National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 2275

Chlorinated Pesticide Solution-II in 2,2,4-Trimethylpentane

This Standard Reference Material (SRM) is a solution of eight chlorinated pesticides in 2,2,4-trimethylpentane (isooctane) intended primarily for use in the calibration of chromatographic instrumentation. A unit of SRM 2275 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentrations of Constituent Pesticides: The certified concentration values, expressed as mass fractions, for eight 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. 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 accounted for by NIST [1]. The measurands are listed in Table 1 and the certified values are metrologically traceable to the SI derived unit for mass fraction, expressed as milligrams per kilogram.

Information Values: A summary of the gravimetric and gas chromatographic measurements for SRM 2275 is provided in Table 2. This information is **NOT** to be used as a substitute for NIST certified values. Information values cannot be used to establish metrological traceability.

Additional Information: Table 3 lists alternative names, Chemical Abstracts Service (CAS) nomenclature, and registry numbers of the certified components. Also, a representative chromatogram from the gas chromatographic analysis is shown in Figure l.

Expiration of Certification: The certification of **SRM 2275** is valid, within the measurement uncertainty specified, until **31 January 2030**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The 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 or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification on this SRM was under the direction of M.M. Schantz and S.A. Wise formerly of NIST.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.B. Schiller of NIST and S.D. Leigh of the NIST Statistical Engineering Division.

Partial support for the preparation and certification of this SRM was provided by the National Oceanic and Atmospheric Administration, National Ocean Service, Center for Coastal Monitoring and Assessment (Silver Spring, MD).

Analytical measurements of the SRM were performed by R.M. Parris and L.K. Walton both formerly of NIST, and D.L. Poster of NIST.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 22 April 2020
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

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.

Opening of Ampoule: 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 values in Table 1 to be valid within the stated uncertainties. Because of the volatility of 2,2,4-trimethylpentane, certified values are not applicable to material stored in ampoules that have been opened for more than 5 minutes, even if they are resealed.

PREPARATION AND ANALYSIS

SRM Preparation: Pesticides used in the preparation of this SRM were obtained from the Office of Reference Materials, Laboratory of the Government Chemist (United Kingdom); Ultra Scientific (North Kingston, RI); and AccuStandard (New Haven, CT). The pesticide solution was prepared at NIST by weighing and mixing the individual pesticides and 2,2,4-trimethylpentane. The weighed components were added to the 2,2,4-trimethylpentane and mixed until completely dissolved and homogenized. The total mass of this solution was measured and the concentrations calculated for the components (see Table 2). These gravimetric concentrations were adjusted for the consensus purity estimation of each component that was determined using capillary gas chromatography with flame ionization detection, 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 that were then flame sealed.

SRM Analysis: Aliquots from nine ampoules selected using a random stratified sampling scheme were analyzed in duplicate by using capillary gas chromatography with electron capture detection employing an immobilized non-polar stationary phase column. The two deuterated internal standards added to each sample for quantification purposes were: endosulfan-I-d_4 and 4,4'-DDT-d_8. Calibration solutions consisting of weighed amounts of the pesticides (adjusted for the consensus purity estimation) and internal standard compounds in 2,2,4-trimethylpentane were chromatographically analyzed to determine analyte response factors. The analytical values determined for the compounds also are given in Table 2.

Table 1. Certified Concentrations of Chlorinated Pesticides in SRM 2275

Compound	Concentration (mg/kg)[a]	Concentration (µg/mL)[b]
alpha-HCH	3.00 ± 0.15	2.07 ± 0.10
beta-HCH	2.98 ± 0.12	2.054 ± 0.083
Oxychlordane	2.86 ± 0.12	1.976 ± 0.083
trans-Chlordane (*gamma*-Chlordane)	2.954 ± 0.060	2.038 ± 0.041
Endosulfan-I	2.880 ± 0.067	1.987 ± 0.046
Endosulfan-II	2.943 ± 0.069	2.031 ± 0.048
cis-Nonachlor	2.938 ± 0.076	2.027 ± 0.052
Endosulfan sulfate	2.926 ± 0.087	2.019 ± 0.060

[a] Each result is expressed as the certified value ± the expanded uncertainty. The certified value is the unweighted average of the concentrations determined by gravimetric and chromatographic measurements. The expanded uncertainty, at the 95 % level of confidence, is calculated as, $U = ku_c$, where u_c is a combined standard uncertainty calculated according to the ISO/JCGM and NIST Guides [2] and $k = 2$ is the coverage factor. The value of u_c includes both a correction for estimated purity and an allowance for differences between the concentration determined by gravimetric preparation and chromatographic measurements.

