



Certificate of Analysis

Standard Reference Material[®] 2261

Chlorinated Pesticides in Hexane (Nominal Mass Concentration 2 µg/mL)

This Standard Reference Material (SRM) is a solution of 15 chlorinated pesticides in hexane and is intended primarily for use in the calibration of gas chromatographic instrumentation used for the determination of chlorinated pesticides. A unit of SRM 2261 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Mass Fractions of Constituent Pesticides: The certified mass fractions and estimated uncertainties for 13 of the 15 pesticides are given in Table 1. These values are based on quantities used in the gravimetric preparation of this solution and from the analytical results obtained by using gas chromatography (GC) and are reported in mass fraction units [1]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [2].

Supplemental Information: A summary of the gravimetric and gas chromatographic measurements for SRM 2261 is provided in Table 2. This information is **NOT** to be used as a substitute for NIST certified values. A representative chromatogram from the GC analysis of the original solution is shown in Figure 1. In the “Appendix to SRM Certificate,” alternative names, Chemical Abstracts Service (CAS) Nomenclature, and CAS Registry Numbers of the components are listed in Table A-1.

Expiration of Certification: The certification of SRM 2261 is valid, within the measurement uncertainty specified, until **31 January 2018**, provided the SRM is handled and stored 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 certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

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 R.M. Parris and W.E. May of the NIST Analytical Chemistry Division.

Confirmation analysis and coordination of stability measurements leading to updated certification in 1998 and 2011 were under the direction of M.M. Schantz of the NIST Analytical Chemistry Division.

Statistical analysis was provided by S.B. Schiller of the Statistical Engineering Division.

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

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

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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 between 10 °C and 30 °C.

INSTRUCTIONS FOR USE

Open ampoules carefully to prevent contamination and injury. The ampoules are pre-scored and should **NOT** be opened using a file. 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 concentrations in Table 1 to be valid within the stated uncertainties. Because of the volatility of hexane, certified values are not applicable to material stored in ampoules that have been open for more than 3 minutes, even if they are resealed.

Table 1. Certified Mass Fractions of Chlorinated Pesticides in SRM 2261

Compound ^(a)	Mass Fraction ^(b) (mg/kg)	Mass Concentration ^(c) (µg/mL)
Hexachlorobenzene	3.005 ± 0.014	1.968 ± 0.009
<i>gamma</i> -HCH	3.012 ± 0.015	1.972 ± 0.010
Heptachlor	3.020 ± 0.023	1.977 ± 0.015
Heptachlor epoxide	3.020 ± 0.026	1.977 ± 0.017
<i>cis</i> -Chlordane	3.012 ± 0.019	1.972 ± 0.012
<i>trans</i> -Nonachlor	3.034 ± 0.022	1.986 ± 0.014
Dieldrin	3.012 ± 0.020	1.972 ± 0.013
Mirex	3.041 ± 0.042	1.991 ± 0.028
2,4'-DDE	3.019 ± 0.026	1.976 ± 0.017
4,4'-DDE	3.019 ± 0.015	1.976 ± 0.010
2,4'-DDD	3.013 ± 0.026	1.973 ± 0.017
2,4'-DDT	2.993 ± 0.014	1.959 ± 0.009
4,4'-DDT	3.004 ± 0.018	1.967 ± 0.012

^(a) The CAS Registry Number and CAS Nomenclature of each pesticide component are listed in the Appendix of this certificate.

^(b) The certified value is the equally weighted mean of the gravimetric and average chromatographic concentrations. The uncertainty of the certified value is the half-width of a 95 % confidence interval for the mean, with an allowance for systematic error between the methods [4,5].

^(c) The values listed in mass concentration units were obtained by multiplying the certified value in µg/g (prior to rounding) by the density of the SRM solution at 22.5 °C (0.6547 g/mL). These mass concentrations are for use in the temperature range of 20 °C to 25 °C, and an allowance for the change in the density over this temperature range is included in the uncertainties.

PREPARATION AND ANALYSIS

Pesticides used in the preparation of this SRM were donated by the U.S. EPA Pesticides & Industrial Chemicals Repository, Research Triangle Park, NC and the Office of Reference Materials, Laboratory of the Government Chemist (formerly National Physical Laboratory), United Kingdom. The weighed components were added to the hexane and mixed until completely dissolved and homogenized. The total mass of this solution was then measured. The calculated mass fraction based on the mass of the pesticide compound (adjusted for its consensus purity estimate) in the total mass of the solution is given in Table 2 for 13 of the components. The purity estimations of the chlorinated pesticide components were based on NIST analyses using capillary GC with flame ionization detection (FID), the purity assay information from the component suppliers, and, where appropriate, differential scanning calorimetry. This bulk solution was then chilled to approximately -5 °C, and 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules that were then flame sealed.

