



Certificate of Analysis

Standard Reference Material[®] 3068

Total Chlordane in Methanol

This Standard Reference Material (SRM) is a solution of technical chlordane (Chemical Abstracts Service Registry Number 12789-03-6) in methanol. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of the certified mixture. Because of its miscibility with water, SRM 3068 can also be used to fortify aqueous samples with known amounts of chlordane. A unit of SRM 3068 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

Certified Value of Total Chlordane: The certified value [1,2] is 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 taken into account.

Certified Value of Total Chlordane: 22.35 mg/kg \pm 0.67 mg/kg or 17.84 mg/L \pm 0.54 mg/L

The results are expressed as the certified value \pm the expanded uncertainty. The certified value is the unweighted average of the concentrations determined by gravimetric and gas chromatographic measurements. The expanded uncertainty, U , is calculated as $U = ku_c$, where $k = 2$ is the coverage factor for a 95 % confidence interval. The quantity u_c is the combined standard uncertainty calculated according to the ISO Guide [3]. The value of u_c includes an allowance for differences between the concentration determined by gas chromatographic measurements for various sources of chlordane and gravimetric preparation. The certified value expressed as a volume fraction (in milligrams per liter) was obtained by multiplying the certified value, expressed as a mass fraction, by the measured density (22 °C) of the SRM solution, 0.798 (0.013) g/mL where 0.013 represents one standard deviation (1s) and is incorporated in the volume fraction uncertainty.

Expiration of Certification: The certification of **SRM 3068** is valid, within the measurement uncertainty specified, until **31 March 2020**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Warning and 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) 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 Chemical Sciences Division.

Analytical measurements of the SRM were performed by M.M. Schantz and S.J. Broadwater of the NIST Chemical Sciences Division.

Preparation of SRM 3064 was performed by M.P. Cronise of the NIST Office of Reference Materials and by M.M. Schantz.

Statistical consultation was provided by S.D. Leigh of the NIST Statistical Engineering Division.

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

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Gaithersburg, MD 20899
Certificate Issue Date: 07 December 2012
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Robert L. Watters, Jr., Director
Office of Reference Materials

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WARNING AND INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling and Storage: This material contains total chlordane, and should be handled with care. Use proper disposal methods. Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

Use: Sample aliquots for analysis (minimum sample size of 0.5 mL) 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⁽¹⁾

The chlordane used in the preparation of this SRM was obtained from the former U.S. EPA Repository, Research Triangle Park, NC. The solution was prepared at NIST by weighing and mixing the chlordane into the methanol. The weighed chlordane was added to the methanol and mixed until completely dissolved and homogenized. The total mass of this solution was measured and 1.2 mL aliquots were dispensed into 2-mL amber glass ampoules, which were then flame sealed.

Aliquots from two sets of nine ampoules, selected randomly, were analyzed in duplicate using capillary gas chromatography with electron capture detection and an immobilized non-polar stationary phase column. An internal standard solution containing Polychlorinated Biphenyl (PCB) 103 and PCB 198 was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of chlordane (from the same source as used to prepare SRM 3068 for the first set of nine ampoules and from four different sources for the second set of nine ampoules) and internal standard compounds in methanol were chromatographically analyzed to determine response factors for dominant chlordane peaks relative to the internal standards (See Figure 1). This approach is similar to U.S. EPA Method 505 [4].

REFERENCES

- [1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at <http://www.nist.gov/srm/upload/SP260-136.PDF> (accessed Dec 2012).
- [2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office; Washington, DC (2008); available at <http://www.nist.gov/pml/div684/fcdc/upload/sp811.pdf> (accessed Dec 2012).
- [3] 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 Dec 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/physlab/pubs/index.cfm> (accessed Dec 2012).
- [4] Winfield, T.W.; *Method 505 Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products in Water by Microextraction and Gas Chromatography*; revision 2.0 (1989); available at http://water.epa.gov/scitech/methods/cwa/bioindicators/upload/2007_11_06_methods_method_505.pdf (accessed Dec 2012).

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| Certificate Revision History: 07 December 2012 (Extension of certification period; editorial changes); 14 May 2003 (Original certification 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) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.

