

---

---

# TiO<sub>2</sub> Coupon Reflectivity and Aging Study

Mackenzie Devilbiss  
Scintillator R&D Workshop  
5/19/23

---

---

# Overview

- Aging, reflectance over time studies
  - Coupons: USPN115101
  - Standards: NOvA N-27-09-NC, GoreDRP
  - Extrusions: thin extrusions, polymer and cladding sides
  - Results
- Reflectance metric
  - Defining a metric to decouple POPOP fluorescence over time
  - Reflectance metric over time
  - Stability measurements
  - Results

# Aging Study: Measuring Reflectance Over Time

Quick intro:

- I measured that the Mu2e CRV is aging at a higher overall rate than expected, around 8% per year based on the first year of preliminary cosmic ray data at Wideband
- The reason for high CRV aging rate is not understood, is one component of the CRV bars responsible for aging?
  - Components: cladding, fibers, scintillator
- If the cladding changes in reflectivity over time, could it have a significant impact on the light yield in the CRV bars?... change mean number of bounces that light has with the cladding surface?

# Methodology of Reflectance Over Time

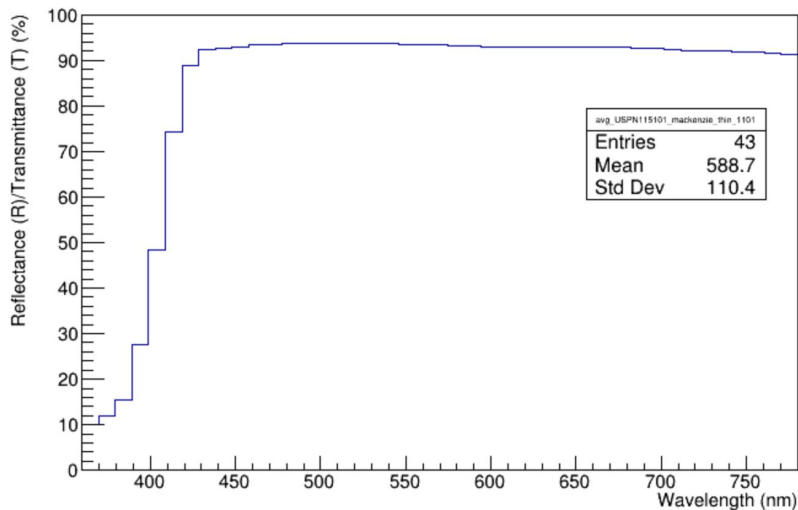
- Using the HunterLabs UltraScan VIS Reflectometer in Lab 6, the reflectivity of different samples has been measured on a ~weekly basis for some time
- If we track the reflectivity over time, do we see a trend?
- Often have multiple samples or take multiple measurements on one sample to get standard deviation errors
- Reflectometer instrument presents data as R/T%, this really just means R

# Measurement Samples and Dates

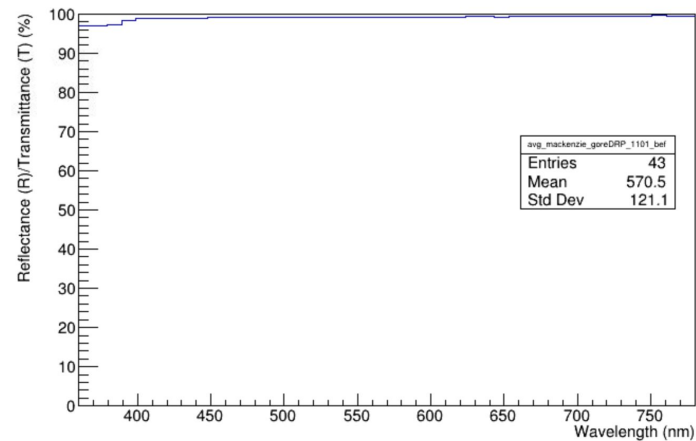
- TiO<sub>2</sub> cladding coupon: USPN115101
  - This is the coupon batch that we received in March 2022 while I was at the lab. This sample has been regularly measured since arrival at Fermilab, beginning 3/17/22
- Standards: NOvA N-27-09-NC and GoreDRP
  - The NOvA sample has always been used to calibrate the reflectometer, it is a well-known sample that has been in use for some time
  - GoreDRP is a highly reflective material with a relatively flat spectrum on the reflectometer. Measurements on this standard began 8/31/22
- Scintillator extrusion samples
  - Machined by Alan to be thin samples, though a small amount of polymer is present on the inner side of the sample. Both sides have been measured since 9/6/22
  - 10 of these samples produced, 5 chosen at random for each measurement

