NIST IR Materials Index Project Update

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Project Goals

1) Demonstrate NIST capabilities for making refraction index measurements of key IR materials at leading-edge absolute accuracies at needed λ ranges.
   - transmitting λs out to ~14 μm
   - some IR materials, e.g. ZnSe, ZnS, BaF_2, transmit in visible

2) Establish reliable generic values for key IR materials at high accuracy that can used by industry.

3) Explore spread of index values among commercial materials.

- Achieving goals 2) and 3) requires meeting certain minimum absolute accuracies.
  - Approximate target $\leq 50 \times 10^{-5}$ in near-mid IR range.
  - Needed accuracies depend on materials and λ-ranges.
  - Higher accuracies for visible λs. For some materials that transmit in visible – must compare to higher accuracy meas.
  - Higher accuracies required to assess index variations.

Delivered Results for one sample of Ge[1]

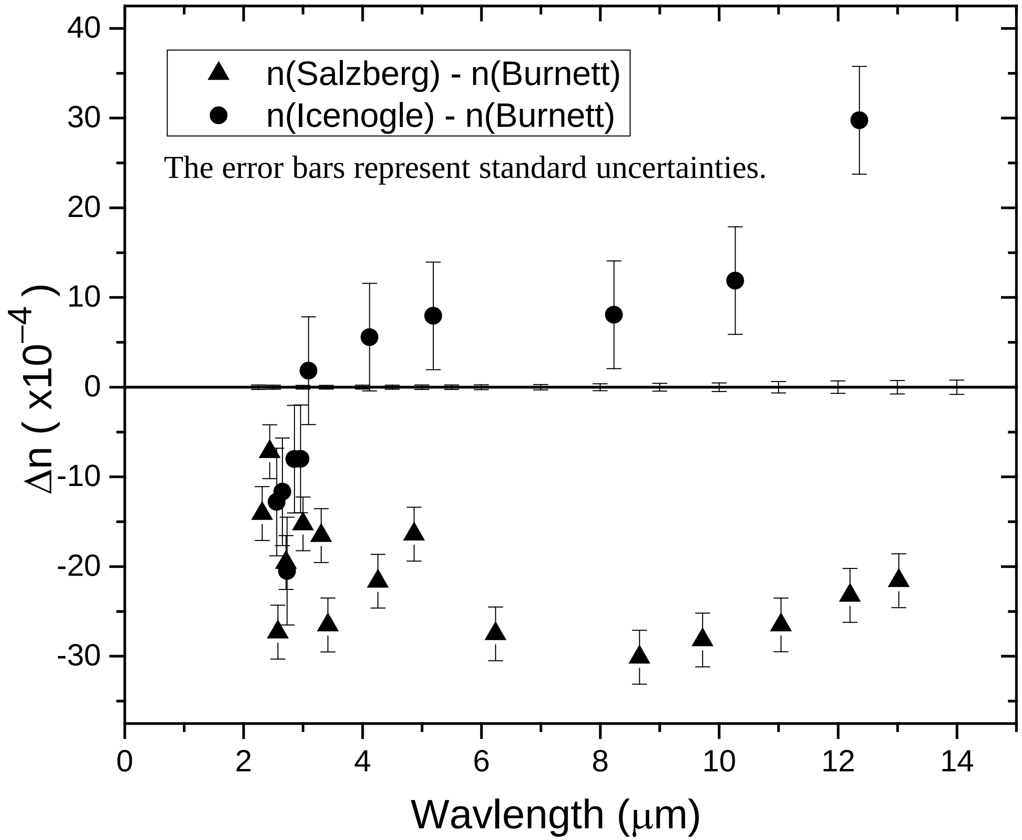
Table 1. Measured relative and absolute indices of refraction, and total standard uncertainties.

λ^{vac}(μm)[a]	λ^{air}(μm)[b]	$Index^{vac}$	$Index^{air}$	σ(x10^{-5})
2.2500	2.2494	4.084061	4.082973	2.5
2.5000	2.4993	4.066938	4.065855	2.2
3.0000	2.9992	4.045788	4.044711	2.1
3.3922	3.3913	4.035707	4.034633	2.0
4.0000	3.9989	4.025809	4.024738	2.2
4.5000	4.4988	4.020564	4.019494	2.3
5.0000	4.9987	4.016859	4.015790	2.4
5.5000	5.4985	4.014126	4.013058	2.6
6.0000	5.9984	4.012056	4.010988	2.7
7.0000	6.9981	4.009179	4.008112	3.0
8.0000	7.9979	4.007305	4.006238	3.9
9.0000	8.9976	4.006008	4.004942	4.3
10.0000	9.9973	4.005056	4.003990	4.8
11.0000	10.9971	4.004362	4.003296	6.4
12.0000	11.9968	4.003801	4.002736	6.9
13.0000	12.9965	4.003375	4.002310	7.5
14.0000	13.9963	4.002958	4.001893	8.0

- Index uncertainty exceeds targets for Ge in range $2.25 \leq \lambda < 14$ μm. But:
  1) Made measurements in range 2.0 – 2.25 μm - index and uncertainty increase rapidly in this range for $\lambda \leq 2.25$ mm. Didn't report.
  2) Prelim. meas. of other samples showed large variations in this range.

