Standards Development Tasks

Task	Responsibility	
	OEOSC	IRMSWG
Materials standards	X	
Supporting standards		
Refractive index measurement	X	
dn/dT	X	
Attenuation coefficient	X	
Inhomogeneity	X	
Inclusions	X	
Curve fitting	X	
Sampling protocols		X
Sample specifications		X
Inclusion measurement instrument specification		X
Refractometer upgrades		X
Planning, procurement		X
Measurement statements of work		X

IRMSWG Organization and Meetings

- Work Breakdown
  - ASC OP/TF6 (OEOSC)
    - Write the materials standards standards
    - Coordinate with other TF developing supporting standards

  - IRMSWG (SPIE)
    - Meetings will consist of status reports, technical evaluations, recommendation
    - Advise TF6 regarding instrumentation and measurement methods
    - Work the details of the measurements
      - Test plans
      - Instrument development /upgrades
      - Sampling protocols
      - Sample specs and procurement
    - Identify funding sources

- Meetings
  - Concurrent with TF6?
  - Photonics West (Jan/Feb)?
  - DSS (April)?
  - Optics and Photonics (August)?

IRMSWG Sub-committees

Sub-committee	Lead
Test plans/SOWs	
Instruments	
Sampling Protocols	
Sample Specs/Procurement	
Funding	

Subcommittee Responsibilities

Determine sampling protocol – sampling frequency, configuration
Define sample tracking methods
Identify instrument development/upgrades required
Define specifications for instrument development/upgrades
Select sample fabricators, measurement labs
Write SOWs

IR Materials of Primary Interest

Material	Material Class	Transparency Waveband (nm)	Operational Temperature (^oC)
Aluminum Oxi-nitride (ALON)	Ceramic (polycrystalline)	250-5400	-40 – 100+
Barium Fluoride	Crystalline	190-12000	-40 – 100+
Calcium Fluoride	Crystalline	190-10000	-40 – 100+
Gallium Arsenide	Crystalline	1500-20000	-40 – 100+
Germanium	Crystalline	2000-17000	-40 – 70
Silicon	Crystalline	1500-8500	-40 – 100+
Magnesium Aluminate (Spinel)	Ceramic (polycrystalline)	450-6300	-40 – 100+
Zinc Selenide	CVD (polycrystalline)	550-20000	-40 – 100+
Zinc Sulfide	CVD (polycrystalline)	500-16000	-40 – 100+

Potential IR Materials of Interest

Material	Material Class	Transparency Waveband (nm)	Upper Operation Temperature (^oC)
Lithium Fluoride	Crystalline	140-9000	-40 – 100+
Magnesium Fluoride	Crystalline	140-7500	-40 – 100+
Magnesium Oxide	Crystalline	250-8500	-40 – 100+

Suggested Precision Levels

Parameter	Precision Level
Refractive Index	±50 ppm
Abbe Number	± 0.2%
dn/dT	± 5%
Internal Absorption Coefficient	± 5%
Homogeneity	± 5 ppm
Minimum inclusion size	20 μm

Potential NIST Roles

- **Fund the measurements**
- **Host a consortium to fund the measurements**
- **Define traceability criteria**
- **Participate in the development of standards**
- **Perform measurements**

Round Robin (1 of 10)