# Reflectometer Spectra Examples

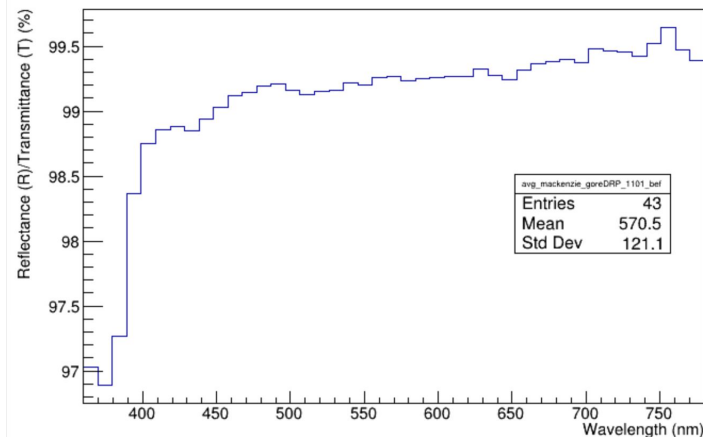
avg\_USPN115101\_mackenzie\_thin\_1101



avg\_mackenzie\_goreDRP\_1101\_bef



avg\_mackenzie\_goreDRP\_1101\_bef



# Average Results and Interpretation

- These results are averages across all wavelengths from 1D histograms of slopes from all wavelengths, sign indicates direction of aging
- TiO<sub>2</sub> coupons:
  - Thin side:  $-0.41\% \pm 0.12\%$  lost per year
  - Thick side:  $-0.22\% \pm 0.10\%$  lost per year
- Extrusion samples:
  - Polymer side:  $+0.87\% \pm 1.2\%$  gained per year
  - Cladding side:  $-0.58\% \pm 0.99\%$  lost per year
- Standards:
  - NoVA N-27-09-NC:  $+0.62\% \pm 0.23\%$  gained per year
  - GoreDRP:  $+0.16\% \pm 0.14\%$  gained per year

# Average Results and Interpretation

- These results are averages across all wavelengths from 1D histograms of slopes from all wavelengths, sign indicates direction of aging
- TiO<sub>2</sub> coupons:
  - Thin side:  $-0.41\% \pm 0.12\%$  lost per year
  - Thick side:  $-0.22\% \pm 0.10\%$  lost per year
- Extrusion samples:
  - **Polymer side:  $+0.87\% \pm 1.2\%$  gained per year**
  - Cladding side:  $-0.58\% \pm 0.99\%$  lost per year
- Standards:
  - **NoVA N-27-09-NC:  $+0.62\% \pm 0.23\%$  gained per year**
  - GoreDRP:  $+0.16\% \pm 0.14\%$  gained per year

**Ignore these two samples:**

**Polymer side of extrusion samples likely have POPOP fluorescence**

**NOvA sample not well understood now, is old and not sure why positive slope**



# Average Results and Interpretation

- These results are averages across all wavelengths from 1D histograms of slopes from all wavelengths, sign indicates direction of aging
- TiO<sub>2</sub> coupons:
  - **Thin side: -0.41% ± 0.12% lost per year**
  - **Thick side: -0.22% ± 0.10% lost per year**
- Extrusion samples:
  - Polymer side: +0.87% ± 1.2% gained per year
  - **Cladding side: -0.58% ± 0.99% lost per year**
- Standards:
  - NoVA N-27-09-NC: +0.62% ± 0.23% gained per year
  - **GoreDRP: +0.16% ± 0.14% gained per year**

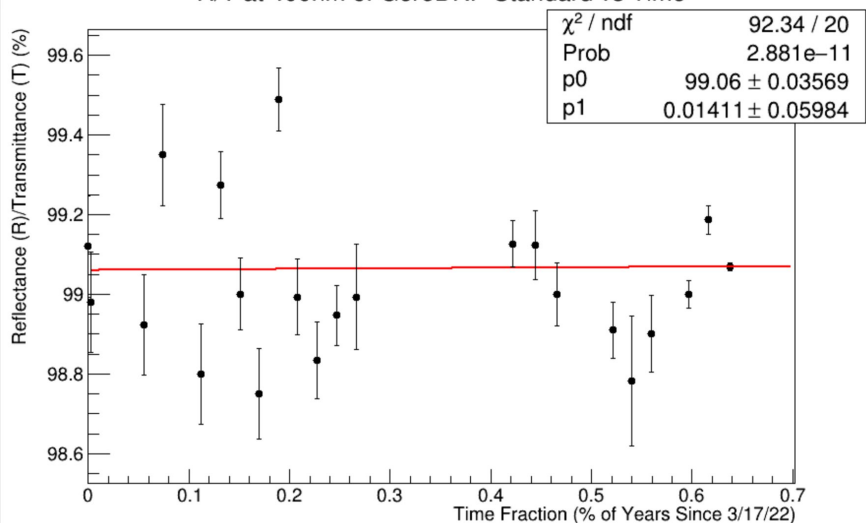
**TiO<sub>2</sub> coupon samples give reasonable slopes, as does the cladding side of the extrusions**

