National Institute of Advanced Industrial Science and Technology
National Metrology Institute of Japan

Reference Material Certificate
NMIJ CRM 8152-b
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 17034:2016 and ISO/IEC 17025:2017. 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, concentration of phthalates expressed as mass fractions, 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%.

<table>
<thead>
<tr>
<th>CAS No.</th>
<th>Certified value, Mass fraction (mg/kg)</th>
<th>Expanded uncertainty, Mass fraction (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diethyl phthalate</td>
<td>84-66-2</td>
<td>879</td>
</tr>
<tr>
<td>Dibutyl phthalate (Di-n-butyl phthalate)</td>
<td>84-74-2</td>
<td>879</td>
</tr>
<tr>
<td>Benzyl butyl phthalate</td>
<td>85-68-7</td>
<td>871</td>
</tr>
<tr>
<td>Dicyclohexyl phthalate</td>
<td>84-61-7</td>
<td>861</td>
</tr>
<tr>
<td>Bis(2-ethylhexyl) phthalate (Di-2-ethylhexyl phthalate)</td>
<td>117-81-7</td>
<td>876</td>
</tr>
</tbody>
</table>

Analysis
The certified values of this CRM were based on the analytical results of phthalates by isotope dilution-gas chromatography/mass spectrometry (ID-GC/MS).

Metrological Traceability
The certified values of this CRM were determined by IDMS as a primary method of measurement. Diethyl phthalate (NMIJ CRM 4022-b), di-n-butyl phthalate (NMIJ CRM 4023-a), benzyl butyl phthalate (NMIJ CRM 4029-a), dicyclohexyl phthalate (NMIJ CRM 4027-a), and di-2-ethylhexyl phthalate (NMIJ CRM 4024-a) were used as the primary standards of the calibration solution for quantification. 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 amber glass bottle is sealed with argon gas in an aluminum-layered bag.

Homogeneity
The homogeneity of the CRM was determined by analyzing 10 bottles randomly sampled from 998 bottles. The inhomogeneity of the analyte was evaluated by ANOVA. The homogeneity is reflected in the uncertainty of the certified value.

Instructions for Storage
This CRM should be stored at a temperature of 35 °C or less and shielded from light. Do not contact this CRM 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 as soon as possible after the opening of a bottle. More than 0.1 g of the resin should be used.

Precautions for Handling
Keep away from heat and ignition sources. Wear 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, lubricant, plasticizer (acetyl tributyl citrate; CAS No. 77-90-7, eight phthalates and di-2-ethylhexyl adipate) and other polymer additives.

Technical Information
This CRM contains dimethyl phthalates, diisobutyl phthalate, dioctyl phthalate and bis(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 described in Analysis, using commercially available reagents as calibration standards.

<table>
<thead>
<tr>
<th>Substance</th>
<th>CAS No.</th>
<th>Informational value, Mass fraction (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dimethyl phthalate</td>
<td>131-11-3</td>
<td>886</td>
</tr>
<tr>
<td>Diisobutyl phthalate</td>
<td>84-69-5</td>
<td>883</td>
</tr>
<tr>
<td>Dioctyl phthalate (Bis(o-octyl) phthalate)</td>
<td>117-84-0</td>
<td>904</td>
</tr>
<tr>
<td>Bis(2-ethylhexyl) adipate       (Di-2-ethylhexyl adipate)</td>
<td>103-23-1</td>
<td>892</td>
</tr>
</tbody>
</table>

NMIJ Analysts
The technical manager and the production manager for this CRM is N. Hanari, and the analysts are S. Matsuyama, Y. Orihara, and Y. Aoyagi.
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.

February 20, 2020

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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