

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 8302-a

No. +++

Biodiesel Fuel (Palm Oil-Based)



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 the calibration of instruments, and validation of analytical methods and instruments used for the quantification of the following components in biodiesel fuel (fatty acid methyl esters) samples and similar materials.

Certified Values

The certified values of this CRM are given in the tables 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	393	25

Substance	Certified value Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)
Na	1.26	0.22
Mg	0.83	0.11
K	0.72	0.15
Ca	1.01	0.16
P	2.09	0.35
S	7.17	0.51

Analysis

The certified values of this CRM were the weighted means of the results obtained from the following methods:

(1) Water:

- Coulometric and volumetric Karl Fischer (KF) titration.

(2) Na:

- Inductively coupled plasma tandem quadrupole mass spectrometry (ICP-MS/MS); pretreatment: acid digestion; quantification: standard addition.
- High resolution inductively coupled plasma mass spectrometry (HR-ICP-MS); pretreatment: acid digestion; quantification: standard addition.
- Flame atomic absorption spectrometry (FL-AAS); pretreatment: xylene dilution; quantification: absolute calibration curve

(3) Mg, K and Ca:

- Isotope dilution (ID-) ICP-MS/MS; pretreatment: acid digestion.
- ICP-MS/MS; pretreatment: acid digestion; quantification: standard addition.

(4) P:

- ICP-MS/MS; pretreatment: acid digestion; quantification: standard addition.

- Flow injection-inductively coupled plasma mass spectrometry (ICP-MS); pretreatment: xylene dilution; quantification: standard addition.
 - Inductively coupled plasma optical emission spectrometry (ICP-OES); pretreatment: xylene dilution; quantification: standard addition.
- (5) S:
- ID-ICP-MS/MS; pretreatment: acid digestion.
 - ICP-MS/MS; pretreatment: acid digestion; quantification: standard addition.
 - ID-ICP-MS/MS; pretreatment: ethanol dilution.
 - Combustion ion chromatography; quantification: standard addition.

Metrological Traceability

- (1) Water: The certified value was determined by two validated methods. Because the calibration solutions for the measurements were prepared from ultrapure water, the purity of which was evaluated by the freezing point depression method (primary method of measurement) in NMIJ, and prepared gravimetrically using a JCSS (Japan Calibration Service System)-calibrated balance, the certified value is traceable to the International System of Units (SI).
- (2) Na, Mg, K, Ca, and P: Each certified value was determined by isotope dilution-mass spectrometry as the primary method of measurement and/or by other validated analytical methods. Their calibration standards were prepared from JCSS standard solutions by a gravimetric method using a JCSS calibrated electronic balance. Therefore, the certified values are traceable to SI.
- (3) S: The certified value was determined by isotope dilution-mass spectrometry as the primary method of measurement and by other validated analytical methods. For the ICP-MS/MS measurements, calibration standards were prepared from a JCSS standard solution by a gravimetric method using a JCSS calibrated electronic balance. For the combustion ion chromatography, the calibration standards were prepared gravimetrically by diluting thiophene with a JCSS-calibrated balance. The purity of the thiophene was determined by the differential scanning calorimetry (DSC) and impurity analysis in NMIJ. Therefore, the certified value is traceable to SI.

Mutual Recognition Arrangement under Metre Convention

The certified values of the mass fraction of water, Na, Mg, K, Ca, and P of this CRM are recognized for international equivalence based on the Mutual Recognition Arrangement under the Metre Convention (CIPM MRA). The calibration measurement capabilities (CMC) of NMIJ related to this CRM are 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 this CRM remains unopened and is stored in accordance with the instructions given in this certificate.

Description of the Material

This CRM is a biodiesel fuel (fatty acid methyl esters) made from palm oil. This CRM is in the form of yellow viscous liquid at room temperature. This CRM of ca. 15 mL in net volume is kept in an amber glass ampoule with argon gas.

Instructions for Storage

This CRM should be stored at temperatures of 15 °C to 30 °C and protected from light.

Instructions for Use

This CRM is for laboratory use only. Prior to use, the ampoule should be shaken gently and opened after several minutes. To avoid sample evaporation and moisture absorption, the CRM should be used promptly once the ampoule is opened. For measurement of the water content, use a gas-tight syringe to take the sample, and it is recommended that the opening be sealed as shown in “Technical Information” to reduce moisture absorption since the water content changes depending on the handling environment.

Precautions for Handling

Wear a mask, gloves, and other personal protective equipment when handling. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

The raw material of this CRM is biodiesel fuel (fatty acid methyl esters) that was made from palm oil. Commercial methanol and an element standard solution in biodiesel fuel, "Custom blend multi-element Standard (Na, K, Mg, Ca: 200 µg/g and P: 400 µg/g, containing Na-, K-, Mg-, Ca-alkylbenzene sulfate and alkyl phosphate)", were added to the material at NMIJ. The spiked biodiesel fuel was homogenized by mechanical mixing and 15 mL of the mixture was sealed in an amber ampoule under argon gas.

Technical Information

For the measurement of water content, a plastic paraffin film was attached to the ampoule just after breaking it open at 35 % relative humidity (25 °C). The sample was taken from the bottom of the ampoule using a gas-tight syringe. When comparing the water content measured immediately after opening and after 30 min, the increase was ca. 15 mg/kg and the obtained results remained within the uncertainty limit of the certified value.

The density of this CRM measured using a vibrating U-tube densimeter at the time of the certification was 0.875 g/cm³ (15 °C). The mass fraction of methanol measured using a headspace gas chromatograph/ mass spectrometer at the time of the certification was 564 mg/kg.

NMIJ Analysts

The technical manager for this CRM is NUMATA M., the production manager is KITAMAKI Y., and the analysts are NAKAMURA K., NUMATA M., KITAMAKI Y., ZHU Y., NARUKAWA T., INAGAKI S., HANARI N., FUJITA Y., KANO Y., KIHARA M., NAKAMURA S., MORII N., TANIGUCHI S., MATSUO M., IWASAWA R. and KUDO I.

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.

Note

This CRM was developed in collaboration with National Institute of Standards and Technology (NIST) with support of a grant from Japan-U.S. Cooperative Project of on Energy and Environmental Technology Research/Standardization (Japan-U.S. Environmental Technology Research Cooperation, 2013-2014) by the Ministry of Economy, Trade and Industry.

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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Revision history

May 30, 2018: Certified value of kinetic viscosity was eliminated based on long-term stability monitoring.

The description on Mutual Recognition Arrangement under Meter Convention was added.

April 24, 2019: Certified value of density was eliminated based on long-term stability monitoring. The density at the time of the certification was added in "Technical Information."

September 28, 2023: Mass fraction of methanol, which had been Indicative value, was described in "Technical Information".

Sample