National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 2260a

Aromatic Hydrocarbons in Toluene

This Standard Reference Material (SRM) is a solution of aromatic hydrocarbons, primarily polycyclic aromatic hydrocarbons (PAHs) ranging in molecular mass from 128 u to 302 u, in toluene. This SRM is intended for use in the calibration of chromatographic instrumentation used for the determination of aromatic hydrocarbons, primarily PAHs. A unit of SRM 2260a consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Mass Fractions of Constituents: Certified mass fraction values and estimated uncertainties are given in Table 1 along with the Chemical Abstract Service (CAS) Registry Numbers. The certified values are based on quantities used in the gravimetric preparation of this solution and from the analytical results obtained by using gas chromatography (GC) and are reported in mass fraction units [1]. 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].

Expiration of Certification: The certification of **SRM 2260a** is valid, within the measurement uncertainty specified, until **01 May 2031**, provided the SRM is handled and stored in accordance with instructions given in this certificate (See "Instructions for Storage, Handling, 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 online) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was under the direction of S.A. Wise of the NIST Chemical Sciences Division and M.M. Schantz, formerly of NIST.

Preparation and analytical measurements of the SRM were performed by D.L. Poster of the NIST Material Measurement Laboratory Office and M.M. Schantz.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh, formerly of NIST.

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

Carlos A. Gonzalez, Chief
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Certificate Revision History on Last Page

Steven J. Choquette, Director
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INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10 °C and 30 °C.

Handling: This material contains PAHs, many of which have been reported to have mutagenic and/or carcinogenic properties, and should be handled with care. Use proper disposal methods.

Use: Open ampoules carefully to prevent contamination and injury. The ampoules are pre-scored and should **NOT** be opened using a file. 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 uncertainties. Because of the volatility of toluene, certified values are not applicable to material stored in ampoules that have been open for more than 5 minutes, even if they are resealed.

PREPARATION AND ANALYSIS[(1)]

The compounds used in the preparation of this SRM were obtained from commercial sources or as Certified Reference Materials (CRMs) from the Institute for Reference Materials and Measurements (formerly BCR), Geel, Belgium. The solution was prepared at NIST by weighing and mixing the individual compounds and toluene. The weighed components were added to the toluene and mixed overnight. The total mass of this solution was measured, and the mass fractions were calculated from this gravimetric procedure. These gravimetric concentrations were adjusted for the purity estimation of each component, which was either determined by using flame ionization capillary GC with two stationary phases of different polarities and differential scanning calorimetry or through the use of CRMs. For select PAHs (naphthalene, benz[*a*]anthracene and benzo[*a*]pyrene), purity values and SI traceability for mass fraction were confirmed at NIST in October 2017. 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 stratified random sampling scheme, were analyzed in duplicate by using gas chromatography/mass spectrometry (GC/MS) with a moderately polar 50 % (mole fraction) phenyl methylpolysiloxane phase and a shape-selective liquid crystalline phase (50 % dimethyl/liquid crystalline polysiloxane). The internal standard solution added to each sample for quantification purposes was prepared from SRMs 2269 and 2270, Perdeuterated PAH-I and Perdeuterated PAH-II, respectively, in Hexane/Toluene. Calibration solutions consisting of weighed amounts of the compounds and the internal standard solution in toluene were chromatographically analyzed to determine analyte response factors.

During stability testing in November 2011, the dibenzo[*a,h*]pyrene content was found to be lower than originally certified. Therefore, the certified mass fraction of dibenzo[*a,h*]pyrene has been removed from the certificate, and because of the observed instability, a new value is not provided.

[(1)]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately 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.

Table 1. Certified Mass Fractions and Mass Concentrations of Components in SRM 2260a

