National Institute of Standards & Technology

Certificate

Standard Reference Material® 2036

Near-Infrared Wavelength/Wavenumber Reflection Standard

This Standard Reference Material (SRM) is a certified transfer standard intended for the verification and calibration of wavelength (λ) and wavenumber (ν) scale near-infrared (NIR) spectrometers operating in diffuse reflectance mode. SRM 2036 is a combination of a glass physically contacted with a piece of sintered polytetrafluoroethylene (PTFE). The combination of rare earth oxide glass with a nearly ideal diffuse reflector provides reflection-absorption bands that range from 15 % reflectance (R) to 40 % R. SRM 2036 is certified for the 10 % band fraction centroid of seven bands spanning the spectral region from 975 nm to 1946 nm (air wavelength). In addition, it is certified for the 10 % band fraction centroid location of the same seven bands in the spectral region from 10 300 cm^{-1} to 5 130 cm^{-1} at 8 cm^{-1} resolution (vacuum wavenumber). A unit of SRM 2036 consists of the optical filter-PTFE assembly mounted in an optical holder, contained in a wooden box.

The glass is a mixture of the following mole fractions: 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 oxide (SiO_2), and zirconium oxide (ZrO_2). The optical filter is 25 mm in diameter and 1.5 mm thick. The sintered PTFE is 25 mm in diameter and approximately 6 mm thick.

Certified Band Locations: The certified vacuum-wavenumber locations for the seven reflection-absorption bands spanning the range from 10 300 cm^{-1} to 5 130 cm^{-1} are listed in Table 1. The certified values for the NIR air-wavelength band locations for the seven reflection-absorption bands from 976 nm to 1946 nm are listed in Table 2. 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 fully investigated or taken into account [1].

Expiration of Certification: The certification of **SRM 2036** is valid, within the measurement uncertainty specified, until **30 November 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.

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 2036 was conducted by S.J. Choquette with assistance from A. Gaigalas and A. Urbas of the NIST Biosystems and Biomaterials Division.

The SRM filter glass was cut and polished by J. Fuller of the NIST Fabrication Technology Division.

Statistical consultation was provided by S. Leigh of the NIST Statistical Engineering Division.

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

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Certificate Revision History on Last Page

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: To maintain the integrity of SRM 2036, the filter should only be handled in its optical mount.

Storage: While not in use, the SRM should be stored in the container provided or one with similar or better mechanical protection.

Use: SRM 2036 is intended for the calibration of diffuse reflectance spectrometers. The method of use for SRM 2036 will vary with the type of accessory. Generally, the reference spectrum may be obtained from the exposed PTFE portion of the SRM (or a similar piece of sintered PTFE). The sample spectrum is then acquired from the glass side of this SRM. The reflectance spectrum of the standard is then calculated as the ratio of the sample to the reference spectrum. For air-wavelength measurements the data density should be equal to or less than 0.5 nm per data point. For vacuum-wavenumber measurements, the data density should be no less than 1.0 cm^{-1} per data point. These data spacings ensure that the centroid location method itself does not contribute significantly to the location bias. Correction to absolute reflectance or for specular reflectance is not necessary as the bias for these is included in the location uncertainties. However, the front surface Fresnel reflection of the SRM may give reflectance values exceeding 100 % especially with fiber probes when referenced to the bare PTFE reference. This will not affect the band location estimates within the stated uncertainties.

Measurement of the SRM under a dry nitrogen purge is highly recommended. If a nitrogen purge is not available, the locations of band 1 and band 3 may differ from the certified values. Acquire the reflectance spectrum at a temperature of 23 °C ± 3 °C. Compare each measured band location to its certified value listed in the appropriate Table 1 or Table 2. Band locations in Table 1 are vacuum-wavenumber values, while those in Table 2 are air-wavelength values. To convert the values in Table 1 to air wavenumber, the appropriate correction for the index of refraction of air must be applied [2]. Taking into account the certification uncertainty of each band of SRM 2036, any statistically significant differences between the measured and certified band locations may then be used to recalibrate the spectrometer wavenumber/wavelength scale.

