National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3069

Organochlorine Pesticides-I in Acetone

Standard Reference Material (SRM) 3069 is a solution of 22 organochlorine pesticides in acetone intended primarily for use in the calibration of chromatographic instrumentation used for the determination of the components in the certified mixture. Because of its miscibility with water, SRM 3069 can also be used to fortify samples with known amounts of the 22 organochlorine pesticides. A unit of SRM 3069 consists of five 2 mL ampoules, each containing 1.2 mL of solution.

Certified Concentration of Constituents: The certified concentration values [1,2] for the 22 organochlorine 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 with electron capture detection (GC-ECD). 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.

Supplemental Information: Chemical Abstracts Service (CAS) Registry Numbers of the certified components are listed in Table 2.

Expiration of Certification: The certification of this SRM is valid until **31 July 2011**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Coordination of the technical measurements leading to the certification was under the direction of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

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Preparation and analytical measurements of the SRM were performed by M.P. Cronise of the NIST Standard Reference Materials Program and M.M. Schantz and C.R. Mack of the NIST Analytical Chemistry Division.

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

INSTRUCTIONS FOR USE

Handling: This material contains organochlorine pesticides 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.

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 value to be valid within the stated uncertainty. Because of the volatility of methanol, 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: The organochlorine pesticides used in the preparation of this SRM were obtained from commercial sources. The solution was prepared at NIST by weighing and mixing the individual components into the acetone. The weighed components were added to the acetone and mixed until completely dissolved and homogenized. The total mass of this solution was measured and the concentration calculated from this gravimetric procedure. These gravimetric concentrations were adjusted for the consensus purity estimation of each component, which was determined by using capillary gas chromatography with flame ionization detection and 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, which were then flame sealed.

SRM Analysis: Aliquots from nine ampoules, selected according to a modified, random number generator scheme, were analyzed in duplicate by using GC-ECD employing an immobilized non-polar (5 % phenylmethylpolysiloxane) stationary phase column. An internal standard solution containing 2,2',4,6'-tetrachlorobiphenyl (PCB 52), 2,2',4,5',6-pentachlorobiphenyl (PCB 103), and 2,2',4,4',5,5'-hexachlorobiphenyl (PCB 153) was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of the 22 organochlorine pesticides and the three internal standard compounds in acetone were chromatographically analyzed to determine analyte response factors.

Table 1. Certified Concentrations of the 22 Organochlorine Pesticides in SRM 3069

Compound	Concentration					
	mg/kg[a]			mg/L[b]		
Aldrin	2.08	±	0.05	1.63	±	0.04
cis-Chlordane	4.08	±	0.12	3.21	±	0.09
trans-Chlordane	3.49	±	0.08	2.74	±	0.07
4,4'-DDE	4.72	±	0.11	3.71	±	0.08
4,4'-DDD	7.56	±	0.18	5.94	±	0.14
4,4'-DDT	9.02	±	0.18	7.09	±	0.14
Dieldrin	2.82	±	0.06	2.22	±	0.05
Endosulfan I	4.66	±	0.10	3.66	±	0.08
Endosulfan II	5.29	±	0.12	4.15	±	0.09
Endrin	5.52	±	0.15	4.34	±	0.12
β-HCH	4.34	±	0.09	3.41	±	0.07
δ-HCH	5.66	±	0.16	4.45	±	0.13
γ-HCH	4.22	±	0.12	3.31	±	0.10
Heptachlor	5.12	±	0.11	4.02	±	0.08
Heptachlor epoxide	2.71	±	0.06	2.13	±	0.05
Hexachlorobenzene	4.39	±	0.09	3.45	±	0.07
Hexachlorocyclopentadiene	5.49	±	0.16	4.31	±	0.13
Methoxychlor	101	±	3	79.4	±	2.0
cis-Nonachlor	4.66	±	0.11	3.66	±	0.08
trans-Nonachlor	5.52	±	0.11	4.34	±	0.09
Propachlor	4.25	±	0.10	3.34	±	0.08
Trifluralin	5.34	±	0.11	4.20	±	0.09

[a] The results are expressed as the certified value ± an 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 Guide [1] 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 concentrations determined by gravimetric preparation and chromatographic measurements.

