National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1491a

Methyl-Substituted Polycyclic Aromatic Hydrocarbons in Toluene

This Standard Reference Material (SRM) is a solution of 18 methyl-substituted polycyclic aromatic hydrocarbons (PAHs), from methylnaphthalenes to methylchrysenes, in toluene. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of methyl-substituted PAHs. A unit of SRM 1491a consists of five 2-milliliter ampoules, each containing approximately 1.2 mL of solution.

Certified Mass Fraction Values: A NIST certified value is a value for which NIST has the highest confidence in its accuracy and that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified mass fraction values and estimated uncertainties for the 18 constituents are given in Table 1 along with the Chemical Abstract Service (CAS) Registry Numbers. Certified concentration values based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography are also included in Table 1. The measurands are the total mass fraction values and concentration values listed in Table 1. The values are metrologically traceable to the International System of Units (SI) derived units for mass fraction (expressed as micrograms per gram) and concentration (expressed as micrograms per milliliter).

Expiration of Certification: The certification of **SRM 1491a** is valid, within the measurement uncertainty specified, until **01 May 2031**, 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 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 was 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.

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Gaithersburg, MD 20899
Certificate Issue Date: 13 May 2021
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING. STORAGE. AND USE

Handling: This material contains methyl-substituted polycyclic aromatic hydrocarbon compounds, many of which have been reported to have mutagenic and/or carcinogenic properties, and 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.

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

Table 1. Certified Mass Fraction Values and Concentrations Values of SRM 1491a

Compound	CAS Registry No.[a]	Mass Fraction Values µg/g[b]	Concentration Values µg/mL[c]
1-Methylnaphthalene	90-12-0	1.758 ± 0.041	1.520 ± 0.035
2-Methylnaphthalene	91-57-6	2.030 ± 0.096	1.756 ± 0.083
1,2-Dimethylnaphthalene	573-98-8	1.990 ± 0.071	1.721 ± 0.061
1,6-Dimethylnaphthalene	575-43-9	1.607 ± 0.036	1.390 ± 0.031
2,6-Dimethylnaphthalene	581-42-0	1.564 ± 0.053	1.353 ± 0.046
1-Methylphenanthrene	832-69-9	2.243 ± 0.028	1.940 ± 0.024
2-Methylphenanthrene	2531-84-2	2.396 ± 0.018	2.072 ± 0.016
3-Methylphenanthrene	832-71-3	2.134 ± 0.010	1.846 ± 0.009
9-Methylphenanthrene	883-20-5	2.288 ± 0.019	1.979 ± 0.016
2-Methylanthracene	613-12-7	1.355 ± 0.010	1.172 ± 0.009
1,7-Dimethylphenanthrene	483-87-4	1.962 ± 0.027	1.697 ± 0.023
1-Methylfluoranthene	25889-60-5	1.116 ± 0.011	0.965 ± 0.010
3-Methylfluoranthene	1706-01-0	1.190 ± 0.014	1.029 ± 0.012
1-Methylpyrene	2381-21-7	1.089 ± 0.013	0.942 ± 0.011
4-Methylpyrene	3353-12-6	1.026 ± 0.012	0.887 ± 0.010
Retene	483-65-8	2.079 ± 0.032	1.798 ± 0.028
3-Methylchrysene	3351-31-3	1.132 ± 0.026	0.979 ± 0.022
6-Methylchrysene	1705-85-7	1.200 ± 0.014	1.038 ± 0.012

[a] Chemical Abstracts; Fourteenth Collective 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 mean of the concentrations determined by gravimetric and chromatographic measurements. The expanded 95 % uncertainty uses a coverage factor of 2 (except a coverage factor of 4 for 4-methylpyrene) and includes both correction for estimated purity and allowance for differences between the concentration determined by gravimetric preparation and chromatographic measurements [1].

[c] The concentrations listed in µg/mL 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 concentrations 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.

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 the Community Bureau of Reference (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 concentrations 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 gas chromatography with two stationary phases of different polarities and differential scanning calorimetry, or through the use of CRMs. The bulk solution was then chilled to approximately −5 °C and 1.2 mL aliquots were dispensed into 2-milliliter 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 gas chromatography/mass spectrometry with a moderately polar 50 % 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 SRM 2269 and SRM 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.

REFERENCES

[1] 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).

[2] 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, p. 571 (2000).

Certificate Revision History: 13 May 2021 (Change of expiration date; editorial changes); 14 December 2016 (Change of expiration date; editorial changes); 31 January 2005 (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.

[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.