



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3076

Aroclor 1232 in Transformer Oil

This Standard Reference Material (SRM) is a solution of Aroclor 1232 (Chemical Abstracts Registry Number 11141-16-5) in transformer oil. This SRM is intended primarily for calibrating chromatographic instrumentation and methods of analysis used for the determination of Aroclor 1232 and polychlorinated biphenyls (PCBs) in transformer oil. A unit of SRM 3076 consists of five 2-mL ampoules, each containing approximately 1.2 mL of transformer oil.

Certified Concentration of Aroclor 1232: The certified concentration [1,2] given below is based on results obtained from the gravimetric preparation of this solution and from the analytical results determined 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.

Aroclor 1232 4252 mg/kg \pm 114 mg/kg or 3789 mg/L \pm 106 mg/L

The result is 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, 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 Aroclors and gravimetric preparation. The concentration expressed as a volume fraction (in mg/L) was obtained by multiplying the certified value, expressed as a mass fraction, by the measured density (22 °C) of the SRM solution, 0.8912 g/mL \pm 0.0205 g/mL, where 0.0205 represents one standard deviation (1s) and is incorporated in the volume fraction uncertainty.

Expiration of Certification: The certification of this SRM lot is valid until **31 July 2012**, 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 otherwise modified.

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.

The coordination of the technical measurements leading to the certification was under the direction of D.L. Poster and M.M. Schantz of the NIST Analytical Chemistry Division.

Analytical measurements of the SRM were performed by D.L. Poster of the NIST Analytical Chemistry Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 23 May 2003

John Rumble, Jr., Chief
Measurement Services Division

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

Partial support for the preparation and certification of this Standard Reference Material was provided by the U.S. Environmental Protection Agency Office of Water, Office of Enforcement and Compliance Assurance, and Office of Research and Development.

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.

NOTICE AND WARNING TO USERS

Handling and Storage: This material is a solution of a PCB mixture. PCB-containing materials are reported to be toxic. Extreme caution and care should be exercised during the handling of SRM 3076. Contact your regional EPA office for information regarding proper disposal. Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

INSTRUCTIONS FOR 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 value to be valid within the stated uncertainty. 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 ANALYSIS¹

The Aroclor 1232 used in the preparation of this SRM was obtained from U.S. EPA. The SRM was prepared at NIST by weighing and mixing Aroclor 1232 into transformer oil (Univolt 60, Exxon). The Aroclor was added to the transformer oil and mixed until completely dissolved and homogenized. The total mass of this solution was measured and aliquots (1.2 mL) were dispensed into 2-mL amber glass ampoules, which were then flame sealed.

Aliquots from nine ampoules, selected randomly, were analyzed using capillary gas chromatography with electron capture detection and an immobilized non-polar (5 % phenyl methylpolysiloxane) stationary phase column. An internal standard solution containing hexachlorobenzene and mirex was added to each sample for quantification purposes. Prior to gas chromatography, samples were placed on aminopropyl solid phase extraction columns and eluted with hexane. The concentrated eluants were then placed on a semi-preparative aminopropylsilane column using hexane as the mobile phase. Calibration solutions consisting of weighed amounts of Aroclor 1232 and internal standard compounds in transformer oil were chromatographically analyzed to determine response factors for Aroclor 1232 relative to each internal standard. The results for Aroclor 1232 are based on the areas of the dominant Aroclor PCB peaks relative to the internal standard peaks (Figure 1). This approach is similar to U.S. EPA Method 505 (Analysis of organohalide pesticides and commercial polychlorinated biphenyl (PCB) products in water by microextraction and gas chromatography, revision 2.0).

¹Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately 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.

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.; *Definition of Terms and Modes Used at NIST for Value-Assessment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office, Washington, DC (2000).
- [2] 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 (1995).
- [3] *Guide to the Expression of Uncertainty in Measurement*; ISBN 72-67-10188-9, 1st Ed., ISO, Geneva, Switzerland (1993); 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://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>.

