NIST Physics Laboratory

OPTICAL TECHNOLOGY DIVISION

Hemispherical-directional Integrating Sphere for High Temperature Reflectance Factor Measurement

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Outline

I. Application: Non-Contact Method for Sample Temperature

II. Hemispherical-directional Reflectance Factor Sphere Design

III. Monte Carlo Modeling and Optimization of Sphere Design

IV. Constructed Sphere & HDRF Performance Results

V. Application Measurement Results: Emittance & Temperature

VI. Conclusions

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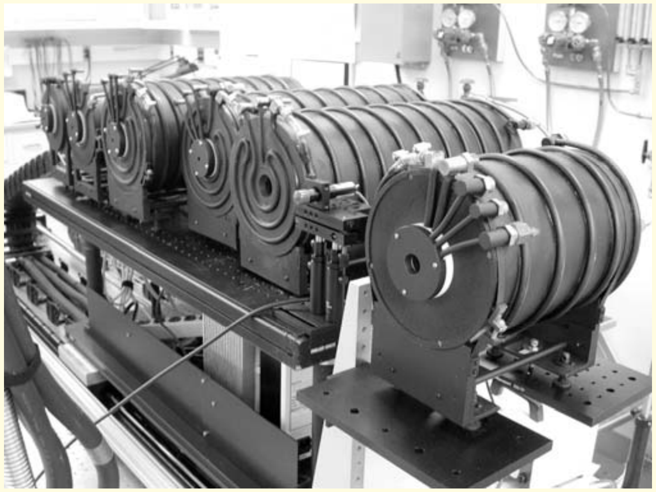
New Capability: Infrared Spectral Emittance



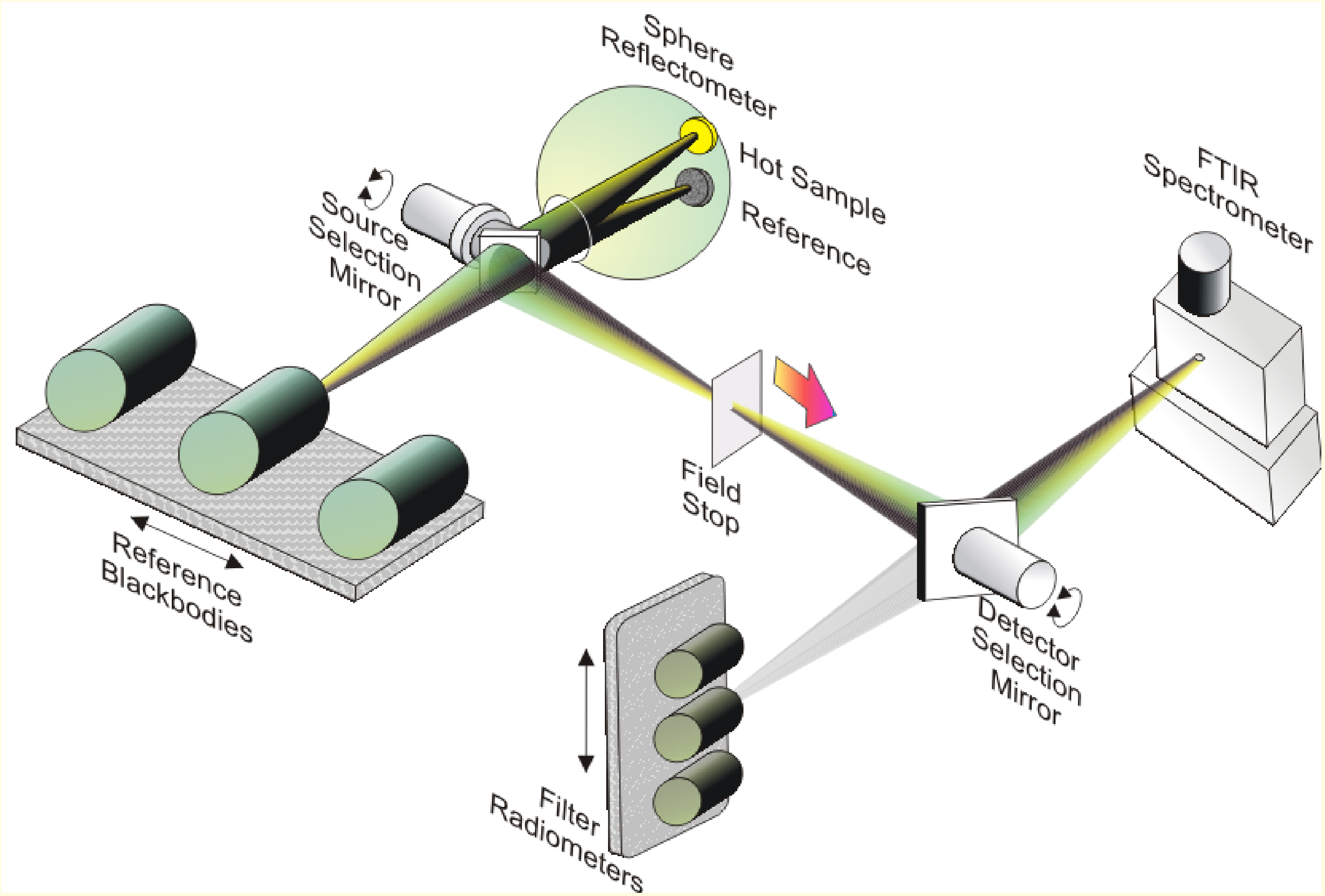
- Compare radiances of samples and reference blackbody source
- Need to know sample and blackbody temperatures
- Sample temperature can be dominant component of uncertainty

$$\varepsilon(\lambda,T)=\frac{V(\lambda,T)}{V_{BB}(\lambda,T_{BB})}\left(e^{\frac{c_2}{\lambda\cdot T}}-1\right)\Big/\left(e^{\frac{c_2}{\lambda\cdot T_{BB}}}-1\right)$$

Sample Temperature Measurement

- Sample temperature required for spectral emittance determination
- Our primary method for sample T ≥ 200 °C is non-contact
  - Secondary method of embedded thermocouple for backup/validation
- Method first developed at INRIM (IMGC) - Italy:
  - M. Batuello, F. Lanza, and T. Ricolfi, "*A simple apparatus for measuring the normal spectral emissivity in the temperature range 600 – 1000ºC*", Proc. 2nd Intl. Symp. Temp. Meas. Ind. Sci. (IMEKO TC12), Suhl (GDR), 1984, pp 125-130.
- Uses Near-IR integrating sphere, filter radiometers & reference blackbodies
- Primary advantage: obtain temperature of sample surface area of interest in direct fashion

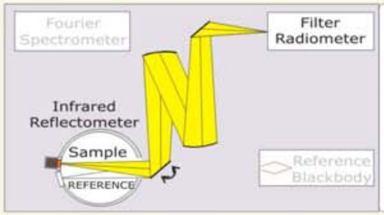
IR Emittance Measurement System



HIGH TEMPERATURE EMITTANCE REALIZATION STEPS

1 MEASURE SAMPLE REFLECTANCE

MEASUREMENT AT FEW LASER WAVELENGTHS

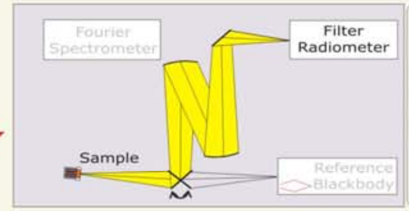


Compare reflectance of the sample with reference plaque by means of spherical laser reflectometer and radiometer

$$\varepsilon_{\lambda}(\lambda_{laser}, T_{sample}) = 1 - \rho_{reference} \times \frac{r_{sample}}{r_{reference}}$$

Where r is the measured response and $\rho(\lambda)$ is known reflectance of the reference plaque.

