



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1492

Chlorinated Pesticides in Hexane

This Standard Reference Material (SRM) is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of the certified compounds. This SRM is a solution of 15 chlorinated pesticides in hexane, with certified concentrations for 14 of the 15 pesticides. A unit of SRM 1492 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentrations of Constituent Pesticides: The certified concentrations and estimated uncertainties for 14 of the 15 pesticides are given in Table 1. These values are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography. Table 2 summarizes the calculated and chromatographically determined concentrations. Alternate names, Chemical Abstracts Service Nomenclature, and Registry Numbers of the certified components are listed in an appendix to the SRM 1492 Certificate of Analysis.

Expiration of Certification: The certification of SRM 1492 is valid, within the measurement uncertainty specified, until **31 January 2018**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

NOTICE AND WARNING TO USERS

Handling: This material contains chlorinated pesticide compounds, many of which have been reported to have toxic, mutagenic and/or carcinogenic properties, and should be handled with care. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

Instructions for Use: Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified values in Table 1 to be valid within the stated uncertainty. Because of the volatility of hexane, certified values are not applicable to material stored in ampoules that have been opened for more than three minutes, even if they are resealed.

Preparation and original analytical determinations were performed by R.M. Parris and F.R. Guenther of the NIST Analytical Chemistry Division.

The coordination of the technical measurements leading to the original certification was under the direction of S.N. Chesler and W.E. May of the NIST Analytical Chemistry Division.

Conformational analysis and coordination of stability measurements leading to updated certification in 1998 was under the direction of M.M. Schantz of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Robert L. Watters, Jr., Chief
Measurement Services Division

Gaithersburg, MD 20899
Certificate Issue Date: 25 January 2008
See Certificate Revision History on Page 3

Statistical consultations were provided by S.B. Schiller of the NIST Statistical Engineering Division.

Partial support for the preparation and certification of this Standard Reference Material was provided by the National Oceanographic and Atmospheric Administration, National Ocean Service, Office of Oceanography and Marine Assessment.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Table 1. Certified Concentrations of Chlorinated Pesticides in SRM 1492

| Compound | Concentration | |
|-------------------------|--|--|
| | ($\mu\text{g}/\text{kg}$) ^(a) | (ng/mL) ^(b) |
| Hexachlorobenzene | 308 \pm 2 | 205 \pm 2 |
| gamma-HCH | 310 \pm 2 | 207 \pm 2 |
| Heptachlor | 299 \pm 7 | 200 \pm 5 |
| Heptachlor epoxide | 307 \pm 7 | 204 \pm 5 |
| <i>cis</i> -Chlordane | 305 \pm 3 | 203 \pm 2 |
| <i>trans</i> -Nonachlor | 297 \pm 5 | 198 \pm 4 |
| Dieldrin | 307 \pm 4 | 205 \pm 3 |
| Mirex | 306 \pm 3 | 204 \pm 2 |
| 2,4'-DDE | 303 \pm 3 | 202 \pm 2 |
| 4,4'-DDE | 306 \pm 3 | 204 \pm 2 |
| 2,4'-DDD | 299 \pm 4 | 200 \pm 3 |
| 4,4'-DDD | 296 \pm 3 | 197 \pm 2 |
| 2,4'-DDT | 307 \pm 3 | 205 \pm 3 |
| 4,4'-DDT | 302 \pm 3 | 202 \pm 2 |

^(a) The certified value is the weighted average of the gravimetric and chromatographic concentrations. The uncertainty of the certified value is the half-width of an approximate 95 % confidence interval, plus an allowance for bias between analytical techniques.

^(b) The concentrations, in ng/mL units, were obtained by multiplying the certified value by the measured density of the SRM solution at 22 °C (0.667 g/mL). These concentrations are for use over the temperature range of 20 °C to 25 °C.

PREPARATION AND ANALYSIS

Pesticides used in the preparation of this SRM were obtained from the U.S. EPA Pesticides & Industrial Chemicals Repository, Research Triangle Park, NC and the Office of Reference Materials, Laboratory of the Government Chemist, United Kingdom. The pesticide solution was prepared at NIST by weighing and mixing the individual pesticides and hexane. The weighed components were added to the hexane and mixed until completely dissolved and homogenized. The total mass of this solution was measured and the concentrations calculated from this gravimetric procedure are given in Table 2 for 14 of the components. These gravimetric concentrations were adjusted for the consensus purity estimation of each component, which was determined using flame ionization high resolution gas chromatography, differential scanning calorimetry, and the purity assay information from the component suppliers. This bulk solution was then chilled to approximately -5 °C and 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules, which were then flame sealed.

Aliquots from twelve randomly selected ampoules were analyzed in duplicate by using electron capture capillary gas chromatography employing an immobilized non-polar stationary phase column. The four PCB internal standards added to each sample for quantification purposes were: PCB's 28, 66, 105, and 180 [1]. Calibration solutions consisting of weighed amounts of the pesticides (adjusted for the consensus purity estimation) and internal standard compounds in hexane were chromatographically analyzed to determine analyte response factors. The analytical values determined for 14 of the compounds also are given in Table 2.

