National Institute of Standards & Technology

Certificate

Standard Reference Material® 2035a

Ultraviolet-Visible-Near-Infrared Wavelength/Wavenumber Transmission Standard

This Standard Reference Material (SRM) is a certified transfer standard intended for the verification and calibration of the wavenumber/wavelength scale of ultraviolet-visible (UV-Vis) and near infrared (NIR) spectrometers operating in transmission mode. A unit of SRM 2035a consists of an optical filter, 25 mm in diameter and 1.5 mm thick, that should be handled in its original optical mount and stored in the wooden container provided.

SRM 2035a is a glass filter consisting of mole fractions of 3.00 % holmium oxide (Ho_2O_3), 1.30 % samarium oxide (Sm_2O_3), 0.68 % ytterbium oxide (Yb_2O_3), and 0.47 % neodymium oxide (Nd_2O_3) in a matrix containing lanthanum oxide (La_2O_3), boron oxide (B_2O_3), silicon dioxide (SiO_2), and zirconium dioxide (ZrO_2).

Certification: 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. In this certificate, spectral features are referred to as *bands* if their location is determined by the center-of-gravity (COG) or centroid algorithm, whereas those determined by a five point cubic polynomial fit are referred to as *peak* locations. SRM 2035a is certified for the 10 % band fraction centroid of seven bands spanning the spectral region from 10 300 cm^{-1} to 5 100 cm^{-1} at 8 cm^{-1} resolution (vacuum wavenumber). The same seven bands are certified for the 10 % band fraction centroid location in the spectral region from 971 nm to 1960 nm at 1 nm and 3 nm spectral bandwidths (air wavelength). The certified absorption band locations for these seven bands are given in Table 1 for vacuum wavenumber and Table 2 for air wavelength. SRM 2035a is certified for thirteen additional peak locations in the spectral region from 334 nm to 805 nm at 1 nm and 3 nm spectral bandwidths. The certified transmittance peak locations for these thirteen peaks are given in Table 3 for air wavelength.

Expiration of Certification: The certification of **SRM 2035a** is valid, within the measurement uncertainties specified, until **01 January 2022**, 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) will facilitate notification.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were provided by S.J. Choquette of the NIST Biosystems and Biomaterials Division.

The production and certification of SRM 2035a was conducted by S.J. Choquette and A.A. Urbas of the NIST Biosystems and Biomaterials Division. Additional measurements were supplied by L. Hanssen and C. Cooksey of the NIST Sensor Science Division.

Statistical consultation was provided by S.D. Leigh and H.-K. Liu of the NIST Statistical Engineering Division.

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

Anne Plant, Chief
Biosystems and Biomaterials Division

Gaithersburg, MD 20899
Certificate Issue Date: 06 March 2014
Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling and Storage: To maintain the integrity of SRM 2035a, the filter should only be handled in its optical mount. While not in use, the SRM should be stored in the original wooden container provided or one with similar or better mechanical protection.

Use: Carefully insert SRM 2035a into the sample beam of the spectrometer being tested. Measurements under a dry (air or nitrogen) purge are highly recommended for NIR measurements. If a dry purge is not available, the locations of band 1 and band 3 may differ significantly from the certified values. Acquire the absorbance/transmittance spectrum, referenced to air at a nominal temperature between 20 °C and 26 °C. Compare measured band or peak locations to its certified value listed in the appropriate table (Tables 1, 2 and 3). Band locations in Table 1 (NIR) are vacuum wavenumber values at constant 8 cm^{-1} resolution. Investigations have shown that these band locations remain within the stated uncertainties for resolutions higher than 8 cm^{-1} (e.g., 4 cm^{-1}, 2 cm^{-1}, etc.); however, they are not certified for use with resolutions lower than 8 cm^{-1}. Band locations in Table 2 (NIR) are air wavelength values at spectral bandwidths of 1 nm and 3 nm. Peak locations in Table 3 (UV-Vis) are air wavelength values at spectral bandwidths of 1 nm and 3 nm. Comparison should be made to the spectral bandwidth more representative of the spectrometer being used. Taking into account the certification uncertainty of each band of SRM 2035a, any statistically significant differences between the measured and certified band locations may be used to recalibrate the spectrometer wavenumber/wavelength scale. For air wavelength measurements, a data point spacing equal to or less than 0.5 nm is recommended. For vacuum wavenumber measurements, a data spacing equal to or less than 1.0 cm^{-1} is recommended.

