National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2483

Single-Wall Carbon Nanotubes (Raw Soot)

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical and instrumental methods of analysis of carbon nanotubes. A unit of SRM 2483 consists of a bottle containing approximately 250 mg of nanotube soot.

Certified Mass Fraction Values: Certified mass fraction values, reported on a dry-mass basis [1,2], for barium (Ba), cerium (Ce), chlorine (Cl), cobalt (Co), dysprosium (Dy), europium (Eu), gadolinium (Gd), lanthanum (La), molybdenum (Mo), and samarium (Sm) are listed in Table 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 [3].

Reference Values: Reference mass fraction values are listed in Table 2 [2]. Additional reference values are listed in Tables 3 and 4, respectively. Reference values are noncertified values that are 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 [3].

Information Values: Information mass fraction and other values are provided in Table 5 [2] and Figures 1 through 5. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [3].

Expiration of Certification: The certification of **SRM 2483** is valid, within the measurement uncertainty specified, until **30 June 2021**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). However, 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.

Coordination of the technical measurements for certification was accomplished under the direction of J.A. Fagan of the NIST Polymers Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Analytical measurements for homogeneity testing and certification of this SRM were performed by J.A. Fagan of the NIST Polymers Division; R. Zeisler, R.L. Paul, R. Spatz, L.L. Yu, J. L. Mann, and W.R. Kelly of the NIST Analytical Chemistry Division; A.R. Hight Walker of the NIST Radiation and Biomolecular Physics Division, E. Mansfield, R. Geiss, and S. Hooker of the NIST Materials Reliability Division, and A. Vladar of the NIST Semiconductor and Dimensional Metrology Division.

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

Eric K. Lin, Chief
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Certificate Issue Date: 14 November 2011

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Certified Values

Certified values are reported below using instrumental neutron activation analysis (INAA), cold neutron Prompt Gamma Activation Analysis (PGAA), thermal neutron PGAA and inductively coupled plasma mass spectrometry (ICP-MS) [2].

Table 1. Certified Mass Fractions Values for SRM 2483 (Dry-Mass Basis)[a]

	Mass Fraction		Units
Ba[b,c]	119.0	± 3.4	mg/kg
Ce[b,c]	192.7	± 7.3	mg/kg
Cl[b,d]	0.2125	± 0.0089	%
Co[b,d]	0.963	± 0.017	%
Dy[b,c]	8.36	± 0.17	mg/kg
Eu[b,c]	2.27	± 0.13	mg/kg
Gd[c,d]	10.57	± 0.95	mg/kg
La[b,c]	104.0	± 4.0	mg/kg
Mo[b,d]	3.406	± 0.029	%
Sm[b,c,d]	13.09	± 0.90	mg/kg

[a] The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor $k = 2$ (approximately 95 % confidence), calculated by combining a between-method variance with a pooled, within-method variance following the ISO Guide [4-9].
[b] INAA.
[c] ICP-MS.
[d] PGAA.

Reference Values

Reference values for mass fractions of additional elements are presented in Table 2 [2]. Table 3 reports values measured for the material via thermogravimetry. Table 4 reports the measured average value for the G/D band ratio as measured by Raman scattering.

Table 2. Reference Mass Fractions Values for SRM 2483 (Dry-Mass Basis)[a]

	Mass Fraction		Units
Al[b]	723	± 19	mg/kg
Mg[b]	0.1150	± 0.0011	%
Mn[b]	4.482	± 0.041	mg/kg
Na[b]	0.1187	± 0.0036	%
Th[b,c]	25.7	± 4.4	mg/kg
V[b]	6.89	± 0.14	mg/kg
W[b,c]	7.50	± 1.22	mg/kg

[a] The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor $k = 2$ (approximately 95 % confidence), calculated by combining a between-method variance with a pooled, within-method variance following the ISO Guide [4-10].
[b] INAA.
[c] ICP-MS.

Table 3. Reference Thermogravimetry-Derived Values for SRM 2483[a]

	Value		Units
Oxidation Temperature	482.2	± 1.2	°C
Residual Mass[b]	7.09	± 0.33	%

[a] The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor $k = 2$, calculated by combining a between-method variance with a pooled, within-method variance following the ISO Guide [4-6,9-10].
[b] Dry-mass basis.

