Welcome

- ASC OP/TF6 (OEOSC)
  - Write the standards related to infrared materials
  - Coordinate with other task forces in developing and supporting standards
- IRMSWG (SPIE)
  - Advise TF6 regarding instrumentation and measurement methods
  - Meetings will consist of status reports, technical evaluations, recommendation
  - Work the details of the measurements
  - Test plans
  - Instrument development /upgrades
  - Sampling protocols
  - Sample specs and procurement
  - Identify funding sources
- All documents from this meeting will be posted to:
  - http://www.optstd.org/op1%202015meetings.htm

ANSI ASC OP – Task Force 6: IR Materials Standards and SPIE IR Materials Standards Working Group

Wednesday, August 12, 2015
08:00 – 10:00 PDT

SPIE Optics & Photonics

Marriott Marquis San Diego Marina
333 West Harbor Dr.
San Diego, CA 92101
Carlsbad Room

Draft Agenda

1	Welcome, Introductions, and appointment of note taker	A. Phenis
2	Adoption of agenda	A. Phenis
3	Approval of previous meeting minutes	A. Phenis
4	Reports	
5	Refractometer measurements update (NIST)	J. Burnett
5a	Pilot study sample status update	A. Phenis
5b	Submitted projects update (OEOSC)	A. Krisiloff
5c	Other reports and updates	Project Leaders
5d	Group Discussion Topics	A. Phenis
6	Time and Place for next meeting - suggestions	A. Phenis
7	Adjourn	A. Phenis

Approval of previous meeting minutes

- Previous meeting documents and minutes can be found at

  http://www.optstd.org/op1%202015meetings.htm

Reports

Refractometer measurement update

J. Burnett – NIST

NIST IR Materials Pilot Study - Update

John Burnett, Leonard Hanssen, and Simon Kaplan
National Institute of Standards and Technology
Physical Measurement Laboratory
john.burnett@nist.gov

Outline

1. NIST Commitment to IR Materials Index Pilot Study
2. NIST Refractometry System
3. Materials
4. Ge Measurements
5. Longer Term Measurement Plans

NIST Commitment to IR Materials Index Pilot Study

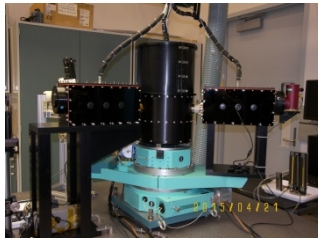
In response to request IRMSWG, NIST (with director's blessing) committed in 2013 to participate in IR Materials Index Pilot Study with a direction of substantial resources to make most accurate index measurements of key IR materials.

- Upgraded sub-ppm-accuracy UV-vis Min-Dev refractometry facility for λ to 14 μm.
- Goal - Maintain UV-vis capability while achieving highest possible IR accuracy for various IR materials with different transmission ranges and other optical properties.
- Completed and tested key components in Jan 2015.
- Worked with Gary Wiese (Lockheed) to develop optimal specs. for key materials:

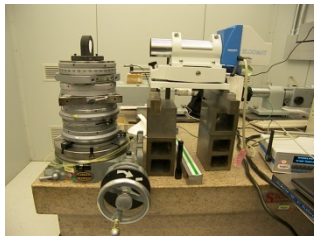
  Ge (single X'tal), ZnSe, ZnS, Si (single X'tal), CaF_2, BaF_2, IRG26 (As_2Se_3) (glass), GASIR1 (Umicore glass), and others. Developed priority list - Ge at top.
- Started receiving materials (multiple samples from different boule locations). Developed specific measurement strategy for first materials Ge.
- Jan. – June, NIST completed other commitments. Started on Ge in July.

Index Measurement Issues

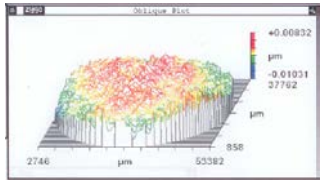
High-Accuracy Index Measurements: Determine index from minimum-deviation-angle. Actually, requires numerous measurements w/ numerous sources of error.



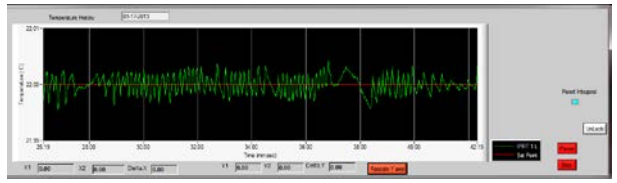
Dev Angle
Goniometer w/ calibrated encoder
Alignment issues
($\Delta\theta \leq 0.2$ arc-sec)



Prism Apex Angle
Auto collimator + encoder
($\Delta\theta \leq 0.2$ arc-sec)

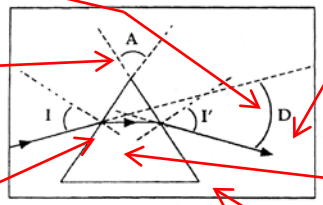


Prism Surface Flatness
Zygo Interferometer
(Wavefront RMS $\lambda/40$)



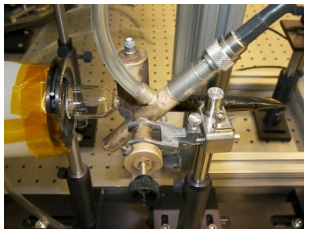
Temp Control – dn/dT
TWP and MPGa calibration
($\Delta T \leq 5$ milli K)

At Minimum Deviation Angle

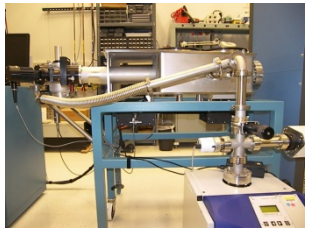


$$n(\lambda) = \frac{\sin\left(\frac{A + D(\lambda)}{2}\right)}{\sin\left(\frac{A}{2}\right)} \cdot n_{gas}(\lambda)$$

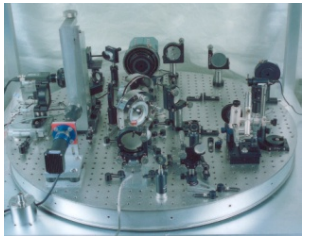
λ Calibrations
Spectral calibration lamps
($\Delta\lambda \leq$ 0.01-0.1 nm)



Material Absorb.
Transmission spectrometer
($A_{10} \leq 0.01$/cm)

