the definitions given in **JIS K 0094**, **JIS K 0114**, **JIS K 0123**, **JIS K 0211** and **JIS K 0215**, and the following definitions apply.

a) **dioxins** a generic term of tetra-through octachlorodibenzo-para-dioxins, tetra-through octachlorodibenzofurans and dioxin-like PCBs

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

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

d) **PCDDs** Polychlorodibenzo-p-dioxins

e) **PCDFs** Polychlorodibenzofurans

f) **TeCDDs** Tetrachlorodibenzo-p-dioxins

g) **PeCDDs** Pentachlorodibenzo-p-dioxins

h) **HxCDDs** Hexachlorodibenzo-p-dioxins

i) **HpCDDs** Heptachlorodibenzo-p-dioxins

j) **OCDD** Octachlorodibenzo-p-dioxin

k) **TeCDFs** Tetrachlorodibenzofurans

l) **PeCDFs** Pentachlorodibenzofurans

m) **HxCDFs** Hexachlorodibenzofurans

n) **HpCDFs** Heptachlorodibenzofurans

o) **OCDF** Octachlorodibenzofuran

p) **isomers substituted by chlorine at 2,3,7,8-positions** total of 17 types of isomers substituted by chlorine at 2,3,7,8-positions as shown below

They are composed of 7 types of tetra-through octachlorodibenzo-para-dioxins, and 10 types of tetra-through octachlorodibenzofurans.

1) **Tetra- to octachlorodibenzo-para-dioxin** (**PCDDs**)

2,3,7,8-Tetrachlorodibenzo-para-dioxin (2,3,7,8-TeCDD)

1,2,3,7,8-Pentachlorodibenzo-para-dioxin (1,2,3,7,8-PeCDD)

1,2,3,4,7,8-Hexachlorodibenzo-para-dioxin (1,2,3,4,7,8-HxCDD)

1,2,3,6,7,8-Hexachlorodibenzo-para-dioxin (1,2,3,6,7,8-HxCDD)

1,2,3,7,8,9-Hexachlorodibenzo-para-dioxin (1,2,3,7,8,9-HxCDD)

1,2,3,4,6,7,8-Heptachlorodibenzo-para-dioxin (1,2,3,4,6,7,8-HpCDD)

Octachlorodibenzo-para-dioxin (OCDD)

2) **Tetra- to octachlorodibenzofuran (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-Hexachlorodibnzofuran (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)

Octachlorodibenzofuran (OCDF)

q) **PCBs** Polychlorobiphenyl

r) **TeCBs** Tetrachlorobiphenyl

s) **PeCBs** Pentachlorobiphenyl

t) **HxCBs** Hexachlorobiphenyl

u) **HpCBs** Heptachlorobiphenyl

v) **dioxin-like PCBs (DL-PCB)** polychlorobiphenyl (PCBs) which include compounds where ortho positions (2,2′,6 and 6′) are not substituted by chlorine (non-ortho compounds) and mono-ortho compounds having one substituted chlorine at the ortho position (mono-ortho compounds)

It is also called coplanar PCB.

1) **Non-ortho compounds**

3,4,4′,5-Tetrachlorobiphenyl [3,4,4′,5-TeCB (IUPAC* No. 81)]

Note * Abbreviation of International Union of Pure and Applied Chemistry.

3,3′,4,4′-Tetrachlorobiphenyl [3,3′,4,4′-TeCB (IUPAC No. 77)]

3,3′,4,4′,5-Pentachlorobiphenyl [3,3′,4,4′,5-PeCB (IUPAC No. 126)]

3,3′,4,4′,5,5′-Hexachlorobiphenyl [3,3′,4,4′,5,5′-HxCB (IUPAC No. 169)]

2) **Mono-ortho compounds**

2,3,3′,4,4′-Pentachlorobiphenyl [2,3,3′4,4′-PeCB (IUPAC No. 105)]

2,3,4,4′,5-Pentachlorobiphenyl [2,3,4,4′,5-PeCB (IUPAC No. 114)]

2,3′,4,4′,5-Pentachlorobiphenyl [2,3′,4,4′,5-PeCB (IUPAC No. 118)]

2′,3,4,4′,5-Pentachlorobiphenyl [2′,3,4,4′,5-PeCB (IUPAC No. 123)]

2,3,3′,4,4′,5-Hexachlorobiphenyl [2,3,3′,4,4′,5-HxCB (IUPAC No. 156)]

2,3,3′,4,4′,5′-Hexachlorobiphenyl [2,3,3′,4,4′,5′-HxCB (IUPAC No. 157)]

2,3′,4,4′,5,5′-Hexachlorobiphenyl [2,3′,4,4′,5,5′-HxCB (IUPAC No. 167)]

2,3,3′,4,4′,5,5′-Heptachlorobiphenyl [2,3,3′,4,4′,5,5′-HpCB (IUPAC No. 189)]

w) **the minimum limit of detection for the apparatus** the minimum value that can be detected with the GC/MS used for the measurement

x) **the minimum limit of detection for the analytical method** the minimum value that can be detected in the procedure of the operation from pretreatment of the sample to the measurement with GC/MS

y) **the minimum limit of detection for the sample** the minimum concentration that can be detected for the sample

z) **the minimum limit of determination for the apparatus** the minimum value that can be determined with the GC/MS used for the measurement

aa) **the minimum limit of determination for the analytical method** the minimum value which can be determined in the procedure of the operation from pretreatment of the sample to the measurement with GC/MS

In general, approximately 3-fold errors may be estimated for the value in the vicinity of the minimum limit of the detection, to compare with the values obtained near the minimum limit of the determination.

ab) **the minimum limit of determination for the sample** the minimum concentration that can be determined in the sample

ac) **TEF** 2,3,7,8-TeCDD toxic equivalency factor

ad) **TEQ** 2,3,7,8-TeCDD toxic equivalency quantity

ae) **RRF** relative response factor

4 Outline of the analytical method After the dioxins in industrial water and waste water shall be extracted, they shall be cleaned up, identified and determined using a GC/MS. The flow diagram of the analysis is shown in figure 1.

Remarks : Because dioxins are highly toxic, inhalation, accidental ingestion or direct dermal contact with them shall be avoided as much as possible. The pretreatment room and laboratory shall be well-ventilated, and the management of waste water and wastes shall also be sufficiently carried out. Inhalation or accidental ingestion of other reagents, solvents, etc. may be detrimental to the health of technicians and for this reason, the utmost care shall be taken when handling them and the laboratory shall be sufficiently ventilated.