National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3001

Toluene in Methanol

(Nominal Mass Fraction – 0.01 g/g)

This Standard Reference Material (SRM) is a gravimetrically prepared single-compound solution (toluene) in methanol intended primarily for the calibration of instrumentation and validation of methods for volatile organic compound (VOC) determinations. Because of its miscibility with water, this SRM can also be used to fortify aqueous samples with known amounts of the VOC. A unit of SRM 3001 consists of two 5 mL sealed borosilicate glass ampoules of a gravimetrically prepared solution of toluene in methanol. Approximately 2.5 mL of this SRM mixture is supplied in each 5 mL glass ampoule.

Certified Value: 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 [1]. The certified concentration value for toluene, reported as a mass fraction, is given below.

Toluene (mass fraction): 0.0101 g/g ± 0.0004 g/g

The certified value is an unweighted mean of the results from three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance [2] with a pooled, within-method variance following the ISO and NIST Guides [3].

Reference Value: A NIST reference value is a non-certified value that is the best estimate of the true value; however, the value does not meet NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision and may not include all sources of uncertainty [1]. A reference value for density is provided for the calculation of volume to assist in the transfer of material during gravimetric dilutions of the SRM.

Density of the SRM Solution at 22 °C: 0.79042 g/mL ± 0.00001 g/mL

The reference value is the mean of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Students t-distribution corresponding to the associated degrees of freedom and 95 % confidence level [3].

Expiration of Certification: The certification of **SRM 3001** is valid, within the measurement uncertainty specified, until **31 January 2015**, provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). 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 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.

The overall coordination and direction of the technical work required for this SRM certification were performed by F.R. Guenther and M.M. Schantz of the NIST Analytical Chemistry Division.

Stephen A Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 30 November 2009
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

The analytical measurements leading to the certification of this SRM were performed by B.A. Benner, Jr., T.L. Green, F.R. Guenther, C.R. Mack, M.M. Schantz, B.J. Porter, and S-k. Wong of the NIST Analytical Chemistry Division.

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

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

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

SRM Preparation: This SRM was prepared by NIST using precise gravimetric mass determinations. Chemicals used in the preparation were received from commercial sources and were assessed for purity at NIST using differential scanning calorimetry (DSC), gas chromatography with flame ionization detection (GC/FID), and gas chromatography with mass spectrometric detection (GC/MS).

Toluene Concentration Value Assignment: The certified value for toluene is based on the gravimetric preparation of the SRM and analysis by GC/FID and GC/MS of randomly selected ampoules from the lot. The analytical method was calibrated using four calibration standards independently prepared by gravimetry.

INSTRUCTIONS FOR USE

Storage: Sealed ampoules should be stored in the dark at temperatures between 10 °C and 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. It is recommended that aliquots be withdrawn at temperatures between 20 °C and 25 °C. Each ampouled solution must be opened in a clean, dry environment and processed without delay. Each ampouled solution of the SRM is intended for use immediately after opening and may not be reused, even if resealed.

Preparation of Working Standard Solutions by Mass: Great care must be used in handling this SRM. Dilution of this SRM should be made gravimetrically (weighed) and not by volumetric means. (Volume may be calculated for transfer purposes only.). It is recommended that the SRM material be transferred in a gas-tight syringe to a septum-sealed container containing the diluent. It is critical that the SRM be injected slowly beneath the surface of the diluent. The syringe should be weighed before injecting the material and weighed again after solution transfer. This allows subtraction of the mass of the syringe and any SRM material that remains in the syringe. The amount of toluene added to the diluent can then be determined from the mass added and the certified value.

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: Washington, DC (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/upload/SP260-136.PDF.

[2] Levenson, M.S., Banks, D.L., Eberhardt, K.R., Gill, L.M., Guthrie, W.F., Liu, H.k., Vangel, M.G., Yen, J.H., and Zhang, N.F., "An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM," J. Res. Natl. Inst. Stand. Technol. 105, pp. 571–579, (2000).

[3] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf; 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/.

Certificate Revision History: 30 November 2009 (Extended certificate expiration date, updated certified value, editorial changes); 13 July 2005 (This technical revision reports an extension in the certificated period); 15 June 2001 (Original certificate date).

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; or via the Internet at http://www.nist.gov/srm.