DEPARTMENT OF COMMERCE UNITED STATES OF AMERICA

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2975

Diesel Particulate Matter (Industrial Forklift)

This Standard Reference Material (SRM) is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs) and nitro-substituted PAHs (nitro-PAHs) in diesel particulate matter and similar matrices. In addition to certified and non-certified values for selected PAHs and nitro-PAHs, non-certified values are provided for total extractable mass, particle-size distribution, and specific surface area; supplemental information on mutagenic activity is also provided. All of the chemical constituents for which certified and non-certified values are provided are naturally present in the diesel particulate material. A unit of SRM 2975 consists of a bottle containing 1 g of the diesel particulate matter collected from an industrial diesel-powered forklift.

SRM 1975 Diesel Particulate Extract [1], which is a dichloromethane extract of the diesel particulate matter, was prepared from the same lot of material as SRM 2975. A second diesel particulate material, SRM 1650b Diesel Particulate Matter [2], which was originally issued in 1985, is representative of heavy-duty diesel engine particulate emissions.

Certified Mass Fraction Values: Certified mass fraction values are provided for PAHs in Table 1. The certified values for the PAHs are based on the agreement of results obtained at NIST from two or more independent analytical methods [3]. 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 [3]. Metrological traceability is to the International System of Units (SI) unit of mass expressed as derived unit of mass fraction.

Non-Certified Values: Non-certified mass fraction values are provided for nitro-PAHs in Table 2, additional PAHs in Table 3 and nitro-PAHs for specific method conditions in Table 4. Some PAHs and nitro-PAHs are listed more than once in the table depending on the extraction conditions used (see "Preparation and Analysis"). Non-certified values for total extractable mass and the particle-size distribution are provided in Tables 5 and 6, respectively. A non-certified value for specific surface area of SRM 2975 as determined by N_2 gas adsorption is provided in Table 7. Non-certified values are the best estimates of the true value; however, the values do not meet the 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 analytical methods. Non-certified values are traceable to the measurement processes and standards used by NIST.

Period of Validity: **SRM 2975** is valid, with 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 Use"). The value assignments are nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its validity. 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 SRM 2975 was under the leadership of S.A. Wise of the NIST Chemical Sciences Division and M.M. Schantz, formerly of NIST.

Carlos A. Gonzalez, Chief
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Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Analytical measurements for the certification of SRM 2975 were performed by D.L. Poster of the Material Measurement Laboratory Office, L.C. Sander, of the NIST Chemical Sciences Division and H.M. Bamford, D. Bezabeh, M. Lopez de Alda, M.M. Schantz, P. Schubert, and L. Walton, formerly of NIST. Specific surface area and porosity data were provided by P. Scheepers of the Department of Epidemiology at Katholieke Universiteit Nijmegen, Nijmegen, The Netherlands. The particle-size distribution data were provided by Honeywell, Inc., Clearwater, FL.

The diesel particulate material was provided by M.E. Wright of the Donaldson Company, Inc., Minneapolis, MN.

Statistical consultation was provided by N.A. Heckert, of the NIST Statistical Engineering Division and S.D. Leigh and M.G. Vangel, formerly of NIST.

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This material is a naturally occurring diesel particulate material and contains constituents of known and unknown toxicities and mutagenicities; therefore, extreme caution and care should be exercised during its handling and use.

Storage: Store SRM 2975 in its original bottle at approximately 25 °C (room temperature) and keep away from direct sunlight.

Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed thoroughly. The recommended minimum sample size is 100 mg.

PREPARATION AND ANALYSIS(1)

Sample Collection and Preparation: The diesel particulate material used to prepare SRM 2975 was obtained from M.E. Wright of the Donaldson Company, Inc., Minneapolis, MN. The material was collected from a filtering system designed specifically for diesel-powered forklifts [4]. The diesel particulate material was received at NIST in a 55-gallon drum. The material was removed from the drum and homogenized in a V-blender for 1 h and then stored in polyethylene bags. A total of 13.7 kg of diesel particulate material was homogenized; a total of 5.65 kg of material was extracted for preparation of SRM 1975 [1] and the remaining diesel particulate material was bottled for distribution as SRM 2975.