2 MEASURE SAMPLE RADIANCE



Compare radiance of the sample with blackbody by means of filter radiometer with known relative response

$$r_{samp} = G \times \int_{\lambda_0-\Delta}^{\lambda_0+\Delta} R(\lambda) \times \varepsilon_{\lambda}(\lambda, T_{samp}) \times L_{plank}(\lambda, T_{samp}) d\lambda$$

$$r_{BB} = G \times \int_{\lambda_0-\Delta}^{\lambda_0+\Delta} R(\lambda) \times \varepsilon_{BB}(\lambda, T_{BB}) \times L_{plank}(\lambda, T_{BB}) d\lambda$$

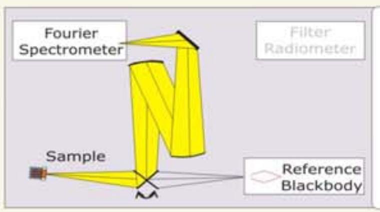
Where r is the measured response, $R(\lambda)$ is the responsivity of the radiometer and G is the geometrical factor.

3 CALCULATE SAMPLE TEMPERATURE

Compute TRUE temperature of the sample surface, using:

- data and equations as shown in Step 2,
- emissivity data from Step 1,
- known blackbody temperature;
- filter radiometer spectral response.

4 MEASURE SPECTRAL EMISSIVITY



Measure sample emissivity, comparing its radiance with blackbody by means of the FT Spectrometer

$$\varepsilon(\lambda, T_{sample}) = \varepsilon_{BB}(\lambda, T_{BB}) \times \left(\frac{L_{sample}}{L_{BB}}\right)_{meas} \times \left(\frac{L_{plank}(\lambda, T_{BB})}{L_{plank}(\lambda, T_{sample})}\right)_{calc}$$

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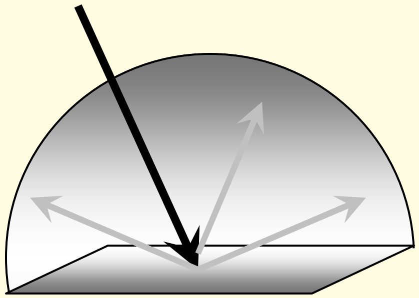
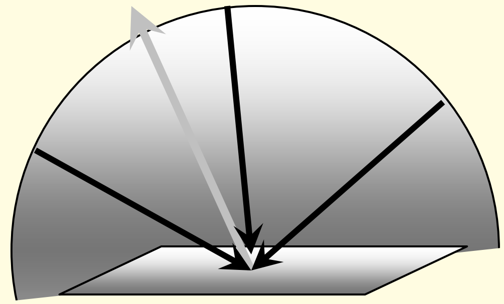
"Diffuse" Reflectance



Directional-Hemipsherical Reflectance

DHR

- Single direction illumination
- Hemispherical collection
- = output flux/input flux
- Requires uniform collection



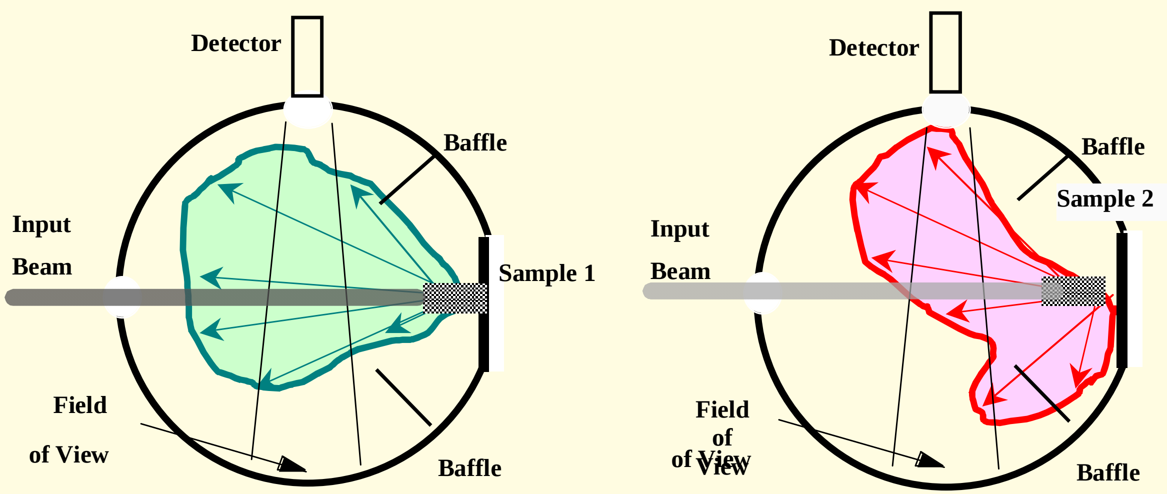
Hemispherical-Directional Reflectance Factor

HDRF

- Hemispherical illumination
- Directional collection (small solid angle)
- = output flux/flux from ideal diffuser
  output flux/(input flux*proj. solid angle)
- Requires uniform radiance illumination

(DHR) Sphere Design for Relative Reflectance Measurements: How to Handle First Reflection from Sample?

- Design philosophy: treat light reflected from sample and reference in identical fashion
- Effect: Sample scatters light (BRDF) in arbitrary fashion different from reference
- Problem: Detectors often have limited field-of-view (FOV) and stronger response for light within FOV
- Solution: Use baffles to control light interchange between sample/reference and ports/detector field-of-view (FOV)
- Goal: To make throughput to the detector independent of the sample BRDF



Isotropic Sphere Design Concept*

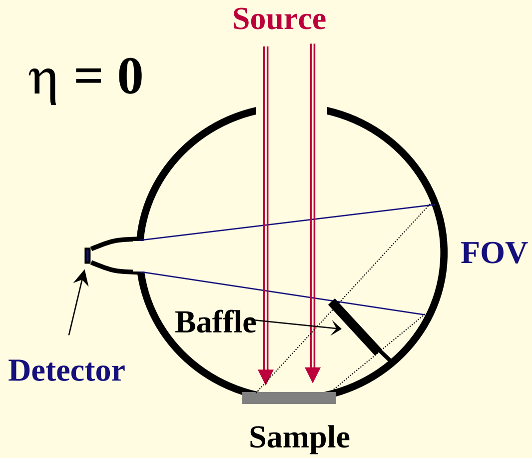
- **Design must treat sample and reference reflected light equally for accurate relative measurements and be independent of scattering distribution (BRDF)**

