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

Reference Material Certificate
NMIJ CRM 8112-a
No. +++

Heavy Metals (Cd, Cr, Hg, Pb) in ABS Resin - Low 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 analysis and validating analytical methods and instruments during the quantitative determination of Cd, Cr, Hg and Pb in ABS resin and similar polymers.

Certified Values
The certified values of Cd, Cr, Hg and Pb in this CRM are given in the table below. The drying instruction 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%.

<table>
<thead>
<tr>
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<th>Certified value, Mass fraction (mg/kg)</th>
<th>Expanded uncertainty, Mass fraction (mg/kg)</th>
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</thead>
<tbody>
<tr>
<td>Cd</td>
<td>9.383</td>
<td>0.223</td>
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<tr>
<td>Cr</td>
<td>94.47</td>
<td>1.11</td>
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<tr>
<td>Hg</td>
<td>94.10</td>
<td>2.87</td>
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<tr>
<td>Pb</td>
<td>94.98</td>
<td>0.87</td>
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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, Hg and Pb),
(2) Dry-ashing digestion followed by open-system dissolution using nitric acid and hydrogen dioxide / inductively coupled plasma optical emission spectrometry (Cd, Cr and Pb),
(3) Microwave digestion using sulfuric acid and nitric acid / inductively coupled plasma mass spectrometry (Cd, Cr, Hg and Pb).

Metrological Traceability
Each certified value of this CRM was determined by more than one method, including isotope dilution – mass spectrometry as a primary method of measurement with NMIJ primary standard solutions of Cd, Cr, Hg and Pb. The certified values 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
This certificate is valid for 3 years 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 small pellets. The net mass of 25 g is kept in a brown glass bottle.

Homogeneity
The homogeneity of this CRM was determined by analyzing 14 bottles selected at approximately same intervals in the order of bottling. The elements (Cd, Cr, Hg 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 by slow rolling. It should be dried for one hour at 80 °C and then maintained at room temperature for one hour in a silica-gel desiccator. The recommended sample mass is 0.10 g or more for one analysis. Please note that this sample contains hundreds mg/kg of Br.

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 (deca brominated biphenyl ether) 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
Commercial ABS resin and powders of CdO, PbCrO₄, chromium(III) acetylacetonate and HgS were mixed and then pellets were produced from extruding the mixture. The extruding process was repeated two more times. These master pellets produced were mixed with commercial ABS resin and the extruding process was repeated three times.

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

<table>
<thead>
<tr>
<th>Revision history</th>
<th>Details</th>
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<tbody>
<tr>
<td>March 10, 2011</td>
<td>The expiration of certification was extended from March 31, 2012 to March 31, 2017. The description on Mutual Recognition Arrangement (CIPM MRA) was added.</td>
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<tr>
<td>April 1, 2015</td>
<td>“Metrology Management Center” was renamed to “Center for Quality Management of Metrology.”</td>
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<tr>
<td>November 12, 2015</td>
<td>The description in “Expiration of Certification” was changed to “3 years after the date of shipment.”</td>
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