National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 2895

Ethanol-Water Solution (nominal 0.2 % by mass)

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 concentration of 0.2 % by mass. SRM 2895 is intended primarily for use in the calibration of instruments and techniques used for the determination of ethanol in blood. This concentration along with five other levels (nominal concentrations of 0.02 %, 0.04 %, 0.08 %, 0.1 %, 0.2 %, and 0.3 % by mass) are available as a set in SRM 1828b and as individual solutions in SRMs 2891 through 2896, respectively. A second SRM suite, SRM 1847, is intended primarily for the calibration of instruments and techniques used for the determination of ethanol in breath. SRM 1847 is a set of three levels (nominal concentrations of 2 %, 6 %, and 25 % by mass). These three levels are also available as individual solutions in SRMs 2897 through 2899, respectively. A unit of SRM 2895 consists of five 2-milliliter ampoules, each containing approximately 1.2 mL of solution.

Certified Concentration of Ethanol: The certified concentration 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 accounted for by NIST.

Ethanol Certified Concentration Value: 0.1701 % ± 0.0014 % (by mass)

The results are expressed as the certified value ± the expanded uncertainty. Certified values are unweighted means of concentrations determined by gravimetric preparation and chromatographic and titrimetric measurements [1]. 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-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guides [2]. 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 this SRM 2895 is valid, within the measurement uncertainties specified, until **31 January 2014**, provided the SRM is handled, stored, and used in accordance with the instructions given in this certificate (see "Notice and Warning to Users"). However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the rights 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 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 Analytical Chemistry Division.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 26 March 2004

John Rumble, Jr., Chief
Measurement Services Division

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.

Partial funding support for the preparation and certification of this Standard Reference Material was provided by the National Institute of Justice (NIJ) and managed through the NIST Office of Law Enforcement Standards (OLES).

Preparation of and analytical measurements on the SRM were performed by J.V. Goodpaster and M.M. Schantz of the NIST Analytical Chemistry Division and M.P. Cronise and C.N. Fales of the NIST Measurement Services Division. Additional analytical measurements were performed by M. Archer of the National Metrology Laboratory, Pretoria, South Africa and L.G. Mackay and D.G. Burke of the National Analytical Reference Laboratory, Sydney, Australia.

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.

NOTICE AND WARNING TO USERS

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.

Instructions for Use: Sample aliquots for analysis should be withdrawn **immediately** after opening the ampoules and should be processed without delay for the certified concentration value to be valid within the stated uncertainty. Because of the volatility of ethanol, the certified concentration 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

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 flame ionization capillary gas chromatography 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-milliliter 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)[1]. 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 (Pretoria, South Africa) 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 equipment, instruments, or materials are identified in this certificate in order to specify 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.

The concentration of this solution was also determined at the National Analytical Reference Laboratory (Sydney, Australia) using an exact matching isotope dilution-gas chromatographic/mass spectrometric (ID-GC/MS) method. The ID-GC/MS method utilized carbon-13 labeled ethanol and GC/MS analysis with a Chrompack porous layer PoraPlot Q column, 27.5 m × 0.32 mm id (Varian, Mulgrave, Victoria, Australia)[1]. Duplicate samples from six ampoules were analyzed using the ID-GC/MS method.

REFERENCES

[1] 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).

[2] *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 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 at http://www.nist.gov/srm.