

Towards a new diffuse reflectance scale at NRC

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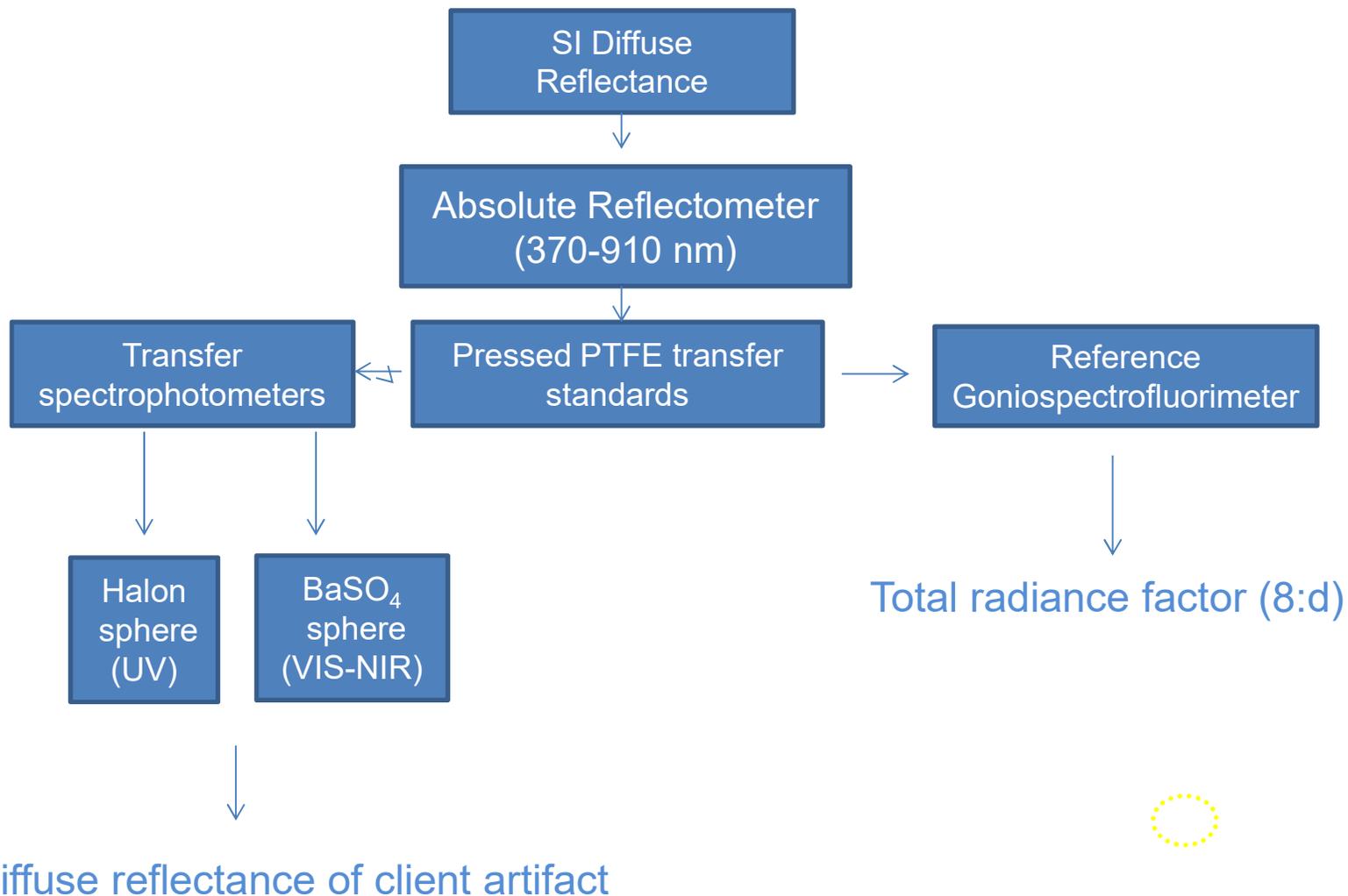
Outline

1. Realizing a diffuse reflectance scale
2. The Sharp-Little method for absolute reflectance
3. Design objectives and solutions
4. Some challenges due to integrating sphere non-idealities

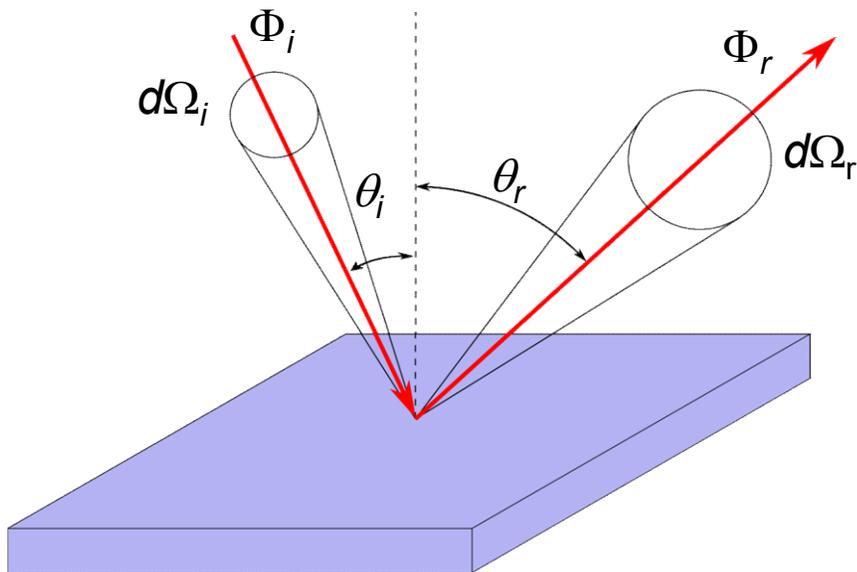
Applications of diffuse reflectometry

- Colorimetry
 - Paper
 - Textiles
 - Paint
- Photovoltaics
- Spectroscopy
 - Materials science, biology, geology, pharmaceuticals,....
- Commercial instruments widely available

Diffuse reflectance traceability chain at NRC



Diffuse Reflectance



$$\rho(\Omega_i, \theta_i, \Omega_r, \theta_r) = \frac{\Phi_o(\Omega_i, \theta_i, \Omega_r, \theta_r)}{\Phi_i(\Omega_i, \theta_i)}$$

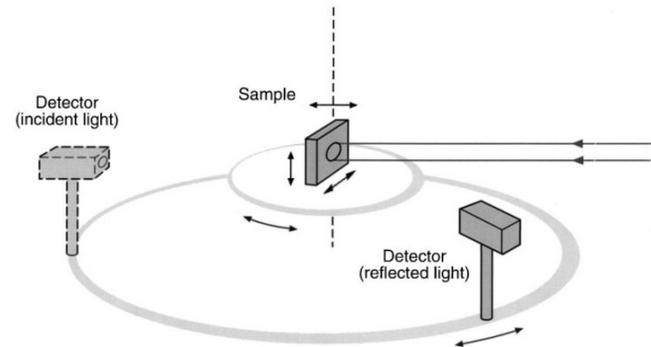
$$\Omega_i = 2\pi, \theta_o \sim 0^\circ \rightarrow (d; 0^\circ)$$

$$\theta_i \sim 0^\circ, \Omega_o = 2\pi \rightarrow (0^\circ; d)$$

} CIE
recommended

How to measure absolute reflectance?

