

# New Integrating Sphere for Absolute and Relative Measurements of Directional-Hemispherical Reflectance in the NIST ROSI Facility

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# The Team



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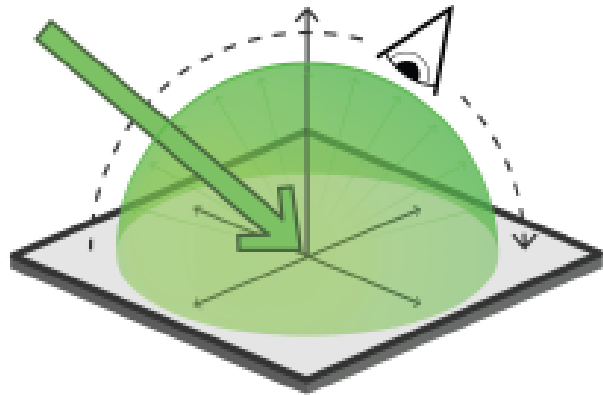
Thom Germer

- Directional/hemispherical reflectance and ROSI sphere
- Uncertainty budget components for relative measurements
- Future work

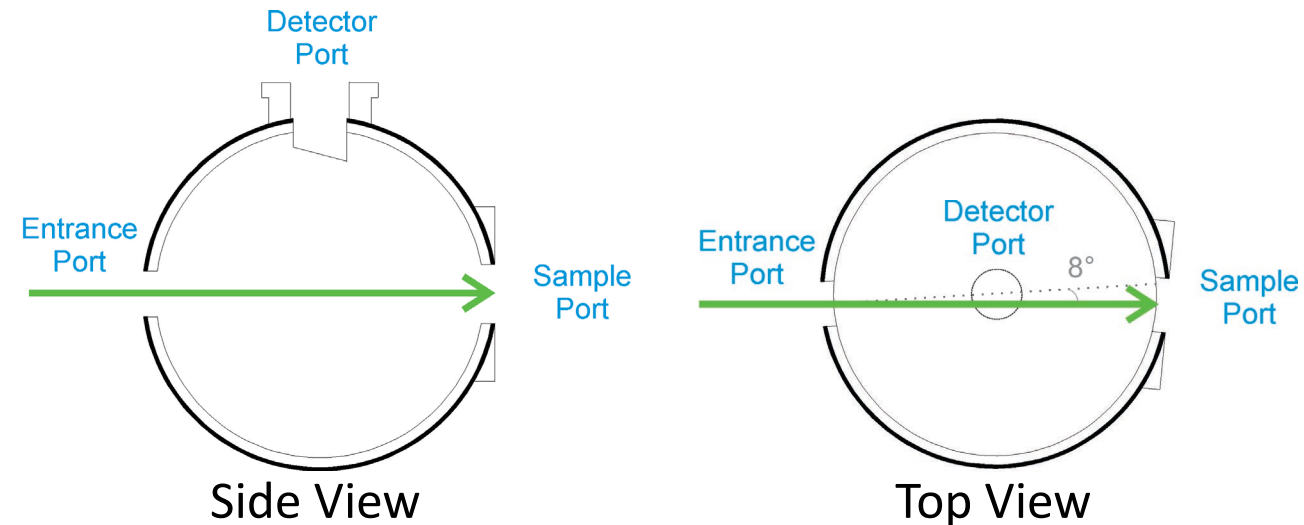
# Directional/hemispherical reflectance and ROSI Sphere