Table 4. Reference G/D Raman Peak Ratio for 514.5 nm Excitation for SRM 2483[a]

	Value
G/D Ratio	17.2 ± 1.0

[a] The G/D reference value is the mean of 57 ratios from one technique. Literature reports [11] suggest that approximately 30+ measurements are necessary to ensure that an average G/D value accurately describes a bulk sample. The expanded uncertainty is calculated as $U = k$ (standard deviation divided by square root of 57) for $k = 2$, two standard deviations of the mean. The standard deviation of the 57 ratios is 3.7.
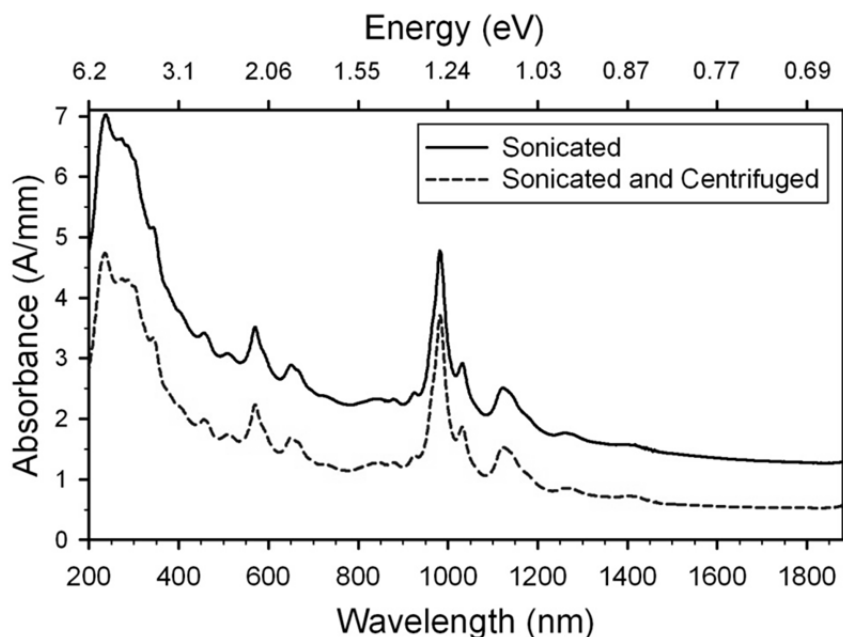
Informational Values
Additional measurements and data were obtained to further characterize the material and are provided as information values. Measured concentrations for additional elements are reported in Table 5 [2]. Optical absorbance spectra covering the ultraviolet, visible and near-infrared (UV-VIS-NIR) spectrum are provided in Figure 1. A NIR fluorescence excitation-emission plot is given in Figure 2. Raman spectra for excitation at 514.5 nm, 532.1 nm and 632.8 nm are presented in Figure 3. Scanning electron microscopy images are provided in Figure 4, and a transmission electron microscopy (TEM) in Figure 5.

Table 5. Information Mass Fractions Values for SRM 2483 (Dry-Mass Basis)

|	Mass Fraction	Units
As[a]	12.5	mg/kg
B[b]	74.7	mg/kg
C[b]	94.6	%
Ca[a]	0.303	%
Cu[a]	186	mg/kg
H[b]	0.38	%

[a] INAA.
[b] PGAA.