Measurement Conditions: The diffuse reflectance certification measurements for the NIR spectral region $(10\,300 \text{ cm}^{-1}$ to $5\,130 \text{ cm}^{-1})$ were made using Bruker IFS66 and Bio-Rad FTS-60 Fourier transform (FT) spectrometers.[1] The IFS66 spectrometer was equipped with a bifurcated-fiber-optic diffuse-reflectance probe. The FTS-60 spectrometer was equipped with a custom integrating sphere assembly that enabled the measurement of the total (8° sampling geometry) and diffuse components of the reflectance spectrum of SRM 2036. Details about this spectrometer can be found in references 3 and 4. The IFS66 spectrometer calibration was validated with ambient water vapor bands, SRM 2065 Ultraviolet-Visible-Near-Infrared Transmission Wavelength/Wavenumber Standard (compositionally identical to SRM 2036), and SRM 1920a Near Infrared Reflectance Wavelength Standard from 740 nm to 2000 nm. The FTS-60 spectrometer was calibrated using ambient water vapor and carbon dioxide bands.

The dispersive air-wavelength NIR measurements were performed on a PerkinElmer Lambda 900 (PE900) spectrometer equipped with a 60 mm integrating sphere and a Varian Cary 5e spectrometer equipped with a 110 mm integrating sphere assembly. Both spectrometers were used to measure the total and diffuse components of this standard. The air-wavelength axis of the PE900 spectrometer was calibrated in the NIR using the emission lines of the internal deuterium lamp (second order of the grating), and using SRM 2065 for transmittance and SRM 1920a for diffuse reflectance. The Cary 5e was calibrated using the same emission lines and standards as the PE900.

The transmission spectra of the optical glass only (without the PTFE backing) were measured on the IFS66 spectrometer operated in transmission mode. SRM 2036 is **NOT** intended for use as a transmission wavelength/wavenumber standard. However, the homogeneity of this melt and similarity of its band positions to SRM 2065 were determined using transmission measurements. For these transmission measurements, the IFS66 was calibrated in vacuum-wavenumber units using SRM 2517 Wavelength Reference Absorption Cell-Acetylene [5]. The calibration of the spectrometer was validated using water vapor bands. Details of the measurements and data analysis for both FT and dispersive measurements can be found in references 6 through 8.

Wavenumber and Wavelength Band Location Methodology: The method used to determine the certified NIR wavenumber and wavelength band locations of SRM 2036 is the centroid (center-of-gravity) technique [9–11]. If another technique is used, a comparison with the certified values **may not be valid**. In this certificate, positions determined with the centroid algorithm are referred to as *band* locations. For SRM 2036

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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.

centroid calculations, a 10 % fraction of the band was used for both wavenumber and wavelength reflection-absorbance data. Further information on the use of this algorithm with other NIST SRMs can be found in reference 10.

Certified Wavenumber Band Locations: The certified vacuum-wavenumber locations listed in Table 1 were obtained at 8 cm^{-1} constant wavenumber resolution and are certified for operation between 21 °C and 26 °C. The certified values represent the mean band locations from both the IFS66 fiber probe measurements and the FTS-60 total and diffuse-component-only measurements. The location bias for each of these modes of operation is well within the short-term precision variance of these instruments operated in diffuse reflectance. Atmospheric water vapor is a significant source of variance for band 3, and this band should be used with caution when calibrating unpurged commercial spectrometers with SRM 2036.

Certified Wavelength Band Locations: The certified values for the NIR air-wavelength band locations for the seven reflection-absorption bands for 3 nm, 5 nm, and 10 nm spectral slit width (SSW) resolution at 21 °C are listed in Table 2. The certified values of the air-wavelength band locations represent the mean band locations from both the Cary 5e and PE900 spectrometers operated in both total (specular and diffuse) and diffuse-only collection geometries. As for the FT measurements, the location bias for each of these modes of operation of the SRM was within the short-term precision variance of the dispersive spectrometers used for certification of this standard. The NIR reflectance spectrum of SRM 2036 is illustrated in Figure 1.

