[This document is just explanation translated from the original Japanese certificate and some information is omitted from it.]

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan

Reference Material Certificate

NMIJ CRM 7904-a
No. +++

Polychlorinated Biphenyls in Fuel Oil

National Institute of Advanced Industrial Science and Technology
AIST

This certified reference material (CRM) was produced based on NMIJ's quality system in compliance with JIS Q 0034 (ISO GUIDE 34), for use in controlling the precision of analysis or confirming the validity of analytical methods or instruments during analysis of polychlorinated biphenyls (PCBs) in mineral oil samples and similar materials.

Certified Values

The certified values, expressed as mass fractions (dry-mass basis), are given in the following table. The expanded uncertainty was determined using coverage factor ($k = 2$), corresponding to an estimated confidence interval of approximately 95 %.

Certified values of PCB congeners*

	CAS No.	Certified value Mass fraction (μg/kg)	Expanded uncertainty Mass fraction (μg/kg)	Analytical Method
CB8 (2,4'-dichlorobipheny)	34883-43-7	575	29	1,2,3,4
CB28 (2,4,4'-trichlorobiphenyl)	7012-37-5	173	8	1,2
CB52 (2,2',5,5'-tetrachlorobiphenyl)	35693-99-3	184	10	1,2,4
CB101 (2,2',4,5,5'-pentachlorobiphenyl)	37680-73-2	167	13	1,2,3,4
CB118 (2,3',4,4',5- pentachlorobiphenyl)	31508-00-6	124	7	1,2,3,4,5
CB138 (2,2',3,4,4',5'- hexachlorobiphenyl)	35065-28-2	133	14	1,2
CB153 (2,2',4,4',5,5'- hexachlorobiphenyl)	35065-27-1	174	7	1,2,4,5
CB180 (2,2',3,4,4',5,5'- heptachlorobiphenyl)	35065-29-3	151	8	1,2,3,4,5
CB194(2,2',3,3',4,4',5,5'- octachlorobiphenyl)	35694-08-7	37	3	1,2,3,4,5
CB206(2,2',3,3',4,4',5,5',6-nonachlorobiphenyl)	40186-72-9	9.2	1.6	1,2,3,4,5

*IUPAC number

Determination of Certified Values

Each certified value was calculated from PCB concentrations in a PCB solution determined by the following analytical methods, PCB concentrations in CRM 7905-a (blank oil), and mass ratio between the PCB solution and CRM 7905-a.for the preparation of the CRM 7904-a. The methods for determinations of PCB concentrations in the CRM 7905-a are described in its certificate.

Analytical methods:

1. [ID-GC/MS］Column: HT8-PCB (Kanto Chemical), Mass resolution: 10 000 (EI, SIM)
2. [ID-GC/MS］Column: DB-XLB (Agilent Technologies), Mass resolution: 10 000 (EI, SIM)
3. [ID-GC/MS］Column: DB-1701 (Agilent Technologies), Mass resolution: 10 000 (EI, SIM)
4. [ID-GC/MS］Column: DB-1MS (Agilent Technologies), Mass resolution: 10 000 (EI, SIM)
5. [ID-GC/MS］Column: DB-XLB (Agilent Technologies), Mass resolution: 3 000 (NCI, SIM)

Traceability

The certified values were determined by IDMS as a primary method of measurement. Because the calibration solution for the measurements was prepared from NMIJ standard solutions of CB28 (CRM 4206-a1), CB153 (CRM 4207-a1) and CB194 (CRM 4209-a1); and high purity other PCB congeners of which purities were evaluated in NMIJ, the certified values are

traceable to the International System of Units (SI).

Expiration of Certification

The certification of this CRM is valid until March 31, 2017, provided that the material is unopened and stored in accordance with the instructions given in this certificate.

Sample Form

This CRM is fuel oil (JIS K2205 class 1-2). The form of CRM is 2.5 mL of black oil sealed in an amber ampoule with argon gas.

Homogeneity

The homogeneity of the CRM was determined by analyzing 10 ampoules selected by random sampling of 600 ampoules. The PCB congeners were determined by the normal phase liquid chromatography and ID-GC/MS method, and the inhomogeneity was evaluated by ANOVA. The inhomogenity of analytes is not significant and is reflected in the uncertainty of the certified value.