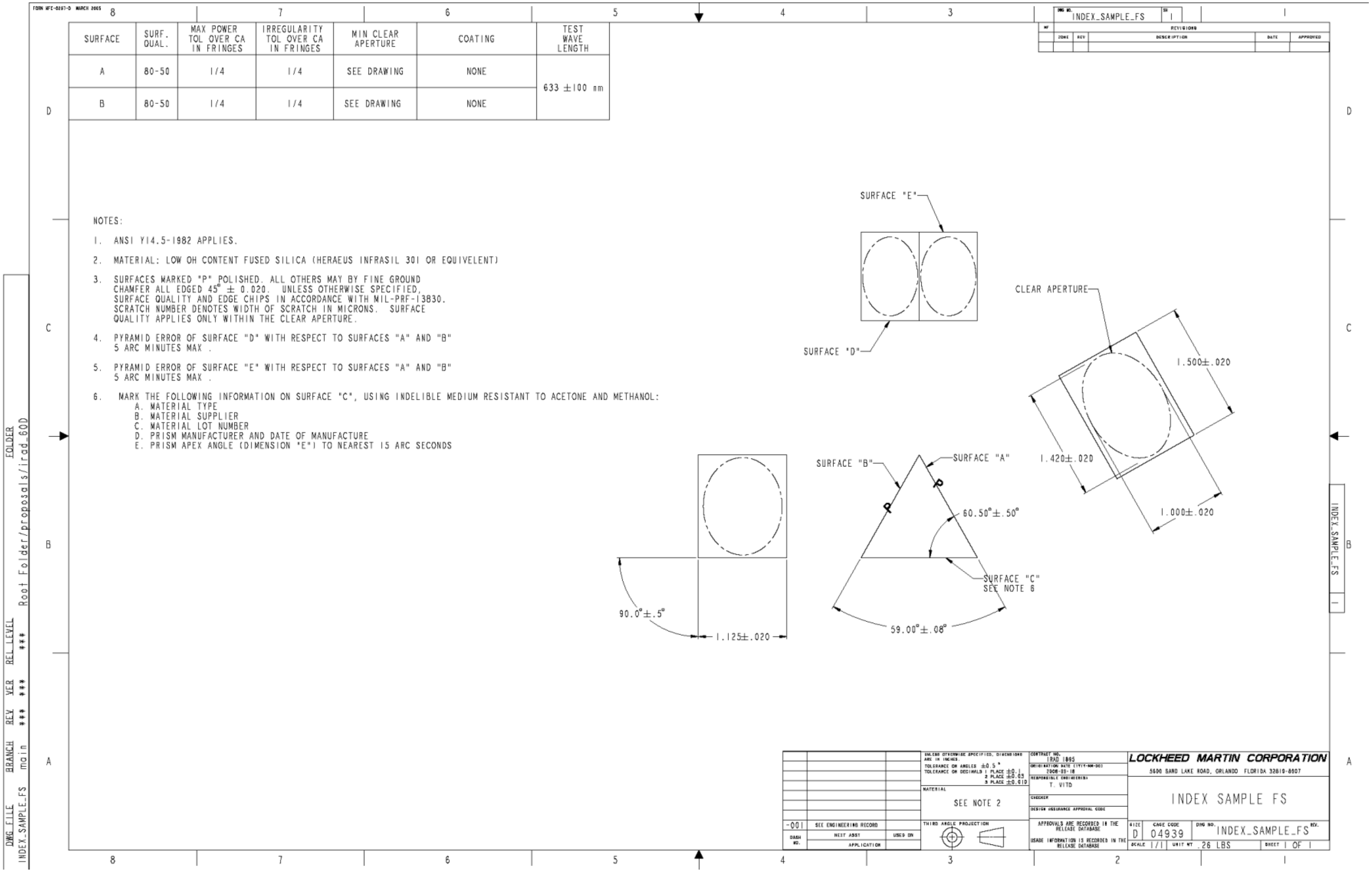
- **ZnSe and Fused Silica only, one prism each**
  - **ZnSe: high index, VIS to LWIR**
  - **Fused Silica (Infrasil 301): low index, UV to MWIR**

- **Protocol**
  - **Minimum deviation method**
  - **Three labs participated**
    - **M3 Measurement Solutions**
    - **WPAFB**
    - **NASA Goddard SFC**
  - **Room temperature only (20 $\pm$ 0.5^{o}C)**
  - **Measure each prism at least 3 times, different operator each time preferred**
  - **Fit results to 3-term Sellmeier formula**

- **Report**
  - **Measured prism angle**
  - **Tabulated results**
  - **Sellmeier formulas**
  - **Measurement error estimate and measurement accuracies**

Round Robin (2 of 10)

