Bringing the Robotic Optical Scattering Instrument (ROSI) On Line for Calibrations

Heather Patrick

CORM 2017

August 1, 2017

The Team

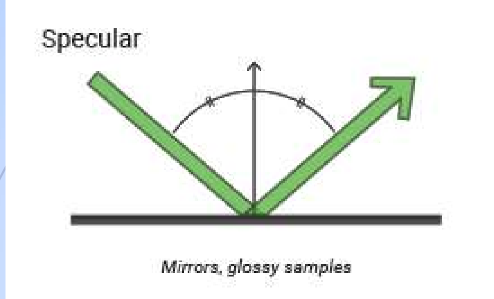
Clarence Zarobila,
Heather Patrick,
Catherine Cooksey

Thom Germer

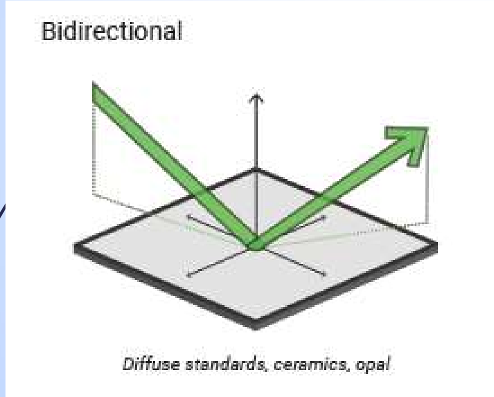
Cameron Miller, Group Leader

…with thanks to many co-authors and collaborators at NIST!

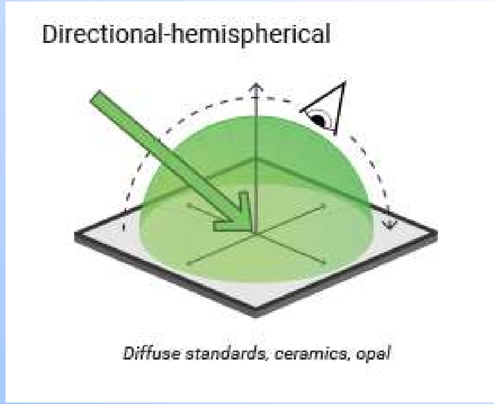
Reflectance Geometries



$\theta_i = \theta_r$ Reflectance $\rho(\lambda,\mathrm{pol})$
In-plane



Specific incident and view combination
BRDF($\theta_i,\phi_i,\theta_r,\phi_r,\lambda$,pol), Reflectance Factor
Can be out-of-plane

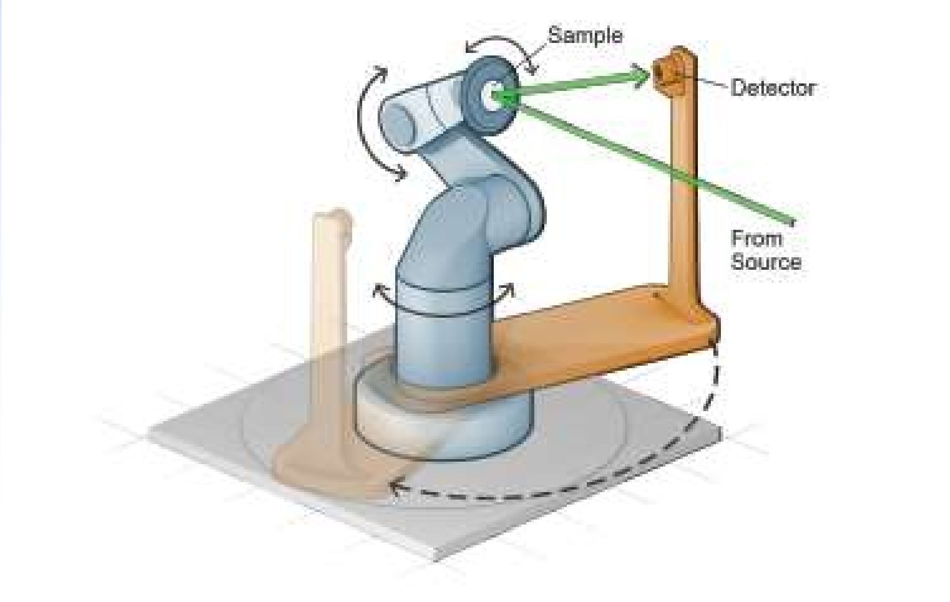


Fixed incident, collect over full hemisphere
Integrate BRDF, or use sphere

Thanks to Sean Kelley for drawings!

