



JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 0311 : 2005

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

ICS 13.040.40; 71.040.40

Reference number : JIS K 0311 : 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 0311 : 1999** is replaced with this Standard.

In this revision, this Standard has been extensively looked over and the revision has been carried out about definitions, sampling of sample gas, pretreatment of sample, identification and determination, report of results and quality control of measurement data.

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 0311:2005, First English edition published in 2005-11

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.

© JSA 2005

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

Printed in Japan

NH/AT

Contents

	Page
1 Scope	1
2 Normative references	1
3 Definitions	2
4 Outline of the analytical method.....	4
5 Gas sampling	6
5.1 Outline of gas sampling	6
5.2 Gas sampling apparatus.....	6
5.3 Reagents	8
5.4 Preparations for sampling of sample gas.....	10
5.5 Sampling amount of sample gas	12
5.6 Sampling procedure.....	12
5.7 Recovery and storage of the sample	13
5.8 Calculation of gas sampling volume	14
5.9 Report of gas sampling	14
6 Pretreatment of the sample.....	14
6.1 Outline for the pretreatment of the sample	14
6.2 Reagents	17
6.3 Equipment and apparatus	19
6.4 Procedures for pretreatment	19
7 Identification and determination.....	28
7.1 Outline of the identification and the determination.....	28
7.2 Reagents and apparatus.....	28
7.3 Measurement	31
7.4 Identification and determination	36
7.5 The minimum limit of detection and the minimum limit of determination	39
7.6 Confirmation of the recovery ratio	42

8	Report of the results	43
8.1	Methods of expression for analytical results	43
8.2	Unit of concentration	43
8.3	Change to toxicity equivalency (TEQ)	43
8.4	Treatment of numerical values	44
9	Quality control of the measured data	46
9.1	Assurance of reliability of measured data	46
9.2	Requirements for operation of the measurement	50
9.3	Record of measurement operation	53
9.4	Report on the precision control	54
Annex 1 (normative)	Sample gas sampling apparatus	64
Annex 2 (informative)	Example for use of internal standard substance	71

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

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 exhaust gas which are generated by combustion, chemical reactions and discharged to a flue, stack or duct (hereafter referred to as “duct”) in stationary source emissions 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 the 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 under for dioxin-like PCBs.

2 Normative references The following standards contain provisions which, through reference in this Standard, 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 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 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 *High purity nitrogen*
- JIS K 8040 *Acetone for pesticide residue and polychlorinated biphenyl analysis*
- JIS K 8105 *Ethylene glycol*
- JIS K 8117 *Dichloromethane for pesticide residue and polychlorinated biphenyl analysis*
- JIS K 8180 *Hydrochloric acid*
- JIS K 8550 *Silver nitrate*

JIS K 8574 *Potassium hydroxide*

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*

JIS Z 8808 *Methods of measuring dust concentration in flue gas*

3 Definitions For the purposes of this Standard, the definitions given in **JIS K 0095**, **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-para-dioxins
- e) **PCDFs** Polychlorodibenzofurans
- f) **TeCDDs** Tetrachlorodibenzo-para-dioxins
- g) **PeCDDs** Pentachlorodibenzo-para-dioxins
- h) **HxCDDs** Hexachlorodibenzo-para-dioxins
- i) **HpCDDs** Heptachlorodibenzo-para-dioxins
- j) **OCDD** Octachlorodibenzo-para-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-dioxins (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 octachlorodibenzofurans (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)
 - Octachlorodibenzofuran (OCDF)
- q) **PCBs** Polychlorobiphenyls
- r) **TeCBs** Tetrachlorobiphenyls
- s) **PeCBs** Pentachlorobiphenyls
- t) **HxCBs** Hexachlorobiphenyls
- u) **HpCBs** Heptachlorobiphenyls
- v) **dioxin-like PCBs (DL-PCBs)** polychlorobiphenyls (PCBs) which include compounds where ortho positions (2,2',6 and 6') are not substituted by chlorine (non-ortho compounds) and compounds having one substituted chlorine at the ortho position (mono-ortho compounds) That 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 gas** the minimum concentration that can be detected for the sample gas
- z) **the minimum limit of the determination for the apparatus** the minimum value that can be determined by the GC/MS used for the measurement
- aa) **the minimum limit of the 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 vicinity of the minimum limit of the detection, to compare with the values obtained near the minimum limit of the determination.
- ab) **minimum limit of determination for the sample gas** the minimum concentration that can be determined in the sampling gas
- ac) **TEF** 2,3,7,8-TeCDD toxic equivalency factor
- ad) **TEQ** 2,3,7,8-TeCDD toxic equivalent quantity
- ae) **RRF** relative response factor

4 Outline of the analytical method The dioxins in exhaust gas shall be collected by "collection filter" with using a cylindrical filter and such like, "absorption collection" with using a linked absorption bottle (impinger) and "adsorption collection" by an adsorption column. After extracting them from collection part, 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 room 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.