Figure 1. Informational UV-VIS-NIR absorbance spectra for SRM 2483 dispersed at a concentration of 1 mg/mL in a 2 % mass/volume solution of sodium deoxycholate (black solid line, see methods). The spectrum of the surfactant solution has been subtracted from each of the spectra. As measured at 775 nm, the supernatant retains approximately half of the initial absorbance after centrifugation-based processing to remove non-nanotube components and poorly dispersed material. For both the dispersed suspension of nanotubes and the centrifuged dispersion (dashed line), multiple peak features are clearly identifiable to individual nanotube species, defined by the chiral vector indices (n,m) that describe the orientation of the tubular graphitic structure and the nanotube diameter. Dominant peak features are due to the (6,5) nanotube at 343 nm, 568 nm, and 982 nm. Other distinct peaks are identifiable to the semiconducting (7,5), (7,6), (8,3), (8,4), (9,1) and the metallic (6,6) and (7,4) nanotubes. Other species are also present but are less readily apparent.
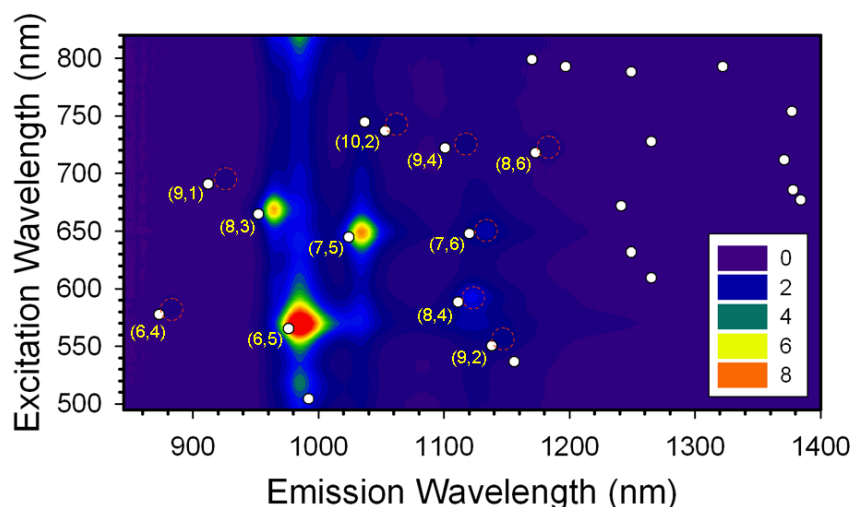