Certification Uncertainty: The expanded uncertainty (U_{95}) for the wavenumber and wavelength band locations given in Tables 1 and 2 are determined from the appropriate combination of component standard uncertainties (i.e., estimated standard deviations), with a coverage factor based on the Student's t-distribution, 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 % [12]. Components of the uncertainty for the vacuum-wavenumber band locations include: instrument bias (two certification spectrometers), calibration of the NIST FT spectrometers, location shift due to temperature, water vapor interference, and the short-term precision variance. Components of the uncertainties for the air-wavelength band locations include: instrument bias (two reference instruments) and the location uncertainty of the wavelength calibration standards.

Table 1. Certified Band Locations[a,b], Vacuum Wavenumber at 8 cm^{-1} Resolution

Band	Centroid Location $\pm$ U_{95} (cm^{-1})		
1[c]	5139.4	$\pm$	0.34
2	6803.9	$\pm$	0.94
3	7312.7	$\pm$	0.61
4	8179.0	$\pm$	0.36
5	8682.0	$\pm$	1.65
6	9293.9	$\pm$	1.30
7	10244.9	$\pm$	0.70

[a] Band locations determined using a centroid method with a band fraction of 0.1; see Figure 1 for band identification.
[b] Uncertainties represent U_{95}, the expanded uncertainty calculation using a T statistic where n = 3, T = 4.3, in accordance with reference 12.
[c] See Information Values section for additional information pertaining to Band 1 centroid location.

Table 2. Certified Band Locations[b,c], Air Wavelength

Band	3 nm SSW $\pm U_{95}$ (nm)			Standard Error of Mean[c] (nm)	5 nm SSW $\pm U_{95}$ (nm)			Standard Error of Mean[c] (nm)	10 nm SSW $\pm U_{95}$ (nm)			Standard Error of Mean[c] (nm)
7	976.0	±	0.3	(0.02)	976.0	±	0.2	(0.01)	975.9	±	0.6	(0.01)
6	1075.7	±	0.2	(0.01)	1075.7	±	0.9	(0.01)	1075.8	±	2.2	(0.03)
5	1151.4	±	0.1	(0.01)	1151.2	±	1.0	(0.01)	1151.0	±	3.4	(0.01)
4	1222.1	±	0.4	(0.02)	1222.1	±	0.3	(0.02)	1222.1	±	0.9	(0.03)
3	1367.1	±	0.4	(0.01)	1367.2	±	0.5	(0.01)	1367.3	±	0.2	(0.02)
2	1469.6	±	0.4	(0.02)	1469.6	±	1.7	(0.02)	1469.5	±	3.7	(0.03)
1[d]	1945.7	±	0.3	(0.04)	1945.8	±	0.7	(0.02)	1945.6	±	1.5	(0.01)

[a] Band locations determined using a centroid method with a band fraction of 0.1; see Figure 1 for band identification.
[b] Uncertainties represent U_{95}, the expanded uncertainty calculation using a T statistic where n = 3, T = 4.3, in accordance with reference 12. The 95 % confidence interval of the air-wavelength bands of SRM 2036 is determined by the uncertainty of the calibration of the reference instruments with SRM 2065.
[c] The number in parentheses is the standard error of the mean centroid band location.
[d] See Information Values section for additional information pertaining to Band 1 centroid location.

Information Values: 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 [1]. Information values are intended to provide additional information on the matrix. The approximate location of the band edges used to calculate band fractions in vacuum wavenumber (cm^{-1}) and air wavelength (nm) are given in Table 3. Regarding Band 1, the certified band locations were determined using peak fractions associated with the band edge listed in Table 3. However, an inflection point arising from a shoulder peak is evident in the reflectance spectrum of SRM 2036 at approximately 1995 nm (5 130 cm^{-1}) in Figure 1. This inflection point can be used for the determination of a 10 % peak fraction for Band 1 but studies have shown that the centroid band location in this case will typically fall outside the certification uncertainties. These studies revealed a bias in centroid band location for Band 1 using a 10 % peak fraction based on this shoulder peak compared to that used for the certified values of approximately +0.4 nm for wavelength (e.g., 1946.1 nm for 3 nm SSW) and –0.9 cm^{-1} for wavenumber (e.g., 5138.5 cm^{-1}).

