National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan

Reference Material Certificate

NMIJ CRM 8123-a
No. +++

Heavy Metals (Cd, Cr, Hg, Pb) in PVC Resin Pellet - 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 quantitative determination of Cd, Cr, Hg, and Pb in polyvinylchloride 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 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	95.62	1.39
Cr	949.0	9.7
Hg	937.0	19.4
Pb	965.5	6.6

Analysis

Each certified value 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, Cr, 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 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

The certificate is valid until March 31, 2021, provided that the material remains unopened and is stored in accordance with the

Sample

instructions given in this certificate.

Sample Form

This CRM is in the form of a small pellets and 25 g in net mass is kept in a brown glass bottle.

Homogeneity

Regarding the elements (Cd, Hg, and Pb), 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 were determined by microwave digestion using sulfuric acid and nitric acid/inductively coupled plasma mass spectrometry (using an external calibration curve and an internal standard). Regarding Cr, the homogeneity of the CRM was determined by analyzing 7 bottles selected at approximately equal intervals on the basis of the order of bottling. The element was determined by microwave digestion using sulfuric acid and nitric acid/isotope dilution–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 1 h at 80 °C and then maintained at room temperature for 1 h in a silica-gel desiccator. The recommended sample mass is 0.10 g or more for one analysis.

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 (decabrominated 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

The base resin was prepared by mixing a commercial PVC resin with diisononyl phthalate (DINP), a stabilizer, and so on. The base resin and powders of CdO, $PbCrO_4$, Cr(III) acetylacetonate, and HgS were mixed and then pellets were produced from the mixtures. The pellet-producing process was repeated two more times.

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.

Sample

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,

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."

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Sample