[b] The concentration in mg/L was obtained by multiplying the certified value, expressed as a mass fraction, by the measured density of the SRM solution at 22 °C (0.7857 g/mL). This concentration is 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 uncertainty.

Table 2. Compound Names and CAS Registry Numbers for the Components in SRM 3069[a]

Common Name	Chemical Name	CAS Registry Number
Aldrin	1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-, (1α,4α,4aβ,5α,8α,8aβ)-1,4:5,8-dimethanonaphthalene	309-00-2
cis-Chlordane	1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a hexahydro-(1α,2α,3aα,4β,7β,7aα) 4,7-methano-1H-indene	5103-71-9
trans-Chlordane	1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro-(1α,2β,3aα,4β,7β,7aα) 4,7-methano-1H-indene	5103-74-2
4,4'-DDE	1,1'-(dichloroethenylidene)bis(4-chloro)-benzene	72-55-9
4,4-DDD	1,1'-(2,2-dichloroethylidene)bis(4-chloro)-benzene	72-54-8
4,4'-DDT	1,1'-(2,2,2-trichloroethylidene)bis(4-chloro)-benzene	50-29-3
Dieldrin	3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a- octahydro-, (1aα,2β,2aα,3β,6β,6aα,7β,7aα)-2,7:3,6-dimethanonaphth (2,3-b)oxirene	60-57-1
Endosulfan-I	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,hexahydro-, 9a-3-oxide, (3α,5aβ,6α,9α,9aβ)-6,9-methano-2,4,3-benzodioxathiepin	959-98-8
Endosulfan-II	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-, 3-oxide, (3α,5aα,6β,9βα,9aα)-6,9-methano-2,4,3-benzodioxathiepin	33213-65-9
Endrin	3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-, (1aα,2β,2aβ,3α,6α,6aβ,7β,7aα)-2,7:3,6-dimethanonaphth(2,3-b)oxirene	72-20-8
β-HCH	1,2,3,4,5,6-hexachloro-, (1α,2β,3α,4β,5α, 6β isomer)-cyclohexane	319-85-7
δ-HCH	1,2,3,4,5,6-hexachloro-, (1α,2α,3α,4β,5α, 6β isomer)-cyclohexane	319-86-8
γ-HCH	1,2,3,4,5,6-hexachloro-, (1α,2α,3β,4α,5α, 6β isomer)-cyclohexane	58-89-9
Heptachlor	1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methano-1H-indene	76-44-8
Heptachlor epoxide	2,3,4,5,6,7,7-heptachloro-1a,1b,5,5a,6,6a-hexahydro-, (1aα,1bβ,2α,5α,5aβ,6aα)-2,5-methano-2H-indeno(1,2-b)oxirene	1024-57-3
Hexachlorobenzene (HCB)	Hexachlorobenzene	118-74-1

Table 2 (cont.). Compound Names and CAS Registry Numbers for the Components in SRM 3069[a]

Common Name	Chemical Name	CAS Registry Number
Hexachlorocylopentadiene	Hexachlorocylopentadiene	77-47-4
Methoxychlor	1,1'-(2,2,2-trichloroethylidene)bis(4-methyoxy)-benzene	72-43-5
cis-Nonachlor	1,2,3,4,5,6,7,8,8-nonachloro-2,3,3a, 4,7,7a-hexahydro-(2α) 4,7-methano-1H-indene	5103-73-1
trans-Nonachlor	1,2,3,4,5,6,7,8,8-nonachloro-2,3,3a, 4,7,7a-hexahydro-(2β) 4,7-methano-1H-indene	39765-80-5
Propachlor	2-chloro-N-(1-methylethyl)-N-phenyl-acetamide	1918-16-7
Trifluralin	2,6-dinitro-N,N-dipropyl-4-(trifluoromethyl)-benzenamine	1582-09-8

[a] Chemical Abstracts, Thirteenth Collective Index, Index Guide, American Chemical Society, Columbus, OH (1996).

REFERENCES

[1] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R; Guenther; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definition of Terms and Modes Used at NIST for Value-Assessment of Reference Materials for Chemical Measurements*, NIST Special Publication 260-136 (2000).

[2] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*, NIST Special Publication 811, 1995 Ed. (1995).

[3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland (1993); see also Taylor, B.N. and 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://physics.nist.gov/Pubs/.

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 http://www.nist.gov/srm.