During stability testing in August 1998, the aldrin content was found to be lower than originally certified. Therefore, the certified concentration of aldrin has been removed from the certificate, and because of its observed instability, a new value is not provided. A representative chromatogram from the GC analysis of the original solution is shown in Figure 1 on page 4.

Table 2. Summary of Results^(a)

| Compound | Concentrations | |
|-------------------------|---|--|
| | Gravimetric ^(b) ($\mu\text{g}/\text{kg}$) | GC/ECD ^(c) ($\mu\text{g}/\text{kg}$) |
| Hexachlorobenzene | 307 | 309 \pm 2 |
| gamma-HCH | 310 | 310 \pm 2 |
| Heptachlor | 297 | 301 \pm 3 |
| Heptachlor epoxide | 306 | 307 \pm 2 |
| <i>cis</i> -Chlordane | 304 | 306 \pm 2 |
| <i>trans</i> -Nonachlor | 296 | 297 \pm 2 |
| Dieldrin | 305 | 308 \pm 4 |
| Mirex | 304 | 308 \pm 4 |
| 2,4'-DDE | 302 | 304 \pm 2 |
| 4,4'-DDE | 306 | 307 \pm 3 |
| 2,4'-DDD | 299 | 301 \pm 3 |
| 4,4'-DDD | 296 | 297 \pm 3 |
| 2,4'-DDT | 307 | 308 \pm 2 |
| 4,4'-DDT | 302 | 302 \pm 2 |

^(a) The summary of results given above is presented for use only as background information.

^(b) Calculated concentration based on the mass of the pesticide added to the total mass of the solution.

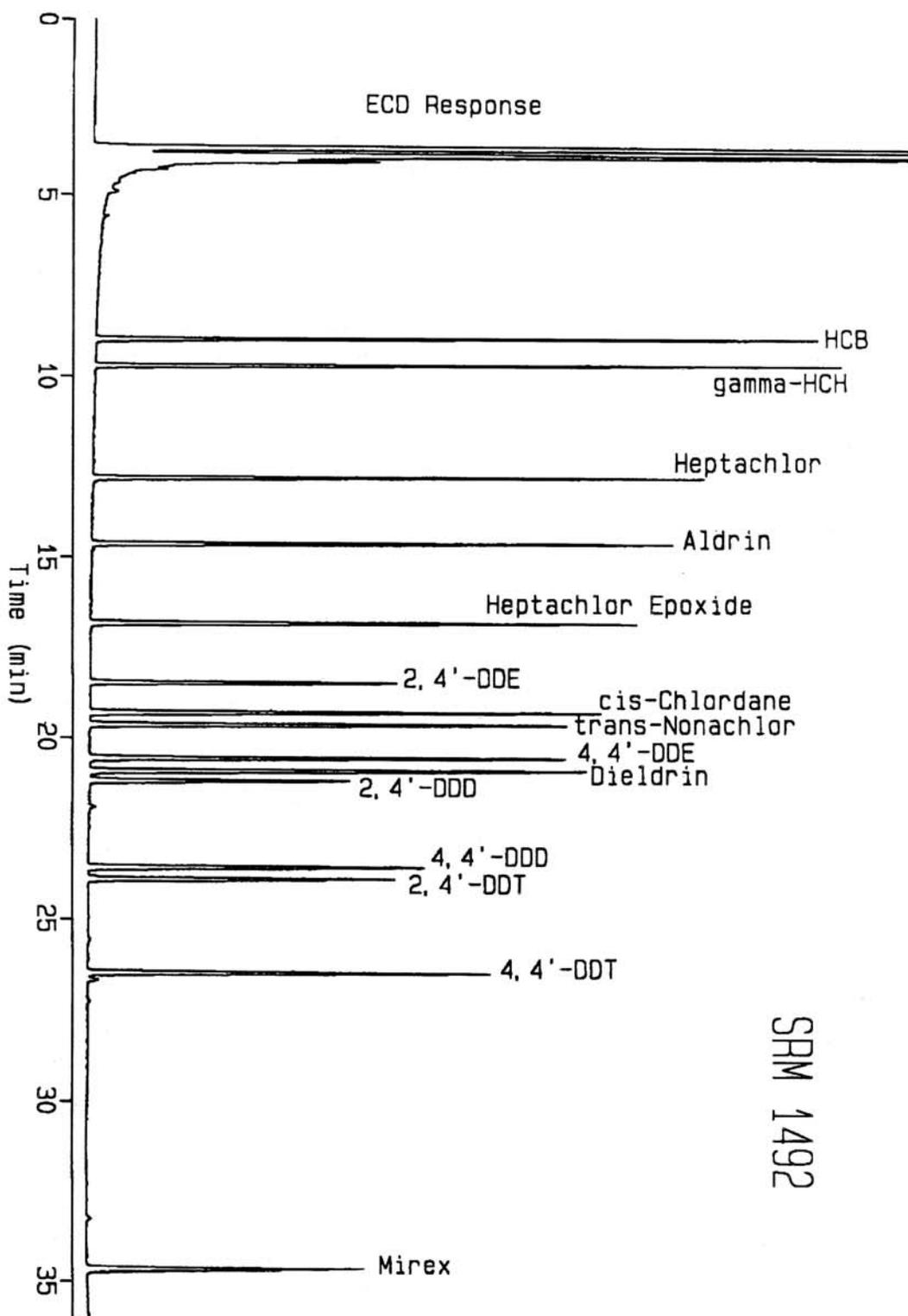
^(c) Concentrations determined by using gas chromatography with electron capture detection. The listed uncertainties represent one standard deviation of a single measurement for these results and recognize only the within-method variability.

