Status Update of the Infrared Optical Properties of Materials Program

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Outline

I. Infrared Optical Properties Measurement Program Overview

II. New Specular Reflectance Standard Reference Materials & Intra-Comparison

III. Refractive Index Characterization of Polymers

IV. Summary

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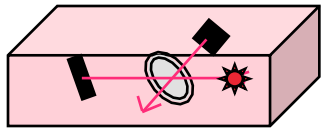
NIST Infrared Optical Properties Measurement Program

FT-IR Spectrometers



IR Lasers

Fixed λ — Tunable λ

Measurement Methodologies

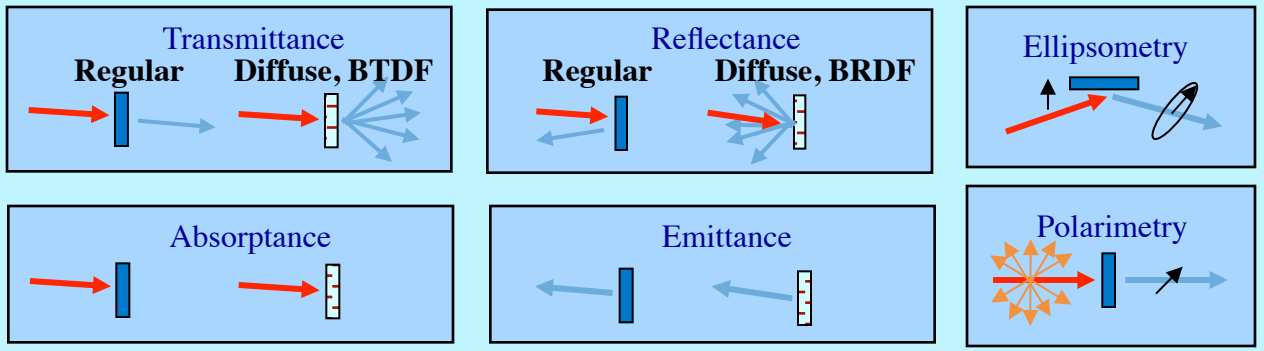
- Modeling Instruments & Methods
- Error Sources Evaluation
- Absolute Methods ρ, τ, α, ε

Specialized Accessories

- Integrating Spheres
- Goniometer & BRDF
- Refractometer
- Radiance Comparator
- Cavity Reflectometer