Polycyclic Aromatic Hydrocarbons (PAHs)

The general approach used for the value assignment of the PAHs in SRM 2975 consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM), toluene, or toluene/methanol mixture, cleanup of the extracts using solid-phase extraction (SPE), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) for analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC) and (2) gas chromatography/mass spectrometry (GC/MS) for analysis of the PAH fraction on four stationary phases of different selectivity (i.e., a 5 % [mole fraction] phenyl-substituted methylpolysiloxane phase, a 50 % phenyl-substituted methylpolysiloxane phase, a non-polar, extra low bleed proprietary phase, and a smectic liquid crystalline stationary phase).

Multiple sets of GC/MS results, designated as GC/MS (Ia and Ib), GC/MS (II), GC/MS (III), GC/MS (IVa, IVb, and IVc), and GC/MS (Va through Vd), were obtained using three columns with different selectivities for the separation of PAHs.

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(1)Certain commercial instruments, materials, or processes 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 instruments, materials, or processes identified are necessarily the best available for the purpose.

GC/MS (Ia and Ib): For GC/MS (I) analyses, duplicate subsamples of 100 mg from eight bottles of SRM 2975 were extracted with toluene:methanol (1:1 volume fraction) using PFE as described by Schantz et al. [5]. The excess volume in the PFE cells was filled with clean sodium sulfate. The extracts were concentrated to about 0.5 mL and placed on an aminopropylsilane SPE cartridge from which they were eluted with 20 mL of 2 % DCM in hexane (volume fraction). The eluant was concentrated and then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness; DB-5MS, Agilent Technologies, Wilmington, DE), and these results are designated as GC/MS (Ia). A subset of 8 of the 16 extracts from GC/MS (Ia) were also analyzed on a 50 % (mole fraction) phenyl-substituted methylpolysiloxane stationary phase (0.25 mm i.d. × 60 m, 0.25 µm film thickness; DB-17MS, Agilent Technologies), and these results are designated as GC/MS (Ib).

GC/MS (II through V): For the GC/MS (II) analyses, 100 mg samples of SRM 2975 were extracted with DCM using PFE; the extracts were processed and analyzed as described above for GC/MS (Ia). GC/MS (III) was identical to GC/MS (II) except that Soxhlet extraction with DCM for 18 h was used instead of PFE. For the GC/MS (IV) analyses, subsamples of 40 mg to 100 mg of SRM 2975 were extracted with DCM using PFE and the extracts processed as described above for GC/MS (I). The processed extracts were then analyzed by GC/MS using three different columns: 5 % phenyl methylpolysiloxane [GC/MS (IVa)], 50 % phenyl methylpolysiloxane [GC/MS (IVb)], and a 0.2 mm i.d. × 25 m (0.15 µm film thickness) smectic liquid crystalline phase [SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT; GC/MS (IVc)].

In addition, a study investigating the effect of increasing the temperature and pressure used for PFE on the extraction efficiency for PAHs was performed. In this study, the solvent used was toluene although a 9:1 toluene:methanol (volume fraction) was also evaluated. The PFE conditions used included: 100 °C with 13.8 MPa; 100 °C with 20.7 MPa; 200 °C with 13.8 MPa; and 200 °C with 20.7 MPa [methods GC/MS (Va) through GC/MS (Vd), respectively]. Following an SPE step similar to GC/MS (I) above, the processed extracts were analyzed using a non-polar, extra low bleed proprietary phase (0.25 mm i.d. × 60 m, 0.25 µm film thickness).

Reversed-Phase Liquid Chromatography with Fluorescence Detection (LC-FL): For the LC-FL analyses, subsamples of approximately 200 mg from each of six bottles of SRM 2975 were Soxhlet-extracted for 20 h using 200 mL of DCM. The extracts were concentrated and processed through aminopropylsilane SPE cartridges as described above for the GC/MS analyses. The processed extract was further fractionated using normal-phase LC on a semi-preparative aminopropylsilane column (µBondapak NH_2, 9 mm i.d. × 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [6–8]. Four fractions were collected containing PAHs of relative molecular mass 178 and 202 (fraction 1), 228 (fraction 2), 252 and 276 (fraction 3), and 278 (fraction 4). All of the PAH fractions were analyzed using a 5 µm particle-size polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength-programmed fluorescence detection [7–9]. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the diesel particulate samples prior to solvent extraction for use as internal standards for quantification purposes.

