National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 2278

Deuterated Organic Acids in Methanol:Methylene Chloride

This Standard Reference Material (SRM) is a solution of seven deuterated organic acids in methanol:methylene chloride. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of organic acids. A unit of SRM 2278 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentrations of Constituents: The certified concentration values and estimated uncertainties for the seven constituents, expressed as mass fractions, are given in Table 1 along with the Chemical Abstract Service (CAS) Registry Numbers. The certified concentration values are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography. 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.

Expiration of Certification: The certification of SRM 2278 is valid, within the measurement uncertainties specified, until **31 December 2017**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for 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 L.C. Sander of the NIST Analytical Chemistry 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.

Preparation and analytical measurements of the SRM were performed by M.M. Schantz of the NIST Analytical Chemistry Division and M.P. Cronise and C.N. Fales of the Standard Reference Materials Group.

Support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 22 October 2008

Robert L. Watters, Jr., Chief
Measurement Services Division

NOTICE AND WARNING TO USERS

Handling: This material should be handled with care. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

Instructions for Use: Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **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 the solvent, certified values are not applicable to material stored in ampoules that have been opened for more than 3 minutes, even if they are resealed.

PREPARATION AND ANALYSIS

The compounds used in the preparation of this SRM were obtained from commercial sources. The solution was prepared at NIST by weighing and mixing the individual compounds, methanol, and methylene chloride. The weighed components were added to the methanol (2.5 L, exact mass known). The methylene chloride (2.5 L, exact mass known) was then added, and the mixture was stirred overnight. The total mass of this solution was measured, and the concentrations were calculated from this gravimetric procedure. These gravimetric concentrations were adjusted for the purity estimation of each component, which was determined using flame ionization capillary gas chromatography with two stationary phases of different polarities. From the bulk solution, 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules, which were then flame sealed.

Aliquots from nine ampoules selected using a stratified, random sampling scheme were analyzed in duplicate by using capillary gas chromatography with flame ionization detection (GC-FID) following methylation of the acids. A non-polar stationary phase was used for the GC-FID analysis. The internal standards added to each sample for quantification purposes were non-deuterated organic acids. Calibration solutions consisting of weighed amounts of the compounds (adjusted for the purity estimation) and the internal standard compounds in methanol:methylene chloride were chromatographically analyzed to determine analyte response factors.

Table 1. Certified Concentrations of Components in SRM 2278

Compound name	CAS Registry # [a]	Concentration µg/g [b]
Benzoic acid-d_5	1079-02-3	16.23 ± 0.38
Decanoic acid-d_{19}	88170-22-3	16.22 ± 0.37
Hexanoic acid-d_{11}	95348-44-0	43.73 ± 1.15
Myristic acid-d_{27}	60658-41-5	16.67 ± 0.38
Palmitic acid-d_{31}	39756-30-4	18.72 ± 0.40
Phthalic acid-d_4	87976-26-9	17.08 ± 0.41
Succinic acid-d_4	14493-42-6	16.92 ± 0.35

[a] Chemical Abstracts, Fourteenth Collective Index. Index Guide, American Chemical Society, Columbus, Ohio, 2001.

[b] The results are expressed as the certified value ± the expanded uncertainty. The certified value is the average of the concentrations determined by gravimetric and chromatographic measurements. The expanded 95% uncertainty uses a coverage factor of 2 and includes both correction for estimated purity and allowance for differences between the concentration determined by gravimetric preparation and chromatographic measurements [1].

REFERENCES

[1] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); see 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).

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.