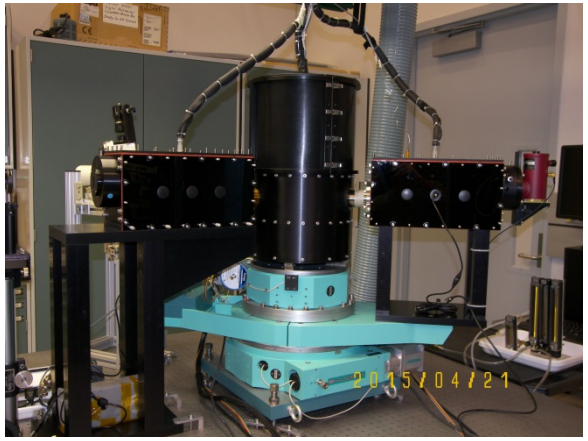


Index Homogeneity
n variation on ingot $\Rightarrow$
n variation on sample
Vis/UV interferometer
($\Delta n \leq 1 \times 10^{-7}$)

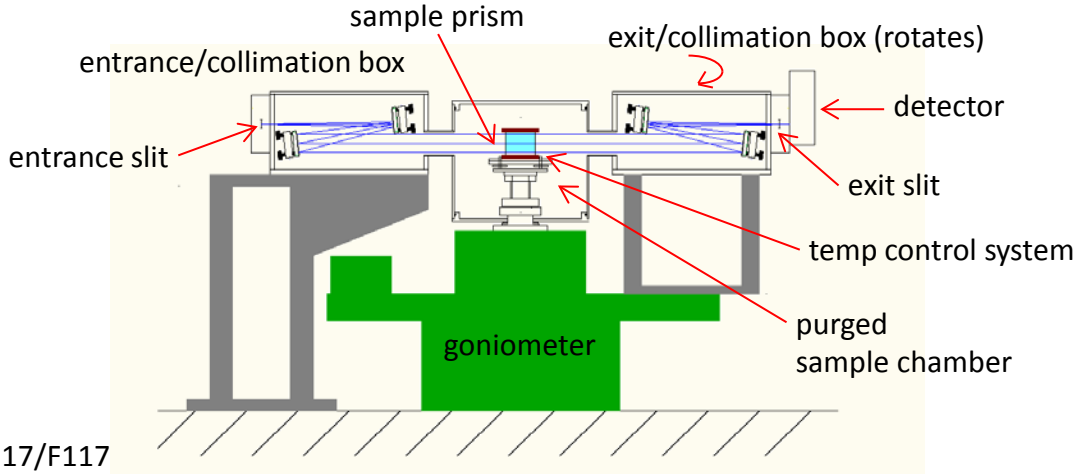


Stress Birefringence
grown-in or external
(stress-optic coeff. - π_{ijkl})
Polarimeter
(1 nm/cm [$\Delta n \leq 1 \times 10^{-7}$])

NIST VUV/IR (0.12 – 15 μm) Refractometry System



NIST Refractometry Facility located at NIST AML 217/F117



- All reflective optics + purge housing + sources/detectors ⇒ operates VUV (120 nm) through 14 μm.
- Temperature range - operates normally near room T (15 - 25 °C).
- Specs.:
  - FL=0.5 m, F/# = 10.
  - Goniometer - calibrated angle encoder scale: absolute angle uncertainty ≤ 0.2 arc-sec.
  - Feedback temperature control of sample to ≤ 5 mK, purge gas to to ≤ 20 mK.

  ⇒ Absolute index uncertainty depends on λ and sample properties, e.g., size, apex angle, surface figure.

  Typically: VUV/vis (0.12 – 0.8 μm): 1-σ ~ 5×10^{-7} - 5×10^{-6} + dn/dT in range T (15 - 25 °C).

  Near-IR (0.8 – 5.0 μm): 1-σ ~ 5×10^{-6} - 4×10^{-5}

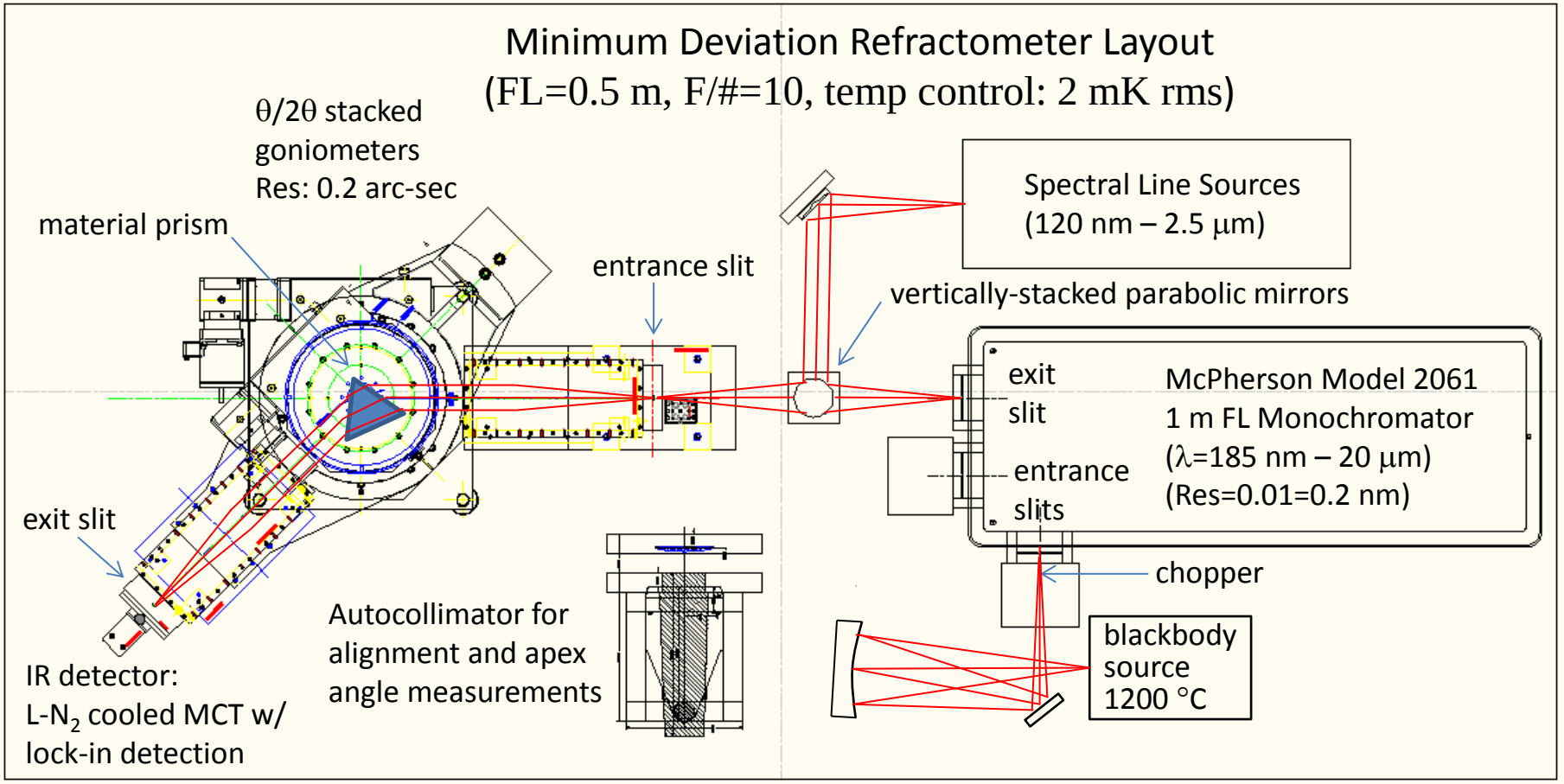
  Mid-IR (5.0 – 14 μm): 1-σ ~ 4×10^{-5} - 8×10^{-5}
- Delivering high-accuracy index results to industries (Litho) for lens design and sample variations.
- NIST focus has been highest-accuracy NOT throughput. Laborious process, ~1 week/sample.
  - Developing index database for values for important optical materials.

