Infrared hemispherical reflectance measurements in the 2.5 µm to 50 µm wavelength region using an FT spectrometer

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2. The upgraded hemispherical reflectance facility, based on an FT spectrometer
3. Sorting the issue of sample heating.
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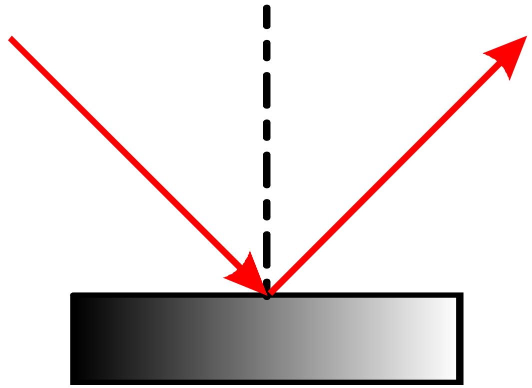
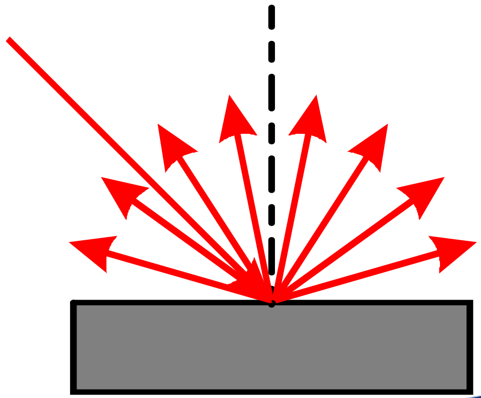
What do we measure?

- Regular reflectance



- Directional Hemispherical reflectance



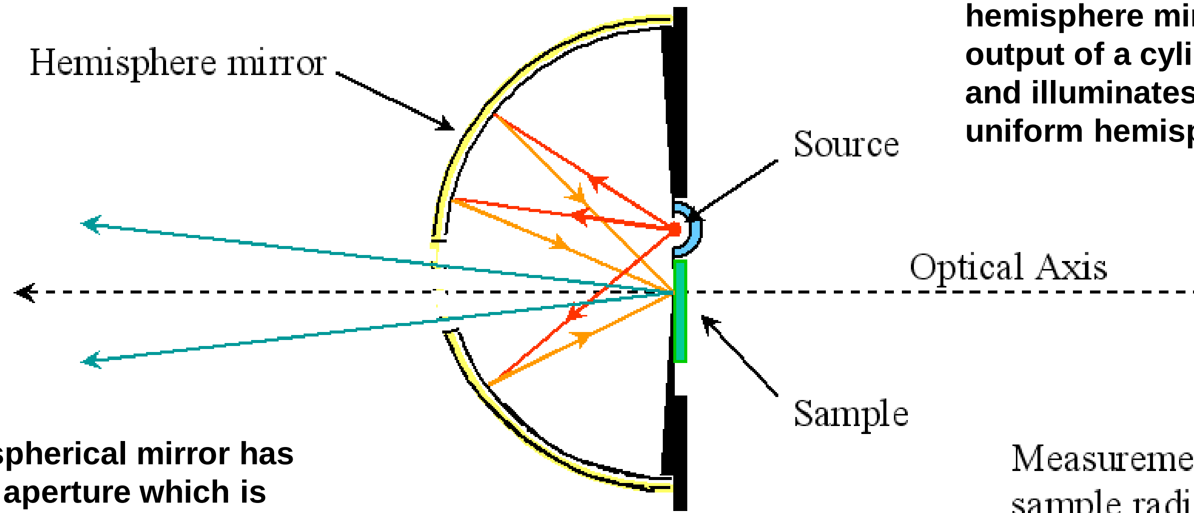
Introduction

- Two methods are currently being used by NMIs for directional - hemispherical reflectance measurements at ambient temperatures in the infrared:
- NIST use an integrating sphere-based method. Losses in the integrating sphere limit measurements to wavelengths below 19 μm.
- NPL has traditionally used a hemispherical reflector which allows measurements to wavelengths over 50μm, but the method also has some drawbacks.
- Aim: To describe recent improvements to the NPL hemispherical reflectance facility.

The NPL hemispherical reflectance infrared measurement facility

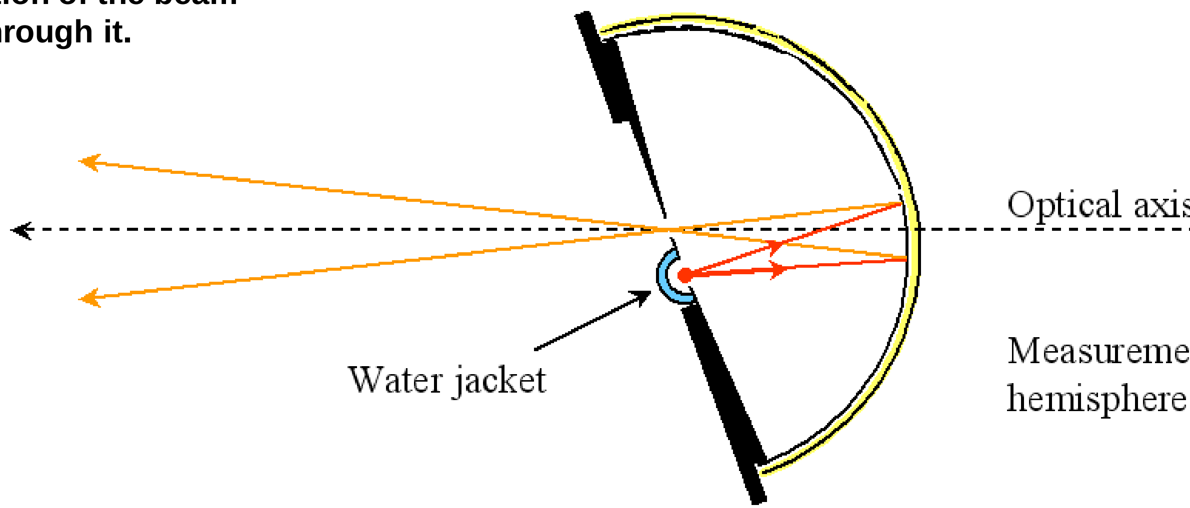
- Comprised of:
  - the absolute hemispherical reflectometer and
  - a diffraction grating-based spectrophotometer.

The reflectometer is based on a hemisphere mirror which collects the output of a cylindrical Oppermann source and illuminates the test sample with a uniform hemisphere of radiation.



The hemispherical mirror has one small aperture which is only slightly larger than the cross section of the beam passing through it.

Measurement of sample radiance $L_s(\lambda)$



Measurement of hemisphere radiance $L_h(\lambda)$

reflectance of sample = $L_s(\lambda) / L_h(\lambda)$

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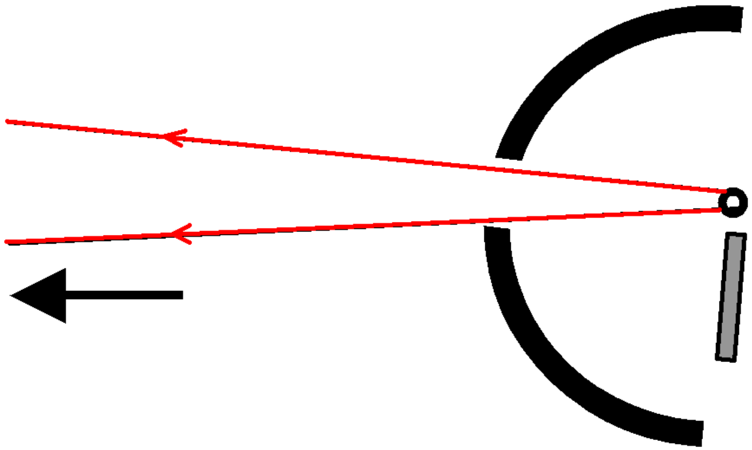
The NPL hemispherical reflectance measurement facility

- By rotating and sliding the hemisphere to reproducible positions, the hemispherical mirror radiance (R) and reflected sample radiance (S) are be measured.

- The radiance directly from the source is also measured with and without the sample present, to take into account inter-reflections between the source and the test sample.



A Perkin-Elmer 580B IR grating spectrophotometer used to be used to spectrally analyse the hemispherical reflectometer output in the 2.5 μm to 56 μm wavelength range.

The sample compartment of the Perkin-Elmer 580B spectrophotometer

The grating spectrophotometer was replaced by an FT spectrometer

- FT spectrometers offer advantages over grating-based instruments which makes them faster, particularly in the IR.
  - Fellgett (multiplex) advantage
  - Jacquinot advantage
  - Connes advantage

Description of the FT spectrometer

A KBr beamsplitter is used to cover the 2.5 μm - 22 μm wavelength range.

- A multilayer Mylar beamsplitter is used to cover the 16 μm - 50 μm wavelength range.
- Full coverage from 2.5 μm to 50 μm.
- A gold-black-coated deuterated L-alanine triglycine sulphate (DLATGS) pyroelectric detector with a KBr window is used with the KBr bemsplitter.
- A similar detector, but with a polythene window, is used with the Mylar beamplitter.

The FT spectrometer next to the enclosure

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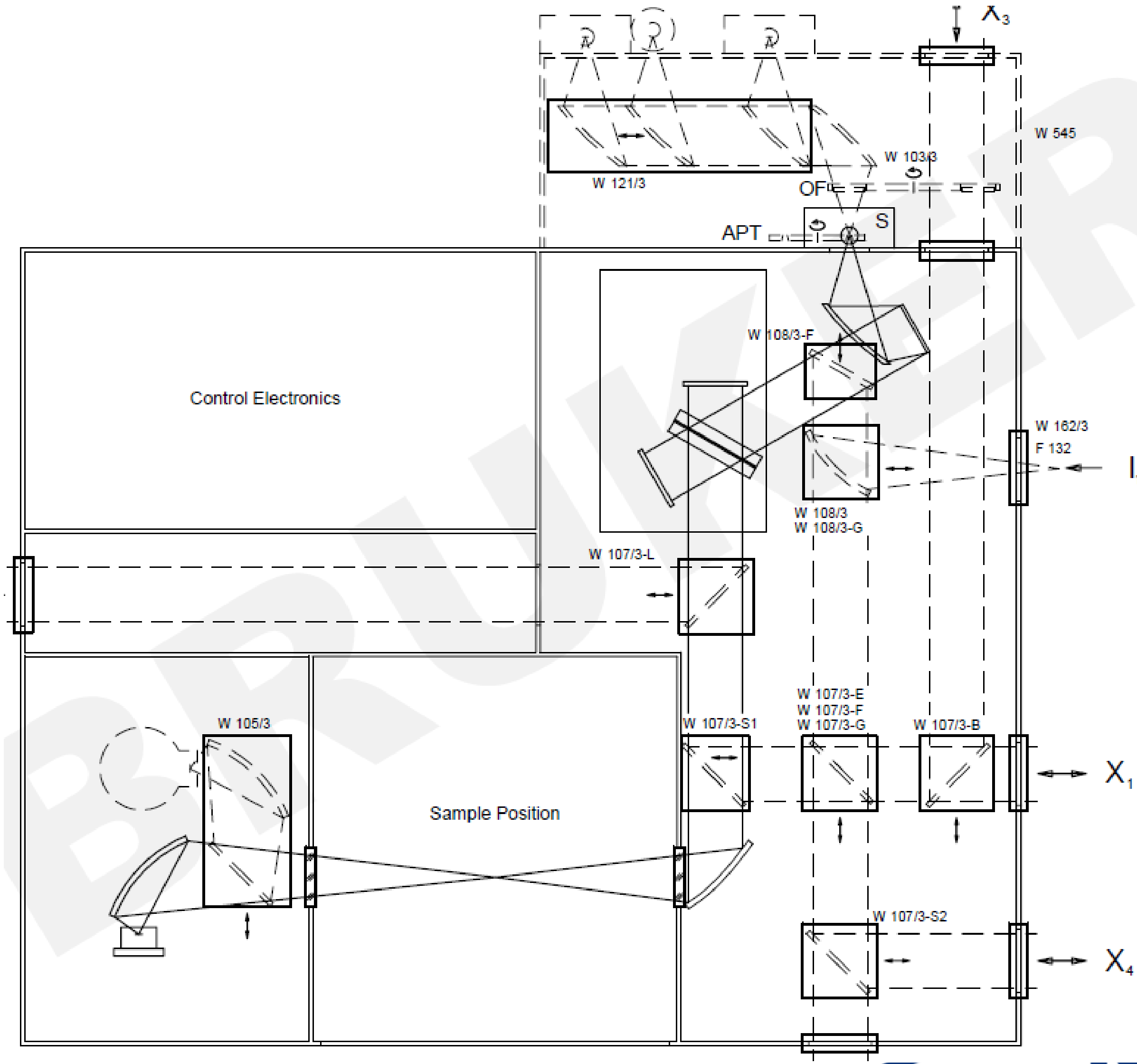
System alignment

- The standard alignment of the spectrometer aligns a beam from an internal source through the interferometer optics and brings it to a focus in the internal sample compartment. This beam is then refocused on the active area of the detector(s).
- Once this has been carried out, visible radiation is used to illuminate an aperture placed at the focus in the sample compartment, which then forms an image at the emission port of the spectrometer (a mirror is used to switch from the internal source to the emission port). The visible radiation is then used to align the relay optics and the AHR (in all of its required orientations). The source is removed from the sample compartment, the ‘dummy’ beamsplitter is replaced by the appropriate beamsplitter, and the AHR and its relay optics are properly aligned.

