



# National Institute of Standards and Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1828c

#### 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 1828c is intended for use in the calibration of instruments and techniques used for the determination of ethanol in aqueous solutions. A unit of SRM 1828c 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 %. Each ampoule contains approximately 1.2 mL of solution.

**Certified Values:** The certified mass fraction values expressed as percent and associated uncertainties for each of the six solutions are given in Table 1. 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]. The certified values are based on the agreement of results obtained at NIST using two analytical techniques.

**Information Values:** Information values for the density at 20 °C for each of the six solutions are given in Table 2. An information value is a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [1]. Information values cannot be used to establish metrological traceability.

**Expiration of Certification:** The certification of **SRM 1828c** is valid, within the measurement uncertainty specified, until **30 April 2037**, 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 or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was under the direction of J.A. Murray and W.A. MacCrehan of the NIST Chemical Sciences Division.

Preparation of and analytical measurements on the SRM were performed by J. Murray and B. Lang of the NIST Chemical Sciences Division.

Statistical consultation and evaluation of the data were provided by N.-F. Zhang, of the NIST Statistical Engineering Division.

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

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Certificate Issue Date: 22 December 2017

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## 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.

**Metrological Traceability for Certified Values:** The certified mass fraction of ethanol for each of the six solutions is given in Table 1. Metrological traceability is to the SI derived unit for mass fraction, expressed as percent, through the mass balance purity assessment of neat material, determined by gas chromatography with flame ionization detection (GC-FID) using two separate columns and Karl Fischer analysis, and the certified value of SRM 2897a *Ethanol-Water Solution (Nominal Mass Fraction 2 %)* used to prepare calibrants.

Table 1. Certified Values of Ethanol in Water for the Six Levels Comprising SRM 1828c

Nominal Mass Fraction Level (%)	Mass Fraction <sup>(a)</sup> (%)
0.02	0.01729 ± 0.00023
0.04	0.03725 ± 0.00047
0.08	0.07663 ± 0.00097
0.1	0.0977 ± 0.0013
0.2	0.2037 ± 0.0027
0.3	0.2985 ± 0.0037

<sup>(a)</sup> Values are expressed as  $x \pm U_{95\%}(x)$ , where  $x$  is the certified mass fraction value (expressed as percent) and  $U_{95\%}(x)$  is the expanded uncertainty of the certified value [2,3]. The true value of the analyte is believed to lie within the interval  $x \pm U_{95\%}(x)$  with 95 % confidence. To propagate this uncertainty, treat the certified values as a normally distributed random variable with mean  $x$  and standard deviation  $U_{95\%}(x)/2$ .

Table 2. Information values for density of SRM 1828c at 20 °C.

Nominal Mass Fraction Level (%)	Density (g/mL)
0.02	0.9982
0.04	0.9982
0.08	0.9981
0.1	0.9980
0.2	0.9979
0.3	0.9977

## PREPARATION AND ANALYSIS<sup>(1)</sup>

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 12 h. Aliquots of 1.2 mL were dispensed into 2 mL amber glass ampoules that had been evacuated with argon and then flame sealed.

Aliquots from twelve 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. The ethanol used to prepare the calibration solutions were adjusted for the mass balance purity estimation of the ethanol, which was determined using GC-FID with two stationary phases of different polarities, and Karl Fischer analysis for water content.

In addition to the GC-FID analyses, the mass fraction of each of the six solutions was determined by using gas chromatography-mass spectrometry (GC-MS) on an Rxi-1301Sil MS column, 60 m x 0.25 mm id, 1.0 µm film thickness (Restek Technologies, Bellefonte, PA). The internal standard added to each sample for quantification purposes was 1-propanol. Calibration solutions consisting of weighed amounts of SRM 2897a and the internal standard compound in organic-free water were chromatographically analyzed to determine analyte response factors. Aliquots from six ampoules were analyzed in duplicate by GC-MS.

The density of all six solutions was estimated at 20 °C using a density meter with an oscillating U-tube sensor.

## 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 Dec 2017).
- [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 (2008); available at [http://www.bipm.org/utls/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed Dec 2017); 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 Dec 2017).
- [3] Zhang, N.F.; *Calculation of the Uncertainty of the Mean of Autocorrelated Measurements*; Metrologia, Vol. 43, pp. S276-S281 (2006).

*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](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*

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<sup>(1)</sup> 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.