PAH Isomers of Relative Molecular Mass (M_r) 302: For the determination of the relative molecular mass (M_r) 302 isomers, samples of approximately 100 mg each were extracted using PFE at 100 °C with DCM as the extraction solvent. The extracts were then concentrated with a solvent change to hexane, passed through an aminopropyl SPE cartridge, and eluted with 40 mL of 10 % DCM in hexane (volume fraction). The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substitued methylpolysiloxane phase (0.25 µm film thickness; DB-17MS). Perdeuterated dibenzo[*a,i*]pyrene was added to the diesel particulate matter prior to extraction for use as an internal standard.

Homogeneity Assessment for PAHs: The homogeneity of SRM 2975 was assessed by analyzing duplicate samples of 100 mg each from eight bottles selected by stratified random sampling. Samples were processed and analyzed as described above for GC/MS (I). Statistically significant differences among bottles were observed for the PAHs at the 100 mg sample size, and this source of uncertainty has been incorporated in the calculation of the uncertainty associated with the assigned values.

Nitro-Substituted Polycyclic Aromatic Hydrocarbons (Nitro-PAHs)

SRM 2975 was analyzed at NIST for the determination of nitro-PAHs. Three sets of three, four, and five samples of SRM 2975 (≈ 100 mg each) were spiked with the following perdeuterated nitro-PAHs for use as internal standards: 9-nitroanthracence-d_9, 3-nitrofluoranthene-d_9, 1-nitropyrene-d_9, and 6-nitrochrysene-d_{11}. The samples were extracted using PFE with DCM or toluene as the extraction solvent.

Following concentration, each sample was processed through an aminopropylsilane SPE cartridge using 40 mL of 20 % DCM in hexane (volume fraction). The concentrated eluant was then subjected to normal-phase LC using a semi-preparative amino/cyano-phase column with a mobile phase of 20 % DCM in hexane to isolate the nitro-PAH fraction. The nitro-PAH fraction was analyzed by GC with negative chemical ionization mass spectrometry (GC/NCI-MS) using a 0.25 mm i.d. × 30 m fused silica capillary column containing a 5 % diphenyl-substituted dimethylsiloxane phase (0.25 µm film thickness) or a 50 % phenyl-subsituted methylpolysiloxane phase (0.25 µm film thickness). Methane was used as the NCI-MS collision gas. The effect of increasing the temperature and pressure used in PFE was also investigated at NIST for the nitro-PAHs using 100 °C with 13.8 MPa; 100 °C with 20.7 MPa; 200 °C with 13.8 MPa; and 200 °C with 20.7 MPa.

The concentration of 1-nitropyrene was also measured at the Department of Epidemiology at Katholieke Universiteit Nijmegen, Nijmegen, The Netherlands using GC/MS [10].

Value Assignment for PAHs and Nitro-PAHs

The current value assignment of PAHs and nitro-PAHs in SRM 2975 is based on the measurements used when SRM 2975 was issued in 2000 and on recent additional measurements using different extraction conditions. Previous studies on solvent extraction of diesel particulate matter for determination of PAHs indicated that PFE at 100 °C removed higher quantitites of higher relative molecular mass PAHs than does Soxhlet extraction [5]. Recent studies on the extraction of PAHs and nitro-PAHs from diesel particulate matter [11–13] have shown that using PFE at 200 °C removes higher quantities of some (not all) PAHs and nitro-PAHs than using PFE at 100 °C. As a result of these studies, value assignment for PAHs and nitro-PAHs in SRM 2975 is based on measurements using PFE at both 100 °C and 200 °C and on limited measurements using Soxhlet extraction. In cases where the quantities of the individual PAHs and nitro-PAHs determined do not change with the extraction method or temperature, the measurements were combined and the resulting values are denoted as certified values. When different results are obtained using PFE at 100 °C or at 200 °C, the values are reported for both temperatures, and they are denoted as non-certified values. These non-certified values should be considered as “method dependent” values because they are dependent on the extraction method and temperature.

Total Extractable Mass

For the determination of total extractable mass, six subsamples of approximately 1 g to 2 g of SRM 2975 were Soxhlet-extracted for 18 h with DCM. The extract was concentrated to approximately 20 mL and then filtered to remove particulate matter. Aliquots of 100 µL to 150 µL were placed in tared aluminum foil pans; the DCM was evaporated until constant mass was obtained, and then the mass of the remaining residue was determined. The total extractable mass non-certified value is provided in Table 5.