ROSI

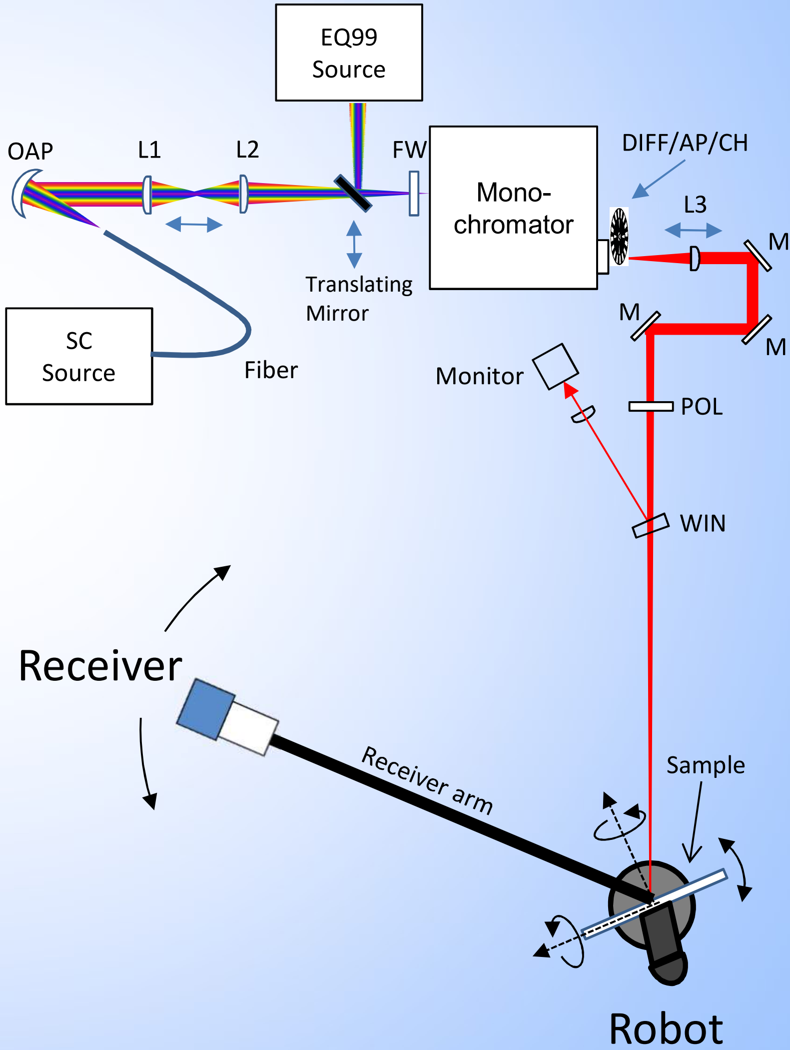
- Next generation reference reflectometer
- Designed to replace current instrument, STARR
- Multi-angle, multi-wavelength scattering and reflectance
- Includes new features
  - Improved source power
  - Out-of-plane capabilities
  - Improved sample handling
  - Full automation of sources and detectors



Light Sources	Tunable, based on laser-driven light source and super-continuum source with monochromator
Detectors	Silicon UV/VIS and extended indium-gallium arsenide (EIGA) for SWIR
Wavelength Range	250 nm to 2400 nm Polarization available
Bandwidth	14 nm
Geometries	• 0° to 80° incident/viewing angles (specular/bidirectional) • Optional out-of-plane viewing (bidirectional)
Sample size	• 50 mm to 300 mm • Up to 6 kg
Beam size	10 mm
Measurands	• Reflectance ρ • Reflectance factor R • BRDF f_r

ROSI Source/Receiver Detail

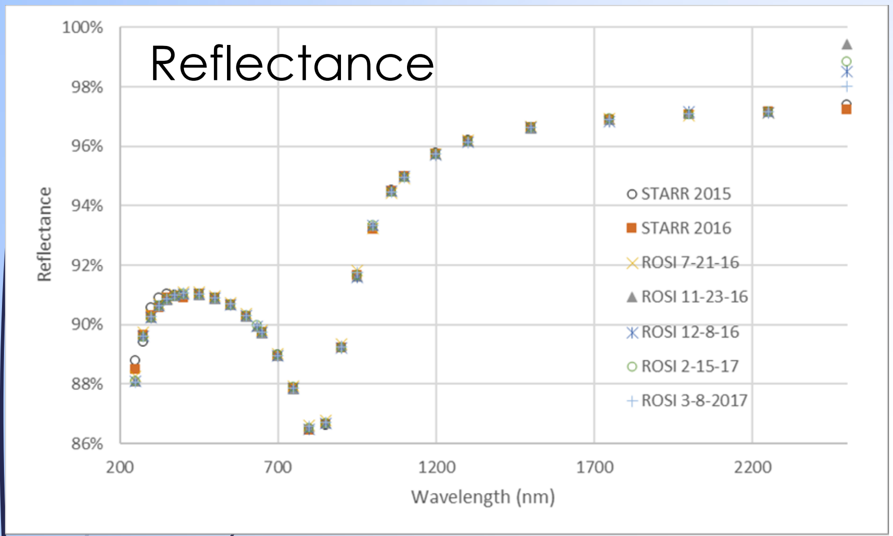
- **Light Source:**
  - Broadband input to monochromator, switchable SC/EQ99
  - Tunable output 250 nm – 2400 nm
  - Automated source switching
- **Receiver:**
  - Si detector for UV/VIS
  - Extended InGaAs (EIGA) for SWIR
  - Switch at push of a button