PREPARATION AND ANALYSIS[(1)]

Preparation: The SRM glass composition was melted at Schott Glass, Duryea, PA. The finished SRM filters were supplied to NIST by Avian Technologies, Sunapee, NH.

Measurement Conditions: Certification measurements for NIR vacuum wavenumber band locations were performed on Bruker Vertex-70, Bruker IFS-66, Bruker IFS-66v, and Bomem DA3 FT spectrometers. The Bruker Vertex-70, Bruker IFS-66, and Bruker IFS-66v FT spectrometers were calibrated in vacuum wavenumber units using an acetylene gas cell or ambient water vapor bands. The Bomem DA3 FT spectrometer was calibrated using ambient water vapor and carbon dioxide bands. Certification measurements for NIR air wavelength band locations were performed on PerkinElmer Lambda 900, PerkinElmer Lambda 1050, Cary 6000i dispersive spectrometers, and a Bruker Vertex-70 FT spectrometer. The air wavelength axis of the PerkinElmer Lambda 900 was calibrated using emission lines of Kr, Ar, and Ne atomic emission pen lamps in first and second grating order. The air wavelength axis of the PerkinElmer Lambda 1050 was calibrated per the instrument instructions using the emission lines of the internal D_2 source in the second grating order. The air wavelength axis of the Cary 6000i was calibrated using emission lines of Hg and Ar from an internal atomic emission calibration lamp. The Bruker Vertex-70 FT was calibrated in air wavelength by applying the appropriate correction for the refractive index of air [1] to the calibrated vacuum wavenumber axis. Certification measurements for UV-Vis air wavelength were performed on PerkinElmer Lambda 900 and Cary 6000i commercial dispersive spectrometers, the NIST Materials Measurement Laboratory High Accuracy Spectrophotometer (HASII), and an additional in-house constructed spectrometer system. The air wavelength axes of all spectrometers were calibrated using lines from atomic emission calibration lamps. The PerkinElmer Lambda 900 and HASII were calibrated using Hg and Kr emission lines. The Cary 6000i was calibrated using Hg and Ar emission lines. The in-house constructed spectrometer was calibrated using Hg, Ar and Kr emission lines.

Wavenumber and Wavelength Band/Peak Location Methodology: The method used to determine the certified NIR wavenumber (υ) and wavelength (λ) band locations of SRM 2035a is the center-of-gravity (COG) technique [2–5]. *If another technique is used, a comparison with the certified values will not be valid*. In this certificate, positions determined with the centroid algorithm are referred to as *band* locations. Values listed in Tables 1 and 2 are band locations. For SRM 2035a COG calculations, 10 % fractions of the bands were used for both wavenumber and wavelength data. Further information on the use of this algorithm with other NIST SRMs can be found in References [6–8]. The method used to determine the certified UV-Vis wavelength (λ) peak locations of SRM 2035a was a cubic polynomial fit to five points spanning the transmittance peak minimum. Values listed in Table 3 are peak locations.

[(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.

Certified Values: 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 [9]. The certified vacuum wavenumber locations for the seven absorption bands spanning the range from 10 300 cm^{-1} to 5 100 cm^{-1} are listed in Table 1. These values were obtained at 8 cm^{-1} constant wavenumber resolution and are certified for operation between 20 °C and 26 °C. The certified values represent the consensus mean band locations from measurements on four Fourier-transform (FT) spectrometer systems. The NIR absorbance spectrum of SRM 2035a in vacuum wavenumber is shown in Figure 1. Atmospheric water vapor is a significant source of variation for band 3, and this band should be used with caution when calibrating commercial spectrometers with SRM 2035a.

The certified values for the NIR air wavelength band locations for the seven absorption bands from 971 nm to 1960 nm for 1 nm and 3 nm spectral slit width (SSW) resolutions are listed in Table 2 and are certified for operation between 20 °C and 26 °C. The certified values of the air wavelength band locations represent the mean band locations from three scanning dispersive spectrometer systems and one FT spectrometer system. The NIR absorbance spectrum of SRM 2035a in air wavelength is illustrated in Figure 2.

