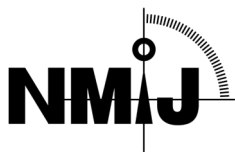


National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 4222-d02  
No. +++

## Water in Mesitylene (0.1 mg/g)

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 the validating analytical methods and instruments, and calibrating 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 (1,3,5-trimethylbenzene)) 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	131	3

**Analysis**

The certified value of this CRM is the weighted mean of the analytical results obtained by coulometric and volumetric KF titrations, 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 it of ca. 8 mL in net volume is sealed in an amber glass ampule.

**Homogeneity**

The homogeneity of this CRM was determined by analyzing water in 11 ampules selected by random sampling from 226 subdivided ampules. The inhomogeneity of the analyte was evaluated by ANOVA and is reflected in the uncertainty of the certified value.

**Instructions for Storage**

This CRM should be stored at a temperature between 15 °C and 30 °C in a clean place and shielded from light.

**Instructions for Use**

Before opening the ampule shake gently, and leave to stand for several minutes. To avoid sample evaporation, and moisture sorption or desorption, this CRM should be used promptly once the ampule is opened. Samples should be taken from the ampule using a gas-tight syringe, and the rubber cap accompanying this CRM. It is recommended that all the operations from ampule 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 ampule 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 when handling this CRM. The use, handling, storage and disposal of this CRM should be conducted according to laws regulating the components of this CRM. Refer to the Safety Data Sheet (SDS) on this CRM before use.

**Preparation**

This CRM was subdivided by Kanto Chemical Co., Inc. Mesitylene was first stirred in the glass bottle under ambient air. Once the remaining water content achieved a constant value, 8 mL of the solution was dispensed into an amber glass ampule and the ampule was then sealed.

**Technical Information**

The density of this CRM measured with an oscillation-type density meter at room temperature is 0.8610 g/cm<sup>3</sup> (25 °C), 0.8651 g/cm<sup>3</sup> (20 °C), and 0.8692 g/cm<sup>3</sup> (15 °C).

**NMIJ Analysts**

The technical manager and production manager for this CRM are HANARI N. and INAGAKI S., respectively. Analytical measurements for the certification of this CRM were performed at NMIJ by INAGAKI S., NUMATA M., SUZUKI T., ASAKAI T. and IWASAWA R.

**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, 2020

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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Phone: +81-29-861-4059; Fax: +81-29-861-4009, <https://unit.aist.go.jp/nmij/english/refmate/>

## 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 ampule gently and stand for a few minutes.
3. Open the ampule and attach the supplemental rubber cap immediately.
4. Insert an injection needle into the cap to avoid depressurizing the ampule 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.

Sample