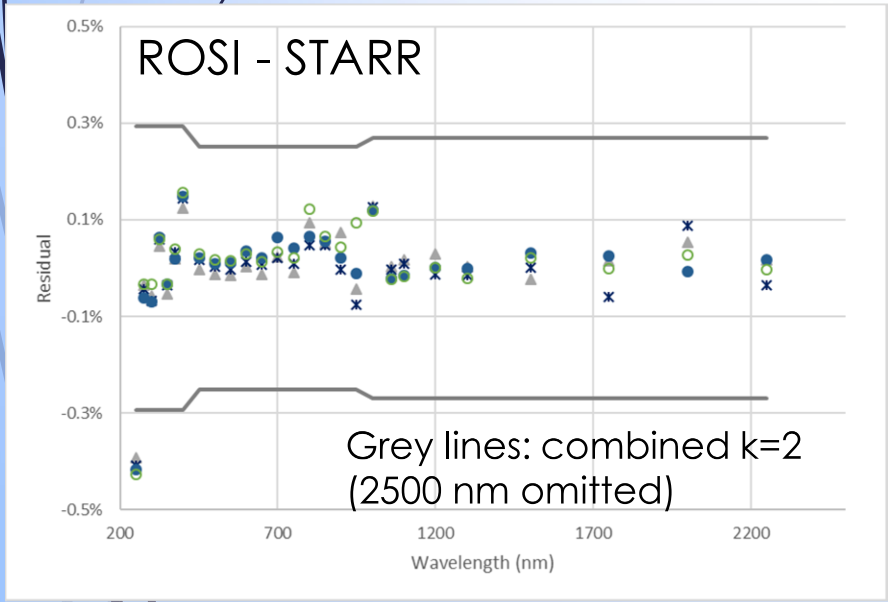


Calibration Comparison Overview

- Current reference instrument for reflectance, STARR, has been well validated throughout years of international comparisons but is nearing end of service life
- ROSI source and receiver UV upgrade complete early 2016; enabled duplication of STARR customer calibrations on ROSI
- Initially Al first surface mirror SRM 2003 (99G32) with several months of data
- ROSI repeated STARR calibrations for 3 mirrors
  - C1 1" dielectric mirror
  - C2 1" Al mirror
  - C3 2" protected Al mirror
- Recently measured additional check standards
  - SN 67 2nd surface, and SRM 2011 090204 also measured on ROSI and STARR
- One diffuse (BRDF) calibration also repeated, topic for a later date
- Error budget and lessons learned!

Comparisons – SRM 2003 Check



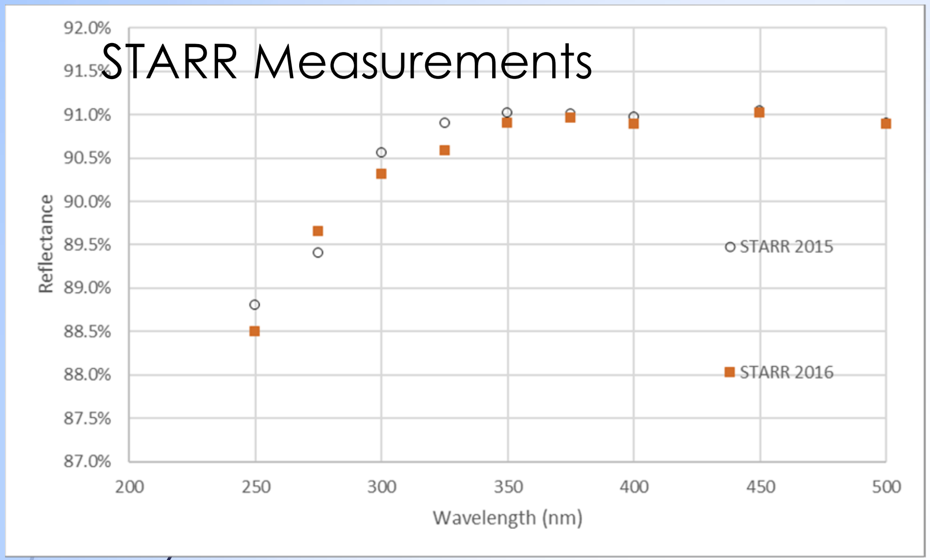


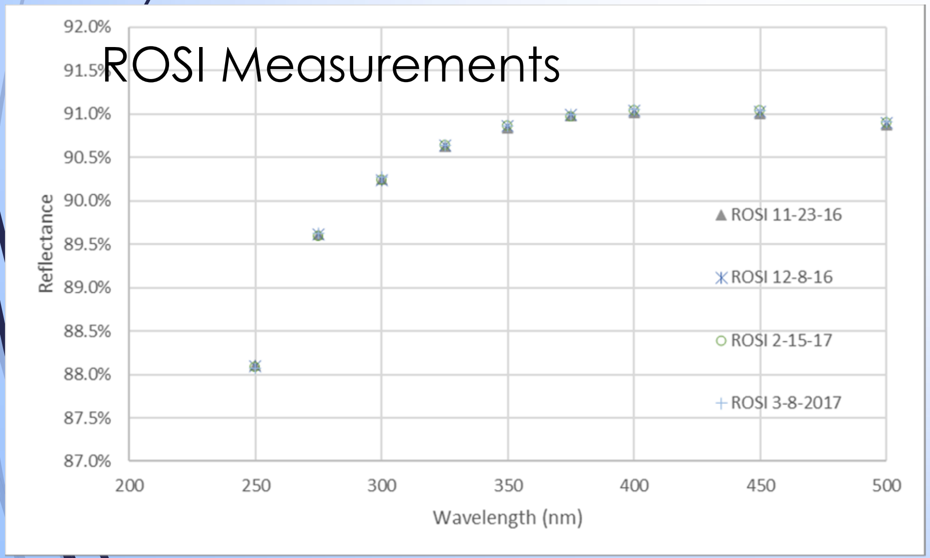
- SRM 2003 series
- 1st surface Al 2" mirror, ROSI check standard
- Comparing all data to STARR 2016 measurement