**Use GoreDRP as standard, this is very stable as expected**

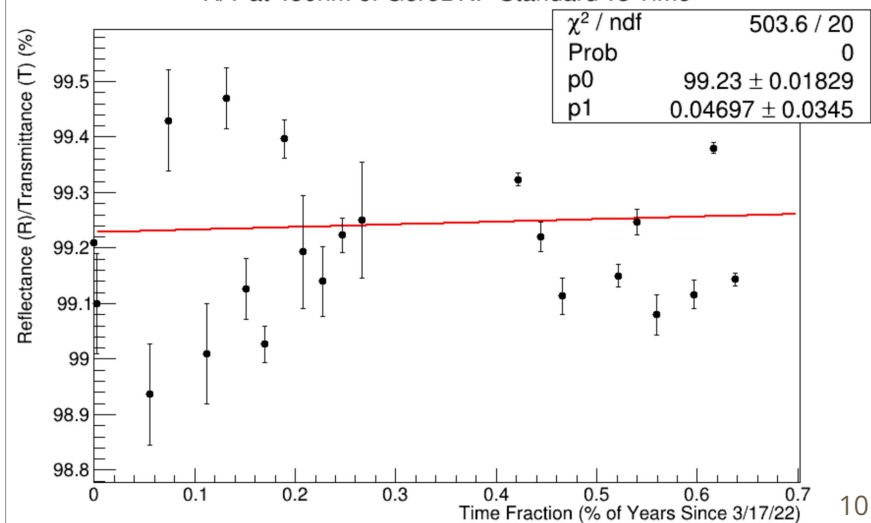
# GoreDRP Standard Plots

- As mentioned on previous slide, the GoreDRP standard is a very stable sample with nearly no signs of aging over the measurement period
- Error = standard deviation of repeated measurements on one sample

