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

Reference Material Certificate
NMIJ CRM 8133-a
No. +++

Heavy Metals (Cd, Cr, Hg, Pb) in PP Resin Pellet- High Concentration

This certified reference material (CRM) was produced in accordance with the NMIJ’s management system, and in compliance with JIS Q 0034 (ISO GUIDE 34). This CRM is intended for use in controlling the precision of analysis and for validating analytical methods and instruments during the quantitative determination of Cd, Cr, Hg and Pb in polypropylene 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>
<th>Substance</th>
<th>Certified value, Mass fraction (mg/kg)</th>
<th>Expanded uncertainty, Mass fraction (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cd</td>
<td>94.26</td>
<td>1.39</td>
</tr>
<tr>
<td>Cr</td>
<td>895.2</td>
<td>9.6</td>
</tr>
<tr>
<td>Hg</td>
<td>941.5</td>
<td>19.6</td>
</tr>
<tr>
<td>Pb</td>
<td>949.2</td>
<td>7.5</td>
</tr>
</tbody>
</table>

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 / inductively coupled plasma atomic emission spectrometry (Cd and Pb).
(3) Microwave digestion using 70% nitric acid / inductively coupled plasma atomic emission 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 the 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 until March 31, 2021, provided that the material remains unopened and stored in accordance with the instructions given in this certificate.

Sample Form
This CRM consists of small pellets in a brown glass bottle. The net mass is 25 g.

Homogeneity
The homogeneity of this CRM was determined by analyzing 14 bottles selected at approximately equal intervals on the basis of the order of bottling. The elements (Cd, Cr, Hg and Pb) were determined by microwave digestion using 70% nitric acid / inductively coupled plasma mass spectrometry (using an external calibration curve and an internal standard). The homogeneity of each element is reflected in the uncertainty of the certified value.

Instructions for Storage
This CRM should be kept at room temperature (15 °C to 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 tens 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 1 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 Method
The base resin was prepared by mixing a commercial PP resin and powders of CdO, PbCrO₄, chromium(III) acetylacetonate, and HgS were mixed and then pellets were produced from the mixture. The pellet-producing process was repeated two more times.

NMIJ Analysts
The technical and production manager is A. Hioki, and the analysts are M. Ohata and A. Hioki.

Technical Information
Customer registration on the NMIJ Website (given below) will facilitate notification of any revision of the information given above. 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, 2015
Ryoji Chubachi
President
National Institute of Advanced Industrial Science and Technology
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

Revision history
August 10, 2012: The expiration of certification was extended from March 31, 2013 to March 31, 2021.
April 1, 2015: “Metrology Management Center” was renamed to “Center for Quality Management of Metrology”