[1]Burnett, J.H., Kaplan, S.G., Stover, E., and Phenis, A., Refractive index measurements of Ge," Proceedings of SPIE 9974 (2016).

Comparison With Extensively Used Measurements

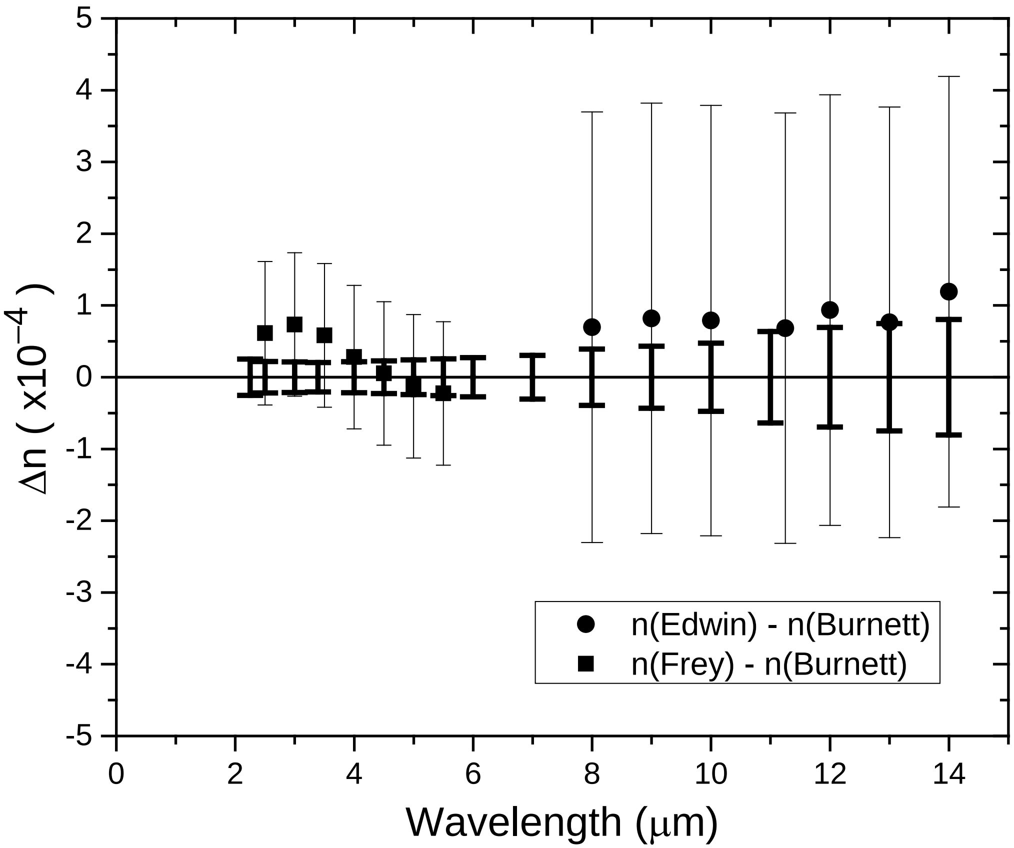


- Plot of differences between the measurements of Salzberg et al.[1] and of Icenogle et al.,[2] and values of the Sellmeier formula of this work.
  - Results used in IR Refs.: *Handbook of Optical Constants of Solids* (Palik) and *Handbook of Infrared Optical Materials* (Klocek) and in commonly used commercial optics design software.
- Values differ well outside 1-σs. Could be due to differences in material quality.

[1]Salzberg, C. D. and Villa, J. J., "Index of Refraction of Germanium," J. Opt. Soc. Am. 48, 579 (1958).

[2]Icenogle, H. W., Platt, B. C., and Wolfe, W. L., "Refractive indexes and temperature coefficients of germanium and silicon," Appl. Opt. 15(10), 2348-2351 (1976).

