



# Certificate of Analysis

## Standard Reference Material<sup>®</sup> 3067

### Toxaphene in Methanol

This Standard Reference Material (SRM) is a solution of technical toxaphene (Chemical Abstracts Registry Number 8001-35-2) in methanol. This SRM is intended primarily for calibrating chromatographic instrumentation used for the determination of technical toxaphene. Because of its miscibility with water, SRM 3067 can also be used to fortify aqueous samples with known amounts of toxaphene. A unit of SRM 3067 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

**Certified Concentration of Toxaphene:** The certified concentration [1] given below is based on the gravimetric preparation of this solution and from 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 [2].

Certified Concentration of Toxaphene: 26.1 mg/kg  $\pm$  1.0 mg/kg or 20.9 mg/L  $\pm$  0.8 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 gravimetry and gas 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 [3] and  $k = 2$  is the coverage factor. The value of  $u_c$  includes an allowance for differences between the concentration determined by gas chromatographic measurements for various sources of toxaphene and gravimetric preparation. The concentration, expressed as a mass 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, 0.800 g/mL with a standard deviation (1s) of 0.016 g/mL, which is incorporated in the volume fraction uncertainty.

**Reference Mass Fraction Values:** Reference mass fraction values are provided in Table 1 for 22 toxaphene congeners. Reference values are noncertified values that are estimates of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2].

**Expiration of Certification:** The certification of **SRM 3067** is valid, within the measurement uncertainties specified, until **31 July 2016**, 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) will facilitate notification.

The overall direction and 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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*See Certificate Revision History on Last Page*

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.

Preparation of the SRM was performed by M.P. Cronise of the NIST Measurement Services Division and M.M. Schantz of the NIST Analytical Chemistry Division.

Analytical measurements of the SRM were performed by S.J. Broadwater, M.M. Schantz, and S.S. Vander Pol of the NIST Analytical Chemistry Division.

Partial support for the preparation and certification of this SRM was provided by the U.S. Environmental Protection Agency (EPA) Office of Water, Office of Enforcement and Compliance Assurance, and Office of Research and Development.

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

## **INSTRUCTIONS FOR HANDLING, STORAGE, AND USE**

**Handling:** This material contains toxaphene and should be handled with care. Use proper disposal methods.

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

**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 and reference values to be valid within the stated uncertainty. Because of the volatility of methanol, certified and reference 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<sup>1</sup>**

The toxaphene 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 toxaphene into methanol. The weighed toxaphene 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, argon-flushed, amber glass ampoules, which were then flame sealed.

For the analysis of total toxaphene, 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 hexachlorobenzene and PCB 209 was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of toxaphene (from the same source as used to prepare SRM 3067 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 toxaphene peaks relative to the internal standards. This approach is similar to EPA Method 505 (Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl [PCB] Products in Water by Microextraction and Gas Chromatography, revision 2.0).

For the analysis of the toxaphene congeners (Table 1), individual toxaphene congeners were purchased from commercial sources. Two aliquots from three ampoules of SRM 3067 were gravimetrically diluted with toluene followed by the gravimetric addition of <sup>13</sup>C-labeled *trans*-chlordane for use as an internal standard. The samples were analyzed by using gas chromatography with mass spectrometric detection operated in the negative chemical ionization mode (GC/NCI-MS). The samples were analyzed on two columns: a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.25 μm film thickness; DB-5MS, Agilent Technologies, Wilmington, DE) and a 0.25 mm i.d. × 60 m fused silica capillary column with a proprietary phase (0.25 μm film thickness; DB-XLB, Agilent Technologies) [4]. An example chromatogram from the DB-XLB analysis is shown in Figure 1.

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<sup>1</sup>Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 1. Reference Concentrations Values for Selected Toxaphene Congeners