The Bruker Equinox 55 FTS FT spectrometer



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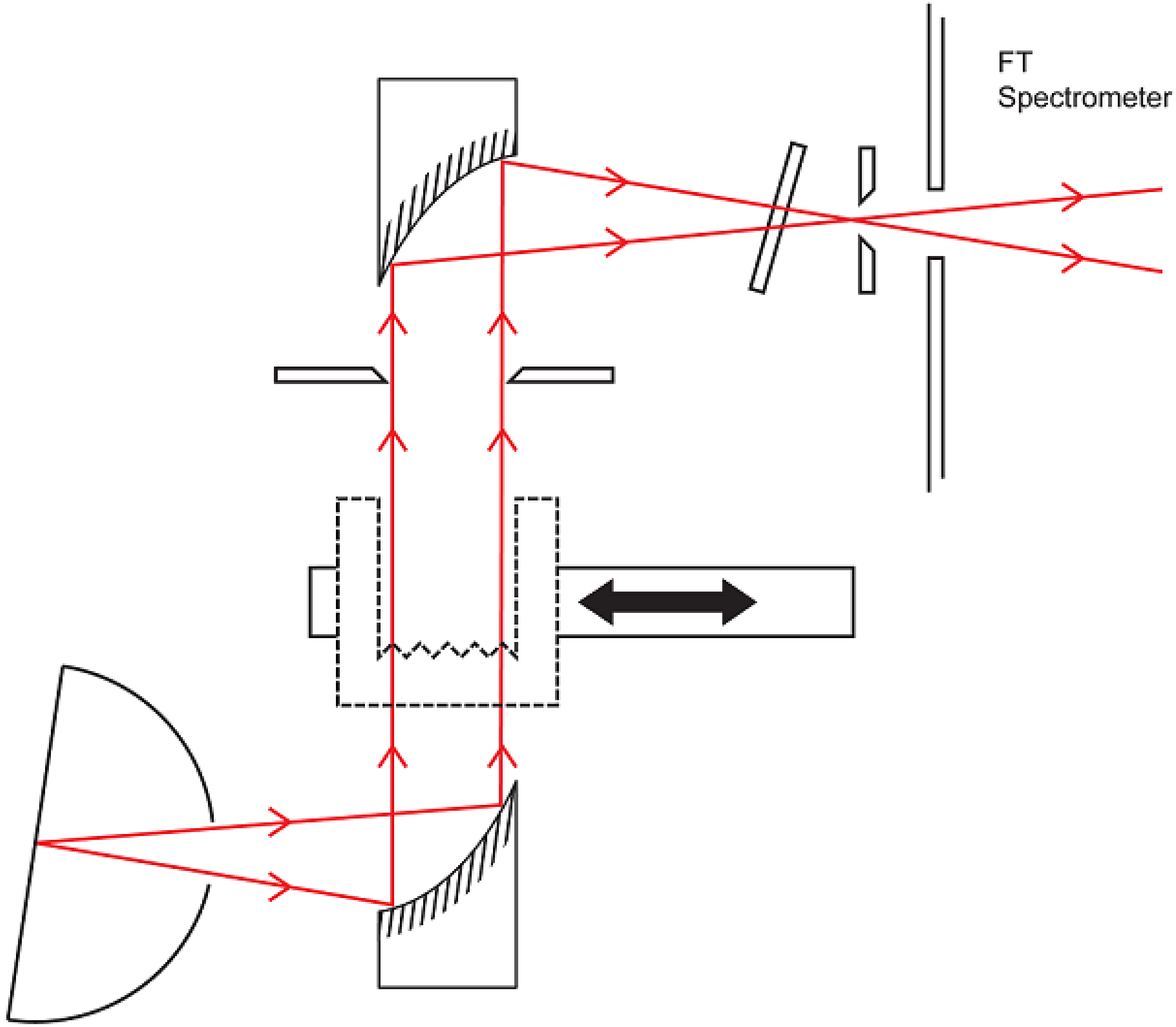
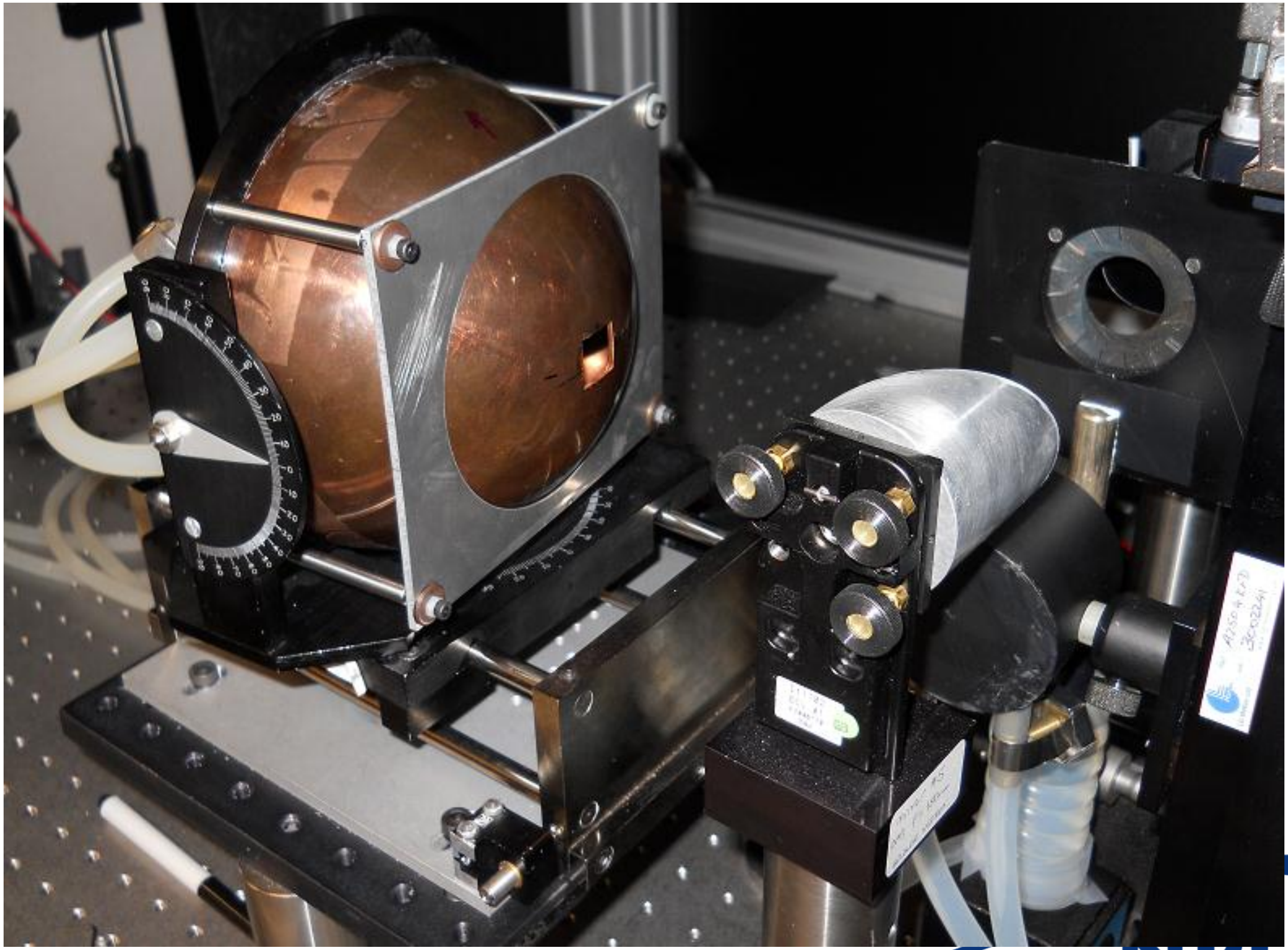
Layout of relay optics of new facility