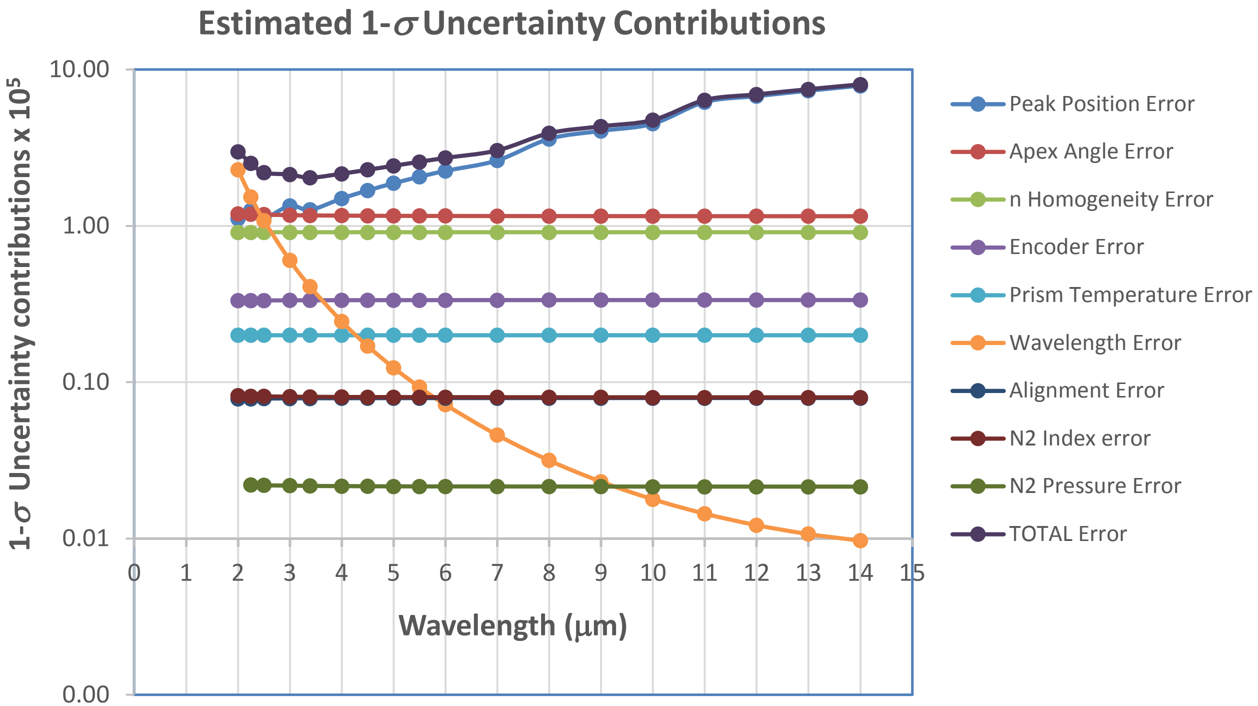
Comparison With More Recent Measurements



- Plot of. differences between the measurements of Edwin et al.[3] and Frey et al.,[4] and values of the Sellmeier formula of this work. The error bars represent standard uncertainties.

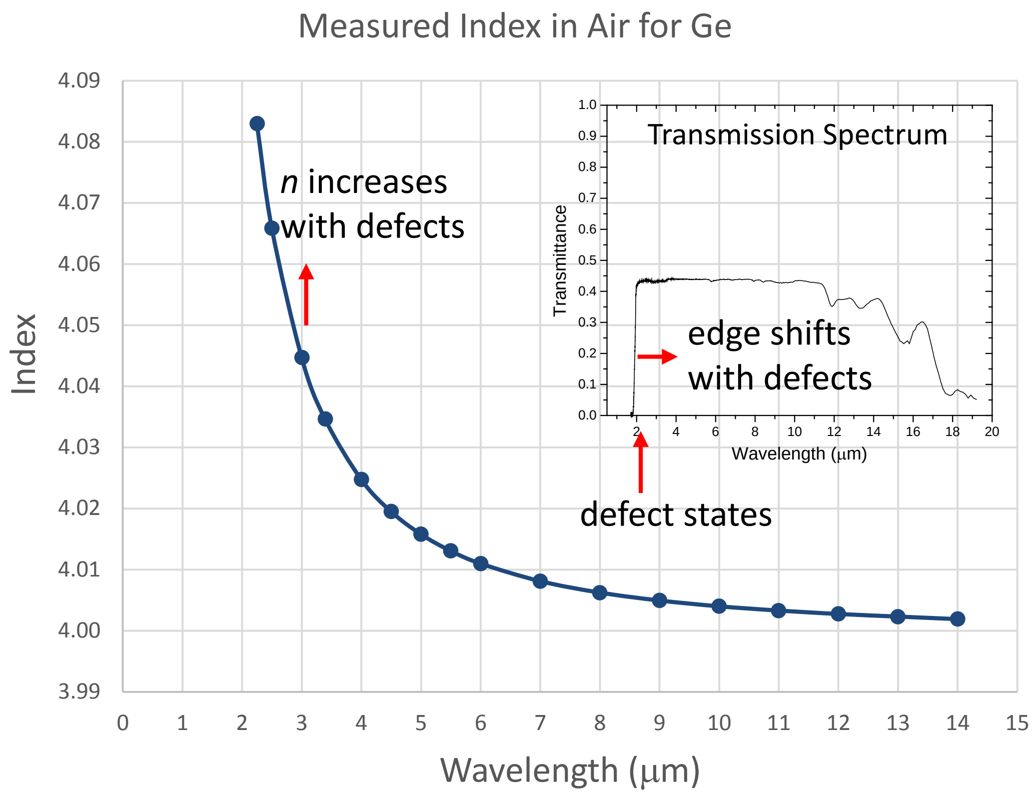
[3]Edwin, R. P., Dudermel, M. T., and Lamare, M., "Refractive index measurements of a germanium sample," Appl. Opt. 17(7), 1066-1068 (1978).
[4]Frey, B. J., Leviton, D. B., Madison, T. J., "Temperature-dependent refractive index of silicon and germanium," Proc. SPIE 6273, 62732J (2006).