R/T at 400nm of GoreDRP Standard vs Time

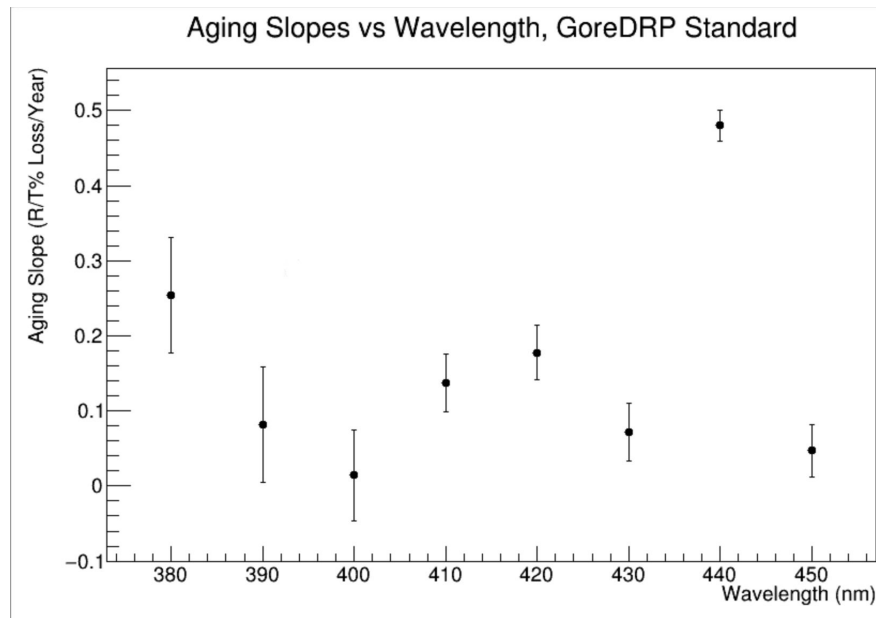


R/T at 450nm of GoreDRP Standard vs Time



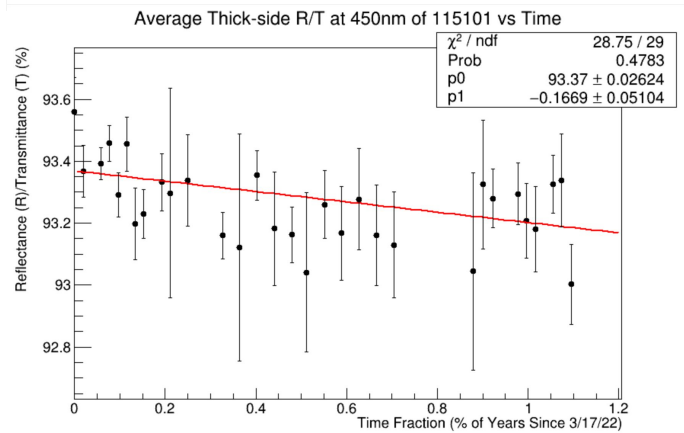
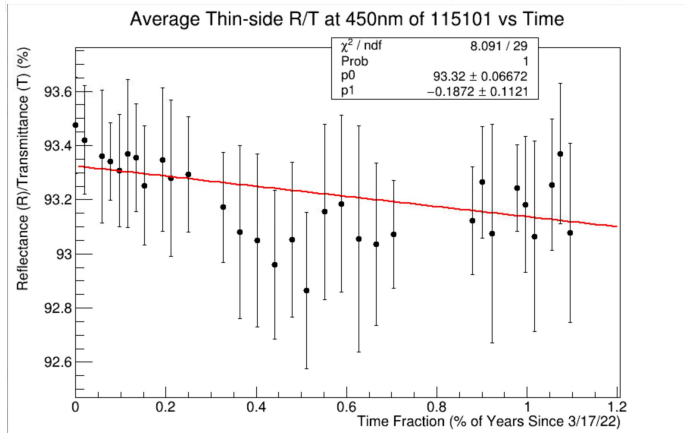
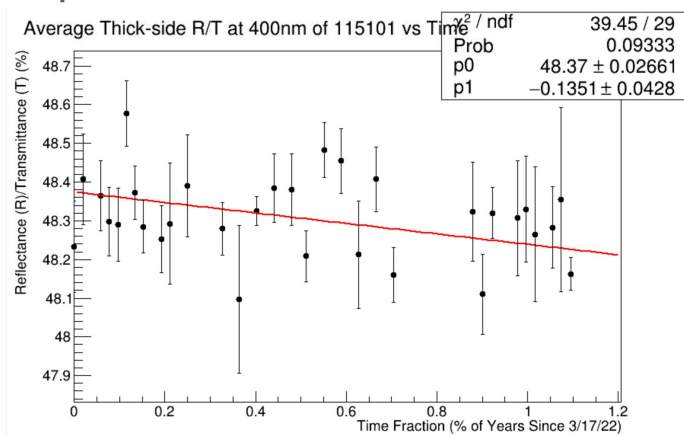
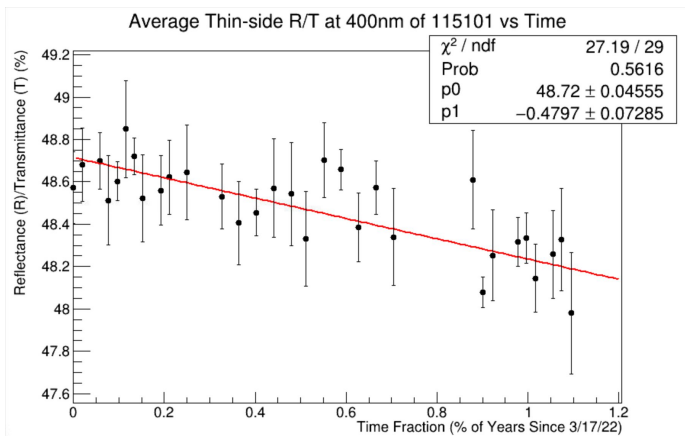
# GoreDRP Summary by Wavelength

- Most wavelengths have aging slope of 0.1%
- Besides  $\lambda = 440\text{nm}$ , not much structure in different wavelengths
- Error here is ROOT error on the slope parameter from previous linear fits



