[This document is just explanation translated from the original Japanese certificate and some information is omitted from it.]

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) was produced on the basis of NMIJ's management system under JIS Q 0034 (ISO GUIDE 34), and can be used to control the precision of analysis or to confirm the validity of analytical methods or instruments during the X-ray fluorescence analysis of Cd, Cr, Hg and Pb in ABS resin or similar polymers.

## **Certified Values**

The certified values of Cd, Cr, Hg and Pb in this CRM are given in the following Table. This CRM should be used without any drying. Each expanded uncertainty was determined with the coverage factor k = 2; it defines an interval estimated to have a level of confidence of approximately 95%.

	Certified value,	Expanded uncertainty,
	mass fraction (mg/kg)	mass fraction (mg/kg)
Cd	93.7	3.1
Cr	890.6	10.7
Hg	952	40
Pb	943.5	9.2

## **Determination of Certified Values**

Each certified value was determined by the following analytical method:

(1) Microwave digestion using sulfuric acid and nitric acid / isotope dilution – inductively coupled plasma mass spectrometry.

#### **Metrological Traceability**

Each certified value was determined by isotope dilution – mass spectrometry as a primary method of measurement with NMIJ primary standard solutions of Cd, Cr, Hg and Pb and is traceable to the International System of Units (SI).

## Mutual Recognition Arrangement (CIPM MRA)

The measurement capabilities used for this CRM are covered in the Calibration and Measurement Capabilities registered in the BIPM database (<a href="http://kcdb.bipm.org/AppendixC/default.asp">http://kcdb.bipm.org/AppendixC/default.asp</a>) under the Metre Convention on the basis of the Mutual Recognition Arrangement (CIPM MRA) for national measurement standards and for calibration and measurement certificates issued by NMIs.

## **Expiration of Certification**

The certification of this CRM is valid until March 31, 2021, provided that the CRM is unopened and stored in accordance with the instructions given in this certificate.

#### Sample Form

The form of this CRM is a disk with diameter of 30 mm and thickness of 2 mm, kept in a plastic case.

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## **Homogeneity**

The homogeneity of the CRM was determined by analysing 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.

#### **Notice for Storage**

This CRM should be kept at room temperature (15 degree C - 35 degree C) and should not be shined directly upon.

#### Notice for Use

In terms of the homogeneity, the certified values of this CRM represent the sample concentrations of the surface area more than  $20.0 \text{ mm}^2$  (an area corresponding to a circle with diameter of 5.0 mm). In measuring X-ray fluorescence, X-ray should be radiated on the clear side on which there is no circle mark with diameter of 6 mm formed during the injection-molding process. Please note that this sample contains tens mg/kg of Br.

#### **Preparation Method**

Commercial PP resin and powders of CdO, and PbCrO $_4$ , 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 of 2 mm and diameter of 30 mm were produced by the injection-molding method.

#### **NMIJ Analysts**

The technical and production manager was A. Hioki and the analyst was M. Ohata.

## **Technical Information**

If important changes on this CRM including any change of certified values happen, it will be informed to the customers, who can easily obtain such information by "CRM user registration" through the home page. Technical information on this CRM will be obtained from the home page and the other contact routes (*vide infra*).

### **Reproduction of Certificate**

In case of producing a duplicate of this certificate, the fact of reproduction should be clearly indicated on it.

March 24, 2009

Hiroyuki Yoshikawa
President,
National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact
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Revision history

August 10, 2012 The expiration of certification was extended from March 31, 2014 to March 31, 2021.

August 10, 2012 The description on Mutual Recognition Arrangement (CIPM MRA) was added.

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