



National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material[®] 2898a

Ethanol-Water Solution (Nominal Mass Fraction 6 %)

This Standard Reference Material (SRM) is a solution of ethanol (ethyl alcohol: Chemical Abstracts Service [CAS] Registry Number 64-17-5) in water at a nominal mass fraction of 6 %. SRM 2898a is intended primarily for use in the calibration of instruments and techniques used for the determination of ethanol. A unit of SRM 2898a consists of five 10-milliliter ampoules, each containing approximately 10 mL of solution.

Certified Mass Fraction of Ethanol: The certified mass fraction value given below is based on results obtained from the gravimetric preparation of the solution and from the analytical results determined using gas chromatography with flame ionization detection (GC-FID). 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].

Ethanol Certified Mass Fraction Value: 6.01 % \pm 0.13 %

The results are expressed as the certified value \pm the expanded uncertainty. The certified value is an unweighted mean of mass fractions determined by gravimetric preparation and chromatographic measurements. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence. The expanded uncertainty is calculated as $U = ku_c$, where u_c incorporates the observed difference between the results from the methods and their respective uncertainties, as well as an uncertainty component related to purity determination, consistent with the ISO Guide and with its Supplement 1, and $k = 2$ is a coverage factor corresponding to approximately 95 % confidence [2–4].

Expiration of Certification: The certification of **SRM 2898a** is valid, within the measurement uncertainty specified, until **30 April 2025**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Handling, Storage, and Use”). However, 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.

Coordination of the technical measurements leading to the certification of this SRM was under the direction of L.C. Sander of the NIST Chemical Sciences Division.

Preparation of and analytical measurements on the SRM were performed by M.M. Schantz of the NIST Chemical Sciences Division and M.P. Cronise of the NIST Office of Reference Materials.

Statistical analysis was provided by J.H. Yen of the NIST Statistical Engineering Division.

Support aspects involved with the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Certificate Issue Date: 30 May 2013

Robert L. Watters, Jr., Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: The solution contains ethanol in water at the stated concentration. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10 °C and 30 °C.

Use: Sample aliquots for analysis should be withdrawn **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 ethanol, the certified value is **NOT** applicable to material stored in ampoules that have been opened for more than 2 min, even if they are resealed.

PREPARATION AND ANALYSIS⁽¹⁾

The solution was prepared at NIST by weighing and mixing known masses of ethanol and organic-free water. The solution was capped and mixed overnight (a minimum of 16 h). The total mass of the solution was measured, and the mass fraction was calculated from this gravimetric procedure. The gravimetric mass fraction was adjusted for the purity estimation of the ethanol, which was determined using GC-FID using two stationary phases of different polarities, differential scanning calorimetry, and Karl Fischer analysis for water content. The bulk solution was chilled slightly, and 10 mL aliquots were dispensed into 10-milliliter glass ampoules, which were then flame sealed.

Duplicate aliquots from ten ampoules, selected using a stratified random sampling scheme, were analyzed by GC-FID on a relatively polar DB-wax column, 15 m × 0.45 mm id, 0.85 µm film thickness (Agilent Technologies, Wilmington, DE, USA). The internal standard added to each sample for quantification purposes was 1-propanol. Calibration solutions consisting of weighed amounts of ethanol and the internal standard compound in organic-free water were chromatographically analyzed to determine analyte response factors.

REFERENCES

- [1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.S.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at <http://www.nist.gov/srm/publications.cfm> (accessed May 2013).
- [2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2013); 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://www.nist.gov/pml/pubs/tn1297/index.cfm> (accessed May 2013).
- [3] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to Expression of Uncertainty in Measurement; Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed May 2013).
- [4] Efron, B.; Tibshirani, R. J.; *An Introduction to the Bootstrap*; Chapman & Hall, London, UK (1993).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.

⁽¹⁾ Certain commercial equipment, instruments, or materials are identified in this certificate to adequately 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.