- Goniorefectometric methods (NPL, HUT/MIKES)



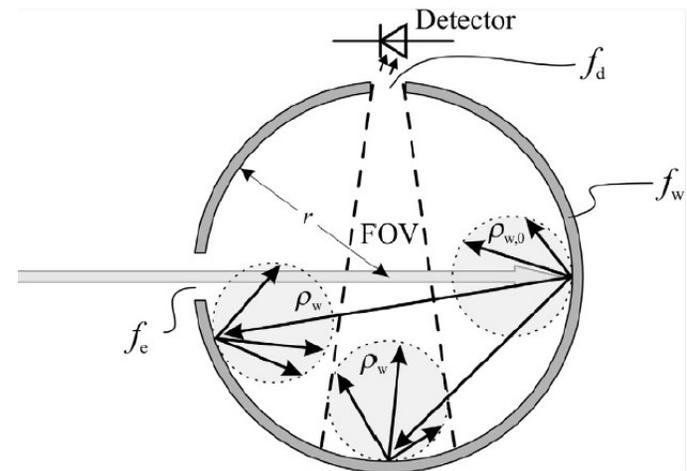
- Integrating sphere-based methods:

3rd Taylor

Sharp-Little (NRC, KRISS, NIM)

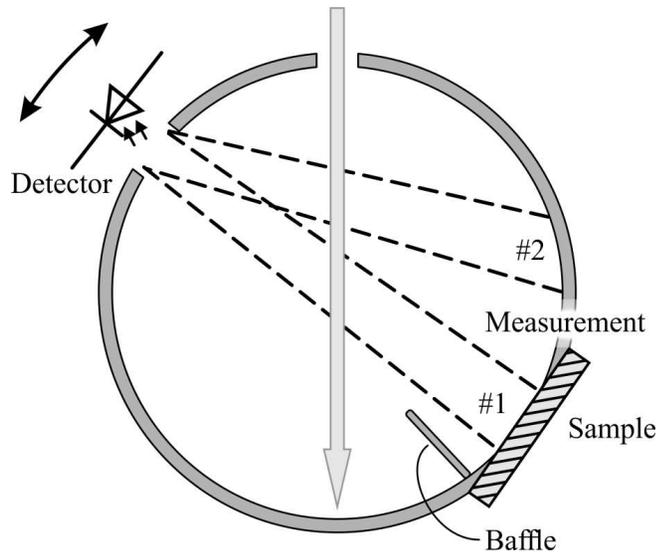
Double sphere/van den Akker (NIST)

Korte-Schmidt (PTB)



CIE Publication 44:1979

The Modified Sharp-Little method



Realizes $(d;0^\circ)$
absolute reflectance scale

Modified method corrects
for sphere openings,
average wall reflectance

Total sphere area: a_0

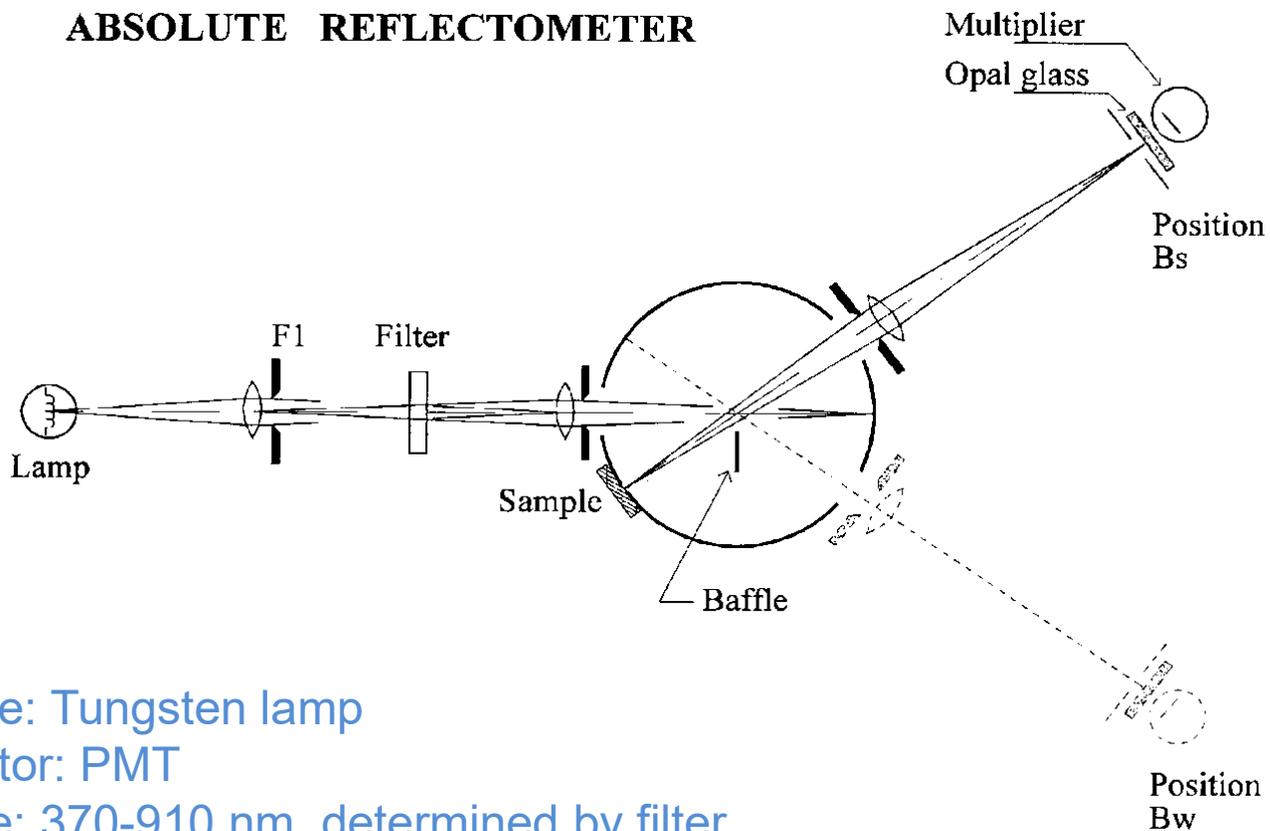
Wall area: a_1

Sample area: a_2

$$\rho_S = \frac{B_1}{B_2} \frac{a_0}{a_1 + a_2}$$

The NRC Absolute Reflectometer

ABSOLUTE REFLECTOMETER



Source: Tungsten lamp

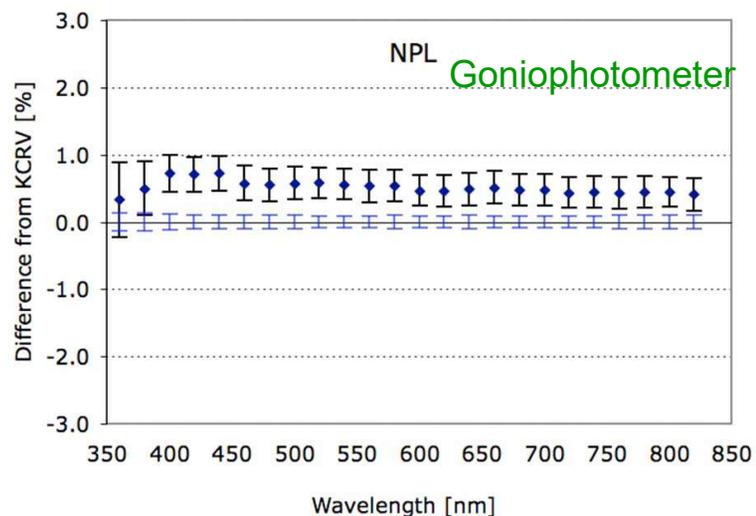
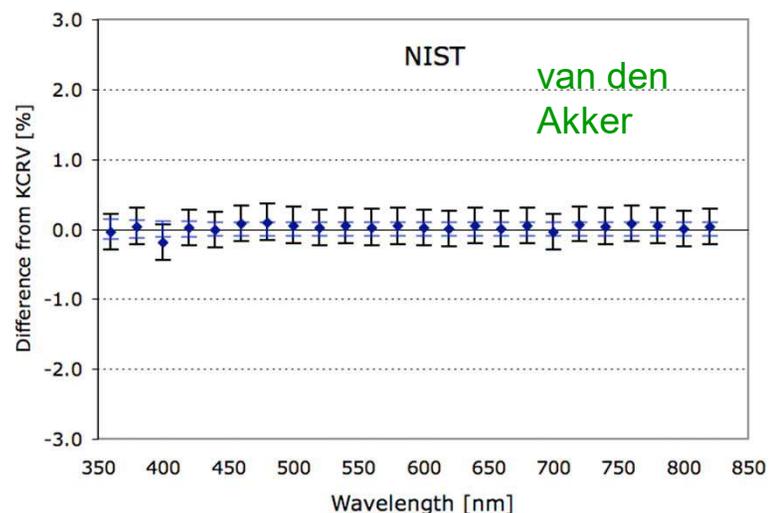
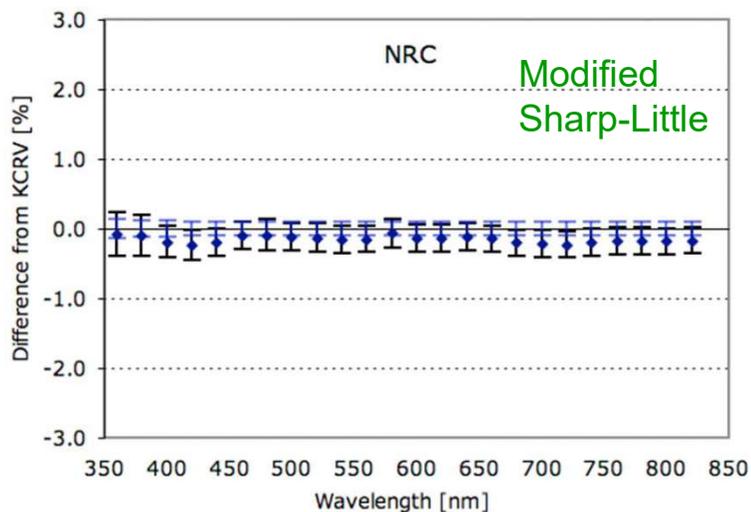
Detector: PMT