Lessons learned

- Not enough light for 2500 nm
  - Fianium generally much brighter than lamp
  - But, falls off quickly at very longest wavelengths
- Mid-November, made modification to monitor optics, improved agreement
- ROSI reproducibility generally better than 0.1%

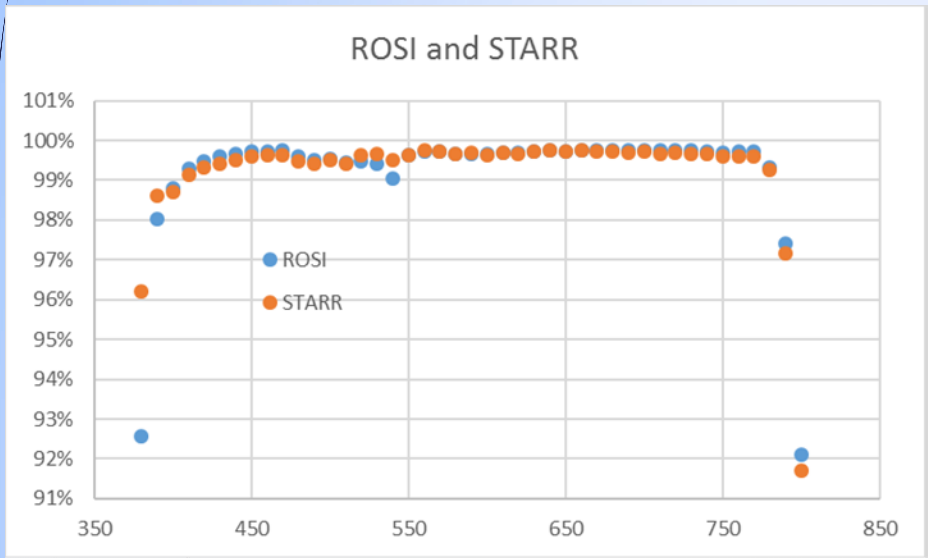
Improved Reproducibility in UV

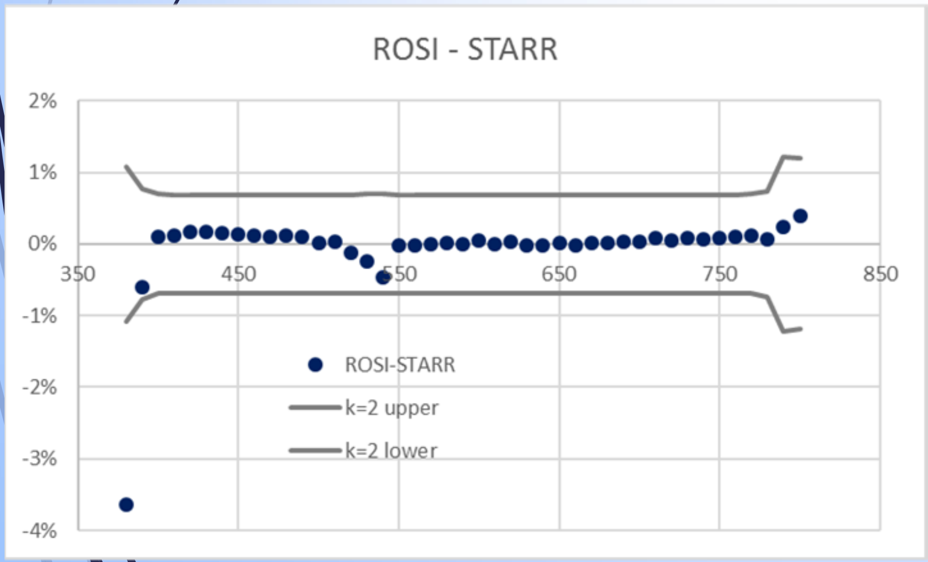




- STARR reproducibility is on 0.1% level for visible, but worse in UV
- ROSI reproducibility generally better than 0.1%, even in UV
- ROSI has more power at UV wavelengths than in STARR, due to EQ99 source
- ROSI does not have to switch lamps at 400 nm