The hemispherical reflectometer



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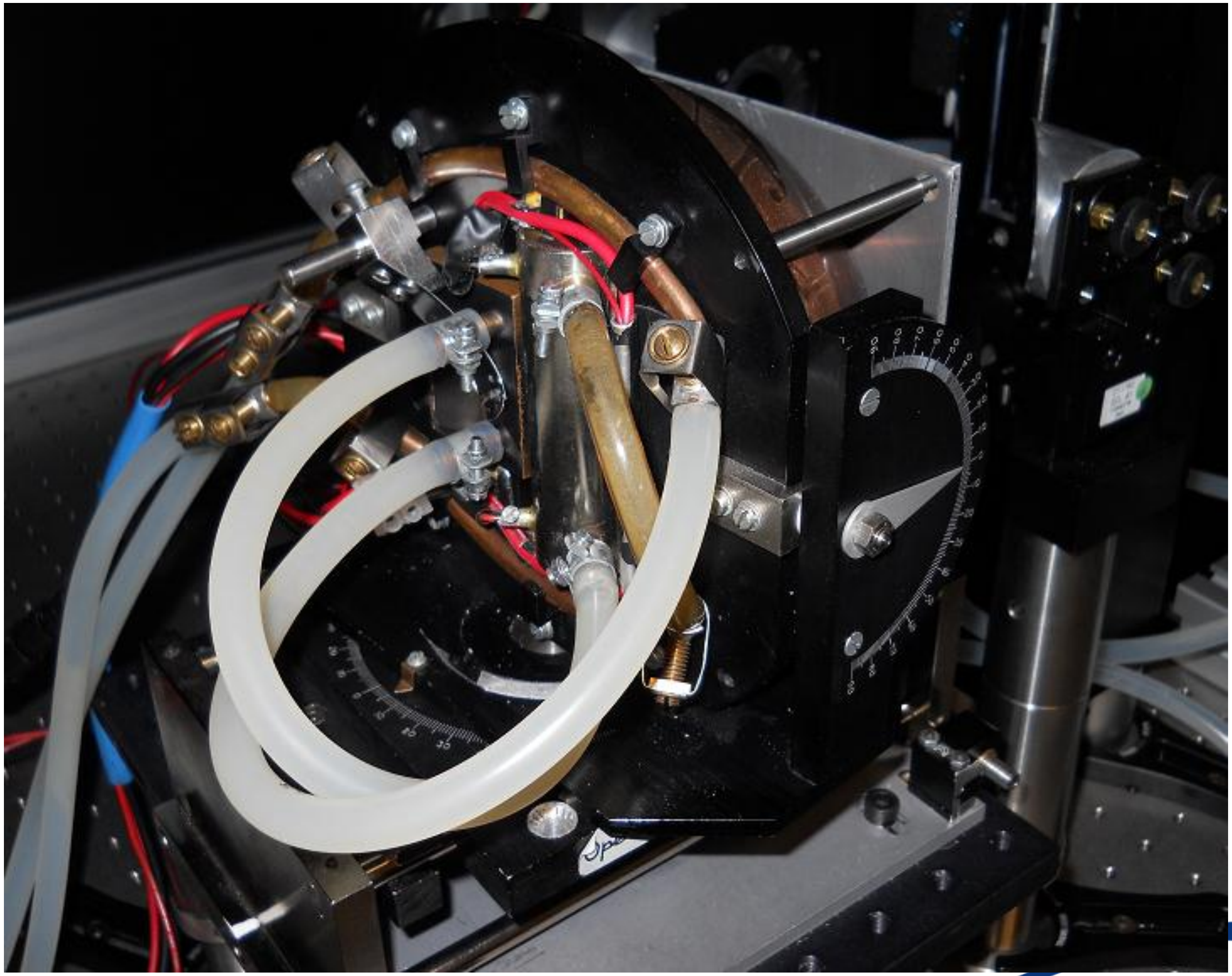
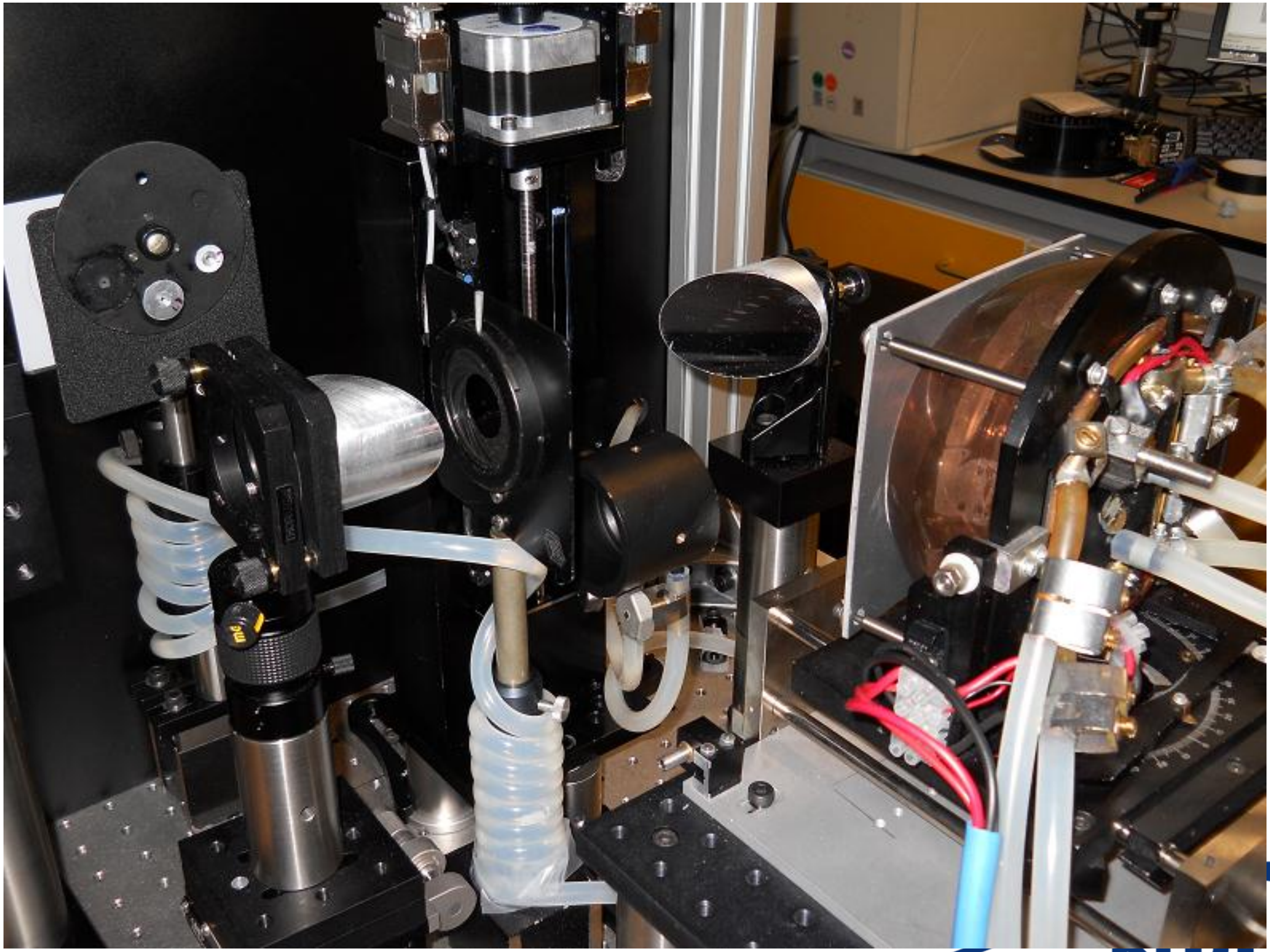
The rear of the Hemispherical reflectometer



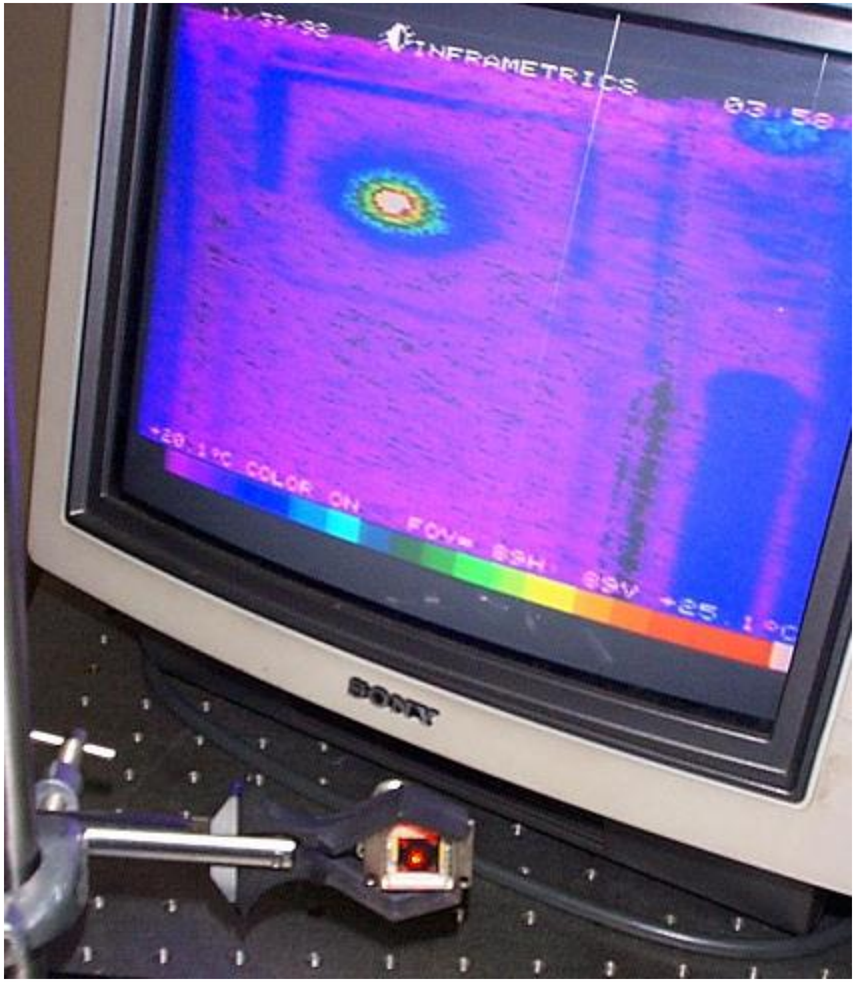
The relay optics and blackbody

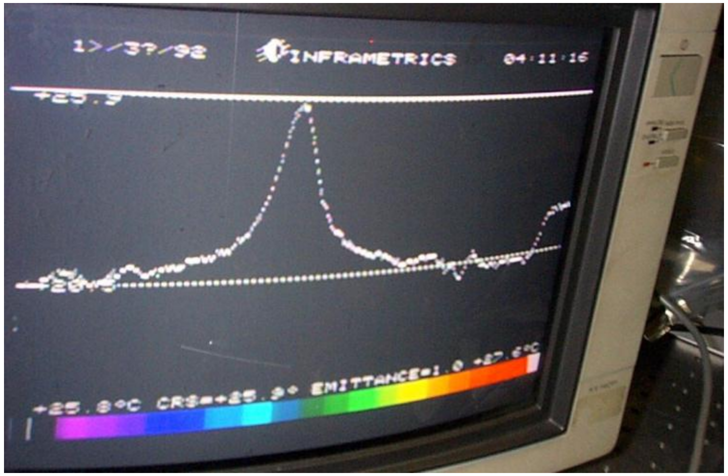


Heating of the test sample

- Very high irradiance levels reach the sample when the unfiltered output of the Oppermann source is used to illuminate the test sample.
- This is a particular problem in absorptive samples because radiation is absorpted and the temperature of the sample increases.
- The contribution due the thermal radiation emitted by the sample itself can become a significant problem for wavelengths longer than about 20μm.
- There is no means to differentiate the component of the detected radiation which was reflected by the sample AND the radiation emitted by the sample due to the increase of its temperature due to heating by the source output.

Pyro illuminated by He-Ne laser





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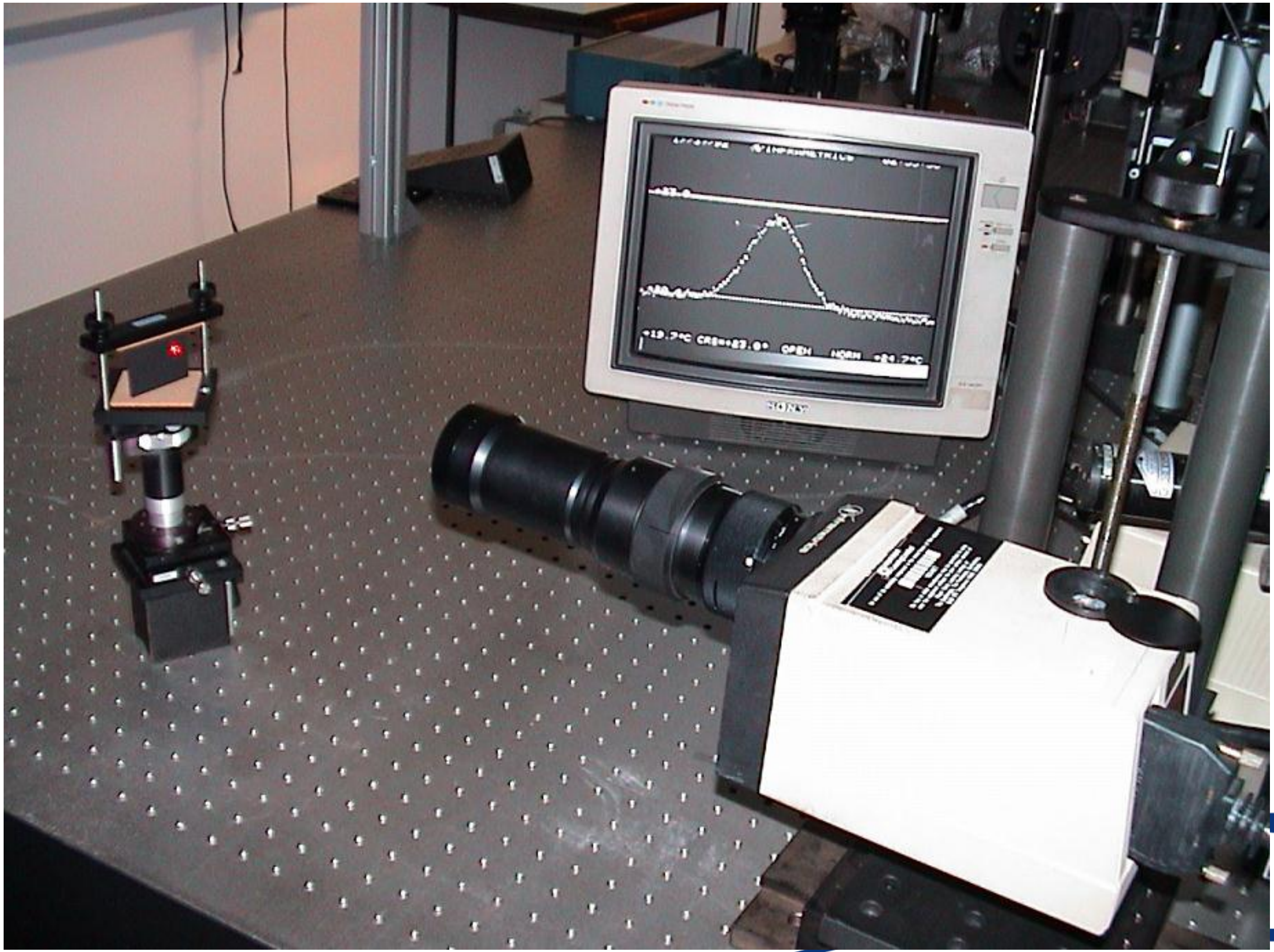
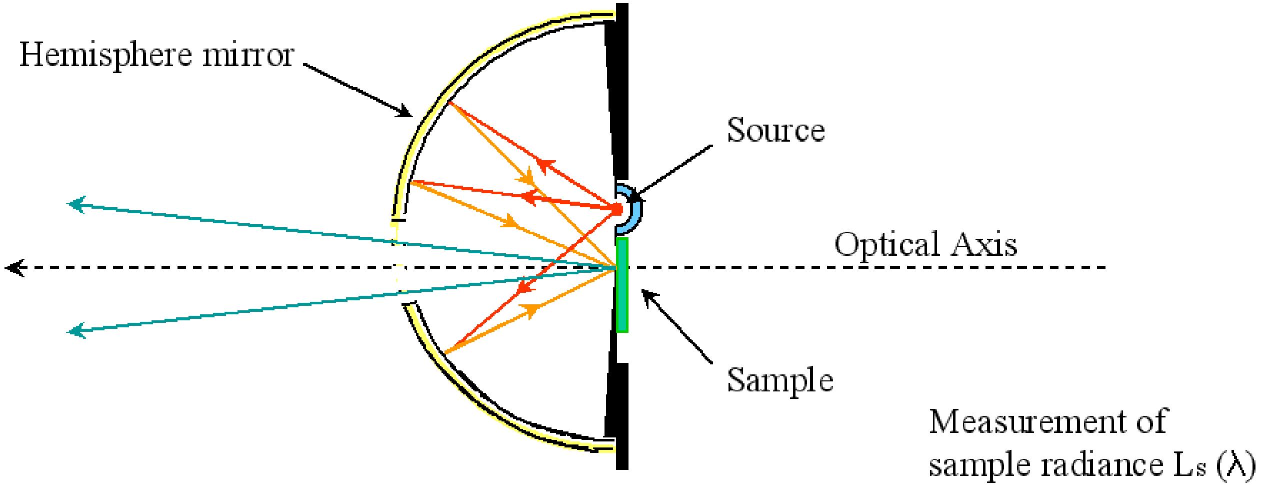
Heating of a Nextel paint sample