Materials, Coatings, Optical Components, Cavities & Detectors

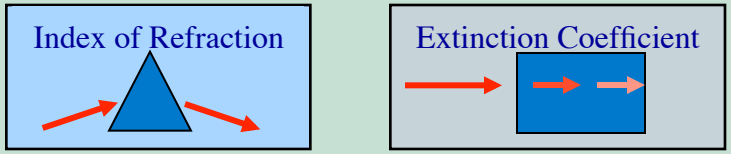
Direct Measurements & Properties



Support Mechanisms

- Calibration Service
- Physical Standards
- Inter-Comparisons
- Reference Data

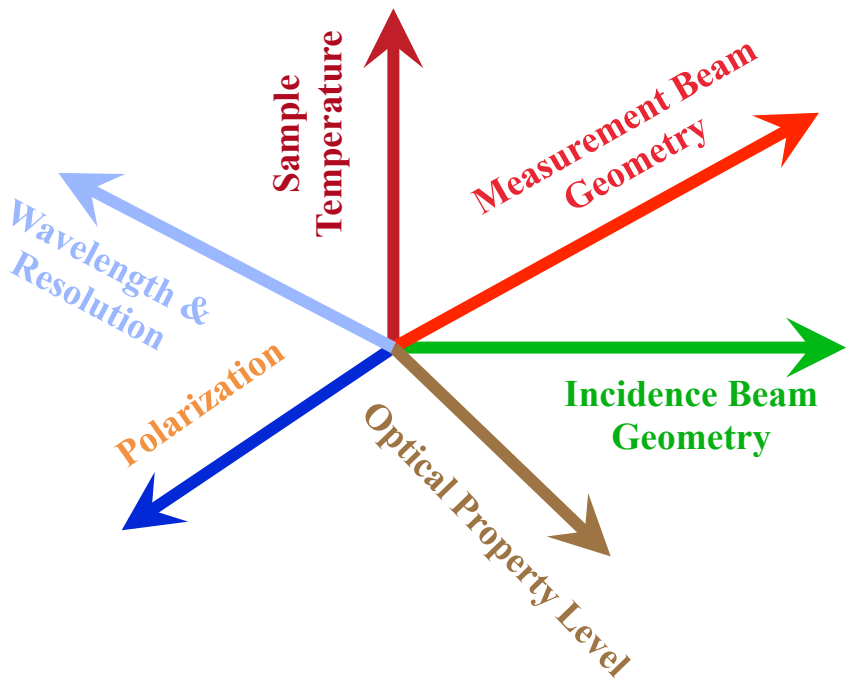
Additional Quantities



Support Communities

External:	Internal:
Industry	Detectors
Government	Sources
Academia	Materials

Critical Variables Addressed

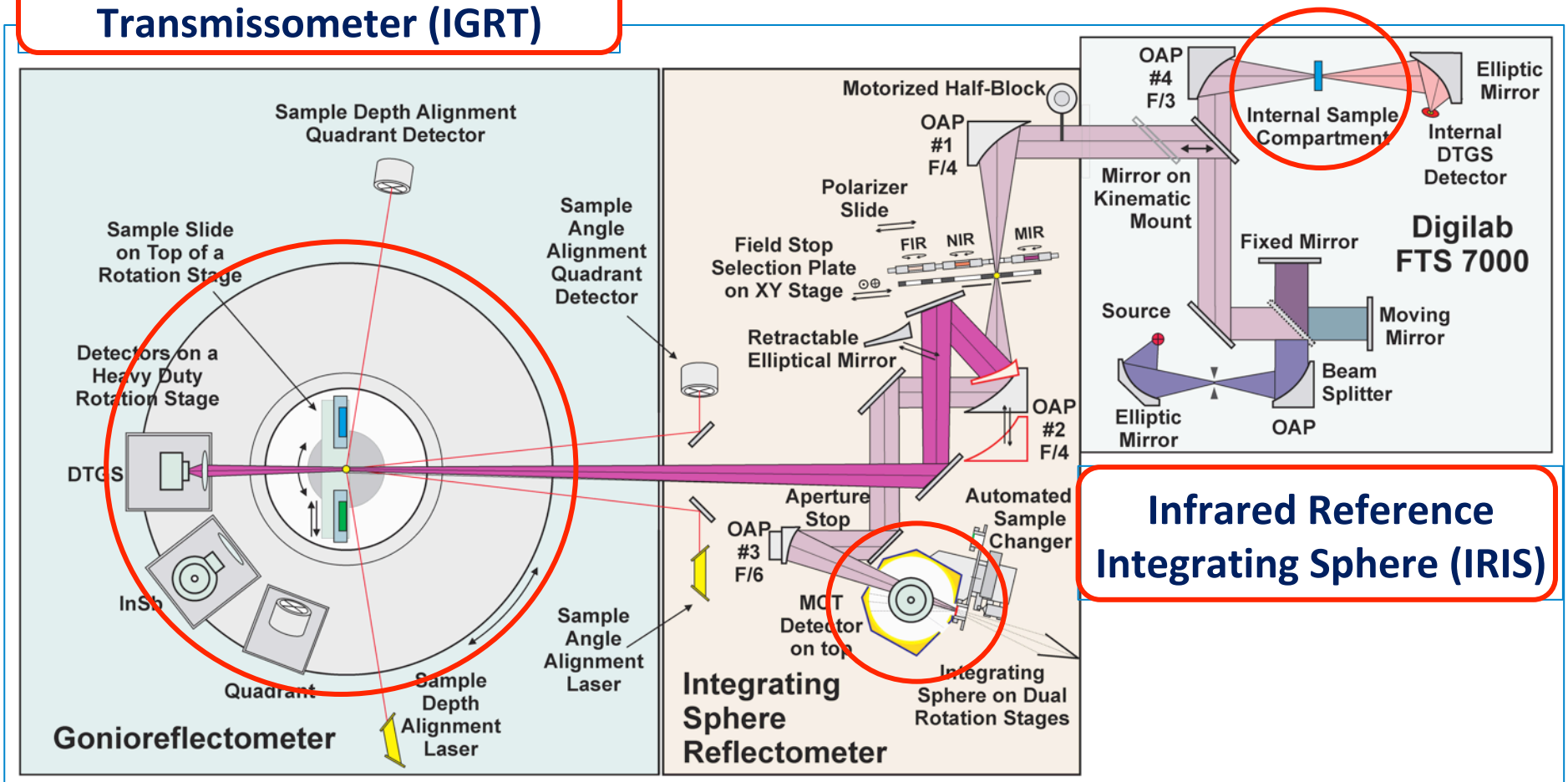


Typical Sample Types Characterized

- **Windows**: transmittance / emittance
- **Mirrors**: reflectance / emittance
- **Filters**: in-band spectral transmittance & out-of-band level
- **Coatings** (especially **blacks**): diffuse reflectance, emittance
- **Cavities** & structured surfaces (for blackbodies, radiometers): emittance
- **New** (& old) IR **optical** materials: index of refraction
- **All materials**, especially undergoing processing: emittance / reflectance
- **Polarizers**: extinction coefficient, Mueller Matrix elements

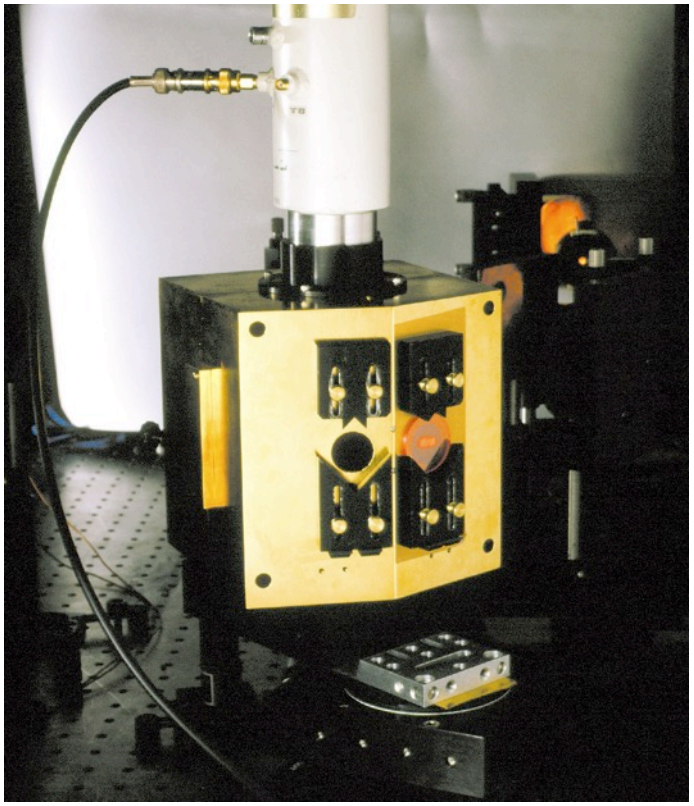
NIST Fourier Transform Infrared Spectrophotometry System (FTIS)

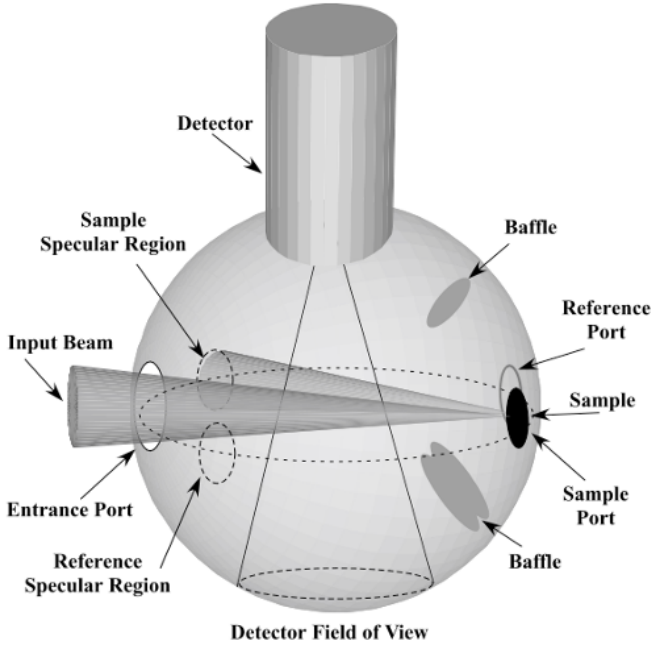
Infrared Gonio-Reflectometer Transmissometer (IGRT)



Infrared Reference Integrating Sphere (IRIS)

