



National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material[®] 2268

Carbon-13 Labeled Levoglucosan in Ethyl Acetate

This Standard Reference Material (SRM) is a solution of carbon-13 labeled levoglucosan (1,6-anhydro-beta-d-glucose-¹³C₆) in ethyl acetate. This SRM is intended primarily for use as an internal standard or surrogate internal standard solution that is used to spike both the unknown sample and a calibration or external standard solution of non-labeled levoglucosan. A unit of SRM 2268 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentration of Carbon-13 Labeled Levoglucosan: The certified concentration value [1,2] given below is 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.

Carbon-13 Labeled Levoglucosan

62.2 mg/kg \pm 4.4 mg/kg

The results are expressed as the certified value \pm the expanded uncertainty. The certified value is the unweighted average of the concentration determined by gravimetric and chromatographic methods. The expanded uncertainty, at a 95 % level of confidence, is calculated as $U = ku_c$, where u_c is a combined standard uncertainty calculated according to the ISO and NIST Guides [3] and $k = 2$ is the coverage factor. The quantity u_c represents, at the level of one standard deviation, the combined effects of the uncertainty due to purity assessment and an allowance for differences between the concentration determined by gravimetric preparation and chromatographic measurements.

Expiration of Certification: The certification of this SRM lot is valid until **30 September 2016**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the right to withdraw, amend, or extend this certification at any time.

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.

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The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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Certificate Issue Date: 12 March 2007

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

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 between 10 °C and 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 value to be valid within the stated uncertainty. Because of the volatility of ethyl acetate, certified values are not applicable to material stored in ampoules that have been opened for more than 4 minutes, even if they are resealed.

PREPARATION AND ANALYSIS

The carbon-13 labeled levoglucosan used in the preparation of this SRM was obtained from a commercial source. The solution was prepared at NIST by weighing and mixing the carbon-13 labeled levoglucosan and ethyl acetate. The weighed component was added to the ethyl acetate and mixed overnight. The total mass of this solution was measured, and the concentration was calculated from this gravimetric procedure. The gravimetric concentration was adjusted for the purity estimation of the carbon-13 labeled levoglucosan, which was determined using flame ionization capillary gas chromatography with two stationary phases of different polarities. This bulk solution was then chilled to approximately -5 °C, 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 stratified, random sampling scheme were analyzed in duplicate by using capillary gas chromatography with mass spectrometric detection (GC/MS) in the electron impact ionization mode and a non-polar 5 % phenyl methylpolysiloxane phase following derivatization using N,O-bis-(trimethylsilyl)trifluoroacetamide (BSTFA) catalyzed with 1 % trimethylchlorosilane (TMCS) [4]. The internal standard added to each sample for quantification purposes was non-labeled levoglucosan (CAS # 498-07-7). Briefly, the levoglucosans were converted to their corresponding trimethylsilyl derivatives by adding 20 µL each of pyridine and a 1 % mixture of TMCS in BSTFA. These sample mixtures were heated to 60 °C for 2 h, after which enough ethyl acetate was added to bring the final sample volume to approximately 1 mL. The ethyl acetate solution was then analyzed by GC/MS. Calibration solutions consisting of weighed amounts of the carbon-13 labeled levoglucosan (adjusted for the purity estimation) and the internal standard compound in ethyl acetate were chromatographically analyzed to determine analyte response factors.

REFERENCES

- [1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; and 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).
- [2] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (1995).
- [3] 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).
- [4] Larsen, R.K., III; Schantz, M.M.; Wise, S.A.; *Determination of Levoglucosan in Particulate Matter Reference Materials*; Aerosol Sci. Technol.; Vol. 40, pp. 781–787 (2006).

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://nist.gov/srm>.