



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3008

Methylene Chloride in Methanol

(Nominal Mass Fraction, 0.01 g/g)

Standard Reference Material (SRM) 3008 is 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. One unit of SRM 3008 consists of two 5 mL sealed borosilicate glass ampoules of a gravimetrically prepared single compound solution in methanol. Approximately 2.5 mL of this SRM mixture is supplied in each 5 mL glass ampoule.

Certified Value: The certified concentration value [1,2] for methylene chloride, reported as a mass fraction, is given below.

Methylene Chloride (mass fraction): 0.009973 g/g \pm 0.000062 g/g

The uncertainty in the certified value is expressed as an expanded uncertainty, $U = ku_c$, calculated according to the methods in the ISO Guide [3]. The coverage factor, $k = 2$, is used to obtain an expanded uncertainty with an approximate confidence level of 95 %. The quantity u_c represents, at the level of one standard deviation, the combined effects of the uncertainty due to purity assessment and an allowance for differences between the concentration determined by gravimetric preparation and chromatographic measurements.

Reference Value: A reference value [1] 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: 0.79256 g/mL \pm 0.00003 g/mL at 22 °C

Expiration of Certification: The certification of this SRM is valid, within the measurement uncertainties specified, until **01 July 2004** provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Instructions for Use). Certification is nullified if the SRM is contaminated or otherwise modified.

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.

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

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

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD
Certificate Issue Date: 10 January 2002

John Rumble, Jr., Acting Chief
Standard Reference Materials Program

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.

The analytical measurements leading to the certification of this SRM were performed by T.L. Green, F.R. Guenther, and C.R. Mack of the NIST Analytical Chemistry Division.

Statistical consultation was provided by S.D. Leigh of the NIST Statistical Engineering 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 selective detection (GC/MSD).

Methylene Chloride Concentration Value Assignment: The certified value for methylene chloride was determined from the gravimetric preparation of the SRM, and analysis of randomly selected ampoules from the lot using GC/FID. 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 ampoule 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 Standard Working Solutions by Mass: Great care must be exercised in handling this SRM. Dilution of this SRM must 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 methylene chloride added to the diluent can be determined from the mass added and the certified value.

REFERENCES

- [1] May, W.E. et al., "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/>.

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>.