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

Reference Material Certificate
NMIJ CRM 4001-b
No. +++
Ethanol

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 instruments, and validation of analytical techniques and instruments during ethanol analysis.

Certified Values
The certified values of purities (amount-of-substance fraction and mass fraction) are given in the table below. The uncertainty of each 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, Amount-of-substance fraction (mol/mol)</th>
<th>Expanded uncertainty, Amount-of-substance fraction (mol/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethanol</td>
<td>64-17-5</td>
<td>0.999</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<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>Ethanol</td>
<td>64-17-5</td>
<td>1.000</td>
</tr>
</tbody>
</table>

Analysis
The certified values (amount-of-substance fraction) were determined by the freezing point depression method carried out using adiabatic calorimeters operated in accordance with the fractional melting method. The combined standard uncertainty was estimated as a combination of standard uncertainties in purity determination, homogeneity test, and stability test.
The certified values (mass fraction) were determined by converting the purity (amount-of-substance fraction) using the estimated average molecular weights of impurities. The combined standard uncertainty was estimated as a combination of standard uncertainties in purity determination, homogeneity test, and stability test.

Metrological Traceability
The certified values (amount-of-substance fraction) were determined by the freezing point depression method carried out using adiabatic calorimeters and are traceable to the International System of Units (SI). Temperature (platinum resistance thermometer), voltage (digital multimeter), resistance (standard resistor), and heating duration (universal counter) of the adiabatic calorimeters were calibrated and were traceable to the SI.
The certified values (mass fraction) were determined by converting the purity (amount-of-substance fraction) using the estimated average molecular weight of impurities. The concentrations of impurities were determined using a gas chromatography with flame ionization detection (GC-FID) and Karl-Fisher (KF) titration. GC-FID was calibrated using solutions prepared gravimetrically with a JCSS-calibrated balance, and the KF method was validated by NMIJ. The certified values were traceable to the SI.

Indicative Values
The concentration of carbon 14 ($^{14}$C) in this CRM was determined via the collaborative analysis carried out using accelerator mass spectrometers and liquid scintillation counters. The indicative values of $^{14}$C concentration represented as “percent Modern
Carbon” (pMC: Stuiver & Polach (1977) Radiocarbon, 19(3), 355-363) and specific radioactivity are given in the following table. The quoted uncertainties are the half-width of the expanded uncertainty intervals calculated using the standard deviation of reported results and a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

“Specific radioactivity” used in this section means radioactivity per unit mass of carbon. The half-life of $^{14}$C was assumed to be 5730 years (Godwin (1962) Nature, 195, 1984).

<table>
<thead>
<tr>
<th></th>
<th>Indicative value</th>
<th>Expanded uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td>pMC ($A_{abs}/A_{abs} \times 100$: on July 1, 2011)$^1$</td>
<td>108.0 %</td>
<td>1.6 %</td>
</tr>
<tr>
<td>pMC ($A_{SN}/A_{ON} \times 100$)$^2$</td>
<td>105.8 %</td>
<td>1.6 %</td>
</tr>
<tr>
<td>Specific radioactivity (on July 1, 2011)$^3$</td>
<td>0.2440 Bq/g-carbon</td>
<td>0.0035 Bq/g-carbon</td>
</tr>
</tbody>
</table>

1) $A_{abs}$ denotes the standard “modern” $^{14}$C concentration. It is defined as $^{14}$C concentration in atmospheric CO$_2$ in 1950, and corresponds to 0.2260 Bq/g-carbon. Practically, NIST SRM 4990c (oxalic acid) is often used as the standard for calculating $A_{abs}$. In that case, $A_{abs}$ is the specific radioactivity or $^{14}$C/$^{12}$C value of SRM 4990c normalized to –25 per mil for $^{13}$C, corrected for decay since 1950, and multiplied by 0.7459. $A_S$ denotes the specific radioactivity or $^{14}$C/$^{12}$C value of this CRM in 2011 (not normalized for $^{13}$C).

