

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 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 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, linear perfluorooctanesulfonic acid expressed as mass fractions, is given in the table below. The uncertainty of the certified value is the expanded uncertainty obtained by multiplying the combined standard uncertainty by a coverage factor (k) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Substance	CAS No.	Certified value Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)
Perfluorooctanesulfonic acid (Heptadecafluoro-1-octanesulfonic acid)	1763-23-1	33.1	5.0

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;

- [Dissolution] Solvent, tetrahydrofuran
[Clean-up] Reprecipitation of polymer by addition of methanol
[LC/MS/MS] Column, Octadecylsilyl (ODS); electrospray ionization (ESI); multiple reaction monitoring (MRM)
- [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.

Description of the material

This CRM is ABS resin containing potassium perfluorooctanesulfonate, potassium perfluorobutanesulfonate, sodium perfluorooctanoate, dechlorane plus and tetrabromobisphenol A, and 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.

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 mass fraction of this substance in this CRM was estimated by LC/MS/MS. This CRM contains chlorine (321 mg/kg) and bromine (575 mg/kg). Mass fractions 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., AOYAGI 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 customers. 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.

Date of Shipment: Xxxxx xx, 20xx

8155a00 No. +++-140326-220526

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.”