



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3064

Endothall in Water

This Standard Reference Material (SRM) is a solution of endothall (Chemical Abstracts Service Registry Number 145-73-3) in water. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of endothall. SRM 3064 can also be used to fortify aqueous samples with known amounts of endothall. A unit of SRM 3064 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

Certified Value: The certified value given below 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 Endothall: 40.0 mg/kg \pm 1.1 mg/kg

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 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 [1]. The value of u_c includes both a correction for estimated purity and an allowance for differences between the concentration determined by gravimetric preparation and chromatographic measurements.

Expiration of Certification: The certification of **SRM 3064** 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"). However, the certification is nullified if the SRM is damaged, contaminated, or 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 C.R. Mack 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 and C.R. Mack.

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

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

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Gaithersburg, MD 20899
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Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

WARNING AND INSTRUCTIONS FOR HANDLING, STORAGE, AND 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.

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.

PREPARATION AND ANALYSIS⁽¹⁾

The endothall monohydrate used in the preparation of this SRM was obtained from a commercial source. The solution was prepared at NIST by weighing and mixing the endothall monohydrate into the water. The weighed endothall monohydrate was added to the water and mixed until completely dissolved and homogenized. The total mass of this solution was measured. The gravimetric concentration was adjusted for the consensus purity estimation of the endothall, which was determined using capillary gas chromatography with flame ionization detection (GC-FID). This bulk solution was then chilled and 1.2-mL aliquots were dispensed into 2-mL amber glass ampoules, which were then flame sealed.

Aliquots from nine ampoules, selected randomly, were analyzed in duplicate by using capillary GC-FID employing an immobilized non-polar (100 % dimethylpolysiloxane) stationary phase column. An internal standard solution containing dicamba (3,6-dichloro-2-methoxybenzoic acid) was added to each sample for quantification purposes followed by the addition of acidified acetone [2]. Calibration solutions consisting of weighed amounts of endothall and internal standard in water plus acidified acetone were chromatographically analyzed to determine the response factor for endothall relative to dicamba.

REFERENCES

- [1] 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).
- [2] Carlson, R.; Whitaker, R.; Landskov, A.; Chapter 31; *Endothall in Analytical Methods for Pesticides and Plant Growth Regulators: New and Updated Methods*; G. Zweig, Ed; Academic Press: New York, NY, pp. 327–340 (1978).

Certificate Revision History: 06 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.