# Directional/hemispherical reflectance

Directional-hemispherical



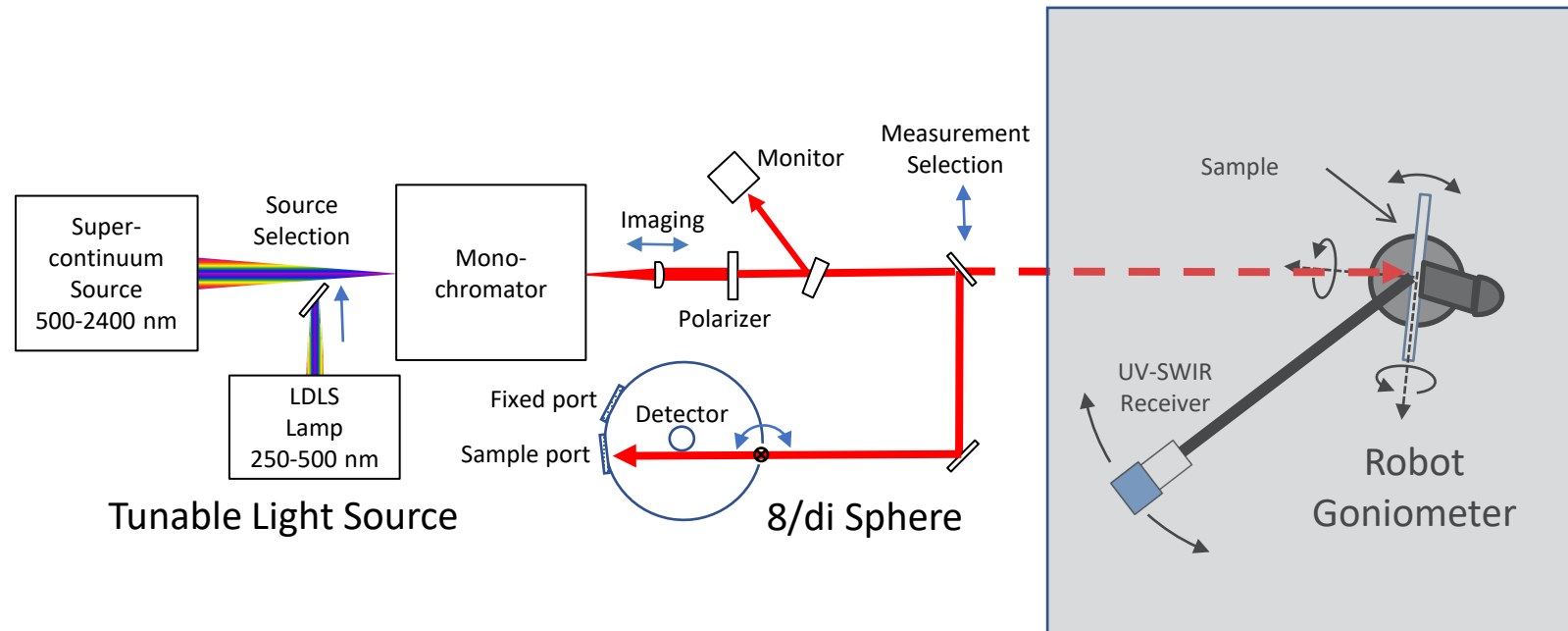
Typical Integrating Sphere Setup



- Most common diffuse reflectance geometry
- Usually 8/di (8° incidence, specular included), also 0/de, 6/di
- Only reflectance geometry for which there is a BIPM Key Comparison
- Historically provided by NIST STARR facility from 250 nm – 2500 nm

- Tunable light source and broadband detector(s)
- Directional/hemispherical relative to a standard:
  1. Measure with a pressed PTFE standard on sample port
  2. Measure with the sample under test on sample port
  3. Ratio

