

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 1583

#### Chlorinated Pesticides in 2, 2, 4-Trimethylpentane

This Standard Reference Material (SRM) is intended primarily for calibrating methods used in the determination of the chlorinated pesticides certified in this SRM. It can also be used for the purpose of adding known amounts of these pesticides to a sample in recovery studies or to fortify samples with known concentrations of these pesticides. The Chemical Abstracts Service (CAS) Nomenclature, common names, and CAS Registry Number of the six pesticide components are listed in Table 1.

Certified Concentrations of the Pesticides: The certified concentrations and estimated uncertainties of the pesticides are shown in Table 2.

Each value is based on the concentration calculated from the mass of the pesticide added to a known mass of 2,2,4-trimethylpentane (isooctane) and on the analytical results obtained by using capillary gas chromatography with electron capture detection (GC/ECD). The pesticides used were procured as being 99+ percent pure and GC and GC/MS analyses supported these claims. Table 3 shows the calculated concentrations and the concentrations obtained by the analytical methods used in the certification. A noncertified concentration of heptachlor epoxide, which is given for information only, is also listed in Table 3.

#### NOTICE AND WARNING TO USERS

Handling: Pesticide-containing materials are reported to be toxic and should be handled with care. Proper disposal methods should be used.

Expiration of Certification: This certification is valid within the specified uncertainty limits for one year from the date of purchase. In the event that the certification should become invalid before then, purchasers will be notified by NBS.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10 to 30 °C.

Use: Samples of the SRM for analysis should be withdrawn from ampoules (at  $23 \pm 5$  °C) immediately after opening and used without delay for the certified values listed in Table 2 to be valid within the stated uncertainty. Certified values are not applicable to material in ampoules stored after opening, even if they are resealed.

Preparation and analytical determinations were performed at the Center for Analytical Chemistry, Organic Analytical Research Division, by S.N. Chesler, D.P. Enagonio, L.R. Hilpert, R.M. Parris, and C.R. Vogt.

Consultation on the statistical design of the experimental work and evaluation of the data was provided by K.R. Eberhardt of the Statistical Engineering Division.

The coordination of the technical measurements leading to the certification was under the direction of S.N. Chesler, W.E. May, and R.M. Parris.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

## PREPARATION AND ANALYSIS

Pesticides and 2,2,4-trimethylpentane were obtained from commercial sources. The pesticide solution was prepared at NBS by weighing and mixing the individual pesticides and 2,2,4-trimethylpentane. This solution was dispensed into 2-mL amber ampoules which were then flame sealed. Aliquots from randomly selected ampoules were analyzed with a gas chromatograph equipped with an injector splitter and a 30 m x 0.25 mm nonpolar, immobilized phase wall-coated open-tubular column. A constant current Ni<sup>63</sup> electron capture detector was used for these analyses. Quantitative results were obtained by using 2,4',5-trichlorobiphenyl and 2,2',4,5,5'-pentachlorobiphenyl as internal standards (IS). Calibration solutions consisting of weighted amounts of the pesticides and IS compounds in 2,2,4-trimethylpentane were chromatographed to determine analyte response factors.

Table 1

Chemical Abstracts Service (CAS) Nomenclature and Registry Number<sup>a</sup>

<u>Pesticide</u>	<u>CAS Nomenclature</u>	<u>CAS Registry Number</u>
$\gamma$ -BHC Lindane	(1 $\alpha$ ,2 $\alpha$ ,3 $\beta$ ,4 $\alpha$ ,5 $\alpha$ ,6 $\beta$ )-1,2,3,4,5,6-hexachlorocyclohexane	58-89-9
$\delta$ -BHC	(1 $\alpha$ ,2 $\alpha$ ,3 $\alpha$ ,4 $\beta$ ,5 $\alpha$ ,6 $\beta$ )-1,2,3,4,5,6-hexachlorocyclohexane	319-86-8
Aldrin	(1 $\alpha$ ,4 $\alpha$ ,4 $\alpha\beta$ ,5 $\alpha$ ,8 $\alpha$ ,8 $\alpha\beta$ )-1,2,3,4,10,10-hexachloro-1,4,4 $\alpha$ ,5,8,8 $\alpha$ -hexahydro-1,4:5,8-dimethanonaphthalene	309-00-2
Heptachlor Epoxide	2,3,4,5,6,7,7-heptachlor-1 $\alpha$ ,1 $\beta$ ,5,5 $\alpha$ ,6,6 $\alpha$ -hexahydro-2,5-methano-2H-indeno[1,2-b]oxirene	1024-57-3
4,4'-DDE p,p'-DDE	1,1'-(dichloroethenylidene)bis[4-chlorobenzene]	72-55-9
4,4'-DDT p,p'-DDT	1,1'-(2,2,2-trichloroethylidene)bis[4-chlorobenzene]	50-29-3

<sup>a</sup>Chemical Abstracts, Tenth Collective Index, Index Guide, American Chemical Society, Columbus, Ohio, 1982.

Table 2  
Certified Concentrations of Pesticides in SRM 1583

Compound <sup>a</sup>	----- Concentration <sup>b</sup> -----	
	<u>μg/g</u>	<u>μg/mL<sup>c</sup>, 23 °C</u>
γ-BHC	1.11 ± 0.01	0.77 ± 0.01
δ-BHC	0.76 ± 0.01	0.53 ± 0.01
Aldrin	0.86 ± 0.01	0.59 ± 0.01
p,p'-DDE	1.23 ± 0.03	0.85 ± 0.02
p,p'-DDT	1.90 ± 0.10	1.31 ± 0.07

<sup>a</sup>See Table 1 for CAS Nomenclature.

<sup>b</sup>For each compound, the certified value is the mean of the calculated and GC/ECD determinations. The corresponding uncertainty represents the symmetrical interval about the certified value which encompasses the 95 percent confidence interval from the analyses that utilized gas chromatography with electron capture detection.

<sup>c</sup>The concentration and uncertainty expressed in mass/volume units are applicable for use of this material at 23 °C. Since the density of 2,2,4-trimethylpentane changes with temperature, the listed concentration will change by up to 1 percent of the value listed if the SRM is used at other temperatures in the 14.6 °C to 31.4 °C range. See "Selected Values of Properties of Hydrocarbons and Related Compounds," American Petroleum Institute Research Project 44, Thermodynamic Research Center, Texas A&M University, Table 3d, page 1, October 1952.

Table 3  
Summary of Results

Compound	----- Concentration, μg/g -----	
	<u>Calculated<sup>a</sup></u>	<u>GC/ECD<sup>b</sup></u>
γ-BHC	1.112 ± 0.001	1.109 ± 0.010
δ-BHC	0.768 ± 0.001	0.761 ± 0.007
Aldrin	0.861 ± 0.001	0.861 ± 0.010
p,p'-DDE	1.231 ± 0.001	1.232 ± 0.025
p,p'-DDT	1.900 ± 0.002	1.899 ± 0.100
Heptachlor Epoxide <sup>c</sup>	---	0.997 ± 0.016

<sup>a</sup>Uncertainty expressed as ± 2σ based on estimates of the precision of the weighings for the two balances used.

<sup>b</sup>Uncertainty expressed as 95 percent confidence interval.

<sup>c</sup>This value is for information only and is not certified.