Information values for the NIR vacuum-wavenumber temperature coefficients and 0 °C degree intercepts are given in Table 4. SRM 2036 has additional absorbance bands between 334 nm and 1000 nm. The locations of these bands are currently not certified. Information values on the location of these bands are provided in Table 5 for a SSW of 5 nm. The ultraviolet-visible reflectance spectrum of SRM 2036 is illustrated in Figure 2.

Table 3. Information Values Approximate Location of Band Edge, Wavenumber (cm^{-1}) and Wavelength (nm).

Band	cm^{-1}	nm
1	4550	2180
2	6650	1503
3	7160	1396
4	8560	1168
5	8560	1168
6	8935	1119
7	10365	964

Table 4. Information Values for Temperature Coefficients: Parameters for 4 cm^{-1} Resolution Spectra[a].

Band	$\overline{\beta_i}$, (cm^{-1}/°C)	$\overline{\alpha}_{i,SRM}$, (cm^{-1})
1	−0.0494(15)[b]	5139.77(12)
2	0.0878(16)	6802.16(5)
3	0.012(6)	7312.41(12)
4	0.0597(10)	8176.78(6)
5	−0.0395(15)	8682.5(2)
6	−0.0751(19)	9295.84(15)
7	0.0179(7)	10245.12(3)

[a] All band locations can be described by a band-specific linear model: $b_i = \alpha_i + \beta_i T$, where b_i denotes the estimated band location, T is the temperature in °C of the filter during acquisition of the spectrum, β_i is the change in band location per °C (temperature coefficient slope) for the ith band, and α_i is the extrapolated location of the band at 0 °C (temperature coefficient intercept).

[b] The digits in parentheses denote the combined standard uncertainty of the last reported digit of the coefficient.

Table 5. Information Band Location Values for Measured in Reflectance at 5 nm SSW.

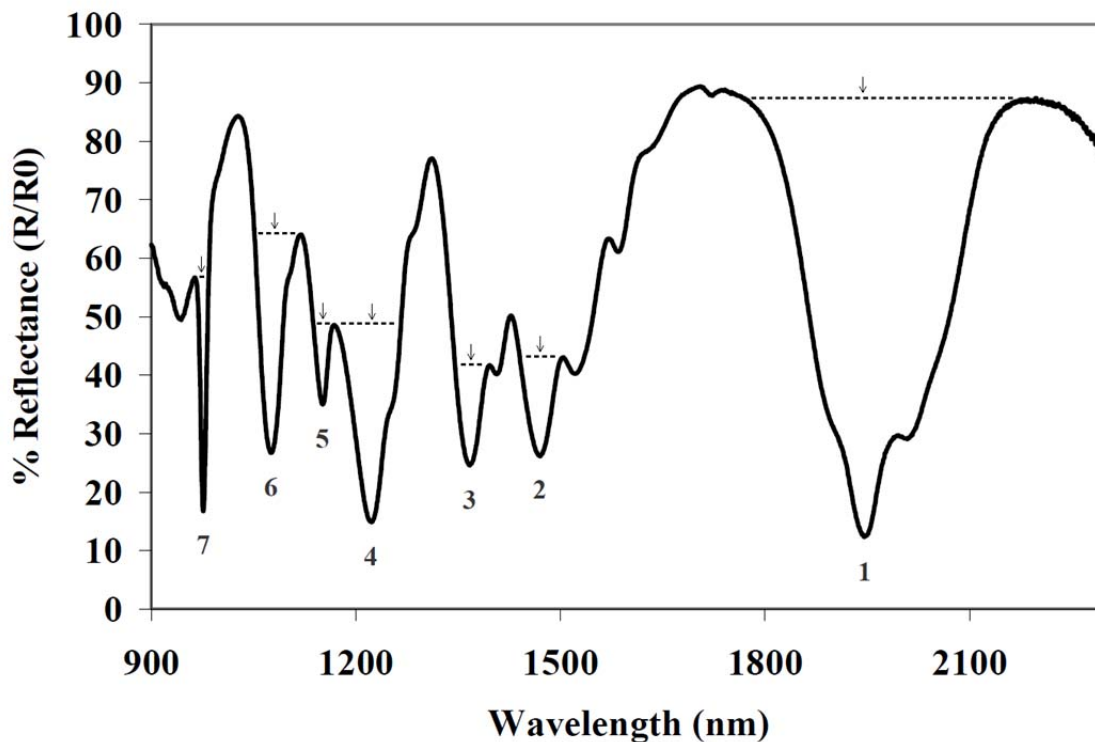| Band	Wavelength (nm)
20	333.8
19	346.1
18	361.3
17	374.3
16	385.8
15	402.6
14	418.2
13	485.3
12	538.2
11	583.3
10	642.7
9	747.6
8	804.2