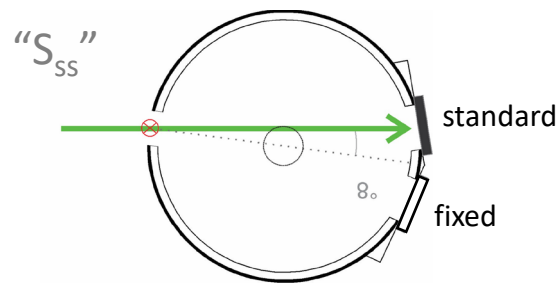
# ROSI Integrating Sphere



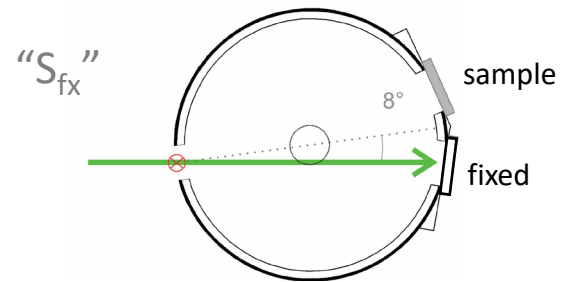
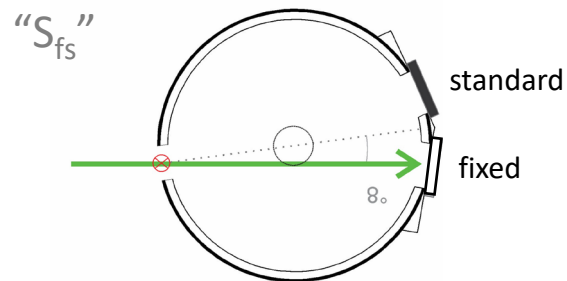
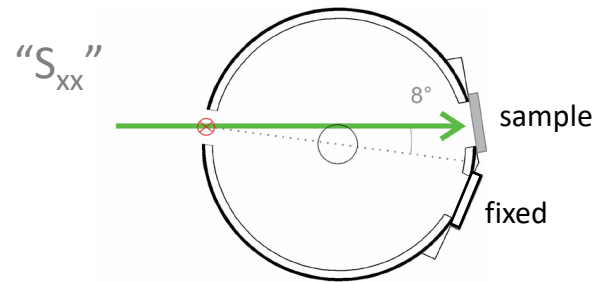
- 290 mm (ID) sintered PTFE sphere, 2 sample ports
- Share tunable light source with robot goniometer, 250 nm – 2400 nm
- Sphere rotates about input port to choose illuminated port
- Second rotation about sphere axis also available for future absolute method
- Top detector port accepts UV-VIS (current) or SWIR (future) detectors

# Measurement Method, Relative Measurements

Step 1: Standard



Step 2: Sample Under Test



- Sequential measurements of standard, sample under test on “sample” sphere port
- “Fixed” port has fixed white sample
- Step 1: measure the pressed PTFE standard and fixed sample
- Step 2: measure the sample under test and fixed sample
- Fixed measurement accounts for any change in sphere efficiency due to sample/std

Combining measurements:

$$R_{simpl} = \frac{S_{xx}}{S_{fx}} \cdot \frac{S_{fs}}{S_{ss}} R_{std}$$

# Uncertainty budget for relative measurements

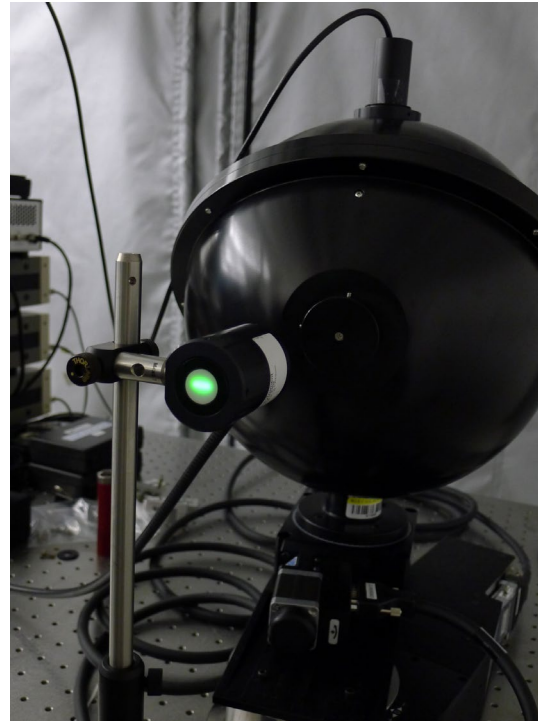
# Uncertainty Components, Relative Measurements

- Reference Standard accuracy
- Wavelength accuracy
- *Out-of-band stray light in source*
- *Fluorescence in sphere or sample*
- Reproducibility
- Other: polarization, source bandwidth, source drift, geometry effects, signal noise