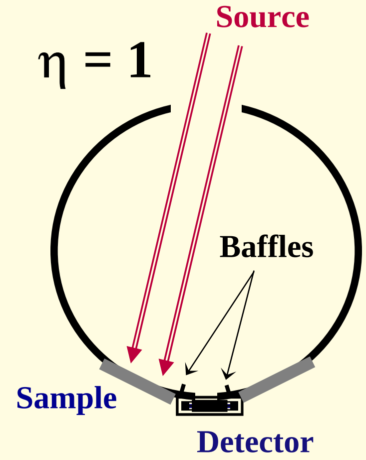
- **Conclusion: best designs "force" sample and reference η to be the same**
  - **Where η is the fraction of reflected light going into the FOV.**
  - **Three possibilities, $\eta = 0$, $(\eta = 1/2)$, and $\eta = 1$**

K. A. Snail and L. M. Hanssen, "Integrating sphere designs with isotropic throughput", Applied Optics **28 no. 10, 1793 (1989).*

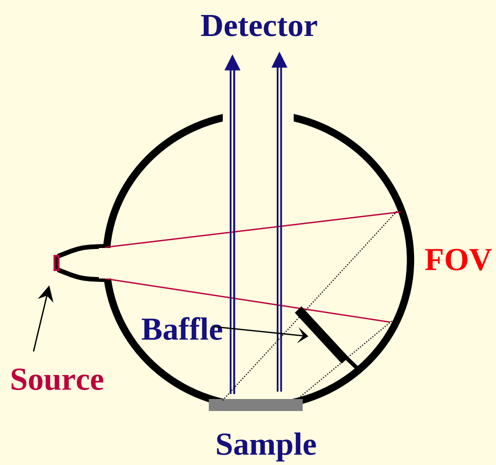
Isotropic Sphere Designs

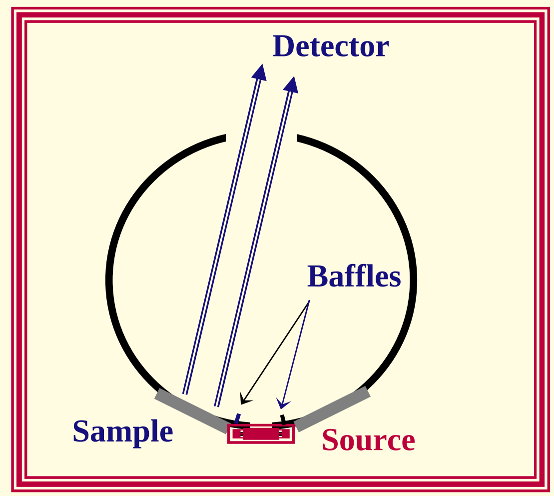
DHR Designs





HDR Designs





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Modeling of HDRF Integrating Sphere Using Monte Carlo Methods*

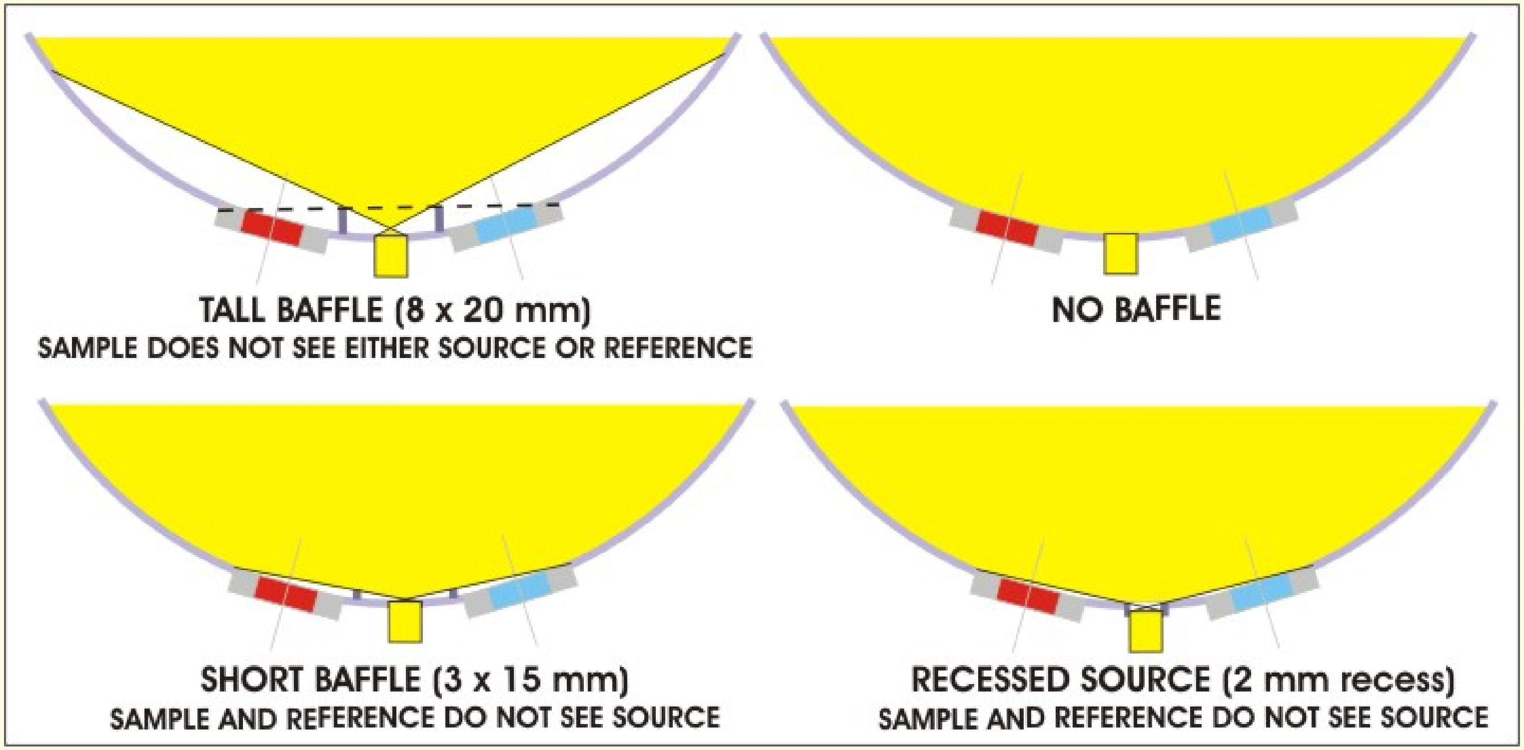
- Monte Carlo Modeling Software Description:
  - Employs backward ray-tracing, importance sampling, other methods for high speed calculations 10^7 rays / run
  - Sample & reference have specular/diffuse or real BRDF
  - Source has $\mathrm{Cos}^n(\theta)$ form
  - Sphere wall & other ports have specular/diffuse (current version)
- Output Products:
  - Hemispherical distributions of spectral radiance falling onto sample center
  - Measured spectral reflectance for samples w/ specular-diffuse & real BRDF
  - Integrating sphere throughput