Particle-Size Information

Dry particle-size distribution measurements for SRM 2975 were obtained as part of a collaborative effort with Honeywell, Inc., Clearwater, FL. A Microtrac particle analyzer, which makes use of light-scattering techniques, was used to measure the particle-size distribution of SRM 2975 [14]. Briefly, a reference beam was used to penetrate a field of particles and the light that scatters in the forward direction from the field is measured. The particle size as a volume distribution is derived via a computer-assisted analysis. From these data, the total volume, average size, and a characteristic width of the particle-size distribution were calculated.

Specific Surface Area and Porosity

The specific surface area and porosity were determined based on nitrogen gas adsorption measurements [15]. The gas adsorption measurements were performed on a NOVA-1200 instrument, Quantachrome Corp., Boynton Beach, FL at 77 K after the samples were outgassed for 24 h at 120 °C under vacuum. The nitrogen isotherms were analyzed using the Brunauer-Emmet-Teller (BET) equation [16] to obtain the surface area (Table 8) and the Barrett-Joyner-Halenda (BJH) method [17] to obtain the porosity. Based on the BJH method, SRM 2975 shows a wide distribution of mesopores, but with substantial outer area. The pore diameter of the particles in SRM 2975 range from 4 nm to 35 nm with the greater number of particles at about 20 nm.

Mutagenicity Activity

The mutagenicity of solvent extracts of SRM 2975 has not been determined; however, the non-certified values for mutagenicity of SRM 1975 were determined by using the the *Salmonella typhimurium*/mammalian microsome mutagenicity assay and should be applicable to extracts of SRM 2975. SRM 1975 was prepared by Soxhlet extraction using DCM of multiple samples of SRM 2975. Non-certified values for mutagenic activity for SRM 1975 are reported in the Certificate of Analysis for SRM 1975 [1] and described in detail by Hughes et al. [18].

Table 1. Certified Mass Fraction Values for Selected PAHs in SRM 2975

	Mass Fraction(a) (mg/kg)
2-Methylphenanthrene(b,c,d,e)	2.22 ± 0.21
1-Methylphenanthrene (b,c,d,e,f,g)	0.923 ± 0.057
Triphenylene(e,h,i)	5.32 ± 0.24
Benzo[*j*]fluoranthene (g,i,j)	0.819 ± 0.093
Benzo[*a*]fluoranthene(e,g,h)	0.066 ± 0.007
Dibenz[*a,h* + *a,c*]anthracene(e,f,i)	0.523 ± 0.047
Picene(e,f,g,i)	0.902 ± 0.091
Dibenzo[*a,e*]pyrene(e,g)	0.599 ± 0.024
Dibenzo[*b,k*]fluoranthene(e,g)	2.54 ± 0.08

(a) The certified values are weighted means of the mass fractions from two to six analytical methods [19]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within-method variances with a between-method variance [21] following the ISO/JCGM Guide [22,23].

(b) GC/MS (Ia) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with toluene:methanol mixture (100 °C with 13.8 MPa)

(c) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa)

(d) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM

(e) GC/MS (V) on non-polar extra low bleed proprietary phase after PFE with toluene at two temperatures and two pressures (100 °C with 13.8 MPa; 100 °C with 20.7 MPa; 200 °C with 13.8 MPa; and 200 °C with 20.7 MPa)

(f) GC/MS (IVa) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa)

(g) GC/MS (IVb) on 50 % phenyl-substituted methylpolysiloxane phase of same extracts as GC/MS (IVa)

(h) GC/MS (IVc) on a smectic liquid crystalline phase of same extracts as GC/MS (IVa)

(i) LC FL of isomeric PAH fractions after Soxhlet extraction with DCM

(j) GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase of selected extracts from GC/MS (Ia)

Table 2. Non-Certified Mass Fraction Values for Selected Nitro-PAHs in SRM 2975

	Mass Fraction(a) (mg/kg)
9-Nitrophenanthrene(b)	0.466 ± 0.013
3-Nitrophenanthrene(c)	0.190 ± 0.007
2-Nitrofluoranthene(c)	0.231 ± 0.032
3-Nitrofluoranthene(b)	3.80 ± 0.24
1-Nitropyrene(b)	35.2 ± 2.2
7-Nitrobenz[*a*]anthracene(b)	3.57 ± 0.32
6-Nitrochrysene(b)	2.45 ± 0.33

(a) The non-certified values are weighted means of the mass fractions from two to six analytical methods [19]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [21] following the JCGM Guide [22,23].