⁽¹⁾Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

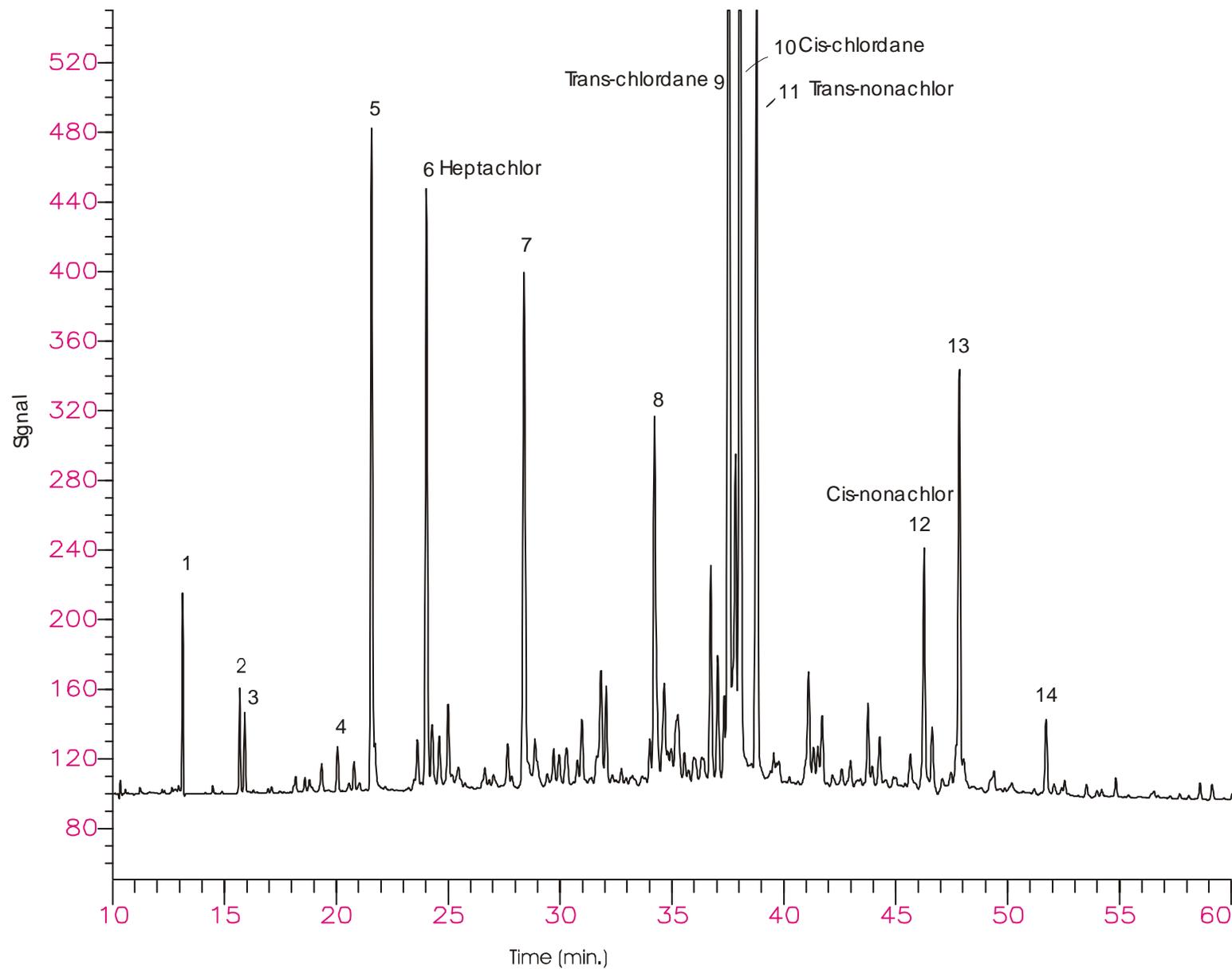


Figure 1: Five of the main components in technical chlordane are heptachlor (1.1 mg/kg), *trans*-chlordane (1.7 mg/kg), *cis*-chlordane (2.6 mg/kg), *trans*-nonachlor (1.6 mg/kg), and *cis*-nonachlor (0.3 mg/kg). The concentrations given are for informational purposes only.