Sources of Uncertainty



- Dominant source of *n* uncertainty at short λ (2.0 – 2.5 μm) due to λ uncertainty. $\Delta\lambda$ (1-σ) = 0.2 nm over full λ range. Diffraction-limited except below 2.5 μm.
- For Ge for $\lambda = 2.0$ μm, Δn (1-σ) = 3×10^{-5}.
  $\Rightarrow$ This cloaks interesting sample-to-sample variations.
- σ_λ contribution rises rapidly for shorter λ.
- This situation gets a lot worse for other materials which transmit at shorter λ, e.g., Si, ZnS, ZnSe, $BaF_{2,}$ CaF_2, etc.
- Need to improve this to satisfy goals 2) and 3).

Index Differences Between Samples

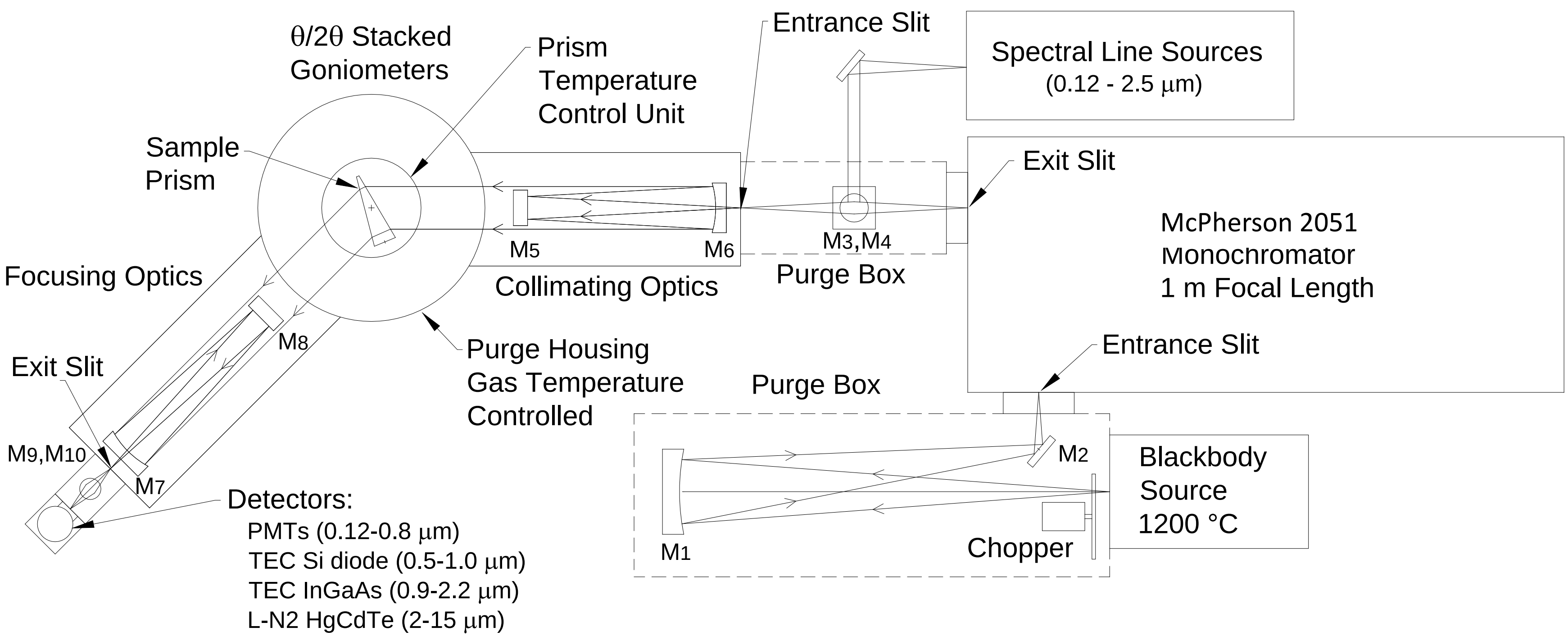


Reason care about short λ measurements: sample differences most pronounced there.

- Index "diverges" near the absorption edge $\lambda \leq 2$ µm (electronic band transitions).
- Defect states associated with band edges.
  - $\Rightarrow$ Affects absorption edge and thus index.
  - $\Rightarrow$ Index near absorption edge is most sensitive to impurities, defects, stress, etc.
- Expect sample index dispersion differences most near absorption edge 2 - 3 µm. This is where the "action is" for sample-to-sample index comparison.