IUPAC Name	AV Code [5]	Parlar Number [6]	Mass Fraction ( $\mu\text{g}/\text{kg}$ )
2-exo,3-endo,6-exo,8,9,10-hexachlorobornane <sup>(a)</sup>	B6-923		22.2 $\pm$ 0.7 <sup>(b)</sup>
2,2,5,5,8,9,10-heptachlorobornane <sup>(c)</sup>	B7-495		122 $\pm$ 6 <sup>(b)</sup>
2,2,5-endo,6-exo,8,9,10-heptachlorobornane <sup>(c)</sup>	B7-515	32	491 $\pm$ 8 <sup>(b)</sup>
2-endo,3-exo,5-endo,6-exo,8,9,10-heptachlorobornane <sup>(a,c)</sup>	B7-1001		104 $\pm$ 8 <sup>(d)</sup>
2-exo,3-endo,5-exo,8,9,10,10-heptachlorobornane <sup>(a,c)</sup>	B7-1450		107 $\pm$ 3 <sup>(d)</sup>
2,2,3-exo,8,8,9,10-heptachlorocamphene <sup>(a,c)</sup>		25	155 $\pm$ 20 <sup>(d)</sup>
2,2,3-exo,8,8,9,9,10-octachlorocamphene <sup>(a,c)</sup>		31	140 $\pm$ 11 <sup>(d)</sup>
2,2,3-exo,5-endo,6-exo,8,9,10-octachlorobornane <sup>(a,c)</sup>	B8-531	39	136 $\pm$ 11 <sup>(d)</sup>
2,2,5-endo,6-exo,8,8,9,10-octachlorobornane <sup>(a,c)</sup>	B8-806	42a	401 $\pm$ 6 <sup>(d)</sup>
2,2,5-endo,6-exo,8,9,10,10-octachlorobornane <sup>(c)</sup>	B8-810	49a	221 $\pm$ 3 <sup>(b)</sup>
2-endo,3-exo,5-endo,6-exo,8,8,10,10-octachlorobornane <sup>(a,c)</sup>	B8-1413	26	128 $\pm$ 10 <sup>(d)</sup>
2-endo,3-exo,5-endo,6-exo,8,9,10,10-octachlorobornane <sup>(c)</sup>	B8-1414	40	287 $\pm$ 4 <sup>(b)</sup>
2-endo,3-exo,6-exo,8,8,9,10,10-octachlorobornane <sup>(c)</sup>	B8-1471		18.6 $\pm$ 1.0 <sup>(b)</sup>
2-exo,3-endo,5-exo,8,9,9,10,10-octachlorobornane <sup>(c)</sup>	B8-1945	41	116 $\pm$ 1 <sup>(b)</sup>
2-exo,5,5,8,9,9,10,10-octachlorobornane <sup>(a,c)</sup>	B8-2229	44	147 $\pm$ 2 <sup>(d)</sup>
2,2,3-exo,5,5,8,9,10,10-nonachlorobornane <sup>(a,c)</sup>	B9-715	58	261 $\pm$ 20 <sup>(d)</sup>
2,2,3-exo,5,5,9,9,10,10-nonachlorobornane <sup>(c)</sup>	B9-718		86.7 $\pm$ 2.8 <sup>(b)</sup>
2,2,3-exo,5-endo,6-exo,8,9,10,10-nonachlorobornane <sup>(a)</sup>	B9-743		58.7 $\pm$ 2.3 <sup>(b)</sup>
2,2,5,5,8,9,9,10,10-nonachlorobornane <sup>(a,c)</sup>	B9-1025	62	407 $\pm$ 17 <sup>(d)</sup>
2,2,5-endo,6-exo,8,8,9,10,10-nonachlorobornane <sup>(a,c)</sup>	B9-1046	56	98.2 $\pm$ 3.2 <sup>(d)</sup>
2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-nonachlorobornane <sup>(a,c)</sup>	B9-1679	50	166 $\pm$ 7 <sup>(d)</sup>
2-exo,3,3,5-exo,6-endo,8,9,10,10-nonachlorobornane <sup>(a,c)</sup>	B9-2006		55.0 $\pm$ 4.9 <sup>(d)</sup>

<sup>(a)</sup> GC/NCI-MS analysis on a DB-5MS column.

<sup>(b)</sup> The reference values are the means of results obtained using one analytical technique. The expanded uncertainty,  $U$ , is calculated as  $U = ku_c$ , where  $u_c$  is one standard deviation of the analyte mean, and the coverage factor,  $k = 2.57$ , is determined from the Student's  $t$ -distribution corresponding to the associated degrees of freedom and a 95 % confidence level for each analyte.

<sup>(c)</sup> GC/NCI-MS analysis on a DB-XLB column.

<sup>(d)</sup> The reference value is a weighted mean of the results from two analytical methods [7]. The uncertainty listed with each value is an expanded uncertainty about the mean [8,9], with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method variance following the ISO Guide [3].

