



National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 3071

#### Glyphosate in Water

Standard Reference Material (SRM) 3071 is a solution of glyphosate (Chemical Abstracts Registry Number 1071-83-6) in water intended primarily for use in the calibration of chromatographic instrumentation used for the determination of glyphosate. This SRM can also be used to fortify aqueous samples with known amounts of glyphosate. A unit of SRM 3071 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

**Certified Concentration of Glyphosate:** The certified concentration value [1,2] given below is based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using liquid chromatography (LC). 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.

Glyphosate       $7.76 \pm 0.33$  mg/kg

The results are expressed as the certified value  $\pm$  an expanded uncertainty. The certified value is the unweighted average of the concentration determined by gravimetric and chromatographic methods. The expanded uncertainty, at a 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 concentration in mg/L is the same as the certified concentration based on the density of the solution at 22 °C (1.0 g/mL).

**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 (see Instructions for Use). 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.

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.

Coordination of the technical measurements leading to the certification was under the direction of L.C. Sander and M.M. Schantz of the NIST Analytical Chemistry Division.

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.

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

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Preparation and analytical measurements of the SRM were performed by M.P. Cronise of the NIST Standard Reference Materials Group and J. Brown Thomas and L.C. Sander of the NIST Analytical Chemistry 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. 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<sup>1</sup>

**SRM Preparation:** The glyphosate used in the preparation of this SRM was obtained from a commercial source. The solution was prepared at NIST by weighing and mixing the glyphosate into the water. The weighed glyphosate was added to the water 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 glyphosate, which was determined using LC with ultraviolet (UV) detection at 196 nm. Aliquots (1.2 mL) of the bulk solution 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 LC with UV detection at 196 nm employing an anion exchange column (4.6 x 250 mm, 5 :m particle diameter; Wescan Instruments, Inc., Santa Clara, CA, USA). Isocratic elution (96 % aqueous buffer solution of 0.0062 M potassium dihydrogen phosphate and 4 % methanol; pH adjusted to 2 with 85 % phosphoric acid) at a flow rate of 2.0 mL/min was used for the determination of glyphosate in the solution [4]. The concentration of glyphosate in the SRM was determined by using the external standard method, based on response factors determined from analyses of four calibration solutions of the compound.

## REFERENCES

- [1] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R; Guenther; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *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. and 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/>.
- [4] Morlier, L.W.; Tomkins, D.F.; *Liquid Chromatographic Determination of Glyphosate in Water-Soluble Granular Formulations: Collaborative Study*; J. AOAC Intl., Vol. 80, pp. 464-468 (1997).

*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](mailto:srminfo@nist.gov); or via the Internet <http://www.nist.gov/srm>.*

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<sup>1</sup>Certain commercial equipment, instruments, or materials are identified in this certificate in order 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.