The certified values for the UV-Vis air wavelength peak locations for the thirteen peaks from 334 nm to 805 nm for 1 nm and 3 nm spectral slit width (SSW) resolutions are listed in Table 3 and are certified for operation between 20 °C and 26 °C. The certified values of the air wavelength peak locations represent the mean band locations from four scanning dispersive spectrometer systems. The UV-Vis transmittance spectrum of SRM 2035a in air wavelength is illustrated in Figure 3.

Certification Uncertainty: The expanded uncertainties ($U_{95\%}$) for the wavenumber and wavelength band and peak locations given in Tables 1, 2 and 3 are determined from the appropriate combination of instrument-specific standard uncertainties to define the interval within which the unknown value of the band/peak can be asserted to lie with a level of confidence of approximately 95 % [10–13]. Components of the uncertainty for the wavenumber and wavelength band and peak locations include: within-instrument variation, between-instrument bias, calibration of the NIST FT and scanning dispersive spectrometer systems, location shifts due to temperature, water vapor interference, and short-term precision variance (i.e., estimated standard deviations). For the NIR wavenumber and wavelength band locations, the Type A uncertainty components were determined independently for each band and the combined, band specific uncertainties were expanded by a coverage factor ($k = 3.184$) based on the Student's t-distribution and number of spectrometers contributing to the certification. For the UV-Vis peak locations, a pooled uncertainty across all peaks was estimated for the Type A component and the combined overall uncertainty was expanded by a coverage factor ($k = 2$).

Table 1. Certified Values for NIR Band Locations[a] and Uncertainties[b] for Vacuum Wavenumber at 8 cm^{-1} Resolution.

Band	Band Location (cm^{-1})		
1	5138.91	±	0.35
2	6805.70	±	0.81
3	7314.06	±	0.33
4	8179.61	±	0.73
5	8682.42	±	0.14
6	9294.56	±	0.30
7	10245.59	±	0.16

[a] Band locations determined using a centroid method with a band fraction of 0.1; see Figure 1 for band identification.

[b] The listed uncertainties represent the expanded uncertainty ($U_{95\%}$) calculation ($k = 3.184$) in accordance with reference 10. The certified values represent the consensus mean band locations from measurements on four Fourier transform (FT) spectrometer systems and the measurand is the band location at 8 cm^{-1} resolution. The certified values are metrologically traceable to the SI unit of reciprocal meters (m^{-1}), expressed as reciprocal centimeters (cm^{-1}).
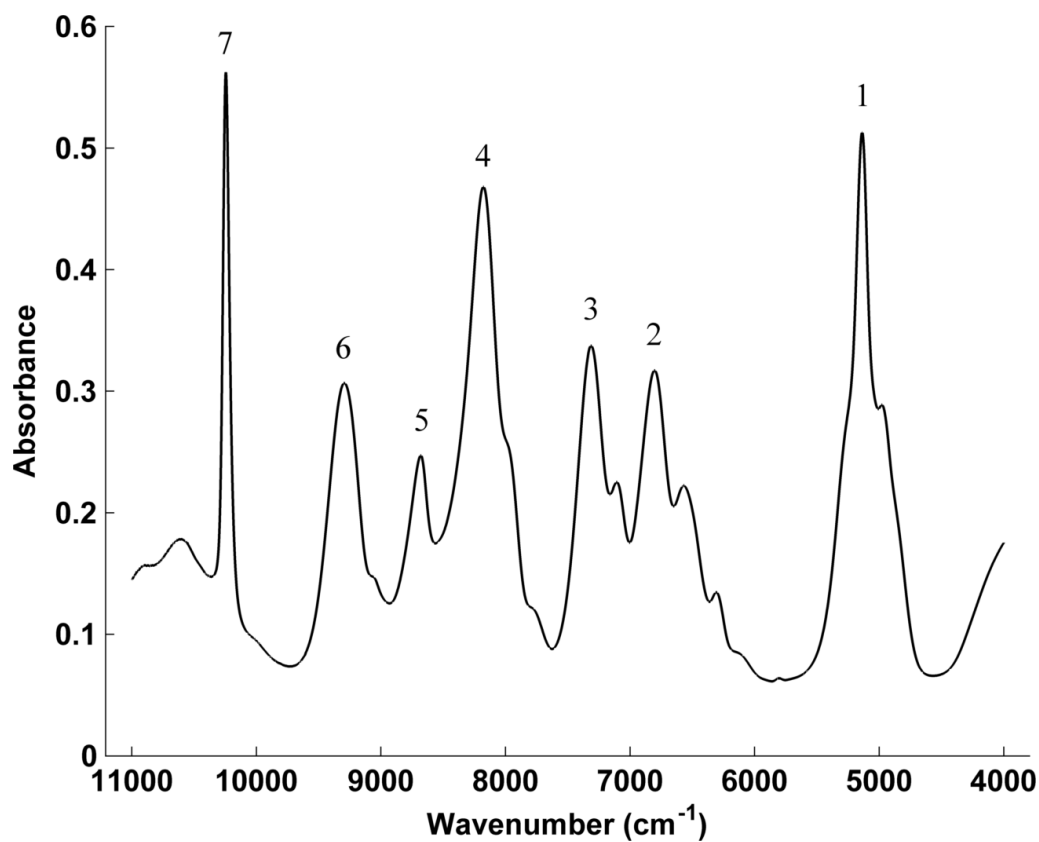

