Temperature Calibration of Thermal/Optical Carbon Analyzer

John Watson (john.watson@dri.edu)

Judith C. Chow
Xiaoliang Wang
Dana L. Trimble
Steven D. Kohl

Desert Research Institute, Reno, NV

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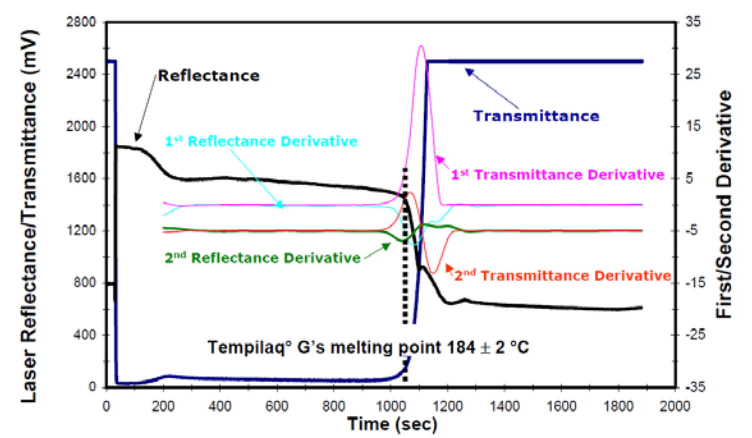
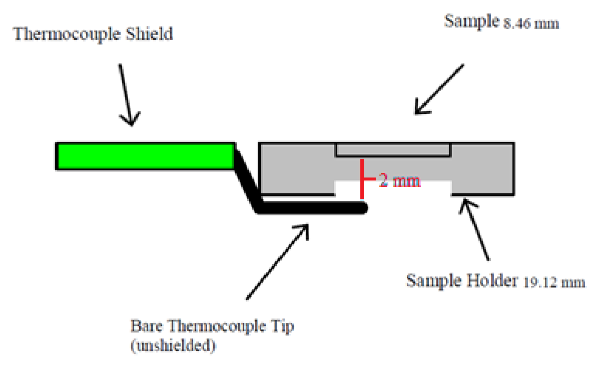
Objectives

- Review current temperature calibration method with Tempilaq*



(Tempilaq's melting point 184 ± 2 ºC)



- Introduce new calibration method using infrared emission spectroscopy

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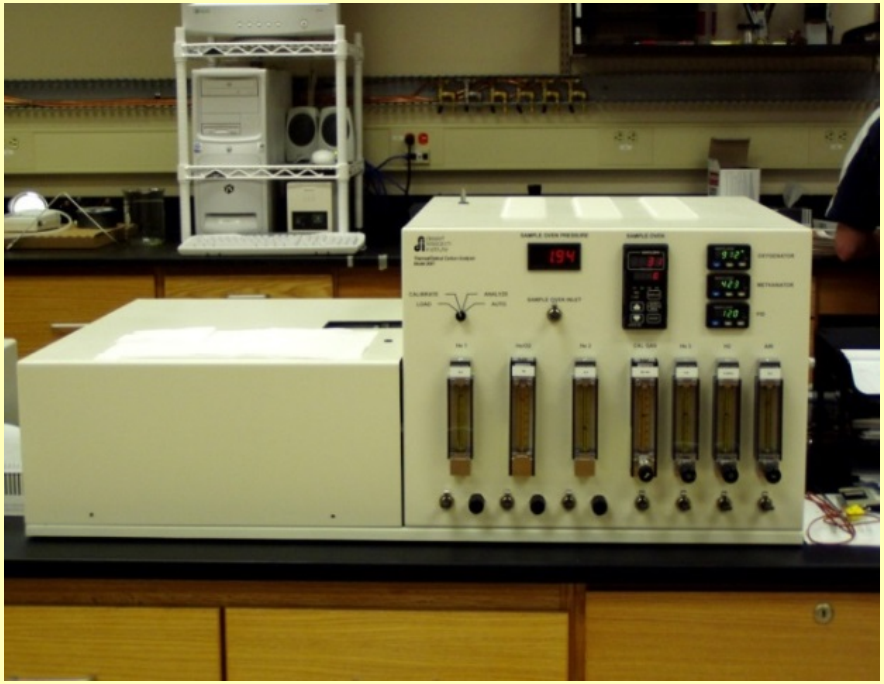
* Quick-drying temperature indicating liquids

Temperature calibration was initiated for Model 2001 during 2004/2005

DRI/OGC (1987-2004)

DRI Model 2001 (2005-2015)



Advantages of Temperature Calibration with Tempilaq

- Use temperature-sensitive compounds that change appearance when they reach target temperatures (i.e., 121, 184, 253, 510, 704, and 816°C)

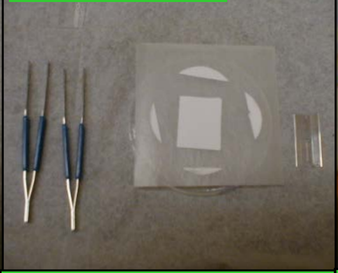
- Relate filter sample temperature to the sensor (i.e., thermocouple) temperature

Disadvantages of Temperature Calibration with Tempilaq*

- Procedures are cumbersome (~8 hours/calibration)

1. Collect supplies.

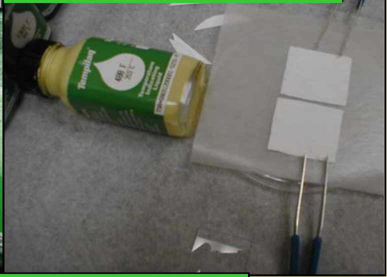
2. Cut the filter.