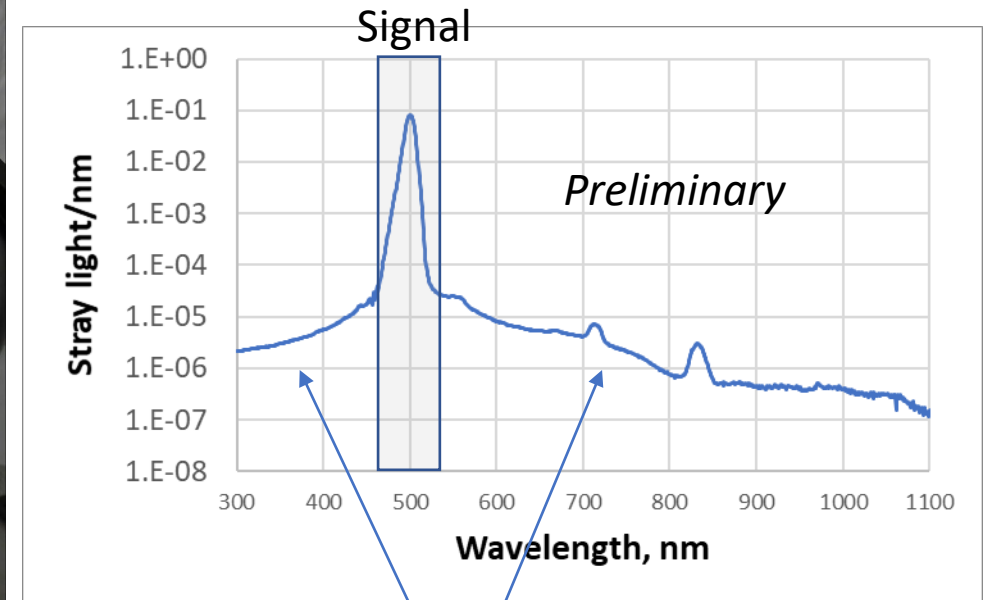
# Out-of-band stray light: measurement



Spectrometer (300 nm – 1100 nm)



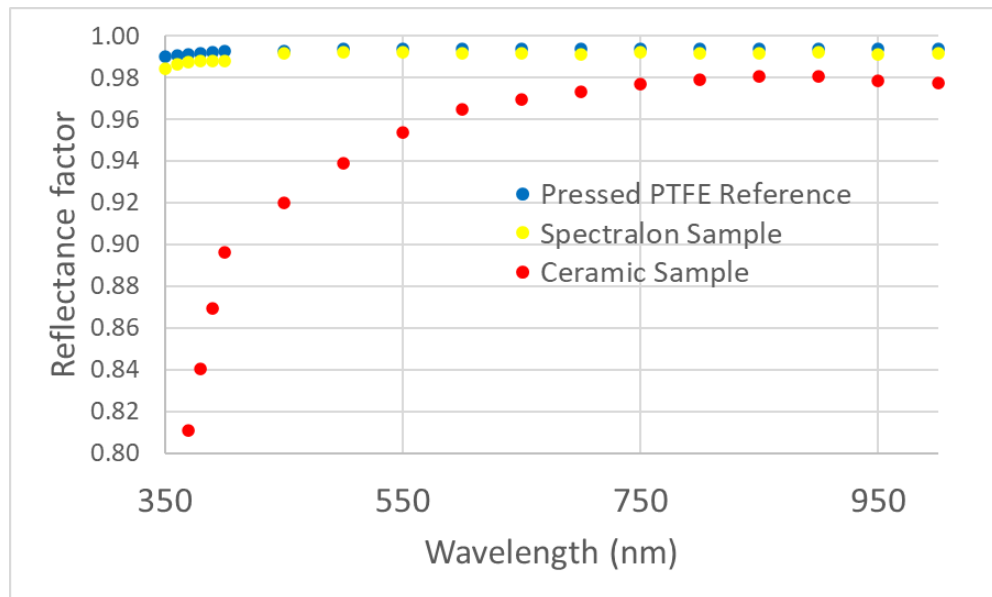
Incident on Irradiance Head



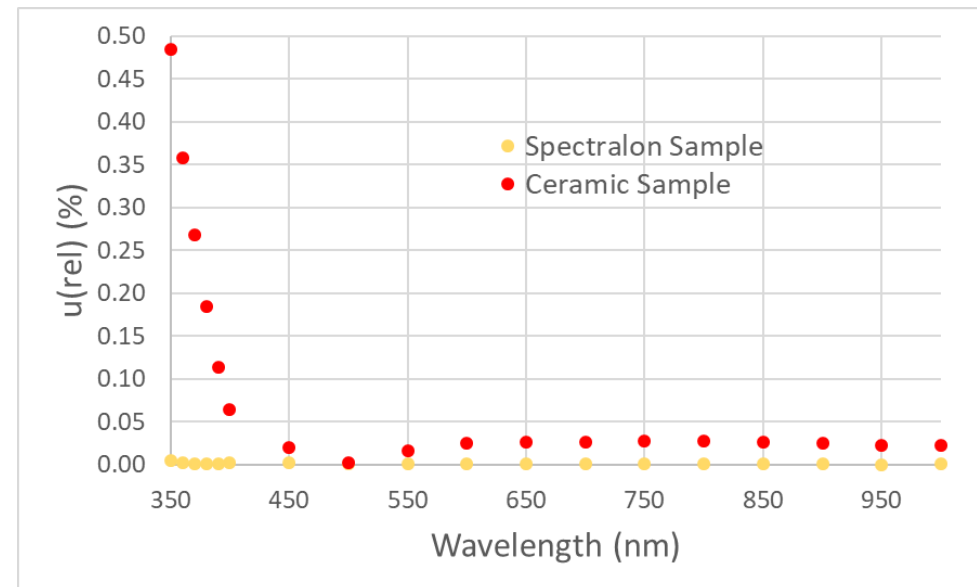
- Measure spectral stray light with spectrometer
- Tune ROSI source to wavelengths from 320 nm to 1030 nm
- At each wavelength, short integration measurement for signal, long integration measurement for stray
- Out-of-band stray light expressed as fractional level/nm relative to the signal light

