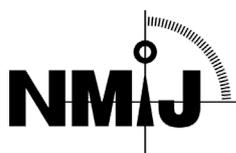


National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 8156-a(02)

No. +++



Polyvinyl Chloride (Phthalate Esters in PVC Resin Pellet Low 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 for validating analytical methods and instruments during quantification of phthalates in polyvinyl chloride resin and similar materials.

Certified Values

The certified values, mass fractions of phthalates, are given in the table below. The uncertainty of the certified value is the expanded uncertainty obtained by multiplying the combined standard uncertainty by a coverage factor (k) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Substance	CAS No.	Certified value Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)
Diethyl phthalate	84-66-2	93	18
Dipropyl phthalate (Di- <i>n</i> -propyl phthalate)	131-16-8	90	15
Dibutyl phthalate (Di- <i>n</i> -butyl phthalate)	84-74-2	92	17
Diisobutyl phthalate	84-69-5	91	14
Benzyl butyl phthalate	85-68-7	90	14
Dipentyl phthalate (Di- <i>n</i> -pentyl phthalate)	131-18-0	94	16
Dihexyl phthalate (Di- <i>n</i> -hexyl phthalate)	84-75-3	93	17
Dicyclohexyl phthalate	84-61-7	92	15
Bis(2-ethylhexyl) phthalate (Di-2-ethylhexyl phthalate)	117-81-7	95	16

Analysis

The certified values of this CRM were means of the analytical results of phthalates by isotope dilution-gas chromatography/mass spectrometry (ID-GC/MS) and isotope dilution-liquid chromatography/mass spectrometry (ID-LC/MS).

Metrological Traceability

The certified values of this CRM were determined by isotope dilution-mass spectrometry which was one of the primary methods of measurement. Diethyl phthalate (NMIJ CRM 4022-b), di-*n*-propyl phthalate (NMIJ CRM 4025-a), di-*n*-butyl phthalate (NMIJ CRM 4023-a), benzyl butyl phthalate (NMIJ CRM 4029-a), di-*n*-pentyl phthalate (NMIJ CRM 4026-a),

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di-*n*-hexyl phthalate (NMIJ CRM 4027-a), dicyclohexyl phthalate (NMIJ CRM 4028-a), and di-2-ethylhexyl phthalate (NMIJ CRM 4024-a), and diisobutyl phthalate whose purity was evaluated with quantitative NMR at NMIJ were used as the primary standards of the calibration solution for quantification prepared by gravimetric preparation. The certified values, therefore, are 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 this remains unopened and CRM is stored in accordance with the instructions given in this certificate.

Description of the Material

This CRM is in the form of a grayish white pellets of polyvinyl chloride and a 10 g net mass is kept in an amber glass bottle. The amber glass bottle is sealed with argon gas in an aluminum-layered bag.

Instructions for Storage

This CRM should be stored in a clean place at temperatures of 15 °C to 35 °C and protected from light. Do not contact by placing this CRM horizontally to other plastics such as a bottle cap for long period.

Instructions for Use

This CRM is for laboratory use only. This CRM should be used promptly once a bottle is opened. Considering the homogeneity, a minimum sample mass of 0.2 g should be used to ensure valid results.

Precautions for Handling

Keep away from heat and ignition sources. Wear personal protective equipment, such as a safety mask and gloves when handling. The use, handling, and storage of this CRM should be performed while observing the laws regulating the components of this CRM. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

This CRM was prepared by excluding from the mixture of polyvinyl chloride resin, stabilizer (2-ethylhexyl 4,4-dibutyl-10-ethyl-7-oxo-8-oxa-3,5-dithia-4-stannatetradecanoate; CAS No. 10584-98-2), twelve phthalates, lubricant, and other polymer additives. The excluding process was conducted by Tokuyama Sekisui Co., Ltd.

Technical Information

This CRM contains dimethyl phthalate, diheptyl phthalate, and dioctyl phthalate. The mass fractions of them on February 24, 2022 were given in the table below. These values were determined by the methods described in “Analysis”, using commercially available reagents as calibration standards.

Substance	CAS No.	Mass fraction (mg/kg)
Dimethyl phthalate	131-11-3	95
Diheptyl phthalate (Di- <i>n</i> -heptyl phthalate)	3648-21-3	93
Dioctyl phthalate (Di- <i>n</i> -octyl phthalate)	117-84-0	97

NMIJ Analysts

The technical manager and the production manager for this CRM are HANARI N., and the analysts are HANARI N., MATSUYAMA S., AOYAGI Y., ORIHARA Y., NAKAMURA K., YAMAZAKI E., YAMAZAKI T., KITAMAKI Y. and ITOH N.

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Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customers. 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.

March 14, 2025

ISHIMURA Kazuhiko
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:
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