3. Split the quartz-fiber filter.

4. Choose a Tempilaq melting point liquid.

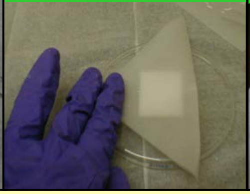
5. Prepare the Petri dish set.



6. Spread the Tempilaq evenly.

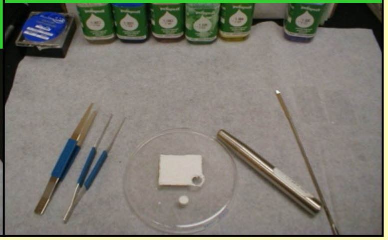
7. Spread Tempilaq on both sides.

8. Put both slides back and cover with membrane substrate.



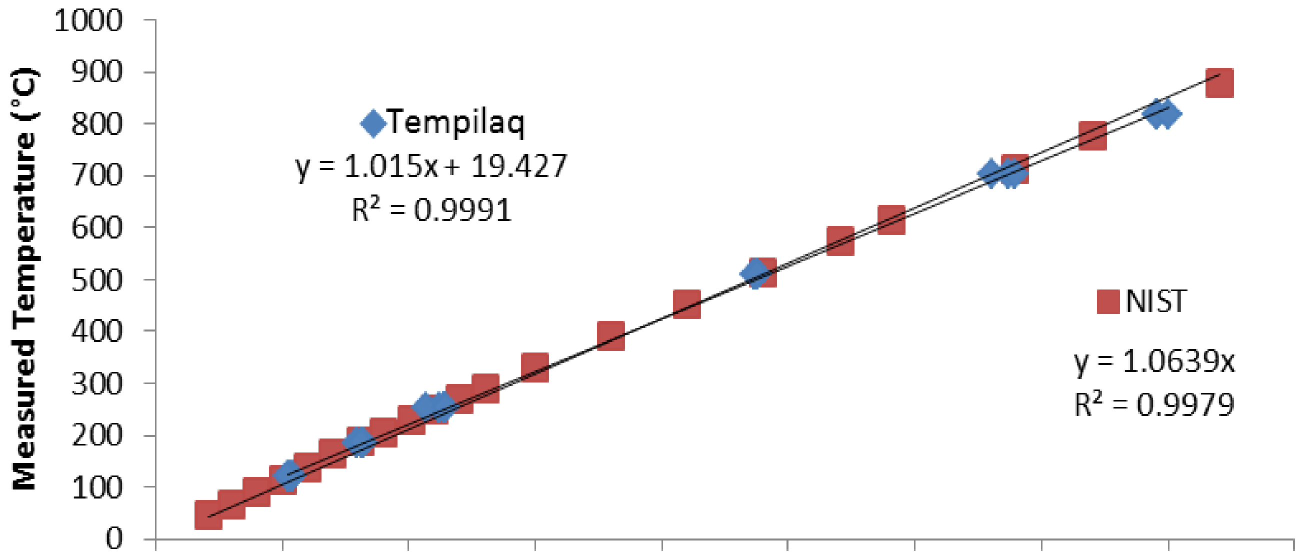
9. Firmly press entire Tempilaq sample to ensure homogeneity within the quartz-fiber filter.

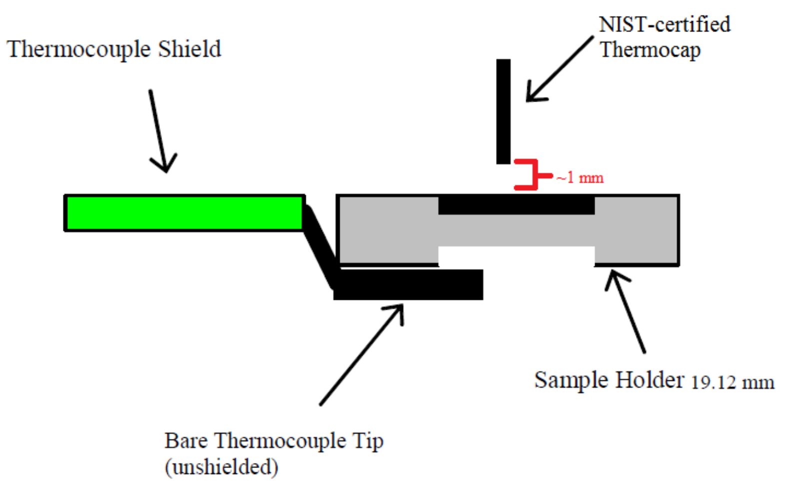
10. Take a punch for analysis.