Figure 2. Informational NIR fluorescence spectrum for SRM 2483 dispersed in sodium deoxycholate solution through sonication and then centrifuged for purification (see methods used for absorbance measurements). Prior to the fluorescence measurement the nanotube dispersion was diluted to an absorbance of 0.12 absorbance units per cm at 775 nm into 1 % sodium deoxycholate/D_2O solution. The emission intensity has been corrected for the excitation intensity and emission side wavelength-dependent sensitivity. Dominant peak features are emission from the (6,5), (7,5), and (8,3) chiralities. The (6,4), (7,6), (8,4), (9,1), (9,2), (9,4), (10,2), and (8,6) are also present in sufficient quantity to be easily detected (red dashed circles with labeled chiral indices denote observed minor peaks). Other chiralities are likely present, but in quantities too small to display fluorescence on this intensity scale (the intensity is normalized by the (6,5) emission peak divided by ten). For reference, empirical excitation-emission peak locations for different Single-Wall Carbon Nanotube (SWCNT) structures, as assigned in the literature [12], are noted by white dots on the figure. The diameter range of the clearly observed species is from 0.692 nm for the (6,4) chirality, to 0.966 nm for the (8,6) chirality. Note: the NIR fluorescence measurement technique is only sensitive to semiconducting nanotube chiralities.
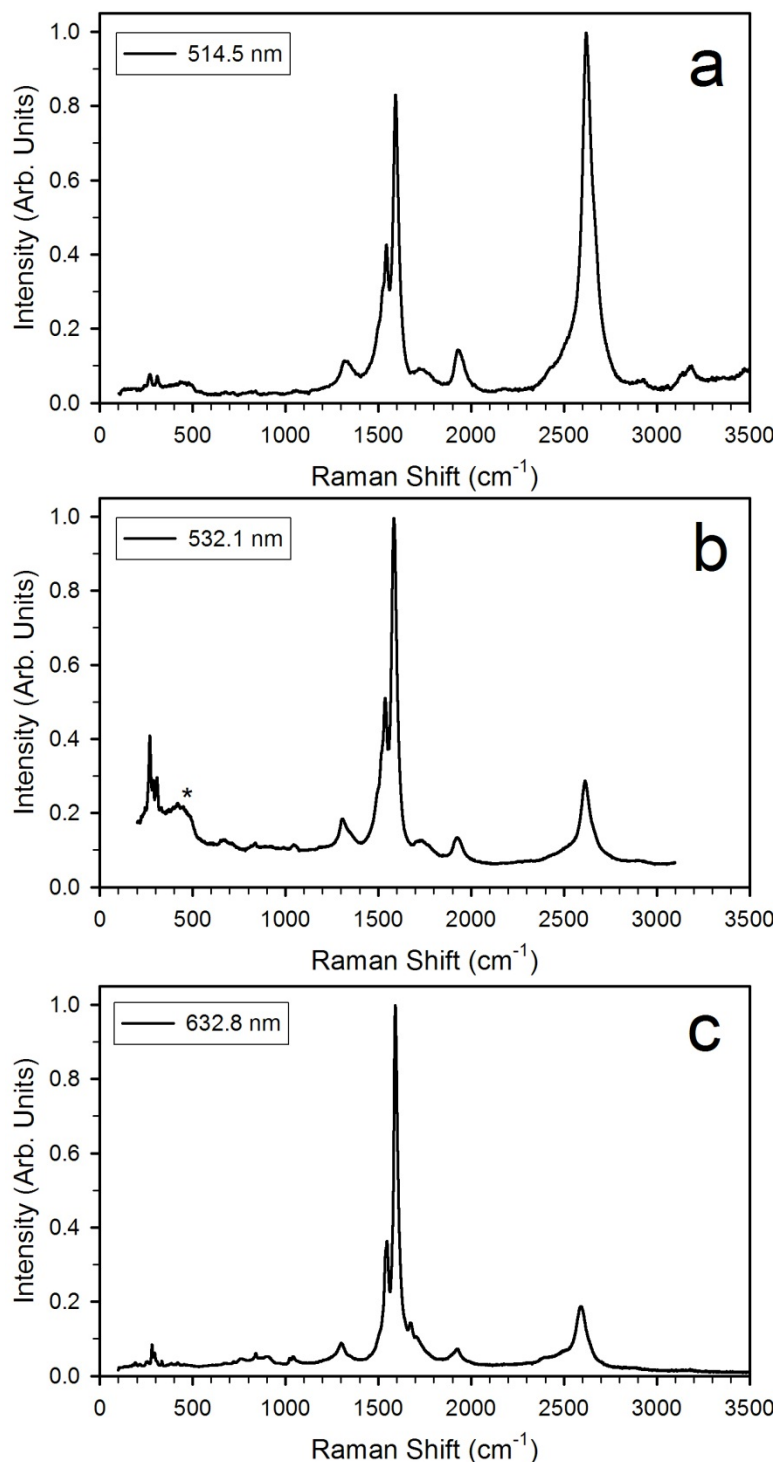
