



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3065

Chlorinated Herbicides-I in Methanol

Standard Reference Material (SRM) 3065 is a solution of eight chlorinated herbicides, (free acid form), in methanol intended primarily for use in the calibration of chromatographic instrumentation used for the determination of the certified components in the mixture. Because of its miscibility with water, SRM 3065 can also be used to fortify samples with known amounts of the eight chlorinated herbicides. A unit of SRM 3065 consists of five 2-mL ampoules, each containing 1.2 mL of solution.

Certified Concentration of Constituents: The certified concentration values [1,2] for the eight chlorinated herbicides are given in Table 1. These values are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography with mass spectrometric detection (GC/MS). 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.

Supplemental Information: Chemical Abstracts Service (CAS) Registry Numbers of the certified components are listed in Table 2.

Expiration of Certification: The certification of this SRM lot is valid until **31 March 2013**, 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 modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

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.

Analytical measurements of the SRM were performed by M.M. Schantz and C.R. Mack of the NIST Analytical Chemistry Division.

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

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Preparation of the SRM were performed by M.P. Cronise of the NIST Measurement Services Division and by M.M. Schantz and C.R. Mack of the NIST Analytical Chemistry Division.

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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 contains chlorinated herbicides and 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 ANALYSIS¹

SRM Preparation: The chlorinated herbicides used in the preparation of this SRM were obtained from commercial sources. The solution was prepared at NIST by weighing and mixing the individual components into the methanol. The weighed components were 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 are given in Table 2 for the components. These gravimetric concentrations were adjusted for the consensus purity estimation of each component, which were determined by using capillary gas chromatography with flame ionization detection and differential scanning calorimetry. 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 randomly, were analyzed in duplicate by using GC/MS employing an immobilized non-polar (5 % phenylmethylpolysiloxane) stationary phase column. An internal standard solution containing 2,2',4,5',6-pentachlorobiphenyl and 2,2',3,3',4,5,5',6-octachlorobiphenyl was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of the eight chlorinated herbicides and the two internal standard compounds in methanol were chromatographically analyzed to determine analyte response factors.

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.

Table 1. Certified Concentrations of the Eight Chlorinated Herbicides in SRM 3065

Compound	Concentration	
	mg/kg ^a	mg/L ^b
Acifluorfen	78.8 ± 1.9	62.4 ± 1.5
Bentazon	82.6 ± 2.0	65.4 ± 1.6
2,4-D	134.2 ± 3.2	106.2 ± 2.5
2,4-D Butyl Ester	73.1 ± 1.8	57.9 ± 1.4
Dalapon	110.4 ± 2.8	87.4 ± 2.2
Dicamba	96.0 ± 2.0	76.0 ± 1.6
Picloram	126.2 ± 3.0	99.9 ± 2.4
2,4,5-TP (Silvex)	217.8 ± 5.0	172.4 ± 4.0

^a The results are expressed as the certified value ± the 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 concentration determined by gravimetric preparation and chromatographic measurements.

^b The 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 (0.7914 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.

Table 2. Compound Names and CAS Registry Numbers for the Components in SRM 3065^a

Common Name	Chemical Name	CAS Registry Number
Acifluorfen	5-(2-chloro-4-(trifluoromethyl)phenoxy)-nitrobenzoic acid	50594-66-6
Bentazon	3-(1-methylethyl)-2,2-dioxido-1H-2,1,3-benzothiadiazin-4(3H)-one	25057-89-0
2,4-D	2,4-dichlorophenoxy-acetic acid	94-75-7
2,4-D butyl ester	2,4-dichlorophenoxy-acetic acid butyl ester	94-80-4
Dalapon	2,2-dichloropropanic acid	75-99-0
Dicamba	3,6-dichloro-2-methoxy-benzoic acid	1918-00-9
Picloram	4-amino-3,5,6-trichloro-2-pyridinecarboxylic acid	1918-02-1
2,4,5-TP (Silvex)	2-(2,4,5-trichlorophenoxy)-propionic acid	93-72-1

^a Chemical Abstracts, Thirteenth Collective Index, Index Guide, American Chemical Society, Columbus, Ohio, 1996.

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 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>.