Prism Fabrication Drawing



SURFACE	SURF. QUAL.	MAX POWER TOL OVER CA IN FRINGES	IRREGULARITY TOL OVER CA IN FRINGES	MIN CLEAR APERTURE	COATING	TEST WAVE LENGTH
A	80-50	1/4	1/4	SEE DRAWING	NONE	633 ±100 nm
B	80-50	1/4	1/4	SEE DRAWING	NONE	

NOTES:

1. ANSI Y14.5-1982 APPLIES.
2. MATERIAL: LOW OH CONTENT FUSED SILICA (HERAEUS INFRASIL 301 OR EQUIVELENT)
3. SURFACES MARKED "P" POLISHED. ALL OTHERS MAY BY FINE GROUND CHAMFER ALL EDGED 45° ± 0.020. UNLESS OTHERWISE SPECIFIED, SURFACE QUALITY AND EDGE CHIPS IN ACCORDANCE WITH MIL-PRF-13830. SCRATCH NUMBER DENOTES WIDTH OF SCRATCH IN MICRONS. SURFACE QUALITY APPLIES ONLY WITHIN THE CLEAR APERTURE.
4. PYRAMID ERROR OF SURFACE "D" WITH RESPECT TO SURFACES "A" AND "B" 5 ARC MINUTES MAX .
5. PYRAMID ERROR OF SURFACE "E" WITH RESPECT TO SURFACES "A" AND "B" 5 ARC MINUTES MAX .
6. MARK THE FOLLOWING INFORMATION ON SURFACE "C", USING INDELIBLE MEDIUM RESISTANT TO ACETONE AND METHANOL:
    - A. MATERIAL TYPE
    - B. MATERIAL SUPPLIER
    - C. MATERIAL LOT NUMBER
    - D. PRISM MANUFACTURER AND DATE OF MANUFACTURE
    - E. PRISM APEX ANGLE (DIMENSION "E") TO NEAREST 15 ARC SECONDS

UNLESS OTHERWISE SPECIFIED, DIMENSIONS ARE IN INCHES.
TOLERANCE ON ANGLES ±0.5°
TOLERANCE ON DECIMALS 1 PLACE ±0.1
2 PLACE ±0.03
3 PLACE ±0.010

MATERIAL: SEE NOTE 2

CONTRACT NO. IRAD 1895
ORIGINATION DATE (YYYY-MM-DD) 2008-09-18
RESPONSIBLE ENGINEER(S): T. VITO
CHECKER
DESIGN ASSURANCE APPROVAL CODE
APPROVALS ARE RECORDED IN THE RELEASE DATABASE
USAGE INFORMATION IS RECORDED IN THE RELEASE DATABASE

LOCKHEED MARTIN CORPORATION
5600 SAND LAKE ROAD, ORLANDO FLORIDA 32819-8907

INDEX SAMPLE FS

SIZE D
CAGE CODE 04939
DWG NO. INDEX_SAMPLE_FS
REV.
SCALE 1/1
UNIT WT .26 LBS
SHEET 1 OF 1

-001 SEE ENGINEERING RECORD
DASH NO.
NEXT ASSY
USED ON
APPLICATION
THIRD ANGLE PROJECTION

Round Robin (3 of 10)

Measurement Wavelengths

Material	Wavelength Intervals	
	Wavelength Range (nm)	Max Interval (nm)
Fused Silica	400-800	50
	800-2000	100
	2000-4000	500
Zinc Selenide	550-800	50
	800-2000	100
	2000-12000	500

Round Robin (4 of 10)

Laboratory	Reported Prism Apex Angle	
	Fused Silica	ZnSe
M3M	59.17156	28.99852
AFRL	59.17048	28.9982
NASA	59.17143	28.99851
Total Variation	0.00108 (3.9”)	.00032 (1.2”)

Round Robin (5 of 10)

