National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2285

Ignitable Liquids Test Mixture

This Standard Reference Material (SRM) is intended primarily for use in the calibration of chromatographic instrumentation used for the classification of an ignitable liquid residue. SRM 2285 is a solution of 15 compounds, including even carbon number aliphatic hydrocarbons from hexane to tetracosane, toluene, *p*-xylene, 2-ethyltoluene, 3-ethyltoluene, and 1,2,4-trimethylbenzene in methylene chloride. A unit of SRM 2285 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Mass Fraction Values: The certified values for the 15 constituents are given in Table 1. These 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 uncertainty have been investigated or taken into account [1].

Information Concentration Values: Information values for concentrations, expressed as percent volume fractions, are provided in Table 2 for the components. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed [1].

Expiration of Certification: The certification of **SRM 2285** is valid, within the measurement uncertainty specified, until **30 March 2023**, 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) will facilitate notification.

Overall direction and coordination of technical measurements leading to certification were performed by M.M. Schantz and S.A. Wise of the NIST Chemical Sciences Division.

Preparation and analytical measurements of the SRM were performed by J.V. Goodpaster, B.J. Porter, and M.M. Schantz of the NIST Chemical Sciences 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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Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
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INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This material contains alkane and aromatic compounds and should be handled with care. Use proper disposal methods, see material safety data sheet.

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

PREPARATION AND ANALYSIS[(1)]

The compounds used in the preparation of this SRM were obtained from Aldrich (Milwaukee, WI), EM Science (Gibbstown, NJ), Mallinckrodt (Phillipsburg, NJ), and JT Baker (Phillipsburg, NJ). The weighed components were added to the methylene chloride and mixed until completely dissolved and homogenized. The total mass of this solution was measured, and the mass fractions were calculated from this gravimetric procedure. These mass fractions were adjusted for the purity estimation of each component, which was determined using gas chromatography with flame ionization detection (GC-FID) on two stationary phases of different polarities and differential scanning calorimetry. The 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 random stratified sampling scheme, were analyzed in duplicate by using GC-FID with two stationary phases of different polarities. The internal standard *n*-heptadecane was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of the compounds (adjusted for the purity estimation) and the internal standard compound in methylene chloride were chromatographically analyzed to determine analyte response factors. A representative chromatogram from the GC-FID analysis on each stationary phase is shown in Figure 1.

Certified Mass Fraction Values: The results are expressed as the certified value ± the expanded uncertainty. The certified value is the unweighted average of the mass fractions determined by gravimetry and chromatographic measurements. The expanded 95 % uncertainty uses a coverage factor, *k*, of 2 and incorporates both correction for estimated purity and allowance for differences between the mass fractions determined by gravimetric preparation and chromatographic measurements [2]. The Chemical Abstract Service (CAS) Registry Numbers are given in Table 1.

Table 1. Certified Mass Fractions of Components in SRM 2285

Compound	CAS Registry No.[(a)]	Mass Fraction (mg/g)
n-hexane	110-54-3	1.004 ± 0.038
n-octane	111-65-9	1.130 ± 0.040
n-decane	124-18-5	1.015 ± 0.023
n-dodecane	112-40-3	1.371 ± 0.031
n-tetradecane	629-59-4	1.307 ± 0.030
n-hexadecane	544-76-3	1.064 ± 0.030
n-octadecane	593-45-3	1.250 ± 0.043
n-eicosane	112-95-8	1.382 ± 0.047
n-docosane	629-97-0	1.356 ± 0.032
n-tetracosane	646-31-1	1.481 ± 0.046
toluene	108-88-3	1.249 ± 0.046
p-xylene	106-42-3	1.360 ± 0.042
2-ethyltoluene	611-14-3	1.284 ± 0.028
3-ethyltoluene	620-14-4	1.243 ± 0.026
1,2,4-trimethylbenzene	95-63-6	1.249 ± 0.031

[(a)] Chemical Abstracts, Eleventh Collective Index. Index Guide, American Chemical Society: Columbus, OH (1986).

[(1)] Certain commercial equipment, instruments, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