Figure 1. SRM 2036 Diffuse Reflectance Spectrum. Specular-excluded spectrum acquired at 5 nm spectral slit width. The dashed lines and arrows indicate the base of the band from which the 10 % band fractions were calculated for band location certification. They do not represent the 10 % band fractions. See Information Values section for additional information pertaining to Band 1 centroid location.
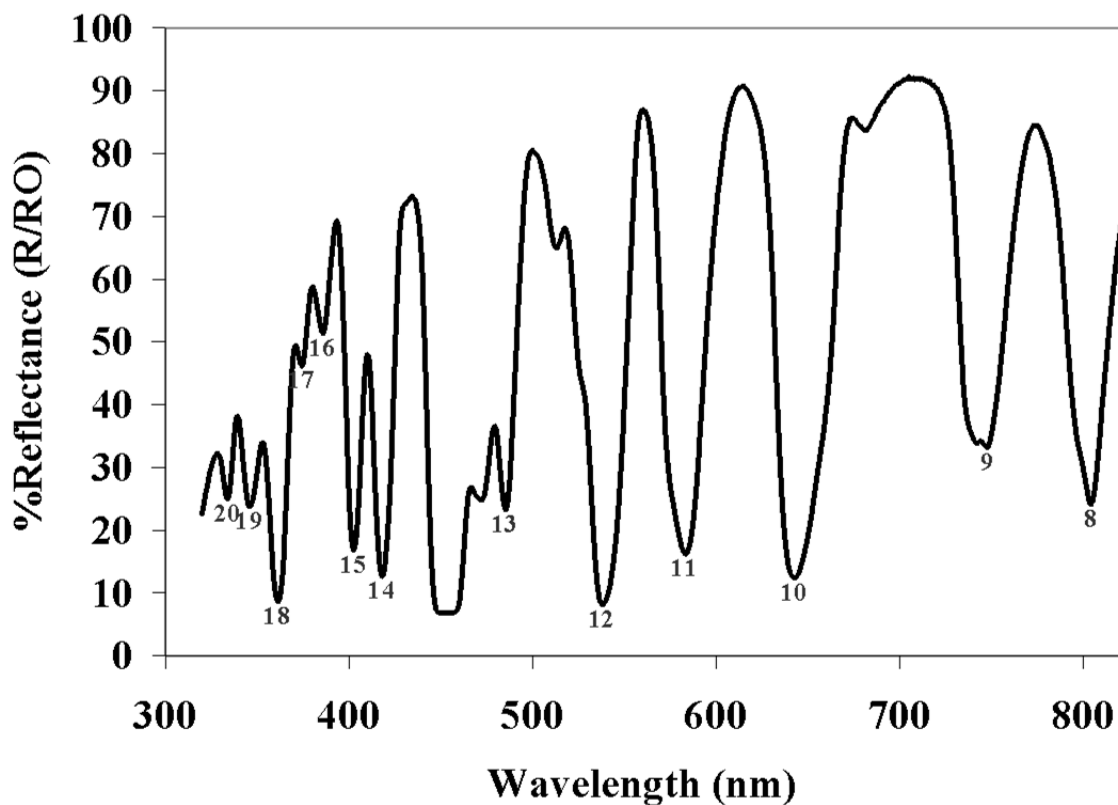

Figure 2. SRM 2036 Diffuse Reflectance Spectrum. Specular-included spectrum acquired at 5 nm spectral slit width.

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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.