

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 8102-a
No. +++

Heavy Metals (Cd, Cr, Pb) in ABS Resin (Low-concentration Pellets)

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 confirming the validity of analytical methods and instruments during the quantitative determination of Cd, Cr, and Pb in ABS resin and similar polymers.

Certified Values

The certified values of Cd, Cr, and Pb in this CRM are given in the table below. The drying method is described in this certificate. 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 value, Mass fraction (mg/kg) | Expanded uncertainty, Mass fraction (mg/kg) |
|----|---|--|
| Cd | 10.77 | 0.20 |
| Cr | 27.87 | 0.35 |
| Pb | 108.9 | 0.89 |

Analysis

Each certified value was determined by the following analytical methods and is traceable to the International System of Units (SI):

- (1) Microwave digestion using sulfuric acid and nitric acid / isotope dilution mass spectrometry (Cd, Cr, and Pb)
- (2) Dry-ashing digestion followed by open-system dissolution using nitric acid and hydrogen peroxide / inductively coupled plasma mass spectrometry (Cd, Cr, and Pb)
- (3) Microwave digestion using nitric acid and perchloric acid / inductively coupled plasma atomic emission spectrometry (Cd and Pb).

Metrological Traceability

Each certified value was determined by multiple methods including the isotope dilution-mass spectrometry as a primary method of measurement using NMIJ primary standard solutions of Cd, Cr, and Pb, and is traceable to the International System of Units (SI).

Mutual Recognition Arrangement under Meter Convention

This certificate is consistent with the calibration and measurement capabilities (CMCs) that are included in Appendix C of the Mutual Recognition Arrangement (MRA) drawn up by the International Committee for Weights and Measures (CIPM). Under the MRA, all participating institutes recognize the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C (as for Appendix C of MRA, see <http://kcdb.bipm.org/AppendixC/default.asp>).

Expiration of Certification

This certificate is valid until March 31, 2022, provided that the material remains unopened and is stored in accordance with the

instructions given in this certificate.

Sample Form

This CRM is in the form of small pellets and it of ca. 25 g in net volume is kept in an amber glass bottle.

Homogeneity

The homogeneity of the CRM was determined by analyzing 12 bottles selected at approximately same intervals in the order of bottling. The elements (Cd, Cr, and Pb) were determined by microwave digestion using sulfuric acid and nitric acid–inductively coupled plasma mass spectrometry. The homogeneity of each element is reflected in the uncertainty of the certified value.

Instructions for Storage

This CRM should be stored at a temperature between 15 °C and 35 °C, and shielded from light.

Instructions for Use

Prior to use, the sample should be mixed well by slow rolling. It should be dried for 1 h at 80 °C and then allowed to stand at room temperature for 1 h in a silica-gel desiccator. The recommended minimum sample mass for analysis is 0.10 g. Please note that this sample contains metallic elements other than the certified ones (Cd, Cr, and Pb).

Precautions for Handling

Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

Commercial ABS resin was powdered by cryomilling and mixed with CdO and PbCrO₄ powders. Small pellets were then produced from the resulting mixture using a twin screw kneading extruder. The pellet production process was repeated two more times.

Technical Information

The following values are shown as information only. Interlaboratory analysis using the candidate reference material of this CRM was carried out in 19 laboratories (18 laboratories for Cr). For each element, the median and estimate of standard deviation for the result distribution calculated on the basis of dispersion from the median are summarized in the table below.

| | Median of interlaboratory analysis, Mass fraction (mg/kg) | Estimate of standard deviation of result distribution, Mass fraction (mg/kg) |
|----|--|--|
| Cd | 10.31 | 0.53 |
| Cr | 26.64 | 1.64 |
| Pb | 106.6 | 5.5 |

NMIJ Analysts

For this CRM, the technical and production managers are HIOKI A. and the analyst is OHATA M.

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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Phone: +81-29-861-4059; Fax: +81-29-861-4009, <https://unit.aist.go.jp/nmij/english/refmate/>

Revision history

- February 26, 2009: The expiration date of this certification was extended to March 31, 2015, from March 31, 2010. The description on Mutual Recognition Arrangement under Meter Convention was added.
- January 20, 2014: The expiration date of this certification was extended to March 31, 2022, from March 31, 2015.
- April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."