National Institute of Advanced Industrial Science and Technology
National Metrology Institute of Japan
Reference Material Certificate
NMIJ CRM 4074-a
No. +++
Trichloroacetic Acid

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 the calibration of analytical instruments, quality control of analytical instruments, and validation of analytical techniques and instruments.

Certified Value
The certified value of this CRM is purity (the mass fraction), 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>Substance</th>
<th>CAS No.</th>
<th>Certified value, Mass fraction (kg/kg)</th>
<th>Expanded uncertainty, Mass fraction (kg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trichloroacetic Acid</td>
<td>76-03-9</td>
<td>0.999</td>
<td>0.002</td>
</tr>
</tbody>
</table>

Analysis
The certified value of this CRM was a mean value of mass fraction of trichloroacetic acid obtained by the freezing point depression method based on the stepwise scan method using a differential scanning calorimeter (DSC) and that obtained by the neutralization titrimetry. The combined standard uncertainty of the certified value was estimated by combining standard uncertainties derived from purity determination, deviation between the methods, homogeneity test, and stability test.

Metrological Traceability
The certified value of this CRM is determined by the primary methods of measurement, namely the freezing point depression method and the titrimetry. The purity (mass fraction) is obtained by the freezing point depression method using a DSC while scales of temperature and enthalpy of the DSC are calibrated with NMIJ CRM 5401-a (cyclohexane) and NIST SRM 2232 (indium). The purity (mass fraction) is obtained by the titrimetry using sodium hydroxide aqueous solution as a titrant which is calibrated with NMIJ CRM 3004-a (amidosulfuric acid). Balances calibrated through the Japan Calibration Service System (JCSS) are used in weighing for the determination. Therefore, the certified value is traceable to the SI.

Expiration of Certification
This certificate is valid for one year from the date of shipment, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

Sample Form
This CRM is in the form of a white solid at room temperature. This CRM is sealed in an amber glass vial with argon gas and the vial is sealed in an aluminum laminated bag with argon gas. The net amount is 1.5 g for each vial.

Homogeneity
Ten vials were sampled from the total of 300 subdivided vials by using the stratified random sampling method in order of subdivision for each of the two homogeneity tests: the high-performance liquid chromatography and the Karl-Fischer titrimetry. Area percentages of trichloroacetic acid were measured by the high-performance liquid chromatography while water content was measured by the Karl-Fischer titrimetry. The evaluated variation of purity among the vials due to inhomogeneity has been...
incorporated into the uncertainty of the certified value. It is concluded, therefore, that this CRM is homogeneous within the range of the uncertainty of the certified value.

**Instructions for Storage**
This CRM should be stored in a cold (around -20 °C) and light shielded place.

**Instructions for Use**
This CRM is for laboratory use only. The vials of this CRM should be allowed to warm to room temperature and shaken well before opening. This CRM should be used as soon as possible once a vial is opened. This CRM, after being warmed to room temperature, should be opened in as a low humidity condition as possible since it is deliquescent. More than 0.2 g of the material should be used in each analysis.

**Precautions for Handling**
Keep away from heat and ignition sources. Wear personal protective equipment such as safety glasses, safety mask and safety gloves when handling. Refer to the safety data sheet (SDS) on this CRM before use.

**Preparation Method**
This CRM was prepared by KANTO CHEMICAL CO., INC. For packaging, each amber glass vial was filled with 1.5 g of trichloroacetic acid was filled into an amber glass vial. The vial was then sealed in an aluminum layered bag.

**Information**
This CRM contains dichloroacetic acid as an impurity. The mass fraction of dichloroacetic acid determined by the high performance liquid chromatography was 0.44 g/kg at the time of certification.

**NMIJ Analysts**
The technical manager for this CRM is M. Numata. The production manager is Y. Shimizu and analysts are Y. Shimizu, T. Yamazaki, N. Saito, Y. Kitamaki, X. Bao, and S. Nakamura.

**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.

March 8, 2017
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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