IR Refractometry System

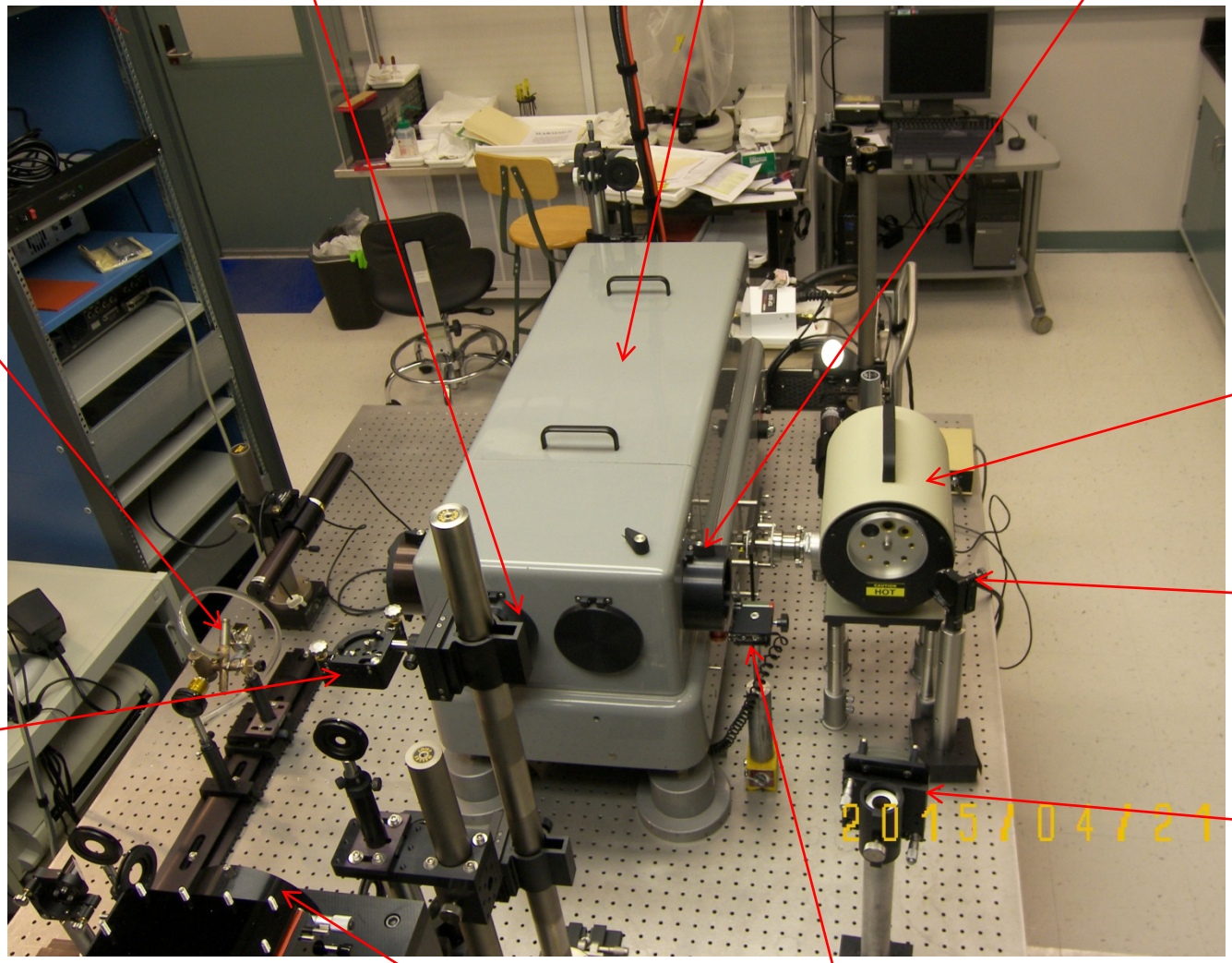
λ range to IR – λ=0.12-14 μm (T=15-25 °C)

(Consistent w/ ISO/TC 172/SC 3 N223 Test Method for IR Index)