- Tempilaq contaminates the oven and requires instrument recalibration

NIST-certified thermocouple* gives shows small deviations for high temperatures, possibly due to different heating characteristics compared to the sample





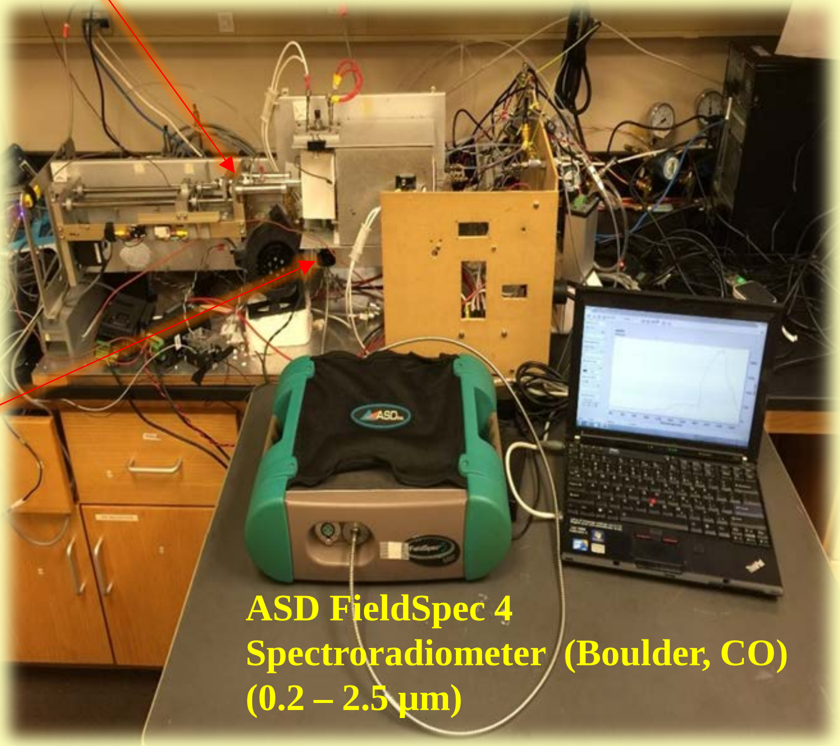
- Indirect method to measure sample temperature, serves as QC check

A graphite disc is a blackbody that simulates heating characteristics of the quartz filter

Make graphite punches

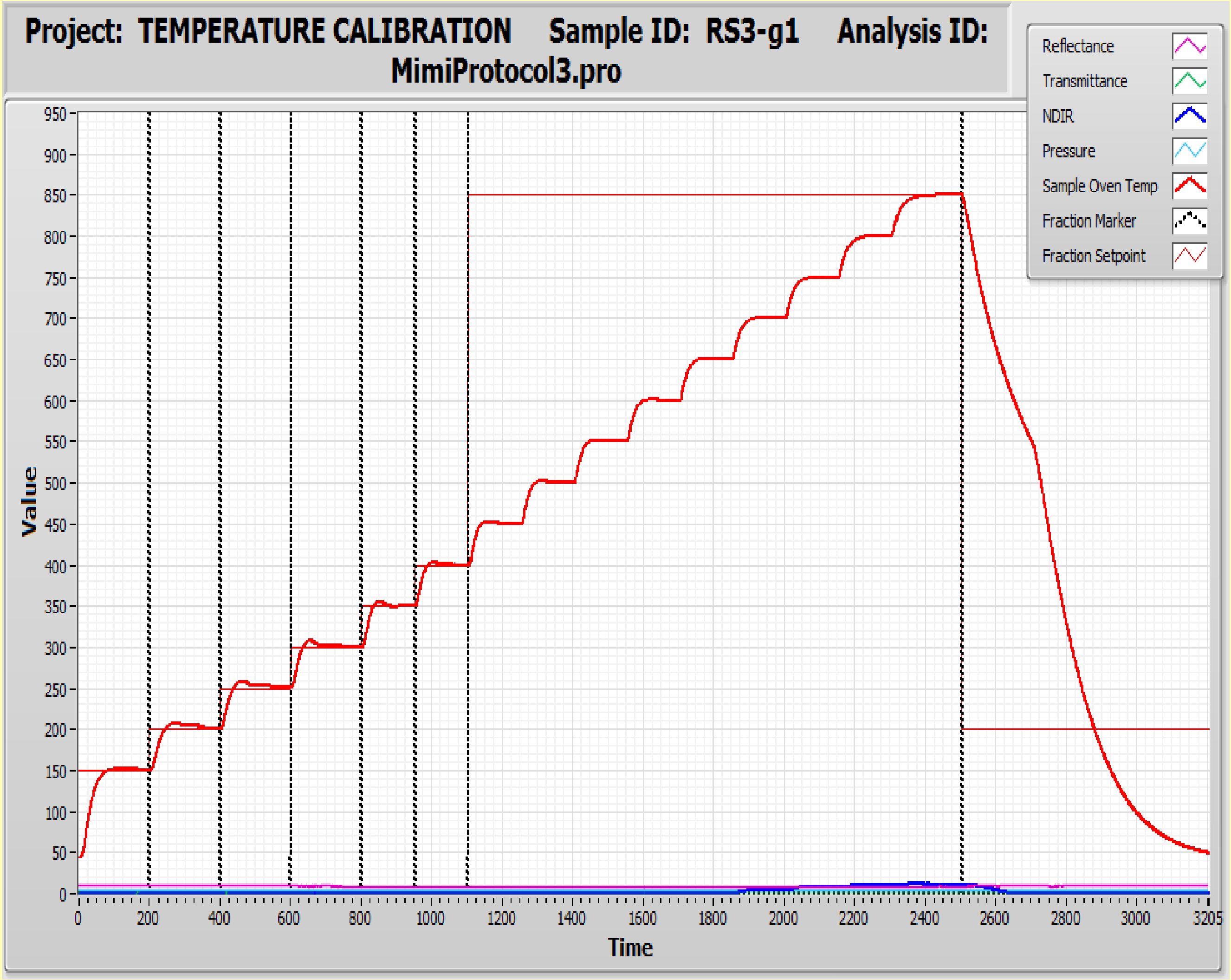
Load a punch into carbon analyzer

Interface the transmittance light pipe with spectrometer



ASD FieldSpec 4 Spectroradiometer (Boulder, CO) (0.2 – 2.5 µm)

