



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3061

Chloral Hydrate in Methanol

Standard Reference Material (SRM) 3061 is a solution of chloral hydrate (Chemical Abstracts Registry Number 302-17-0) in methanol intended primarily for use in the calibration of chromatographic instrumentation used for the determination of chloral hydrate. Because of its miscibility with water, this SRM can also be used to fortify aqueous samples with known amounts of chloral hydrate. A unit of SRM 3061 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentration of Chlorate Hydrate: 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 gas chromatography. 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.

Chloral Hydrate 31.0 ± 1.0 mg/kg or 24.5 ± 0.8 mg/L

The results are expressed as the certified value \pm the 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 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. 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 (0.791 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.

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.

The coordination of the technical measurements leading to the certification was under the direction of S.A. Wise and M.M. Schantz 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.

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

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 23 May 2003

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

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.

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 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 chloral hydrate used in the preparation of this SRM was obtained from a commercial source. The solution was prepared at NIST by weighing and mixing the chloral hydrate into the methanol. The weighed chloral hydrate was 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 is summarized below. These gravimetric concentrations were adjusted for the consensus purity estimation of the chloral hydrate, which was determined 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 capillary gas chromatography with electron capture detection employing an immobilized non-polar (5 % phenylmethylpolysiloxane) stationary phase column. An internal standard solution containing trichloroethylene was added to each sample for quantification purposes. Following addition of the internal standard solution, the solution was diluted with methyl *tert*-butyl ether for analysis. Calibration solutions consisting of weighed amounts of chloral hydrate and internal standard compound in methanol were chromatographically analyzed also following dilution with methyl *tert*-butyl ether to determine the response factor for chloral hydrate relative to trichloroethylene.

¹Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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>.