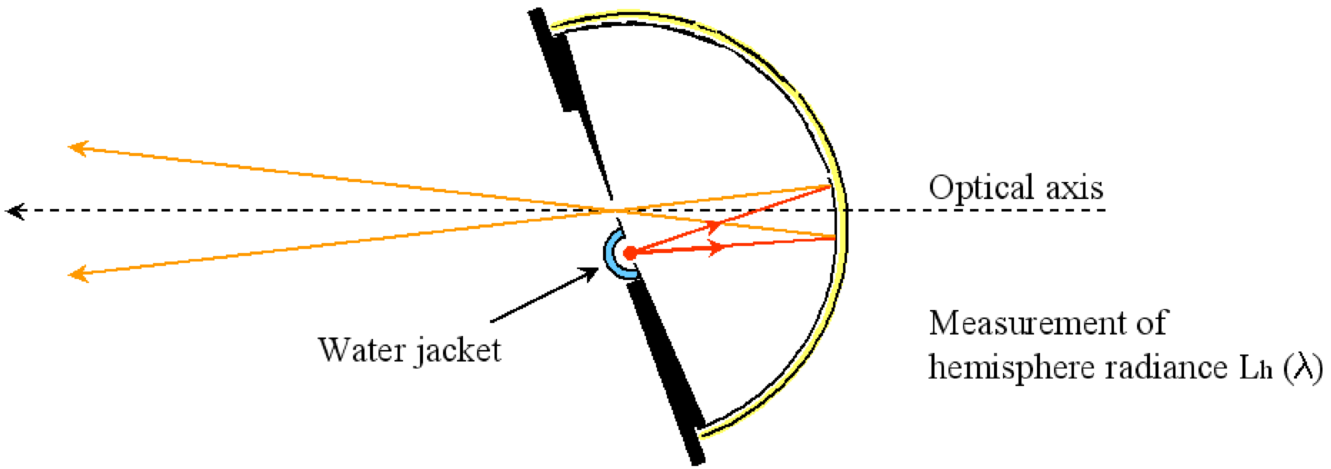


Eliminating the effects of sample heating

- The effect of the thermal emission of the test sample was studied by measuring the reflectance using the Oppermann source at different temperatures.
- For black samples, the reflectance decreased gradually (at long wavelengths) as the temperature of the source decreased and asymptotically reached a minimum level (see later).
- This requires the source to operate at relatively low temperatures, necessitating long measurement times for wavelengths longer than 10 μm.
- During this time the response of the interferometer and relay optics may drift. To correct for this, a blackbody (seen earlier) which is mounted on a PC-driven linear translation stage is periodically inserted and is viewed by the spectrometer.
- The blackbody is kept at a temperature stable to better than $\pm$0.05 ^{o}C using flowing water.

$$S = R_{sample} R_{hemi}\ e_{glowbar}\ L_{glowbar}(\lambda, T) + (1 - R_{sample}) L_{sample}(\lambda) - L_{bb}(\lambda)$$



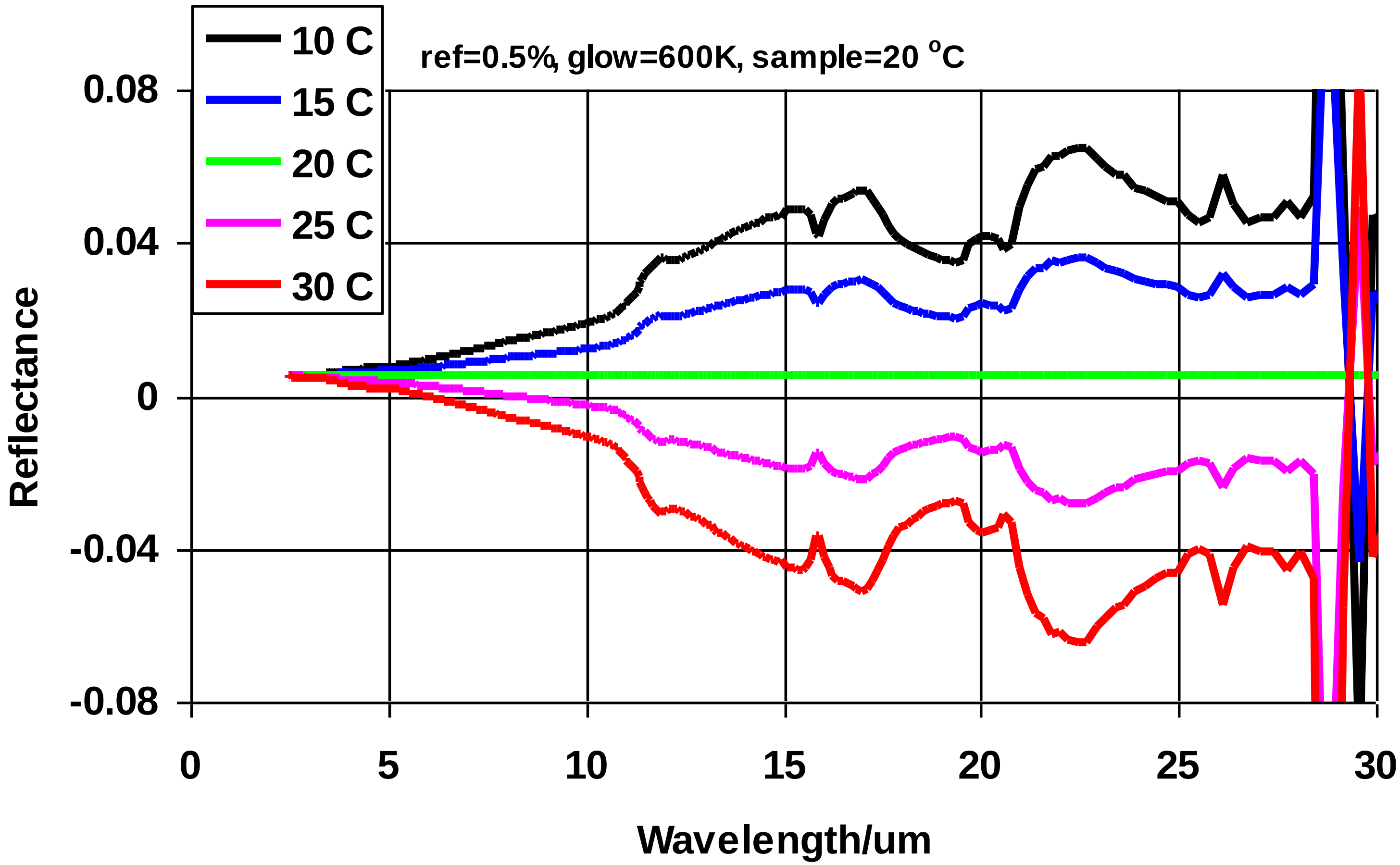


$$R = R_{hemi}(\lambda)\ e_{glow}(\lambda)\ L_{glow}(T) - L_{bb}(\lambda)$$

$$Ratio = \frac{R_{sample}\, R_{hemi}\, e_{glowbar}\, L_{glowbar}(\lambda, T) + (1 - R_{sample}) L_{sample}(\lambda) - L_{bb}(\lambda)}{R_{hemi}(\lambda)\, e_{glowbar}(\lambda)\, L_{glowbar}(T) - L_{bb}(\lambda)}$$

$$Ratio = R_{sample} + \frac{(1 - R_{sample}) L_{sample}(\lambda) - L_{bb}(\lambda)}{R_{hemi}(\lambda)\, e_{glowbar}(\lambda)\, L_{glowbar}(T) - L_{bb}(\lambda)}$$

Calculated reflectance for different reference blackbody temperatures



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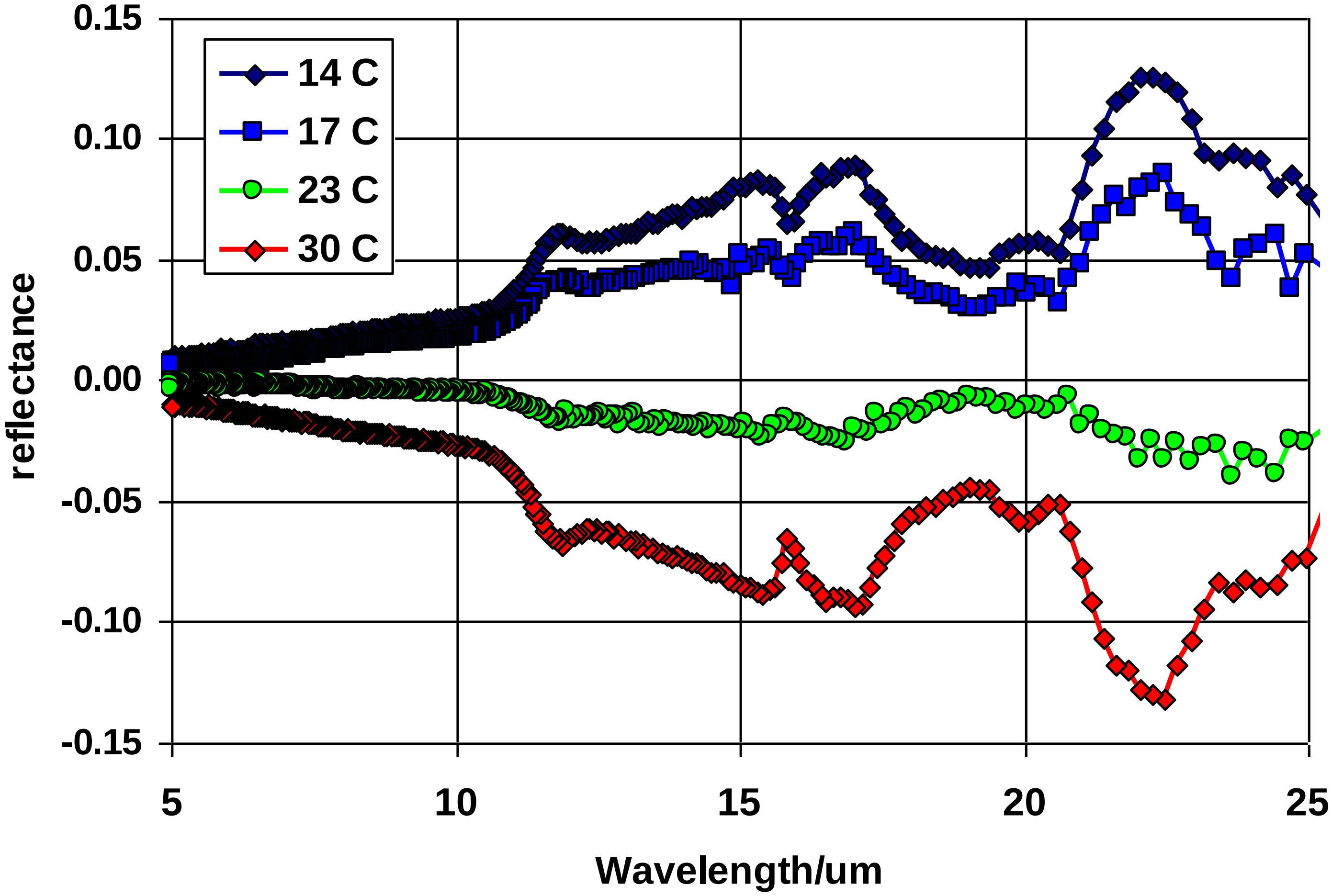
Measurement of the reflectance of the VA150 at four different reference blackbody temperatures