Measurement Variability

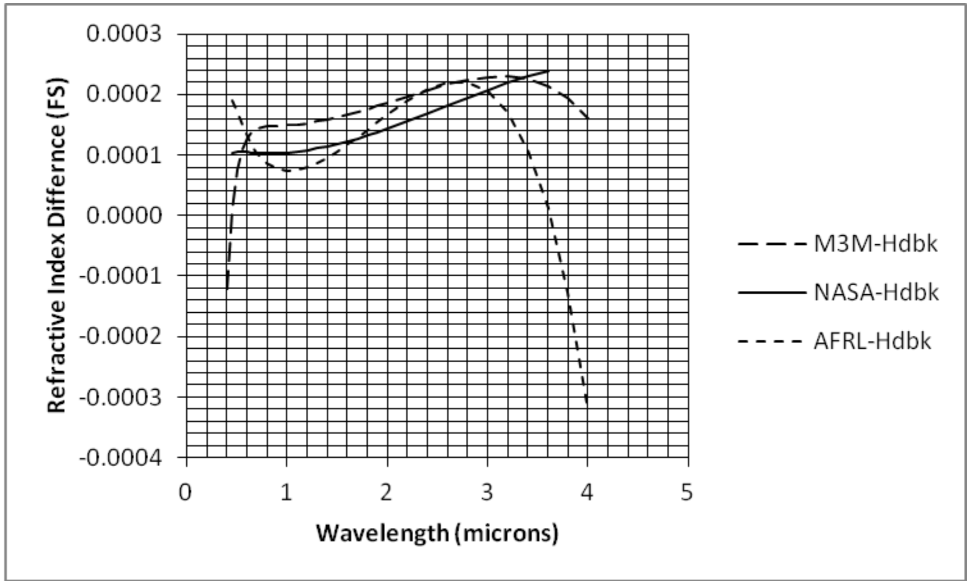
Fused Silica			
Laboratory	PV (average)	SD (average)	RMS Fit Error
M3M (3)	0.000039	0.000021	0.000019
AFRL (15)	0.000080	0.000022	0.000058
NASA (5)	0.000016	0.000006	0.000005

ZnSe			
Laboratory	PV (average)	SD (average)	RMS Fit Error
M3M (3)	0.000095	0.000049	0.000022
AFRL (15)	0.000500	0.000157	0.000848
NASA (3+)	0.000068	0.000031	0.000023

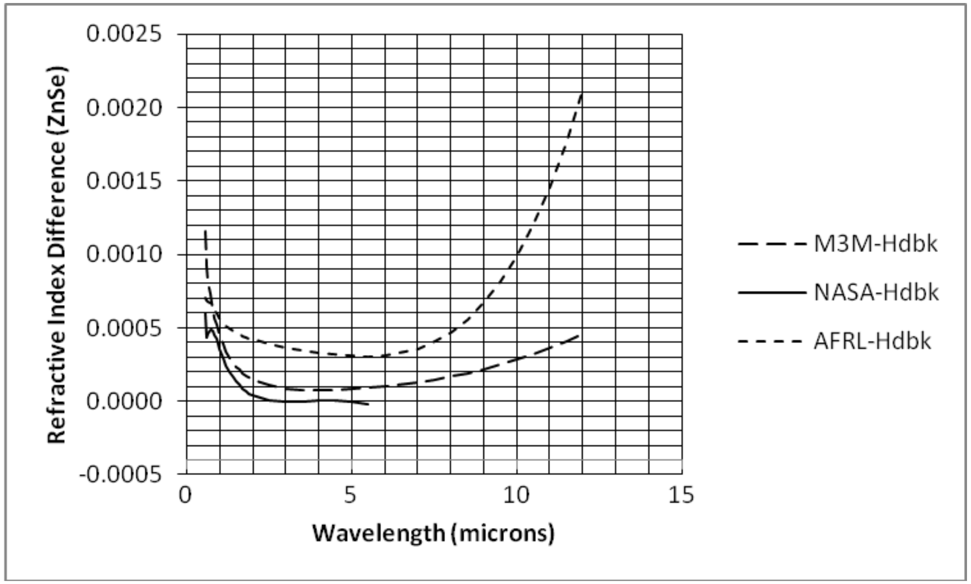
Round Robin (6 of 10)

Measurement Reproducibility (Sellmeier Fits)