(b) Three methods using GC/NCI-MS on either a 5 % phenyl-substituted or 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa) and four methods using GC/NCI-MS on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with toluene at two temperatures and two pressures (100 °C with 13.8 MPa; 100 °C with 20.7 MPa; 200 °C with 13.8 MPa; and 200 °C with 20.7 MPa)

(c) Two methods using GC/NCI-MS on a 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa) and four methods using GC/NCI-MS on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with toluene at two temperatures and two pressures (100 °C with 13.8 MPa; 100 °C with 20.7 MPa; 200 °C with 13.8 MPa; and 200 °C with 20.7 MPa)

Table 3. Non-Certified Mass Fraction Values for Selected PAHs in SRM 2975
Based on Extraction Method and Conditions

Extraction Conditions		Mass Fractions(a) (mg/kg)		
Soxhlet Extraction or PFE at 100 °C				
	Naphthalene(b,c,d,e)	1.78	±	0.48
	Biphenyl(e)	0.323	±	0.014(f)
	Fluorene(e,g,h,i)	0.440	±	0.071
	Phenanthrene(b,c,d,e,g,h,i)	17.3	±	1.0
	Anthracene(b,c,e)	0.039	±	0.003
	3-Methylphenanthrene(c,d,e,g,h,i)	1.06	±	0.07
	9-Methylphenanthrene(d,e,j)	0.448	±	0.022
	1,2-Dimethylphenanthrene(c,d)	0.05	±	0.02
	1,6-, 1,7-, 2,5-, and 2,9-Dimethylphenanthrene(c,d)	0.57	±	0.08
	1,8-Dimethylphenanthrene(c,d)	0.06	±	0.02
	2,6-Dimethylphenanthrene(c,d)	0.25	±	0.05
	2,7-Dimethylphenanthrene(c,d)	0.23	±	0.05
	3,6-Dimethylphenanthrene(c,d)	0.18	±	0.02
	Fluoranthene(b,c,d,e,g,h,i)	26.9	±	1.1
	Pyrene(b,c,d,e,g,h,i)	0.94	±	0.11
	1-, 3-, and 7-Methylfluoranthene(c)	0.53	±	0.03
	2-Methylpyrene(c,d)	0.040	±	0.008
	4-Methylpyrene(c,d)	0.022	±	0.005
	Benzo[*c*]phenanthrene(c,d,e,g,h,i,k)	1.15	±	0.21
	Benz[*a*]anthracene(b,c,d,e,g,h,i)	0.330	±	0.037
	Chrysene(b,e,k)	4.58	±	0.17
	Benzo[*k*]fluoranthene(b,c,d,e,h,i,j,k)	0.699	±	0.058
	Benzo[*e*]pyrene(c,d,e,g,h,i,k)	1.14	±	0.06
	Benzo[*a*]pyrene(b,c,d,e,k)	0.053	±	0.003
	Perylene(b,e)	0.059	±	0.005
	Indeno[1,2,3-*cd*]pyrene(b,c,d,e)	1.37	±	0.16
	Benzo[*ghi*]perylene(b,c,d,e,g,h,i)	0.489	±	0.032
	Dibenz[*a,j*]anthracene(b,c,e)	0.337	±	0.071
	Benzo[*b*]chrysene(c,d,e)	0.094	±	0.018
	Coronene(c,e)	1.32	±	0.27
PFE at 200 °C				
	Naphthalene(l)	4.00	±	0.13(f)
	Biphenyl(l)	2.94	±	0.14(f)
	Fluorene(l)	2.88	±	0.19
	Phenanthrene(l)	20.7	±	0.3
	Anthracene(l)	0.049	±	0.002(f)
	3-Methylphenanthrene(l)	1.38	±	0.08
	9-Methylphenanthrene(l)	0.587	±	0.040
	Fluoranthene(l)	30.9	±	0.5
	Pyrene(l)	1.52	±	0.20
	Benzo[*c*]phenanthrene(l)	1.64	±	0.10(f)
	Benz[*a*]anthracene(l)	0.966	±	0.043(f)
	Chrysene(l)	5.73	±	0.11(f)
	Benzo[*k*]fluoranthene(l)	1.75	±	0.09(f)
	Benzo[*e*]pyrene(l)	2.28	±	0.18
	Benzo[*a*]pyrene(l)	0.766	±	0.039(f)
	Perylene(l)	0.082	±	0.006
	Indeno[1,2,3-*cd*]pyrene(l)	2.13	±	0.12(f)
	Benzo[*ghi*]perylene(l)	1.59	±	0.09(f)
	Dibenz[*a,j*]anthracene(l)	0.433	±	0.008(f)
	Benzo[*b*]chrysene(l)	0.210	±	0.011(f)
	Coronene(l)	2.12	±	0.08(f)