Figure 1. NIR Absorbance Spectrum of SRM 2035a in Vacuum Wavenumber with Band Locations Indicated.

Table 2. Certified Values for NIR Band Locations[a] and Uncertainties[b] for Air Wavelength.

Band	1 nm SSW Band Location (nm)			3 nm SSW Band Location (nm)		
1	1945.7	±	0.4	1945.7	±	0.4
2	1469.1	±	0.2	1469.0	±	0.2
3	1366.8	±	0.1	1366.8	±	0.1
4	1222.2	±	0.2	1222.2	±	0.2
5	1151.5	±	0.1	1151.4	±	0.1
6	1075.6	±	0.1	1075.6	±	0.1
7	975.8	±	0.1	975.9	±	0.1

[a] Band locations determined using a centroid method with a band fraction of 0.1; see Figure 2 for band identification.

[b] The listed uncertainties represent the expanded uncertainty ($U_{95\%}$) calculation ($k = 3.184$) in accordance with reference 10. The certified values represent the mean band locations from three scanning dispersive spectrometer systems and one FT spectrometer system and the measurand is the band location at 1 nm and 3 nm SSW. The certified values are metrologically traceable to the SI unit of meters, expressed as nanometers.
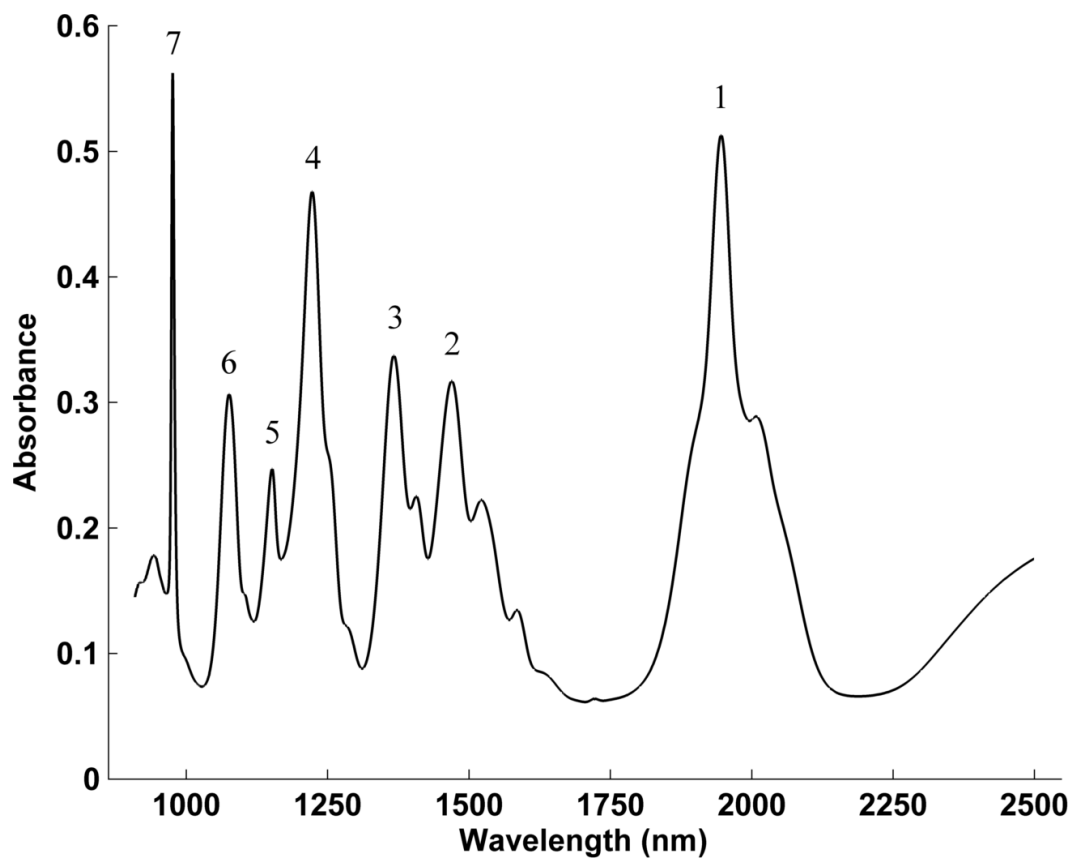