REFERENCE

[1] Ballschmiter, K.; Zell, M.; *Fresenius Z. Anal.*; Chem. Vol. 302, pp. 20-31 (1980).

| |
|--|
| <p>Certificate Revision History: 25 January 2008 (Update of expiration date); 06 August 2003 (Update of expiration date.); 01 December 1998 (Removal of aldrin certification); 17 April 1989 (Original certificate date).</p> |
|--|

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.



SRM 1492

Figure 1. Capillary Gas Chromatogram of NIST SRM 1492.

Appendix to SRM Certificate
Standard Reference Material 1492

The following supplementary information may be of interest in connection with the use of this SRM and is supplied for the convenience of the user.

Table A-1. Name(s), Chemical Abstracts Service (CAS) Registry Numbers, and Nomenclature^(a)

| Compound (Alternative Name) | CAS Registry No. | CAS Nomenclature |
|---|--------------------------------|---|
| Hexachlorobenzene (HCB) | 118-74-1 | hexachlorobenzene |
| gamma-HCH (gamma-BHC) (Lindane) | 58-89-9 | (1 α ,2 α ,3 β ,4 α ,5 α ,6 β)-1,2,3,4,5,6-hexachlorocyclohexane |
| Heptachlor | 76-44-8 | 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methano-1H-indene |
| Aldrin (HHDN) | 309-00-2 | (1 α ,4 α ,4a β ,5 α ,8 α ,8 $\alpha\beta$)-1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-dimethanonaphthalene |
| Heptachlor epoxide | 1024-57-3 | (1 α ,1b β ,2 α ,5 α ,5a β ,6a α)-2,3,4,5,6,7,7-heptachloro-1a,1b,5,5a,6,6a-hexahydro-2,5-methano-2H-iden[1,2-b]oxirene |
| <i>cis</i> -Chlordane | 5103-71-9 (alpha-Chlordane) | (1 α ,2 α ,3a α ,4 β ,7 β ,7a α)-1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro-4,7-methano-1H-indene |
| <i>trans</i> -Nonachlor | 39765-80-5 | (1 α ,2 β ,3 α ,3a α ,4 β ,7 β ,7a α)-1,2,3,4,5,6,7,8,8-nonachloro-2,3,3a,4,7,7a-hexahydro-4,7-methano-1H-indene |
| Dieldrin (HEOD) | 60-57-1 | (1 α ,2 β ,2a α ,3 β ,6 β ,6a α ,7 β ,7a α)-3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-2,7:3,6-dimethanonaph[2,3-b]oxirene |
| Mirex (Dechlorane) (Perchlordecone) | 2385-85-5 | 1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachlorooctahydro-1,3,4-metheno-1H-cyclobuta[cd]pentalene |
| 2,4'-DDE (o,p'-DDE) | 3424-82-6 | 1-chloro-2-[2,2-dichloro-1-(4-chlorophenyl)ethenyl]benzene |
| 4,4'-DDE (p,p'-DDE) | 72-55-9 | 1,1'-(dichloroethylenidene)bis[4-chlorobenzene] |

| | | |
|--------------------------------------|----------|---|
| 2,4'-DDD (o,p'-DDD) (o,p'-TDE) | 53-19-0 | 1-chloro-2-[2,2-dichloro-1-(4-chlorophenyl)ethyl]benzene |
| 4,4'-DDD (p,p'-DDD) (p,p'-TDE) | 72-54-8 | 1,1'-(2,2-dichloroethylidene)bis[4-chlorobenzene] |
| 2,4'-DDT (o,p'-DDT) | 789-02-6 | 1-chloro-2-[2,2,2-trichloro-1-(4-chlorophenyl)ethyl]benzene |
| 4,4'-DDT (p,p'-DDT) | 50-29-3 | 1,1'-(2,2,2-trichloroethylidene)bis[4-chlorobenzene] |

^(a) Chemical Abstracts, Eleventh Collective Index. Index Guide, American Chemical Society, Columbus, Ohio, (1986).