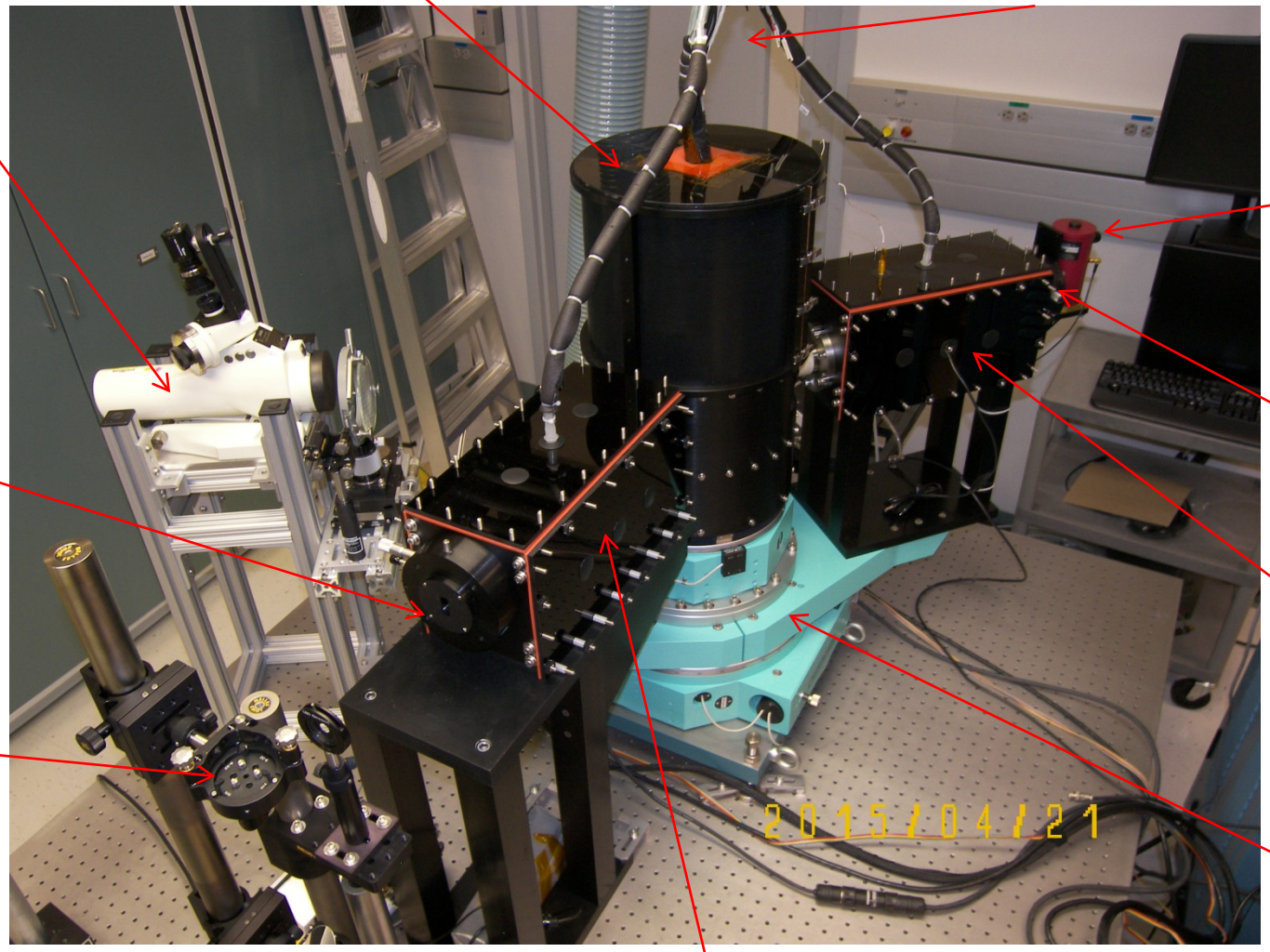
- All reflective optics from source to detector.
- Blackbody source (1200 °C), 1 m FL monochromator - Resolution at λ =5 μm ~ 0.1 nm.
- IR detector (Liquid N_2 cooled MCT), lock-in detection.
- Index accuracy achieves theoretical limit for material, sample geometry, and sample specs.)