2) $A_{ON}$ denotes the standard $^{14}$C concentration at the time of measurement (in 2011). It is the specific radioactivity or $^{14}$C/$^{12}$C value of SRM 4990c normalized to –25 per mil for $^{13}$C, and multiplied by 0.7459. $A_{SN}$ denotes the specific radioactivity or $^{14}$C/$^{12}$C value of this CRM in 2011, normalized to –25 per mil for $^{13}$C. The ratio does not depend on the year of measurement.

3) The specific radioactivity of this CRM equals 0.2260 $A_S/A_{abs}$. The radioactivity concentration in this CRM (Bq/g-sample) is calculated from specific radioactivity by multiplying it with the carbon content of this CRM (0.5214).

Mutual Recognition Arrangement under Meter Convention

This certificate is consistent with the calibration and measurement capabilities (CMCs) that are included in Appendix C of the Mutual Recognition Arrangement (MRA) drawn up by the International Committee for Weights and Measures (CIPM). Under the MRA, all participating institutes recognize the validity of each other’s calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C (as for Appendix C of MRA, see http://kcdb.bipm.org/AppendixC/default.asp).

Expiration of Certification

This certificate is valid until March 31, 2022, provided that the CRM remains unopened and is stored in accordance with the instructions given in this certificate.

Sample Form

This CRM is in the form of a colorless and clear liquid at room temperature. This CRM of 15 mL in net volume is sealed in an amber glass ampoule containing argon gas.

Homogeneity

Ten ampoules were sampled from 300 subdivided ampoules randomly stratified for homogeneity tests by GC-FID and KF. Area percentages of ethanol (determined by GC-FID) and water content (determined by KF) of the CRM were measured and evaluated by homogeneity tests. The evaluated variations between the purities of the different ampoules due to inhomogeneity were taken into account for determining the uncertainty in the certified values, and this CRM is homogeneous within the uncertainty range of the certified values.

Precautions for Storage

This CRM should be stored in a cold (temperature: about –20 °C) and dark place.

Instructions for Use

This CRM is for laboratory use only. The bottle of this CRM should be allowed to warm to room temperature before opening. The CRM should be used promptly once the ampoule is opened.
Instructions for Handling
Keep away from heat and ignition sources. Wear personal protective equipment such as safety glasses, safety mask, and safety gloves in handling. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation
This CRM was purified and subdivided by Kanto Chemical Co., Inc. It was purified by distillation and drying. For packaging, 15 mL of ethanol was filled into an amber glass ampoule in an argon atmosphere.

Technical Information
The concentrations of water and acetaldehyde in this CRM were 108 mg/kg and 2 mg/kg, respectively. These impurities were measured by KF and GC-FID, respectively. The $\delta^{13}$C value determined by isotope-ratio mass spectrometry was $-11.7$ per mil (standard deviation: 0.1 per mil, $n = 3$).

Collaborators
Measurements for the $^{14}$C content and $\delta^{13}$C value were conducted as a collaborative analysis among 13 laboratories (listed below) coordinated by the NMIJ.

Institute of Accelerator Analysis, Ltd.
The General Environmental Technos, Co., Ltd.
Kyushu Environmental Evaluation Association
Geo Science Laboratory, Co., Ltd.
Tokyo Metropolitan Industrial Technology Research Institute
Radioisotope Center, The University of Tokyo
Atmosphere and Ocean Research Institute, The University of Tokyo
Center for Chronological Research, Nagoya University
Japan Radioisotope Association
Nippon Kaiji Kentei Kyokai
Japan Chemical Analysis Center
Paleo Labo, Co., Ltd.
Quantum Radiation Division, National Metrology Institute of Japan

NMIJ Analysts
For this CRM, the technical manager is K. Kato, the person responsible for production is Y. Kitamaki, and the production analysts are Y. Kitamaki, Y. Shimmizu, S. Inagaki, T. Yamanaka, M. Numata, and E. Yoshimura.

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.

April 1, 2015
Ryoji Chubachi
President
National Institute of Advanced Industrial Science and Technology
If you have any questions about this CRM, please contact:
National Institute of Advanced Industrial Science and Technology,
National Metrology Institute of Japan,
Center for Quality Management of Metrology, Reference Materials Office,
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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.”