Range: 370-910 nm, determined by filter

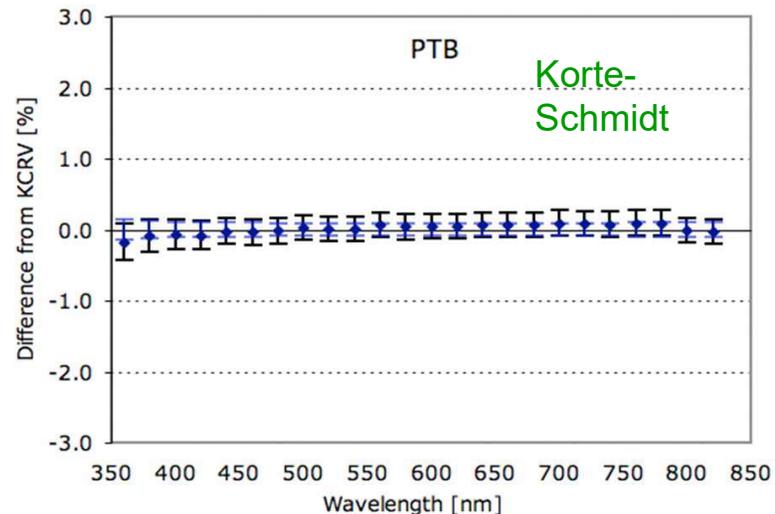
Bandwidth: ~ 10 nm

Sphere coating: BaSO₄

CCPR K5 Key Comparison for Spectral Diffuse Reflectance



Systematic difference using goniophotometric method



Why the Sharp-Little method?

- Established principle
- Comparatively straightforward
- NRC has existing expertise
- Robust (present instrument has operated for 40+ years)
- Demand from paper industry

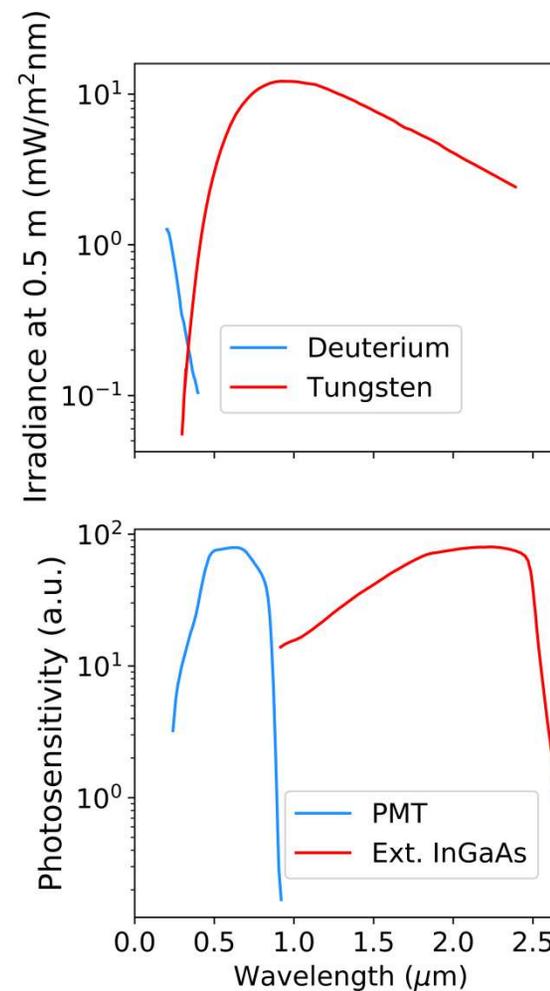
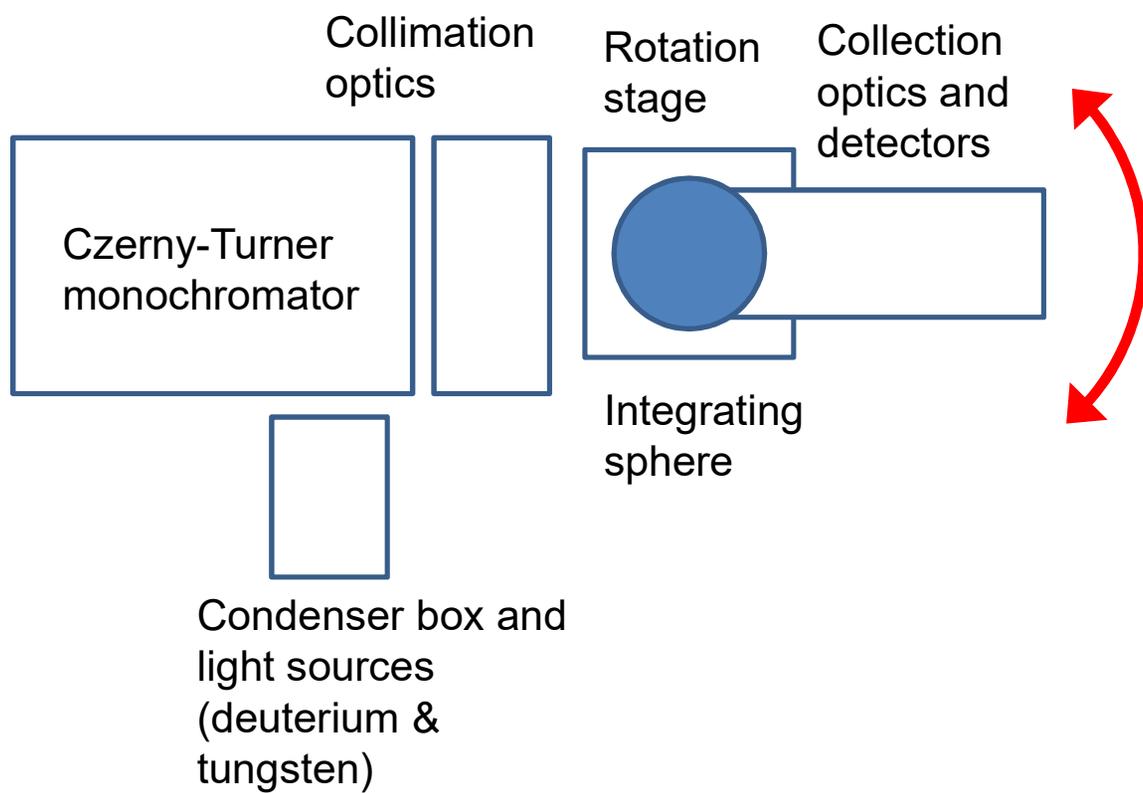
Design Objectives

1. Extend spectral range (250 to 2500 nm)
2. Resolution: 5 nm
3. Full automation
4. Reduce errors due to sphere non-idealities.

