



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1614

#### Dioxin (2,3,7,8-TCDD in Isooctane)

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of analytical methods in the determination of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD). It can also be used to fortify samples with known amounts of 2,3,7,8-TCDD. The <sup>13</sup>C-labeled 2,3,7,8-TCDD can be used as an internal standard in methods based on gas chromatography/mass spectrometry (GC/MS). This SRM consists of separate solutions of unlabeled and labeled 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) in 2,4,4-trimethylpentane (isooctane). Three ampoules contain approximately 1.2 mL each of an isooctane solution of unlabeled 2,3,7,8-TCDD, and three ampoules contain approximately 1.2 mL each of an isooctane solution of <sup>13</sup>C-labeled 2,3,7,8-TCDD.

**Certified Values and Uncertainties:** The certified concentrations and estimated uncertainties of the unlabeled and <sup>13</sup>C-labeled 2,3,7,8-TCDD solutions are given in Table 1. NIST certified values are values for which NIST has the highest confidence in their accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. The certified values are reported in mass fraction (ng/g) units, but are also reported in mass concentration (ng/mL) units for user convenience [2]. The <sup>13</sup>C-labeled solution is certified for the total concentration of all isotopic forms of 2,3,7,8-TCDD. The isotopic purity of the <sup>13</sup>C-labeled material was determined to be an atom fraction of 98.2 % ± 0.1 % <sup>13</sup>C by mass spectrometry. The fully <sup>13</sup>C-labeled compound, 2,3,7,8-TCDD-<sup>13</sup>C<sub>12</sub>, accounts for 80.7 % ± 0.5 % of the 2,3,7,8-TCDD molecules in the sample.

The certified values are the weighted means of gravimetric values, based on the concentration calculated from the mass of 2,3,7,8-TCDD added to a known mass of isooctane and on the analytical results obtained using capillary gas chromatography with electron capture detection (GC/ECD). The uncertainties are two standard deviations of the certified values. The uncertainties include the gravimetric and GC measurement variability and any observed material heterogeneity.

**Expiration of Certification:** This certification of **SRM 1614** is valid, within the measurement uncertainty specified, until **31 January 2019**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). This 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 NIST technical measurements leading to the certification of SRM 1614 was performed by L.R. Hilpert, R.M. Parris, and W.E. May of the NIST Analytical Chemistry Division.

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*See Certificate Revision History on Last Page*

The preparation and analytical determinations were performed in the NIST Analytical Chemistry Division by S.N. Chesler, B. Coxon, L.R. Hilpert, R.M. Parris, R.E. Rebbert, M.J. Welch, and E. White V.

Statistical analysis of the experimental data was performed by R.C. Paule of the NIST Statistical Engineering Division.

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

## NOTICE AND WARNING TO USERS

**Handling:** The toxicity and/or carcinogenicity of 2,3,7,8-TCDD has not been defined; however, this material should be treated as a potential health hazard. Ampoules should be opened and the contents used by persons trained in proper handling techniques. Users in the United States should contact their regional offices of the U.S. Environmental Protection Agency for information regarding proper disposal of these materials; in other countries, users should contact the appropriate organization responsible for public health or environmental control.

Trimethylpentane (isooctane), used as a diluent in this SRM, is stable when stored in closed containers and at room temperature. It will not undergo hazardous polymerization. However, it is highly flammable and should be kept away from oxidizing agents.

**Storage:** Sealed ampoules, as received, should be stored in the dark at temperatures between 10 °C and 30 °C. It is recommended that these materials be stored in a secure area in a double-sealed container.

## INSTRUCTION FOR USE

Samples of the SRM should be withdrawn from ampoules (at 23 °C ± 8 °C) immediately after opening and used without delay for the certified values listed in Table 1 to be valid within the stated uncertainties. Certified values are not applicable to material in ampoules stored after opening, even if they are resealed.

## PREPARATION AND ANALYSIS<sup>1</sup>

Samples of the unlabeled 2,3,7,8-TCDD and <sup>13</sup>C-2,3,7,8-TCDD used in the preparation of SRM 1614 were donated by R. Mitchum, Food and Drug Administration, National Center for Toxicological Research, Jefferson, AK. The unlabeled 2,3,7,8-TCDD was originally obtained from ECO Control Inc., Cambridge, MA; and the <sup>13</sup>C-labeled 2,3,7,8-TCDD from Midwest Research Institute, Kansas City, MO. The purities of the labeled and unlabeled 2,3,7,8-TCDD used in the preparation of this SRM were determined at NIST using mass spectrometry, nuclear magnetic resonance spectrometry, and GC with flame ionization detection. The purities were found to be greater than 95 %.

