

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 4229-a

No. +++



Water in Methylcyclohexane (0.02 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 mass fraction of water in methylcyclohexane 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	18	5

Analysis

The certified value of this CRM was the analytical results obtained by coulometric KF titration.

Metrological Traceability

The certified value of this CRM was determined by the coulometric KF titrator whose electric charge was verified by the Japan Calibration Service System (JCSS)-calibrated standard resistor, voltmeter and frequency counter. The mass of a sample injected to the titrator was measured by JCSS-calibrated balance. The certified value, therefore, is traceable to the International System of Units (SI).

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 methylcyclohexane. 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 15 °C to 30 °C and protected from light.

Instructions for Use

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. A silica gel tube must be inserted into the rubber cap to prevent depressurization of the ampule and moisture sorption during sampling. This CRM is for

Date of Shipment: Xxxxx xx, 20xx

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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 2 L of methylcyclohexane 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 certification were 0.7648 g/cm³ (25 °C), 0.7691 g/cm³ (20 °C) and 0.7734 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 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 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.

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, <https://unit.aist.go.jp/nmij/english/refimate/>

Supplement Water in Methylcyclohexane (0.02 mg/g)

Protocol for measurement (Example)

An example of the procedure for measurement of this CRM is presented 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 a silica gel tube (approximately 2.5 mL size) into the rubber cap to avoid depressurization of the ampule and moisture sorption while sampling.
5. Rinse a gas-tight syringe with approximately 1 mL of the CRM adequately.
6. Take approximately 6.5 mL of the CRM cautiously to avoid 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 by using a precision balance.
10. Detach the silicon chip and inject approximately 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.