DEPARTMENT OF COMMERCE
UNITED STATES OF AMERICA

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2890

Water Saturated 1-Octanol

This Standard Reference Material (SRM) is intended primarily for use in validating methods and calibrating instruments for the measurement of trace concentration levels of water. SRM 2890 is a solution of water saturated 1-octanol which is certified for its water content. A unit of SRM 2890 consists of five 5 mL ampoules, each containing approximately 2 mL of water saturated 1-octanol.

Certified Concentration (mass fraction) of Water Content

47.3 mg/g ± 1.0 mg/g
39.24 mg/mL ± 0.85 mg/mL(a)

(a) A density of 0.82930 g/mL at 21.5 °C ± 0.5 °C was used in the conversion between mass fraction (mg/g) and mass concentration (mg/mL).

The measurand is the water content in SRM 2890. The certified value of the water content is the equally weighted mean of the means of the results obtained from: (1) coulometric Karl Fischer measurements performed at NIST, (2) volumetric Karl Fischer measurements performed at NIST, and (3) an interlaboratory comparison exercise involving 15 collaborating laboratories. The expanded uncertainty in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO/JCGM and NIST Guides [1] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c accounts for within-method and between-method variations. The effective degrees of freedom can be considered infinite; therefore the coverage factor, $k = 2$, is a value for a 95 % confidence interval based on a normal distribution. The certified value is metrologically traceable to the International System of Units (SI) unit of mass, expressed as milligram per gram.

Expiration of Certification: The certification of **SRM 2890** is valid, within the measurement uncertainty specified, until **30 June 2027**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see “Notice and Warning to Users”). 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, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm). Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Preparation and analytical measurements were performed by L.T. Sniegoski and S.A. Margolis, formerly of the NIST Chemical Sciences Division.

Coordination of the technical measurements was under the direction of S.A. Wise of the NIST Chemical Sciences Division and S.A. Margolis.

Statistical consultation was provided by M.S. Levenson, formerly of the NIST Statistical Engineering Division.

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
Certificate Issue Date: 11 June 2025
Certificate Revision History on Page 3

Steven J. Choquette, Director
Office of Reference Materials

NOTICE AND WARNING TO USERS

Safety: Consult the Safety Data Sheet (SDS) for hazard information.

Storage, Handling, and Use: The ampoules are sealed under dry nitrogen and should be stored between 10 °C and 30 °C. After opening an ampoule, caution should be taken not to contaminate the sample. For the certified value to be valid within the stated uncertainty, samples should be withdrawn from the ampoule into a dry syringe.

PREPARATION AND ANALYSIS

Preparation of SRM 2890: 1-Octanol (CAS Number 111-87-5), as supplied by the manufacturer, was stirred over distilled water for 24 h at room temperature (21.5 °C ± 0.5 °C). The octanol-water suspension was allowed to stand and separate for three days. The water saturated octanol (upper layer) was decanted into a second bottle and then dispensed in 2.2 mL aliquots into washed and dried nitrogen filled ampoules, and flame sealed.

Analytical Methods: At NIST two groups of 20 ampoules each were selected from the fill sequence of ampoules by random stratified sampling. One group of ampoules was analyzed by the coulometric Karl Fischer method and the other group of ampoules was analyzed by the volumetric Karl Fischer method; each group of ampoules was analyzed in a random order over four days. The Karl Fischer instruments were calibrated with a series of five standards prepared by the gravimetric addition of increasing amounts of water to dry 1-octanol. The Karl Fischer instruments were also evaluated for systematic biases as described previously [2,3].

An interlaboratory comparison exercise among 15 laboratories and NIST was conducted to determine the water content of SRM 2890. All analyses in the interlaboratory comparison exercise were performed using the coulometric Karl Fischer methods and the reagents routinely used in the participating laboratories. In addition, one laboratory measured the water content by using a head-space/gas chromatography method. Each laboratory analyzed two subsamples from each of two ampoules of SRM 2890. The participating laboratories are listed below.

All Karl Fischer measurements at NIST were performed on a volume basis and the results converted to a mass basis using the density of the solution. The density of SRM 2890 was determined to be 0.82930 g/mL (standard deviation of 0.00049 g/mL, based on six measurements) at 21.5 °C ± 0.5 °C. Evaluation of the effect of temperature on the saturation of the 1-octanol indicated that the water content between 10 °C and 30 °C varied less than 5 % from the value at room temperature (21.5 °C ± 0.5 °C).

Homogeneity Study: Samples from 10 ampoules, selected throughout the fill sequence, were measured in duplicate at NIST in a random order within a single day. No statistically significant variation in the moisture content was detected with fill sequence.

Analysts and Laboratories Participating in the Interlaboratory Comparison Exercise:

P. Griffin; Doble Engineering Co., Watertown, MA
J. Lloyd; United Power Services, Nashville, TN
G. Gauger; Cooper Power Systems, Franksville, WI
G. Thomas; Big Rivers Electric Corp., Henderson, KY
C. Smith; Cosa Instrument Co., Norwood, NJ
F. Mees; UMM Electronics, Indianapolis, IN
J. Sans; S D Meyers, Tallmadge, OH
M. Anderson; Bonneville Power Administration, Vancouver, WA
C. Claiborne; ABB Power T&D Co. Inc., Raleigh, NC
F. Koch; Brinkmann Instruments Inc., Westbury, NY
T. Zazulak; Boston Edison, Boston, MA
G. Humphry; JOAP TSC, Pensacola, FL
T. Shull; Robert Becker Laboratories, Patuxent River, MD
W. Zdrojewski; Mid-Atlantic Regional Materials Test Laboratory, Norfolk, VA
J. Jalbert; Institute de recherche de Hydro-Quebec, Variance, Quebec, Canada

REFERENCES

[1] 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 https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Jun 2025); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Jun 2025).

[2] Margolis, S.A.*; Sources of Systematic Bias in the Measurement of Water by the Coulometric and Volumetric Karl Fischer Methods*; Anal. Chem., Vol. 23, pp. 4864–4871 (1997).

[3] Margolis, S.A.; *Systematic Errors in the Measurement of Moisture by Karl Fischer Methods*; Third Intl. Symp. on Humidity and Moisture, Vol. 2, pp. 133−140, National Physics Laboratory, Teddington, UK (1998).

Certificate Revision History: 11 June 2025 (Change of expiration date; editorial changes); **09 June 2021** (Change of expiration date; editorial changes); **02 July 2015** (Change of expiration date; editorial changes); **31 July 2008** (Update of expiration date; editorial changes); **25 August 1998** (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; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.