Table 3. Non-Certified Mass Fraction Values for Selected PAHs in SRM 2975
Based on Extraction Method and Conditions (continued)

Extraction Conditions	Mass Fractions(a) (mg/kg)		
Soxhlet Extraction or PFE using temperatures between 100 °C and 200 °C			
2-Methylnaphthalene(c,d,e,l)	1.91	±	0.32
1-Methylnaphthalene(c,d,e,l)	0.99	±	0.11
Acenaphthene(e,l)	0.548	±	0.029
Benzo[*ghi*]fluoranthene(e,l)	10.6	±	0.3
Benzo[*b*]fluoranthene(d,i,k)	11.5	±	2.8

(a) The non-certified values, unless otherwise footnoted, are weighted means of the mass fractions from two to six analytical methods [19]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [21] following the ISO/JCGM Guide [22,23].

(b) LC-FL of isomeric PAH fractions after Soxhlet extraction with DCM

(c) GC/MS (IVa) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa)

(d) GC/MS (IVb) on 50 % phenyl-substituted methylpolysiloxane phase of same extracts as GC/MS (IVa)

(e) GC/MS (V) on non-polar extra low bleed proprietary phase after PFE with toluene at one temperature and two pressures (100 °C with 13.8 MPa and 100 °C with 20.7 MPa)

(f) The non-certified value is a weighted mean of average mass fractions, with one average from each of two or more analytical methods [19,20]. The expanded uncertainty is the half width of a symmetric 95 % parametric bootstrap confidence interval [24], which is consistent with the ISO/JCGM Guide [22,23]. The effective coverage factor, $k = 2$.

(g) GC/MS (Ia) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with toluene:methanol mixture (100 °C with 13.8 MPa)

(h) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa)

(i) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM

(j) GC/MS (Ib) on 50 % phenyl-substituted methylpolysiloxane phase of selected extracts from GC/MS (Ia)

(k) GC/MS (IVc) on a smectic liquid crystalline phase of same extracts as GC/MS (IVa)

(l) GC/MS (V) on non-polar extra low bleed proprietary phase after PFE with toluene at one temperature and two pressures (200 °C with 13.8 MPa and 200 °C with 20.7 MPa)

Table 4. Non-Certified Mass Fraction Values for Selected Nitro-PAHs in SRM 2975

Extraction Conditions		Mass Fractions[a] (mg/kg)
PFE using temperatures between 100 °C and 200 °C		
	1-Nitronaphthalene[b]	0.044 ± 0.002
	2-Nitronaphthalene[b]	0.118 ± 0.005
PFE at 100 °C		
	9-Nitroanthracene[c]	3.07 ± 0.28
	8-Nitrofluoranthene[d]	0.60 ± 0.13
	4-Nitropyrene[d]	0.175 ± 0.016[e]
	6-Nitrobenzo[*a*]pyrene[f]	1.36 ± 0.40[e]
	1,3-Dinitropyrene[f]	1.12 ± 0.21
	1,6-Dinitropyrene[f]	2.35 ± 0.51[e]
	1,8-Dinitropyrene[f]	3.10 ± 0.95
PFE at 200 °C		
	9-Nitroanthracene[g]	3.94 ± 0.19[e]

[a] The non-certified values, unless otherwise footnoted, are weighted means of the mass fractions from two to six analytical methods [19]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [21] following the ISO/JCGM Guide [22,23].