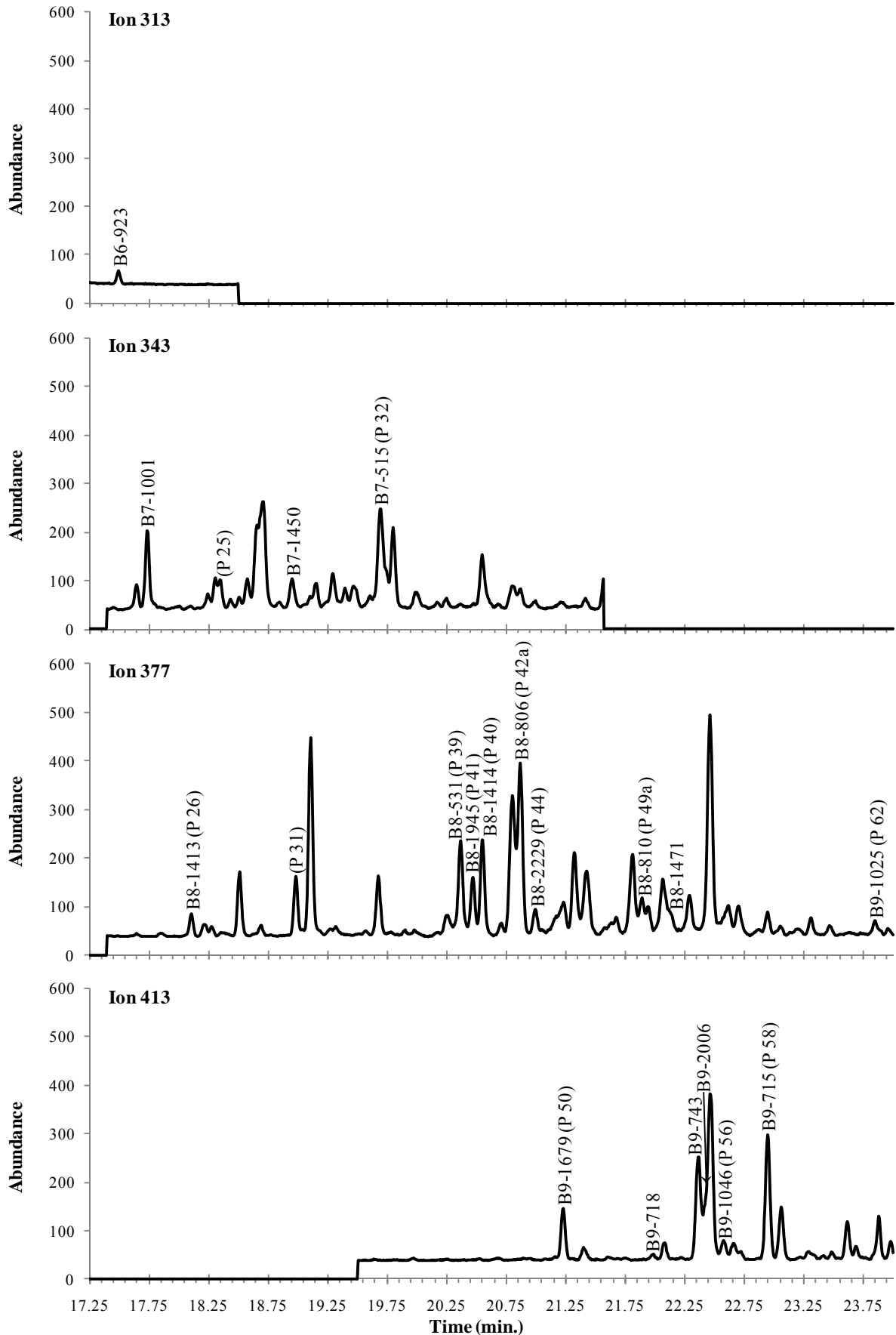


Figure 1. GC/NCI-MS Chromatogram from the DB-XLB analysis showing the congener identification in SRM 3067.

## REFERENCES

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<b>Certificate Revision History:</b> 31 August 2010 (Addition of reference concentration values for selected toxaphene congeners; extension of certification period; editorial changes.); 23 May 2003 (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](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*