A. V. Prokhorov, S. N. Mekhontsev and L. M. Hanssen, "Monte Carlo modeling of an integrating sphere reflectometer", Applied Optics* ***42* *no. 19, 2382 (2003).*

Geometric Parameters of Modeled System

Dimension	Size
Sphere radius	127 mm
Elliptic opening major axes	60 × 46 mm
Source radius	5 mm
Sample and reference radii	9.5 mm
Sample and reference holders radii	17.5 mm
Distance between baffles	30 mm
Baffles height	3 mm
Baffles length	11 mm
Central angle between sample and reference	32°
Viewing angle	10°

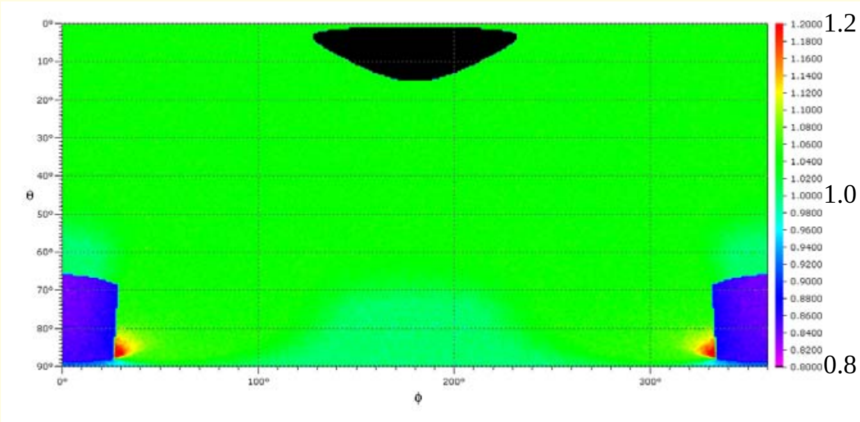
HDR Baffling Design Options Modeled



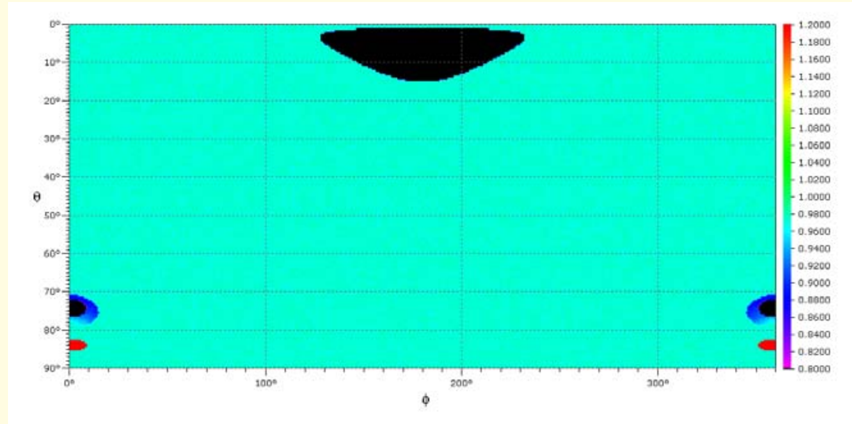
- Goals for evaluation:
  - Best in radiance uniformity
  - Least sensitive to scattering properties of sample