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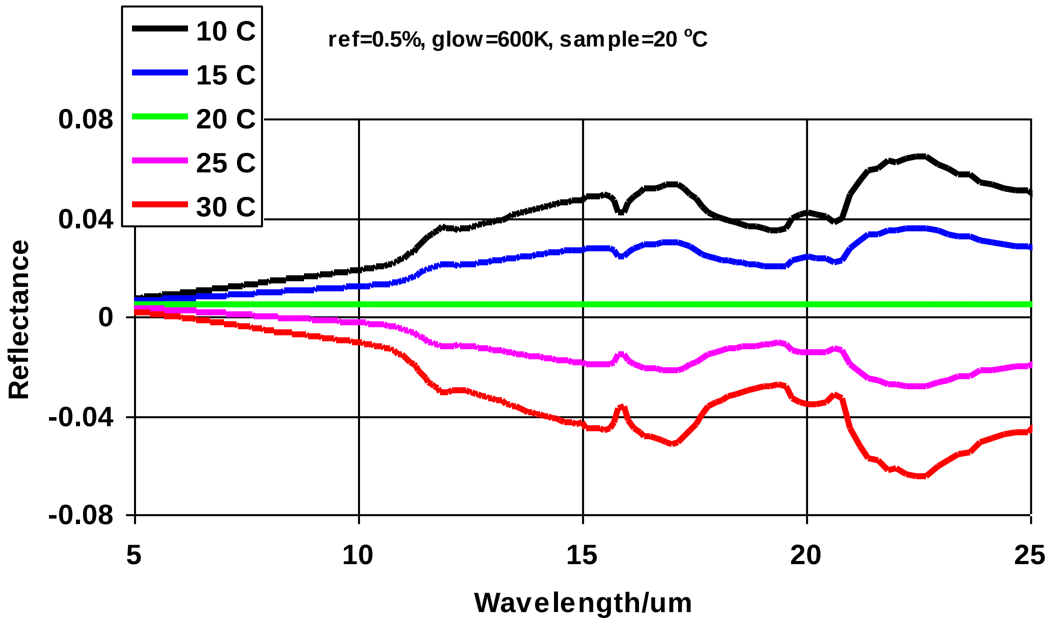

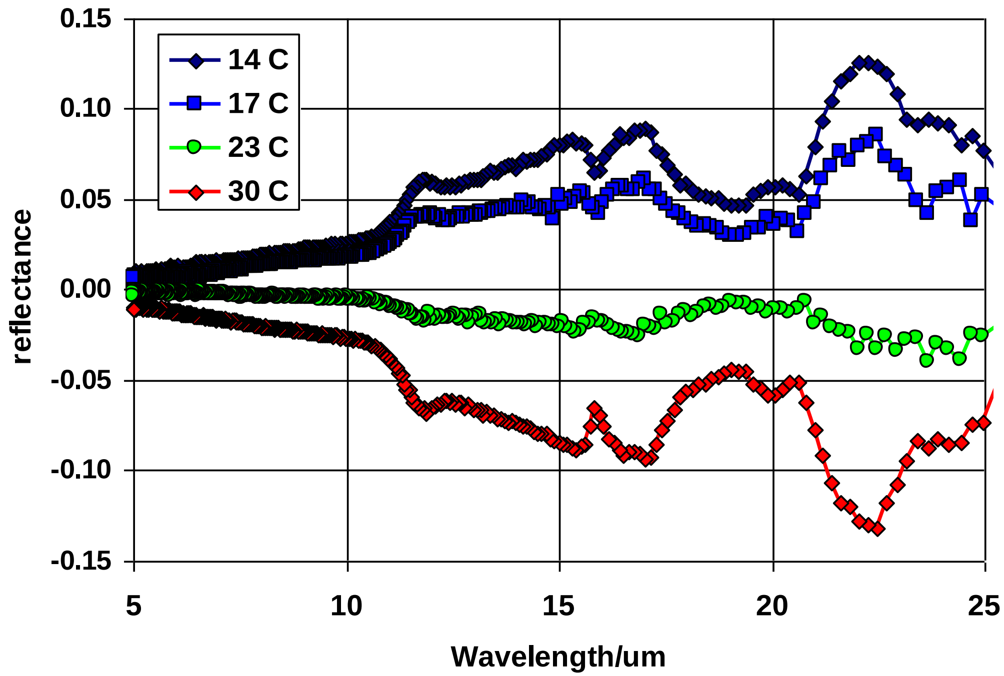

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The true hemispherical reflectance is given by:

$$R_S = \frac{M_H - B}{1 - B}$$

Where:

M_H is the measured reflectance and B is given by:

$$B = \frac{L(T_S) - L(T_{S0})}{(1 - R_G)[L(T_G) - L(T_{G0})]R_H}$$

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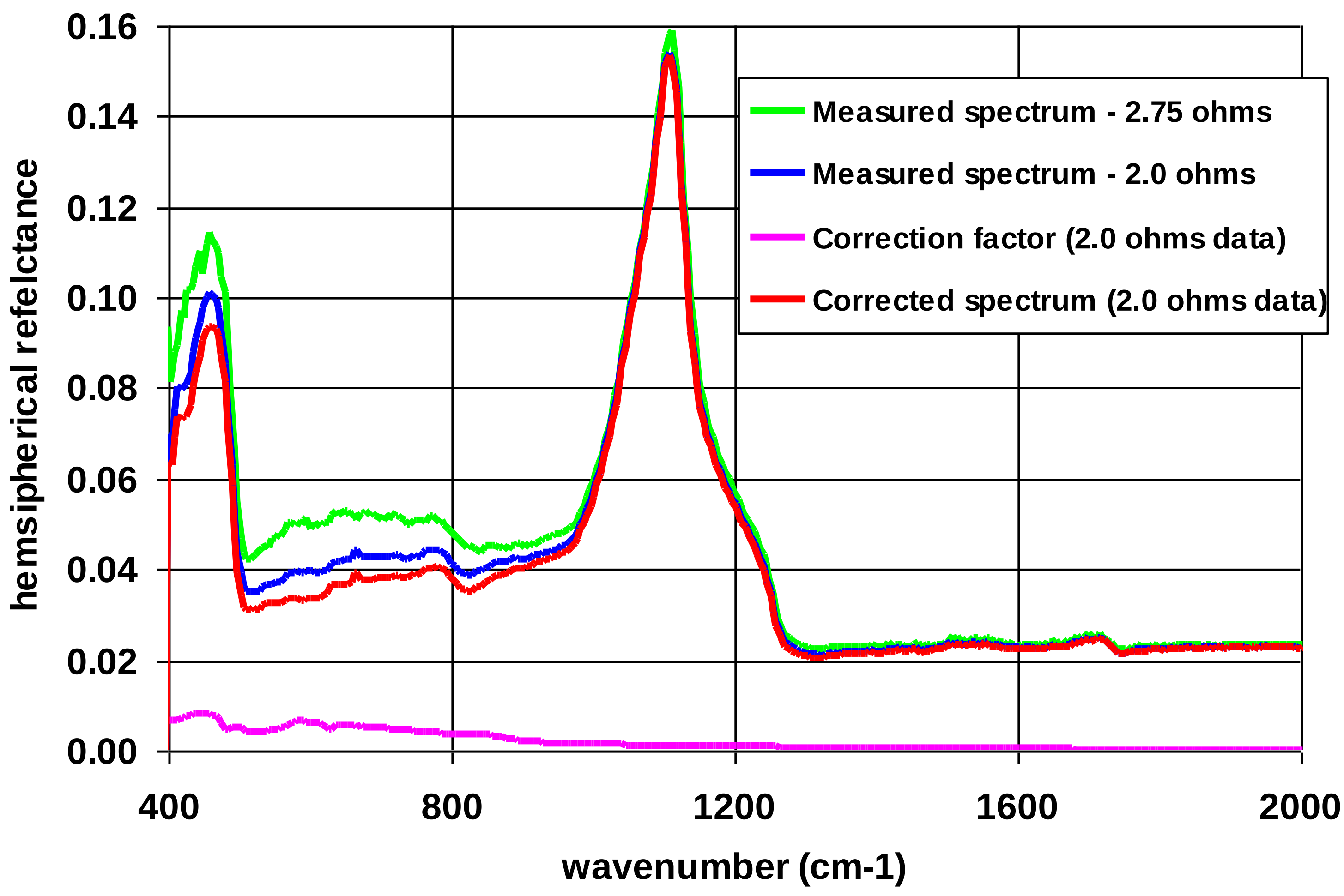
Hemispherical reflectance of Nextel black paint



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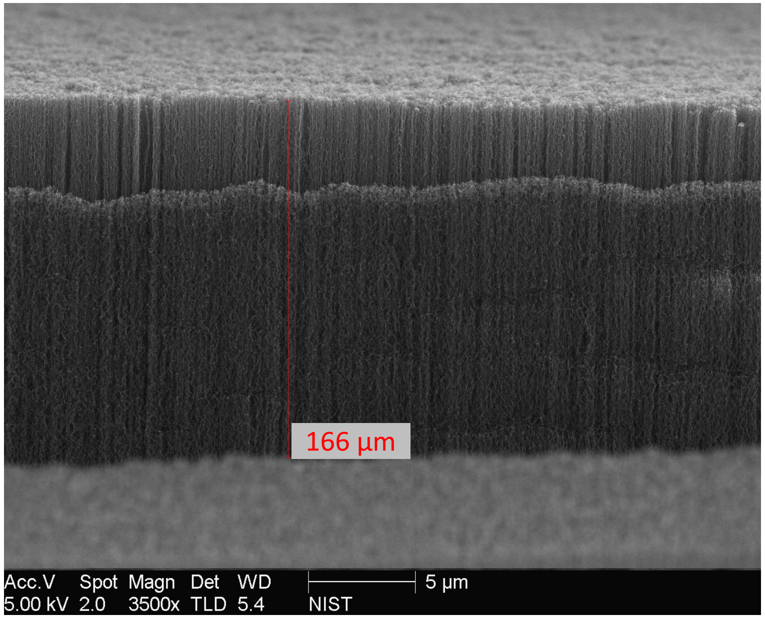
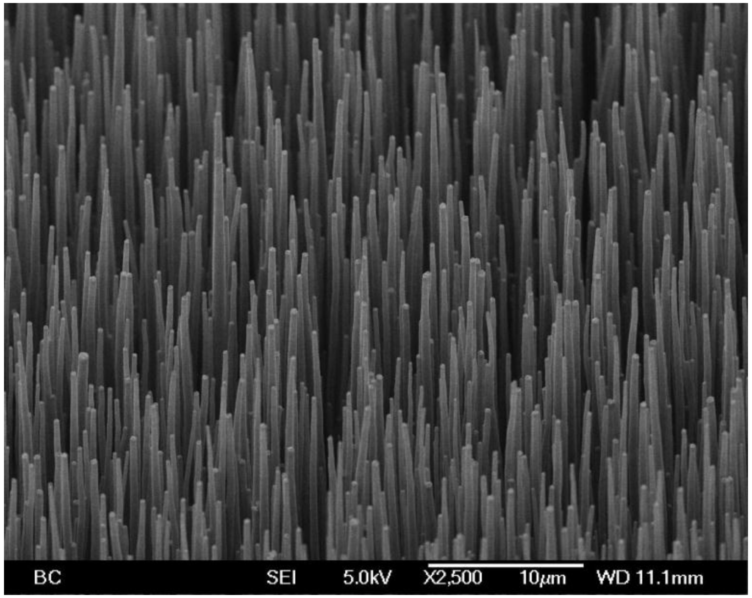
Vertically aligned MWCNT coatings





