

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 7902-a

No. +++



## Polychlorinated Biphenyls in Insulation Oil (High Concentration)

This certified reference material (CRM) was produced in accordance with the NMIJ's management system in compliance with JIS Q 0034 (ISO Guide 34). It is intended for use in controlling the precision of analysis or confirming the validity of analytical methods or instruments during the 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 table below. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor ( $k$ ) of 2, which gives a level of confidence of approximately 95 %.

Certified values of PCB congeners\*

	CAS No.	Certified value, Mass fraction ( $\mu\text{g}/\text{kg}$ )	Expanded uncertainty, Mass fraction ( $\mu\text{g}/\text{kg}$ )	Analytical method
CB3 (4-chlorobiphenyl)	2051-62-9	505	21	1,2,3,4,5
CB8 (2,4'-dichlorobiphenyl)	34883-43-7	576	29	1,2,3,4
CB28 (2,4,4'-trichlorobiphenyl)	7012-37-5	174	7	1,2
CB52 (2,2',5,5'-tetrachlorobiphenyl)	35693-99-3	187	9	1,2,4
CB101 (2,2',4,5,5'-pentachlorobiphenyl)	37680-73-2	169	13	1,2,3,4
CB118 (2,3',4,4',5-pentachlorobiphenyl)	31508-00-6	126	7	1,2,3,4,5
CB138 (2,2',3,4,4',5'-hexachlorobiphenyl)	35065-28-2	134	11	1,2
CB153 (2,2',4,4',5,5'-hexachlorobiphenyl)	35065-27-1	176	7	1,2,4,5
CB180 (2,2',3,4,4',5,5'-heptachlorobiphenyl)	35065-29-3	153	8	1,2,3,4,5
CB194 (2,2',3,3',4,4',5,5'-octachlorobiphenyl)	35694-08-7	38	3	1,2,3,4,5
CB206 (2,2',3,3',4,4',5,5',6-nonachlorobiphenyl)	40186-72-9	9.3	1.6	1,2,3,4,5

\*IUPAC number

**Analysis**

Each certified value was calculated from PCB concentration in a PCB solution determined by the following analytical methods, PCB concentrations in CRM 7903-a (oil matrix), and mass ratio between the PCB solution and CRM 7903-a for the preparation of CRM 7902-a. The methods for the determination of PCB concentrations in CRM 7903-a are described in its certificate.

Analytical methods:

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

**Metrological 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 other high-purity PCB congeners the purities of which were evaluated at NMIJ, the certified values are traceable to the International System of Units (SI).

### Expiration of Certification

This certificate is valid until March 31, 2017, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

### Sample Form

This CRM is electrical insulation oil mainly composed of paraffin oil. It is in the form of yellow viscous oil, and is sealed in amber ampoules (2.5 mL each) 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 normal-phase liquid chromatography and ID-GC/MS, and the inhomogeneity was evaluated by ANOVA. The inhomogeneity of analytes is not significant and is reflected in the uncertainty of the certified value.

### Precautions for Storage

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

### Instructions for Handling

A protective mask, gloves, and other protective equipment should be worn during handling this CRM. It should be handled, stored, and disposed of in accordance with the prevailing laws. Refer to the safety data sheet (SDS) on this CRM before use.

### Preparation Method

This CRM is a commercial insulating oil (JIS C2320 class 1-2) that was used as transformer oil for 26 years and was collected from a transformer (three-phase, 300 kVA). The collected oil was filtered using a glass fiber filtration paper (retention particle diameter: 0.5 µm). Subsequently, CB3 and CB8 reagents and technical PCB, Kaneclor, were added gravimetrically (CB3, 0.501 mg/kg; CB8, 0.504 mg/kg; KC300, 1.27 mg/kg; KC400, 1.29 mg/kg; KC500, 1.29 mg/kg; and KC600, 1.28 mg/kg). The PCB-spiked oil was homogenized by mechanical mixing, and the mixture was sealed in amber ampoules (2.5 mL each) with argon gas.

The oil was collected by the General Environmental Technos Co., Ltd.

### Information Values

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

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

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

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

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

PCB homologue concentrations (mass fractions) in CRM 7902-a based on the official Japanese method

	Concentration (µg/kg)	Expanded uncertainty (µg/kg)	Detection limit** (µg/kg)
(Mono) Chlorobiphenyls	512	33	0.22

Dichlorobiphenyls	635	21	0.19
Trichlorobiphenyls	883	146	0.12
Tetrachlorobiphenyls	1451	421	0.09
Pentachlorobiphenyls	988	150	0.13
Hexachlorobiphenyls	961	115	0.30
Heptachlorobiphenyls	739	89	0.12
Octachlorobiphenyls	230	49	0.13
Nonachlorobiphenyls	13.1	1.8	0.09
Decachlorobiphenyl***	0.14	—	—

\*\*Concentration of each isomer estimated from the lowest detection limit

#### 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<sub>2</sub>-silica gel column and DIOL-silica gel column)

[GC/MS] Column: DB-XLB (Agilent Technologies), Mass resolution: 3000 (NCI, SIM)

#### b. Density

The density of this CRM at room temperature is 0.87741 g/mL (20 °C), 0.87406 g/mL (25 °C), or 0.87072 g/mL (30 °C). The measurement was performed using an oscillation density meter.

#### NMIJ Analysts

For this CRM, the technical and production managers are 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

Customer registration on the NMIJ Website (given below) will facilitate notification of any revision of the information given above. Technical reports regarding this CRM can be obtained from the contact details given below.

#### Reproduction of Certificate

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

April 1, 2015

Ryoji Chubachi  
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:  
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#### Revision history

April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."