

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 7509-a  
No. +++

## Pesticides in Soybean

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 of analytical techniques for the determination of pesticides in soybean samples and similar materials.

**Certified Values**

The certified values of concentrations 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 %.

## Certified values of Pesticides

Pesticides	CAS No.	Certified value Mass fraction ( $\mu\text{g}/\text{kg}$ )	Expanded uncertainty Mass fraction ( $\mu\text{g}/\text{kg}$ )	Analytical Method
Diazinon ( <i>O,O</i> -diethyl <i>O</i> -2-isopropyl-6-methylpyrimidin-4-yl phosphorothioate)	333-41-5	21.7	2.2	1, 2
Fenitrothion ( <i>O,O</i> -dimethyl- <i>O</i> -4-nitro- <i>m</i> -tolyl phosphorothioate)	122-14-5	88	12	1, 2
Chlorpyrifos ( <i>O,O</i> -diethyl <i>O</i> -3, 5, 6-trichloro-2-pyridyl phosphorothioate)	2921-88-2	11.1	2.1	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	20.1	4.6	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 cleaned up by a series of solid phase extraction (octadecylsilanized silica gel and graphite carbon/aminopropylsilanized silica gel).

[GC/MS] Column, DB-5MS; splitless injection; electron 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 organic layer was cleaned up by acetonitrile/hexane partition. The acetonitrile layer was further cleaned up by a solid phase extraction (silica gel).

[GC/MS] Column, DB-35MS; 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 cleaned up by acetonitrile/hexane partition. The acetonitrile layer was further 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 the 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).

### **Mutual Recognition Arrangement under Meter Convention**

This certificate is consistent with the calibration and measurement capabilities (CMCs) that are included in Appendix C of the Mutual Recognition Arrangement (MRA) drawn up by the International Committee for Weights and Measures (CIPM). Under the MRA, all participating institutes recognize the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C (as for Appendix C of MRA, see <http://kcdb.bipm.org/AppendixC/default.asp>).

### **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 soybean, which was grown to contain the four pesticides. This CRM is in the form of a light-yellow powder and it of ca. 10 g in net volume is kept in an amber glass bottle.

### **Homogeneity**

The homogeneity of the CRM was determined by analyzing 10 bottles randomly sampled from 208 bottles. The inhomogeneity of the analytes was evaluated by ANOVA and is reflected in the uncertainty of the certified values.

### **Instructions for Storage**

This CRM should be stored about  $-30^{\circ}\text{C}$  and shielded from light.

### **Instructions for Use**

This CRM should be equilibrated to room temperature before use and should be used promptly, once the bottle is opened. From the homogeneity, more than 1 g of the material should be used.

### **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**

The raw soybean was prepared to contain the target pesticides in Japan. This was freeze-pulverized, mixed, and bottled into 10-g portions. The bottled samples were sterilized by  $\gamma$ -ray irradiation (15 kGy).

### **Collaborator**

A part of the preparation of the 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.

### **NMIJ Analysts**

The technical manager and production manager for this CRM are NUMATA M. and YARITA T., respectively. Analysts are YARITA T., OTAKE T., AOYAGI Y., and KURODA Y.

**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

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

January 11, 2017: The expanded uncertainties were reevaluated based on the results of stability assessment after certification.