

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 7508-a
No. +++

Pesticides in Cabbage

This certified reference material (CRM) was produced in accordance with the NMIJ's management system and in compliance with ISO GUIDE 34:2009 and ISO/ICE 17025:2005. This CRM is intended for use in the validation of analytical methods and instruments during analysis of pesticides (fenitrothion, chlorpyrifos and permethrin) in cabbage samples and similar materials.

Certified Values

The certified values, expressed as mass fractions, are given in the table below. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

Pesticides	CAS No.	Certified value Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)	Analytical Method
Fenitrothion (<i>O</i> , <i>O</i> -dimethyl- <i>O</i> -4-nitro- <i>m</i> -tolyl phosphorothioate)	122-14-5	2.41	0.20	1, 2
Chlorpyrifos (<i>O</i> , <i>O</i> -diethyl <i>O</i> -3, 5, 6-trichloro-2-pyridyl phosphorothioate)	2921-88-2	6.94	0.84	1, 2
Permethrin (3-phenoxybenzyl (1 <i>RS</i> , 3 <i>RS</i> ; 1 <i>RS</i> , 3 <i>RS</i>)-3-(2, 2'-dichlorovinyl)-2, 2-dimethylcyclopropanecarboxylate)	52645-53-1	5.75	0.50	1, 3

Analysis

The certified values of this CRM were based on the analytical results of pesticides by homogenization extraction and isotope dilution-gas chromatography/mass spectrometry (ID-GC/MS) described as follows;

1. [Extraction] Solvent, acetonitrile

[Clean-up] The extract was shaken with sodium chloride and phosphate buffer solution (pH 7.0) in a separatory funnel. The acetonitrile layer was dehydrated and dried, then, toluene/acetonitrile (1:3, v/v) was added. This was cleaned up by a solid phase extraction (graphite carbon/aminopropylsilanized silica gel).

[GC/MS] Column, DB-5MS; splitless injection; electron impact ionization (EI); selected ion monitoring (SIM)

2. [Extraction] Solvent, acetone

[Clean-up] The extract was shaken with ethyl acetate/hexane (1:4, v/v) and saturated sodium chloride aqueous solution in a separatory funnel. The ethyl acetate/hexane (1:4, v/v) layer was dehydrated and dried, then, hexane/acetone (1:1, v/v) was added. This was cleaned up by a solid phase extraction (silica gel).

[GC/MS] Column, DB-17MS; on-column injection; EI; SIM

3. [Extraction] Solvent, acetone

[Clean-up] The extract was shaken with hexane and 10 % sodium chloride aqueous solution in a separatory funnel. The hexane layer was dehydrated and dried, then, hexane was added. This was cleaned up by a solid phase extraction (Florisil).

[GC/MS] Column, DB-35MS; on-column injection; EI; SIM

Metrological Traceability

The certified values of this CRM were determined by IDMS. The purities of the high-purity pesticides were evaluated by NMIJ and the calibration solutions for the determination were prepared from these pesticides. The certified values are traceable to the International System of Units (SI).

Expiration of Certification

This certificate is valid for 3 months from the date of shipment, provided that the material is stored in accordance with the instructions given in this certificate.

Sample Form

This CRM was prepared from cabbage that was grown to contain the four pesticides. This CRM is in the form of a green powder and it of ca. 3 g in net volume is kept in an amber glass bottle.

Homogeneity

The homogeneity of this CRM was determined by analyzing 10 bottles selected by random sampling of 200 subdivided bottles. The inhomogeneity of the analyte was evaluated by ANOVA and was reflected in the uncertainty of the certified value.

Instructions for Storage

This CRM should be stored about -30 °C under dark condition.

Instructions for Use

The CRM should be equilibrated to room temperature before use. More than 0.2 g of the material should be used. If it's necessary, the water that is equivalent to about ten times of sample weight can be added. When the water was added, pay attention to the degradation of target pesticides.

Precautions for Handling

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

Preparation

This CRM was prepared to contain the target pesticides in Japan. The cabbage for the CRM was freeze-pulverized, homogenized, and bottled into 3-g portions. The bottled samples were sterilized by γ -ray irradiation with ^{60}Co and stored about -30 °C until required.

Technical Information

The concentration of etofenprox (2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzyl ether) determined in February, 2014 was 9 mg/kg.

The moisture content assessed by drying the sample in an oven at 105 °C to 110 °C for 24 h was approximately 12 %.

NMIJ Analysts

The technical manager and production manager for this CRM is M. Numata and T. Yarita, respectively. Analysts are T. Otake, T. Yarita, Y. Aoyagi, and Y. Kuroda.

Collaborator

A part of the preparation of CRM was carried out by the General Environmental Technos Co., Ltd under a contract with NMIJ. The homogeneity study was carried out by the Japan Food Research Laboratories under a contract with NMIJ.

Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the

registered customer. 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 by the Research and Development Projects for Application in Promoting New Policy of the Agriculture Forestry and Fisheries of the Ministry of Agriculture, Forestry and Fisheries, Japan.

April 1, 2015

Ryoji Chubachi
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

- March 30, 2012: The uncertainties for fenitrothion, chlorpyrifos and permethrin were changed.
- January 7, 2015: The uncertainties for fenitrothion and permethrin, the certified value and corresponding uncertainty of chlorpyrifos, the information for etofenprox were changed. Note was added.
- April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."