Comparison of Design’s Radiance Uniformity

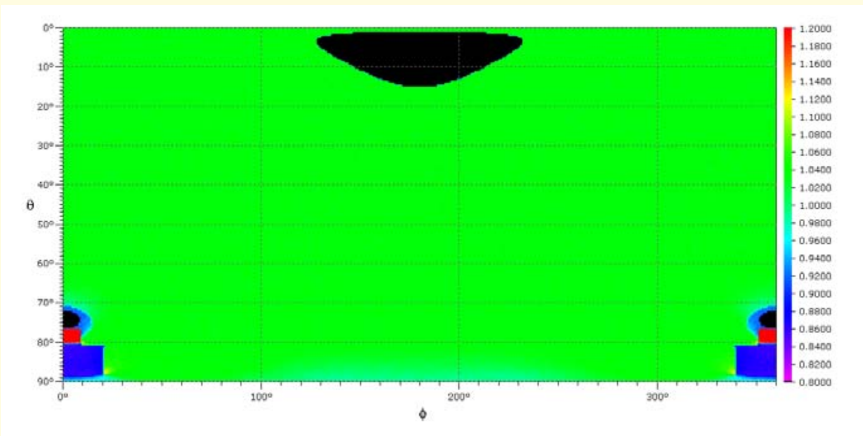
Large Baffle



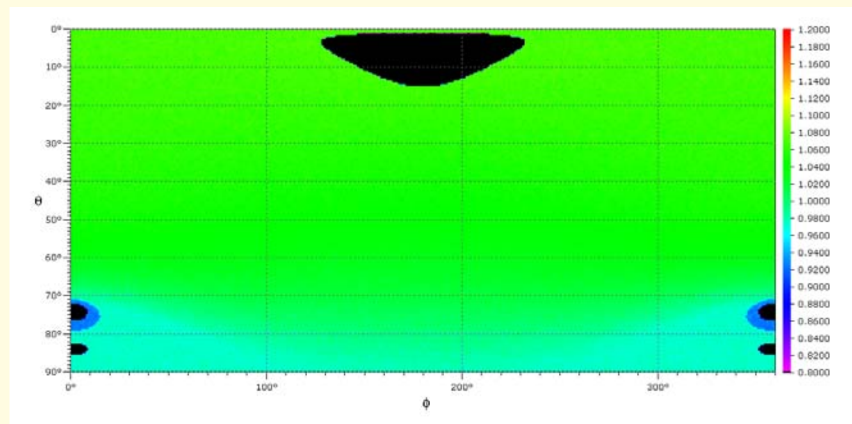
No Baffle



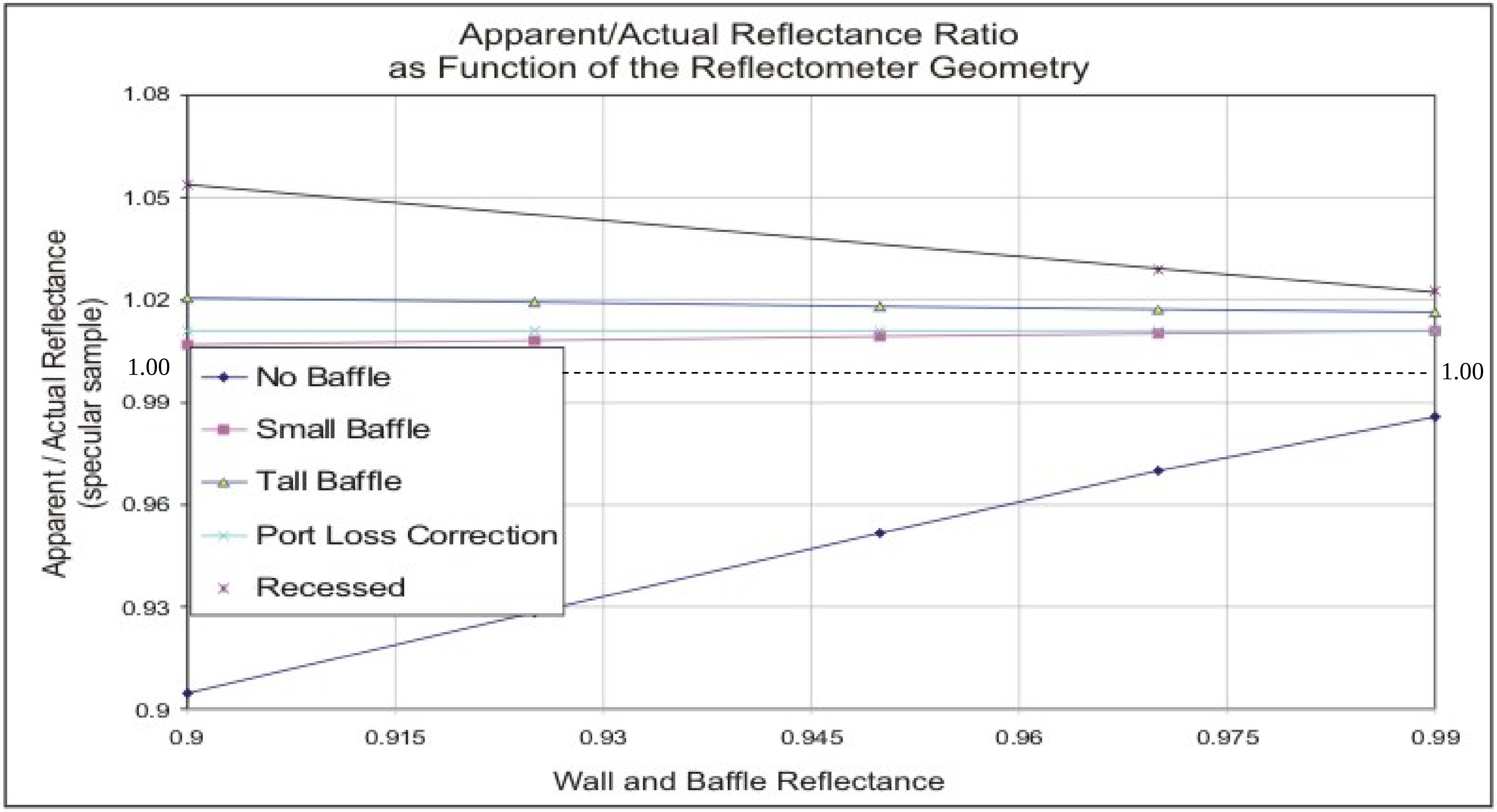
Small Baffle



Recessed Source



Effects of Design on Measured Reflectance for a Specular Sample Compared to a Diffuse Reference



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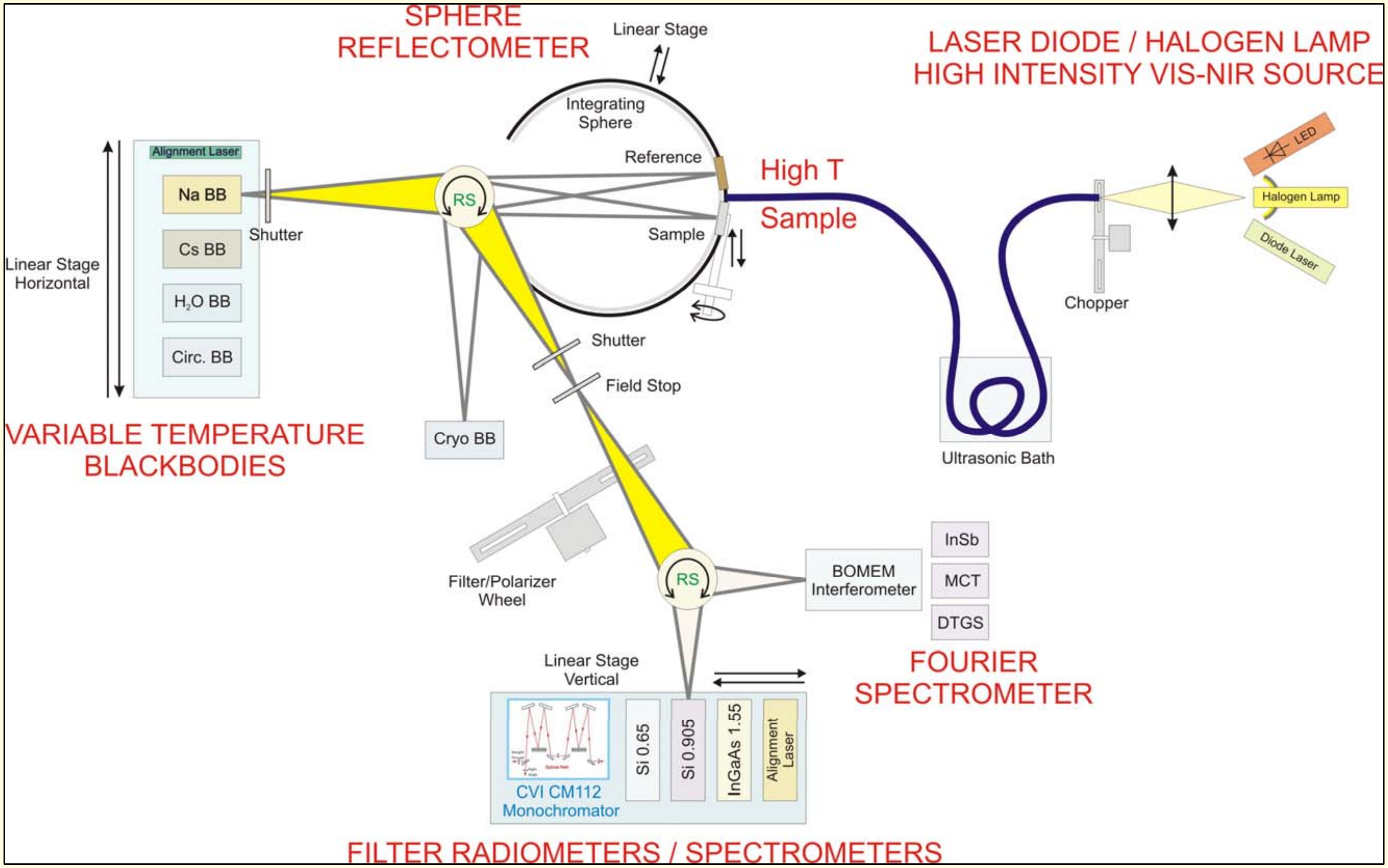
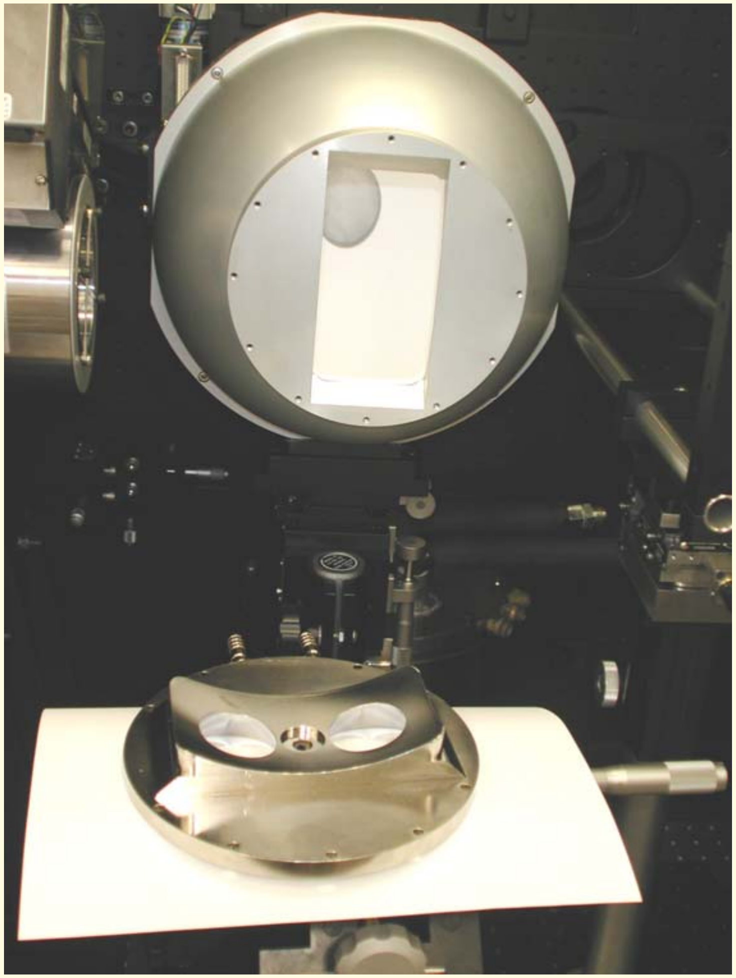
Sample Emittance/Temperature Measurement Setup