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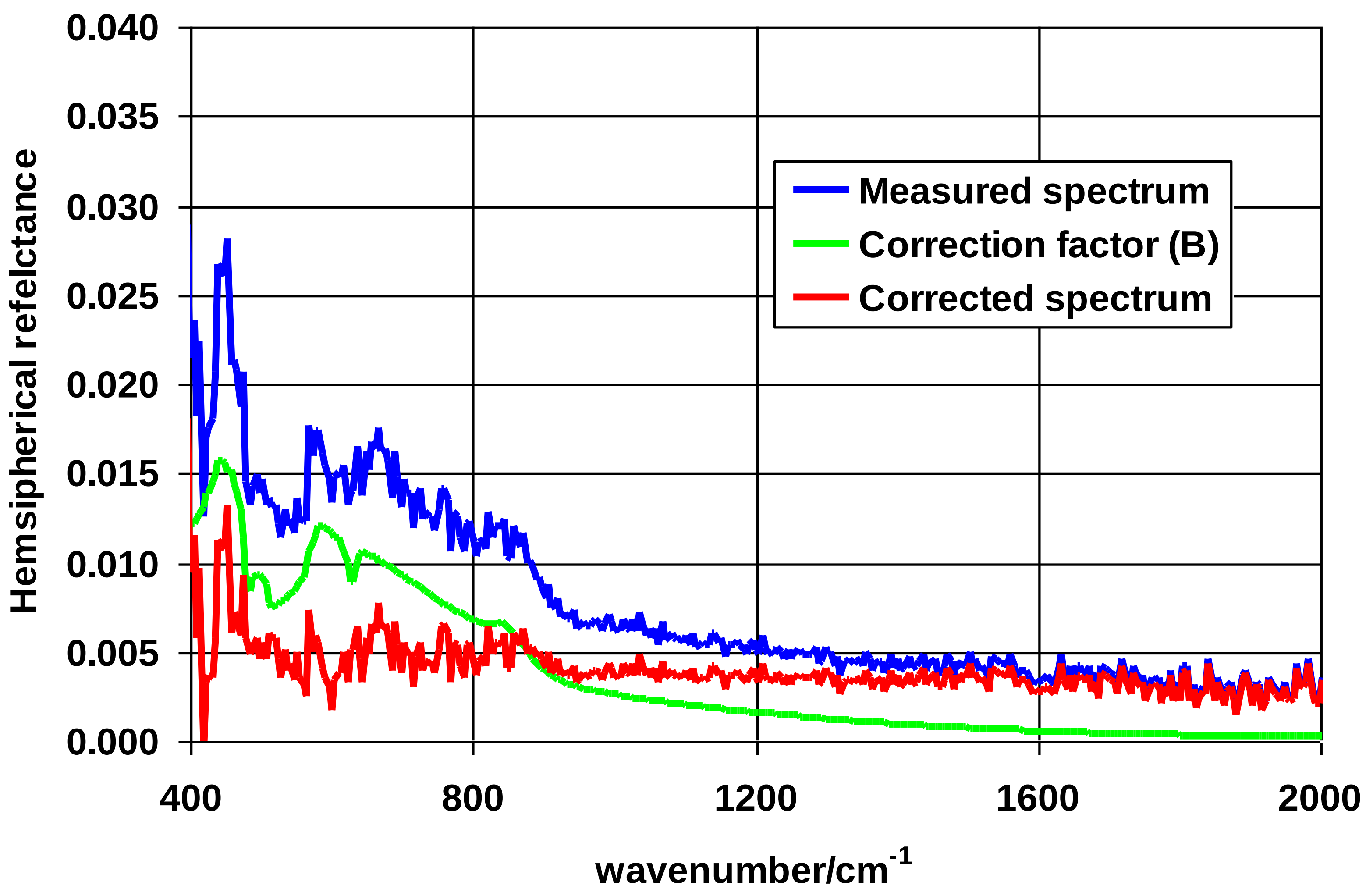
Hemispherical reflectance of 150 μm long vertical carbon nanotube forest (VANTA)



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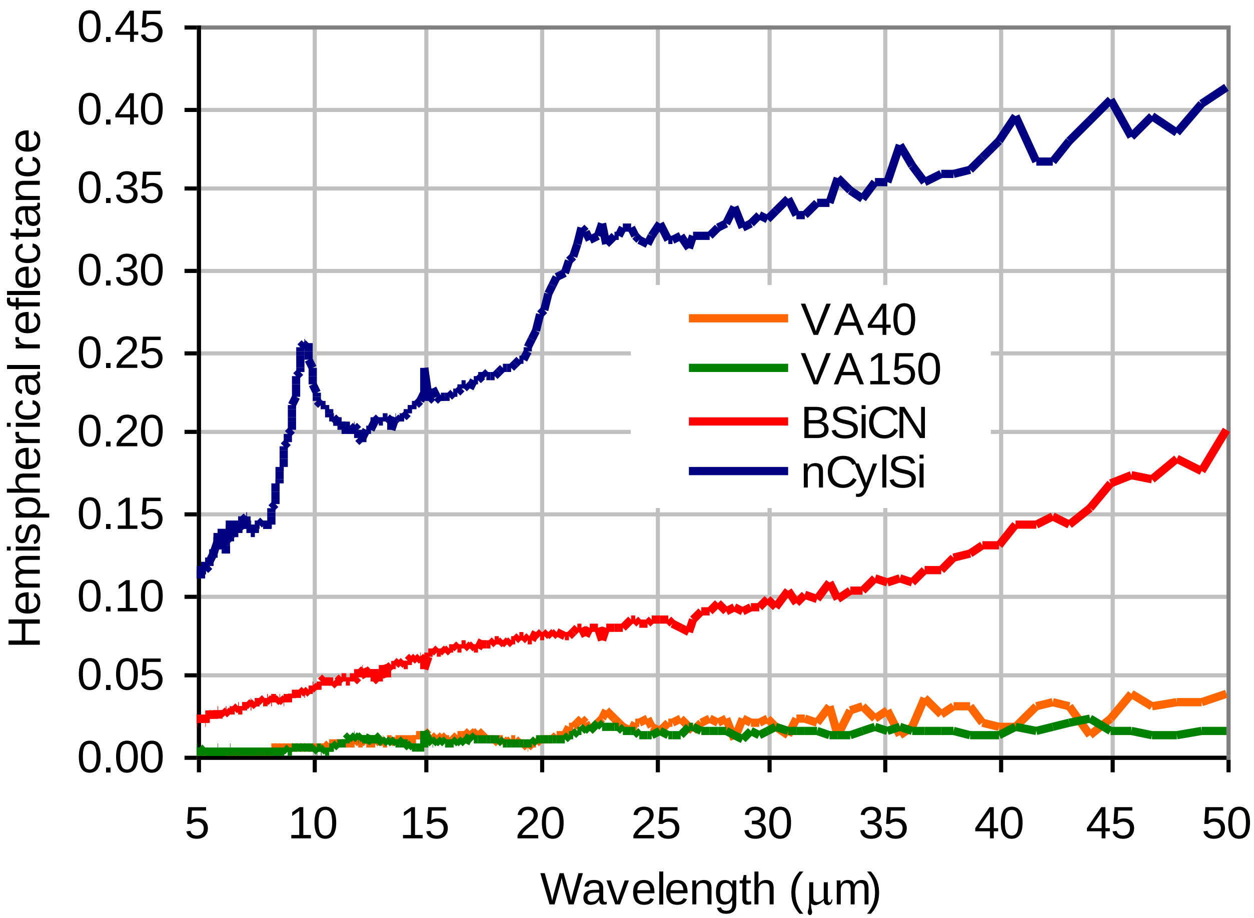
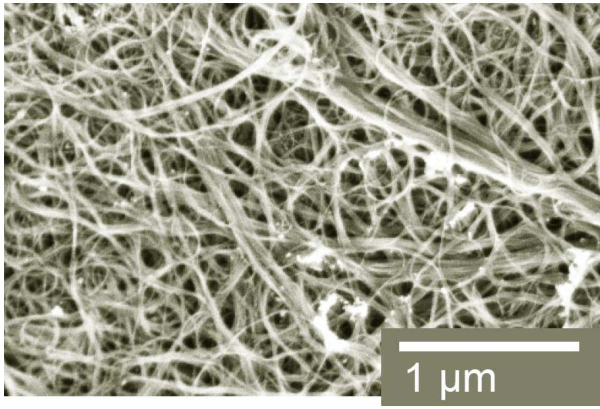
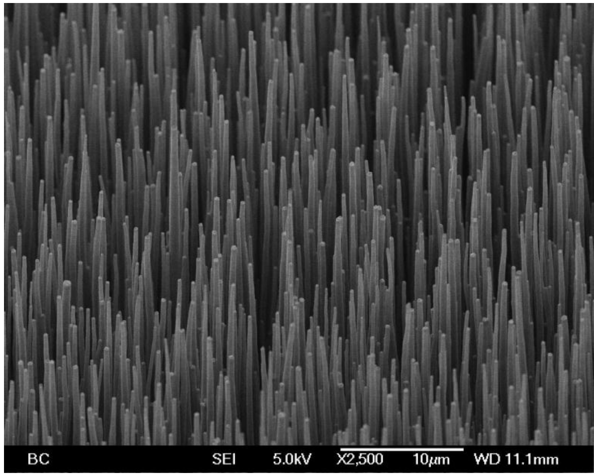
Hemispherical reflectance of MWCNT samples in the 5 μm to 50 μm range.

See poster OPM_OR_001, Result of NPL & NIST collaboration







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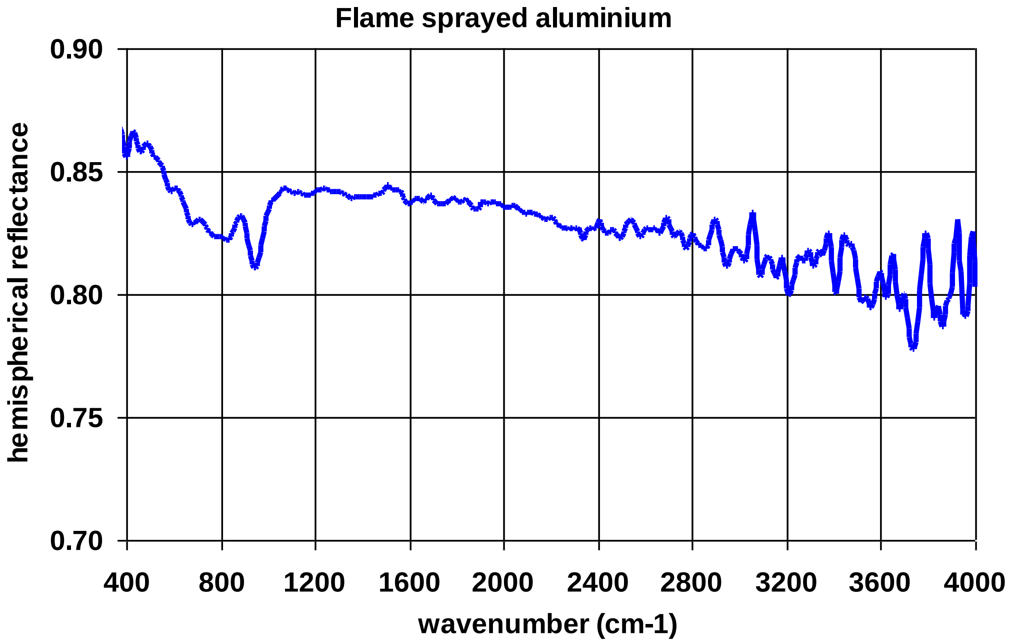