IR Source λ Accuracy

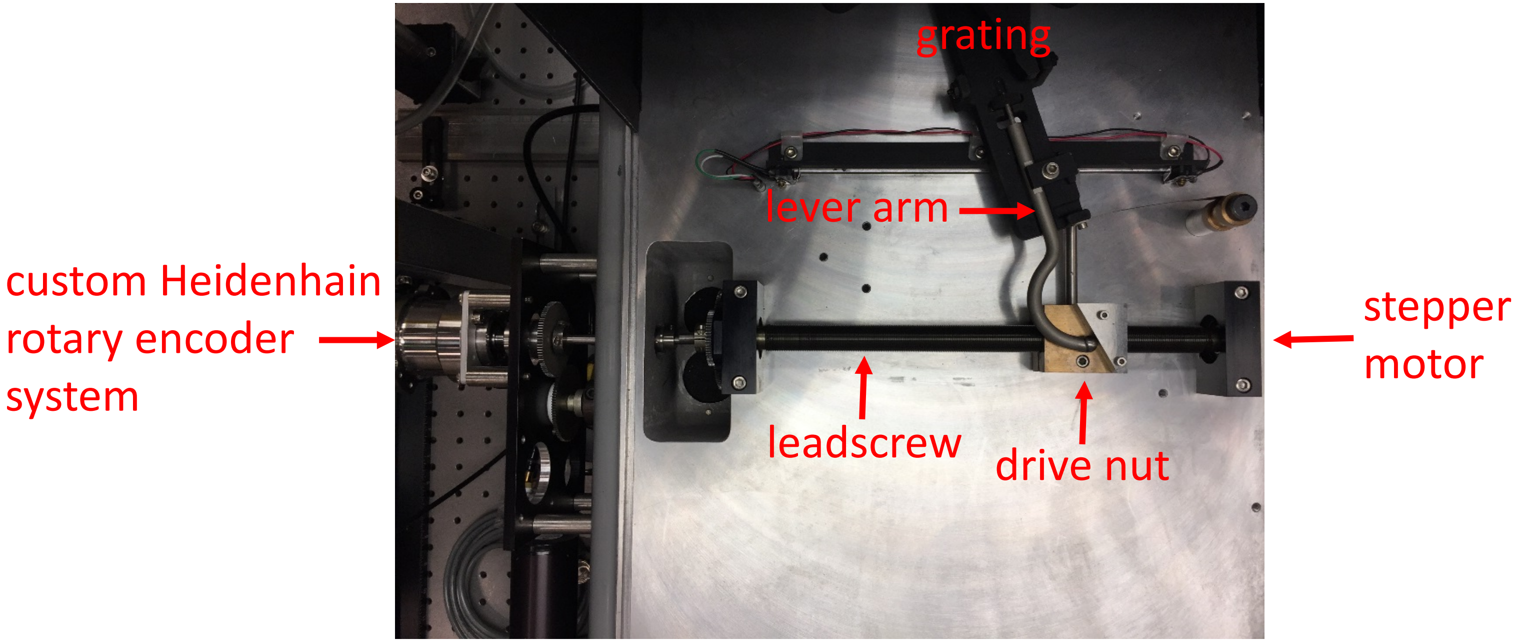
- λ source for project in range 1-14 mm: black-body source with λ-bands created by McPherson 2051 spectrometer.
- Accurate λs determined by calibration of monochromator.



Wavelength Accuracy of Spectrometer

- McPherson 2051 spectrometer: plane-grating, sine-bar drive system.
- In sine drive, λ increases linearly with translation of drive-nut.
  $\Rightarrow \lambda$ increases linearly w/ rot of lead screw, meas. by *rotary* encoder.
- λ-calibration: calibration of spectral λs w/ rotation angle.