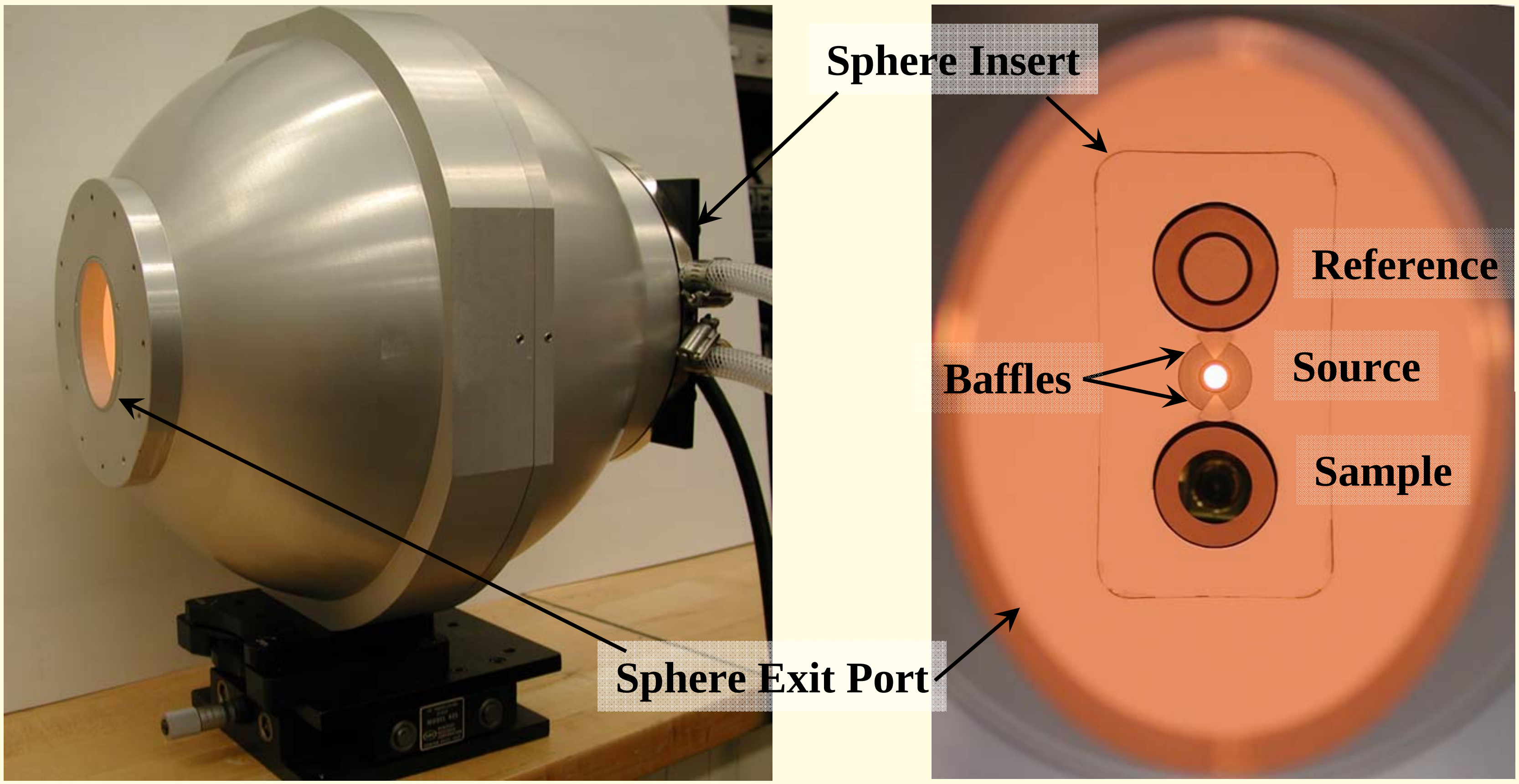
DHR Integrating Sphere: Rear View w/ uncoated Insert