Figure 3. Informational Raman scattering spectra for SRM 2483 with excitation at 514.5 nm, 532.1 nm, and 632.8 nm. Separately collected reference spectra (from an empty vial) for each excitation wavelength have been subtracted from each of the presented spectra to better represent the inherent Raman spectra of the nanotube powder. Multiple peak features corresponding to different SWCNT Raman features, including the radial breathing modes (RBM), D-band, G-band, M-band; iTOLA band, and G′-band (2D), are visible. The starred feature in Figure 3B is an artifact from the imperfect subtraction of the empty vial signal.
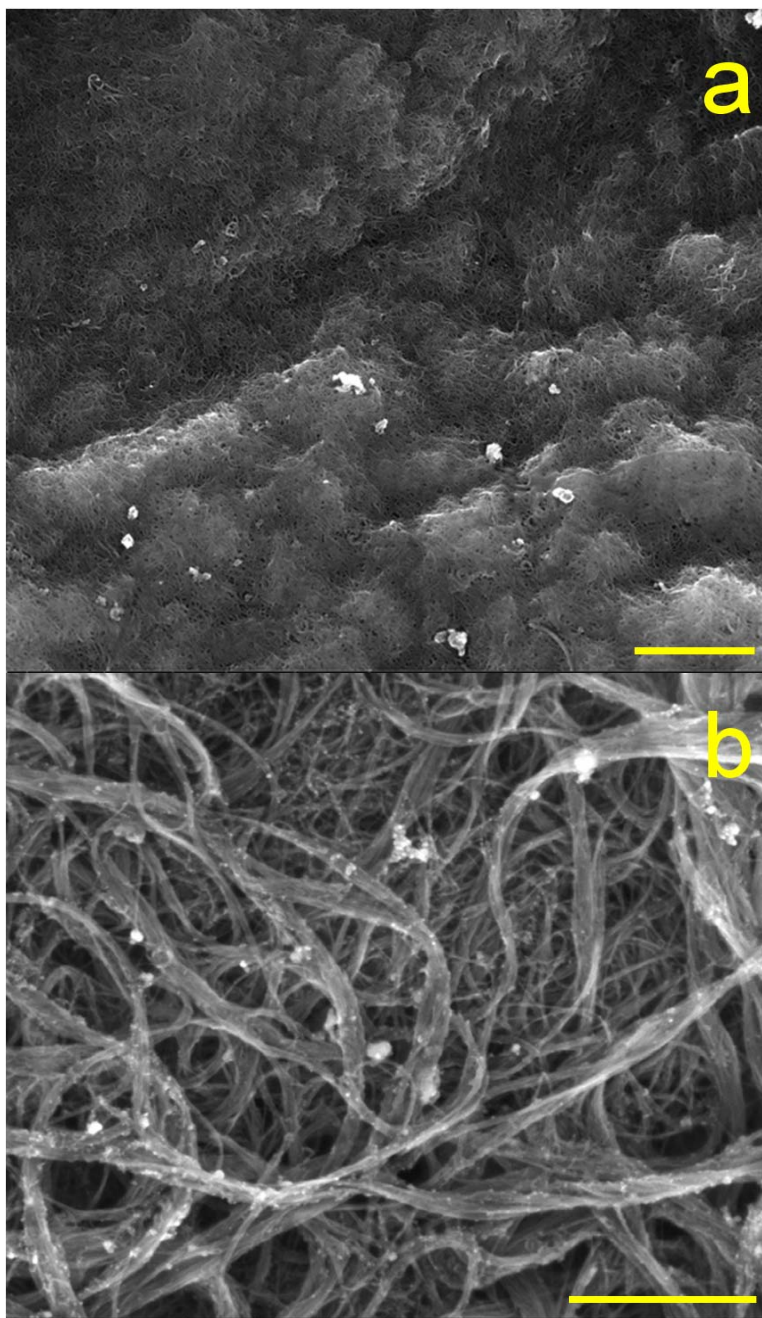
Figure 4. Informational Scanning Electron Microscopy (SEM) images of SRM 2483. The top image (a) is an SEM micrograph of SRM 2483 at low magnification. The underlying roped structure of the nanotube soot is visible, as are heterogeneous (on this scale) impurities. The horizontal field of view is 6.4 μm (scale bar indicates 1 μm). The lower image (b) is a higher magnification micrograph of SRM 2483. The morphology of the nanotubes in the soot is primarily that of large bundles intertwined with smaller bundles, and in homogeneously covered with residual catalyst materials. Liquid preparation was used for this sample. The horizontal field of view is 1.02 μm (scale bar indicates 250 nm).
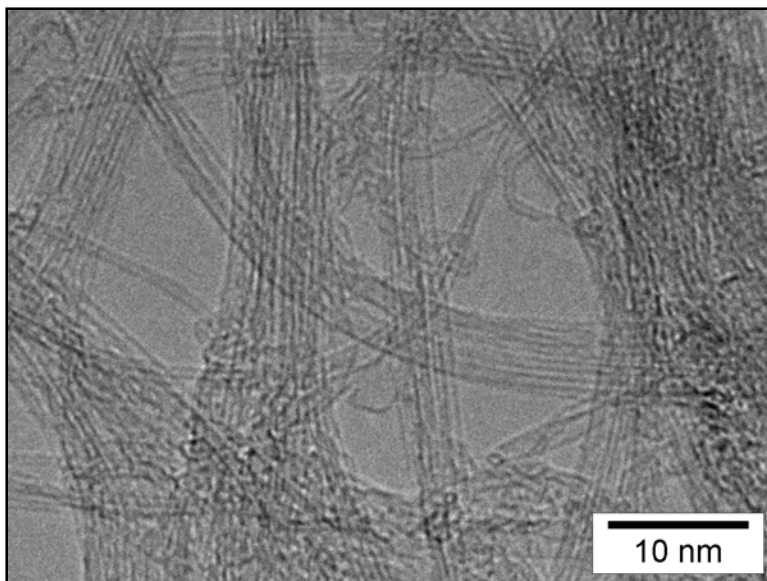
Figure 5. Informational Transmission Electron Microscopy (TEM) micrograph of soot from SRM 2483 deposited onto a TEM grid from chloroform suspension. The cylindrical nanotubes visible in the image are approximately 0.8 nm in diameter [13].

