National Institute of Standards & Technology

Certificate

Standard Reference Material® 1929

Infrared High Reflectance Specular Standard

(Nominal Diameter 25 mm)

Serial Number: Sample

This Standard Reference Material (SRM) is intended primarily for calibrating the reflectance scales of specular reflectometers in the infrared (IR) spectral region from 1.0 µm to 18.0 µm (555 cm^{-1} to 10,000 cm^{-1}). A unit of SRM 1929 consists of a specular finish gold plated copper disk, approximately 3.0 mm thick and 25.3 mm in diameter. Each unit is inscribed with an identifier number; the last two digits of the inscribed number correspond to the serial number assigned by NIST. SRM 1929 is shipped in an anodized aluminum container with protective inserts constructed in such a way as not to impinge upon the front surface of the mirror. The SRM is placed with its front surface face down into the bottom half of the container.

Certified Reflectance Values: SRM 1929 was measured using the NIST Fourier Transform Infrared Spectrophotometer and Infrared Reference Integrating Sphere [1-5]. This instrument measures reflectance using absolute techniques. The measurements are made as a function of wavelength. Each unit of SRM 1929 is independently certified for a wavelength range of 1 µm to 18 µm. This SRM is certified for specular reflectance for near-normal geometries, unpolarized incident light with an average angle of incidence on the sample between 0° and 30°, and any incident geometry between f/1 and f/16, and any collection geometry from near zero solid angle up to the full reflected hemisphere. For each unit of SRM 1929, at each wavelength, the certified value of the reflectance is given in absolute units (between 0 and 1). The measurand is the regular reflectance measured as a function of wavelength (µm) or equivalent wavenumber (cm^{-1}). Metrological traceability is to the NIST infrared regular reflectance scale as described in NIST Special Publication 250-94. The values shown in Figure 1 of this certificate are valid for the ranges mentioned only. The certification data for SRM 1929 Serial Number Sample can be downloaded at the NIST website at https://www-s.nist.gov/srmors/view_detail.cfm?srm=1929.

Expiration of Certification: The certification of **SRM 1929** is valid, within the measurement uncertainty specified, until **31 December 2023**, 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, exposed to excess humidity, 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 or register online) will facilitate notification.

Overall direction and coordination of the technical measurements leading to certification were performed under the supervision of J.P. Rice and L.M. Hanssen of the NIST Sensor Science Division.

Technical measurements leading to certification were performed by S.G. Kaplan and L.M. Hanssen of the NIST Sensor Science Division.

Statistical consultation and evaluation of the data were provided by N.F. Zhang of the NIST Statistical Engineering Division.

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Steven J. Choquette, Director
Office of Reference Materials

Gaithersburg, MD 20899
Certificate Issue Date: 04 April 2019
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Support aspects involved in preparation, certification, and issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling and Storage: SRM 1929 is fragile and must be handled with **care**. Airborne particulates, aromatics, and improper handling may adversely affect the surface conditions. The SRM should always be handled carefully so that nothing is allowed to contact the bare gold surface, including fingers, other than any holder the user may require. Any holder should be designed such that any contact with the holder is limited to the outside edges away from the regions used for measurement. Other than using a clean air bulb to gently remove dust from its front surface, the user should **not** attempt any other cleaning process, as such action may adversely affect the gold coating. When not in use, SRM 1929 should be kept in its original container. For storage, it is advisable to keep the SRM in a desiccator cabinet.

Use: The sample reflectance should be measured using unpolarized light at near normal incidence within the above specified ranges. A measurement of a sample at 45° incidence using polarized light showed a variation of up to ± 0.005 in reflectance for s or p polarization, but only 0.0005 for unpolarized light. If the light source or detector system used for reflectance measurements have significant polarization content or sensitivity and the incidence angle is greater than 10°, then some effort should be made to depolarize the light or average two orthogonal linear polarization measurements.

Certification Data: The certification data, as shown in Figure 1, is provided to the user in the form of a tab-delimited text file. The data is in columnar format, with columns for wavenumber, wavelength, reflectance, and expanded uncertainty, respectively, over the entire spectral range in steps of 4 cm^{-1}.