# Out-of-band stray light: effect

### Example Reflectance Spectra



### Uncertainty from $10^{-5}/\text{nm}$ stray light



$$q_{std} = \frac{SL_{std}}{S_{std}}$$

Fraction of stray light in standard (pressed PTFE reference) signal

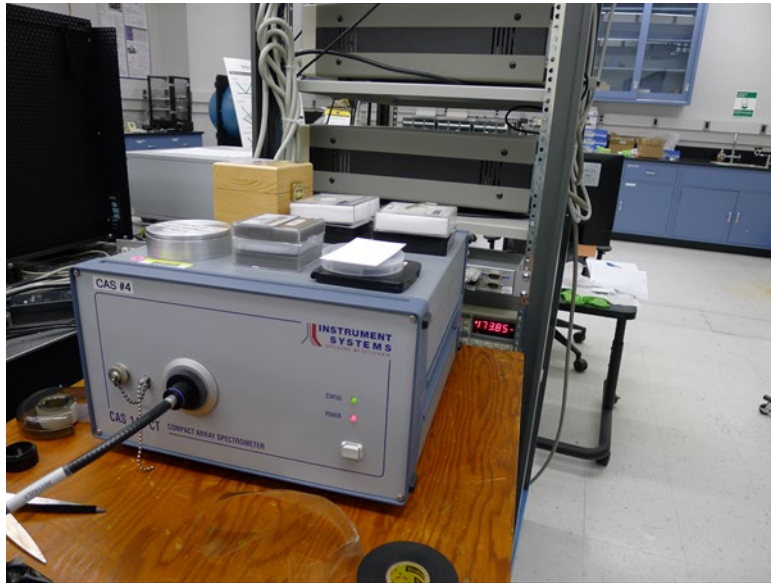
$$q_{smp} = \frac{SL_{smp}}{S_{smp}}$$

Fraction of stray light in sample signal

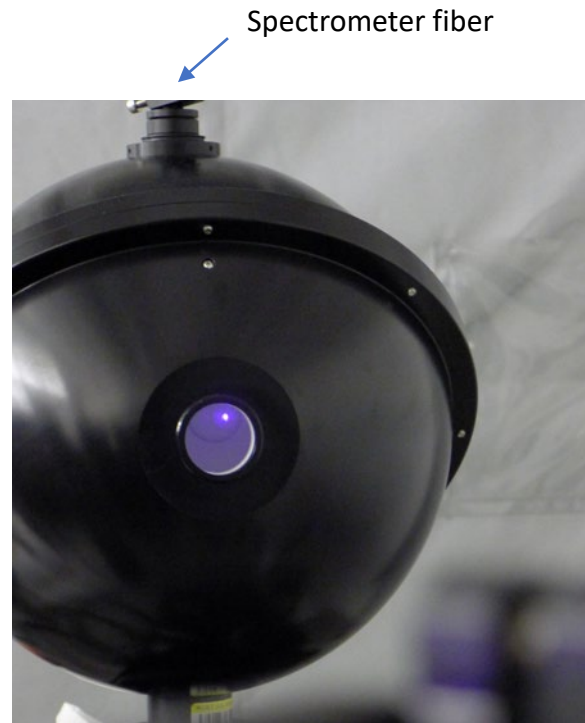
$$u_{rel, stray} = \frac{q_{smp} - q_{std}}{1 + q_{std}}$$