Infrared Reference Integrating Sphere (IRIS)





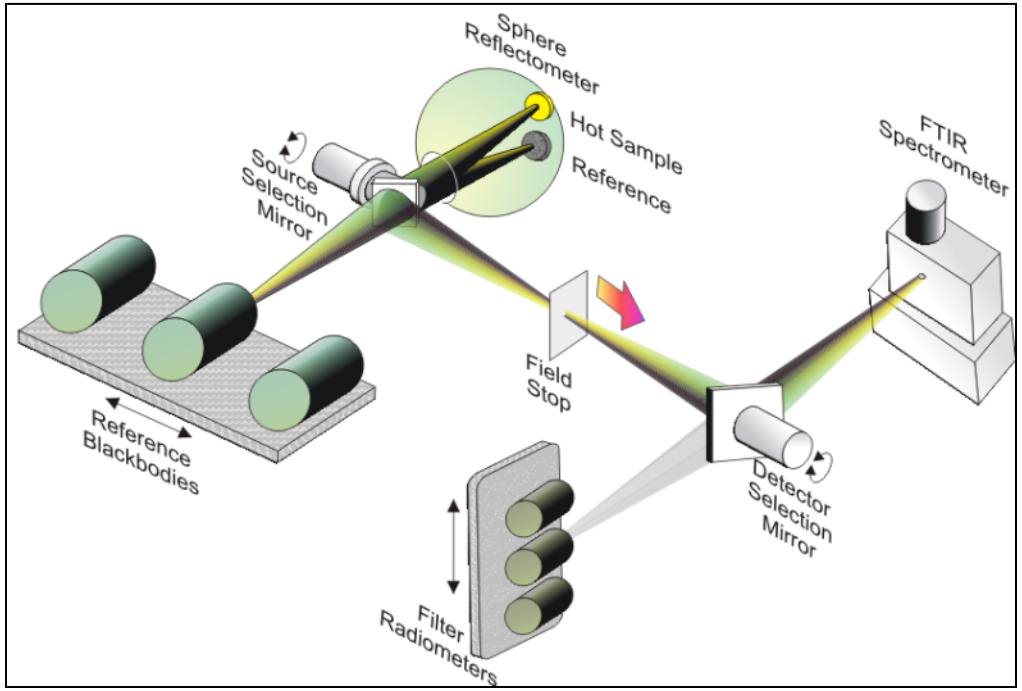
Sphere Specifications

- λ range: 1.0 – 18 µm
- 15 cm diameter
- Gold-plated plasma-sprayed metal coating
- MCT detector w/ concentrator optics
- Spot size 2 - 10 mm
- 8° incidence angle

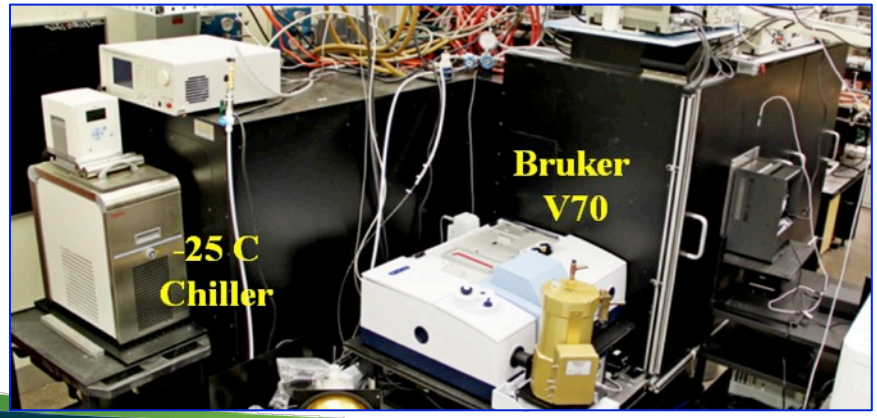
System Capabilities

- Reflectance, Transmittance, Absorptance & Emittance
- Temperatures 15 – 200 °C
- absolute & relative, specular & diffuse
  - R & T direct
  - A & E indirect
- uncertainties (2s):
  - specular: ≤ 0.3%
  - diffuse: 1.5 - 3.5%
  - larger for angle dependent structure
- can measure R of transparent samples
- can sort out scatter from total R & T

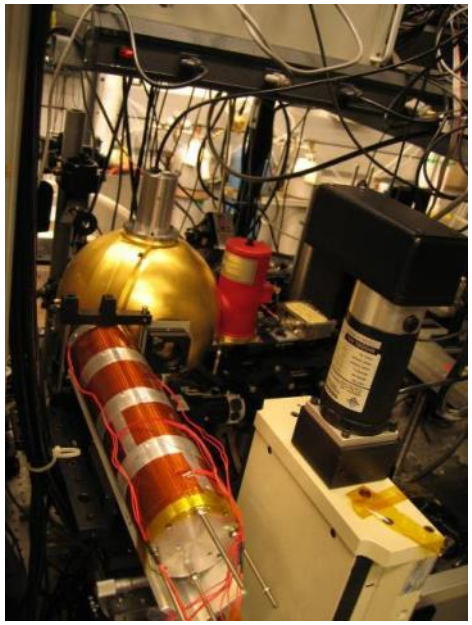
Infrared Spectral Emittance System

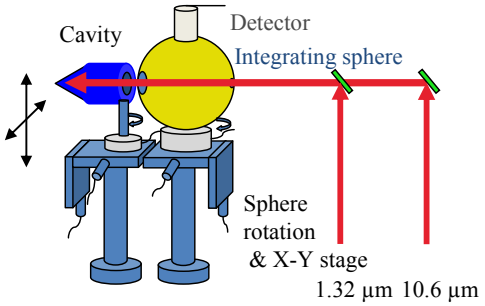


- FT-based Spectral Radiance Comparator (includes Near IR Sphere)
- Purge, polarization; λ range: 1 µm - ≥ 50 µm; *T* range: 200 °C – 900 °C
- Sample heaters, T range: 20 °C to 900 °C
- Filter radiometers: Si (650 nm), Si (900 nm), InGaAs (1550 nm), for T scale transfer & sphere reflectometer
- Sphere reflectometer for sample front surface temperature measurement
- Used for CCT-S1 *Infrared Normal Spectral Emissivity* Supplementary Comparison