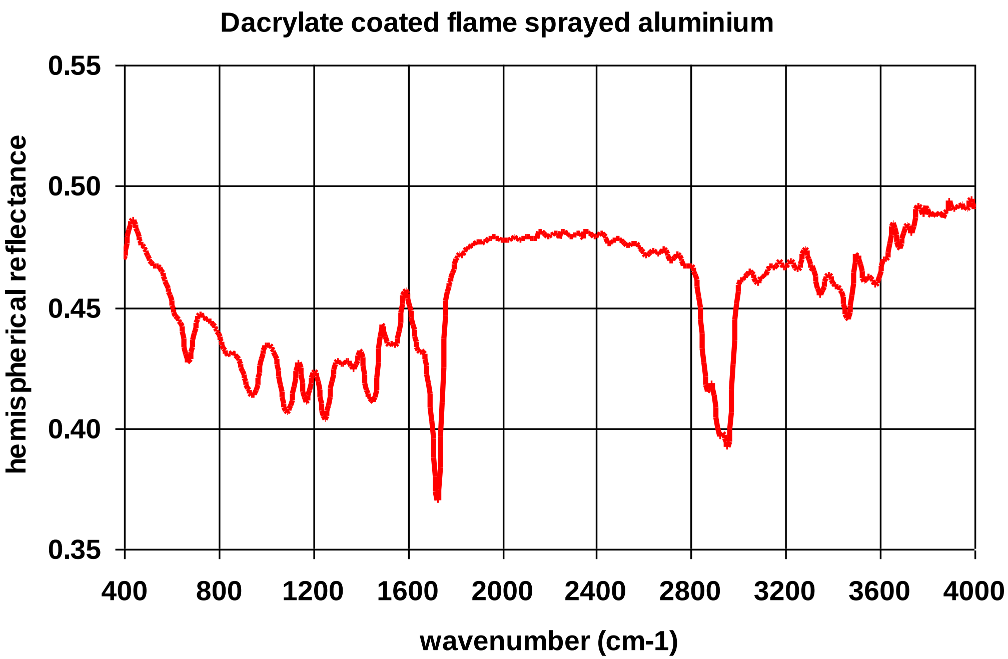

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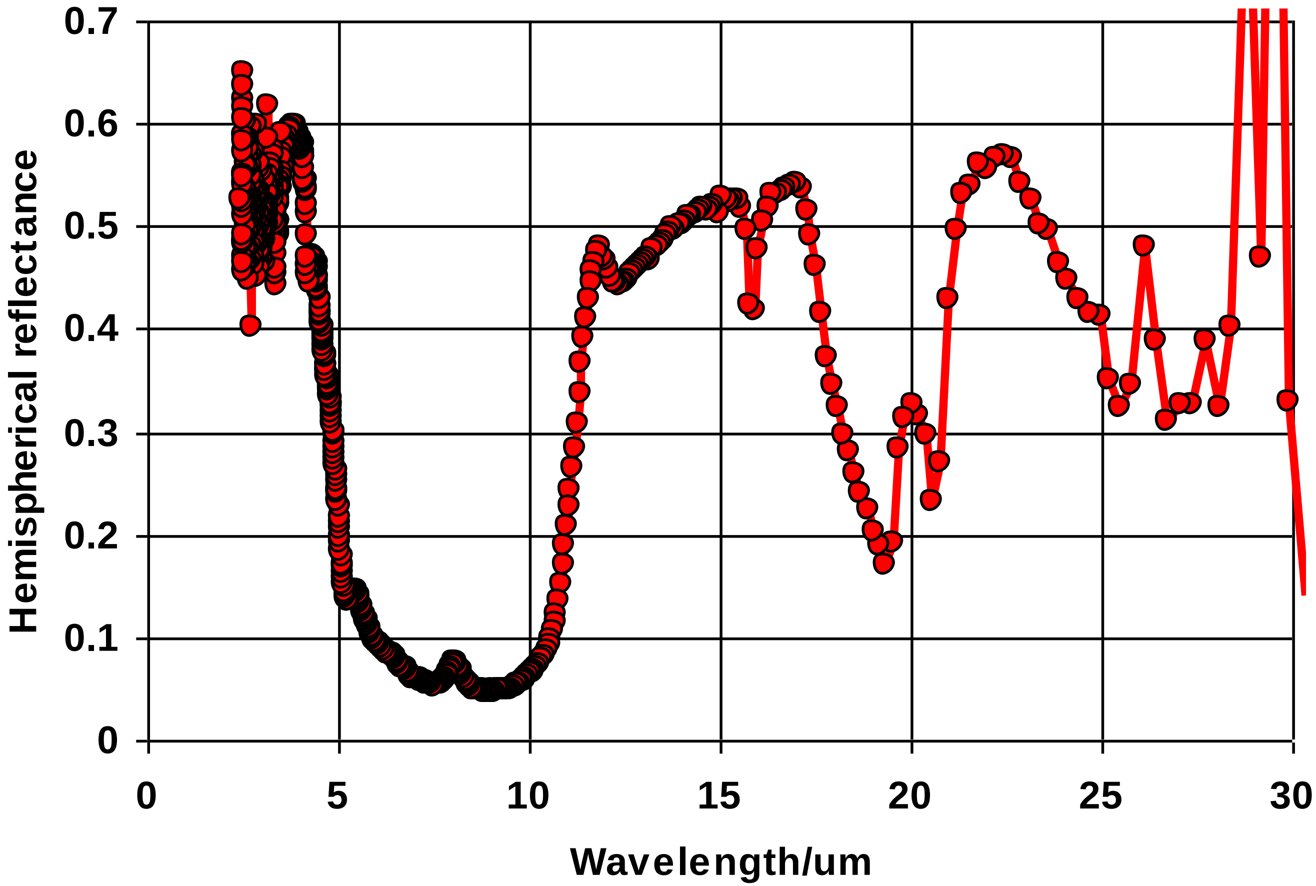
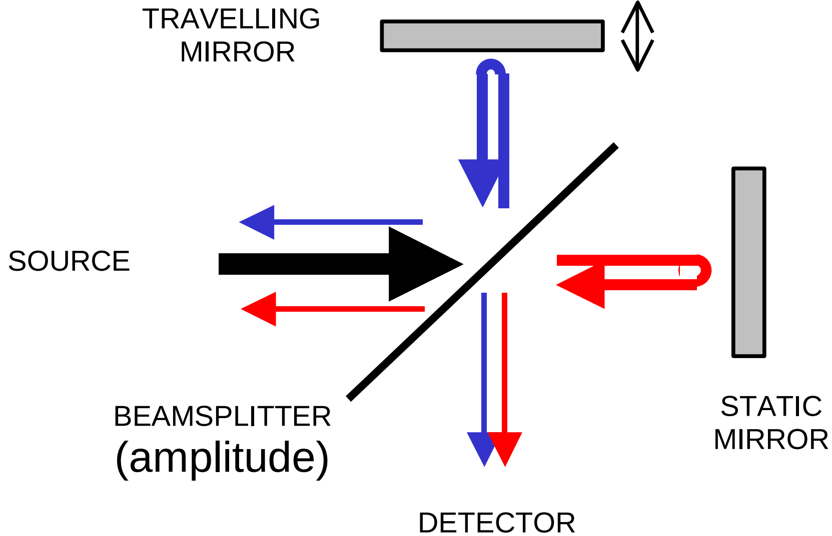
Hemispherical reflectance of the “Opperman” source





- Another issue identified is the treatment of the ‘dark’, or ‘source off’ measurements.
- There are spectral components in the interference functions for the measurements which are in anti-phase to those arising from light from the AHR entering the FTS through the emission port.
- These are due to thermal radiation from the FTS detector and sample compartments passing through the interferometer and being reflected back onto the detector.
- Therefore, all subtraction of ‘dark’ or ‘source off’ measurements, as well as any corrections for drift, must be carried out in the interferogram (time) domain, before Fourier transformation into the frequency (wavelength) domain.

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Advantages of using the hemispherical reflector approach:

High throughput, allows the use of thermal detectors

Measurements can be extended to 50 μm!

Disadvantages of using the hemispherical reflector approach

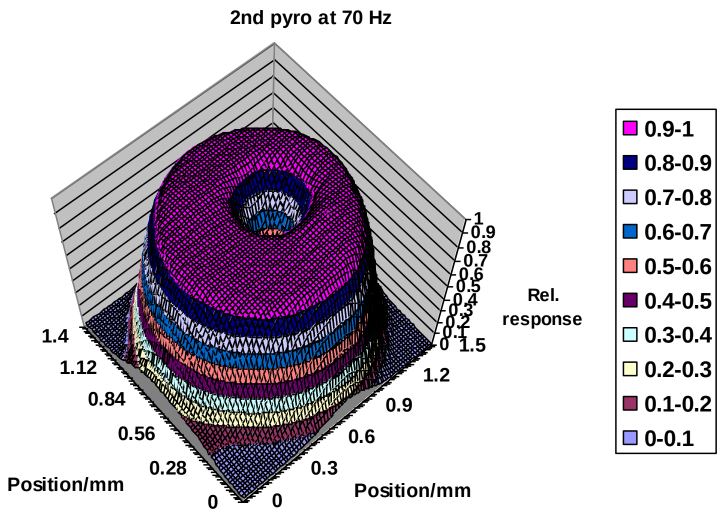
- Heating of the samples.
- Inter-reflections between source and test sample (only an issue for reflective (non-black) samples).
- Facility requires careful alignment. Now we are using visible radiation for alignment

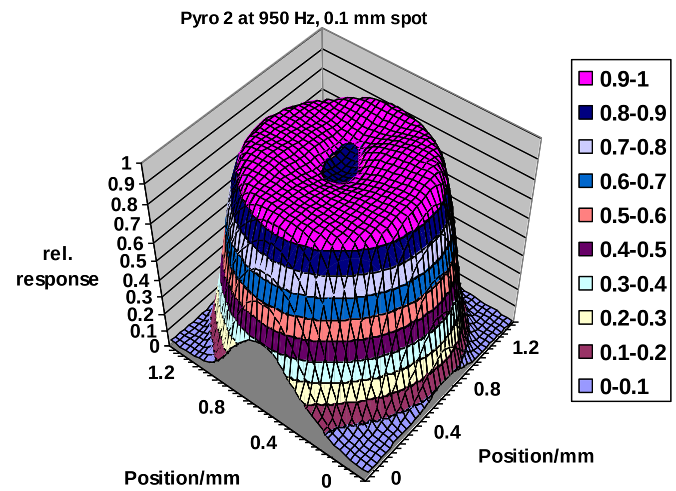
Issues with the hemispherical reflector method (cont).

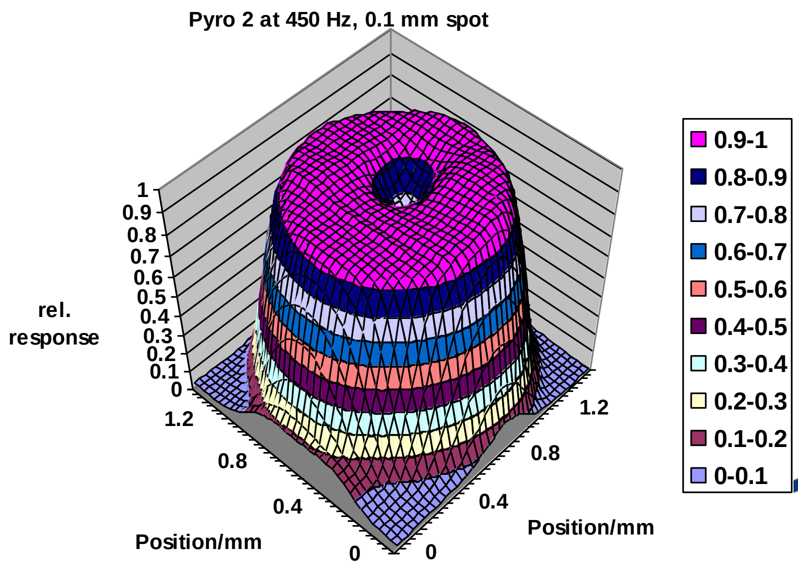
- Optical aberrations of the hemispherical mirror. Ideally an ellipsoidal mirror should be used.
- Source non-uniformity: Spatial non-uniformities in the illumination of the sample (Minimised by using a 1.8 mm diameter stop)
- Source angular illumination: Cylindrical source; some issue in the vertical direction, but small due to 1.8 mm diameter stop.
- Illumination of the sample over +-85^o (dishing). 0.5% of 2π. Correction applied.
- “Hole” on hemisphere. Corresponds to 1.3% of the 2π solid angle. Correction applied.
- Only small part of the sample being “analysed” (1.8 mm diameter spot). Spatial distribution of reflectance measured.
- Optically polished hemispherical reflector: effect of diffuse reflection from hemisphere is negligible.