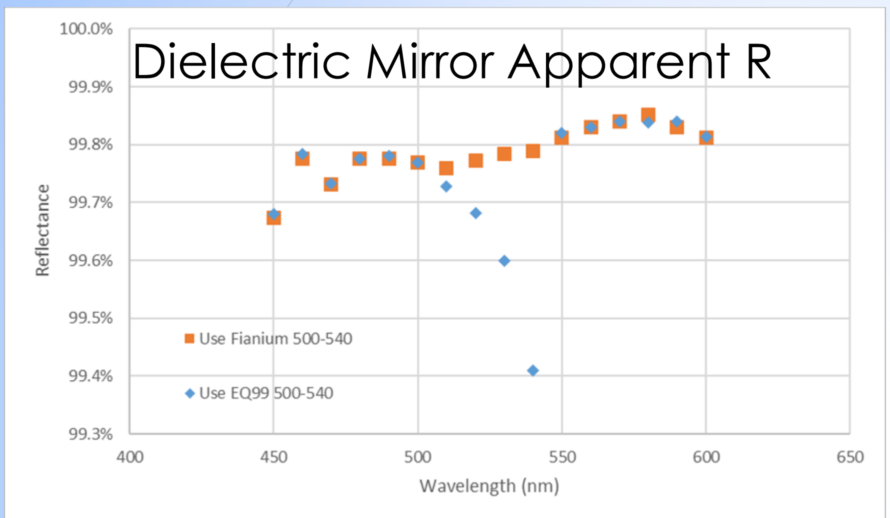
Comparison - C1



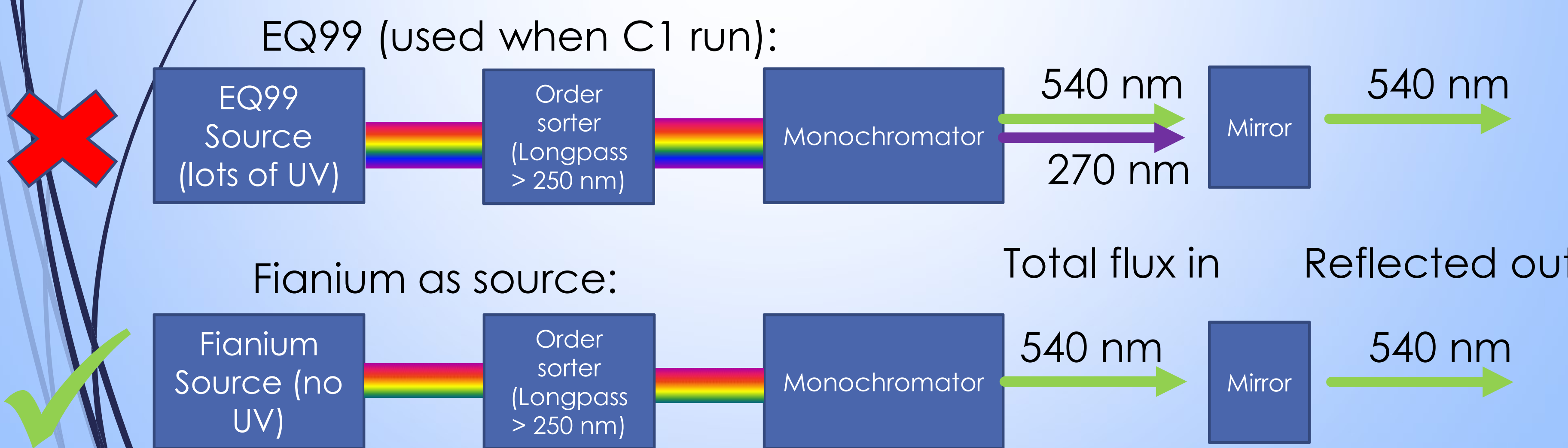


- Dielectric, 1" mirror with sharp roll-off both ends
- A couple of interesting areas…
  - What happened between 510 nm – 540 nm???
  - Discrepancy in UV

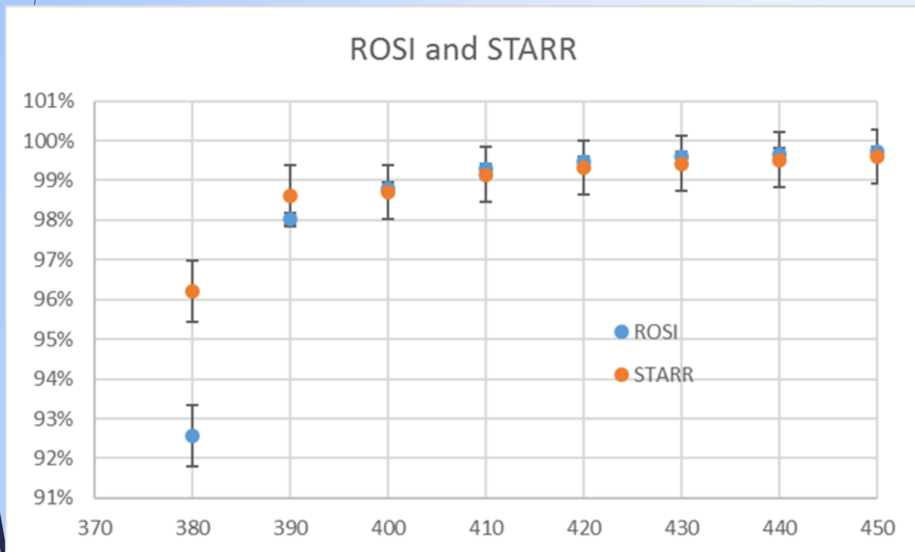
What happened 510-540 nm?