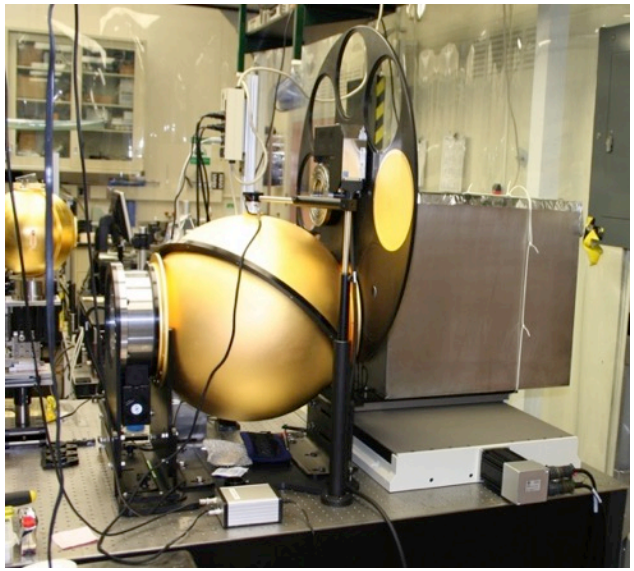
Integrating Sphere Reflectometers for Cavity Reflectance:
Complete Hemispherical Infrared Reflectometer (CHILR)





CHILR I
20 cm dia.

CHILR II
50 cm dia.



- Designed for low level reflectance measurement using gold integrating spheres with small 6 & 12 mm entrance aperture (1/2 angle = 1°) and 50 & 200 mm sample port
- Laser sources: 1.32 µm, 1.55 µm, 3.39 µm, 3.9 µm, 5.0 µm, 10.6 µm, (small tunable range)
- Detectors: MCT, InSb, pyro
- Motorized stages for automated movement of sphere and cavity or input beam
- Map spatial uniformity & angle dependence
- Can measure reflectance down to approx. 10^{-5} (equivalent to emissivity 0.99999)
- Reflectance expanded uncertainties previously estimated 15 - 20% for 10^{-3} to 10^{-5} range
- Radiometer and Blackbody Cavities measured: from 3 mm dia. up to 150 mm dia.

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Calibration Service for IR Optical Property Measurements*

Special tests infrared reflectance, transmittance, and emittance of materials.

SKU 38075S

Availability:

Add to Cart for Price Quote

Primary NIST Technical Contact:
Name: Leonard Hanssen
Phone: (301) 975-2344
Email: Email NIST Technical Contact
Secondary NIST Technical Contact:
Name: Simon Kaplan
Phone: (301) 975-2336
Email: Email NIST Technical Contact

Note: lead time is 90 days.

Qty 1

Add to Cart

Measurements of **infrared spectral reflectance**, **transmittance**, and **emittance** can be made in the wavelength region **1 µm to 20 µm** for submitted test items. In general, measurements are performed on test items with dimensions from 1 cm to 7.5 cm perpendicular to the incident beam. Measurements can be performed in **specular, diffuse, directional-hemispherical**, and bidirectional distribution function (**BRDF**) geometries. Capabilities also exist for performing **variable temperature** measurements from **10 K** to **473 K** (**200 °C**), with **emittance** measurements available from room temperature to **900 °C**. Uncertainty estimates are provided, and depend on the optical characteristics of the submitted test item and the instrument used to perform the measurement. Arrangements for measurements on submitted test items must be made before shipment. The decision to perform the measurements and selection of the instruments to be used rests with NIST. Test items not accepted for measurement will be returned.

* Available at shop.nist.gov

New Infrared Reflectance Standards 1928 & 1929

National Institute of Standards & Technology

Certificate

Standard Reference Material® 1928

Infrared Specular High Reflectance Standard
(Nominal Diameter 51 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 1928 consists of a specular finish gold plated copper disk, approximately 6.0 mm thick and 50.8 mm in diameter. Each unit is inscribed with an identifier number; the two digits after the left "/" of the inscription correspond to the serial number assigned by NIST. SRM 1928 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 1928 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 1928 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 1928, 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 [1]. The values shown in Figure 1 of this certificate are valid for the ranges mentioned only. The certification data for SRM 1928 Serial Number Sample can be downloaded at the NIST website at https://www-s.nist.gov/srmors/view_detail.cfm?srm=1928.

Expiration of Certification: The certification of **SRM 1928** 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.

Gerald T. Fraser, Chief
Sensor Science Division

Gaithersburg, MD 20899
Certificate Issue Date: 26 February 2019

Steven J. Choquette, Director
Office of Reference Materials

SRM 1928

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National Institute of Standards & Technology

Certificate

Standard Reference Material® 1929

Infrared Specular High Reflectance 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.

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Gaithersburg, MD 20899
Certificate Issue Date: 07 September 2018

Steven J. Choquette, Director
Office of Reference Materials

SRM 1929

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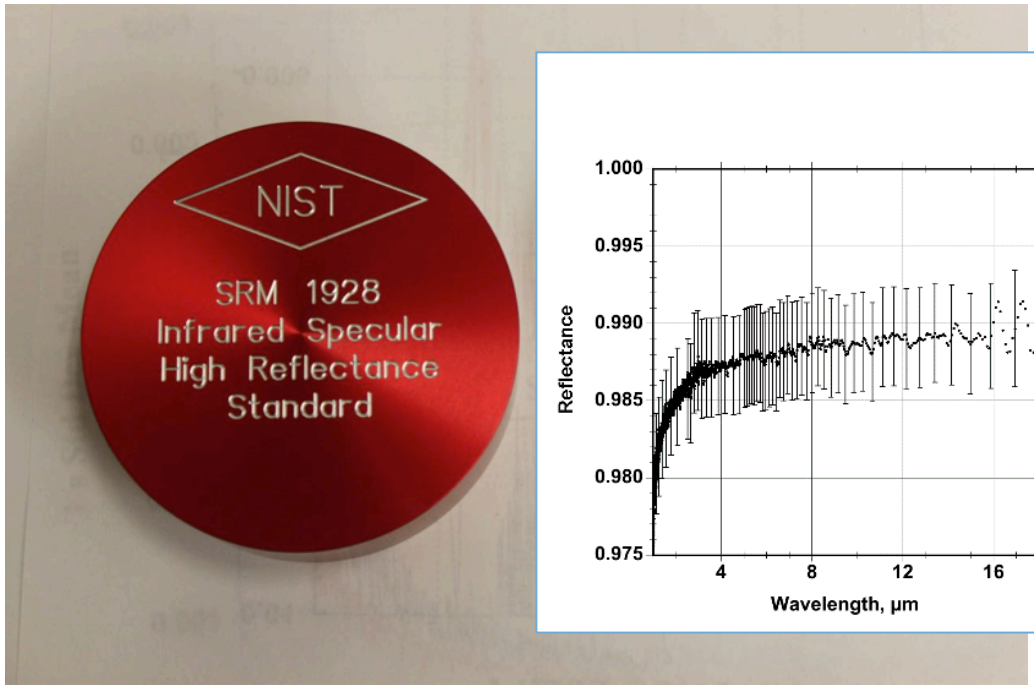
Standard Reference Materials (SRMs) 1928, 1929 Gold Mirror Reflectance Standards



