National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 2896

Ethanol-Water Solution (Nominal Mass Fraction 0.3 %)

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 0.3 % and is intended primarily for use in the calibration of instruments and techniques used for the determination of ethanol. A unit of SRM 2896 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Mass Fraction of Ethanol: The certified value given below is based on results obtained from the gravimetric preparation of the solution and from the analytical results determined using gas chromatography and titrimetry. 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].

Certified Mass Fraction of Ethanol: 0.2980 % ± 0.0030 %

The result is expressed as the certified value ± the expanded uncertainty. The certified value is an unweighted mean of concentrations determined by gravimetric preparation and chromatographic and titrimetric measurements [2]. The uncertainty listed is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the GUM Guide [3]. The uncertainty includes both correction for estimated purity and allowance for differences among the concentrations determined by gravimetric preparation and chromatographic and titrimetric measurements.

Expiration of Certification: The certification of **SRM 2896** is valid, within the measurement uncertainty specified, until **30 April 2023**, provided the SRM is handled and stored in accordance with 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.

The coordination of the technical measurements leading to the certification of this SRM was under the direction of M.M. Schantz and S.A. Wise of the NIST Chemical Sciences Division.

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

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Preparation of and analytical measurements on the SRM were performed by J.V. Goodpaster and M.M. Schantz of the NIST Chemical Sciences Division and M.P. Cronise and C.N. Fales of the NIST Office of Reference Materials. Additional analytical measurements were performed by M. Archer of the National Metrology Laboratory, Pretoria, South Africa.

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Gaithersburg, MD 20899
Certificate Issue Date: 11 April 2013
Certificate Revision History on Last Page

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

Support aspects involved with the issuance of this SRM were coordinated through the NIST 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 mixed overnight (a minimum of 16 h). The total mass of the solution was measured, and the concentration was calculated from this gravimetric procedure. The gravimetric concentration was adjusted for the purity estimation of the ethanol, which was determined using gas chromatography with flame ionization detection (GC-FID) with two stationary phases of different polarities, differential scanning calorimetry, and Karl Fischer analysis for water content. The bulk solution was chilled slightly, and 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules, which were then flame sealed.

Aliquots from nine ampoules, selected using a random stratified sampling scheme, were analyzed in duplicate by using capillary gas chromatography with flame ionization detection 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.

In addition, the concentration of the solution was determined at the National Metrology Laboratory using titrimetry. The ethanol in known masses of the solutions was oxidized to acetic acid using a known mass of standard potassium dichromate solution in the presence of sulfuric acid. The quantity of ethanol in the solution was determined from the quantity of unreacted potassium dichromate in the solution. To determine the quantity of unreacted potassium dichromate, potassium iodide was added to the oxidized mixture, and the liberated iodine was titrated with a sodium thiosulfate solution. Eight titrations were done for this solution.

[1] Certain commercial instruments, materials, or processes are identified in this report to adequately specify 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 instruments, materials, or processes identified are necessarily the best available for the purpose.

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.; *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/upload/SP260-136.PDF (accessed Apr 2013).

[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.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571–579 (2000).

[3] 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/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 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/index.cfm (accessed Apr 2013).

Certificate Revision History: 11 April 2013 (Extension of certification period; editorial changes): **26 March 2004** (Original certificate date).

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.