NIST Refractometry System – Source Side

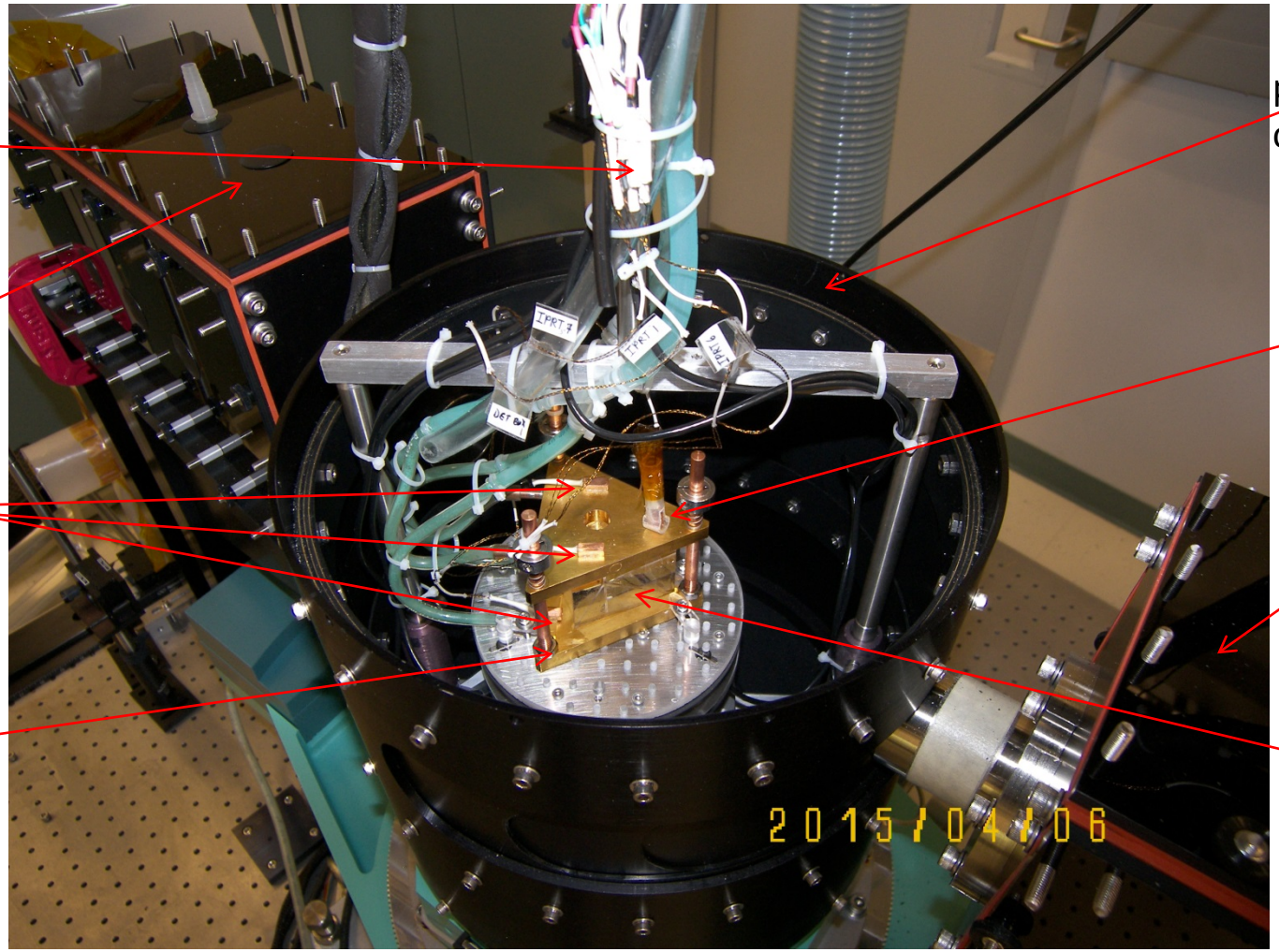


NIST Refractometry System – Refractometer Side



NIST Refractometry System – Sample Chamber

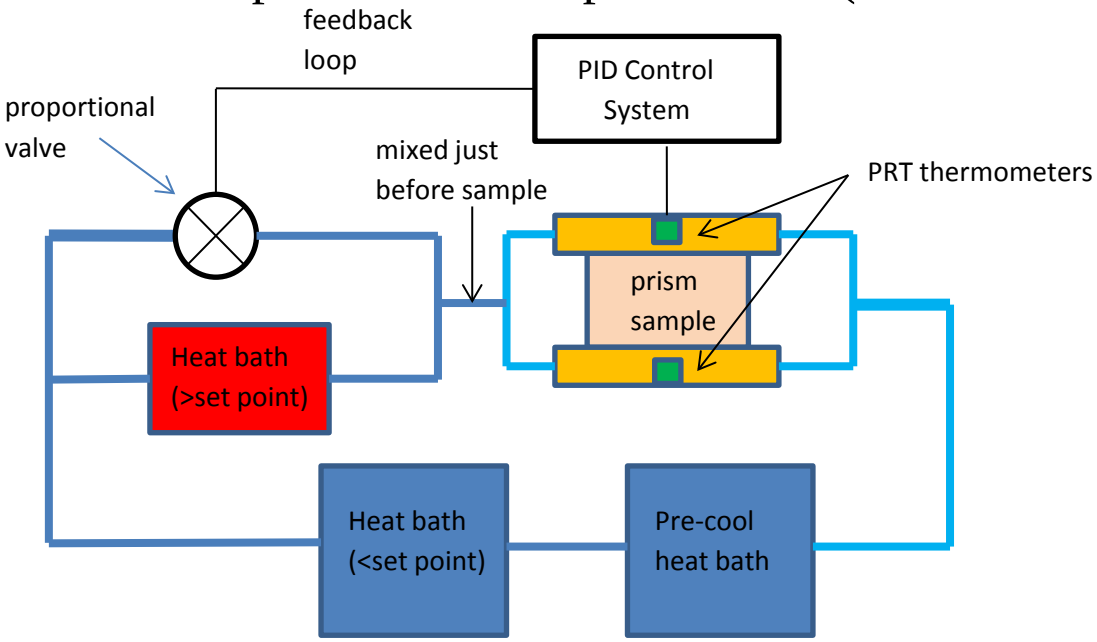
- Sample alignment: (tilt, min. dev.) determined and monitored before, during, and after meas. by laser alignment system.
- *T* control: PID feed-back loops. ΔT: sample < 5 mK; ΔT gas: < 20 mK.

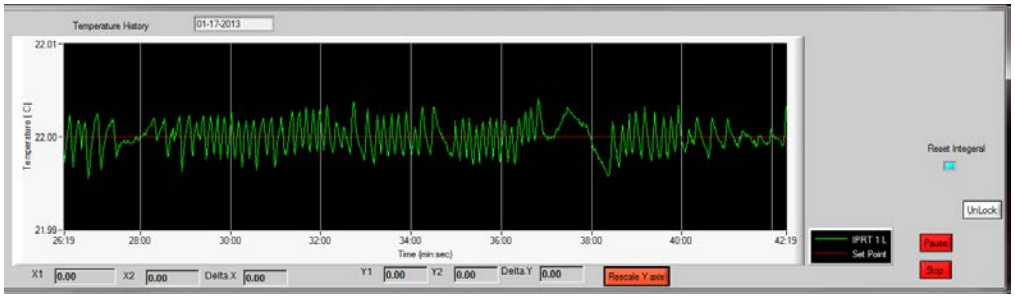


Temperature Control

Sample- and gas-temperature control by PID feedback control loop.

- PRT thermometers calibrated at triple-point water and melting-point of Ga.
- PID control loop using mixing of hot and cold baths.
- Controls absolute temperature of sample < 5 mK (<2 mK RMS).





Calculation of Transmittance Through Air (L=1 m)

HITRAN on the Web

Home | HITRAN survey | Molecules | **Gas mixture spectra** | Cross-Sections | Auxiliary data | Saved spectra | My spectra | References | Information | My profile

Gas mixture spectra: *Parameters for spectrum simulation*

John Burnett [johnhburnett] | Log out

Input selection						Options
Gas mixture: USA model, mean latitude, summer, H=0		Simulation type: Transmittance spectrum				☑ Separate molecules
General parameters:						**Plot scale**
WN_{min}, cm^{-1}: 500	WN_{max}, cm^{-1}: 10000	T, K: 296	P, atm: 1	I_{cut}, cm/mol: 1E-28		◉ Natural ○ Logarithmic
Contour parameters:						
Shape: Voigt	WN_{step}, cm^{-1}: 0.1		Wing, HW: 50			Simulate spectrum
Function parameters:						
Opt.path, m: 1	App.Function (AF): Dirac		App.Resolution, cm^{-1}: 0.1		AF wing, AR: 50	