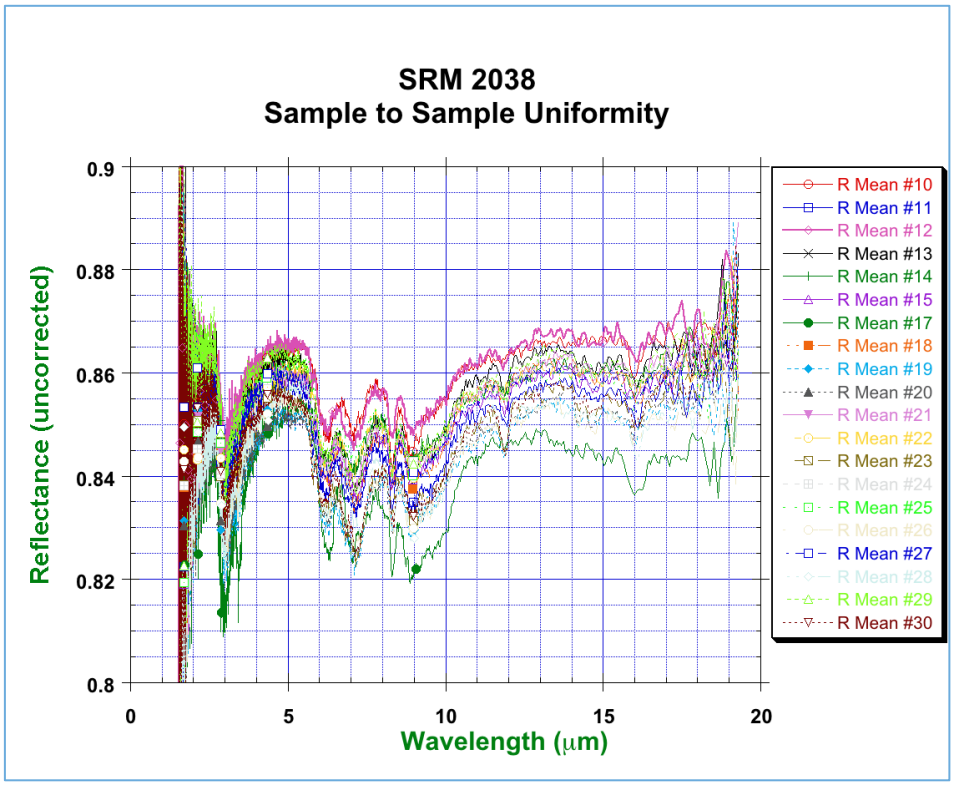
- Electroplated-gold on polished copper substrate mirrors: 1 µm – 18 µm
- Studied over a period of 5 years, material over 10 years
- SRM 1928: 51 mm dia.; SRM 1929: 25 mm dia
- Available for Purchase from NIST

1999 SRM 2038 DHR Standard

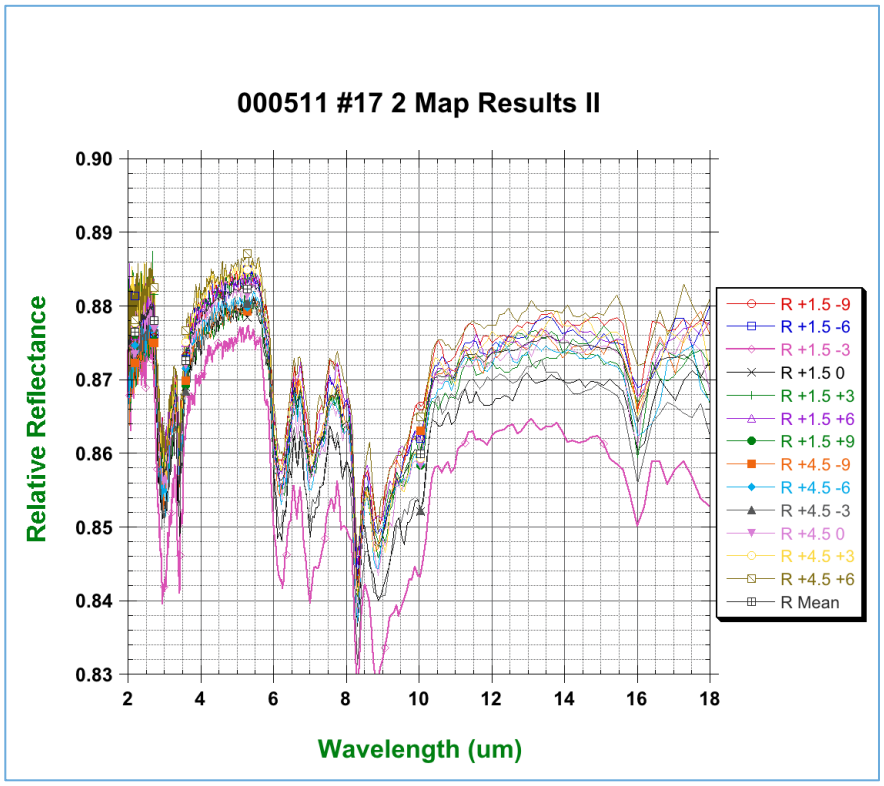
- Set of 30 produced and calibrated
- Coating Properties:
  - Close to ideal diffuser: near Lambertian BRDF
  - High Reflectance
  - Used for NIST Infrared Reference Integrating Sphere