Solutions of the unlabeled 2,3,7,8-TCDD and the <sup>13</sup>C-labeled 2,3,7,8-TCDD were prepared at NIST by weighing and mixing the appropriate compound and isooctane. Each solution was dispensed into 2-mL amber ampoules, which were then flame sealed. Aliquots from randomly selected ampoules were analyzed with a gas chromatograph equipped for split injection and a 30 m × 0.25 mm i.d. wall-coated open-tubular column with a 0.25 μm film of non-polar, immobilized phase. A constant-current electron capture detector (<sup>63</sup>Ni) was used for those analyses. Quantitative results were obtained through the use of 2,2',4,4',5,5',-hexachlorobiphenyl as an internal standard. Calibration solutions consisting of weighted amounts of the analyte and the internal standard compound in isooctane were analyzed chromatographically to determine response factors.

A trichlorodibenzo-*p*-dioxin impurity present in both solutions was quantified using GC/MS with electron impact ionization, selected-ion monitoring, and the method of standard additions. Standard additions of unlabeled 2,3,7-trichlorodibenzo-*p*-dioxin were made to the unlabeled and <sup>13</sup>C-2,3,7,8-TCDD solutions, and although the

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<sup>1</sup>Certain 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.

retention time of the trichlorodibenzo-*p*-dioxin impurity was coincident with that of the 2,3,7-isomer, this was not sufficient to positively identify which isomer was present. Concentrations of the trichlorodibenzo-*p*-dioxin in the SRM solutions are provided, for information only, in Table 2.

Table 1. Certified Values for 2,3,7,8,-TCDD<sup>(a)</sup> in SRM 1614

	Mass Fraction (ng/g) <sup>(b)</sup>	Mass Concentration at 23 °C (ng/mL) <sup>(c)</sup>
2,3,7,8,-TCDD	98.3 ± 3.3	67.8 ± 2.3
2,3,7,8,-TCDD <sup>13</sup> C <sup>(d)</sup>	95.6 ± 1.5	65.9 ± 1.0

- (a) CAS Registry Numbers [3]: 2,3,7,8-TCDD-<sup>12</sup>C<sub>12</sub> [1746-01-6]  
2,3,7,8-TCDD-<sup>13</sup>C<sub>12</sub> [76523-40-5]
- (b) The expanded uncertainty for each certified value is the combined standard uncertainty multiplied by  $k = 2$ . Each combined standard uncertainty includes the gravimetric and GC/ECD 2,3,7,8-TCDD measurement variability, the trichlorodibenzo-*p*-dioxin measurement variability, and for the unlabeled 2,3,7,8-TCDD, the observed sample heterogeneity [4].
- (c) The concentration and uncertainty of expressed in mass concentration units are applicable for use of this material at 23 °C. Since the density of 2,2,4-trimethylpentane changes with temperature, the concentration will change as temperatures other than 23.0 °C. The concentration will change by less than 1 percent of the value listed if the SRM is used as temperatures in the 15 °C to 31 °C temperature range.
- (d) The concentrations given represent the total concentrations for all isotopic forms of 2,3,7,8-TCDD in the solution. The fully <sup>13</sup>C-labeled 2,3,7,8-TCDD accounts for 80.7 % ± 0.5 % of the 2,3,7,8-TCDD molecules in the sample. This value is provided for information only.

Table 2. Information Values for Trichlorodibenzo-*p*-Dioxin in SRM 1614

Solution	Compound	Concentration	
		ng/g	ng/mL (23 °C)
Unlabeled	trichlorodibenzo- <i>p</i> -dioxin- <sup>12</sup> C <sub>12</sub>	(1.5)	(1.0)
Labeled	trichlorodibenzo- <i>p</i> -dioxin- <sup>13</sup> C <sub>12</sub>	(3.9)	(2.7)

## 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.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, U.S. Government Printing Office, Gaithersburg, MD (2000); available at [http://www.cstl.nist.gov/nist839/NIST\\_special\\_publications.htm](http://www.cstl.nist.gov/nist839/NIST_special_publications.htm).
- [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.physics.nist.gov/Pubs>.
- [3] *Chemical Abstracts, Tenth Collective Index*; Index Guide; American Chemical Society: Columbus, OH (1982).
- [4] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: 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/>.

<b>Certificate Revision History:</b> 22 September 2009 (This certificate has been revised to indicate a specific expiration date and editorial changes); 08 July 1985 (Original certificate date).
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*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) 975-2200; fax (301) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet <http://www.nist.gov/srm>.*