Top View of McPherson 2051 Monochromator w/ Rotary Encoder

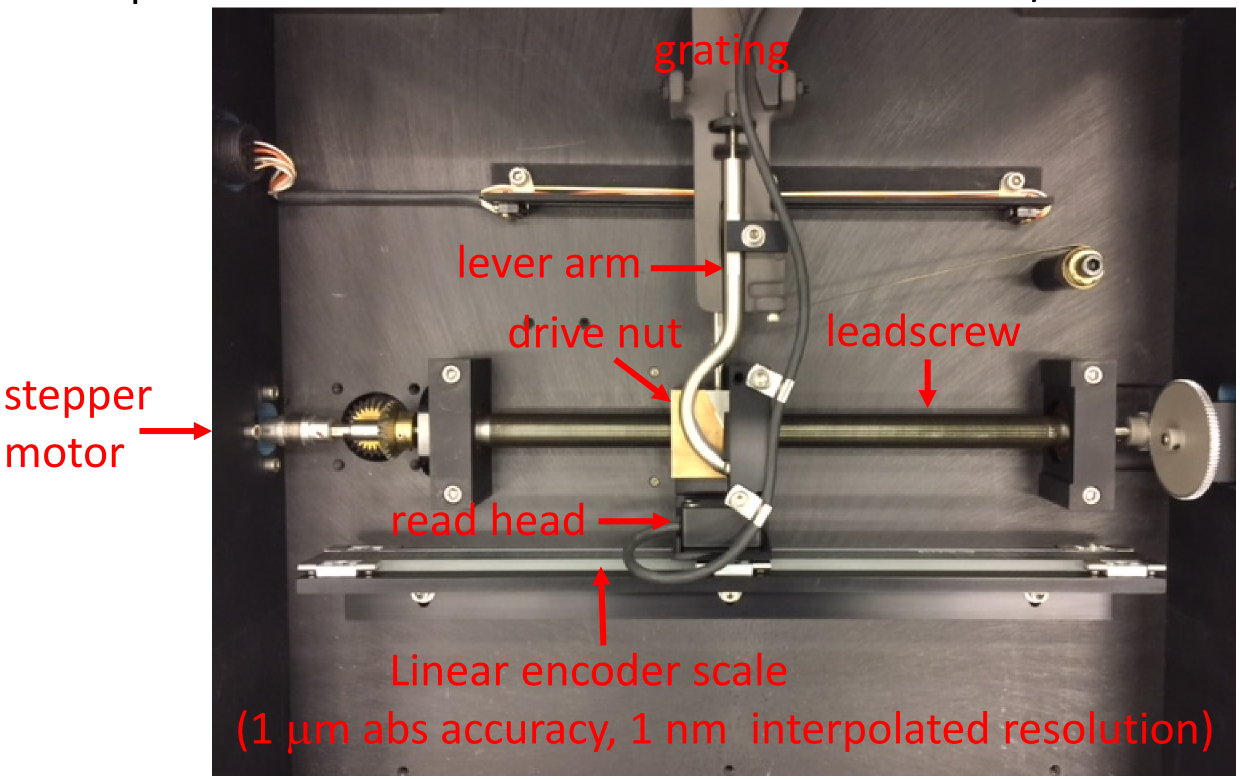


- Commercial spec.: λ reproducibility: 0.01 nm, λ accuracy: 0.05 nm.
- Practical *long-term* (~1 month): reprod.: 0.1 nm, accuracy: 0.2 nm.
  - due to mechanical separation of rotary encoder and grating
- Requires recalibration (1 wk) at least once/mo. Not sustainable!

Modifications to Improve λ-Accuracy

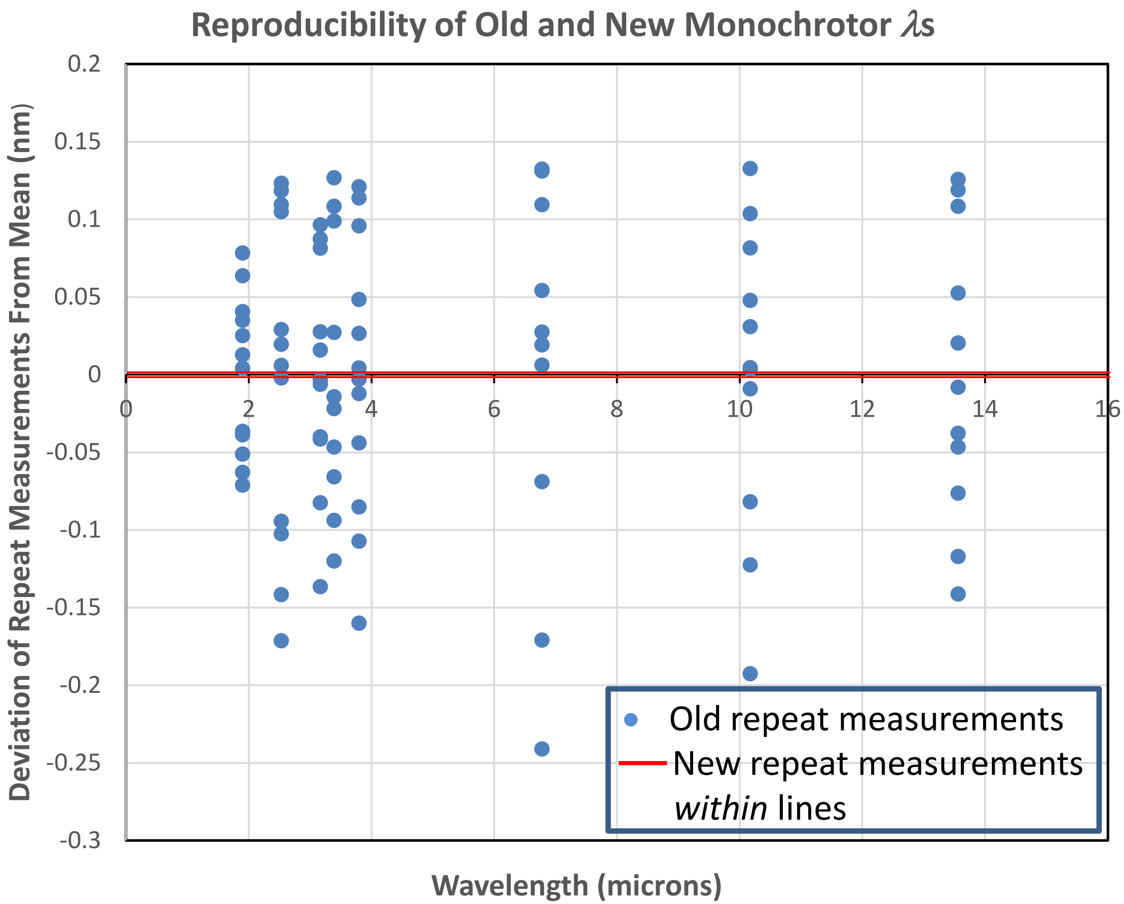
- Replace w/ McPherson 2061 spectrometer: larger *NA*, PB mirrors.
  - More directly connects encoder to grating – not gears.