Uncertainty due to stray light

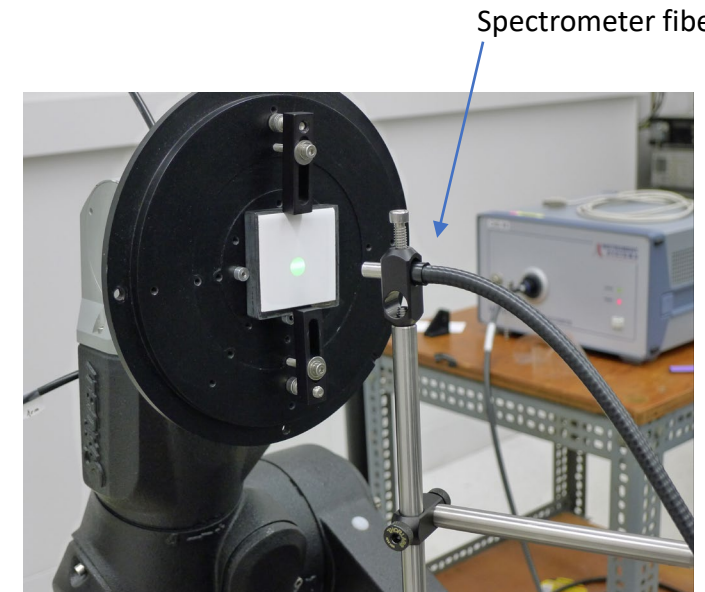
# Sample Fluorescence Measurement



Spectrometer (300 nm – 1100 nm)



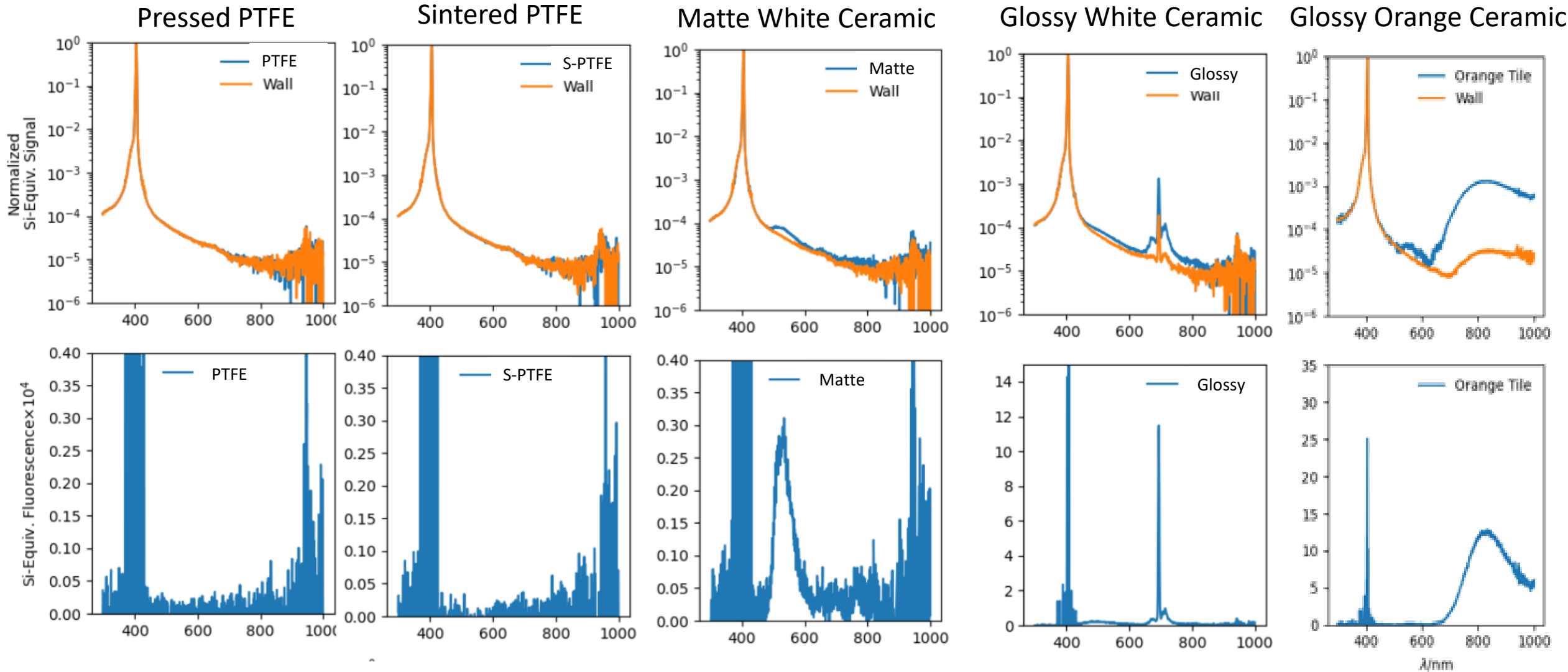
Config 1: 3 mW 405 nm laser excitation, sample on sphere, view fluorescence in sphere



