Date of Shipment: Xxxxx xx, 20xx

4229a00-190220-210527

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 validating analytical methods and instruments and calibrating instruments for quantification of water by means of Karl Fischer (KF) titration.

**Certified Value**

The certified value for this CRM (mass fraction of water in methylcyclohexane) is 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<br>Mass fraction (mg/kg) | Expanded uncertainty<br>Mass fraction (mg/kg) |
|---|---|---|---|
| Water | 7732-18-5 | 18 | 5 |

**Analysis**

The certified value of this CRM is 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 JCSS-calibrated standard resistor, voltmeter and frequency counter. The amount 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 solution. 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 sunlight.

**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. Samples should be taken from the ampule by using a gas-tight syringe and a rubber cap both of which come 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

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 density of this CRM at the time of certification was 0.7648 g/cm$^3$ (25 °C), 0.7691 g/cm$^3$ (20 °C) and 0.7734 g/cm$^3$ (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 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:
National Institute of Advanced Industrial Science and Technology,
National Metrology Institute of Japan,
Center for Quality Management of Metrology, Reference Materials Office,
1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan
Phone: +81-29-861-4059; Fax: +81-29-861-4009, https://unit.aist.go.jp/nmij/english/refmate/

# 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. Set up a KF titrator for the analysis.
2. Shake the ampule gently and leave it to stand for several minutes.
3. Open the ampule and put on a rubber cap, an accessory coming together with the CRM, to the ampule immediately.
4. Insert a silica gel tube (*ca.* 2.5 mL size) into the rubber cap to avoid depressurization of the ampule and moisture sorption during sampling.
5. Rinse a gas-tight syringe with *ca.* 1 mL of the CRM adequately.
6. Take *ca.* 6.5 mL of the CRM cautiously to avoid formation of air bubbles.
7. Wipe the needle of the syringe gently.
8. Point the syringe straight up and take any bubbles out of it. Then attach a silicon chip to its needle.
9. Weigh the syringe by using a precision balance.
10. Take off the silicon chip and inject an aliquot of *ca.* 1.5 mL into an electrolytic cell.
11. Remove the syringe from the cell and attach the silicon chip again.
12. Weigh the syringe again by using the precision balance.
13. Repeat Steps 9 to 12 for 3 to 4 times and analyze the aliquots.