

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 4222-e

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 of this CRM is mass fraction of water in mesitylene (1,3,5-trimethylbenzene) 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 %.

| Substance | CAS No. | Certified value Mass fraction (mg/kg) | Expanded uncertainty Mass fraction (mg/kg) |
|-----------|-----------|--|---|
| Water | 7732-18-5 | 125 | 6 |

Analysis

The certified value of this CRM was 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. The analytical results of coulometric KF titrations were obtained using the coulometric KF titrator whose electric charge was verified by the Japan Calibration Service System (JCSS)-calibrated standard resistor, voltmeter and frequency counter. The analytical results of volumetric KF titrations were obtained using a KF reagent, the titer of which was determined by NMIJ CRM 4228-a. The mass of a sample injected into the titrator was measured by JCSS-calibrated balance. The certified value, therefore, is traceable to the International System of Units (SI).

Mutual Recognition Arrangement under Metre Convention

The certified value of this CRM is recognized for international equivalence based on the Mutual Recognition Arrangement under the Metre Convention (CIPM MRA). The calibration measurement capability (CMC) of NMIJ related to this CRM is registered in the Key Comparison Database (KCDB) (see <https://www.bipm.org/kcdb/>) of the International Bureau of Weights and Measures (BIPM).

Expiration of Certification

This certificate is valid for six months from the date of shipment, provided that the CRM remains unopened and is stored in accordance with the instructions given in this certificate.

Description of the Material

This CRM is a mesitylene. It is in the form of colorless and transparent liquid at room temperature and approximately 8 mL of it

in net volume is sealed in an amber glass ampule.

Instructions for Storage

This CRM should be stored in a clean place at temperatures of 2 °C to 10 °C and protected from light.

Instructions for Use

After taking out the ampule of this CRM from the refrigerator and bringing the ampule back to room temperature, the ampule should be shaken gently and left to stand for several minutes before opening. To avoid sample evaporation, and moisture sorption or desorption, this CRM should be used promptly once the ampule is opened. By using a gas-tight syringe samples should be taken from the ampule setting a rubber cap of which comes together with 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. This CRM is for laboratory use only.

Precautions for Handling

This CRM should be kept away from heat and ignition sources. Personal protective equipment such as eye protection, protective mask and protective gloves should be used when this CRM is handled. This CRM should be used, handled, stored, and disposed of according to laws regulating the components of this CRM. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

Approximately 4.5 L of mesitylene in the glass bottle was exposed to ambient air so as to make residual water reach saturated concentration. After the residual water concentration became constant, 8 mL of the solution was dispensed into an amber glass ampule and the ampule was then sealed.

Technical Information

The densities of this CRM measured with an oscillation-type density meter at the time of the certification were 0.8610 g/cm³ (25 °C), 0.8651 g/cm³ (20 °C) and 0.8692 g/cm³ (15 °C).

NMIJ Analysts

The technical manager for this CRM is HANARI N., the production manager is INAGAKI S., and the analysts are NAKAMURA K., INAGAKI S., SUZUKI T., ASAKAI T. and KIHARA M.

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.

Reproduction of Certificate

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

January 27, 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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Revision history

March 20, 2023: The expanded uncertainty was reevaluated based on the results of stability assessment after certification.

Sample

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