

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 8151-a

No. +++



Polypropylene (Phthalate Esters in PP 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 validating analytical methods and instruments used during the analysis of phthalate in plastics.

Certified Values

The certified values of phthalates in 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)
Di- <i>n</i> -butyl phthalate	84-74-2	963	16
Butyl benzyl phthalate	85-68-7	962	25
Di-2-ethylhexyl phthalate [Bis(2-ethylhexyl) phthalate]	117-81-7	1018	17

Analysis

These certified values were determined by the following analytical methods:

- 1) Extracted in heated toluene and analyzed with gas chromatography/mass spectrometry (isotope dilution method)
- 2) Ultrasonic extraction and analyzed with high-performance liquid chromatography

Metrological Traceability

Each certified value was determined by two methods, including isotope dilution-mass spectrometry as the primary method of measurement, with NMIJ primary standards of phthalates and is traceable to the International System of Units (SI).

Indicative Value

The indicative value of di(2-ethylhexyl) adipate in this CRM is given in the table below. The uncertainty of the indicative 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.	Reference value, Mass fraction (mg/kg)
Di-2-ethylhexyl adipate [Bis(2-ethylhexyl) adipate]	103-23-1	979 ± 57

This indicative value was determined by extraction in heated toluene and analyzed with gas chromatography/mass

spectrometry (isotope dilution method).

Expiration of Certification

This certificate is valid until March 31, 2017, 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. The net mass of each pellet is 5 g and they are kept in a brown glass bottle. The bottle is packed in an aluminum-coated pouch.

Homogeneity

The homogeneity of the CRM was determined by HPLC analysis of 10 bottles randomly selected from 116 bottles. The phthalates were extracted from the pellets (0.10 g each) with the ultrasonic extraction method. The homogeneity is reflected in the uncertainty of the certified value.

Instructions for Storage

This CRM should be stored upright at less than 5 °C in the dark.

Precautions for Use

This CRM should only be used for chemical analysis and should not be touched with bare hands. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation Method

Commercial polypropylene resin, phthalates, and di(2-ethylhexyl)adipate were mixed and then pellets were produced by extruding the mixture. The extruding process was repeated twice.

Information

The informational value of di(*n*-octyl) phthalate in this CRM is given in the table below:

	CAS No.	Informational value, mass fraction (mg/kg)
Di- <i>n</i> -octyl phthalate [Bis(<i>n</i> -octyl) phthalate]	117-84-0	1024

This informational value was determined by the following analytical methods:

- 1) Gas chromatography/mass spectrometry (isotope dilution method)
- 2) High-performance liquid chromatography

NMIJ Analysts

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

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:
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National Metrology Institute of Japan,
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Phone: +81-29-861-4059; Fax: +81-29-861-4009, <https://www.nmij.jp/english/service/C/>

Revision history

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