Fused Silica



Zinc Selenide



Round Robin (7 of 10)

Temperature-Dependent Dispersion Formula (NASA)

$$n^2(\lambda, T) = 1 + \frac{S_1(T)\lambda^2}{\lambda^2 - L_1^2(T)} + \frac{S_2(T)\lambda^2}{\lambda^2 - L_2^2(T)} + \frac{S_3(T)\lambda^2}{\lambda^2 - L_3^2(T)}$$

$$S_1(T) = S_{10} + S_{11}T + S_{12}T^2 + S_{13}T^3 + S_{14}T^4$$

$$L_1(T) = L_{10} + L_{11}T + L_{12}T^2 + L_{13}T^3 + L_{14}T^4$$

$$S_2(T) = S_{20} + S_{21}T + S_{22}T^2 + S_{23}T^3 + S_{24}T^4$$

$$L_2(T) = L_{20} + L_{21}T + L_{22}T^2 + L_{23}T^3 + L_{24}T^4$$

$$S_3(T) = S_{30} + S_{31}T + S_{32}T^2 + S_{33}T^3 + S_{34}T^4$$

$$L_3(T) = L_{30} + L_{31}T + L_{32}T^2 + L_{33}T^3 + L_{34}T^4$$

Round Robin (8 of 10)

Conclusions and Observations

1. **The repeatability of the measurements is significantly influenced by the ability to maintain sufficient signal-to-noise ratio (SNR) across the wavelength range of interest. SNR is a function of both the material transmittance and refractometer characteristics (source, gratings, and detectors). The M3M refractometer is the only laboratory currently able to cover reliably the entire LWIR wavelength range of interest out to 12000nm.**

2. **Given sufficient SNR, the average repeatability of measurements is on the order of 0.00004 or better for VIS/MWIR material (e.g., fused silica), and 0.00010 or better for VIS/MWIR/LWIR material (e.g., ZnSe). The NASA refractometer provides exceptionally good repeatability and Sellmeier fits over its operational wavelength range.**

3. **Based on a comparison of the Sellmeier fits, reproducibility is better than about 0.0005 over a large part of the wavelength range of interest, but degrades significantly when SNR degrades. This suggests the need for SNR standards for refractive index measurements.**

Round Robin (9 of 10)

Conclusions and Observations

4. The typical index repeatability variation is good (i.e., small) enough to provide meaningful measurements of index for optical design and tolerancing. When developing standards on refractive index and dispersion, we should anticipate spectral variations in standard tolerances due to spectral variations in repeatability and reproducibility of the measurements.

5. To minimize Sellmeier fit errors at the ends of the wavelength range of interest, the measurement range should exceed the wavelength range of interest. The amount of excess is a consideration to be addressed during program planning.

6. The NASA refractometer would require upgrades to extend its operation into the LWIR. It appears that the AFRL refractometer would also require upgrades to improve SNR and repeatability above 10000nm. The cost of these upgrades needs to be funded.

Round Robin (10 of 10)

Conclusions and Observations

7. Another practical consideration for a large measurements program is the availability of personnel to execute the program. All the labs have small staffs, generally with one to at most a few personnel available and trained to run the instruments. Both AFRL and NASA personnel are primarily dedicated to priorities other than performing IR material measurements. A high degree of automation is desirable to help mitigate this.

8. The NASA method for gathering and fitting index data over wavelength and temperature has great appeal for efficiently and cost-effectively generating dispersion and dn/dT values.

9. Protocols must be in place to ensure that equipment is calibrated and remains calibrated.

10. The potential for equipment failures is a concern that must be taken into account in program planning.

Required Supporting Standards

Parameter	Standard	Title/Description	Comments
Refractive Index	ISO CD/17328	Optics and photonics — Optical materials and components — Test method for refractive index of infrared optical materials	Under development – Stage 30.20 (Ballot)
Curve Fitting	EDSU 06025	Guide to linear Least Squares curve fitting techniques	Active
dn/dT	None known		ISO CD/17328 could be revised to cover dn/dT
Abbe Number	NA		
Internal Transmittance	ISO 11551	Optics and photonics — Lasers and laser-related equipment — Test method for absorbtance of optical laser components	Active
	ISO 15368	Optics and optical instruments — Measurement of reflectance of plane surfaces and transmittance of plane parallel elements	Active
Inhomogeneity	ISO NP/17411	Optics and photonics — Optical materials and components — Test method for homogeneity of optical glasses by laser interferometry	Under development – Stage 30.20 (Ballot)
Inclusions	None known		