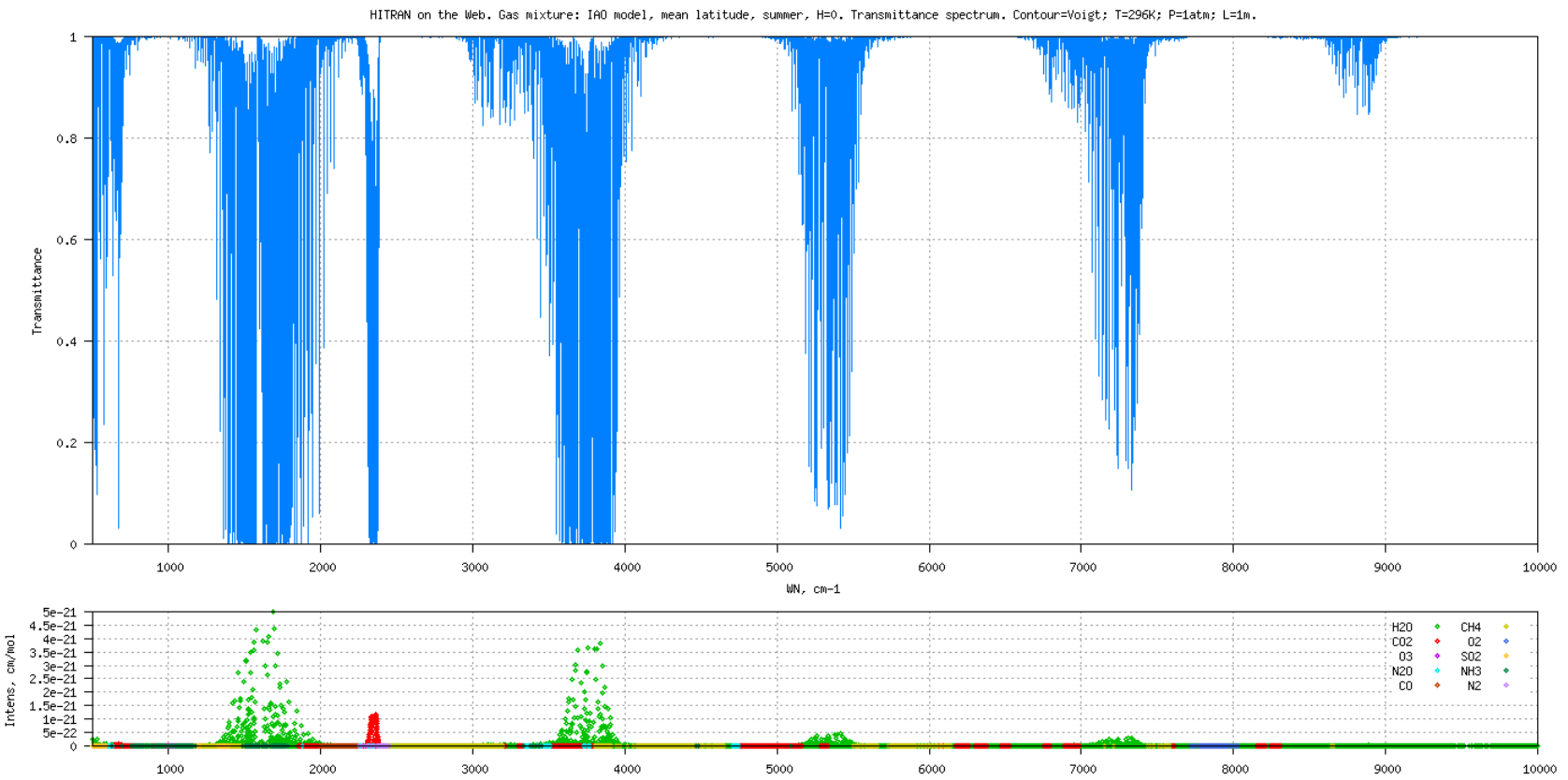
Usage Statistics

All rights reserved © : Harvard-Smithsonian Center for Astrophysics (CFA), Cambridge, MA, USA; V.E. Zuev Institute of Atmospheric Optics (IAO), Tomsk, Russia

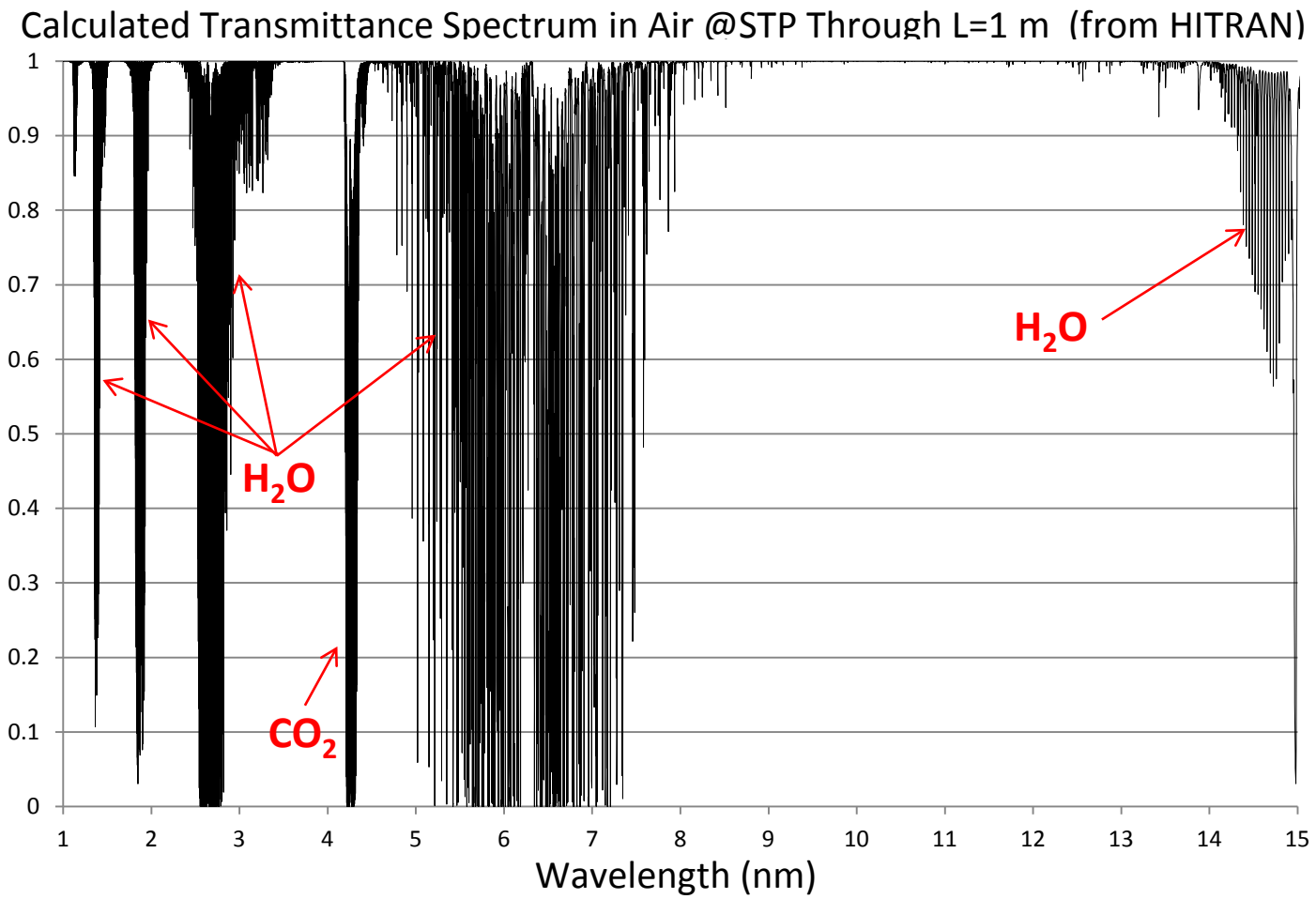
Powered by: ZF ZENO FRAMEWORK

The system development supported by RFBR (grant 09-05-92508-MK_a) and CRDF (grant RUG1-2954-TO-09)

Synthetic spectrum for gas mixture 'IAO model, mean latitude, summer, H=0'. Graphical representation.