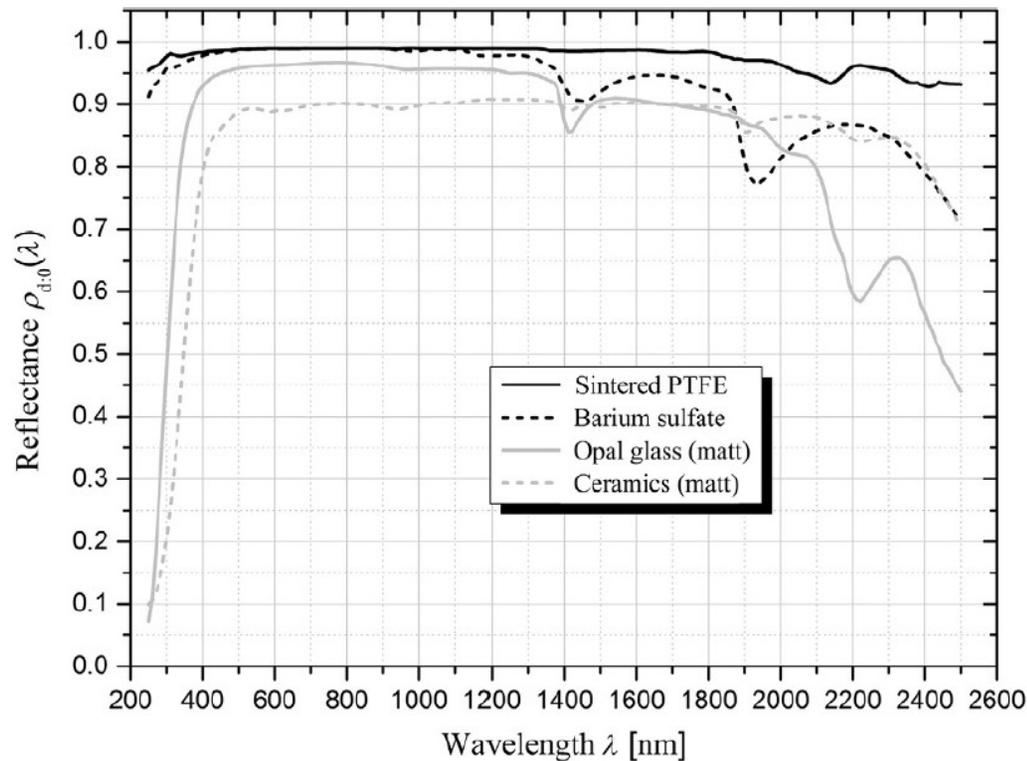
Solutions:

1. Sintered PTFE sphere
2. Multiple source/detectors
3. Monochromator-based system

Schematic design



Sphere material: Sintered PTFE



Sphere multiplier:

$$M = \frac{\rho}{1 - \rho f}$$

PTFE: Enhanced $\rho(d;0^\circ)$
in UV and IR

Integrating sphere challenges

Some assumptions:

- Perfect spherical geometry
- Sample recess is negligible
- Inner surface is Lambertian
- Inner surface is optically homogeneous

$$\rho_S = \frac{B_1}{B_2} \frac{a_0}{a_1 + a_2}$$

Integrating sphere challenges

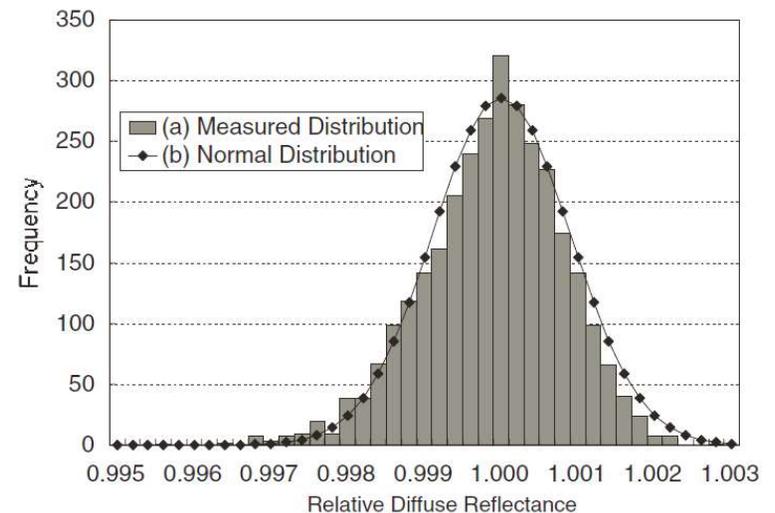
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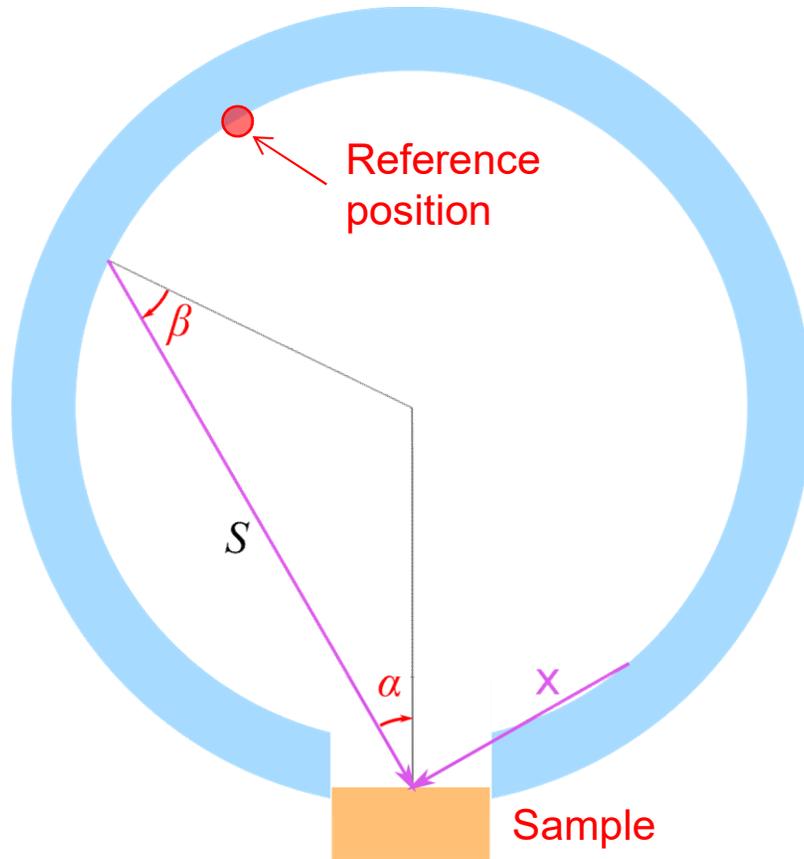
For PTFE:

- 10 mm thickness needed for opacity
- reflectance varies by $\pm 0.2\%$



H. Shitomi *et al.*, Metrologia 40 S185 (2003)

Non-spherical geometry & sample recess



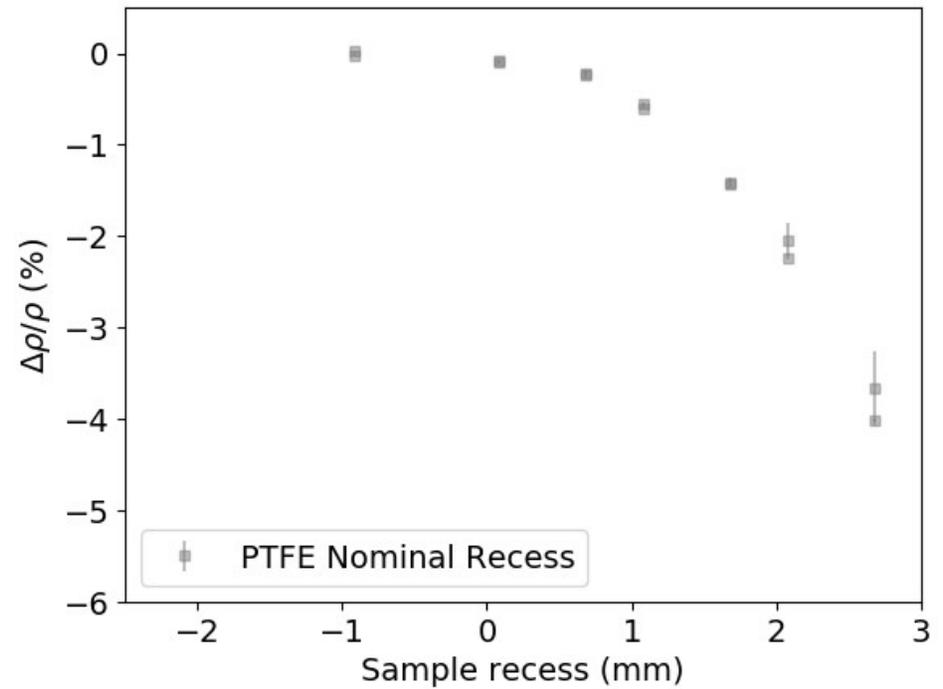
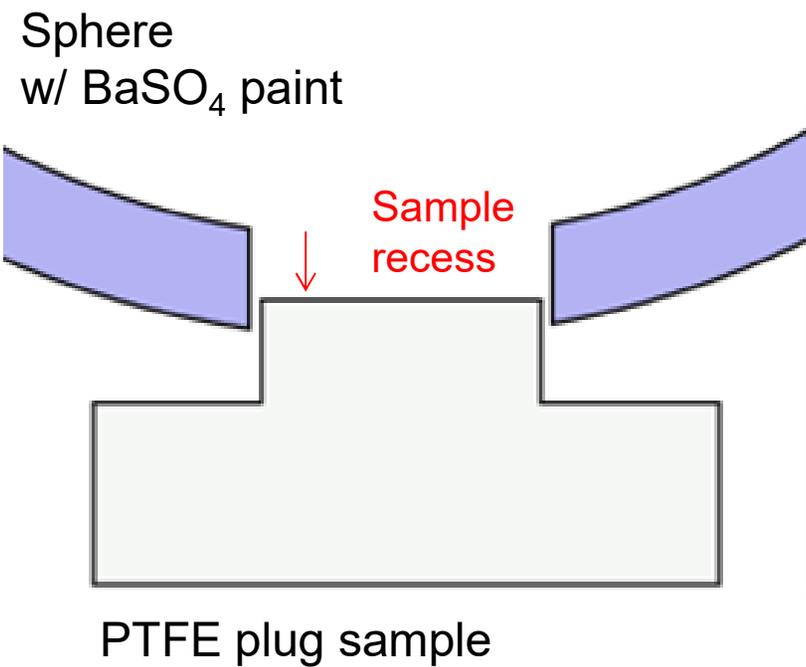
Coefficient of radiative transfer