Study Design Issues

- Single lab or Round robin (R&R)
- Blind test
- Single measurement or multiple measurements of each sample
- Number of samples
- Sampling protocols

IR Materials Standard Structure

Topic	Option 1	Option 2	Option 3
Spec Structure	One standard covers all materials	One standard for each material	One standard for each material class
Transmittance	Transmittance through standard thickness	Attenuation/absorption coefficient	
Inclusions	Number and size		
Temperature dependence of Refractive Index	Index at 20°C (Sellmeier Fit) + dn/dT vs T	Temperature-dependent Sellmeier Fit	
Inhomogeneity	Within a standard blank size		
Inclusions			
Birefringence			
Other Properties			

Measurement Protocols

- Refractive index and Abbe number
  - Wavelength range (overall)
  - Wavelength range (Abbe number)
  - Wavelength interval
  - Temperature range
  - Temperature interval
  - Number of measurements
  - Dispersion curve fitting
  - Reporting format

- Transmittance
  - Wavelength range
  - Wavelength interval
  - Temperature range
  - Temperature interval
  - Number of measurements
  - Reporting format

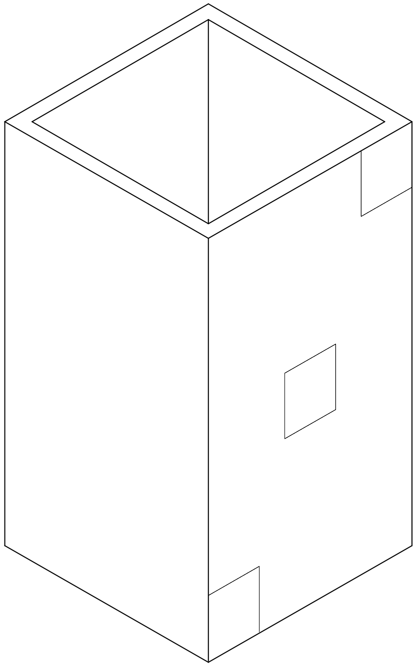
- Homogeneity
  - Wavelength
  - Number of measurements

- Inclusions
  - Minimum dimension
  - Length/width ratio
  - Wavelength range

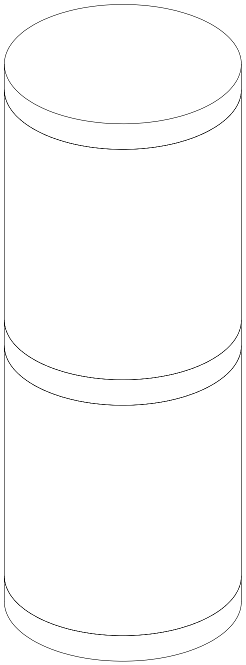
Sampling Protocols

- Sampling frequency (temporal)
- Sample shape and dimensions
- Sample location(s) within batch
- Sample orientation
- Tracking information
  - Material
  - Supplier
  - Batch number/date
  - Sample location/coordinates

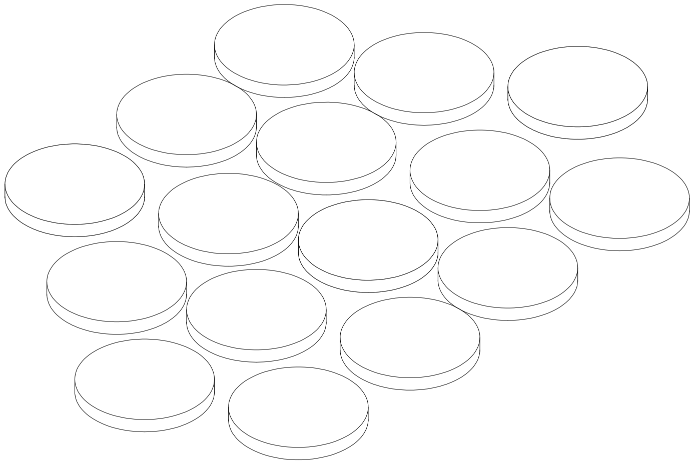
Proposed Sample Locations



CVD Plates



Ingots



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Proposed Sample Configuration Schedule

