National Institute of Advanced Industrial Science and Technology
National Metrology Institute of Japan
Reference Material Certificate
NMIJ CRM 8155-a
No. +++
Perfluoroalkyl Substances in ABS Resin

This certified reference material (CRM) is produced in accordance with the NMJ’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 and for validating analytical methods and instruments during quantification of perfluorooctanesulfonic acid (PFOS) and its salts in ABS resin and similar materials.

Certified Value
The certified value, concentration of linear perfluorooctanesulfonic acid expressed as mass fractions, 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</th>
<th>Expanded uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mass fraction (mg/kg)</td>
<td>Mass fraction (mg/kg)</td>
</tr>
<tr>
<td>1763-23-1</td>
<td>33.1</td>
<td>5.0</td>
</tr>
</tbody>
</table>

The certified value is expressed as free acid, however potassium perfluorooctanesulfonate was added to ABS resin.

Analysis
The certified value of this CRM was based on the analytical results of PFOS by dissolution and isotope dilution-liquid chromatography/tandem mass spectrometry (ID-LC/MS/MS) described as follows;
1. [Dissolution] Solvent, tetrahydrofuran
   [Clean-up] Reprecipitation of polymer by addition of methanol
2. [Dissolution] Solvent, chloroform
   [Clean-up] Reprecipitation of polymer by addition of methanol. Replacement of solvent with distilled water and clean-up by solid phase extraction (ion-exchange).
   [LC/MS/MS] Column, Octadecylsilyl (ODS); electrospray ionization (ESI); multiple reaction monitoring (MRM)

Metrological Traceability
The certified value of this CRM was determined by IDMS. NMIJ CRM 4220-a (Potassium Perfluorooctanesulfonate in Methanol) was used as the calibration solution for the determination. Therefore, the certified value is traceable to the International System of Units (SI). All sample preparations were carried out by gravimetric method, using the balance calibrated by JCSS (Japanese Calibration Service System).

Expiration of Certification
This certificate is valid for 6 months 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 plate about 20 mm square, a thickness of 2 mm and a mass of 1.8 g. Three plates are packed in an aluminum-layered bag.

Homogeneity
The homogeneity of the CRM was determined by analyzing 10 packages randomly sampled from 350 prepared packages. The inhomogeneity of the analyte was evaluated by ANOVA. The homogeneity is reflected in the uncertainty of the certified value.

Instructions for Storage
This CRM should be stored at a temperature between 15 °C and 25 °C and shielded from light.

Instructions for Use
This CRM is for laboratory use only. This CRM should be used as soon as possible after the opening of a bag. When a pulverized sample is analyzed, the homogeneous sample prepared from a whole single plate should be used. More than 0.1 g of the pulverized material should be used.

Precautions for Handling
Keep away from heat and ignition sources. Wear protective equipment, such as a safety mask and gloves when handling. The use, handling and storage of this CRM should be performed while observing the laws regulating the components of this CRM. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation
This CRM was prepared by mixing commercial ABS resin powder, potassium perfluorooctanesulfonate, potassium perfluorobutanesulfonate, sodium perfluorooctanoate, dechlorane plus and tetrabromobisphenol A. The mixing to homogenization was carried out by a kneading machine for a total of two times. The mixed materials were injection-molded with an injection molding machine; then, the injection-molded plates were cut as square plates. This CRM was prepared by the DJK Corporation.

Technical Information
This CRM contains branched perfluorooctanesulfonic acid (17.4 mg/kg). The concentration of this substance in this CRM was estimated by LC/MS/MS. This CRM contains chlorine (321 mg/kg) and bromine (575 mg/kg). Concentrations of these elements in this CRM were estimated by neutron activation analysis performed by using the facilities of the Research Reactor Institute, Kyoto University.

NMIJ Analysts
The technical manager for this CRM is NUMATA M. The production manager is HANARI N. The analysts are HANARI N., ITOH N., IWASAWA R., AYOAGI Y., OHATA M., MIURA T., KATO H., BAO X., and IHARA T.

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.

April 1, 2020

ISHIMURA Kazuhiko
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.”
May 14, 2015: The description in “Expiration of Certification” was changed to “6 months from the date of shipment.”