

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 4222-c

No. +++

Water in Mesitylene (0.1 mg/g)



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/ICE 17025:2005. This CRM is intended for use in the calibration of instruments and confirming the validity of analytical methods and instruments during quantification of water by Karl Fischer (KF) titration.

Certified Value

The certified value for this CRM is concentration (mass fraction of water in mesitylene) 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 %.

	CAS No.	Certified value, Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)
Water	7732-18-5	125	3

Analysis

The certified value of this CRM is the weighted mean of the analytical results obtained by coulometric and volumetric KF titration, where the reciprocal of the uncertainty of the result obtained by each method was used as the weight. The uncertainty between the two methods is reflected in the uncertainty of the certified value.

Metrological Traceability

The certified value of this CRM was determined by the coulometric and volumetric titrations as the primary methods of measurement. To confirm the traceability of an applied current on coulometric KF titration, JCSS-calibrated standard resistors were connected in series to the circuit of the applied current of the KF instrument, and the voltage drop across the resistor was measured by a JCSS-calibrated voltmeter. In addition, by measuring the time using a JCSS-calibrated frequency counter, the traceability of the electric charge (current \times time) applied was ensured. The analytical results of volumetric KF titrations were obtained using a KF reagent, the titer of which was determined by NMIJ CRM 8301-a. As a result, the certified value is traceable to the International System of Units (SI).

Expiration of Certification

This certificate is valid for 6 months 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 clear liquid, and approximately 8.5 mL of CRM is sealed in an amber ampoule.

Homogeneity

The homogeneity of this CRM was determined by analyzing water in 10 ampoules selected by random sampling of 205 subdivided ampoules. The inhomogeneity of the analyte was evaluated by ANOVA and was reflected in the uncertainty of the certified value.

Instructions for Storage

This CRM should be stored in a clean place at a temperature between 15 °C to 30 °C, shielded from lights.

Instructions for Use

Shake gently, and after several minutes, open the ampoule. To avoid sample evaporation, and moisture sorption or desorption, this CRM should be used promptly once an ampoule is opened. Samples should be taken using a gas-tight syringe, and the rubber cap accompanying this CRM. It is recommended that all the operations from ampoule breaking to measurement be performed under conditions of 20 % to 50 % relative humidity. An additional injection needle must be stabbed into the cap in order to ensure the ampoule is not depressurized while sampling.

Precautions for Handling

This CRM is for laboratory use only. Wear personal protective equipment such as safety mask, protective gloves in handling this CRM. The use, handling, storage and disposal of this CRM should be carried out according to the laws regulating the components of this CRM. Handle the CRM according to the Safety Data Sheet (SDS).

Preparation Method

Mesitylene is first stirred in the glass bottle under ambient air. Once the remaining water content reaches a constant value, 8.5 mL of the solution is dispensed and sealed in an amber ampoule.

Information

The density of this CRM measured with an oscillation-type density meter at room temperature is 0.8611 g/cm³ (25 °C), 0.8652 g/cm³ (20 °C), and 0.8693 g/cm³ (15 °C).

NMIJ Analysts

The technical manager and production manager for this CRM is A. Takatsu and S. Inagaki, respectively. Analytical measurements for the certification of this CRM were performed at NMIJ by S. Inagaki, M. Numata, T. Suzuki, T. Asakai, N. Hanari, K. Ishikawa and R. Iwasawa.

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.

April 1, 2015

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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Phone: +81-29-861-4059; Fax: +81-29-861-4009, <https://www.nmij.jp/english/service/C/>

Supplement Water in mesitylene (0.1 mg/g)

Protocol for measurement (example)

An example of a procedure for measurement of this CRM is shown below.

1. Prepare a KF titrator for the analysis.
2. Shake the ampoule gently and stand for a few minutes.
3. Open the ampoule and attach the supplemental rubber cap immediately.
4. Insert a hypodermic needle into the cap to avoid depressurizing the ampoule while sampling.
5. Rinse a gas-tight syringe with *ca.* 1 mL of the CRM adequately.
6. Take *ca.* 6.5 mL of the CRM carefully to avoid the formation of air bubbles.
7. Wipe the needle of the syringe gently.
8. Turn up the syringe and take the bubbles out of it. Then, attach a silicon chip to its needle.
9. Weigh the syringe using a precision balance.
10. Detach the silicon chip and inject *ca.* 1.5 mL of the aliquot into the electrolytic cell.
11. Take off the syringe from the cell and attach the silicon chip again.
12. Weigh the syringe again using a precision balance.
13. Repeat the procedures from No. 9 to 12 and analyze 3 times.