SRM 2038 Set Uniformity Results

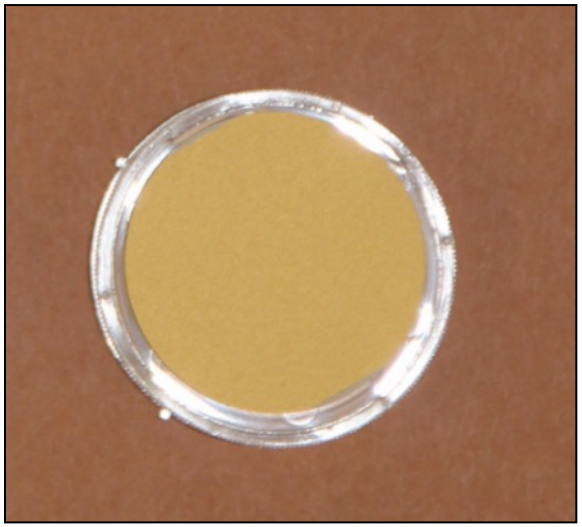
Sample Set Variation

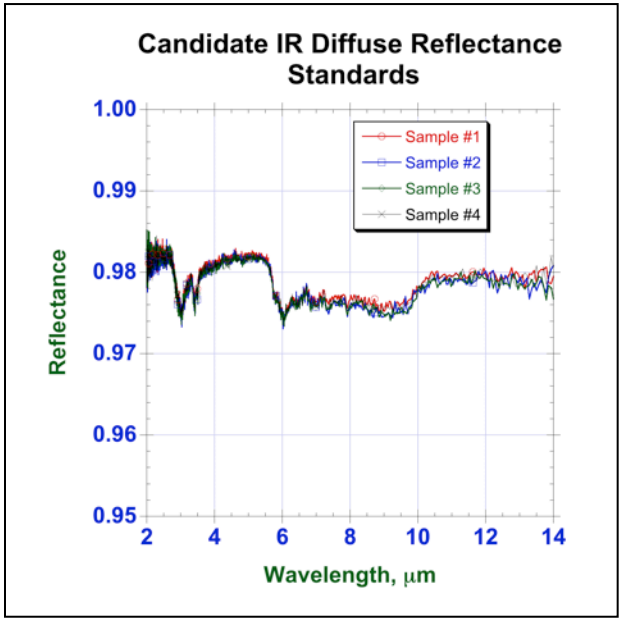


Sample #17 Spatial Uniformity



SRM 2038a Diffuse Infrared High Reflectance (in production)



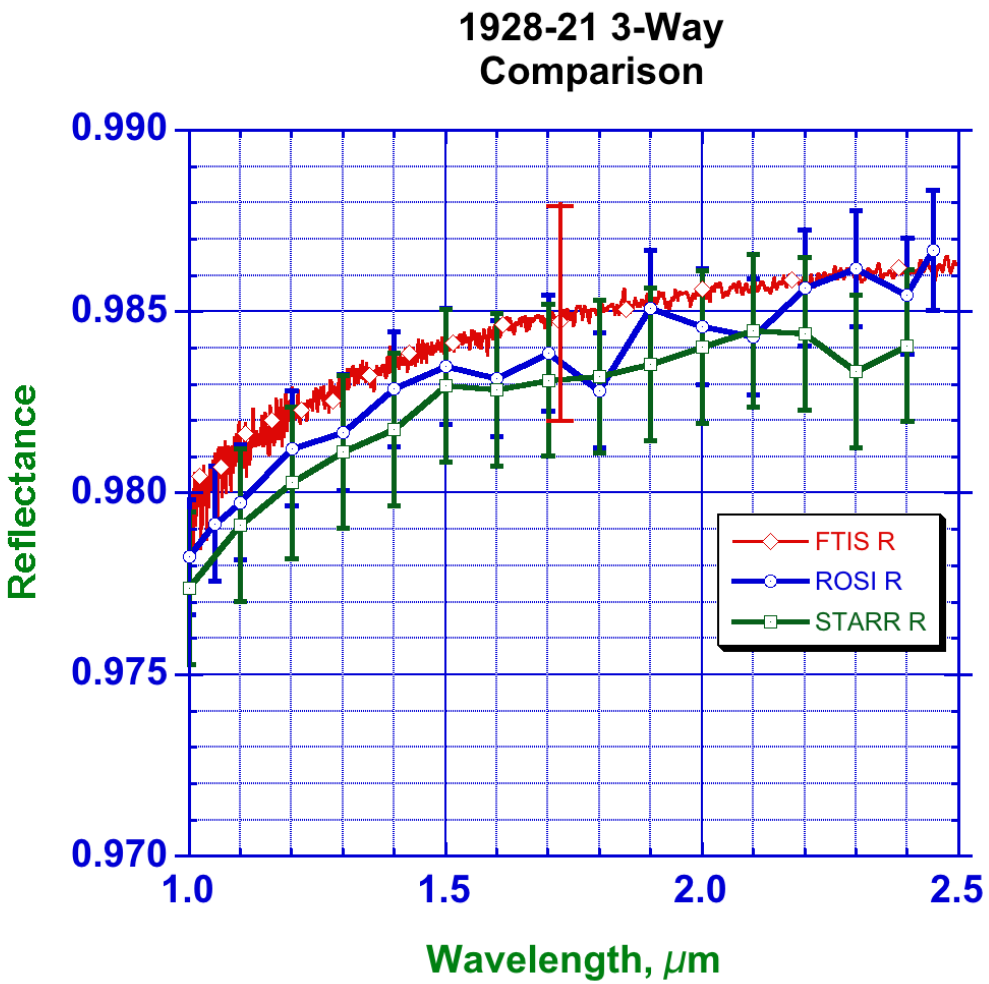


- Electroplated gold over laser etched nickel substrate (replicable)
- Calibrated for near-normal-hemispherical spectral reflectance
- From 2 µm to 18 µm
- Anticipated expanded (*k*=2) uncertainty: 1.5%
- Anticipated availability: FY 2021

NIST Intra-comparison of Specular Reflectance

- 3 instruments at different NIST spectral calibration facilities:
  - Fourier Transform Infrared Spectrophotometry System (FTIS) *Hanssen*
  - Spectral Tri-function Automated Reference Reflectometer (STARR) *Cooksey*
  - Robot Optical Scatter Instrument (ROSI) *Patrick*
- Measurement of SRM 1928-21 @ near-normal (8°) reflectance
- Mutual spectral range of 1.0 to 2.4 µm (1000 – 2400 nm)

Comparison of FTIS, STARR and ROSI



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Broadband refractive index measurements

Motivation

- Energy demand for air conditioning is predicted to overtake heating and grow until at least 2100.[1]
- Passive means of cooling are increasingly important to combat both cause and effects of climate change.
- Passive radiative coolers (PRCs) are an economical approach.