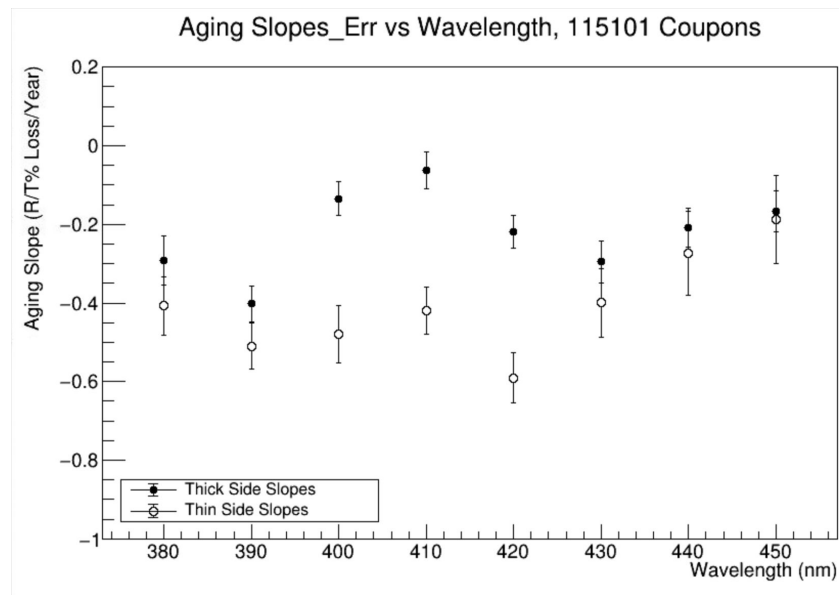
# TiO2 Coupon Plots

- Error = standard deviation of measurements on the 4 different coupons



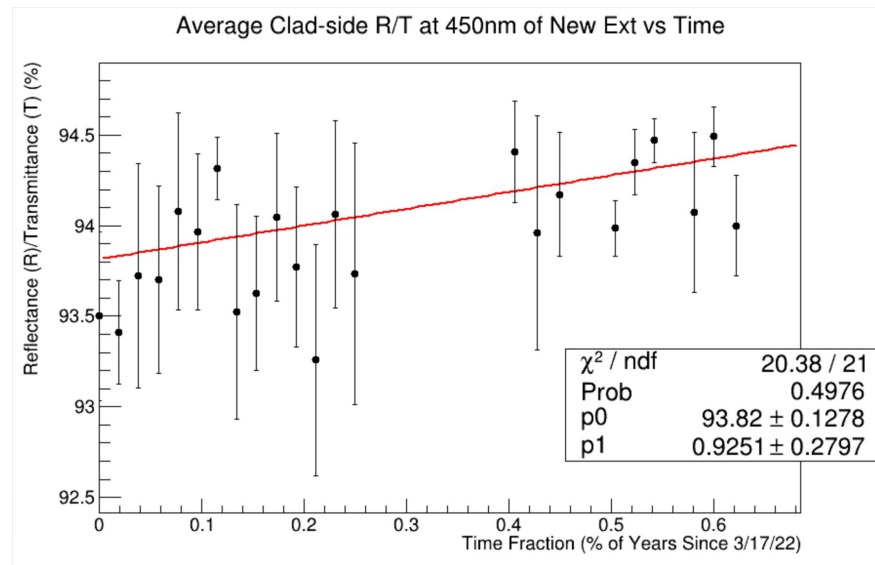
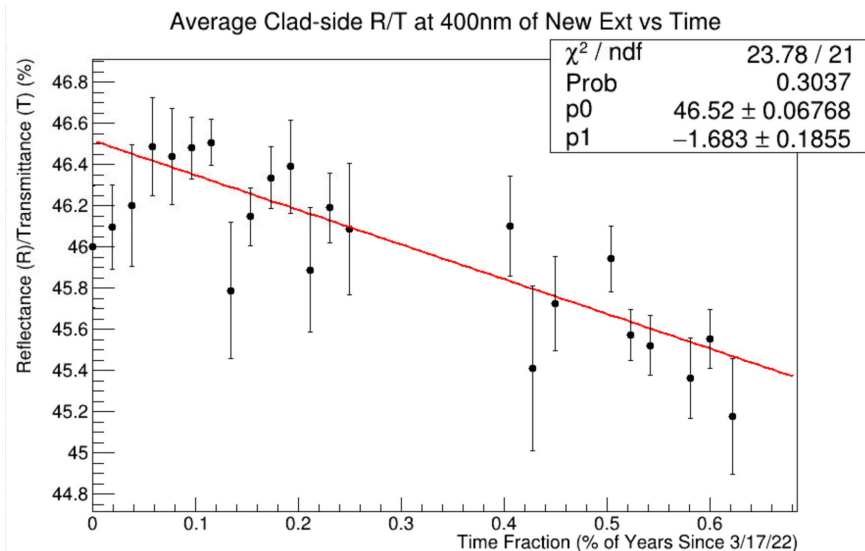
# TiO2 Summary by Wavelength

- Thin side = open circles
- Thick side = filled circles
- Some structure wrt wavelength here? Samples seem to have a peak around  $\lambda = 410\text{nm}$ , dip, then rise together as  $\lambda = 450\text{nm}$
- Slopes do indicate reasonable direction of aging, small negative slopes