$$F \propto \frac{\cos \alpha \cos \beta}{S^2}$$

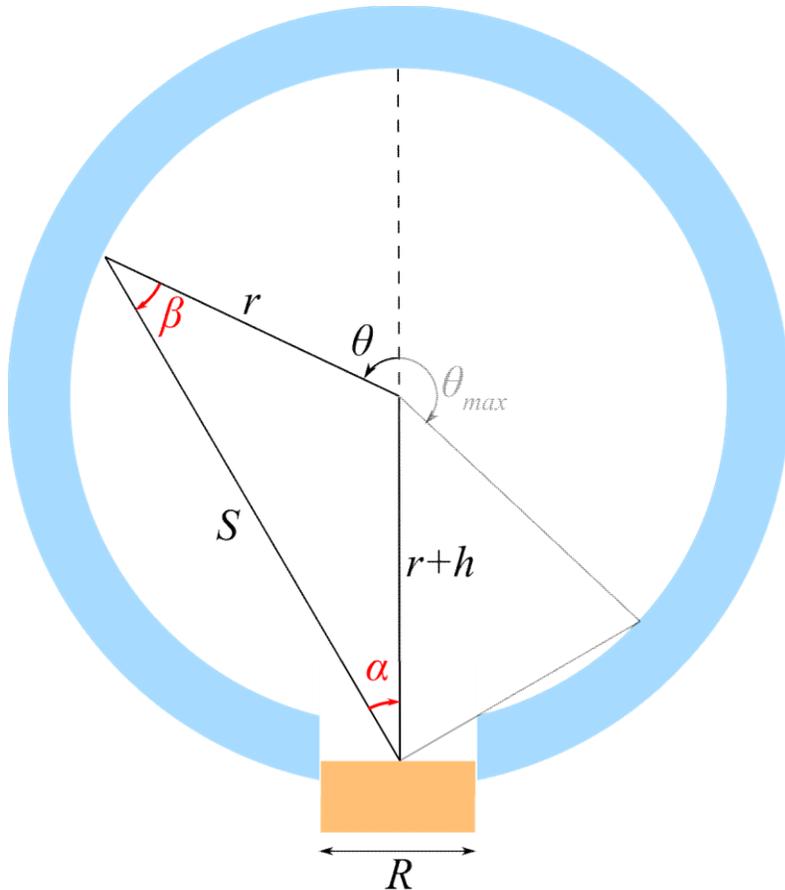
Reduction in flux reaching sample:

1. Increase of S
2. High AOI rays are blocked
3. No 'self-illumination' by flat sample

Example: PTFE



Non-spherical geometry & sample recess



Coefficient of radiative transfer

$$F(\theta, h) \propto \frac{\cos \alpha(\theta, h) \times \cos \beta(\theta, h)}{S(\theta, h)^2}$$

