

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 7510-a
No. +++

Pesticides in Apple

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 the accuracy control of analysis and validation for analytical techniques for the determination of pesticides in apple.

Certified Values

The certified values of concentrations as mass fractions for diazinon, fenitrothion, permethrin, and cypermethrin 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
Diazinon (<i>O,O</i> -diethyl O-2-isopropyl-6-methylpyrimidin- 4-yl phosphorothioate)	333-41-5	2.28	0.54	1, 2
Fenitrothion (<i>O,O</i> -dimethyl- <i>O</i> -4-nitro- <i>m</i> -tolyl phosphorothioate)	122-14-5	3.14	0.72	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	2.81	0.54	1, 3
Cypermethrin (<i>(RS)</i> - α -cyano-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)	52315-07-8	1.55	0.33	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 (pH7.0) 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-17MS; 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-5MS; 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 (for Permethrin) and DB-5MS (for Cypermethrin); on-column injection; EI; SIM

Metrological Traceability

The certified values of this CRM were determined by IDMS as a primary method of measurement. 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 three months from the date of shipment, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

Sample Form

This CRM was prepared from apple, which was grown to contain the four pesticides. This CRM is in the form of a mixture of milky white powder and red powder, and it of ca. 3 g in net volume is kept in an amber glass bottle.

Homogeneity

The homogeneity of this CRM was evaluated by analyzing diazinon, fenitrothion, permethrin, and cypermethrin for 10 vials chosen from total 200 vials. The homogeneity of the analytes was evaluated by ANOVA and is reflected in the uncertainties of the certified values.

Instructions for Storage

This CRM should be stored at a temperature between $-20\text{ }^{\circ}\text{C}$ and $-30\text{ }^{\circ}\text{C}$ under dark conditions.

Instructions for Use

The CRM should be equilibrated to room temperature before use and should be used promptly, once the bottle is opened. 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, attention needs to be paid to the degradation of target pesticides.

Precautions for Handling

Wear a mask, gloves, and other protective equipment 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 apple for the CRM was freeze-dried, pulverized, sieved, and bottled into 3 g portions. The bottled samples were sterilized by γ -ray irradiation with ^{60}Co .

Technical Information

The moisture content assessed by drying the sample in an oven at $105\text{ }^{\circ}\text{C}$ to $110\text{ }^{\circ}\text{C}$ for 24 h was approximately 5 %.

NMIJ Analysts

The technical manager for this CRM is YARITA T. The production manager and analyst are OTAKE T.

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.

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

March 26, 2014: The expanded uncertainties were reevaluated based on the results of stability assessment after certification.

April 1, 2015: “Metrology Management Center” was renamed to “Center for Quality Management of Metrology.”

November 22, 2016: The expanded uncertainties were reevaluated based on the results of stability assessment after certification.