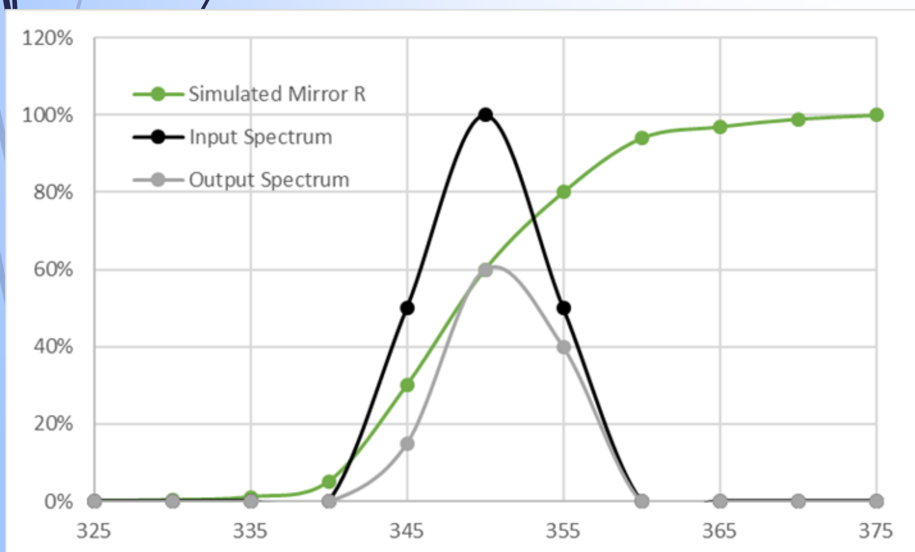


- With C1, used EQ99 up to 540 nm (like blue at left)
- But, order sorting was optimized for Fianium (no UV). Monochromator lets a little 270 nm through!
  - This 270 nm light adds to the total flux measurement in the EQ99 case
- The mirror itself also acts as a filter and does not reflect 270 nm light
  - So, when EQ99 was used, the mirror "reflectance" looked low, because the 270 nm from total flux was not included
- Lesson learned – use Fianium from 500 nm up!



C1 UV Discrepancy





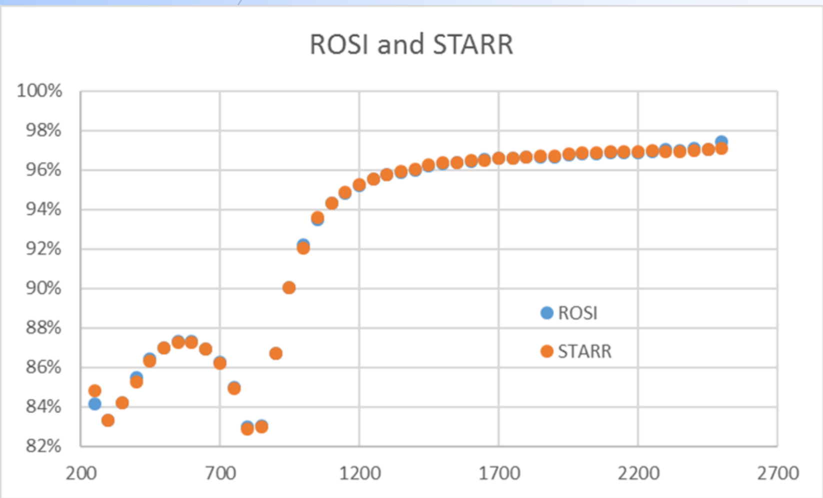
- Outside k = 2 at 380 nm

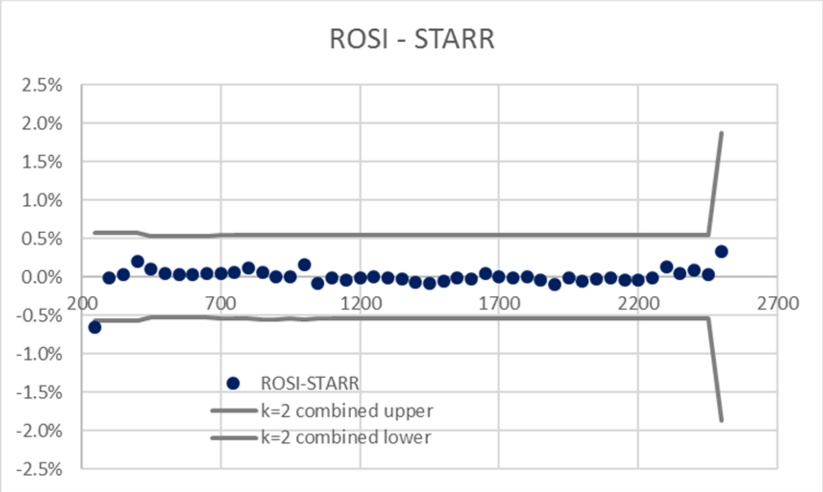
Possible explanations

- Mirror spectrum is not “white”
- STARR and ROSI 14 nm BW
  - Skews result when mirror reflectance is not a linear function of wavelength
  - ROSI/STARR wavelength uncertainty may be underestimating this
- STARR and ROSI likely have different spectral shapes
- Check of STARR wavelength accuracy in UV is less direct than in ROSI
- Believe ROSI will be the more accurate
- Should consider increasing scan beyond customer requested wavelengths, to improve uncertainty estimate