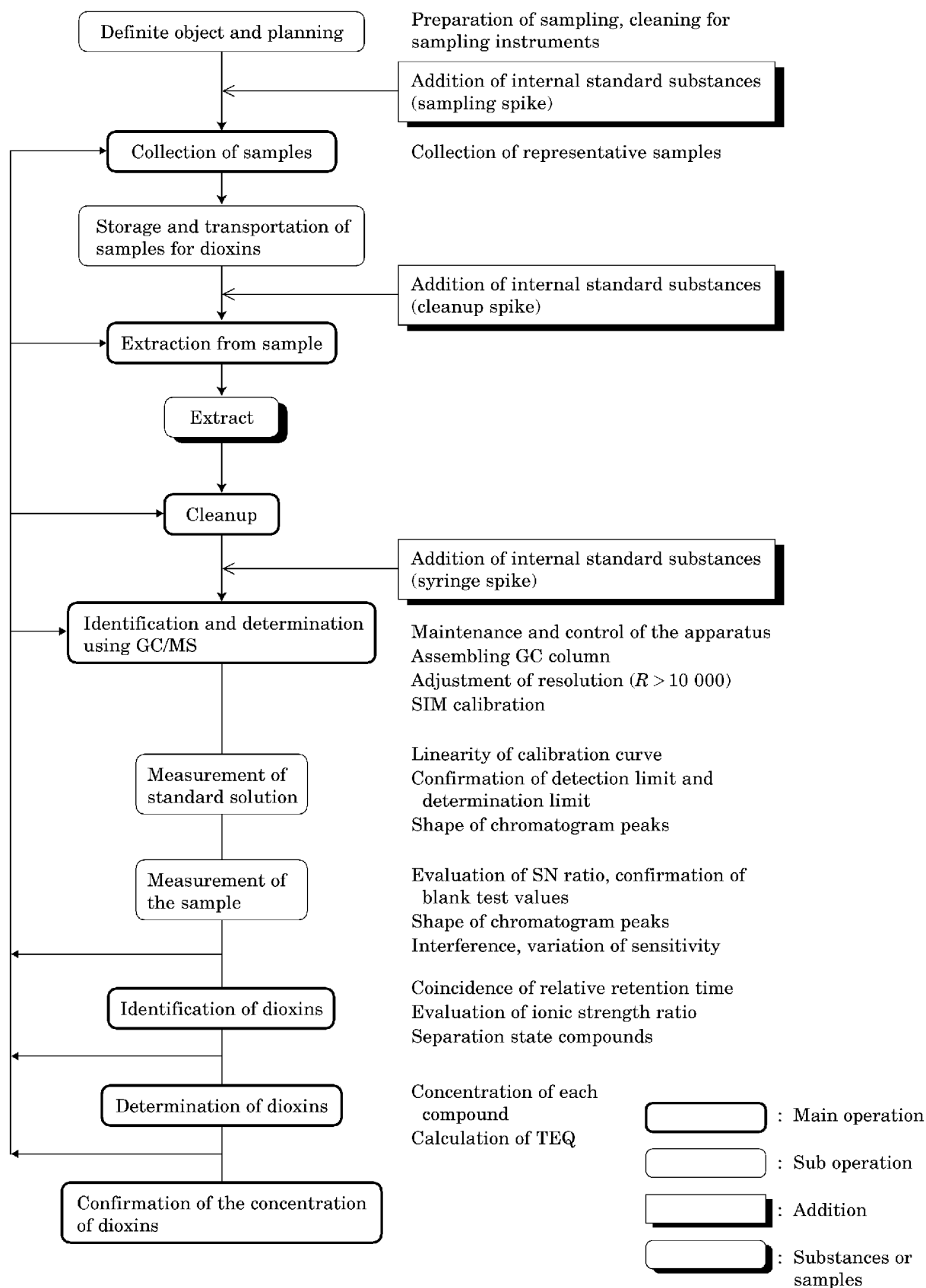


Figure 1 Flow diagram for the determination of dioxins in exhaust gas