



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 2265

#### Nitrated Polycyclic Aromatic Hydrocarbons in Methylene Chloride II

This Standard Reference Material (SRM) is a solution of 16 nitrated polycyclic aromatic hydrocarbons (nitro-PAHs) from nitrophenanthrenes to nitrobenzopyrenes in methylene chloride. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of nitro-PAHs. SRM 2265 is part of a suite of SRMs containing nitro PAHs in various solvents. A unit of SRM 2265 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

**Certified Concentrations of Constituents:** The certified concentration values and estimated uncertainties for 14 nitro-PAHs, expressed as mass fractions, are given in Table 1 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 accounted for by NIST [2].

**Reference Concentration Values:** Reference values, also expressed as mass fractions, for two additional nitro-PAHs 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 this SRM lot is valid until **31 March 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 representative solutions from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Registration (see attached sheet) 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.

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.

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

Stephen A. Wise, Chief  
Analytical Chemistry Division

Robert L. Watters, Jr, Chief  
Measurement Services Division

Gaithersburg, MD 20899  
Certificate Issue Date: 08 March 2007

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.

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

## NOTICE AND WARNING TO USERS

**Handling:** This material contains nitro-PAHs and should be handled with care. Use proper disposal methods. Refer to the corresponding Material Safety Data Sheet (MSDS) for further handling instructions.

**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 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 4 minutes, even if they are resealed.

## PREPARATION AND ANALYSIS<sup>1</sup>

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 (IRMM) (formerly BCR [Community Bureau of Reference], Geel, Belgium). The solution was prepared at NIST by weighing and mixing the individual compounds and methylene chloride. The weighed components were added to the methylene chloride 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 and differential scanning calorimetry. 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 in the negative ion chemical ionization mode and a non-polar 5 % phenyl methylpolysiloxane phase. The internal standards added to each sample for quantification purposes were deuterated nitro-PAHs. Calibration solutions consisting of weighed amounts of the compounds (adjusted for the purity estimation) and the internal standard compounds in methylene chloride were chromatographically analyzed to determine analyte response factors.

---

<sup>1</sup>Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 1. Certified Concentrations of Components in SRM 2265

Compound	CAS Registry No. <sup>(a)</sup>	Concentration $\mu\text{g/g}^{(b)}$	Concentration $\mu\text{g/mL}^{(c)}$
2-Nitrophenanthrene	17024-18-9	3.73 $\pm$ 0.08	4.94 $\pm$ 0.11
3-Nitrophenanthrene	17024-19-0	4.36 $\pm$ 0.11	5.78 $\pm$ 0.15
9-Nitrophenanthrene	954-46-1	3.02 $\pm$ 0.09	4.00 $\pm$ 0.11
2-Nitroanthracene	3586-69-4	2.58 $\pm$ 0.08	3.43 $\pm$ 0.10
9-Nitroanthracene	602-60-8	4.58 $\pm$ 0.15	6.07 $\pm$ 0.20
2-Nitrofluoranthene	13177-29-2	4.12 $\pm$ 0.11	5.46 $\pm$ 0.15
3-Nitrofluoranthene	892-21-7	4.63 $\pm$ 0.10	6.14 $\pm$ 0.13
1-Nitropyrene	5522-43-0	5.21 $\pm$ 0.20	6.91 $\pm$ 0.27
2-Nitropyrene	789-07-1	3.24 $\pm$ 0.08	4.29 $\pm$ 0.10
4-Nitropyrene	57835-92-4	3.30 $\pm$ 0.07	4.38 $\pm$ 0.09
6-Nitrochrysene	7496-02-8	2.91 $\pm$ 0.08	3.85 $\pm$ 0.11
7-Nitrobenz[a]anthracene	20268-51-3	4.80 $\pm$ 0.16	6.36 $\pm$ 0.21
3-Nitrobenzanthrone	17117-34-9	3.31 $\pm$ 0.08	4.39 $\pm$ 0.11
6-Nitrobenzo[a]pyrene	63041-90-7	3.45 $\pm$ 0.14	4.57 $\pm$ 0.19

<sup>(a)</sup> The results are expressed as the certified value  $\pm$  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].

<sup>(b)</sup> The concentrations listed in  $\mu\text{g/mL}$  units were obtained by multiplying the certified values in  $\mu\text{g/g}$  by the density of the solution at 20 °C (1.326 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 2265

Compound	CAS Registry No.	Concentration $\mu\text{g/g}^{(a)}$	Concentration $\mu\text{g/mL}^{(b)}$
1-Nitrobenzo[e]pyrene	91259-16-4	3.16 $\pm$ 0.14	4.19 $\pm$ 0.19
3-Nitrobenzo[e]pyrene	81340-58-1	1.49 $\pm$ 0.26	1.98 $\pm$ 0.34

<sup>(a)</sup> The results are expressed as the certified value  $\pm$  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].

<sup>(b)</sup> The concentrations listed in  $\mu\text{g/mL}$  units were obtained by multiplying the certified values in  $\mu\text{g/g}$  by the density of the solution at 20 °C (1.326 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, 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: Gaithersburg, MD (2000).
- [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 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://physics.nist.gov/Pubs/>.

*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](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*