National Institute of Advanced Industrial Science and Technology  
National Metrology Institute of Japan

Reference Material Certificate  
NMIJ CRM 8109-a  
No. +++

Polybrominated Diphenyl Ethers in Poly(vinyl chloride) Resin

This certified reference material (CRM) is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This CRM is intended for use in controlling the precision of analysis or confirming the validity of analytical methods or instruments during the analysis of decabromodiphenyl ether (DBDE) in poly(vinyl chloride) resin.

Certified Value
The certified value for DBDE in this CRM is 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%.

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Certified value, Mass fraction (mg/kg)</th>
<th>Expanded uncertainty, Mass fraction (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1163-19-5</td>
<td>333</td>
<td>13</td>
</tr>
</tbody>
</table>

Analysis
The certified value of this CRM was determined by the following analytical methods:
1) Gas chromatography / mass spectrometry (isotope dilution method)
2) High performance liquid chromatography (standard addition method)

Metrological Traceability
The certified value of this CRM was determined by two analytical methods described in the “Analysis” based on the purity of DBDE, which was characterized by mass balance method at NMIJ.

Expiration of Certification
This certificate is valid for one year from the date of shipment, provided that the material is stored in accordance with the instructions given in this certificate.

Sample Form
This CRM is in the form of a milky-white disk at room temperature. The diameter, thickness and mass of the disk are about 30 mm, 2 mm and 1.8 g, respectively. One disk is packaged in an aluminum coated pouch.

Homogeneity
The homogeneity of this CRM was evaluated by analyzing 10 disks randomly selected from about 3000 disks by the XRF. The uncertainty in the certified value includes that arising from inhomogeneity.

The homogeneity of this CRM was evaluated by intensities of X-ray fluorescence from bromine in the diameter range of about 10 mm at the center of disks selected. Ten disks were selected almost equal intervals in the order of production for the evaluation. X-ray fluorescence intensities from seven places in one disk were also evaluated. It was assumed that the X-ray fluorescence intensity was proportional to the mass fraction of bromine. The homogeneity of each element is reflected in the uncertainty of the
certified value.

**Instructions for Storage**
This CRM should be kept in the aluminum laminated pouch and should be stored at a temperature around 5 °C in a clean and dark place. It should be kept horizontal and away from stress.

**Instructions for Use**
This CRM should be only used for chemical analysis and should not be touched with bare hands. This CRM should be used more than one disk in disk form. The certified and the information values may change when light such as X-ray is irradiated to this CRM because it can degrade. The certified value and the information value may change, when this CRM is used for the irradiated analysis such as X-ray fluorescence spectroscopy (XRF).

**Precautions for Handling**
Do not use this standard substance for testing/research purposes only. Pay attention to fire and ventilation, wear protective mask, protective gloves, etc. DBDE is designated as Class I Designated Chemical Substances in the Act on the Evaluation of Chemical Substances and Regulation of Their Manufacture, etc. It is also designated as Class I Designated Chemical Substances in the Act on Confirmation, etc. of Release Amounts of Specific Chemical Substances in the Environment and Promotion of Improvements to the Management Thereof (PRTR law). Handle in compliance with these laws. Refer to the safety data sheet (SDS) on this CRM before use.

**Preparation**
This CRM was prepared by Chemical Evaluation and Research Institute, Japan (CERI). Polystyrene resin and DBDE were mixed and extruded, then disks were made by injection molding.

**Technical Information**
The estimated mass fraction of Br associated to the flame retardants in this CRM was 282 mg/kg in 2009. It was estimated from the certified value of this CRM and the composition of the flame retardant added to this CRM determined by HPLC.

**NMIJ Analysts**
The technical managers for this CRM are KINUGASA S. and SAIITO T., the production manager is MATSUYAMA S., and the analysts are KISHINE K., ORIHARA Y., KINUGASA S. and MATSUYAMA S.

**Information**
If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customer. Customer registration on the NMIJ Website (given below) will facilitate notification. 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.

**Note**
The purity of the primary standard was validated in an international comparison in Asian collaboration on reference materials (ACRM). This work was supported by a grant from New Energy and Industrial Technology Development Organization (NEDO).

April 1, 2020

ISHIMURA Kazuhiko
President
National Institute of Advanced Industrial Science and Technology
If you have any questions about this CRM, please contact
National Institute of Advanced Industrial Science and Technology,
National Metrology Institute of Japan,
Center for Quality Management of Metrology, Reference Materials Office,
1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan

Revision history
December 20, 2012: The expiration of certification was extended from “March 31, 2014” to “March 31, 2019.”
April 1, 2015: “Metrology Management Center” was renamed to “Center for Quality Management of Metrology.”
January 12, 2018: The description in “Expiration of Certification” was changed to “one year after the date of shipment.”
Instruction on attachment

Poly(Vinyl Chloride) Resin

This poly(vinyl chloride) resin attached to NMIJ CRM 8109-a was made from poly(vinyl chloride) resin used for the matrix of NMIJ CRM 8109-a. This poly(vinyl chloride) resin was made by injection molding with same production condition of NMIJ CRM 8109-a. This poly(vinyl chloride) was not mixed with decabromodiphenyl ether (DBDE). Storage condition is same as the NMIJ CRM 8109-a.

Appendix
This poly(vinyl chloride) was a result of the project aided from the New Energy and Industrial Technology Development Organization (NEDO) in 2006.

If you have any questions about this material, please contact:
National Institute of Advanced Industrial Science and Technology,
National Metrology Institute of Japan,
Center for Quality Management of Metrology, Reference Materials Office,
1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan
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