National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2266

Hopanes and Steranes in 2,2,4-Trimethylpentane

This Standard Reference Material (SRM) is a solution of five hopanes and five steranes in 2,2,4-trimethylpentane (*iso*-octane). This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of hopanes and steranes. A unit of SRM 2266 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Mass Fraction Values: The certified mass fraction values and estimated uncertainties for three hopanes and four steranes are given in Table 1 along with the Chemical Abstract Service (CAS) Registry Numbers [1]. 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 taken into account [2].

Reference Concentration Values: Reference values, also expressed as mass fractions, for two additional hopanes and one additional sterane are provided in Table 2. The reference values are noncertified values that do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple methods.

Expiration of Certification: The certification of **SRM 2266** is valid, within the measurement uncertainty specified, until **31 March 2026**, 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) will facilitate notification.

Partial funding support for the preparation and certification of this Standard Reference Material was provided by the U.S. Environmental Protection Agency, Office of Research and Development, Human Exposure and Atmospheric Science Division, National Exposure Research Laboratory.

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 Chemical Sciences Division.

Analytical measurements of the SRM were performed by M.M. Schantz of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

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

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Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Steven J. Choquette, Acting Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING, STORANGE AND USE

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.

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 2,2,4-trimethylpentane, certified values are not applicable to material stored in ampoules that have been opened for more than 5 minutes, even if they are resealed.

PREPARATION AND ANALYSIS

The compounds used in the preparation of this SRM were obtained from Chiron AS (Trondheim, Norway). The solution was prepared at NIST by weighing and mixing the individual compounds and 2,2,4-trimethylpentane. The weighed components were added to the 2,2,4-trimethylpentane and mixed 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 this 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/mass spectrometry with a non-polar 5 % phenyl methylpolysiloxane phase. The internal standards added to each sample for quantification purposes were deuterated polycyclic aromatic hydrocarbons. Calibration solutions consisting of weighed amounts of the compounds (adjusted for the purity estimation) and the internal standard compounds in 2,2,4-trimethylpentane were chromatographically analyzed to determine analyte response factors.

Table 1. Certified Concentrations of Components in SRM 2266

Compound	CAS Registry No.	Mass Fraction (µg/g)[a]	Mass Concentration (µg/mL)[b]
17α(H)-22,29,30-trisnorhopane	53584-59-1	4.55 ± 0.23	3.14 ± 0.16
17α(H),21β(H)-30-norhopane	53584-60-4	1.31 ± 0.04	0.90 ± 0.02
17α(H),21β(H)-hopane	13849-96-2	1.70 ± 0.12	1.17 ± 0.08
ααα 20R-cholestane	481-21-0	33.1 ± 1.0	22.8 ± 0.7
αββ 20R-cholestane	69483-47-2	10.61 ± 0.24	7.32 ± 0.17
αββ 20R 24S-methylcholestane	71117-90-3	2.32 ± 0.10	1.60 ± 0.07
ααα 20R 24R-ethylcholestane	62446-14-4	8.58 ± 0.32	5.92 ± 0.22

[a] 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 [3]. The measurand is the certified value for each compound listed. Metrological traceability is to the derived SI unit for mass fraction (expressed as micrograms per gram) and mass concentration (expressed as micrograms per milliliter).

[b] The concentrations listed in micrograms per milliliter were obtained by multiplying the certified values in micrograms per gram by the density of the solution at 20 °C (0.6899 g/mL). A concentration change of less than 1 % will occur for a 5 °C temperature change.

Table 2. Reference Concentrations of Components in SRM 2266

Compound	CAS Registry No.	Mass Fraction (µg/g)[a]	Mass Concentration (µg/mL)[b]
17α(H),21β(H)-22R-homohopane	60305-22-8	0.89 ± 0.03	0.62 ± 0.02
17α(H),21β(H)- 22S-homohopane	60305-23-9	1.54 ± 0.09	1.06 ± 0.06
αββ 20R 24R-ethylcholestane	71117-92-5	3.25 ± 0.15	2.24 ± 0.11

[a] 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 [3]. The measurand is the reference value for each compound listed as determined by the method indicated above. Metrological traceability is to the derived SI unit for mass fraction (expressed as micrograms per gram) and mass concentration (expressed as micrograms per milliliter).

[b] The concentrations listed in micrograms per milliliter were obtained by multiplying the certified values in micrograms per gram by the density of the solution at 20 °C (0.6899 g/mL). A concentration change of less than 1 % will occur for a 5 °C temperature change.

REFERENCES

[1] Chemical Abstracts, Fourteenth Collective Index; Index Guide; American Chemical Society: Columbus, OH (2001).

[2] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition 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/upload/SP260-136.PDF (accessed Apr 2016).

[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 (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2016); 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/index.cfm (accessed Apr 2016).

Certificate Revision History: 04 April 2016 (Change of expiration date; editorial changes); 25 August 2010 (Corrected the name of the analyte ααα 20R-cholestane in Table 1); 12 March 2007 (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.