

Date of Shipment: XXXXX xx, 20xx

4603a00-220224-220224

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 4603-a

No. +++



Potassium Hydrogen Phthalate for Quantitative NMR (^1H)

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 ^1H signal area by nuclear magnetic resonance (NMR) spectroscopy, and validation of analytical methods.

Certified Value

The certified value of this CRM 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 Mass fraction (kg/kg)	Expanded uncertainty Mass fraction (kg/kg)
Potassium Hydrogen Phthalate	877-24-7	0.9998	0.0003

Analysis

The certified value of this CRM is the arithmetic mean of the purity by mass balance approach and purity based on the coulometric acidimetric titration method. In the mass balance approach, organic impurities were analyzed by using a high performance liquid chromatograph with an ultraviolet-visible detector (HPLC-UV), volatile organic compounds were evaluated by using NMR, water content was analyzed by using a Karl-Fischer titrator (KF) and trace metals were evaluated by using an inductively coupled plasma optical emission spectrometer (ICP-OES). For the purity determination by the coulometric acidimetric titration method, potassium evaluated by gravimetric analysis and trace impurities evaluated by ion-chromatography were used. The standard uncertainty was estimated by combining uncertainties due to each analytical method, differences among the two methods, sample homogeneity and stability.

Metrological Traceability

The certified value of this CRM was determined by the mass balance approach, and the coulometric acidimetric titration method which is one of the primary methods. Water content was determined by a KF validated with NMIJ CRM 4228-a. Trace metals were evaluated with an ICP-OES and standard solutions prepared from NMIJ CRM 3600 series by gravimetric mixing. Purity based on the coulometric acidimetric titration method was determined by using the coulometric titrator calibrated by Japan Calibration Service System (JCSS) for the voltage, resistance and time (frequency). Therefore, the certified value is traceable to the International System of Units (SI).

Expiration of Certification

This certificate is valid for one year from the date of shipment, provided that this CRM is stored in accordance with the instructions given in this certificate.

Description of the material

This CRM is potassium hydrogen phthalate in the form of a white powder at a room temperature. Two hundred milligrams of the material was bottled in a brown glass vial and kept in an aluminum-laminated bag.

Instructions for Storage

This CRM should be stored at temperatures from 15 °C to 35 °C, at a relative humidity of 60 % or less and protected from light.

Instructions for Use

From the homogeneity, a minimum sample mass of 5 mg should be used. The CRM is for laboratory use only. The molar mass of this CRM, potassium hydrogen phthalate, (204.2206 ± 0.0034) g/mol ($k = 2$) (IUPAC 2013) can be used.

Precautions for Handling

Refer to the safety data sheet (SDS) on this CRM before use, and wear a protective mask and protective gloves when using this CRM.

Preparation

The raw material was purchased from Kanto Chemical Co., Inc. and subdivided by FUJIFILM Wako Pure Chemical Industries, Ltd. This CRM was bottled into a brown vial and sealed in an aluminum-laminated bag under dry argon atmosphere.

Technical Information

The solubility and chemical shifts in the NMR spectra of this CRM in widely used solvents are shown below. The following solubility and chemical shifts will be changed by temperature or coexisting solutes. Therefore, the overlap between signals originated from the calibrant (this CRM) and measurands has to be checked. This CRM is insoluble in acetonitrile- d_3 , tetrahydrofuran- d_6 , pyridine- d_5 , chloroform- d , dichloromethane- d_2 , acetone- d_6 , benzene- d_6 , toluene- d_8 (less than 0.5 mg/mL). The density of this CRM was 1.64 g/cm³ at 25 °C.

Solvent	Solubility (mg/mL) (25 °C)	Chemical shift (ppm)
		¹ H NMR (δ : 0 ppm (TMS), 25 °C)
Dimethylsulfoxide- d_6	10	7.49, 8.16
D ₂ O	≥ 20	7.58, 7.73
0.1 mol/L NaOD/D ₂ O	≥ 20	7.40, 7.47
Acetic acid- d_4	≥ 20	7.78, 7.99
Methanol- d_4	1	7.57, 8.20

TMS in the table shows the abbreviation for tetramethylsilane.

NMIJ Analysts

The technical manager for this CRM is ITOH N., the production manager is YAMAZAKI T., and the analysts are YAMAZAKI T., SHIMIZU Y., KITAMAKI Y., NAKAMURA S., and BAO X.

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.

Date of Shipment: Xxxxx xx, 20xx

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February 24, 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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Sample