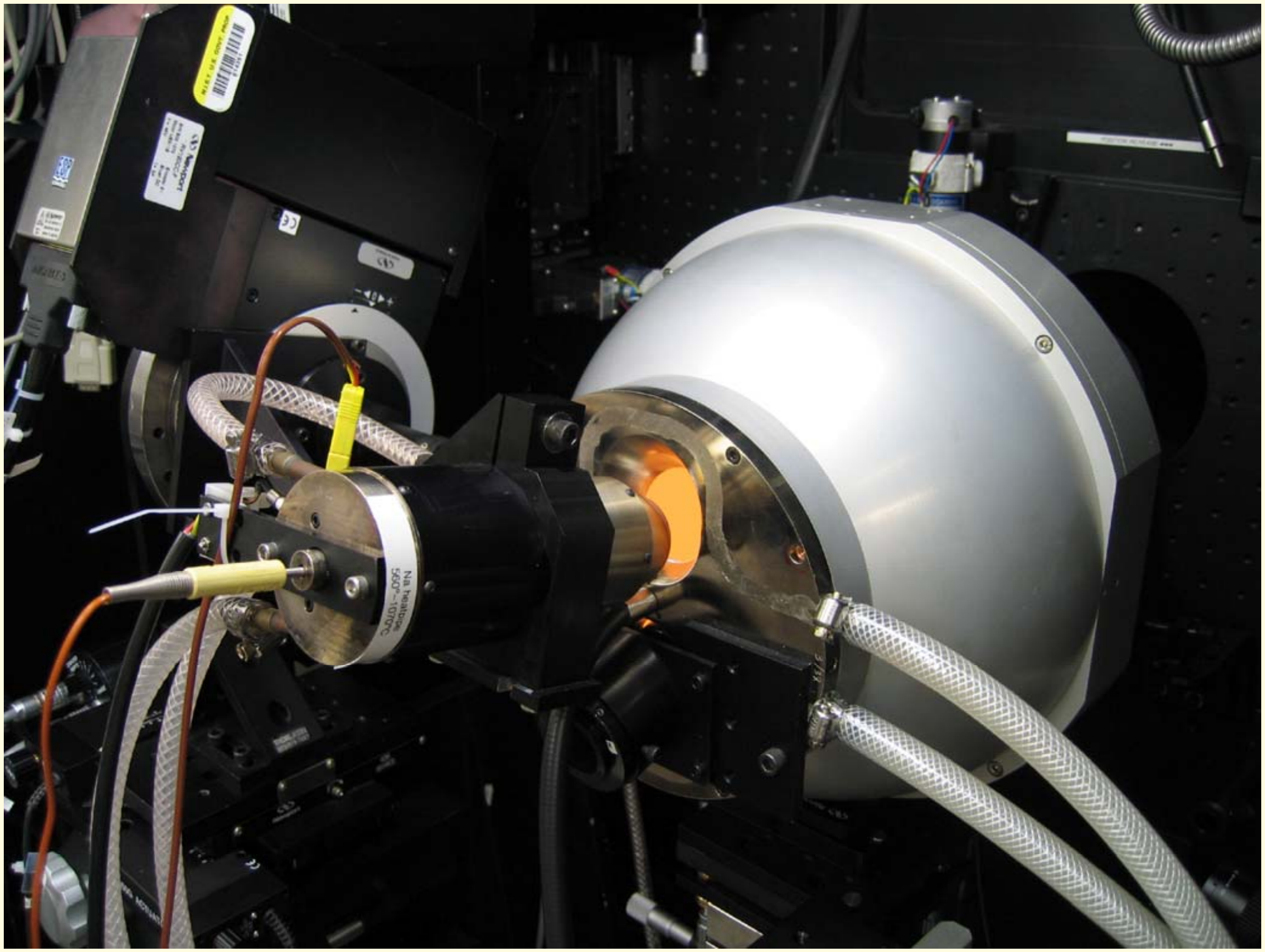
- Sintered PTFE on Main Body
- $BaSO_4$ on insert (future alumina?)
- 250 mm diameter
- Separate insert containing sample, reference and source ports and baffles
- Insert water cooled to accommodate samples up to 1400 K
- Source between sample and reference; minimal size baffles for near 2 π illumination of sphere
- Sample, ref. ports accommodate 9° & normal incidence
- Sample, ref. ports accommodate sample & heater assembly

Integrating Sphere for Sample Temperature Measurement

View through Exit Port



Sample Heater & Sphere



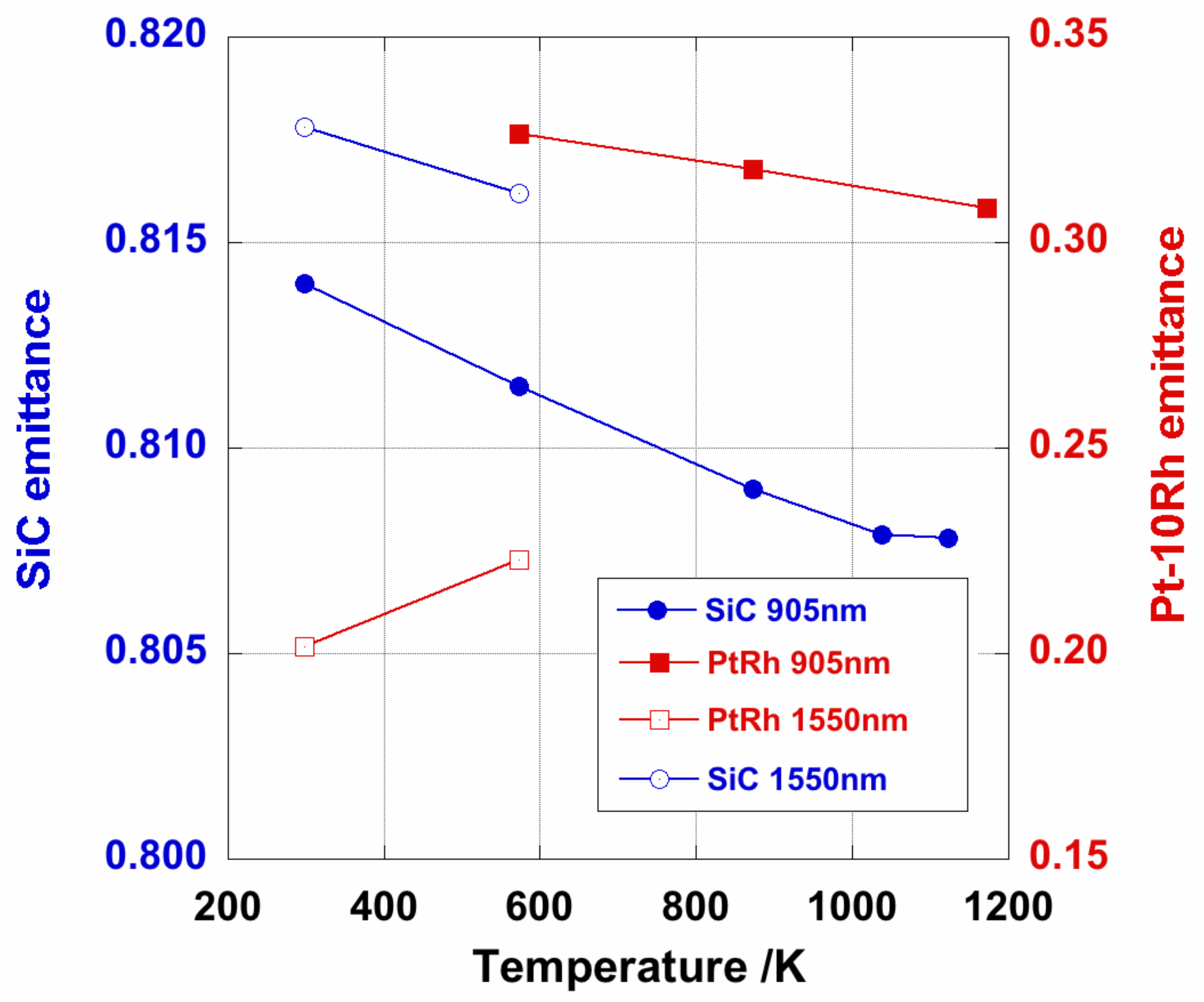
Reflectometer Evaluation using Standard Samples*