Figure 2. NIR Absorbance Spectrum of SRM 2035a in Air Wavelength with Band Locations Indicated.

Table 3. Certified Values for UV-Vis Peak Locations[a] and Uncertainties[b] for Air Wavelength.

Peak	1 nm SSW Peak Location (nm)			3 nm SSW Peak Location (nm)		
8	804.2	±	0.1	804.1	±	0.1
9	747.7	±	0.1	747.7	±	0.1
10	642.5	±	0.1	642.5	±	0.1
11	583.4	±	0.1	583.4	±	0.1
12	537.7	±	0.1	537.9	±	0.1
13	485.6	±	0.1	485.4	±	0.1
14	417.9	±	0.1	418.0	±	0.1
15	402.6	±	0.1	402.4	±	0.1
16	386.2	±	0.1	386.1	±	0.1
17	374.7	±	0.1	374.5	±	0.1
18	360.8	±	0.1	361.3	±	0.1
19	345.3	±	0.1	345.6	±	0.1
20	334.6	±	0.1	334.3	±	0.1

[a] Peak locations determined using a five-point cubic polynomial fit; see Figure 3 for band identification.

[b] The listed uncertainties represent the expanded uncertainty ($U_{95\%}$) calculation ($k = 2$) in accordance with reference 10. For the Type A component, a pooled uncertainty was estimated across all peaks. The certified values represent the mean band locations from four scanning dispersive spectrometer systems and the measurand is the band location at 1 nm and 3 nm SSW. The certified values are metrologically traceable to the SI unit of meters, expressed as nanometers.
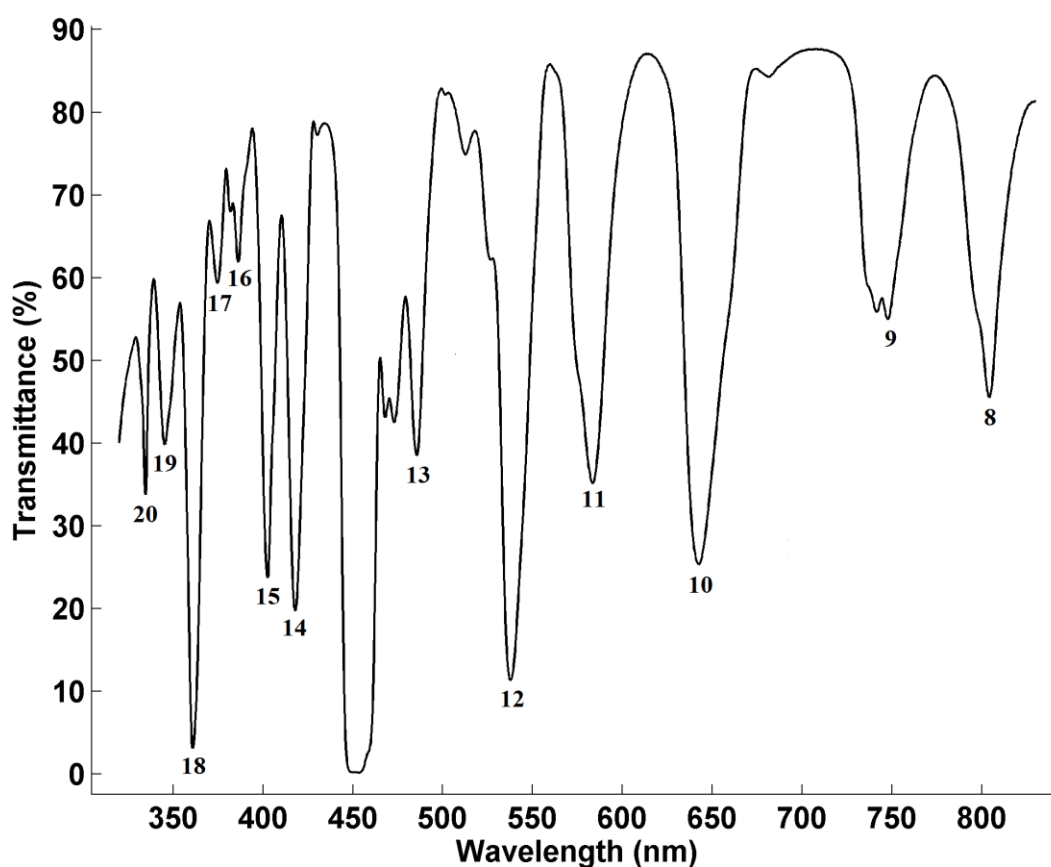