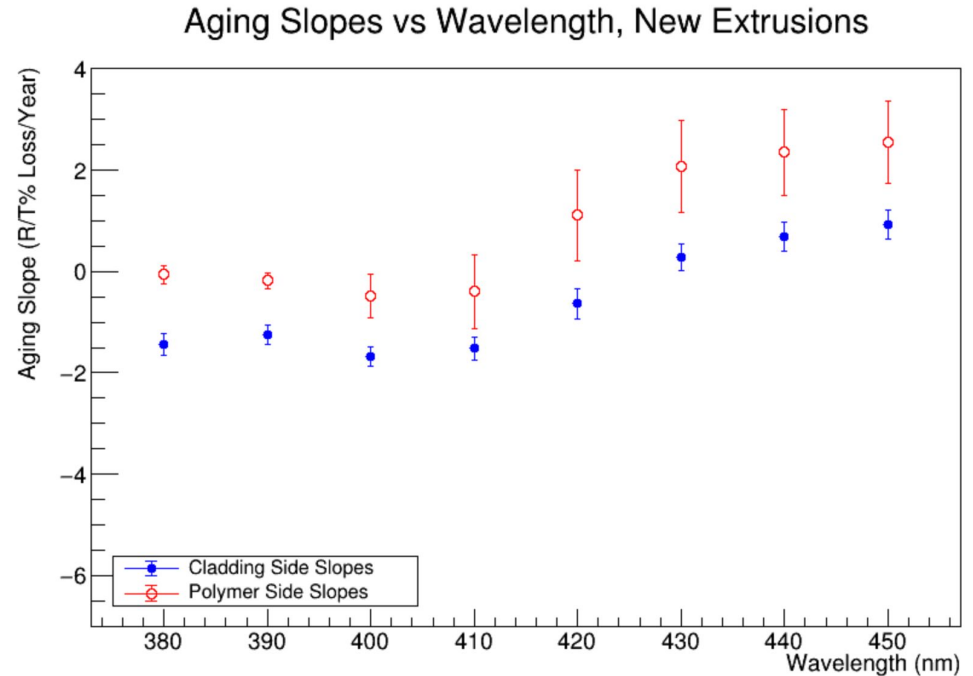
# Extrusion Plots

- Error = standard deviation of 5 sample measurements



# Extrusion Summary by Wavelength

- Focus on blue points for the cladding side
- Similar structure wrt wavelength as the TiO<sub>2</sub> coupon plot, increasing as wavelength increases



# Stability of Reflectometer?

- While discussing the results from these aging plots, the stability of the HunterLabs reflectometer instrument has come under scrutiny
- If the instrument is truly stable, why do we see positive aging slopes?
- Solution/check:
  - Measure reflectivity once an hour for an entire day, take standard deviation
  - Measure reflectivity once a day for an entire week, take standard deviation



# Brian: Stability of Reflectometer

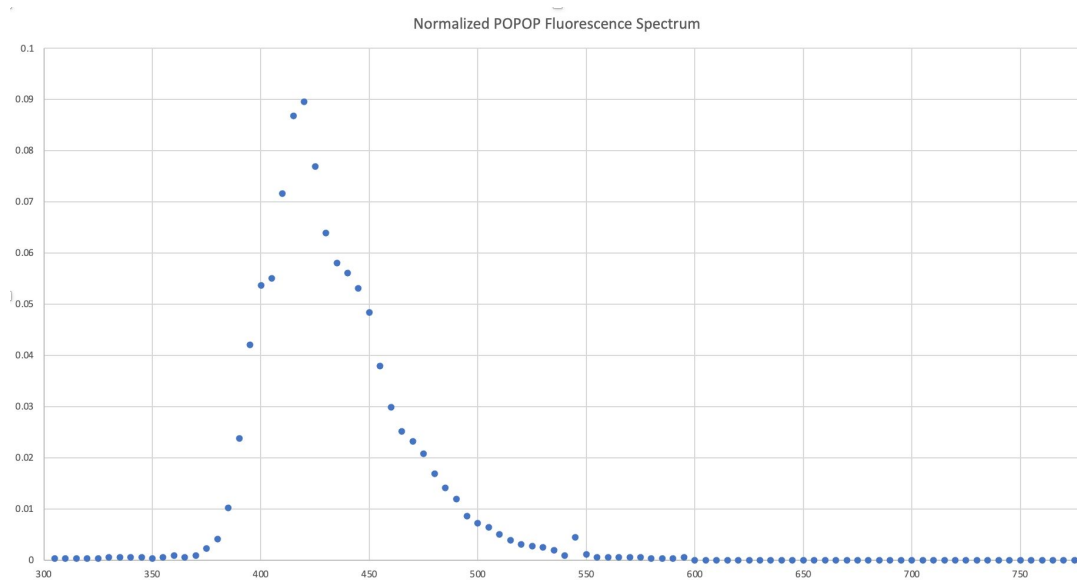
- Results after repeated GoreDRP measurements:
- Standard deviation of repeated daily measurements:  $\pm 0.21\%$
- Standard deviation of repeated weekly measurements:  $\pm 0.07\%$
- As a result, I added a constant error of 0.1% onto the next set of plots...