NOTICE AND WARNING TO USERS

This material should be handled as recommended by the National Institute for Occupational Safety and Health (NIOSH). According to NIOSH, currently there are no studies reported in the literature of adverse health effects in workers producing or using carbon nanotubes or carbon nanofibers. The concern about worker exposure to these materials arises from results of animal studies. Several studies in rodents have shown an equal or greater potency of carbon nanotubes compared to other inhaled particles known to be hazardous to exposed workers (ultrafine carbon black, crystalline silica, and asbestos) in causing adverse lung effects including pulmonary inflammation and fibrosis [14].

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Until required for use, the SRM should be **stored at room temperature** in its original bottle and package, and protected from intense direct light or ultraviolet radiation.

To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum sample quantity of 25 mg to 40 mg is recommended. The soot does not require preparation prior to weighing; however, dehydrating the sample in a desiccator over anhydrous $Mg(ClO_4)_2$ for a period of 8 to 10 days before sampling will minimize variation due to absorbed water content.

Measurements on smaller sample quantities may reveal microheterogeneity of the material. This particularly applies to microscopy measurement methods or optical measurement methods, such as Raman scattering, that sample small focal volumes and thus measure small quantities of material. In both cases, effects from local morphology may influence results on small sample quantities. See the information in the "Homogeneity Assessment" section. Methods were used or adapted from those reported in [15].

PREPARATION AND ANALYSIS[1]

Material Preparation: The material for SRM 2483 was procured from Southwest Nanotechnologies (Norman, OK, USA). Twenty pre-blended containers of 10 g each of SG65 Lot 000-0024 Single-Wall Carbon Nanotubes (SWCNT) powder were received and combined at NIST to form the material for SRM 2483. This material was further blended via mechanical agitation after delivery, and was bottled from the homogenized lot at NIST.

Homogeneity Assessment: The homogeneity of the nanotube soot was tested by multiple techniques to probe both elemental composition homogeneity and structural homogeneity of the nanocarbon fraction. The results obtained by INAA were used to assess the homogeneity of the elemental mass fractions among the test portions. Graphical assay and one-way fixed and random effects analyses of variance (ANOVA) were used to test for significance of run sequence and vial effects. For Co, Dy, La, Mo, and Sm there is a modest component of heterogeneity attributable to vials. Interestingly, the mass fractions of several of the rare earth elements, in particular Sm and La, are strongly correlated with the Mo mass fractions within the samples, whereas other elements only display weak correlation. This could be due to different sources of the elements in the synthesis and purification of the candidate SRM, selective incorporation of Sm and La in the Mo rich catalyst particles, or selective removal of species during purification of the material by the manufacturer. To estimate the magnitude of heterogeneity in the elemental mass fractions, one can use the relative standard deviation due to sampling error. Comparing one significant uncertainty component of the INAA procedure, namely the relative counting uncertainty with the relative standard deviation, the data indicate that there is a sampling error of about 3 % with 25 mg to 40 mg sample masses [2].

Using Raman spectroscopy data, the homogeneity of the bottled soot for nanotube content and morphology was evaluated by both fixed and random effects one-way analyses of variance on the measured Raman (G_{peak}/D_{peak}) ratios. Analyses were performed for three different methodologies of defining G/D from the raw measurements. Both fixed and random analyses for each of the three methodologies strongly support the null hypothesis of no bottle effect on the ratios. For the G_{peak}/D_{peak} methodology, the P-value was 0.60, and the bottle component of variance was effectively zero.

Value Assignment and Uncertainty Analysis: Analyses to establish reference values were conducted at NIST using best practices as determined independently for each measurement method.