	unc	Type	unit	
	k=2			
TYPE A COMPONENTS ESTABLISHED BEFORE EXPERIMENT				
System alignment	3.0	A	%	Relative
TYPE B COMPONENTS ESTABLISHED BEFORE EXPERIMENT				
Cut-out (1.32% of 2π)	0.07	B	%	Relative
Dishing (0.43% of 2π)	0.15	B	%	Relative
Variations in sample irradiance at large angles	0.50	B	%	Relative
Sample heating ($\sigma < 1000$ cm^{-1})	0.25	A	%	Absolute
Validity of CS, CE correction	2.00	A	%	Relative
Baseline uncertainty	0.30	B	%	Absolute
Drift in BB, FT and enclosure temperature, FT response				
FTS				
Detector non-linearity	0.20	B	%	Relative
Spectral aliasing	0.30	B	%	Relative
Inter-reflection effects	0.30	B	%	Relative
Wavenumber accuracy	0.10	B	cm-1	Relative
TYPE A COMPONENTS ESTABLISHED DURING EXPERIMENT				
Repeatability between repeated measurements (St.dev.) covers:		A	%	Relative
Reflectometer positioning during a measurement				
Small variations in sampled sample area				
Instability of the reflectometer source output				
Instability of the instrument over timescale of measurement				
S/N in spectra				
Drift of AHR alignment wrt FT				
Drift of source alignment wrt AHR				
Detector non-uniformity (0.6% maximum)				
Detector temperature variation (± 0.2 °C)				
Other effects due to temperature fluctuations (± 0.2 °C)				
Sample uniformity (if relevant)				

Spatial uniformity of DLATGS pyro at 70 Hz, 450 Hz and 860 Hz







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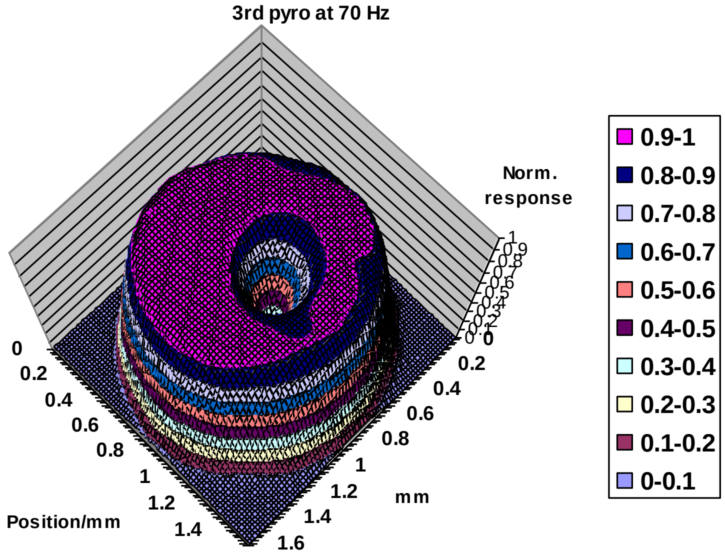
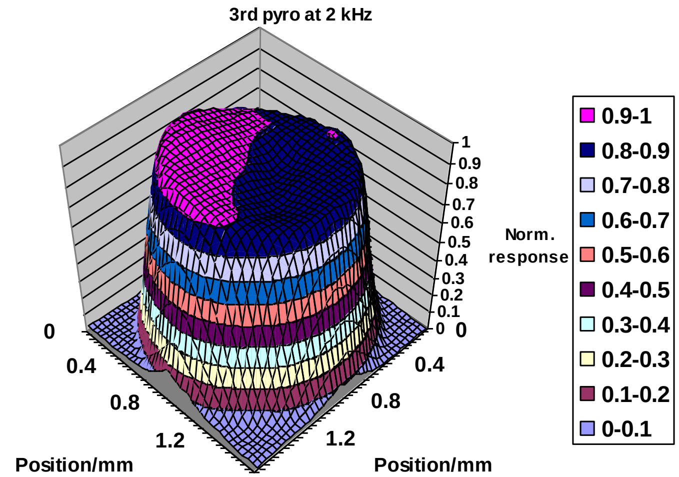
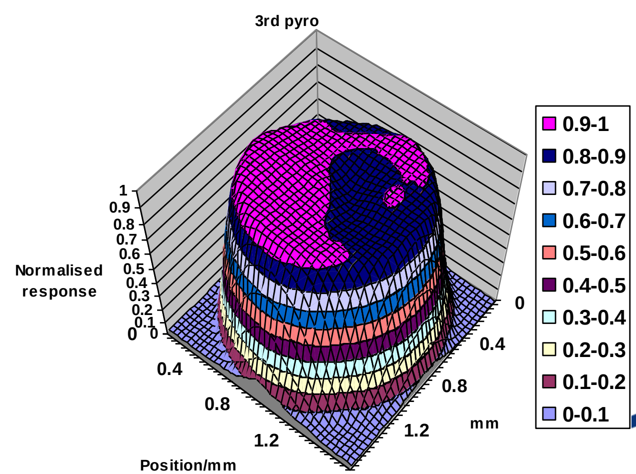
Spatial uniformity of DLATGS pyro at 70 Hz, 900 Hz and 2 kHz







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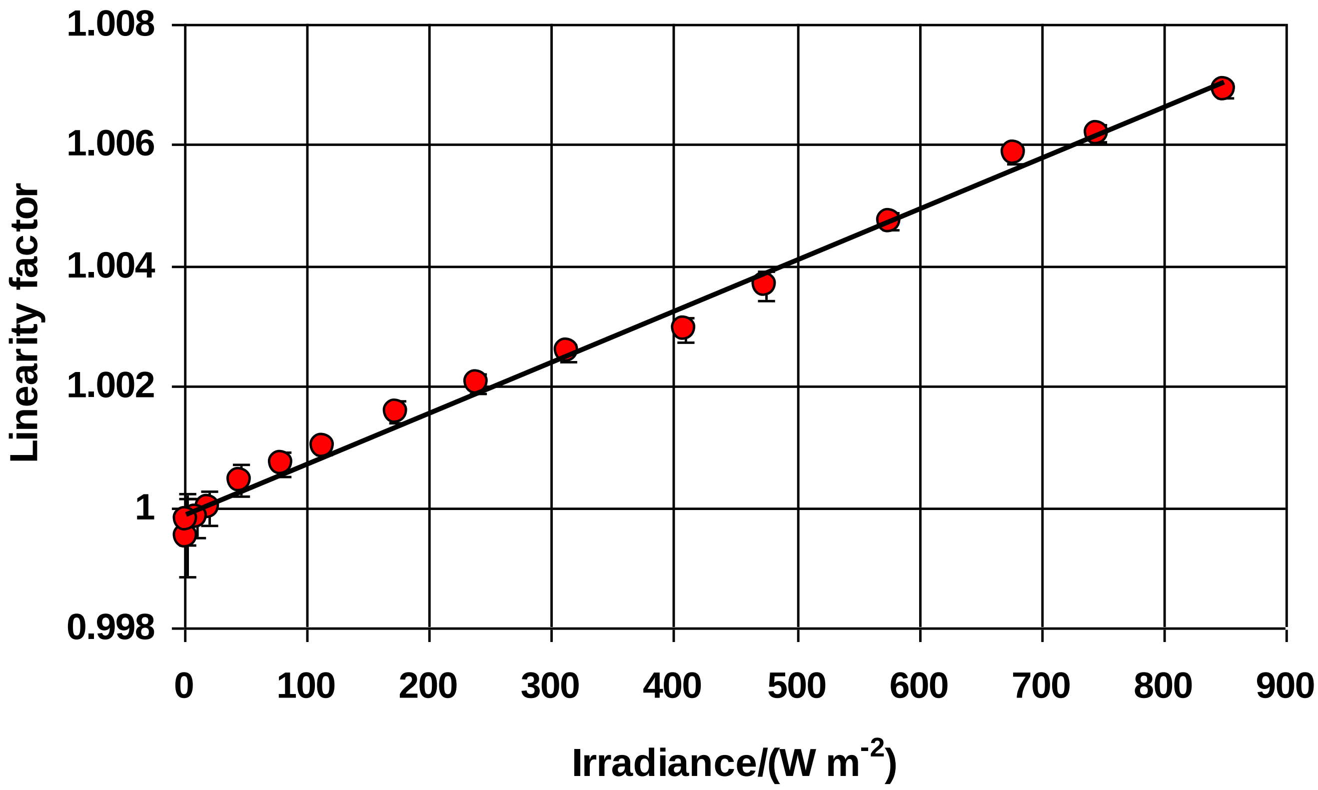
Linearity characteristics of DLATGS detector (1 mm diameter spot)

E. Theocharous, "Absolute linearity measurements on a gold-black-coated deuterated L-alanine-doped triglycine sulphate pyroelectric detector" *Applied Optics*, 47, 3731-3736, 2008.



New IR gonio-reflectometer system

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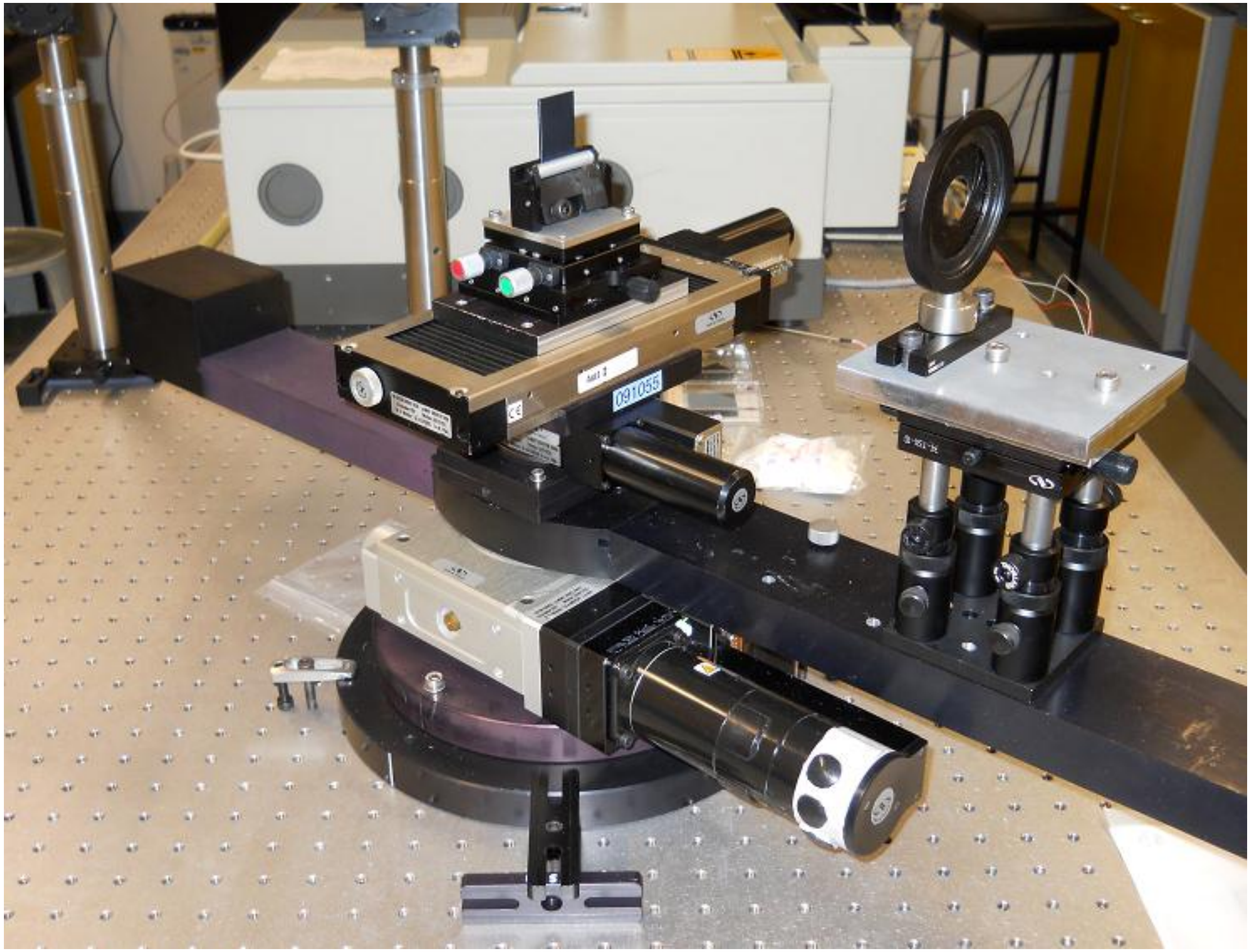
New IR gonio-reflectometer system



Conclusions

- The NPL directional-hemispherical reflectance measurement facility has been upgraded.
- The grating spectrophotometer was replaced by a Fourier transform spectrometer.
- Other improvements include:

  better alignment (using visible radiation),

  use of an intermediate field stop,

  minimisation of the effects of heating of the sample

  correction of the instrumental and relay optics drift by using a blackbody.

  Measurements obtained with the new facility were presented.

* We are open for business

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Thank you for listening

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