



National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material[®] 1828b

Ethanol-Water Solutions (Six Levels)

This Standard Reference Material (SRM) is a set of six levels of ethanol (ethyl alcohol: Chemical Abstracts Service [CAS] Registry Number 64-17-5). SRM 1828b is intended for use in the calibration of instruments and techniques used for the determination of ethanol in solutions. A unit of SRM 1828b consists of six 2 mL ampoules, one ampoule each of the nominal ethanol mass fractions of 0.02 %, 0.04 %, 0.08 %, 0.1 %, 0.2 %, and 0.3 %, labeled SRMs 2891 through 2896, respectively. Each ampoule contains approximately 1.2 mL of solution.

Certified Mass Fractions of Constituents: The certified mass fraction values and estimated uncertainties for each of the six solutions are given in Table 1. The certified values are based on results obtained from the gravimetric preparation of each solution and from the analytical results determined using gas chromatography (GC) 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 [1].

Expiration of Certification: The certification of **SRM 1828b** is valid, within the measurement uncertainty specified, until **30 April 2023**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). 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.

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.

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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 and L.G. Mackay and D.G. Burke of the National Analytical Reference Laboratory, Sydney, Australia.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
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Certificate Revision History on Last Page

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

INSTRUCTIONS FOR HANDLING, STORAGE AND USE

Handling: The solutions contain ethanol in water at the stated concentrations. 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 values in Table 1 to be valid within the stated uncertainty. Because of the volatility of ethanol, the certified values are **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 solutions were prepared at NIST by weighing and mixing known masses of ethanol and organic-free water. Each solution was mixed a minimum of 16 h. The total mass of each solution was measured, and the concentration of each solution was calculated from this gravimetric procedure. These gravimetric concentrations were 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 GC-FID on a relatively polar DB-wax column, 15 m × 0.45 mm id, 0.85 µm film thickness (Agilent Technologies, Wilmington, DE). 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 each of the six solutions 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. A minimum of seven titrations were done for each solution.

The concentrations of three of the solutions (nominal mass concentrations of 0.08 %, 0.1 %, and 0.2 %) were 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). Duplicate samples from six ampoules were analyzed using the ID-GC/MS method.

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

Certified Values: The results are expressed as the certified value $\pm$ the expanded uncertainty. Certified values are unweighted means of concentrations determined by gravimetric preparation and chromatographic and titrimetric measurements [2]. 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 between-method bias with a pooled within-source variance following the ISO 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.

Table 1. Certified Values of Ethanol^(a) in Water for the Six Levels Comprising SRM 1828b

Nominal Mass Fraction Level (%)	Mass Fraction (%)
0.02	0.01951 $\pm$ 0.00018
0.04	0.03900 $\pm$ 0.00046
0.08	0.08023 $\pm$ 0.00074
0.1	0.10084 $\pm$ 0.00083
0.2	0.1701 $\pm$ 0.0014
0.3	0.2980 $\pm$ 0.0030

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://www.nist.gov/srm/publications.cfm> (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 (2008); available at http://www.bipm.org/utls/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/tn1297/index.cfm> (accessed Apr 2013).

Certificate Revision History: 11 April 2013 (Extended certification period; editorial changes); 24 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>.