Top View of McPherson 2061 Monochromator w/ Linear Encoder



- Improves *long-term reproducibility* to: 0.001 nm (100×)
  Improves *practical long-term calibrated accuracy* to: 0.015 nm (>10×)
- Stable calibration: once/year. (Need new calibration for each Temp.)
- Substantially increases sample throughput due to calibration stability!

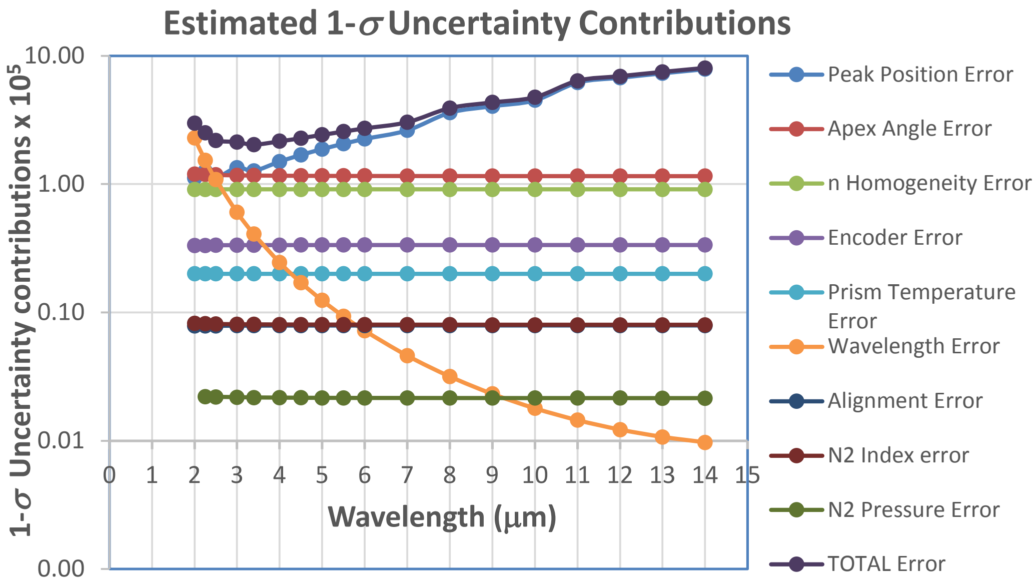
λ Measurement Reproducibility



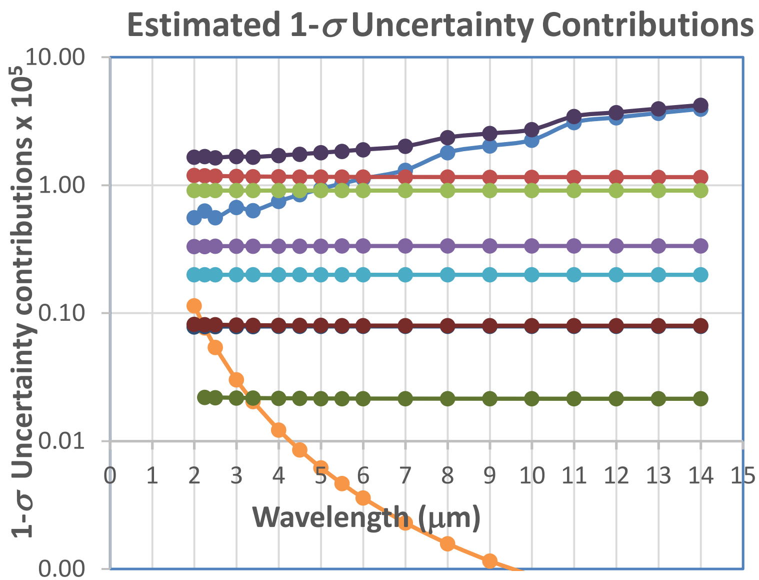
- With previous monochromator, long-term repeatability: σ = 0.1 nm
- With new monochromator, long-term repeatability: σ = 0.001 nm (centroid of scanned peak).
  - with 4th order polynomial fit, calibrated λ uncertainty: $\leq$ 0.015 nm over range 2 – 14 microns