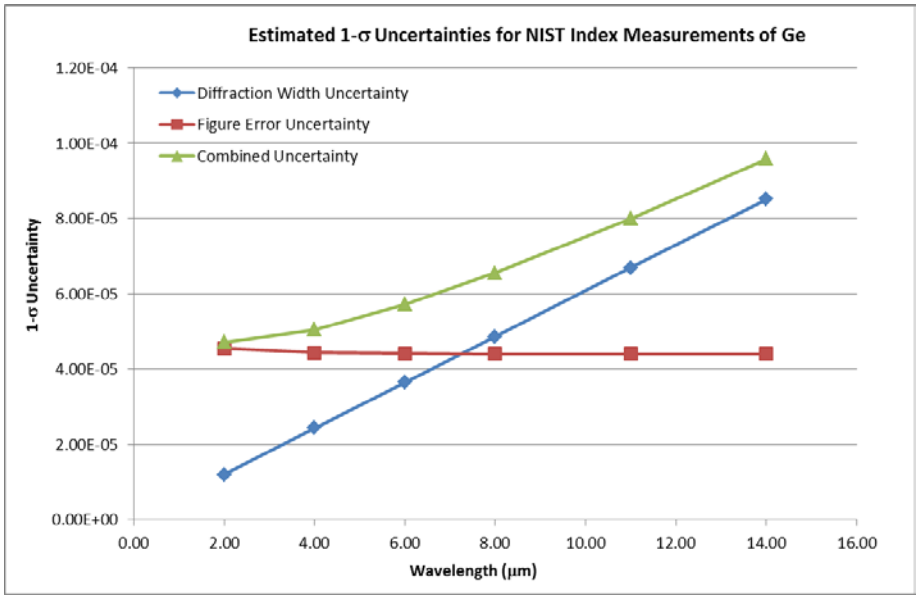
Calculation of Transmittance Through Air (L=1 m)
(same data as above in microns)



- Opaque in substantial regions, w/ trans. windows in regions 3-5 and 8-14 microns.
- Due primarily to vibration/rotation bands of H_20 and to CO_2.
- To obtain accurate index in and near absorption regions, purge with N_2 gas.

General Measurement Strategy

- Determine theoretical uncertainty limit for each sample over transparent range.
  - Depends on sample size, surface figure specs. etc. For example for Ge:



  - As λ gets longer:
    Uncertainty due to diffraction width increases.
    Uncertainty due to figure error decreases.

- Design measurement to ensure uncertainty achieves theoretical limits through range.
  - Requires several gratings w/ different Gr/mm and blazing for signal and resolution.
  - Calibrations of source (black-body source through monochromator).

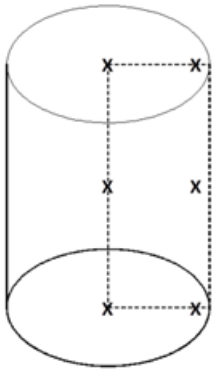
IR Samples – Tentative Schedule

IR Samples Recived by NIST - Tentative NIST Schedule				
Material (Orig Priority)	Supplier	Status	Projected Start Date	Projected Completion
Ge (1) (single crystal)	Photonic Sense GmbH	Received 15 prisms Measurements (6) underway	Measurements underway	2nd week in Oct. '15
ZnSe (2)	II-VI	Received 6 prisms, 6 flats	1st week in Nov. '15	End of Nov. '15
BaF_2 (6) (single crystal)	Hellma Materials GmbH	Received 6 prisms	December '15	
CaF_2 (5) (single crystal)	Hellma Materials GmbH	Received 6 prisms		
BaF_2 (6) (polycrystaline)	ISP Optics	Received 2 prisms, 6 flats		

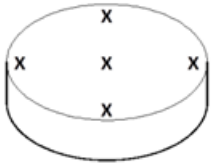
Other IR Materials: ZnS, Si (single X'tal), GASIR1 (Umicore glass), IRG26 (As_2Se_3) (glass), BD2 (glass), GaAs (single X'tal), Spinel, AMTIR5 (As-Se), IR Fused Silica, LiF, MgF_2.

Ge Measurements – Samples

- Goal: for each material, measure samples from various locations in the ingot to assess growth-position dependence.
- IR Sampling protocol of September 2014 – For Ge:



Cylindrical Ingot

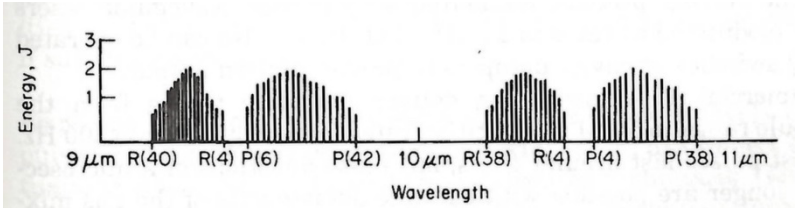


Circular Plate

- For Ge, 15 samples, 5 per layer.
- Due to priorities for now, for Ge, 6 samples is reasonable.
  Will measure the center sample and 1 edge sample from each layer.
- Have started with center layer, center sample.
  The first sample takes longest because finding parameters and optimizing trade-off, such as resolution and S/N at each λ.
- After this, each sample takes about 1 week.

Ge Measurements

- Goal: develop best measurement strategy to get most accurate Sellmeier formula through transmitting range: For Ge: low absorbance in range 2-14 μm.
  - For a given time, trade off between No. λ intervals and No. repeat measurements.
  - Source: 1200 °C black body source.
  - Calibration: Ne lines in (2-3 μm); CO_2 laser vib/rot lines (9 – 11 μm).