[b] Two methods using GC/NCI-MS on a 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa) and four methods using GC/NCI-MS on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with toluene at two temperatures and two pressures (100 °C with 13.8 MPa; 100 °C with 20.7 MPa; 200 °C with 13.8 MPa; and 200 °C with 20.7 MPa)

[c] Three methods using GC/NCI-MS on either a 5 % phenyl-substituted or 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa) and two methods using GC/NCI-MS on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with toluene at one temperatures and two pressures (100 °C with 13.8 MPa and 100 °C with 20.7 MPa)

[d] Two methods using GC/NCI-MS on a 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa)

[e] The non-certified value is a weighted mean of average mass fractions, with one average from each of two or more analytical methods [19,20]. The expanded uncertainty is the half width of a symmetric 95 % parametric bootstrap confidence interval [24], which is consistent with the ISO/JCGM Guide [22,23] and the effective coverage factor, k, equals 2.

[f] Three methods using GC/NCI-MS on either a 5 % phenyl-substituted or 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM (100 °C with 13.8 MPa)

[g] Two methods using GC/NCI-MS on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with toluene at one temperature and two pressures (200 °C with 13.8 MPa and 200 °C with 20.7 MPa)

Table 5. Non-Certified Mass Fraction Value for Total Extractable Mass for SRM 2975

	Mass Fraction[a,b] (%)
Total Extractable Mass	2.7 ± 0.2

[a] Extractable mass as determined from Soxhlet extraction using DCM

[b] The results are expressed as the non-certified value ± the expanded uncertainty. The non-certified value for the total extractable mass is the mean value of six measurements. The uncertainty, computed according to the CIPM approach as described in the NIST and ISO/JCGM Guide [3,22,23], is an expanded uncertainty at the 95 % level of confidence with coverage factor, $k = 2$. The expanded uncertainty defines a range that contains the best estimate of the true value at a level of confidence of approximately 95 %.

Table 6. Non-Certified Values for Particle-Size Characteristics for SRM 2975

Particle Measurement	Value[(a)]
Mean diameter (volume distribution, MV, µm)[(b)]	31.9 ± 0.6
Mean diameter (area distribution, µm)[(c)]	11.2 ± 0.1
Mean diameter (number distribution, µm)[(d)]	1.62 ± 0.01
Surface Area (m^2/cm^3)[(e)]	0.538 ± 0.006

Percentile[(f)]	Particle Diameter[(a)] (µm)
95	110 ± 3
90	70 ± 2
80	44.9 ± 0.8
70	32.4 ± 0.6
60	24.8 ± 0.4
50[(g)]	19.4 ± 0.3
40	15.2 ± 0.2
30	11.7 ± 0.2
20	8.5 ± 0.1
10	5.3 ± 0.1

(a) Each non-certified value is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO/JCGM Guide [3,22,23], is an expanded uncertainty at the 95 % level of confidence with coverage factor, $k = 2$. The expanded uncertainty defines a range that contains the best estimate of the true value at a level of confidence of approximately 95 %.

(b) The mean diameter of the volume distribution represents the center of gravity of the distribution and compensates for scattering efficiency and refractive index. This parameter is strongly influenced by coarse particles.

(c) The mean diameter of the area distribution, calculated from the volume distribution with less influence from the presence of coarse particles than the MV parameter.

(d) The mean diameter of the number distribution calculated from the volume distribution.

(e) Calculated specific surface area assuming solid, spherical particles. This is a computation and should not be interchanged with an adsorption method of surface area determination (see Table 7) as this value does not reflect porosity or topographical characteristics.

(f) The data shown is the percent of the volume that is smaller than the indicated size.

(g) Median diameter (50 % of the volume is less than 19.4 µm)

Table 7. Non-Certified Value for Specific Surface Area of SRM 2975

Specific Surface Area (S)[(a)]	91 m^2/g

(a) Specific surface area determined by multi-point N_2 gas adsorption BET method.

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Certificate Revision History: 02 July 2021 (Change of expiration date; change of nitro-PAHs certified values to non-certified values due to incomplete stability testing from lack of appropriate calibrants; all other measurands previously labeled as reference or information values were converted to non-certified values; editorial changes); 03 December 2013 (Update certified and reference values for PAHs and nitro-PAHs to address the effect of temperature used for extraction; extension of certification period; editorial changes); 19 March 2009 (Addition of reference and information values and update of expiration date); 07 November 2000 (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.