PRC Operating Principle

- Emit in 8-13 µm atmospheric transmission window
- Reflect elsewhere (especially at peak solar wavelengths)

Goal

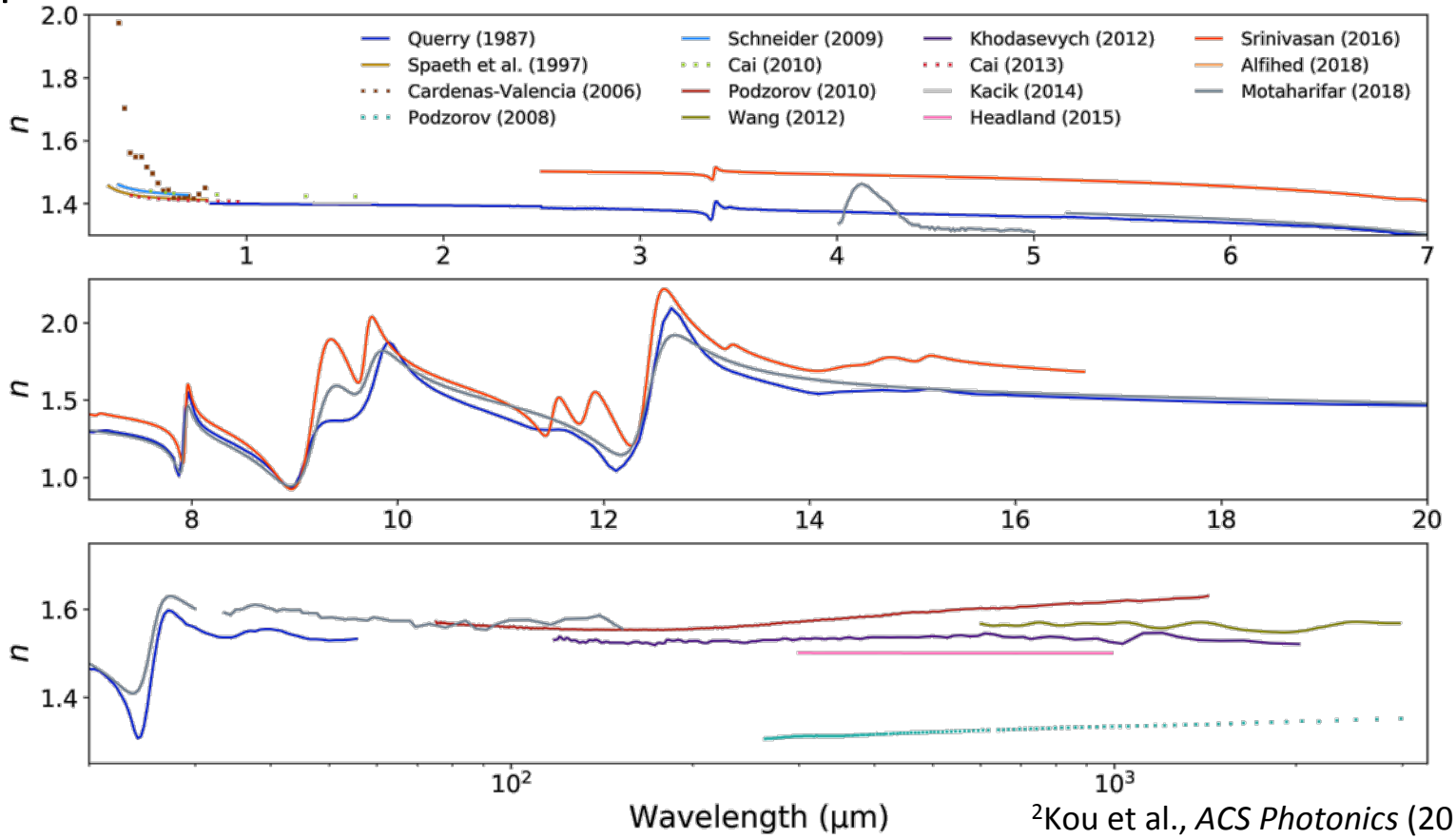
- Simplify design process for PRCs
- Determine complex refractive index from 250 nm to (at least) 125 µm for various candidate materials

[1]Isaac and van Vuuren, *Energy Policy* (2009)

Broadband refractive index measurements

Example: Polydimethylsiloxane

- Used in recent PRC publications[2-3]
- Literature index values are inconsistent

