

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 8136-a  
No. +++)

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

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 X-ray fluorescence analysis of Cd, Cr, Hg and Pb in PP resin Disk and similar polymers.

**Certified Values**

The certified values of Cd, Cr, Hg and Pb in this CRM are given in the table below. This CRM should be used without any drying. 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	93.7	3.1
Cr	890.6	10.7
Hg	952	40
Pb	943.5	9.2

**Analysis**

Each certified value was determined by the following analytical method:

Microwave digestion using sulfuric acid and nitric acid / isotope dilution - inductively coupled plasma mass spectrometry.

**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 from the date of shipment to March 31, 2021, 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 a disk with diameter 30 mm and thickness 2 mm, kept in a plastic case.

**Homogeneity**

The homogeneity of the CRM was determined by analyzing 16 disks. The elements (Cd, Cr, Hg and Pb) were determined by X-ray fluorescence method. The homogeneity of each element is reflected to the uncertainty of the certified value. The homogeneity of lead within a surface was also determined and reflected to the uncertainty of each 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**

From the homogeneity, the certified values of this CRM represent the sample concentrations of the surface area more than 20 mm<sup>2</sup> (an area corresponding to a circle with diameter 5 mm). In measuring X-ray fluorescence, X-ray should be irradiated from the opposite side to the side on which there are 7 circles with diameter 6 mm formed during the injection-molding process. It is necessary to consider that the sensitivity in the X-ray fluorescence is changed depending on a thickness and a kind of the resin. Please note that this sample contains several decades 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**

Commercial PP resin and powders of CdO, PbCrO<sub>4</sub>, chromium(III) acetylacetonate and HgS were mixed and then pellets were produced from extruding the mixture. The extruding process was repeated two more times. From the obtained pellets, the disks with thickness 2 mm and diameter 30 mm were produced by the injection-molding method.

**NMIJ Analysts**

The technical and production managers for this CRM are HIOKI A. and the analysts are OHATA M. and HIOKI A.

**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  
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/xzenglish/refmate/>

Revision history

- August 10, 2012: The expiration of certification was extended from March 31, 2014 to March 31, 2021.  
The description on “Mutual Recognition Arrangement under Meter Convention” was added.
- April 1, 2015: “Metrology Management Center” was renamed to “Center for Quality Management of Metrology.”

Sample