



Certificate of Analysis

Standard Reference Material[®] 3008

Methylene Chloride in Methanol

(Nominal Mass Fraction – 0.01 g/g)

This Standard Reference Material (SRM) is a gravimetrically prepared single-compound solution (methylene chloride) 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 3008 consists of two 5 mL sealed borosilicate glass ampoules of a gravimetrically prepared solution of methylene chloride 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] for Methylene Chloride, reported as a mass fraction, is given below.

Methylene Chloride (mass fraction): 0.0101 g/g \pm 0.0002 g/g

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 value is an unweighted mean of the results from three analytical methods. The uncertainty listed with each 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: 0.79256 g/mL \pm 0.00003 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 Student's t -distribution corresponding to the associated degrees of freedom and 95 % confidence level [3].

Expiration of Certification: The certification of **SRM 3008** 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"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that effect 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.

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Analytical Chemistry Division

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Statistical consultation was provided by S.D. Leigh of the NIST Statistical Engineering 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, B.J. Porter, M.M. Schantz, and S-k.Wong of the NIST Analytical Chemistry Division.

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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).

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 and GC/MS. 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.; 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] 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>.

Certificate Revision History: 17 December 2009 (Extended certificate expiration date, updated certified value, editorial changes); 12 January 2006 (This technical revision reports an extension in the certificated period); 10 January 2002 (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>.