

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 4214-a  
No. +++*p,p'*-DDT, *p,p'*-DDE,  $\gamma$ -HCH in 2,2,4-Trimethylpentane

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 calibration of analytical instruments, quality control of analytical instruments and validation of analytical techniques and instruments in the quantification of chlorinated pesticides.

**Certified Values**

The certified values of this CRM 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 %.

	CAS No.	Certified value Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)
<i>p,p'</i> -DDT (1,1,1-trichloro-2,2-bis[ <i>p</i> -chlorophenyl]ethane)	50-29-3	9.85	0.38
<i>p,p'</i> -DDE (1,1-dichloro-2,2-bis[ <i>p</i> -chlorophenyl]ethylene)	72-55-9	10.01	0.22
$\gamma$ -HCH ((1 $\alpha$ ,2 $\alpha$ ,3 $\beta$ ,4 $\alpha$ ,5 $\alpha$ ,6 $\beta$ )-hexachlorocyclohexane)	58-89-9	10.02	0.13

**Analysis**

The certified values of this CRM were obtained by the multiplying dilution ratio of the starting material (*p,p'*-DDT, etc.) in the gravimetric preparation with the purity of the material. The purity of *p,p'*-DDT was calibrated by the high-performance liquid chromatography using high-purity *p,p'*-DDT, whose purity was determined by the freezing point depression method with differential scanning calorimetry (DSC) and the mass balance method using a gas chromatograph with flame ionization detector (GC-FID). The purity of neat *p,p'*-DDE was determined by the continuous scan method with the DSC and GC-FID. The purity of neat  $\gamma$ -HCH was determined with the GC-FID.

**Metrological Traceability**

The mass fraction of the starting material in the prepared solution was calculated from the gravimetric preparation. The purity of the starting material was determined using the high-purity material that was assessed by the freezing point depression method in combination with the mass balance method. The certified values of this CRM calculated using these results 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**

The certification of this CRM is valid until March 31, 2017, provided that the material remains unopened and is stored in

accordance with the instructions given in this certificate.

### Sample Form

This CRM is in the form of a colorless and clear liquid at room temperature. This CRM of ca. 1 g in net volume is kept in an argon atmosphere in a 2-milliliter brown glass ampule.

### Homogeneity

The homogeneity of the CRM was determined by analyzing eleven ampoules selected by random sampling of 400 subdivided ampoules. The peak area of *p,p'*-DDT, *etc.* of the selected ampoules was measured with the GC-FID. The variation of the results was estimated by ANOVA. The homogeneity of the material is reflected in the uncertainties of the certified values.

### Instructions for Storage

This CRM should be stored at room temperature under clean condition, and shielded from light.

### Instructions for Use

Sample aliquots for analysis should be withdrawn immediately after opening the ampoules.

### Precautions for Handling

Keep away from heat and ignition sources. Wear personal protective equipment such as safety mask and gloves in handling. Use, handle, store and of this CRM should be made observing the laws regulating the components of this CRM. Refer to the safety data sheet (SDS) on this CRM before use.

### Preparation

This CRM was prepared by dissolving the high-purity materials of *p,p'*-DDT, *etc.* with 2,2,4-trimethylpentane.

### Technical Information

Density of this CRM is 0.6918 g/mL at 20 °C. This CRM contains 0.8 µg/kg of *p,p'*-DDMU (1-chloro-2,2-bis(*p*-chlorophenyl)ethylene), 92 µg/kg of *o,o'*-DDT and 17 µg/kg of *o,p'*-DDT. About 9.6 mg/kg of *p,p'*-DDD is also included in this CRM.

### Collaborator

High-purity materials of *p,p'*-DDE and  $\gamma$ -HCH were prepared by Wako Pure Chemical Industries, Ltd.

### NMIJ Analysts

The technical manager for this CRM is YARITA T. The production manager is ISHIKAWA K. The analysts are ISHIKAWA K., HANARI N., SHIMIZU Y., IHARA T., OTSUKA S., IWASAWA R., FUJIKI N., HIGUCHI K. and BAO X.

### 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:  
National Institute of Advanced Industrial Science and Technology,  
National Metrology Institute of Japan,  
Center for Quality Management of Metrology, Reference Materials Office,  
1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan  
Phone: +81-29-861-4059; Fax: +81-29-861-4009, <https://unit.aist.go.jp/nmij/english/refmate/>

Revision history

- April 5, 2011: According to the results of the stability assessment of this CRM, the certified value of  $p,p'$ -DDD was downgraded into an information and the uncertainties of other components were revised.  
The expiration date of this CRM was updated to March 31, 2017.
- April 5, 2014: The description on Mutual Recognition Arrangement under Meter Convention was added.
- April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."