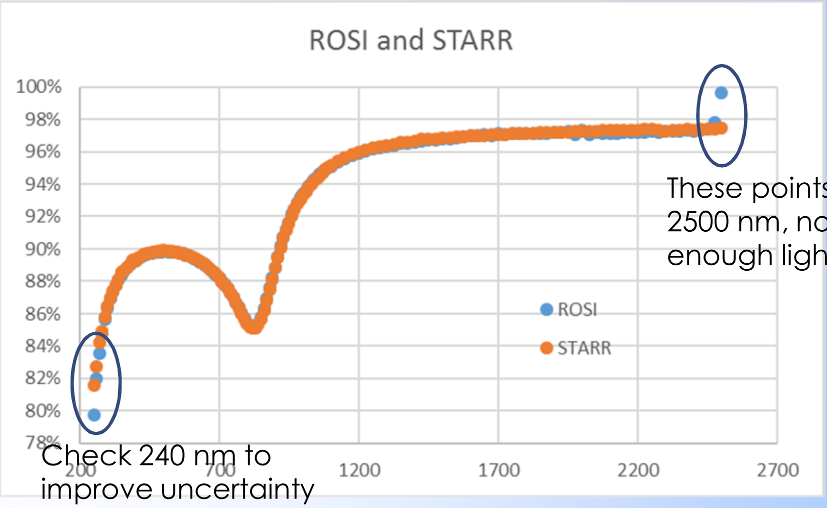
Comparison – C2, C3

Mirror C2



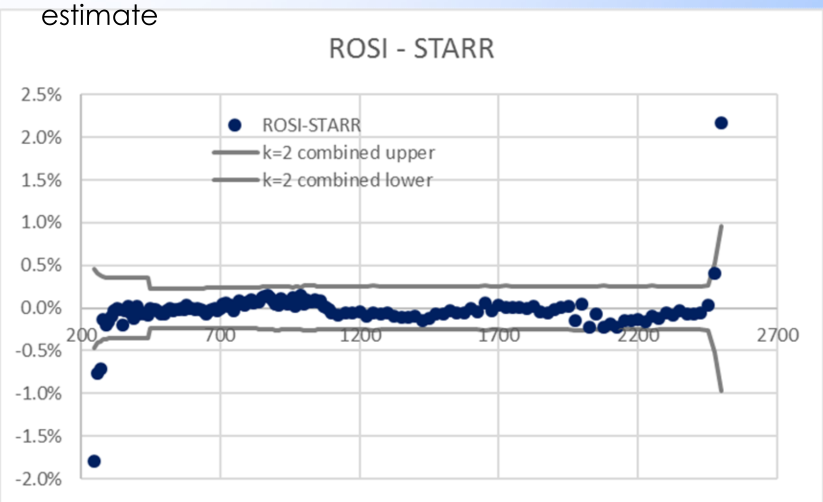


Mirror C3



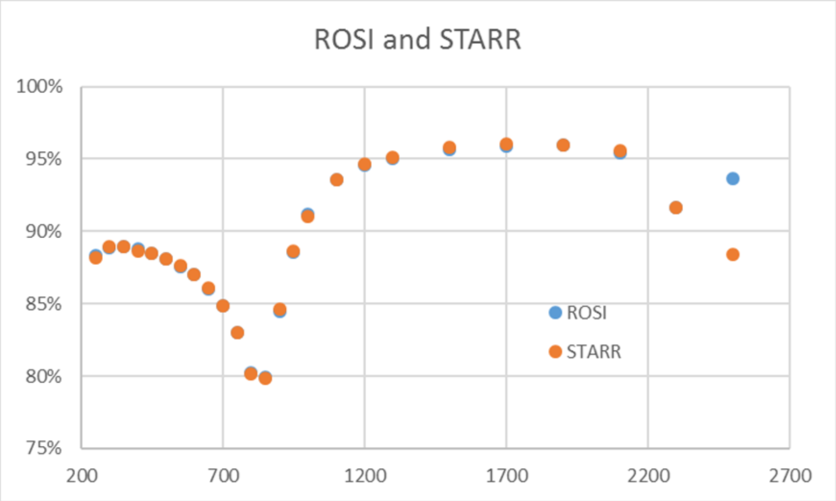
These points 2475, 2500 nm, not enough light

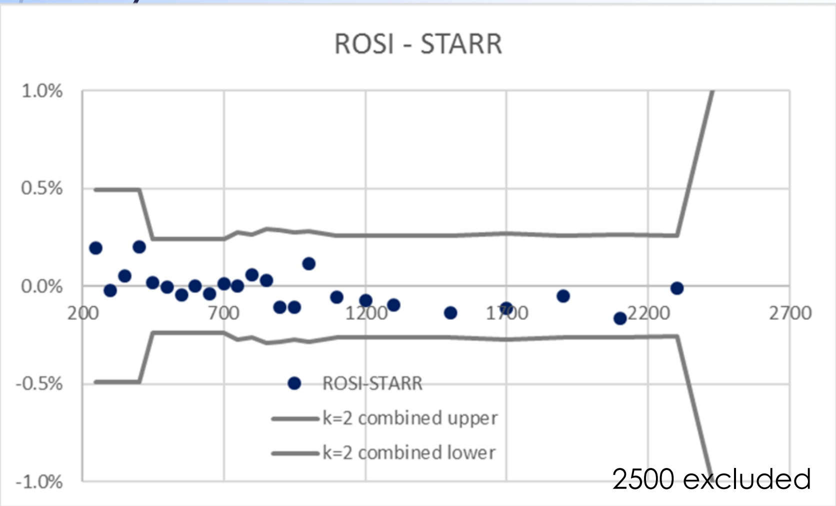
Check 240 nm to improve uncertainty estimate