Aliquots from eight randomly selected ampoules were analyzed in duplicate by GC with electron capture detection (ECD) using an immobilized nonpolar stationary phase capillary column. The four polychlorinated biphenyl (PCB) congener internal standards added to each sample for quantification purposes were PCBs 82, 66, 105, and 180 [3]. Calibration solutions consisting of weighed amounts of NIST SRM 1492 Chlorinated Pesticides in Hexane and the hexane solution of internal standard compounds were chromatographically analyzed to determine analyte response factors. The chromatographically determined mass fractions of 13 of the compounds are also given in Table 2. A capillary gas chromatogram is shown in Figure 1.

During stability testing in August 1998, the aldrin content was found to be lower than originally certified, and during stability testing in November 2011, the 4,4'-DDD content was found to be lower than originally certified. Therefore, the certified mass fractions of aldrin and 4,4'-DDD have been removed from the certificate, and because of the observed instability, new values are not provided.

Table 2. Summary of Calculated and Chromatographic Results^(a)

Compound	Mass Fraction	
	Calculated ^(b) (mg/kg)	GC/ECD ^(c) (mg/kg)
Hexachlorobenzene	3.00	3.01 (0.03)
<i>gamma</i> -HCH	3.00	3.02 (0.02)
Heptachlor	3.01	3.03 (0.03)
Heptachlor epoxide	3.01	3.03 (0.02)
<i>cis</i> -Chlordane	3.00	3.02 (0.03)
<i>trans</i> -Nonachlor	3.03	3.04 (0.03)
Dieldrin	3.00	3.02 (0.03)
Mirex	3.01	3.07 (0.03)
2,4'-DDE	3.00	3.04 (0.03)
4,4'-DDE	3.01	3.02 (0.03)
2,4'-DDD	3.00	3.03 (0.03)
2,4'-DDT	2.99	2.99 (0.03)
4,4'-DDT	3.01	3.00 (0.03)

^(a) The summary of results given above is presented only as background information. These values are not certified.

^(b) Calculated mass fraction based on the mass of the pesticide (adjusted for purity, see text) in the total mass of the solution.

^(c) Mass fractions determined by using GC with electron capture detection. The uncertainties, listed in parentheses, are one standard deviation of a single measurement and only represent within-method variability.