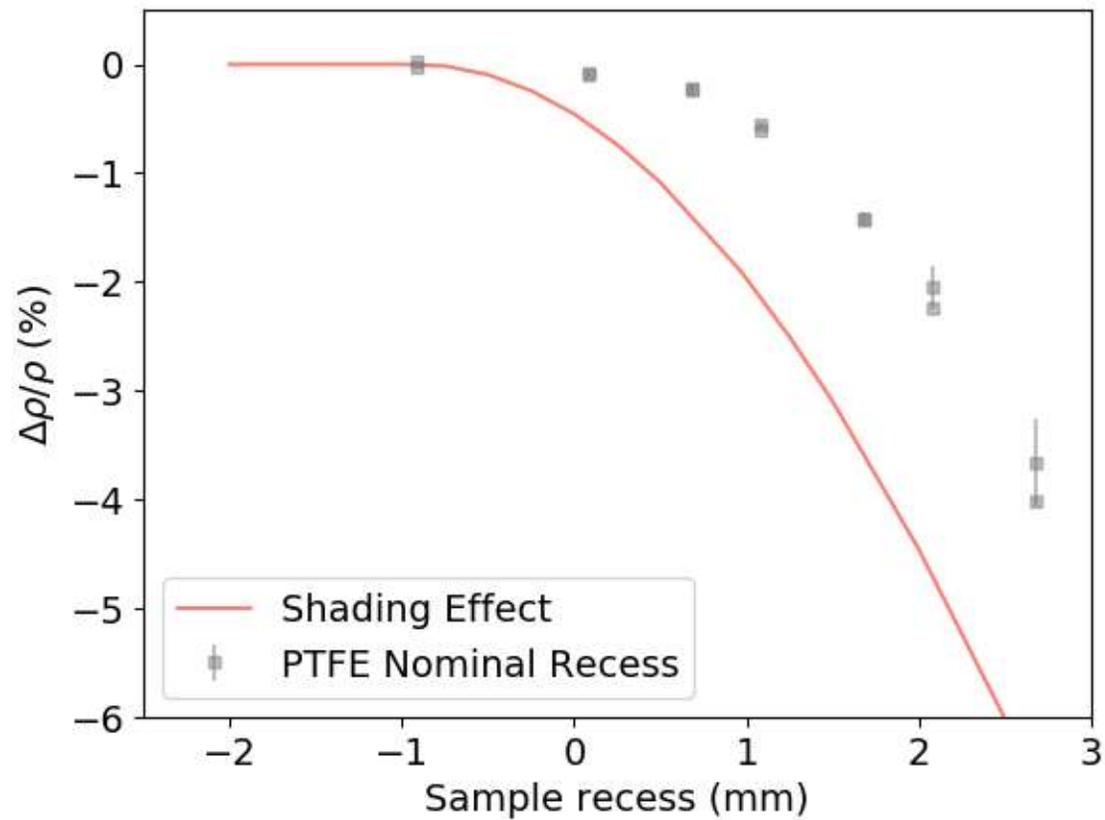
Total sample irradiance

$$E(h) \propto \int_0^{\theta_{\max}} d\theta F(\theta, h) \sin \theta$$

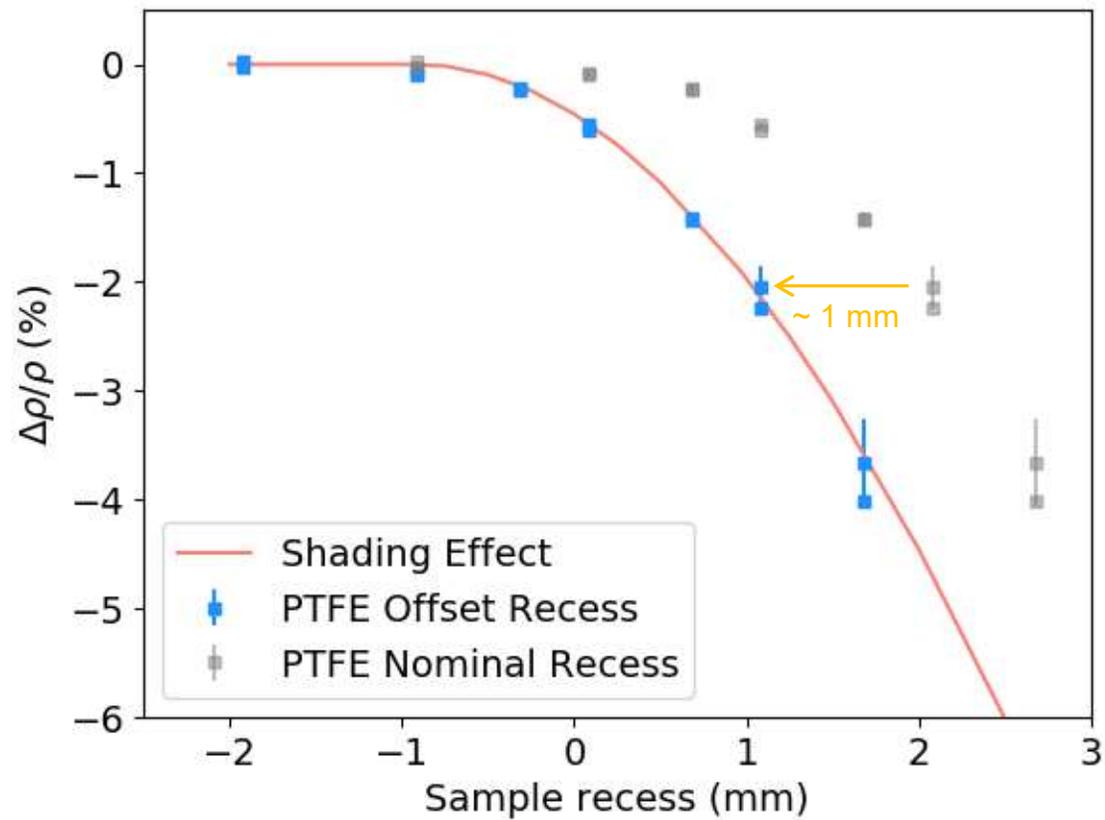
Fractional error in reflectance:

$$\frac{\Delta \rho(h)}{\rho_{\max}} = \frac{E(h)}{E_{\max}} - 1$$

Non-spherical geometry & sample recess

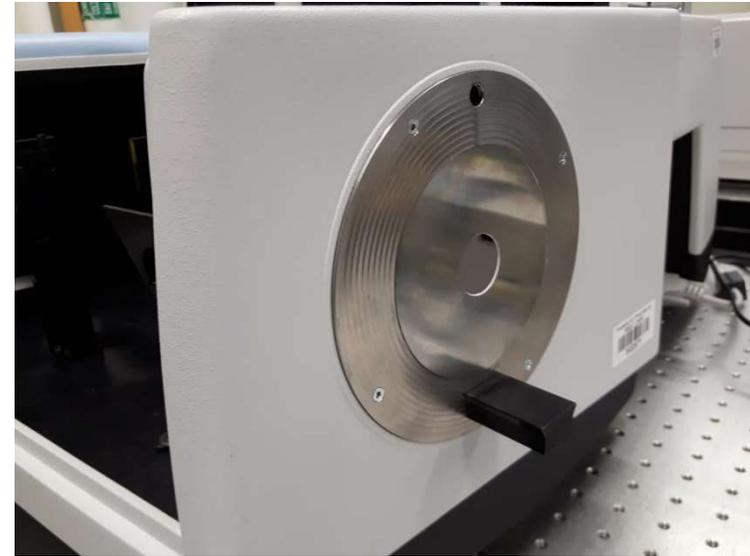
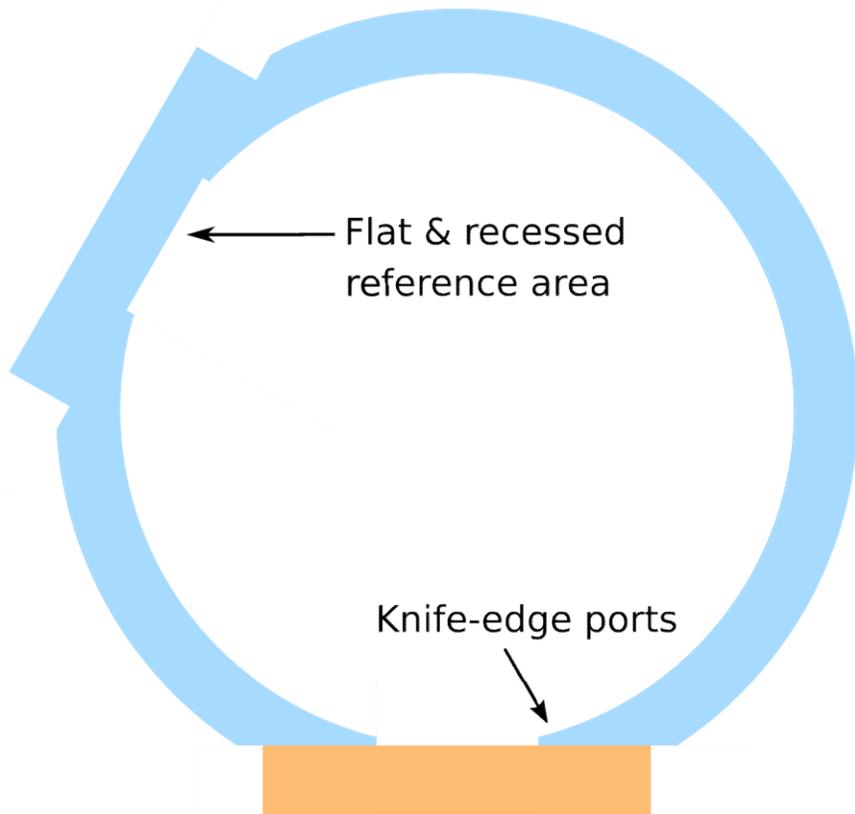


