

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 8152-a
No. +++

Polyvinyl Chloride (Phthalate Esters in PVC Resin Pellet)

This certified reference material (CRM) was produced in accordance with the NMIJ's management system and in compliance with ISO GUIDE 34:2009 and ISO/IEC 17025:2005. This CRM is intended for use in controlling the precision of analysis and confirming the validity of analytical methods and instruments during the analysis of phthalates in plastics.

Certified Values

The certified values for the phthalates of this CRM are given in the table below. 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 %.

| | CAS No. | Certified value, Mass fraction (mg/kg) | Expanded uncertainty, Mass fraction (mg/kg) |
|--|----------|---|--|
| Diethyl Phthalate | 84-66-2 | 918 | 57 |
| Di- <i>n</i> -Butyl Phthalate | 84-74-2 | 934 | 58 |
| Butyl Benzyl Phthalate | 85-68-7 | 897 | 57 |
| Dicyclohexyl Phthalate | 84-61-7 | 896 | 54 |
| Di-2-Ethylhexyl Phthalate (Bis(2-Ethylhexyl) Phthalate) | 117-81-7 | 913 | 58 |

Analysis

The certified values of this CRM were weighted mean of the results of the following analytical methods:

- (1) Isotope dilution–gas chromatography–mass spectrometry.
- (2) High-performance liquid chromatography (HPLC).

Metrological Traceability

Each certified value was determined by two methods, including isotope dilution–mass spectrometry as a primary method of measurement. NMIJ CRM 4022-b (diethyl phthalate), NMIJ CRM 4023-a (di-*n*-butyl phthalate), NMIJ CRM 4029-a (butyl benzyl phthalate), NMIJ CRM 4027-a (dicyclohexyl phthalate), and NMIJ CRM 4024-a (di-2-ethylhexyl phthalate) were used as the primary standards. The certified value of diethyl phthalate is traceable to the International System of Units (SI).

Expiration of Certification

This certificate is valid for one year 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 grayish white pellets. The net mass of 20 g is kept in an amber glass bottle. The bottle is sealed with argon gas in an aluminum-laminated plastic bag.

Homogeneity

The homogeneity of this CRM was determined by HPLC analysis of 10 bottles randomly selected from 595 bottles. About 0.1 g of pellets was dissolved into tetrahydrofuran and it was sedimented by methanol. The supernatant of this solution was analyzed by HPLC. The homogeneity was determined for the sum of mass fraction of di-2-ethylhexyl phthalates and di-*n*-octyl phthalates. The homogeneity of the phthalates is reflected in the uncertainty of the certified value.

Instructions for Storage

This CRM should be kept in the aluminum-laminated bag at a temperature of 35 °C or less in a dark place. Do not contact this CRM to other plastics such as a bottle cap for long period. After use, replace the gas in the bottle and in the plastic bag with argon and keep airtight.

Instructions for Use

From the homogeneity, a minimum sample size of 0.1 g is recommended.

Precautions for Handling

This CRM should only be used for testing/research. Wear a gloves and other protective equipment during handling. This CRM contains Class 1 designated chemical substances listed in the “Act on Confirmation, etc. of Release Amounts of Specific Chemical Substances in the Environment and Promotion of Improvements to the Management Thereof (Law concerning Pollutant Release and Transfer Register / PRTR).” Entrust disposal of this reference material to a professional waste disposal company licensed by prefectural governor. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

This CRM was prepared by excluding from the mixture of poly(vinyl chloride) resin, stabilizer, lubricant, plasticizer (acetyl tributyl citrate; CAS No. 77-90-7, phthalates and di-2-ethylhexyl adipate) and other polymer additives.

Technical Information

This CRM contains dimethyl phthalates, diisobutyl phthalate, di-*n*-octyl phthalate and di-2-ethylhexyl adipate. The mass fractions of them at the time of the certification were given in the table below. These values were determined by methods (1) and (2).

| | CAS No. | Informational value, Mass fraction (mg/kg) |
|--|----------|--|
| Dimethyl Phthalate | 131-11-3 | 913 |
| Diisobutyl Phthalate | 84-69-5 | 901 |
| Di- <i>n</i> -Octyl Phthalate (Bis(<i>n</i> -Octyl) Phthalate) | 117-84-0 | 923 |
| Di-2-Ethylhexyl Adipate (Bis(2-Ethylhexyl) Adipate) | 103-23-1 | 934 |

NMIJ Analysts

The technical manager for this CRM is T. Saito, the production manager is S. Matsuyama, and the analysts are S. Matsuyama and Y. Orihara.

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, 2015

Ryoji Chubachi
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

April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."