Config 2: tunable excitation from ROSI light source, direct view fluorescence

- Samples often presumed free of fluorescence. However, if fluorescence exists it can add unwanted signal to the reflectance measurement
- Fluorescence measurement requires wavelength discrimination on both illumination wavelength and reflected wavelength; use narrow band input and fiber-coupled spectrometer instead of broadband detector
- Two configurations used: On sphere, direct view

# Configuration 1 Results, selected samples



Note: "Wall" is a fixed pressed PTFE sample

- We measure fluorescence as the excess long-wavelength signal, compared to pressed PTFE. We don't believe there is any fluorescence from pressed PTFE
- Spectrometer is not stray-light corrected; the long-wavelength tail on the pressed PTFE itself is attributed to spectrometer stray light
- Sintered PTFE and the sphere wall itself (not shown here) all show the same spectrum as pressed PTFE and we conclude those samples are not fluorescent
- All ceramic samples gave a measurable level of fluorescence for 405 nm excitation wavelength

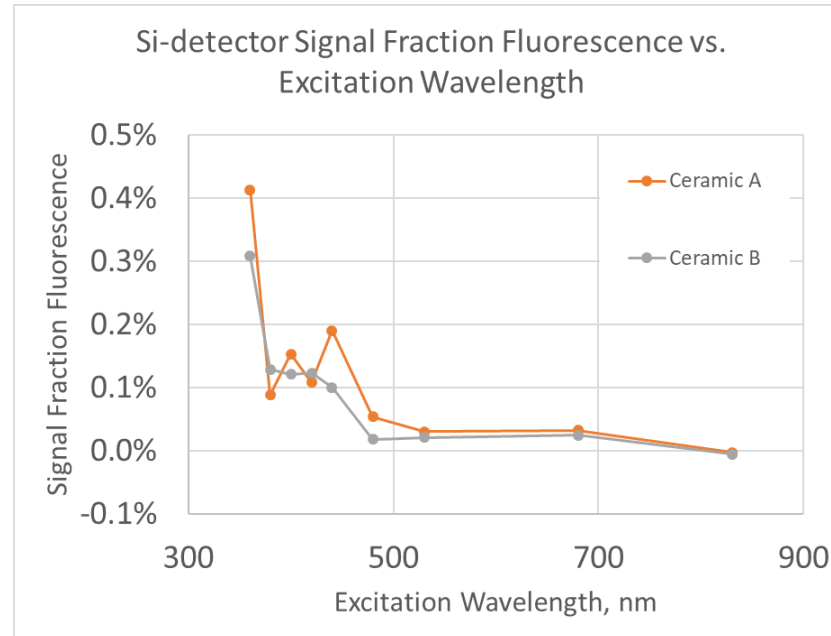
# Fluorescence fraction in signal, 405 nm excitation

Sample type	Si-equivalent fluorescence	Power fraction fluorescence*
Pressed PTFE	NA	NA
Sintered PTFE	< 0.01%	< 0.01%
Matte Ceramic	0.12%	0.07%
Glossy ceramic	0.38%	0.20%
Glossy orange ceramic	20.1%	8.3%

\*the Power fraction fluorescence is different from the Si-equivalent. Power fraction is not weighted by the Si detector responsivity. Power fraction can be considered the true fluorescence level integrated from roughly 450 nm to 1000 nm, relative to reflected signal at the 405 nm incident wavelength.

# Fluorescence Results vs. Wavelength, Config 2

Fraction of signal from fluorescence vs. excitation wavelength, ceramic samples



- Configuration 2 allowed measurement of fluorescence relative to pressed PTFE, vs. excitation wavelength
- Noisier data than configuration 1, but results consistent with configuration 1
- Fluorescence fraction vs. excitation wavelength for 2 ceramic samples plotted
- Leads to error in measured reflectance due to detection of fluorescence by broadband detector during reflectance measurement

# Example Relative Uncertainty Components

Element	Example Ceramic < 420 nm	Sintered PTFE (UV-VIS)
Reference standard	0.2%	0.2%
Wavelength Accuracy	0.6% at 360 nm 0.1% at 420 nm	<0.01%
Stray light	0.36% at 360 nm 0.04% at 420 nm	<0.02%
Fluorescence	0.32% at 360 nm 0.08% at 420 nm	<0.01%
Reproducibility	0.06%	0.06%
Other	0.08%	0.04%
K=2 relative uncertainty	1.6% at 360 nm 0.52% at 420 nm	0.43%