Precautions for Storage

This CRM should be kept at room temperature (below 30 °C) under dark condition.

Precautions for Handling

Wear a mask, gloves and other protective gears during handling. Handle, store and dispose this CRM in accordance with laws.

Preparation Method

Commercial fuel oil (JIS K2205 class 1-2) was filtered with a glass fiber filtration paper (retention particle diameter: 0.5 μm), then CB3, CB8 reagent and technical PCB, Kaneclor were added gravimetrically (CB3, 0.501 mg/kg; CB8, 0.503 mg/kg; KC300, 1.25 mg/kg; KC400, 1.27 mg/kg; KC500, 1.27 mg/kg; and KC600, 1.27 mg/kg were added). The PCB spiked oil was homogenized by mechanical mixing and 2.5 mL of the mixture was sealed in an amber ampoule with argon gas.

Information value

a. Concentrations of PCB homologues obtained by a Japanese official method

Concentrations of PCB homologues were the results by a Japanese official method for the determination of PCBs in waste (The method (1) and (2)): Notification No. 192 of the Ministry of Welfare (1992)). To estimate concentrations of homologues, the GC/MS response factors of PCB isomers are assumed to be same.

If signal-noise ratio (S/N) of the GC/MS signal of a certain congener was lower than 3 or the signal was not higher than the signals obtained from procedural blank tests significantly, concentration of the congener is regarded as zero. The concentrations of each congener estimated from respective lowest detection limits are also shown in the table below.

The expanded uncertainty was determined using coverage factor $k = 2$, corresponding to an estimated confidence interval of approximately 95%. However, uncertainties of PCB concentrations in the calibration solutions, difference of the response factors between isomers are not included.

Because detection limit of decachlorobiphenyl by the analytical method 6 (see below) was lower than the official method, the data obtained by the method 6 was used.

PCB homologue concentrations (mass fractions) in CRM 7904-a based on the Japanese official method

	Concentration (μg/kg)	Expanded uncertainty (μg/kg)	Detection limit ** (μg/kg)
Dichlorobiphenyls	642	11	8.5
Trichlorobiphenyls	871	33	0.21
Tetrachlorobiphenyls	1475	195	0.16
Pentachlorobiphenyls	1047	53	0.15
Hexachlorobiphenyls	986	22	0.23

Heptachlorobiphenyls	732	83	0.28
Octachlorobiphenyls	229	23	0.32
Nonachlorobiphenyls	12.3	0.9	0.15
Decachlorobiphenyl***	0.14	—	—

** Concentration of each isomer estimated from the lowest detection limits

Analytical method***

6. Normal phase chromatography and ID-GC/MS (NCI)*

［Cleanup］ Solid phase extraction (benzene sulfonic acid/silica) and normal phase high performance liquid chromatography（NH_2-silica gel column and DIOL-silica gel column）

［GC/MS］ Column: DB-XLB (Agilent Technologies), Mass resolution: 3 000 (NCI, SIM)

b. Density

Density of this CRM at room temperature is 0.86266 g/mL (20 ℃) , 0.85915 g/mL (25 ℃) and 0.85564 g/mL (30 ℃). The measurement was performed with an oscillational density meter.

NMIJ Analysts

The technical manager and production manager for this CRM is T. Yarita and M. Numata, respectively. Analytical measurements for the certification of this CRM were performed at NMIJ by M. Numata, N. Hanari, K. Ishikawa, Y. Aoyagi, M. Matsuo and S. Otsuka.

Technical Information

Customers will be notified of any revision to this CRM including a change in certified value. Technical information on this CRM can be obtained from the home page and other contact routes (see below).

Reproduction of Certificate

In reproducing this certificate, it should be clearly indicated that the document is a copy.

March 2007
Hiroyuki Yoshikawa
President
National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact
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Note: This certificate is a translation of the original Japanese certificate and is not an official document.

Revision Record
October 20, 2009: The certified vaslue of CB3 (3-chlorobiphenyl) and information value of monochlorobiphenyls were deleted based on the results of the stability monitoring.