# Can we define a metric to decouple wavelength-dependent effects?

- A big unknown in the coupon/extrusion cladding aging problem is whether or not aging is constant among different wavelengths
- My data points to no, I see structure in the wavelength domain
- If we define a metric with respect to POPOP fluorescence at each wavelength, can we effectively 'weight' different wavelengths to minimize this effect?

# POPOP Fluorescence

- Data from Alan
- Measured wrt wavelength using a fluorimeter
- Normalized so area under the curve = 1, previously in arbitrary units

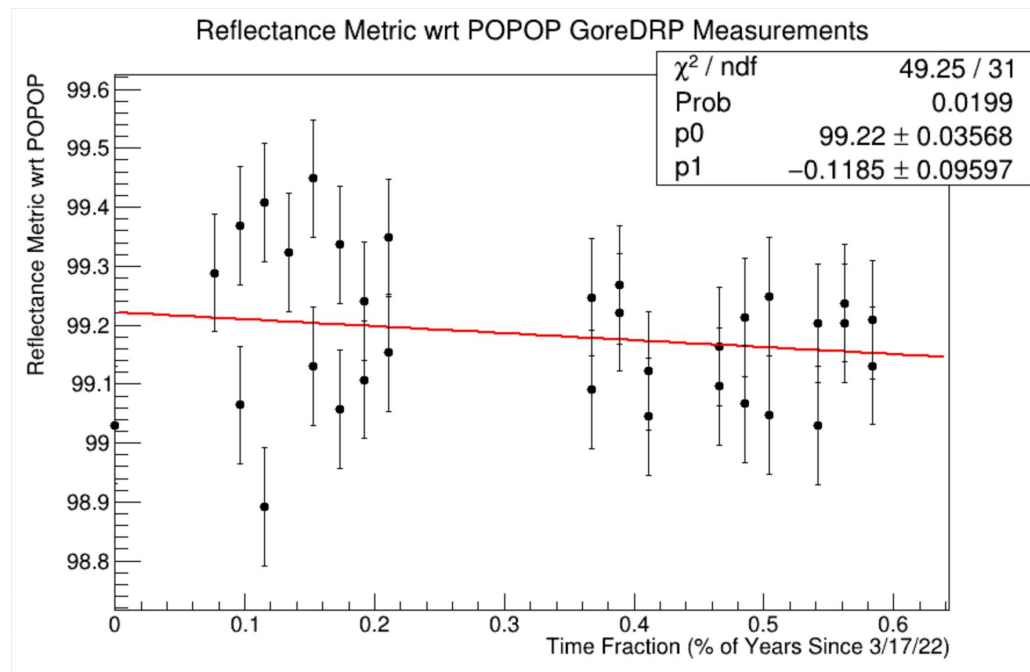


# Reflectance Metric Definition

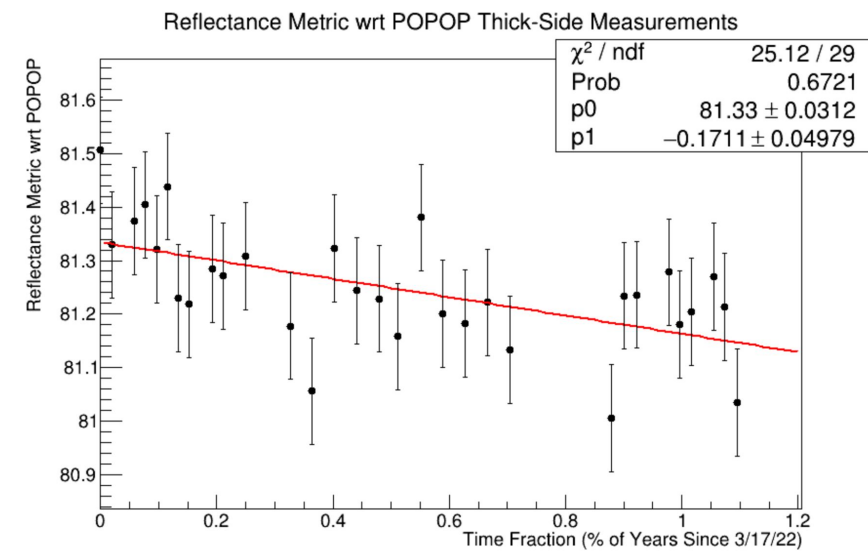