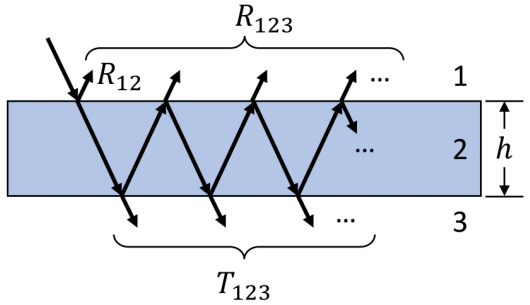


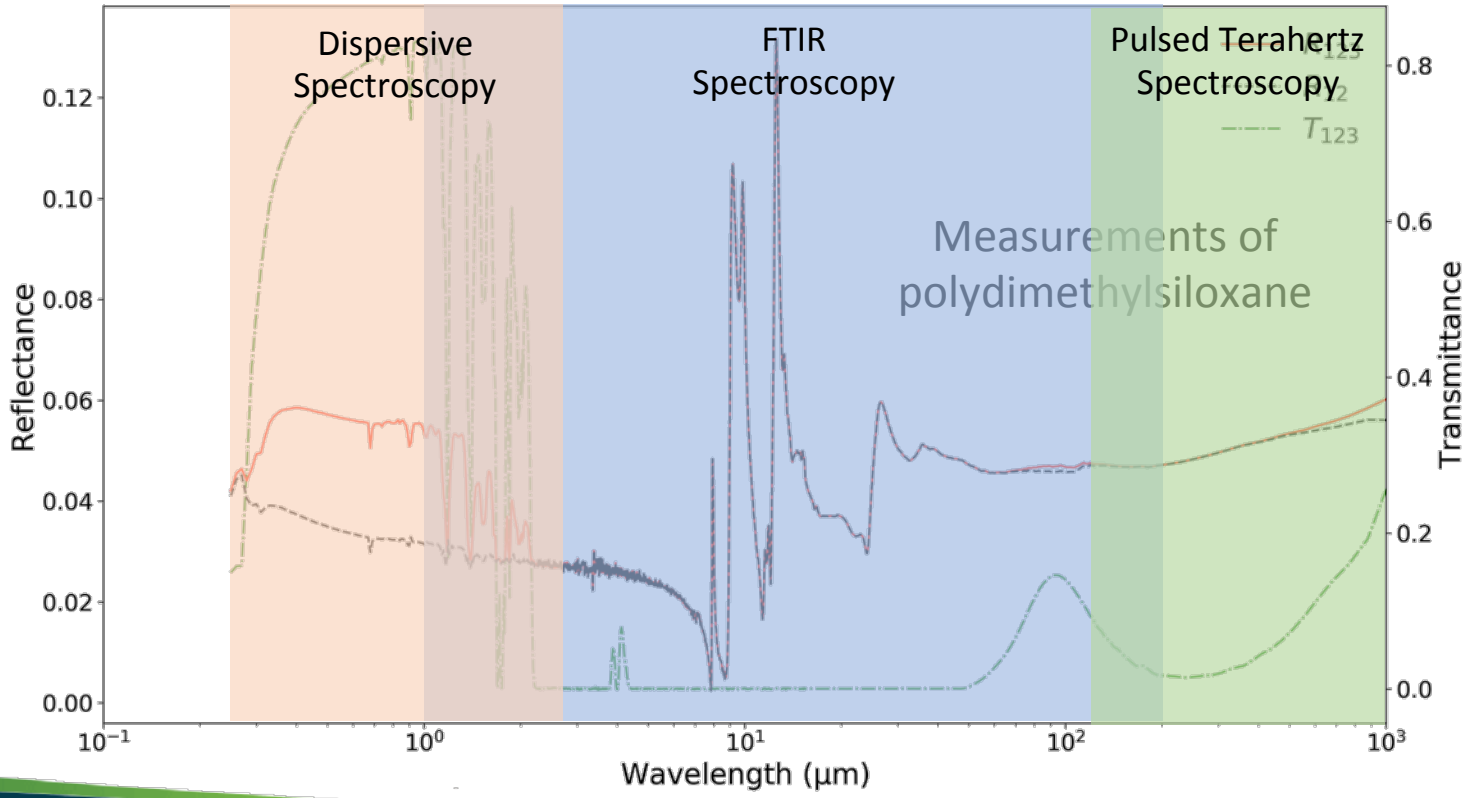
[2]Kou et al., *ACS Photonics* (2017)
[3]Zhou et al., *Nature Sustainability* (2019)

Broadband refractive index measurements

Strategy

- Take RT measurements on thick slab (incoherent internal reflections) of material





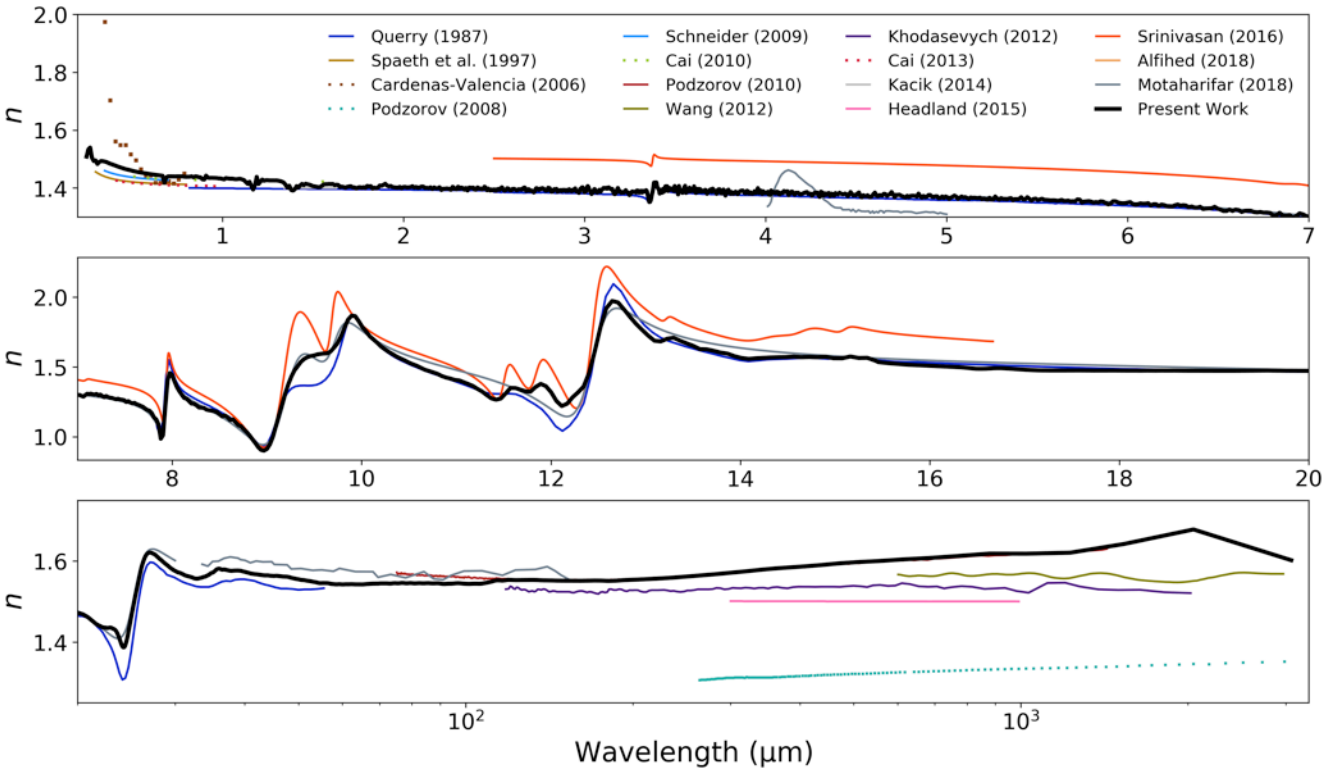
Broadband refractive index measurements

Strategy

- Directly invert $R\downarrow 123$ and $T\downarrow 123$ in regions with non-zero transmittance[4]
- Use Kramers-Kronig relations with $R\downarrow 12$ elsewhere[5]

Current Work

- Error quantification
- Fitting to dielectric function models
- Improved far-infrared FTIR measurements



[4]Nichelatti, *Journal of Optics A* (2002)
[5]Roessler, British Journal of Applied Physics (1965)

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Summary

- NIST continues to pursue expanded and improved measurement capabilities to provide higher accuracy calibration services, standards and data to meet society's needs.
- Areas of recent work include:
  - infrared reflectance standards, infrared index of refraction data
  - emittance and temperature data for selective laser melting in additive manufacturing

Thank-You!