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National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate NMIJ CRM 4049-a

> No. +++ Thiuram

National Institute of Advanced Industrial Science and Technology **AIST** 

This certified reference material (CRM) was produced based on NMIJ's quality system in compliance with JIS Q 0034 (ISO GUIDE 34). Intended uses for this CRM are calibration of analytical instruments and reference materials, quality control of analytical instrument and validation of analytical techniques and instruments.

## **Certified Value**

The certified value for the purity (in mass fraction) of thiuram is given in the following table. The expanded uncertainty was determined using coverage factor k = 2, corresponding to an estimated confidence interval of approximately 95%.

	CAS Number	Certified Value Mass fraction (kg/kg)	Expanded Uncertainty Mass fraction (kg/kg)
Thiuram (Bis(dimethylthiocarbamoyl)disulfide)	137-26-8	0.996	0.002

## **Analytical Methods**

The certified value was determined by subtracting the mass fractions of impurities measured by gas-chromatography with flame ionization detection (GC-FID), high performance liquid chromatography (HPLC) and Karl-Fischer titration (KF) and the mass fraction of thiuram measured by nuclear magnetic resonance (NMR).

## Traceability

The certified value of this CRM is based on the analytical results of subtraction method and NMR. Mass fractions of major impurities including ethyl acetate, tetramethylthiuram monosulfide and sodium dimethyldithiocarbamic acid were determined by using their calibration standards evaluated at NMIJ. The mass fraction of thiuram was obtained by subtracting the mass fractions of the impurities from 1. The mass fraction of thiuram was also evaluated by NMR using 1,4-bis(trimethylsilyl)benzene- $d_4$  calibrated by NMIJ CRM 4039-a (1,4-dichlorobenzene). The certified value obtained by the weighted mean of these values is traceable to the Internaional System of Units (SI).

# **Expiration of Certification**

The certification of this CRM is valid until March 31, 2020, provided that the material is unopened and stored in accordance with the instructions given in this certificate.

## Sample Form

The form of this CRM is white powder and a net amount of 100 mg is enclosed in a 10 mL amber glass vial, and packed in an aluminum-laminated bag.

## Homogeneity

The homogeneity of the CRM was determined by analyzing ten vials randomly selected out of 339 subdivided vials. Area percentages of thiuram measured by HPLC were evaluated to prove the homogeneity of the CRM. Variation of purity (mass fraction) between vials was estimated by the analysis of variance and was reflected in the uncertainty estimation of the certified

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value.

## **Precautions for Storage**

This CRM should be stored in a refrigerator (around 5 °C) at a clean and dark place.

### Precautions for use

Sampling more than 10 mg is recommended for ensuring homogeneity. Keep the CRM at room temperature before opening and use quickly after opening. Do not use the CRM other than testing and research purposes. The use, handling and storage of this CRM should be made observing the laws regulating those of thiuram.

## **Precautions for handling**

Wear protective equipments such as a safety mask and safety gloves in handling.

## **Preparation Method**

Raw material of this CRM was commercially available thiuram pre-selected by the quality evaluation by NMIJ. This CRM was prepared and subdivided into vials by Wako Pure Chemical Industries, Ltd. Vials were packed in laminated aluminum bags under Ar gas atmosphere.

## **Information Values**

This CRM contains ethyl acetate (2.8 g/kg, mass fraction), tetramethylthiuram monosulfide (0.05 g/kg, mass fraction) and dimethyldithiocarbamic acid salt (0.06 g/kg, mass fraction).

#### **NMIJ** Analysts

The technical manager for this CRM is M. Numata. The production manager is K. Ishikawa. The analysts are K. Ishikawa, N. Hanari, T. Saito, S. Otsuka, K. Higuchi, N. Fujiki, R. Iwasawa and T. Miura.

#### **Technical Information**

Customers will be notified of any revision to this CRM including a change in certified value. Technical information on this CRM can be obtained from the home page and other contact shown below.

## **Reproduction of Certificate**

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

March 30, 2010

Tamotsu Nomakuchi 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, Metrology Management Center, Reference Materials Office, 1-1-1, Umezono, Tsukuba, Ibaraki 305-8563, Japan Phone: +81-29-861-4059; Fax: +81-29-861-4009, http://www.nmij.jp/

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