Sources of Uncertainty

Original System

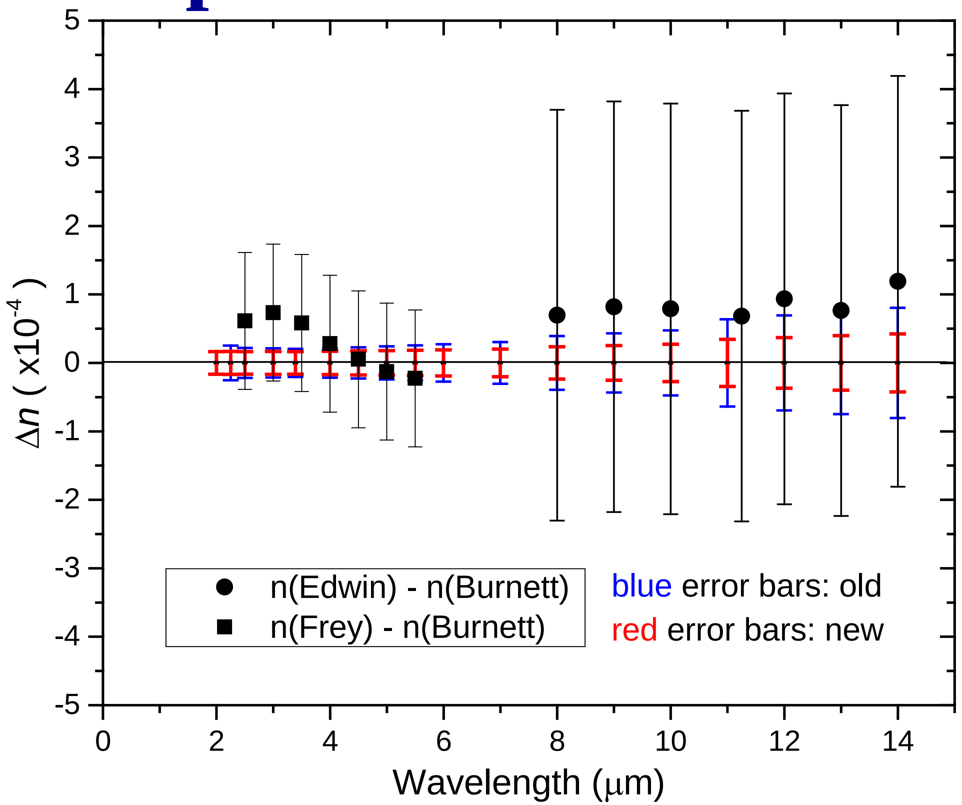


Upgraded System



- With the new monochromator system, the λ-uncertainty makes a negligible contribution to the total index uncertainty σ_{tot}.
  - λ-uncertainty now has *small* impact for other materials at shorter λ.
- At 2.0 µm, total σ for the absolute index drops nearly by half
  σ_{tot} (2.0 µm, original) = 3.0×10^{-5}
  σ_{tot} (2.0 µm, new) = 1.6×10^{-5}

Comparative Uncertainties



- With new λ calibration system, uncertainty improvement about 50%
  $\Rightarrow$ index diffraction-limited (for sample size) to below 1 μm.
  1) Stable calibration: need only once/year, rather than once per/mo.
  2) Much less measurement averaging needed.
  3) No failed measurements due to calibration shifts.
  4) $\Rightarrow$ Increases sample throughput by at least factor of 5.
  5) Enables high-accuracy measurements for visible λs.

Next Steps

- Have started repeat measurements of 6 Ge samples, with index accuracy improved overall. Improvement (×2) at shortest λs.
  - This allows establishing and analyzing index variations within uncertainties.
  - Enables maintaining high accuracy for shorter λ transmitting matls.
  - Much faster sample turn-around time due to stable calibration.
- Complete d*n*/d*T* for Ge.
  - Important data and necessary for proper uncertainty analysis.
- Completion of Ge project for dissemination (publishing) of generic Ge index dispersion (Sellmeier formulas) with range of variations, for commercial use. August 2018.
- Repeat measurement approach with other priority materials, e.g., ZnSe, ZnS, Si, CaF_2, BaF_2, etc.
- Start index measurements of ZnSe samples 0.5 – 14 μm. Need to include Si, InGaAs, and HgCdTe detectors.
  - Expect first results of ZnSe in September 2018.

Anticipated Schedule and Priorities

Priority	Material		Measurment Status
1	Germanium	15 prisms+flats (Photonic Sense)	6 underway, 1 completed, d*n*/d*T* – Complete in 8/18
2	ZnSe	6 prisms+flats (II-VI)	Have characterized samples, start 8/18 - Complete by 12/1/18
3	BaF2	6 prisms - single crystal (Hellma), 2 prisms, 6 flats - polycrystalline (ISP)	Start in 12/18
4	CaF2	6 prisms – single crystal	
5	ZnS		
6	Silicon		
7	GASIR 1		
8	IRG26		
9	BD2		