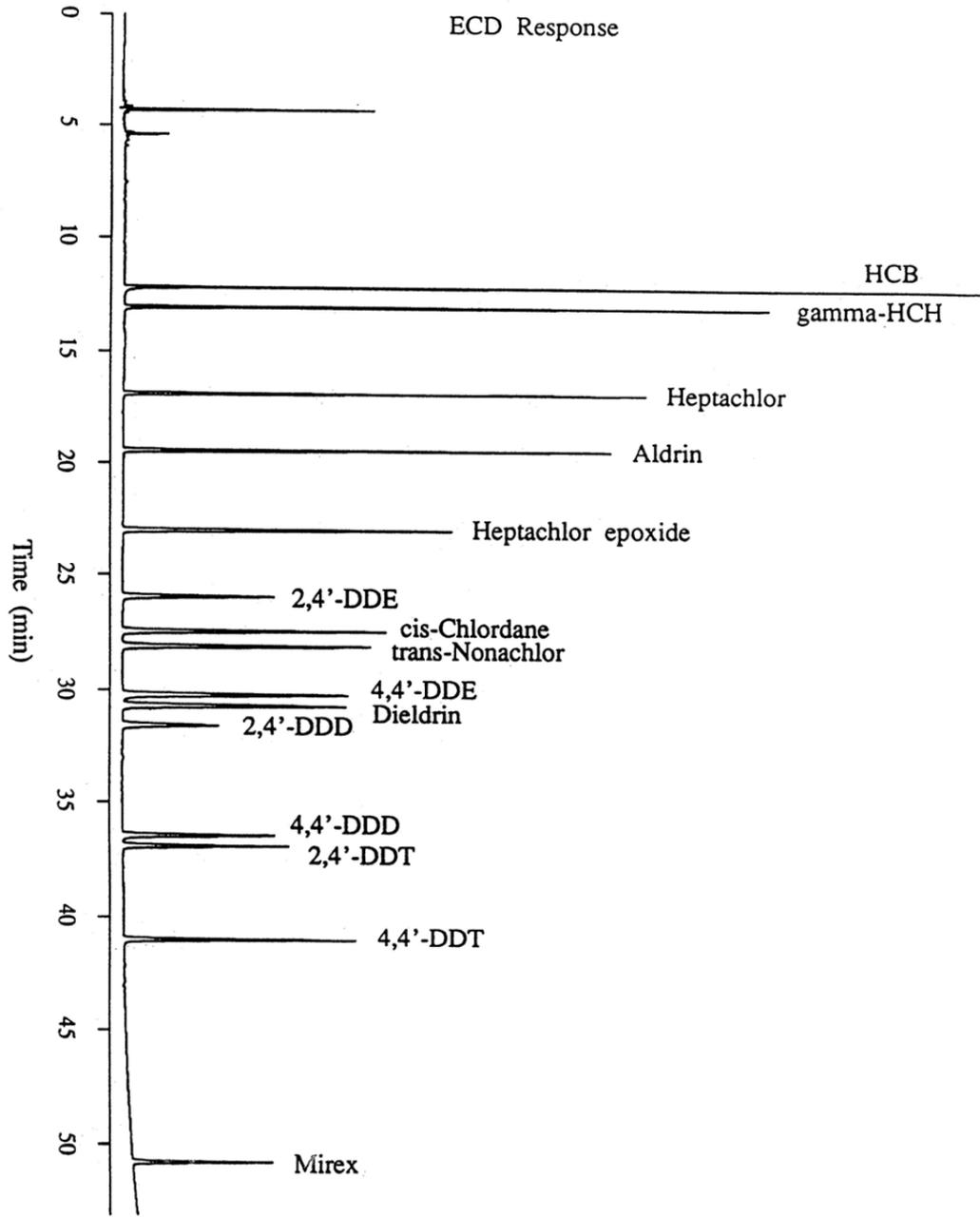


Figure 1. Capillary Gas Chromatogram of NIST SRM 2261.

REFERENCES

- [1] Thompson A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; National Institute of Standards and Technology, U.S. Government Printing Office: Washington, DC (2008); available at <http://www.nist.gov/pml/div684/fcdc/upload/sp811.pdf> (accessed Jan 2012).
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- [3] Ballschmiter, K.; Zell, M.; *Fresenius Z. Anal.; Chem.*, Vol. 302, pp. 20–31 (1980).
- [4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (ISO GUM 1995 with Minor Corrections)*; Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jan 2012); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <http://www.nist.gov/pml/pubs/index.cfm> (accessed Jan 2012).
- [5] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to Expression of Uncertainty in Measurement*; Propagation of Distributions Using a Monte Carlo Method; Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Jan 2012).

Certificate Revision History: 13 February 2012 (Removal of certified value for 4,4'-DDD; editorial changes); 25 January 2008 (Update of expiration date); 06 August 2003 (Update of expiration date); 10 December 1998 (Removal of certified value for aldrin); 10 January 1992 (Original certificate date).
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Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.

Appendix to SRM Certificate
SRM 2261

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. Names, 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 ,8a β)-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-indeno[1,2-b]oxirene
<i>cis</i> -Chlordane (alpha-Chlordane)	5103-71-9	(1 α ,2 α ,3 $\alpha\alpha$,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-dimethanonaphth[2,3-b]oxirene
Mirex (Dechlorane) (Perchlordecone)	2385-85-5	1,1a,2,2,3,3a,4,5,5a,5b,6-dodecachlorooctahydro-1,3,4-metheno-1H-cyclobuta[cd]pentalene
2,4'-DDE (<i>o,p'</i> -DDE)	3424-82-6	1-chloro-2-[2,2-dichloro-1-(4-chlorophenyl)ethenyl]benzene
4,4'-DDE (<i>p,p'</i> -DDE)	72-55-9	1,1'-(dichloroethenyldiene)bis[4-chlorobenzene]
2,4'-DDD (<i>o,p'</i> -DDD) (<i>o,p'</i> -TDE)	53-19-0	1-chloro-2-[2,2-dichloro-1-(4-chlorophenyl)ethyl]benzene

4,4'-DDD (<i>p,p'</i> -DDD) (<i>p,p'</i> -TDE)	72-54-8	1,1'-(2,2-dichloroethylidene)bis[4-chlorobenzene]
2,4'-DDT (<i>o,p'</i> -DDT)	789-02-6	1-chloro-2-[2,2,2-trichloro-1-(4-chlorophenyl)ethyl]benzene
4,4'-DDT (<i>p,p'</i> -DDT)	50-29-3	1,1'-(2,2,2-trichloroethylidene)bis[4-chlorobenzene]

^(a) Chemical Abstract, Eleventh Collective Index, Index Guide, American Chemical Society, Columbus, OH (1986).