

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 8103-a  
No. +++

## Heavy Metals (Cd, Cr, Pb) in ABS Resin - High Concentration Pellet

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 analyses and validating analytical methods and instruments used in the quantitative analysis 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 instructions are described in this certificate. The uncertainty of the certified value is the half-width of the expanded uncertainty intervals 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	106.9	1.37
Cr	269.5	4.5
Pb	1084	9.4

**Analysis**

Each certified value of this CRM was determined by the following analytical methods:

- (1) Microwave digestion using sulfuric acid and nitric acid / isotope dilution-inductively coupled plasma 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), and
- (3) Microwave digestion using nitric acid and perchloric acid / inductively coupled plasma atomic emission spectrometry (Cd and Pb).

**Metrological Traceability**

Each certified value of this CRM is determined by more than one method including the isotope dilution-mass spectrometry, which is the primary method of measurement, with the NMIJ primary standard solutions of Cd, Cr, and Pb. The certified values of this CRM, therefore, are 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**

The certification of this CRM 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 this CRM was determined by analyzing 12 bottles selected at approximately the same intervals in order of bottling. The elements (Cd, Cr and Pb) were determined the by microwave digestion using sulfuric acid and nitric acid / inductively coupled plasma mass spectrometry. The homogeneity of each element has been incorporated into the uncertainties of the certified values. This CRM is homogeneous within the range of the uncertainties of the certified values.

**Instructions for Storage**

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

**Instructions for Use**

The bottle of this CRM should be turned upside down several times to mix the CRM before a sample is taken. The sample taken should be dried for one hour at 80 °C, and then kept at room temperature for one hour in a silica-gel desiccator. The recommended sample mass is 0.10 g or more for each analysis. This CRM contains metallic elements in addition to the certified elements (Cd, Cr, and Pb).

**Precautions for Handling**

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

**Preparation**

Commercially-available cryo-milled ABS resin and CdO and PbCrO<sub>4</sub>, both of which were in the form of powder, were mixed, and from this mixture, pellets were produced. The agitating/mixing/pelletizing process was repeated two more times.

**Technical Information**

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

	Median of inter-laboratory analysis, mass fraction (mg/kg)	Estimated standard deviation of result distribution, mass fraction (mg/kg)
Cd	105.4	4.4
Cr	267.4	13.0
Pb	1080	31

**NMIJ Analysts**

The technical and production managers for this CRM 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

March 24, 2009: The expiration of certification was extended from “March 31, 2010” to “March 31, 2015.”

The description on “Metrological Traceability” and “Mutual Recognition Arrangement under Meter Convention” were added.

January 20, 2014: The expiration of certification was extended from “March 31, 2015” to “March 31, 2022.”

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