Non-spherical geometry & sample recess

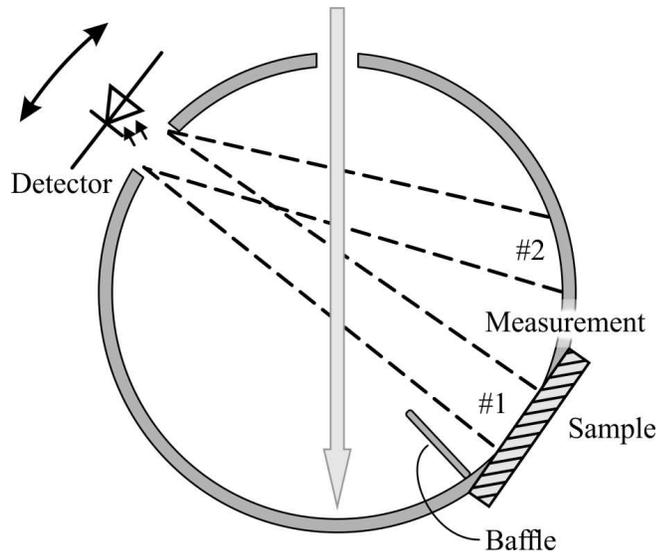


*Offset may be due to roughness/translucency of port edge

Sphere design



Sphere inhomogeneity



Total sphere area: a_0

Wall area: a_1

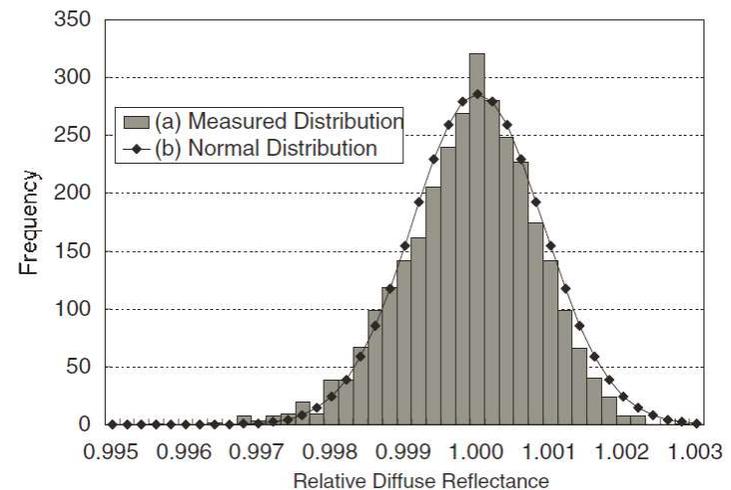
Sample area: a_2

Reference wall location reflectance: ρ_2

Average sphere wall reflectance: ρ_{ave}

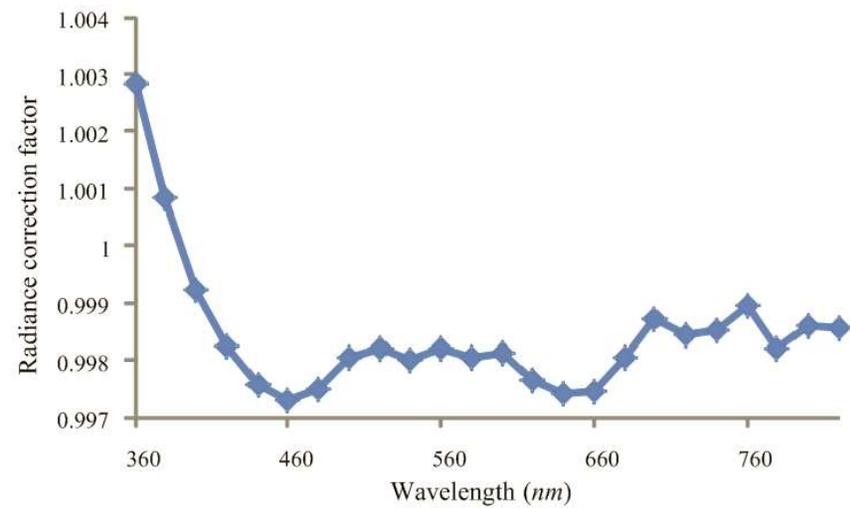
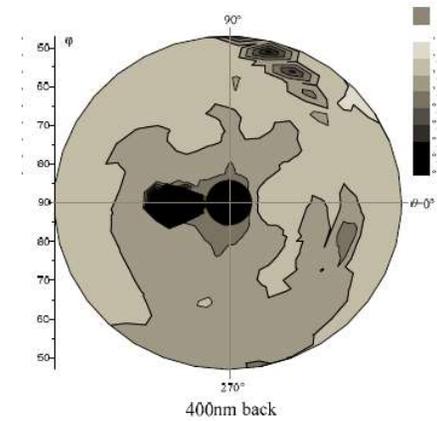
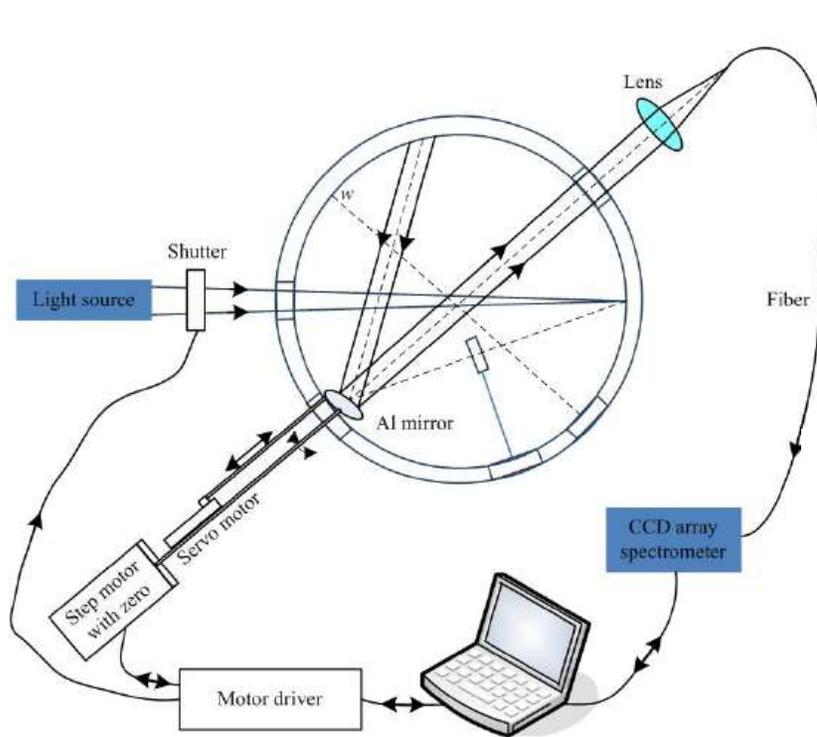
$$\rho_s = \frac{B_1}{B_2} \frac{a_0}{a_1 + a_2} \frac{\rho_2}{\rho_{ave}}$$

Geometric term \downarrow
 Inhomogeneity \uparrow



H. Shitomi *et al.*, Metrologia 40 S185 (2003)

Sphere inhomogeneity scanner



Sun & Ma, Proceedings of SPIE, 2014

Future Work

- Optical & mechanical design (in progress)
- Monochromator & source installation (Fall)
- Integrating sphere & rotation stage (Spring 2018)
- Sphere scanning apparatus
- Participation in CCPR K5 2017 key comparison on Spectral Diffuse Reflectance (long term)

Summary

- A new diffuse reflectance scale is being developed at the NRC
- The new scale will be based on the Sharp-Little Method
- The new system will offer :
 - Improved spectral range and resolution
 - Automation
 - Reduction of errors due to sphere non-ideality.

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Thank you!

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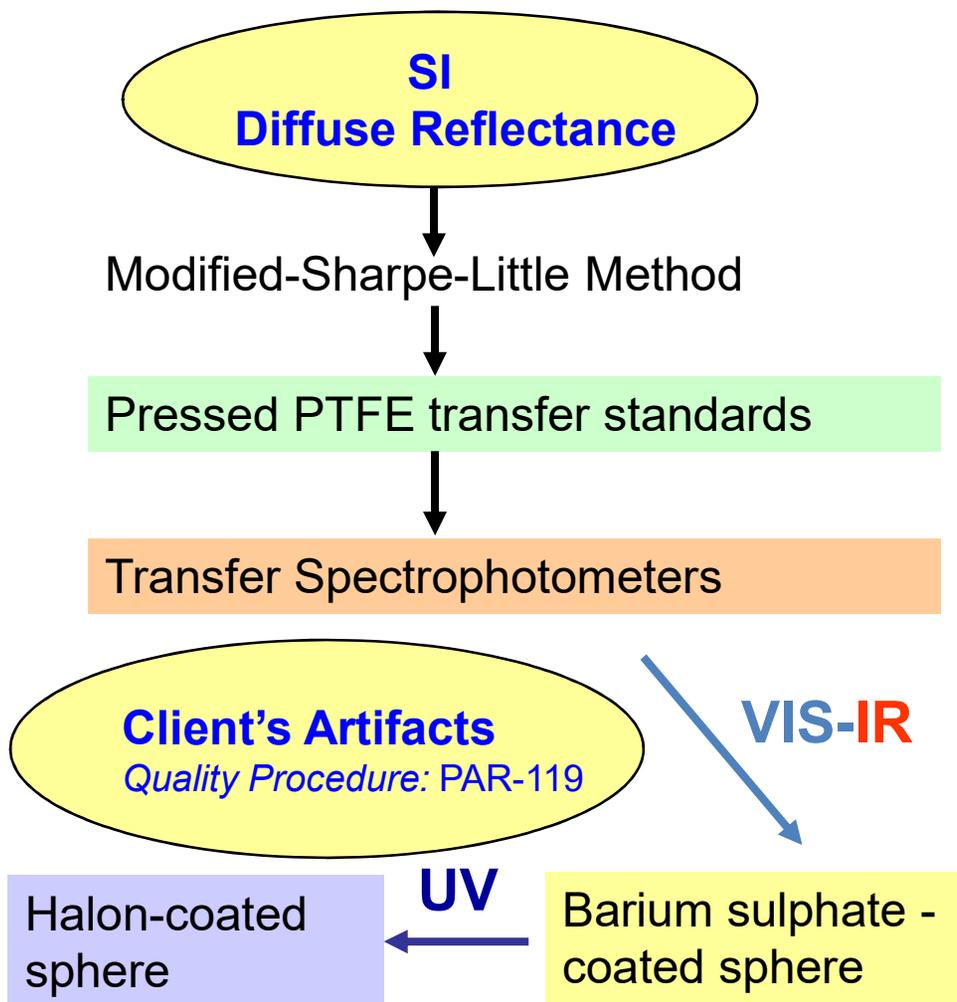


