



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3063

2,3,7,8-Tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) in Methanol

This Standard Reference Material (SRM) is a solution of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) (Chemical Abstracts Registry Number 1746-01-6) in methanol. SRM 3063 is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of 2,3,7,8-TCDD. Because of its miscibility with water, this SRM can also be used to fortify aqueous samples with known amounts of 2,3,7,8-TCDD. A unit of SRM 3063 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentration of 2,3,7,8-TCDD: 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 accounted for by NIST.

2,3,7,8-Tetrachlorodibenzo-*p*-dioxin: 0.410 ± 0.014 mg/kg or 0.324 ± 0.011 mg/L

The results are expressed as the certified value with 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 [3], 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. The certified concentration in mg/L was obtained by multiplying the certified mass value by the measured density of the SRM solution at 22 °C (0.7914 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.

Expiration of Certification: The certification of this SRM lot 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 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.

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.

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.

Support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

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Gaithersburg, MD 20899
Certificate Issue Date: 13 February 2002

John Rumble, Jr., Acting Chief
Standard Reference Materials Program

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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 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 2,3,7,8-TCDD used in the preparation of this SRM was obtained from a commercial source. The solution was prepared at NIST by weighing and mixing the 2,3,7,8-TCDD into the methanol. The weighed 2,3,7,8-TCDD was added to the methanol 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 the 2,3,7,8-TCDD, which was determined using capillary gas chromatography with flame ionization detection. 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 capillary gas chromatography with electron capture detection employing an immobilized non-polar (5 % phenylmethylpolysiloxane) stationary phase column. An internal standard solution containing 1,2,3,4-TCDD was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of 2,3,7,8-TCDD and internal standard compounds in methanol were chromatographically analyzed to determine the response factor for 2,3,7,8-TCDD relative to 1,2,3,4-TCDD.

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-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.; 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>.