Incremental temperature steps (100 – 850ºC, ~50ºC per step) allow for different levels of radiation output

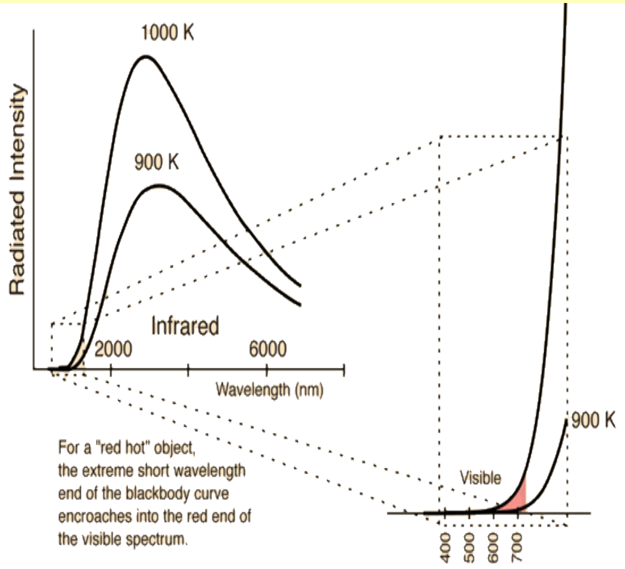


Project: TEMPERATURE CALIBRATION Sample ID: RS3-g1 Analysis ID: MimiProtocol3.pro

Principle of Thermal Radiation and Ratio Pyrometer

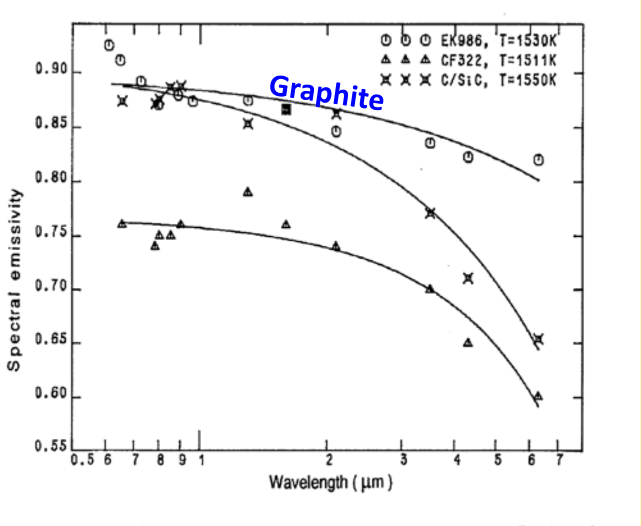
$$\underbrace{L_\lambda\left(\varepsilon_{obj},T_{obj}\right)d\lambda}_{\text{Thermal Radiance}} = \underbrace{\varepsilon_{obj}\left(\lambda,T_{obj}\right)}_{\text{Emissivity}}\underbrace{\frac{2hc^2}{\lambda^5}\frac{1}{e^{\frac{hc}{\lambda kT_{obj}}}-1}}_{\text{Blackbody Radiation}}d\lambda$$

- Radiance signal ratio for two similar wavelengths (SR_{λ_1/λ_2}) is a function of the heated object's temperature (T_{obj} assuming constant emissivity in selected regions)
- Since graphite emissivity is nearly the same for nearby λs, it cancels out when radiances are ratioed



For a "red hot" object, the extreme short wavelength end of the blackbody curve encroaches into the red end of the visible spectrum.