METHODS FOR CERTIFIED VALUE MEASUREMENTS

All elements for which certified and reference values are provided were determined by using at least one of the following methods carried out at NIST: INAA, prompt gamma activation analysis (PGAA), and inductively coupled plasma mass spectrometry (ICP-MS). Mass fraction values are reported on a dry-mass basis. Moisture loss was determined by desiccator drying for eight or more days over anhydrous magnesium-perchlorate.

Instrumental Neutron Activation Analysis
INAA was carried out by the comparator method with known standard samples irradiated and measured under the same conditions as the test portions prepared from the candidate SRM material. Test portions were prepared in duplicate from 22 vials by forming pellets in a 7 mm diameter die of 25 mg to 40 mg from either dried or from material as received. These pellets were immediately weighed and sealed in polypropylene bags for irradiation. Corrections for moisture content were applied to the sample masses as appropriate. The INAA procedure was conducted in a sequence of irradiation and counting steps. A short-time irradiation of individual samples was followed by two counts for elements with short-lived nuclides; this was followed by a long-term irradiation of sample sets and the counts for elements with intermediate and long-lived nuclides [16].

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[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.

Prompt Gamma Activation Analysis
Measurements were performed using both the thermal neutron [17] and cold neutron [18] PGAA spectrometers at the NIST Center for Neutron Research (NCNR). Test portions were prepared from the candidate SRM material by pelletizing approximately 100 mg in a 13 mm die and subsequently weighing and sealing them in bags made from Teflon film. Dried samples were irradiated under vacuum; as-received samples were irradiated in air for 24 h. Appropriate comparator standards were irradiated for shorter periods until counting uncertainty of less than 1 % relative was achieved.

Inductively Coupled Plasma Mass Spectrometry
Test portions of 25 mg to 160 mg of untreated SRM material were digested with the NIST Carius tube method [19]. For measurement each digest was diluted with deionized water to approximately 50 g, and the mass was accurately determined. An aliquot of approximately 2 g of this digest was diluted to approximately 50 g with 1.5 % (volume fraction) HNO_3. Exact masses were determined on a calibrated analytical balance. An ICP-MS equipped with a Peltier-cooled, inert sample introduction system was used for the determination of barium and transition elements in the digests.

METHODS FOR REFERENCE VALUE MEASUREMENTS

Thermogravimetry
Samples of soot were measured during heating in an air environment until the combustible material was consumed. Sample pans were equilibrated to 40 °C before heating. The heating rate was 10 °C/min to 800 °C. Air was introduced at 25 mL/min. The rate of mass loss during this process and the amount of residual mass at 635 °C were recorded. The oxidation temperature is defined as the temperature at which the peak mass loss rate occurs. Data are reported using the mass at 100 °C as the original starting mass (dry-mass basis) to compensate for small variations in the initial water content.

Raman Spectroscopy
Raman scattering measurements were performed for the SRM 2483 using a triple-grating spectrometer with a liquid nitrogen-cooled CCD detector; the excitation source was a 514.5 nm Ar^+ laser with a spot size of roughly 10 µm to 100 µm in diameter. A Raman frequency shift uncertainty of 0.4 cm^{-1} was confirmed by comparison to a spectrum measured for cyclohexane following ASTM E1840-96 [20]. Measurements were made on three different spots in multiple glass vials of soot sampled from bottles of SRM 2483. Each sample was measured over the Raman shift region from 1125 cm^{-1} to 1875 cm^{-1}. Values for the intensities of two Raman features for carbon nanotube materials, the G-band and the D-band, were calculated using several methodologies to define the peak size above the background scattering.

The methodology for defining the ratio G_{peak}/D_{peak} reported in Table 4 is as follows:
1) A baseline for the D-band is defined as the line that maximizes the area under the measured D-band, with the two constraints that the minimum difference (intensity minus fit intensity) between the moving average of the measured scattering value measured over the range 1250 cm^{-1} to 1300 cm^{-1} and 1350 cm^{-1} to 1450 cm^{-1} is zero in each range.
2) The linear fit for the D-band baseline is subtracted from the data, and the peak D-band value (D_{peak}) is assigned as the greatest ordinate value in the range from 1300 cm^{-1} to 1380 cm^{-1}.
3) The peak G value (G_{peak}) is defined as the greatest ordinate scattering value recorded over the 1500 cm^{-1} to 1700 cm^{-1} range minus the average value over the Raman shift range from 1127.1 cm^{-1} to 1135.4 cm^{-1}.
4) The ratio is defined as G_{peak} value divided by D_{peak} value.