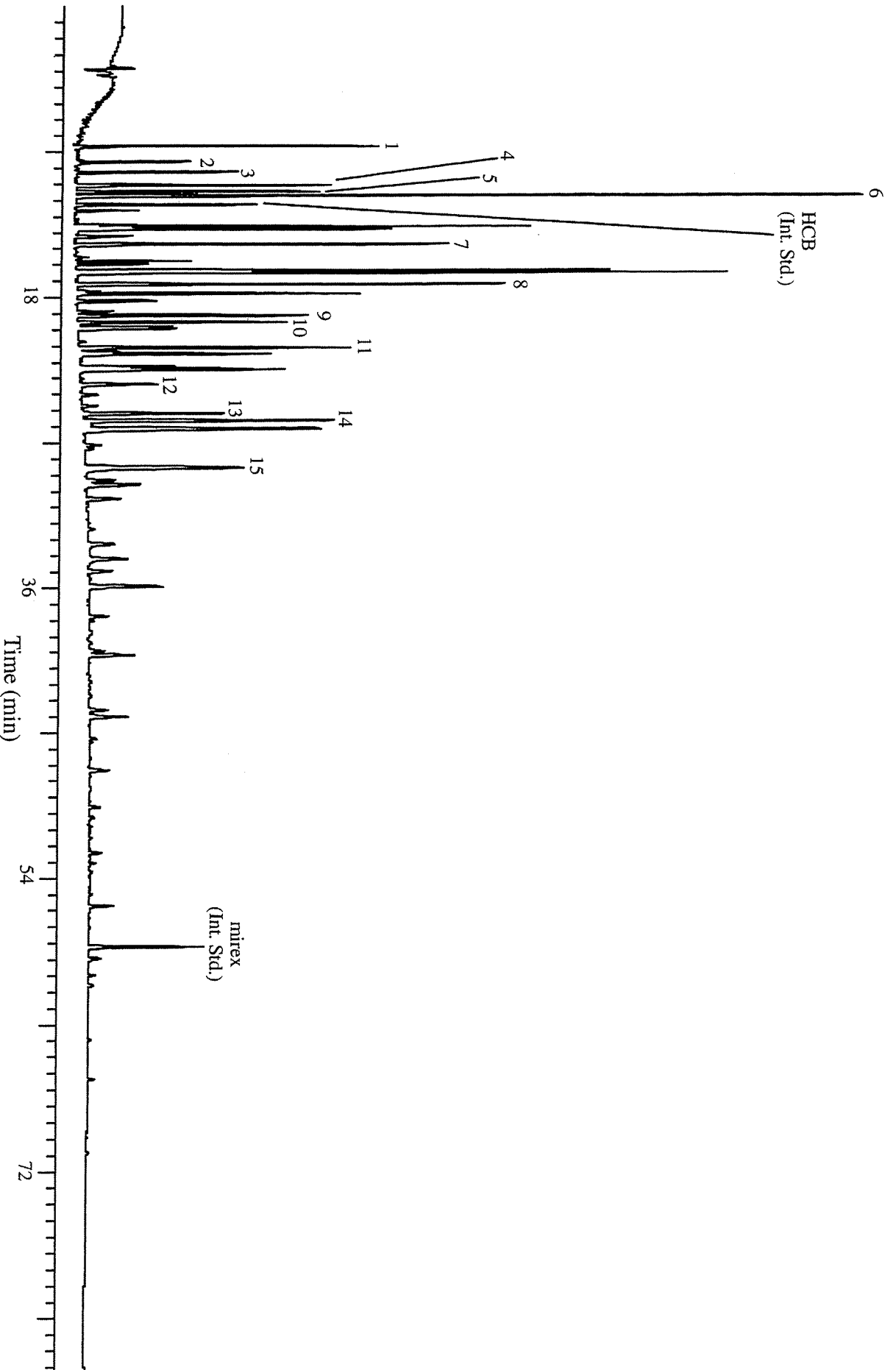


Figure 1. Gas chromatogram of SRM 3076 (Aroclor 1232 in Transformer Oil) with a 5% phenyl methyl polysiloxane column. The peaks used for quantification of the Aroclor mass in transformer oil are shown. (Int. Std.) = internal standard