Figure 3. UV-Vis Absorbance Spectrum of SRM 2035a in Air Wavelength with Peak Locations Indicated.

REFERENCES

[1] Ciddor, P.E.; *Refractive Index of Air: New Equations for the Visible and Near Infrared*; Appl. Opt., Vol. 35(9), pp. 1566–1573 (1996).

[2] ASTM E 1421-99; *Standard Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers*, *Level Zero and Level One Tests*; Annu. Book ASTM Stand., Vol 03.06, (2009).

[3] Cameron, D.G.; Kauppinen, J.K.; Moffatt, D.J.; Mantsch, H.H.; *Precision in Condensed Phase Vibrational Spectroscopy*; Appl. Spectrosc, Vol. 36(3), pp. 245–250 (1982).

[4] Zhu, C.J.; Hanssen, L.M.; *Absorption Line Evaluation Methods for Wavelength Standards*; In Proceedings of the SPIE 3425, Vol. 3425, pp. 111–118 (1998).

[5] Zhu, C.J.; Hanssen, L.M.; *Studies of a Polystyrene Wavenumber Standard for Infrared Spectrophotometry*; In Proceedings of the 11th International Conference on Fourier Transform Spectroscopy, Vol. 430, pp. 491–494 (1998).

[6] Choquette, S.J.; Duewer, D.L.; Hanssen, L.M.; Early, E.A.; *Standard Reference Material 2036 Near-infrared Reflection Wavelength Standard*; Appl. Spectrosc, Vol. 59(4), pp. 496–504 (2005).

[7] Choquette, S.J.; Travis, J.C.; Duewer, D.L.; *SRM 2035: A Rare Earth Oxide Glass for the Wavelength Calibration of Near Infrared Dispersive and Fourier Transform Spectrometers*; In Proceedings of the SPIE Conference on Optical Diagnostic Methods for Inorganic Transmissive Materials, Vol. 3425, pp. 94–102 (1998).

[8] Duewer, D.L.; Choquette, S.J.; O'Neal, L.; Filliben, J.J.; *Rare-earth Glass Reference Materials for Near-infrared Spectrometry: Sources of x-axis Location Variability*; Anal. Chimica Acta, Vol. 490(1-2), pp. 85–98 (2003).

[9] 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 http://www.nist.gov/srm/upload/SP260-136.PDF (accessed March 2014).

[10] 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 (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed March 2014); 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://www.nist.gov/pml/pubs/index.cfm (accessed March 2014).

[11] Dersimonian, R.; Laird, N.; *Meta-analysis in clinical trials*; Controlled Clin. Trials, Vol. 7, pp. 177–188 (1986).

[12] Horn, S.D.; Horn, R.A.; Duncan, D.B; *Estimating Heteroscedastic Variances in Linear-models*; J. Am. Stat. Assoc., Vol. 70(350), pp. 380–385 (1975).

[13] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46(3), pp. 323–331 (2009).

Certificate Revision History: 06 March 2014 (Added certified values for UV-Vis peak locations and uncertainties; editorial changes); 04 May 2012 (Original certificate date).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.