Date of Shipment: Xxxxx xx, 20xx

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

Reference Material Certificate
NMIJ CRM 8156-a
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%.

<table>
<thead>
<tr>
<th>Substance</th>
<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>93</td>
<td>18</td>
</tr>
<tr>
<td>Dipropyl phthalate (Di-n-propyl phthalate)</td>
<td>131-16-8</td>
<td>90</td>
<td>15</td>
</tr>
<tr>
<td>Dibutyl phthalate (Di-n-butyl phthalate)</td>
<td>84-74-2</td>
<td>92</td>
<td>17</td>
</tr>
<tr>
<td>Benzyl butyl phthalate</td>
<td>85-68-7</td>
<td>90</td>
<td>14</td>
</tr>
<tr>
<td>Dipentyl phthalate (Di-n-pentyl phthalate)</td>
<td>131-18-0</td>
<td>94</td>
<td>16</td>
</tr>
<tr>
<td>Dihexyl phthalate (Di-n-hexyl phthalate)</td>
<td>84-75-3</td>
<td>93</td>
<td>17</td>
</tr>
<tr>
<td>Dicyclohexyl phthalate</td>
<td>84-81-7</td>
<td>92</td>
<td>15</td>
</tr>
<tr>
<td>Bis(2-ethylhexyl) phthalate (Di-2-ethylhexyl phthalate)</td>
<td>117-81-7</td>
<td>95</td>
<td>16</td>
</tr>
</tbody>
</table>

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 with GC/MS and LC/MS. 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), di-n-hexyl phthalate (NMIJ CRM 4027-a), dicyclohexyl phthalate (NMIJ CRM 4028-a), and di-2-ethylhexyl phthalate (NMIJ CRM 4024-a) were used as the primary standards of the calibration solution for quantification. The certified values of this CRM are traceable to the International System of Units (SI).
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Expiration of Certification
This certificate is valid for one year from the date of shipment, provided that this 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 in 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 at temperatures of 15 °C to 35 °C or less and protected from light. Do not contact this CRM to other plastics such as a bottle cap for a 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, diisobutyl phthalate, diheptyl phthalate, and dioctyl phthalate. 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>Mass fraction (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dimethyl phthalate</td>
<td>131-11-3</td>
<td>95</td>
</tr>
<tr>
<td>Diisobutyl phthalate</td>
<td>84-69-5</td>
<td>88</td>
</tr>
<tr>
<td>Diheptyl phthalate (Di-n-heptyl phthalate)</td>
<td>3648-21-3</td>
<td>93</td>
</tr>
<tr>
<td>Dioctyl phthalate (Di-octyl phthalate)</td>
<td>117-84-0</td>
<td>97</td>
</tr>
</tbody>
</table>

NMIJ Analyst
The technical manager and the production manager for this CRM are HANARI N., and the analysts are HANARI N., MATSUYAMA S., AOYAGI Y., and ORIHARA Y.

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.
Date of Shipment: Xxxxx xx, 20xx

Reproduction of Certificate
In reproducing this certificate, it should be clearly indicated that the document is a copy.

February 24, 2022

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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