[b] The certified concentrations in micrograms per milliliter units, were obtained by multiplying the certified value by the measured density of the SRM solution at 22 °C (0.6899 g/mL). These concentrations are for use over the temperature range of 20 °C to 25 °C, and an allowance for the change in density over this temperature range is included in the uncertainties.

Table 2. Information Values for Chlorinated Pesticides in SRM 2275[a]

Compound	Concentrations Gravimetric[b] (mg/kg)	Concentrations GC-ECD[c] (mg/kg)
alpha-HCH	2.913	3.09 (± 0.13)
beta-HCH	2.913	3.078 (± 0.086)
Oxychlordane	2.924	2.808 (± 0.083)
trans-Chlordane (*gamma*-Chlordane)	2.953	2.957 (± 0.060)
Endosulfan-I	2.889	2.885 (± 0.048)
Endosulfan-II	2.953	2.935 (± 0.057)
cis-Nonachlor	2.921	2.967 (± 0.061)
Endosulfan sulfate	2.898	2.966 (± 0.059)

[a] Results 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 corrected for the chemical purity.

[c] Concentrations determined by using gas chromatography with electron capture detection (GC-ECD) corrected for the purity of the compounds. The listed uncertainties in parentheses represent one standard deviation of a single measurement for these results and recognize only the within-method variability.

Table 3. Name(s), CAS Nomenclature and Registry Numbers

Compound (Alternative Name)	CAS Nomenclature[a]	CAS Registry Number[a]
alpha-HCH (*alpha*-BHC)	(1α,2α,3β,4α,5β,6β)-1,2,3,4,5,6-hexachlorocyclohexane	319-84-6
beta-HCH (*beta*-BHC)	(1α,2β,3α,4β,5α,6β)-1,2,3,4,5,6-hexachlorocyclohexane	319-85-7
Oxychlordane	2,3,4,5,6,6a,7,7-octachloro-1a,1b,5,5a,6,6a-hexahydro-2,5-methano-2H-indeno(1,2-b)oxirene	27304-13-8
gamma-Chlordane (*trans*-Chlordane)	(1α,2β,3aα,4β,7β7aα)-1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro-4,7-methano-1H-indene	5103-74-2
Endosulfan-I (*alpha*-Endosulfan)	(3α,5aβ,6α,9α,9aβ)-(1,4,5,6,7,7-hexachloro-8,9,10-trinorborn-5-en-2,3-ylenebismethylene)sulphite	959-98-8
Endosulfan-II (*beta*-Endosulfan)	(3α,5aα,6β,9β,9aβ)-(1,4,5,6,7,7-hexachloro-8,9,10-trinorborn-5-en-2,3-ylenebismethylene)sulphite	33213-65-9
cis-Nonachlor	(1α,2α,3α,3aα,4β,7β7aα)-1,2,3,4,5,6,7,8,8-nonachloro-2,3,3a,4,7,7a-hexahydro-4,7-methano-1H-indene	5103-73-1
Endosulfan sulfate	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin,3,3-dioxide	1031-07-8

[a] Chemical Abstracts, Thirteenth Collective Index, Index Guide, American Chemical Society, Columbus, Ohio, 1996.