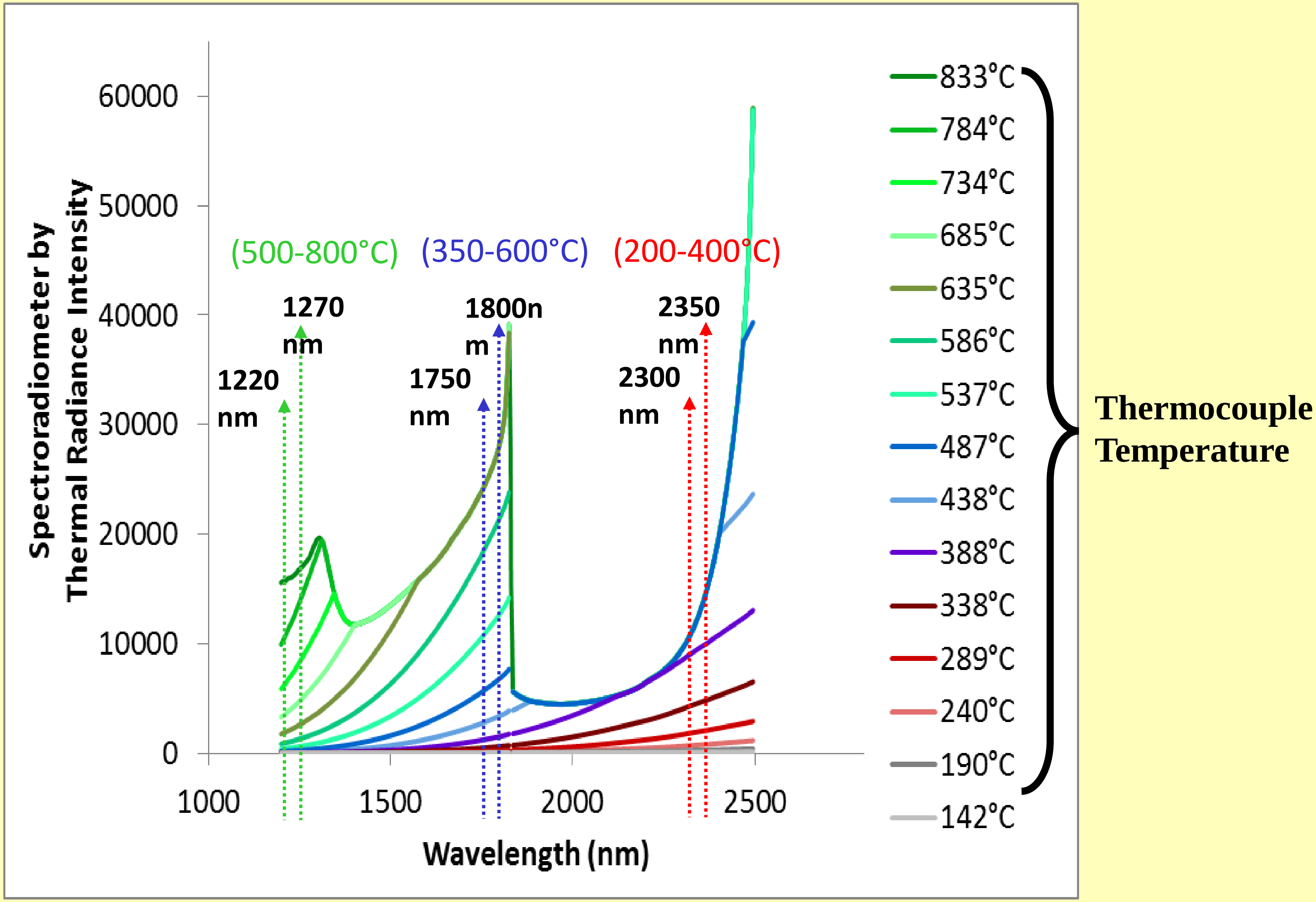
Blackbody Radiation



Spectral normal emissivity of graphite (EK986) carbon/carbon (CF322) and carbon/siliconcarbide (C-SIC) composites

Neuer, G., (1995). Spectral and Total Emissivity Measurements of Highly Emitting Materials. International Journal of Thermophysics 16, 257-265.

Radiation intensity is measured at nearby wavelengths with an IR spectrometer

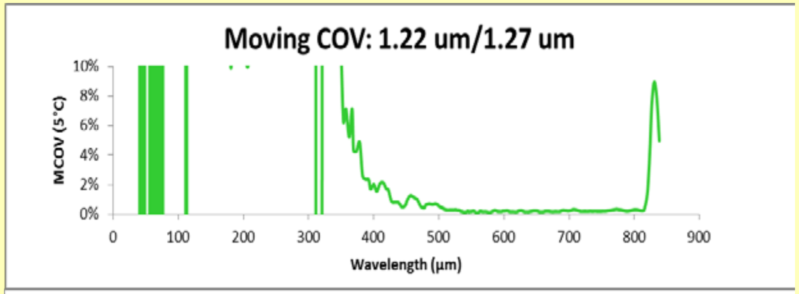


- The spectroradiometer has been calibrated with blackbody standards. Dynamic range of spectroradiometer differs by wavelength.

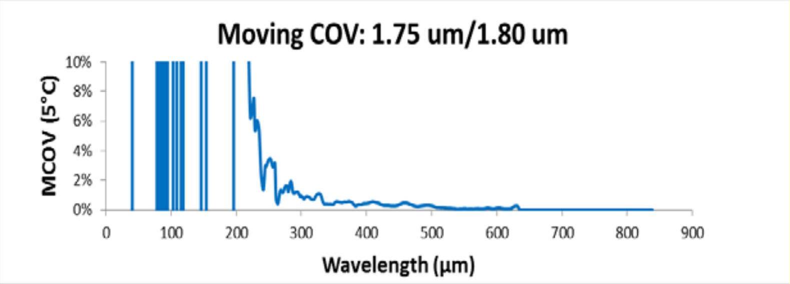
Nearby wavelengths are selected for the best signal ratios (SR_λ)

Moving Coefficient of Variance $\mathrm{MCOV}(T) = \dfrac{\mathrm{Std}(SR_{T-2℃\rightarrow T+2℃})}{\mathrm{Avg}(SR_{T-2℃\rightarrow T+2℃})}$

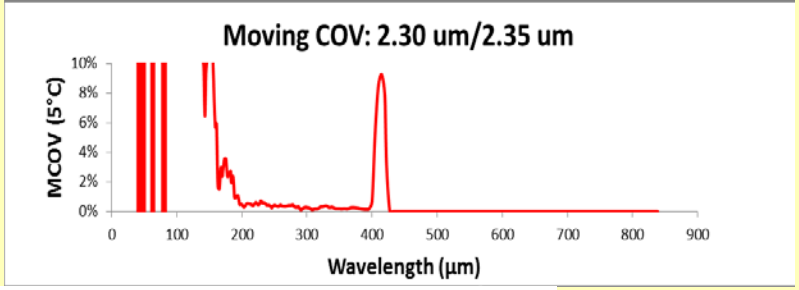
(Assuming that SR varies smoothly within every 4°C interval, MCOV should be small)