Wavelength/ nm	Mirrors Ratio	Diff. %	Specular/ Diffuse	Calibration data	Diff. %	SiC vs. gold	Calibration data	Diff. %
905	1.0004	0.04	1.0102	1.0100	0.02	0.1929	0.1930	-0.05
1550	1.0007	0.07	1.0165	1.0220	-0.55	0.1934	0.1937	-0.16

- Diffuse sample measurement has greater uncertainty (than specular) due to non-uniformity of sphere
- Expanded uncertainty (k = 2) for calibrated standards ~0.1% - 0.5%
- Sphere performance meets design goal

L. M. Hanssen, C. P. Cagran, A. V. Prokhorov, S. N. Mekhontsev, and V. B. Khromchenko, "Use of a High-Temperature Integrating Sphere Reflectometer for Surface-Temperature Measurements", Int. J. Thermophysics* ***28* *no. 2, 566 (2007).*

Emittance Results from Sphere Reflectometer



Emittance Uncertainty Budget

Uncertainty budget of sample spectral emittance			
Reflectometer at 905 nm	**Type**	**Pt-10Rh at 600¼C**	**SiC at 600¼C**
Repeatability of temperature comparison	A	0.05%	0.05%
Sample reflectance			
Repeatability of reflectance comparison	A	0.03%	0.03%
Sample			
Alignment	B	0.19%	0.19%
Temperature	B	0.05%	0.00%
Reflectance reference			
Calibration	B	0.09%	0.09%
Alignment	B	0.19%	0.19%
Sphere reflectometer	B	0.20%	0.20%
Radiometer calibration			
Calibration at FP	B	0.01%	0.01%
Interpolation	B	0.01%	0.01%
Alignment	B	0.00%	0.00%
SSE of interface optics	B	0.04%	0.04%
Combined standard uncertainty of spectral emittance		**0.36%**	**0.35%**
Expanded uncertainty (*k* = 2)		**0.72%**	**0.70%**

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Sample Surface Temperature Uncertainties (using sphere-based method; k=2)

$$\frac{d\varepsilon(\lambda)}{\varepsilon(\lambda)} = \frac{c_2}{\lambda} \cdot \frac{dT(\lambda)}{T(\lambda)^2}$$

SiC		Pt-10Rh	
T [K]	*ΔT [K]*	*T [K]*	*ΔT [K]*
573.75	0.14	573.59	0.15
868.56	0.34	872.76	0.34
1038.81	0.49	1172.75	0.61
1123.61	0.57		

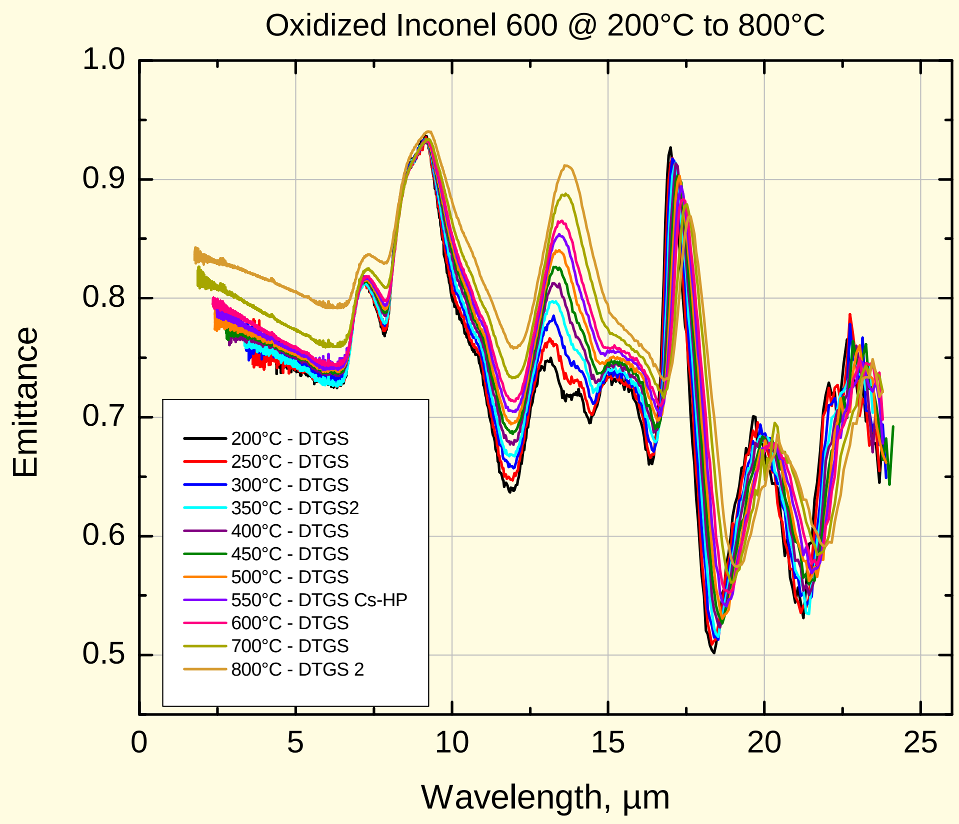
- Using emittance uncertainties from previous table

Temperature Method Comparison/Validation: Non-Contact (Sphere) vs. Contact (TC)

Material	T_{TC} [K]	ε_{tot}	$T_{w/o\ conv.}$ [K]	$T_{w/\ conv.}$ [K]	T_{radio} [K]	$\Delta T_{(radio-conv)}$ [K]
SiC	298.00	0.800	298.00	298.00	---	---
	573.75	0.800	573.71	573.67	573.38	-0.29
	868.56	0.800	868.34	868.25	867.94	-0.32
	1038.81	0.800	1038.36	1038.25	1038.04	-0.21
	1123.61	0.800	1122.99	1122.87	1122.07	-0.80
Pt-10%Rh	573.59	0.096	573.58	573.51	572.96	-0.54
	872.76	0.129	872.69	872.54	871.83	-0.71
	1172.75	0.172	1172.45	1171.21	1171.75	-0.47

- Last column show agreement level of two methods
- Table shows effect of convection loss correction
- Agreement is very good; better than anticipated from uncertainty budgeting

IR Spectral Emittance Example: Oxidized Inconel



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Summary & Conclusions

- We have designed, modeled, constructed, tested and applied an HDRF integrating sphere
- The integrating sphere reflectance performance was validated with calibrated samples.
- The implementation of a sphere-based non-contact temperature measurement method was validated by comparison with contact thermometry.
- The sphere-based method:
  - useful for both specular & diffuse materials
  - advantage for elevated temperatures and poorly conducting materials
  - limited at short wavelengths/lower temperatures due to low sample emission
  - can be adapted to transparent materials