- Sintered PTFE uncertainty dominated by uncertainty of the reference standard
- Wavelength accuracy 0.7 nm  $k=1$ . If reflectance changes rapidly with wavelength (ceramic < 420 nm), this is a significant component
- Spectral stray light and sample fluorescence were also found to be significant for ceramic < 420 nm

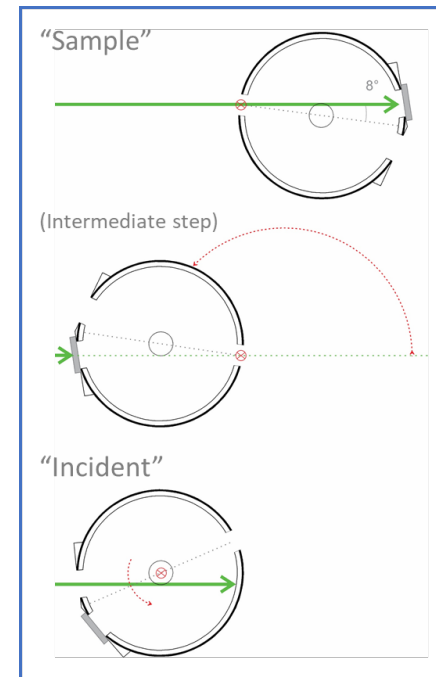
Future work

- Absolute directional/hemispherical measurements using integrating sphere and goniometric methods
- Expansion of wavelength range into the SWIR to 2400 nm
  - ROSI light source already supports this range
  - Extended InGaAs detector to be installed on sphere



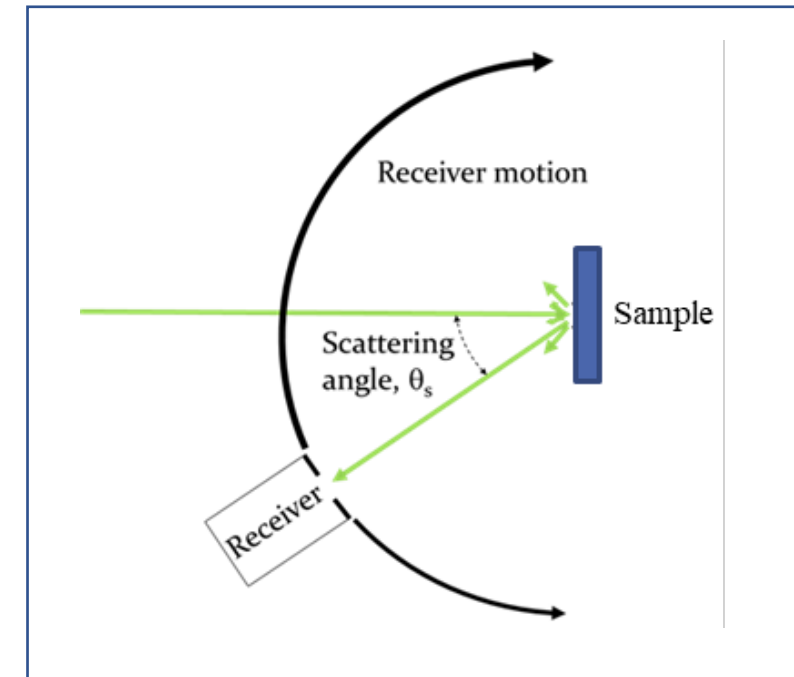
## Methods for Absolute Measurements

### Hanssen et al



- Characterize sphere wall uniformity
- Use knowledge of sample BRDF

### Goniometric



- Use BRDF capability of ROSI robotic arm setup
- Slow, requires many measurement points

*Both methods require careful evaluation of corrections/uncertainties*

# Research Opportunities

- **NRC Postdoctoral Program**
  - Federal employee with 2-year appointment, roughly \$72,000/yr stipend and travel
  - [sites.nationalacademies.org/PGA/RAP](https://sites.nationalacademies.org/PGA/RAP)
- **Other Programs**
  - NIST Professional Research Experience Program (PREP)
  - Summer Undergraduate Research Fellowship Program (SURF)
  - Visiting Scientist and Guest Research Positions



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*Thank you!*