$SR_{1.22\ \mathrm{nm}/1.27\ \mathrm{nm}}$ valid for 500 – 800 °C



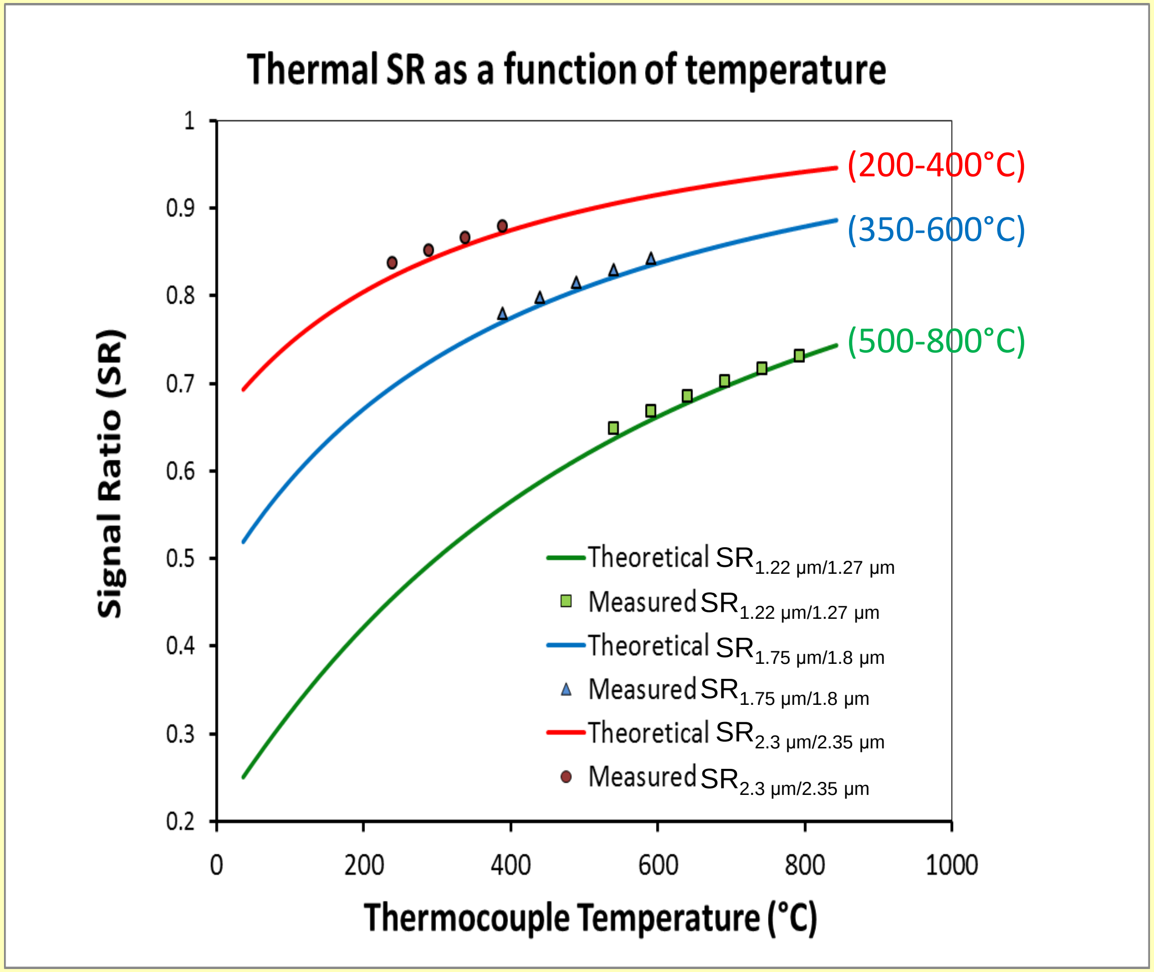
$SR_{1.75\ \mathrm{nm}/1.80\ \mathrm{nm}}$ valid for 350 – 600 °C