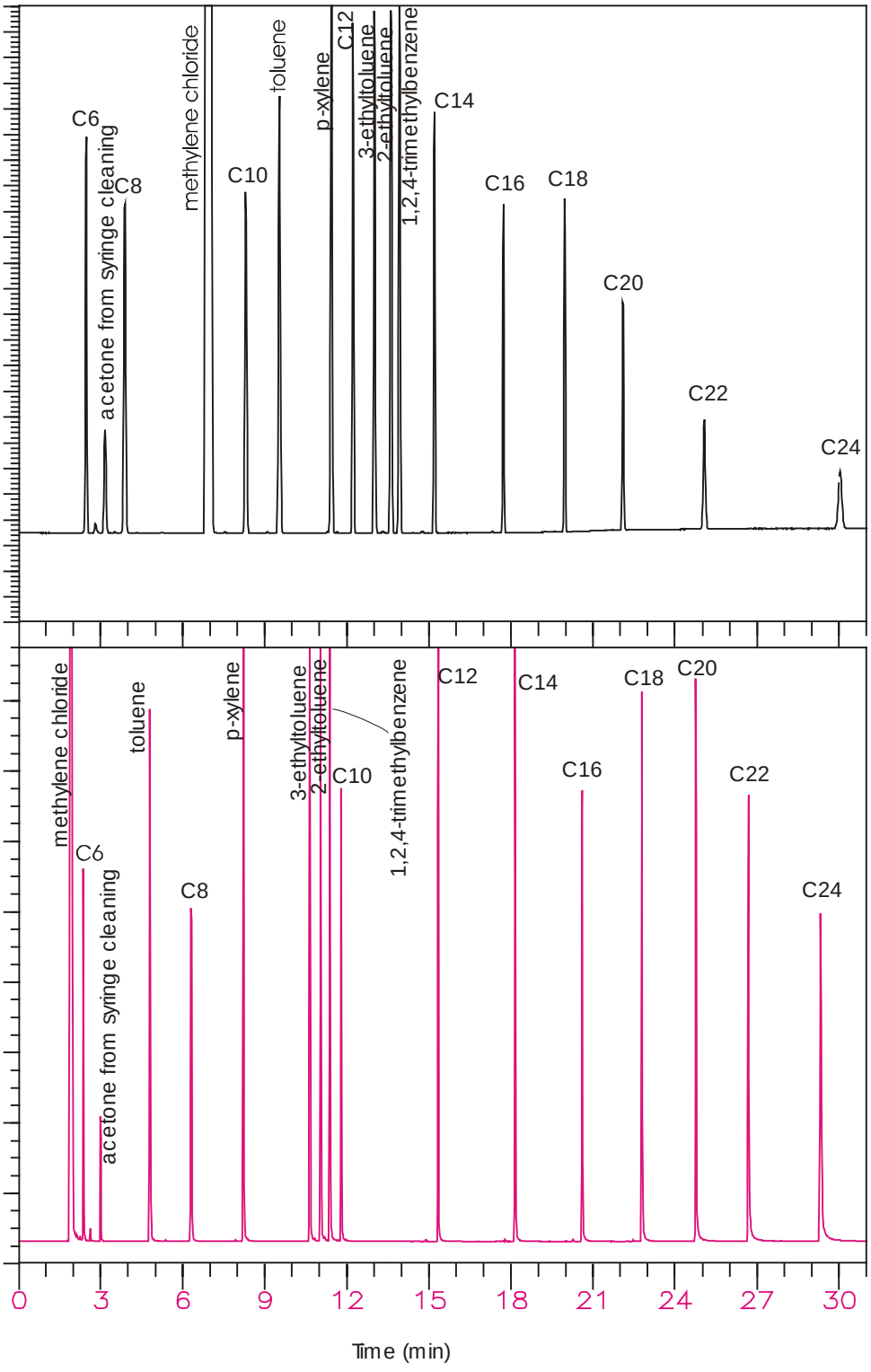

Figure 1. Chromatograms of GC-FID of SRM 2285 for each stationary phase. Upper chromatogram: polyethylene glycol column (15 m × 0.45 mm i.d., 0.85 µm film) with a temperature program from 40 °C (5 minute hold) to 200 °C (10 minute hold) at a rate of 10 °C/min and a constant flow of helium at 1.0 mL/min. Lower chromatogram: 100 % dimethylpolysiloxane column (30 m × 0.25 mm i.d., 0.5 µm film) with a temperature program from 55 °C (6 minute hold) to 250 °C (7 minute hold) at a rate of 10 °C/min and a constant flow of helium at 1.5 mL/min.

Table 2. Information Values (% volume fraction) of Components in SRM 2285

Compound	Information Values[(a)] (% volume fraction)
n-hexane	0.199
n-octane	0.211
n-decane	0.182
n-dodecane	0.240
n-tetradecane	0.224
n-hexadecane	0.180
n-octadecane	0.211
n-eicosane	0.230
n-docosane	0.224
n-tetracosane	0.243
toluene	0.189
p-xylene	0.207
2-ethyltoluene	0.191
3-ethyltoluene	0.188
1,2,4-trimethylbenzene	0.187

[(a)] The information concentration value in % volume fraction is provided for user information only. The values were obtained by multiplying the certified values by the measured density of the SRM solution at 22 °C (1.31 g/mL) and dividing by the densities of the individual compounds as reported in the CRC Handbook of Chemistry and Physics, 60th ed., CRC Press: Boca Raton, FL (1979).

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 May 2013)

[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/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2013); 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 May 2013).

Certificate Revision History: 09 May 2013 (Updated title to conform to current terminology; editorial changes); 05 March 2013 (Extension of certification period; editorial changes); 09 December 2003 (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.