Compound	CAS Registry No.[a]	Mass Fraction (µg/g)	Mass Concentration (µg/mL)[b]
Naphthalene	91-20-3	11.43 ± 0.30[c]	9.89 ± 0.26
Biphenyl	92-52-4	5.61 ± 0.14[c]	4.85 ± 0.12
Acenaphthylene	208-96-8	6.26 ± 0.20[c]	5.41 ± 0.17
Acenaphthene	83-32-9	5.55 ± 0.13[c]	4.80 ± 0.11
Fluorene	86-73-7	4.71 ± 0.11[c]	4.07 ± 0.10
Dibenzothiophene	132-65-0	4.39 ± 0.17[c]	3.80 ± 0.15
Phenanthrene	85-01-8	11.57 ± 0.12[d]	10.01 ± 0.10
Anthracene	120-12-7	3.736 ± 0.054[d]	3.231 ± 0.047
4*H*-Cyclopenta[*def*]phenanthrene	203-64-5	2.32 ± 0.11[c]	2.01 ± 0.10
Fluoranthene	206-44-0	8.324 ± 0.087[d]	7.200 ± 0.075
Pyrene	129-00-0	8.949 ± 0.083[d]	7.741 ± 0.072
Benzo[*ghi*]fluoranthene	203-12-3	3.414 ± 0.045[d]	2.953 ± 0.039
Cyclopenta[*cd*]pyrene	27208-37-3	1.958 ± 0.024[d]	1.694 ± 0.021
Benzo[*c*]phenanthrene	195-19-7	4.608 ± 0.036[d]	3.986 ± 0.031
Benz[*a*]anthracene	56-55-3	4.415 ± 0.078[d]	3.819 ± 0.067
Chrysene	218-01-9	4.62 ± 0.11[c]	4.00 ± 0.10
Triphenylene	217-59-4	4.12 ± 0.16[c]	3.56 ± 0.14
Benzo[*b*]fluoranthene	205-99-2	7.86 ± 0.10[d]	6.80 ± 0.09
Benzo[*j*]fluoranthene	205-82-3	4.145 ± 0.097[c]	3.585 ± 0.084
Benzo[*k*]fluoranthene	207-08-9	3.444 ± 0.036[d]	2.979 ± 0.031
Benzo[*a*]fluoranthene	203-33-8	2.279 ± 0.064[c]	1.971 ± 0.055
Benzo[*e*]pyrene	192-97-2	4.561 ± 0.054[d]	3.945 ± 0.047
Benzo[*a*]pyrene	50-32-8	4.71 ± 0.17[d]	4.07 ± 0.15
Perylene	198-55-0	4.430 ± 0.045[d]	3.83 ± 0.039
Indeno[1,2,3-*cd*]pyrene	193-39-5	4.425 ± 0.030[d]	3.828 ± 0.026
Benzo[*ghi*]perylene	191-24-2	5.669 ± 0.069[d]	4.904 ± 0.060
Dibenz[*a,h*]anthracene	53-70-3	4.555 ± 0.063[d]	3.940 ± 0.054
Dibenz[*a,c*]anthracene	215-58-7	2.912 ± 0.026[d]	2.519 ± 0.022
Dibenz[*a,j*]anthracene	224-41-9	4.539 ± 0.062[d]	3.926 ± 0.054
Picene	213-46-7	3.257 ± 0.047[d]	2.817 ± 0.041
Benzo[*b*]chrysene	214-17-5	4.092 ± 0.033[d]	3.540 ± 0.029
Anthanthrene	191-26-4	2.205 ± 0.029[d]	1.907 ± 0.025
Coronene	191-07-1	2.255 ± 0.033[d]	1.951 ± 0.029
Dibenzo[*b,k*]fluoranthene	205-97-0	1.646 ± 0.068[d]	1.424 ± 0.059
Dibenzo[*a,e*]pyrene	192-65-4	2.277 ± 0.023[d]	1.970 ± 0.020

[a] Chemical Abstracts, Fourteenth Collective Index Guide, American Chemical Society: Columbus, Ohio (2001). Note that dibenzo[*a,h*]pyrene (CAS Registry No. 189-64-0) is present in the solution, but the mass fraction of this compound in solution has not been stable over time.

[b] The values listed in mass concentration units were obtained by multiplying the certified values in µg/g by the density of the solution at 22 °C (0.8649 g/mL). These values are for use in the temperature range of 20 °C to 25 °C, and an allowance for the change in density over this temperature range is included in the uncertainties.

[c] The certified value is the mean of the mass fractions 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 mass fraction determined by gravimetric preparation and chromatographic measurements [3,4].

[d] The certified value is the mean of the mass fractions determined by gravimetric and chromatographic measurements. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 3, calculated by combining a between-method variance [5] with a pooled, within-method variance following the ISO Guide [3,4].

REFERENCES

[1] Thompson A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI);* NIST Special Publication 811; National Institute of Standards and Technology, U.S. Government Printing Office: Washington, DC (2008); available at https://www.nist.gov/pml/special-publication-811 (accessed May 2021).

[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 (2000); available at https://www.nist.gov/system/files/documents/srm/SP260-136.PDF (accessed May 2021).

[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 (2008); available at https://www.bipm.org/en/publications/guides (accessed May 2021); 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 May 2021).

[4] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to Expression of Uncertainty in Measurement*; Propagation of Distributions Using a Monte Carlo Method; Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/en/publications/guides (accessed May 2021).

[5] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.-K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, No. 4, pp. 571–579 (2000).

Certificate Revision History: 11 May 2021 (Change of expiration date; editorial changes); **06 November 2017** (Addition of traceability statement for naphthalene, benz[*a*]anthracene and benzo[*a*]pyrene; editorial changes); **08 April 2013** (Extension of certification period, editorial changes); **13 February 2012** (Removal of certified value for dibenzo[*a,h*]pyrene; editorial revisions); **03 December 2004** (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.