$SR_{2.30\ \mathrm{nm}/2.35\ \mathrm{nm}}$ valid for 200 – 400 °C

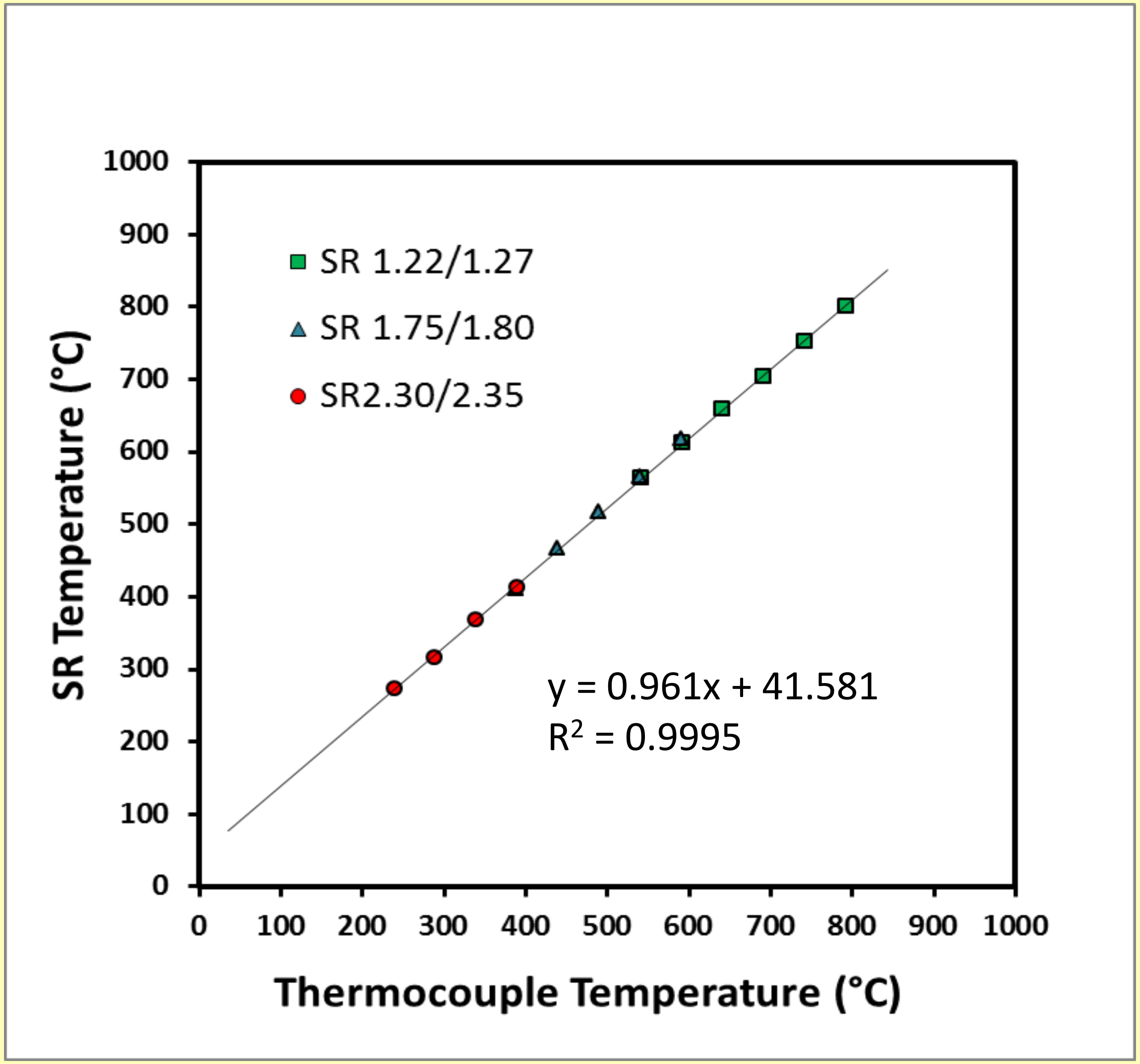
$$SR\,(\lambda_2,\lambda_1) = \text{signal ratio}\,(\lambda_2,\lambda_1) = \frac{L_{\lambda_2}(\varepsilon_{obj},T_{obj})}{L_{\lambda_1}(\varepsilon_{obj},T_{obj})} = \frac{\lambda_1^5}{\lambda_2^5}\,\frac{e^{\frac{hc}{\lambda_1 kT_{obj}}}-1}{e^{\frac{hc}{\lambda_2 kT_{obj}}}-1}$$

Theoretical and measured signal ratios (SRs) correspond well



- Based on the average of five replicate analyses. Only measurements validated for the corresponding wavelength bands (see last slide) are included.

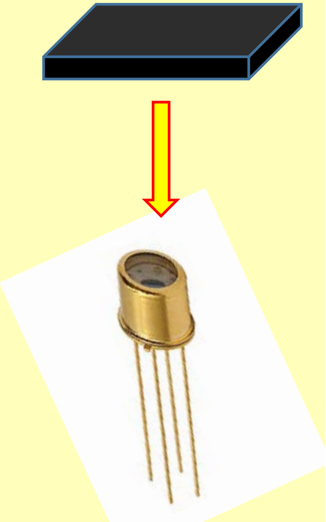
Normal thermocouple underestimates sample (SR) temperature by 10 – 35°C with good correlations



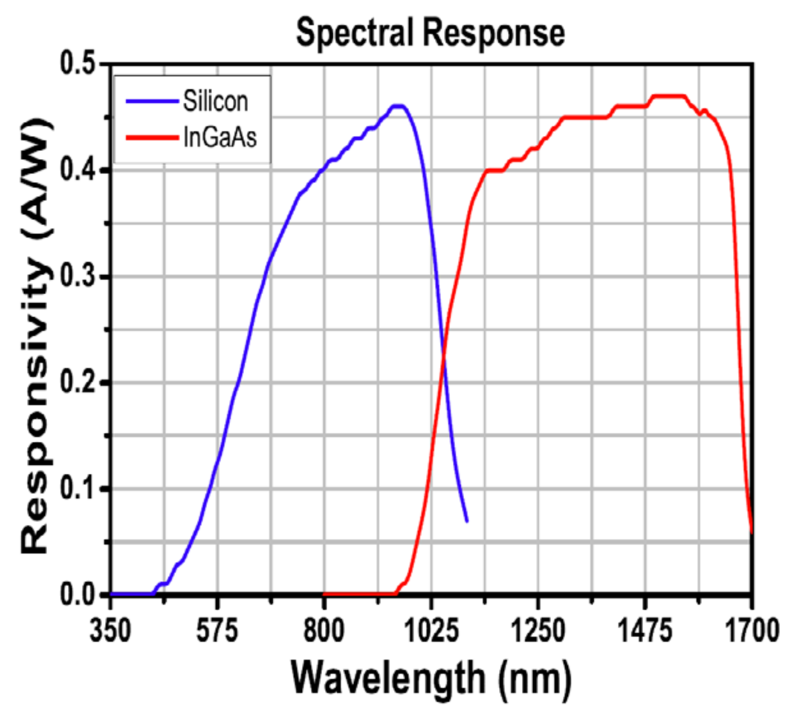
The IR spectrometer demonstrates feasibility, but its >$60k cost makes it impractical