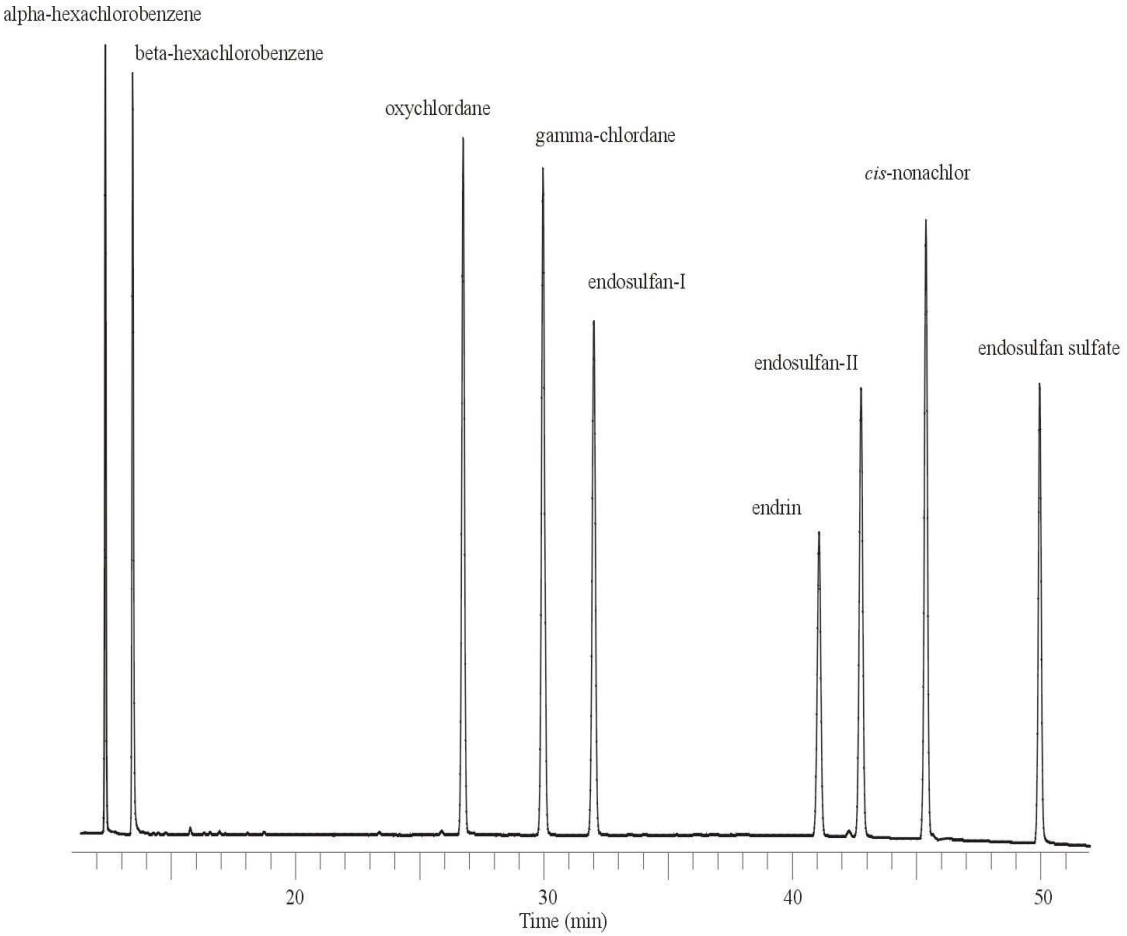

Figure 1. Example of the gas chromatographic elution order of analytes in SRM 2275 on a 5% phenyl-substituted methylpolysiloxane capillary column (60 m × 0.25 mm i.d., 0.25 µm film thickness) with electron capture detection. Note: Endrin concentration is no longer certified in this SRM.

REFERENCES

[1] May, W., Parris, R., Beck, C., Fassett, J., Greenberg, R., Guenther, F., Kramer, G., Wise, S., Gills, T., Colbert, J., Gettings, R., MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at https://www.nist.gov/system/files/documents/srm/SP260-136.PDF (accessed Apr 2020).

[2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2020); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Apr 2020).

Certificate Revision History: 22 April 2020 (Change of expiration date; removal of endrin certified and information values due to observed instability; clarification of storage instructions; editorial changes); 06 November 2009 (This revision reflects at extension of the certification period and editorial changes); 01 August 2001 (Original certificate date).

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 at: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet https://www.nist.gov/srm.