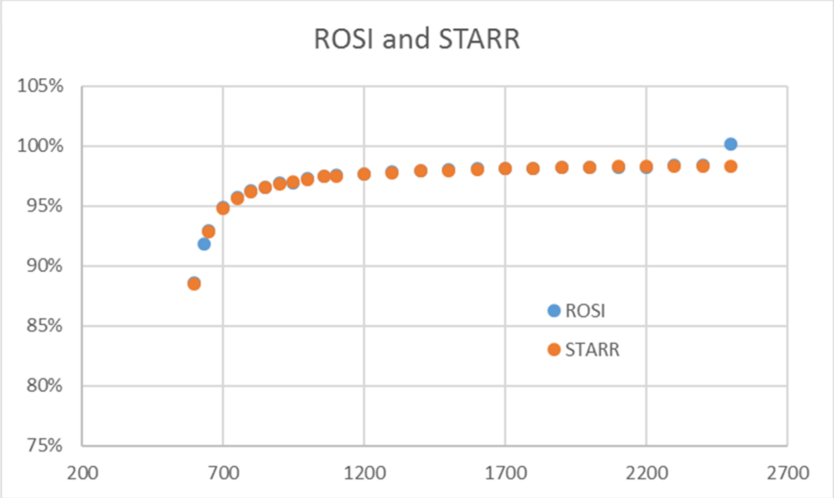
Comparison – SN67, SRM 2011

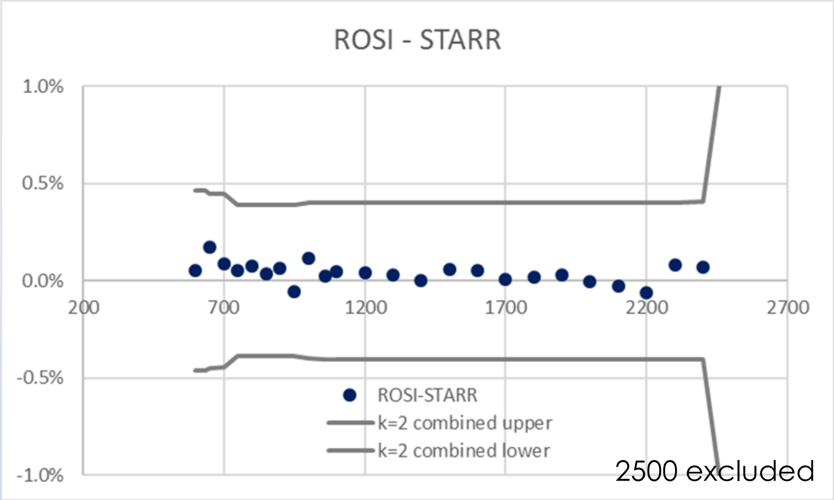
Mirror SN67





Mirror SRM 2011





Uncertainty budget, Specular - ROSI

Prelim uncert ROSI			
Component	Relative Standard Uncert		Notes
	Si Direct Detector	EIGA on Sphere	
Noise	variable	variable	added from data noise and gain ratio uncertainty
Ratio drift	0.05%	0.08%	stability of monitor and detectors over time
Detector uniformity	0.04%	0.01%	checked by translating detectors up and down
Wavelength	variable	variable	added at each wavelength based on R(lambda) slope
Sample uniformity	variable	variable	reflectance vs. illuminated area of sample
Incidence angle	variable	variable	typically small
total k=2	0.13%	0.16%	before variable components added

Notes

- Noise - relative standard uncertainty typically < 0.01% k = 1, usually no gain ratio (fixed gain)
- Wavelength – depends upon slope R(λ) and 0.7 nm k=1 wavelength centroid uncertainty
- Sample uniformity - spot checked at 550 nm wavelength
  - 0.01% for C3, 0.05% for C2
  - ROSI values smaller component than in STARR – beam size?
- Incidence angle
  - Small, 0.07° k=1 angle uncertainty
  - Almost always a very small component (0.01%)
- In spectrally flat region, for mirror, total k = 2 typically < 0.2%

Lessons Learned: STARR and ROSI on 6 mirrors

- Agree within k=2 in spectrally flat regions below 2400 nm
  - At present, will limit ROSI calibrations to 2400 nm and below
- ROSI improved noise and reproducibility in UV
- Mirrors with steep R(λ) in UV can give few % discrepancy between ROSI – STARR
  - Both were designed for spectrally neutral samples
  - Mitigate by better estimate of uncertainty at shortest λ
  - ROSI believed to have improved UV wavelength accuracy
- Good to expand comparison beyond check standards
  - Identified order sorting issue that might have been missed

Outlook

- Plan to begin performing mirror calibrations on ROSI
- ROSI documentation for quality system is complete
  - User manual
  - Measurement assurance manual
- ROSI automation is very functional, have some planned upgrades
  - Adding safeguards for wavelength/grating combination selections
  - Automated run definition generation
  - Possible additional automation of data analysis
- Effort to transfer other calibration types in process

Thank you!