Dual channel photodiode sensors* may be adaptable to this method

Graphite Surface



Thorlabs DSD2
Si/InGaAs Sensor



- The radiance ratio of the two band is dependent on temperature.

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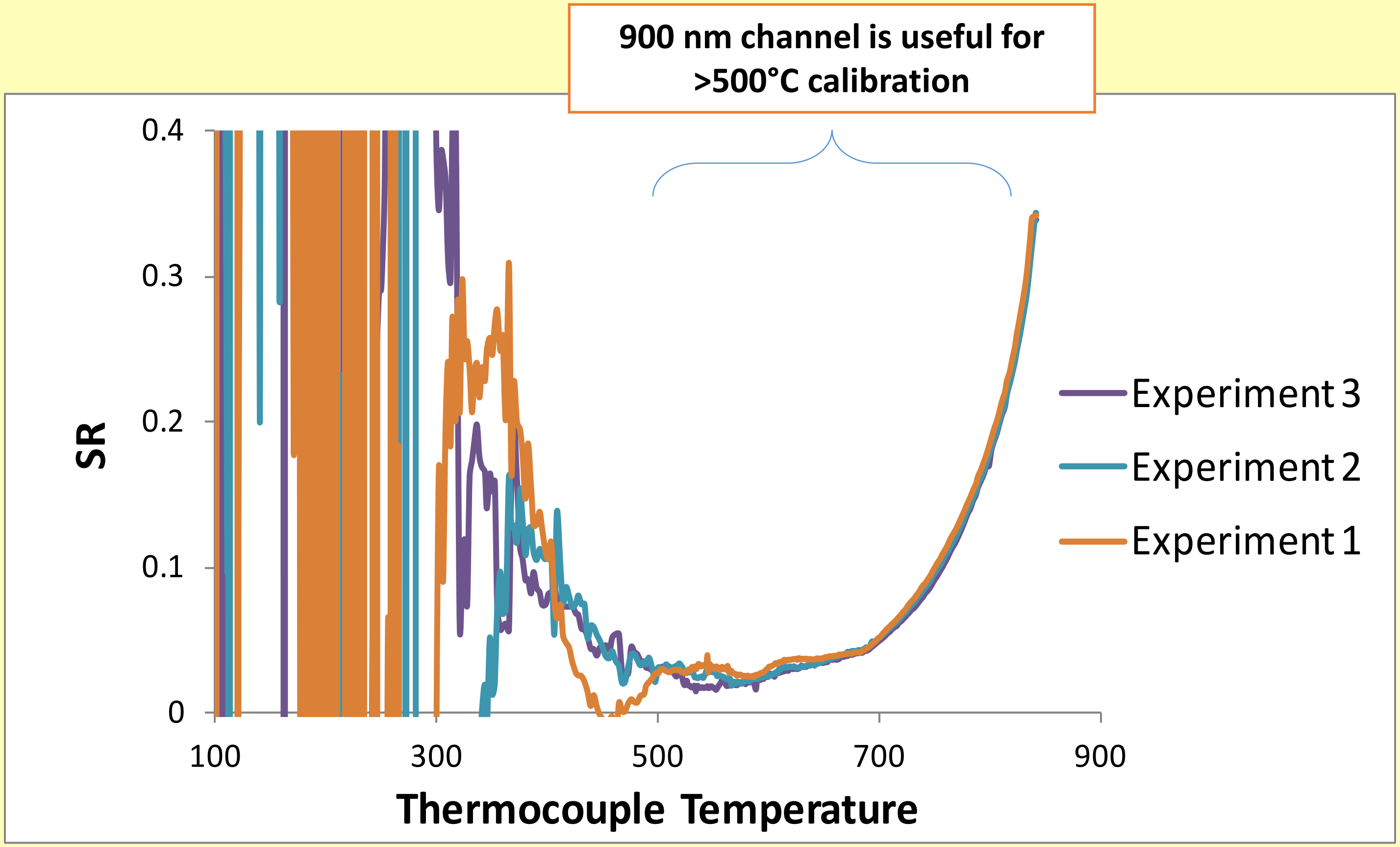
*Two bands centered at 900 and 1500 nm

Photodiode sensors detect a broad band of blackbody radiation and require external calibration for the selected regions (150 – 850 °C)


900 nm channel is useful for >500°C calibration

Conclusion

- Temperature calibration based on signal ratio (SR) of infrared emission spectroscopy provides direct detection of sample temperature and is easy to implement

- Infrared emission spectroscopy produces good sensitivity (<200°C) and stability for λ in the 1 – 3.5 µm range, but equipment is costly

- More tests are needed on less costly sensors (2-3 µm band) and band-pass filters λ