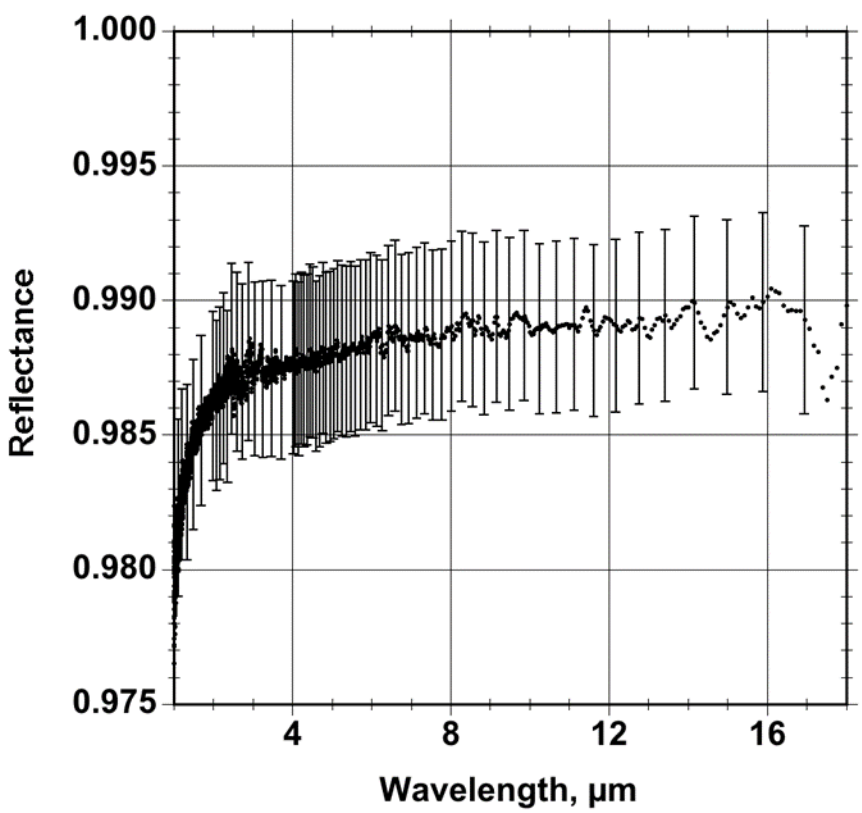

Figure 1. Measured Reflectance Values and Associated Expanded Uncertainties for SRM 1929 Serial Number Sample.

SOURCE, PREPARATION, AND ANALYSIS

Source and Preparation: The SRM samples were produced by Epner Technology Inc. (Brooklyn, NY)[1]. The standard material is an electrodeposited proprietary gold coating over nickel over a polished copper disk.

Certification Measurements: Infrared reflectance measurements were performed using a Digilab 7000S Fourier Transform Spectrometer (FTS) system coupled to a custom integrating sphere apparatus. The instrument room temperature was maintained at 23 °C ± l °C and the humidity ranged from 30 % to 50 % during the measurements. Measurements were made under a purge gas of clean air (with CO_2 and H_2O removed) at atmospheric pressure. Details of the measurement methodology can be found in references 1, 4 and 5. Spectral directional hemispherical reflectance was measured from 2.5 µm through 18.0 µm (555 cm^{-1} through 4000 cm^{-1}) with the setup of the FTS in a mid-IR mode with a KBr beamsplitter and a SiC “globar” source, and in a near-IR mode from 1.0 µm through 2.5 µm (4000 cm^{-1} through 10000 cm^{-1}) with a quartz beamsplitter and a quartz-halogen light source. The spectral resolution was 8 cm^{-1}. A final smoothing was applied to the measured reflectance data, resulting in an effective spectral resolution of 21 cm^{-1}. Measurements were performed with an f/6 cone centered on an average 8° angle of incidence with nominally unpolarized incident light. The focus spot on the sample was 10 mm in diameter. The detector was a liquid-nitrogen-cooled mercury-cadmium-telluride photoconductive element with a KCl lens and gold-coated non-imaging concentrator, viewing a portion of the inner sphere surface. Additional details of the measurement instrumentation and methodology can be found in references 1-5.

Uncertainty Evaluation: The expanded uncertainty, $U_{95\%}(x)$, is provided for each certified reflectance value, x, at each wavelength and shown as error bars in Figure 1. It is the product of the coverage factor, $k = 2$, and the combined standard uncertainty. The true value of the reflectance x is believed to lie within $x \pm U_{95\%}(x)$ with an approximate 95 % confidence. The combined standard uncertainty is the root sum of squares of all the uncertainty components [6-7]. Uncertainty components due to systematic effects are related to the FTS and interface optics and the integrating sphere system. The uncertainties related to the spectrometer and interface optics are wavelength scale error, phase correction error, beam geometry, beam polarization, and system component inter-reflections. Uncertainties related to the integrating sphere system are detector nonlinearity, overfill of the integrating sphere entrance and sample ports, nonequivalence of sample and reference alignment, atmospheric absorption variation, and the local spatial variations of the integrating sphere throughput. Uncertainty components due to random effects are FTS source and alignment stability and detector noise. The uncertainty contributions caused by these effects were evaluated by multiple measurements of the test items. Details on the estimation of these uncertainties are given in Reference 5.

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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.

REFERENCES

[1] Hanssen, L.M; Kaplan, S.G.; Datla, R.U.; *Infrared Optical Properties of Materials* ; NIST Special Publication 250-94 (2015); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.250-94.pdf (accessed Apr 2019).

[2] Kaplan, S.G; Hanssen, L.M; *Infrared Diffuse Reflectance Instrumentation and Standards at NIST*; Anal. Chim. Acta, Vol. 380; pp. 303-310 (1998).

[3] Hanssen, L.M; Kaplan, S.G.; *Infrared Diffuse Reflectance Instrumentation and Standards at NIST*, Anal. Chim. Acta; Vol 380, pp. 289-302 (1998).

[4] Hanssen, L.M, *Integrating-Sphere System and Method for Absolute Measurement of Transmittance, Reflectance, and Absorptance of Specular Samples*," Appl. Opt.; Vol. 40, pp. 3196-3204 (2001).

[5] Chunnilall, C.J.; Clarke, F.J, Hanssen, L.M; Kaplan, S.G.; Smart, M.P.; *NIST-NPL Comparison of Mid-infrared Regular Transmittance and Reflectance*, Metrologia, Vol. 40, pp. S55-S59 (2003).

[6] 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 (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2019);

[7] 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Apr 2019).

Certificate Revision History: 04 April 2019 (Editorial changes); 07 September 2018 (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 https://www.nist.gov/srm.