METHODS FOR INFORMATION VALUE MEASUREMENTS

Ultraviolet-Visible-Near Infrared Absorbance Spectroscopy: Nanotubes from SRM 2483 were dispersed into 2.0 % (mass/volume) sodium deoxycholate solution via sonication (0.635 cm probe, 1 h, 0.9 W/mL); a portion of the sonication suspension was later centrifuged for 2 h at 1884 rad/s and 10 °C (Beckman JA-20 rotor), and the supernatant was collected. The spectrum of the sonicated suspension of nanotubes is representative of the material without any applied purification. The spectrum of the supernatant collected after centrifugation is provided as centrifugation for purification is a common technique that more clearly reveals the locations of the optical transitions. The absorbance spectra were recorded in transmission mode on a dual beam spectrophotometer without the use of an integrating sphere. All samples were measure in a single 1 mm quartz cuvette. The combined absorbance of the surfactant solution and cuvette were measured independently and subtracted from the raw spectra recorded for the sonicated nanotube suspension and the centrifuged nanotube dispersion during data analysis.

NIR Fluorescence

The presented excitation-emission contour plot was measured for the centrifuged nanotube supernatant produced for the absorbance measurements described above. Prior to the fluorescence measurement, the sample was diluted to an absorbance of 0.12 absorbance units per cm at 775 nm with 1.0 % (mass/volume) sodium deoxycholate solution in D_2O. The sample was measured in a 5 mm by 5 mm quartz cuvette on a spectrofluorimeter with an InGaAs array detector. The reported spectra are corrected for the emission train wavelength-dependent sensitivity and the wavelength-dependent excitation power, but not for the in-filter effects of absorbance or Rayleigh scattering within the cuvette volume. Observation of specific peak features is an indication that specific semiconducting nanotubes are present in the sample, although the direct correlation between intensity and concentration is not fully established. The technique is not sensitive to metallic or heavily chemically functionalized nanotubes.

Raman Spectroscopy

Raman spectroscopy measurements were performed at 514.5 nm, 532.1 nm, and 632.8 nm on samples of SRM 2483 in glass vials. Data collection parameters varied with the excitation wavelength. Separately collected reference spectra (from an empty vial under the same collection conditions) for each excitation wavelength have been subtracted from each of the presented spectra to better represent the inherent Raman spectra of the nanotube powder. Note that variation may occur in the ratio of spectral features from these informational spectra due to microheterogeneity of the material in the small volumes typically sampled during Raman measurements.

Scanning Electron Microscopy

Images of SRM 2483 were acquired using SEM. Dry powder was selected using a needle or sharp tweezers and gently smeared across the SEM sample holder. A drop of isopropyl alcohol was used in some cases to improve adherence. Loose remaining material was removed by gently knocking the holder against a hard surface. For images with liquid preparation, a portion of the powder was selected and added to 10 mL of isopropyl alcohol and placed in an ultrasonic bath for 5 min to 30 min. A drop of material was then placed onto a TEM grid and allowed to dry before knocking to remove loose material and measurement. A FEI Helios dual-beam SEM-based measuring system and a high-resolution SEM were used for the reported images.

Transmission Electron Microscopy

Dry SRM 2483 powder was added to chloroform in a 2 mL glass vial, and was sonicated in a bath sonicator for 15 minutes to form a suspension. After sonication, this suspension was immediately deposited onto a thin holey carbon TEM grid using a micropipette to minimize any effect of differential settling of the material. The drop was then allowed to dry completely overnight. A single-tilt sample holder was used for TEM observation. Images were taken at 200 keV under low-dose operating conditions in the TEM. Low dose means that the electron flux onto the samples was restricted with a small condenser aperture (typically 40 micrometers in diameter) and underfocusing the illumination to the minimum that would allow an image to be captured with a 2 s exposure. All images were captured with a one megapixel CCD camera located below the viewing chamber.

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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) 926-4751; email srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.