Test	Sample Configuration
Refractive Index	Prism, 1.5” x 1.0” entrance/exit face
Attenuation Coefficient	Disk, 1” diameter x 0.25” thick
Homogeneity	Disk, 6” diameter x 0.6” thick
Inclusions	Disk, 6” diameter x 0.6” thick

Inhomogeneity Measurement

- **Spatial resolution**
- **Sample configuration**
- **Index resolution**

Inclusion Measurement

- **Spatial resolution**
- **Sample configuration**
- **Index resolution**

Preliminary Cost Estimate
Each material, each supplier

Sample Preparation and Measurement

Sample Type	Est. Cost			
	Material	Sample Fab	Measurement and Report	Total
Refractive Index	NC	$1500	$2000	$3500
Transmittance	NC	$100	$100	$200
Inhomogeneity/Inclusions	NC	$500	$300	$800
			Total	$4500

Other costs

Item	Est. Cost
Inclusion scanner	$250,000
Refractometer upgrades	$50,000

Volunteers

Test Plans	Instrumentation	Sampling	Sample Fab	Funding

Supplemental

INTERNATIONAL STANDARD

ISO 11382

First edition
2010-10-01

Optics and photonics — Optical materials and components — Characterization of optical materials used in the infrared spectral range from 0,78 µm to 25 µm

Optique et photonique — Matériaux et composants optiques — Caractérisation des matériaux optiques utilisés dans la bande spectrale infrarouge de 0,78 µm à 25 µm

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Reference number
ISO 11382:2010(E)

J4413

INTERNATIONAL STANDARD

ISO 11551

Second edition
2003-12-01

Optics and optical instruments — Lasers and laser-related equipment — Test method for absorptance of optical laser components

Optique et instruments d'optique — Lasers et équipements associés aux lasers — Méthode d'essai du facteur d'absorption des composants optiques pour lasers

Reference number
ISO 11551:2003(E)

INTERNATIONAL
STANDARD

ISO
15368

First edition
2001-08-01

Optics and optical instruments — Measurement of reflectance of plane surfaces and transmittance of plane parallel elements

Optique et instruments d'optique — Méthode de mesurage de la réflectance des surfaces planes et de la transmittance des éléments à plan parallèle

Reference number
ISO 15368:2001(E)

Agenda for Joint Meeting of
ASC OP/TF6 and SPIE IR Materials Standards Working Group

August 16, 2012
1400-1630 PDT
San Diego Convention Center
111 w. Harbor Drive
San Diego, CA 92101

1. Welcome and Introductions	**G. Wiese**
2. Adoption of Agenda	**G. Wiese**
3. Working Group Organization and Meetings	**G. Wiese**
4. Follow-up on actions from DSS meeting	
A. NIST role	**L. Hanssen**
B. Materials, wavebands, and temperatures of interest	**All**
5. Standards Development Tasks	**D. Aikens**
A. Review of any internal standards in use	
B. Structure and list of required standards	
C. Plan and schedule for measurements	
D. Assign project leaders	
6. Material Measurement Tasks	**G. Wiese**
A. Structure of measurements	
B. Plan and schedule for measurements	
C. Review Round Robin results	
D. Cost estimate	
E. Material sampling protocols	
F. Inhomogeneity measurements	
G. Inclusion measurement instrumentation	
H. Funding mechanisms	
7. Time and Place for next ASC OP/TF6-SPIE IRMSWG Meeting	**G. Wiese**
8. Adjourn	**G. Wiese**