CO_2 laser vib/rot lines

- Decided on several measurement ranges:
  - 2.0 – 6.0 μm: index every ~ 0.5 μm (9 λs) 150 gr/mm grating w/ 2.0 μm blaze
  - 7.0 – 14.0 μm: index every ~ 1.0 μm (8 λs) 100 gr/mm grating w/ 9.0 μm blaze
  - Achieves resolution limit with reasonable measurement time: 1 week per sample.
- Made preliminary measurements on Ge (middle-center). Determine optimal measurement parameters to achieve index accuracy over λ range 2-14 μm.
  - Need calibration and cross checks so will not report values yet, but consistent.
  - First measurement hardest and most critical. The following measurements faster.
- Expect to complete Ge and distribute data in early October

Longer-Term Measurement Plans

- As measurements are completed, the turn around will become somewhat faster – but probably not by much!
  - NIST facilities set up for high-absolute-accuracy, NOT high-throughput.
  - Expect no faster than 1 sample/week $\Rightarrow$ 1 material/2 months.
    $\Rightarrow$ Multi-year project.
  - May not be reasonable to continue to measure 6 samples per material.
  - We expect that at least for some materials (especially high-quality single crystal, e.g, Ge, Si, BaF_2, CaF_2, NIST will establish generic values.
- Other methods (e.g., prism coupling techniques), faster/commercial apparatus.
  - Not as accurate as Min-Dev method, but good at relative measurements.
  - Hoped that good correlations will be found between *sample differences* found at NIST by the Min-Dev method and by commercial methods.
  - Ideally NIST could measure just one or a few samples for a new material, and sample/batch variations determined by faster commercial methods.
  - NIST intends to publish data from the important materials.
  - Set up NIST materials index database (UV-vis-IR), w/ dn/dTs near 20 °C.
- Longer term: increase T range – cryogenic $\rightarrow$ >> RT.

Acknowledgements

NIST thanks Gary Wiese (Lockheed) and Adam Phenis (Cymer) for their help in setting up the project, developing the material priority lists, helping develop optimal sample specifications, and assembling and encouraging materials suppliers.

Pilot study sample status update

A. Phenis – Cymer, an ASML Company

Study Status

- Testing has been prioritized by the results from a survey done by Gary Wiese (April 2013)
  - The testing order/priority can change based on what is available at NIST.
  - NIST will start at the highest priority and move down the list

Pilot Study Material Status

Measurement Priority	Material	Supplier	Status
1	Germanium	Photonic Sense GmbH	Received, Measurements underway
2	ZnSe	II-VI	Received
3	Multispectral ZnS (Cleartran)	DOW	NA
4	Silicon	Novotech	NA
5	CaF2	Hellma Materials GmbH	Expected delivery May 2015
6	BaF2 (Single Crystal)	Hellma Materials GmbH	Expected delivery May 2015
6	BaF2 (Polycrystalline)	ISP	Samples delivered
7	IRG26 (As2Se3)	Schott	Delayed due to factory fire, Expected deliver TBD
8	GASIR1	Umicore	Expected delivery June 2015

The sample list has shrunk

New Proposed Material Measurement Order

Measurement Priority	Material	Supplier
1	Germanium	Photonic Sense GmbH
2	ZnSe	II-VI
5	CaF2	Hellma Materials GmbH
3	Multispectral ZnS (Cleartran)	DOW
4	Silicon	Novotech
6	BaF2	Hellma Materials GmbH (Single Crystal) or ISP (Polycrystalline)
7	IRG26 (As2Se3)	Schott
8	GASIR1	Umicore
6	BaF2	Hellma Materials GmbH (Single Crystal) or ISP (Polycrystalline)

Submitted projects update

A. Krisiloff – OEOSC and Triptar Lens Company, Inc.

Committee Leaders

- Allen Krisiloff – Secretary, ASC OP and Executive Director, OEOSC
- Adam Phenis – Leader of ASC OP Task Force 6, Infrared Materials
- Dave Aikens – Leader of American TAG Subcommittee SC1, Fundamental Optics
- Leonard Hanssen – Leader of American TAG Subcommittee SC3, Optical Materials

Snapshot of IR Related Standards Under Development

	Domestic (ANSI)	International (ISO)
Definitions and Data		
Spectral Bands (and Abbe Number)	X	
Sampling Protocol	X	
Library of Standard Material Properties	X	
Specification of Quality		
Refractive Index	(x)	X
Homogeneity	(x)	X
Striae	(x)	X
Bubbles and Inclusions	(x)	X
Measurement Methods		
Bubbles/Inclusions		X
Straie		X
Homogeneity		X
Refractive Index	X	
dn/dT	X	
Absorption/Transmission		X

Spectral Bands – OP1.007

- Draft standard OP1.007 was rejected at the OP level
- Several issues came to light
  - Generalized definition for infrared Abbe number (2 degrees of freedom)
  - Odd boundaries for named spectral bands (gaps)
  - Abbe reference wavelengths unrelated to common sources
- Draft is under revision
  - Copied the generalized definition of infrared Abbe number from visible (3 degrees of freedom)
  - Modified named spectral bands to cover all gaps (appeal to many technical communities that utilize the IR)
  - Abbe reference wavelengths identified with laser lines
- Ballot will be resubmitted in September

Sampling protocol– OP1.008

- Draft standard OP1.008 approved by TF6
  - No comments received
- Unrevised draft will be submitted to full OP in September

Library of Standard Properties

- Schema developed by ORA in coordination with Zemax and Lambda
- Waiting for sample data from the pilot campaign to try it out
- Can publish the schema as American National Standard once the schema is confirmed
- May conflict with ISO standards

Specification of Quality

- Refractive Index
- Homogeneity
- Striae
- Bubbles and Inclusions

- Grades defined for visible materials may be useful for IR materials

Methods of Measurement

- Bubbles/Inclusions (ISO - bright field inspection)
- Striae (ISO - shadowgram)
- Homogeneity (ISO - interferometric)
- Refractive Index (ANSI - to be based on ISO 17328)
- dn/dT (part of Refractive Index)
- Absorption/Transmission (ISO - plan to revise ISO 15368)

Other reports and updates

Project Leaders

Group Discussion Topics

A. Phenis – Cymer, an ASML Company

Time and place for next meeting - suggestions

1. Teleconference/Webinar
2. SPIE Photonics West, San Francisco, CA
   - February 13th – 18th, 2016
3. SPIE DSS, Baltimore, MD
   - April 18th – 22nd, 2016

Adjourn

Thank you for coming to this meeting and we look forward to seeing you in future meetings.

All meeting documents will be posted to:
http://www.optstd.org/op1%202015meetings.htm