Design Objectives

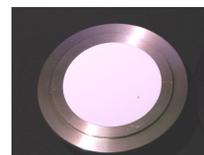
Must meet geometric requirements of ISO 2469 *Paper, board, and pulps*
 - *Measurement of diffuse radiance factor (diffuse reflectance factor)*

Requirement	ISO 2469	NRC Absolute Reflectometer	NRC transfer Instruments
Illumination	Diffuse	Diffuse	Near-normal
Viewing	Normal	Normal	Diffuse
Internal Diameter	≥ 150 mm	200 mm	150 mm
Measured test area:			
Shape	Circular	Circular	Rectangle
Dimensions	28 ± 3 mm	28 mm	8×15 mm ²

Traceability chain



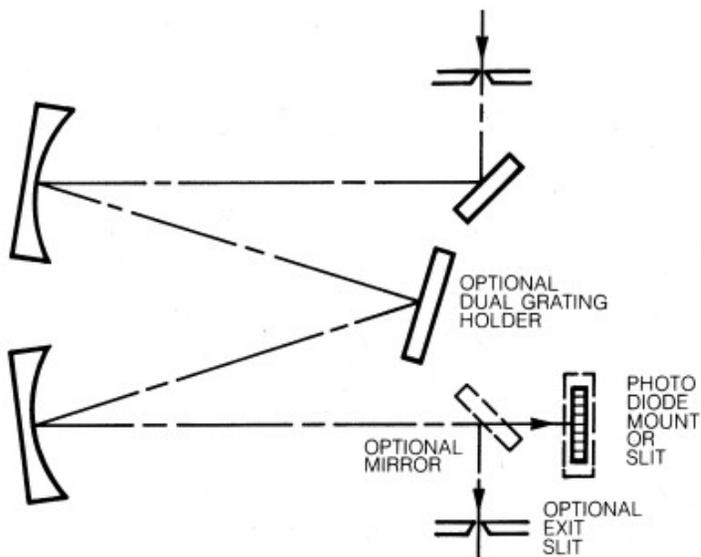
absolute measurements



relative measurements

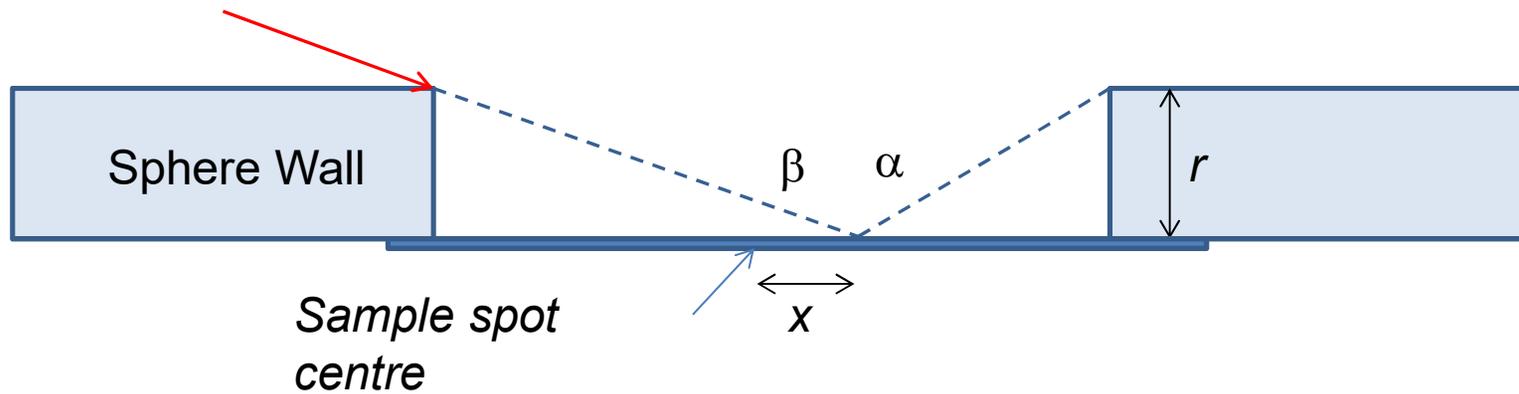


Monochromator: Czerny-Turner-type



- Low stray light levels (1 part in 100, 000)
- Compact
- Robust
- Dual grating:
 - 1200 g/mm (185 – 1300 nm)
 - 600 g/mm (185 – 2600 nm)

Finite spot

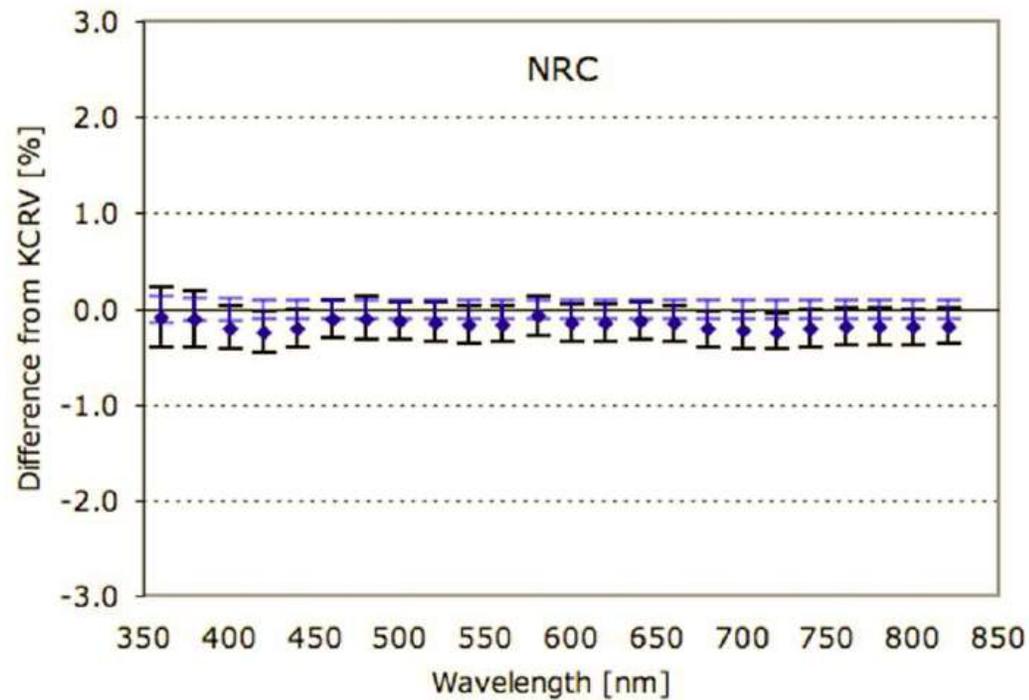


$$\cos \beta = r / \sqrt{r^2 + (R + x)^2}$$

$$\cos \alpha = r / \sqrt{r^2 + (R - x)^2}$$

CCPR K5 Key Comparison for Spectral Diffuse Reflectance

Sample: Spectralon



KCRV: Key Comparison Reference Value

Uncertainty Budget

Table 2b NRC Uncertainties in Spectral Diffuse Reflectance ($k=1$) for the Spectral Range 350 nm to 390 nm.

<i>Uncertainty Component</i>		<i>Type (A or B)</i>	<i>Standard Uncertainty (k=1)</i>	<i>Relative Uncertainty in Reflectance Factor</i>
Signal to Noise		A	0.08 %	0.08 %
Instrument Stability	Short-term	A	0.04 %	0.04 %
Instrument Stability	Long-term	A	0.05%	0.05 %
Wavelength		B	0.15 nm	<0.01 % (S#n); 0.10% (C#n)
Detector Linearity		B	<0.01 %	<0.01 %
Modified Sharp-Little Method				
R > 0.85		B	0.05 %	0.05 %
0.75 < R <= 0.85		B	0.10 – 0.05 %	0.10 – 0.05 %
0.60 < R <= 0.75		B	0.15 – 0.10 %	0.15 – 0.10 %