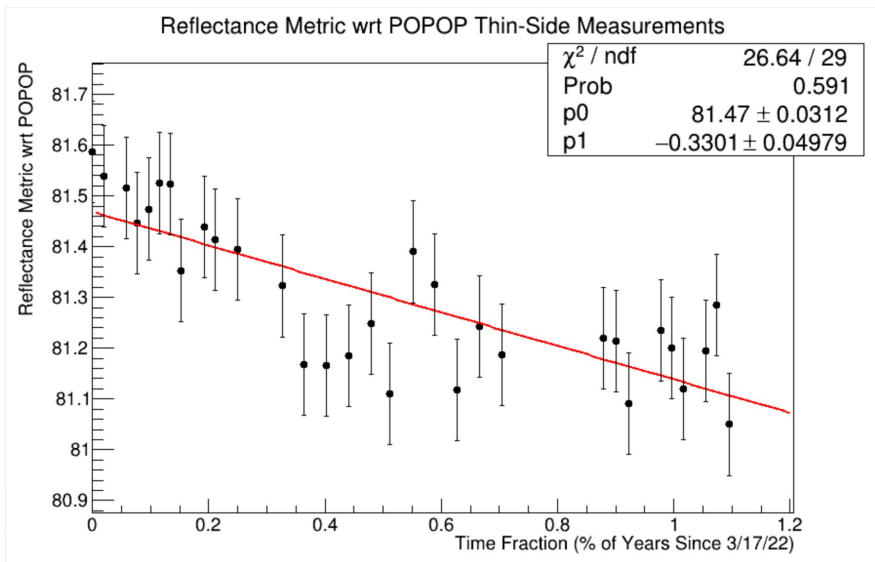
- $R_{\text{metric}} = \sum(\text{over } \lambda)(R_{\text{average}}(\lambda) * \text{POPOP}_{\text{norm}}(\lambda))$
- $R_{\text{metric}}$  is then some number between 0 and 100.... If  $R_{\text{average}}$  was always 100%, then  $R_{\text{metric}} = 100\%$
- In this way,  $\lambda$ s with no POPOP fluorescence are not weighted into sum

# Reflectance Metric Plot - GoreDRP Standard

- Time actually starts 9/20/22
- I tried applying both the daily error and weekly error separately, 0.07% or 0.2%, but since both are constant error, it made no difference on the fit slope. Used 0.1%

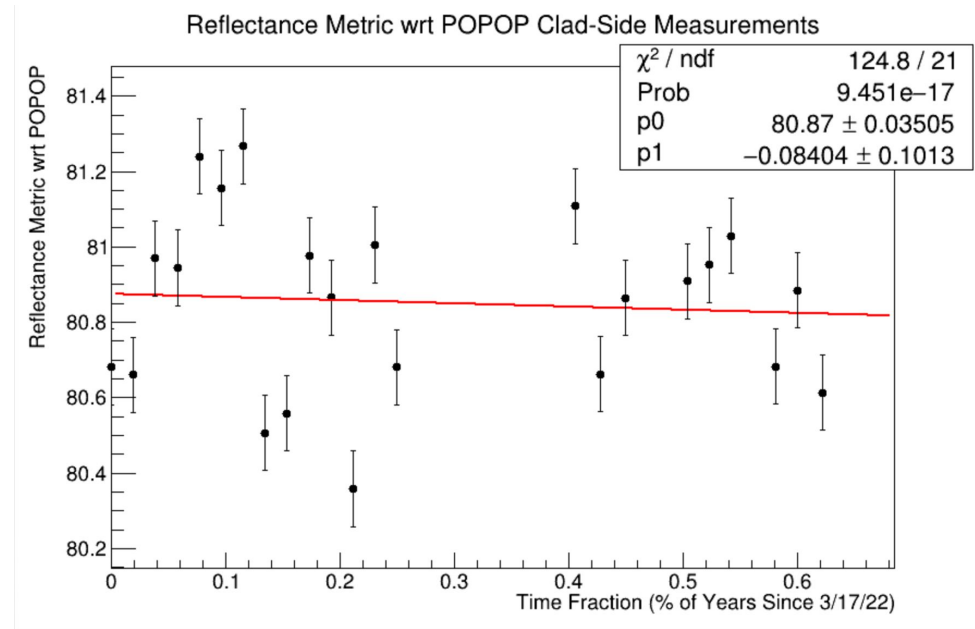


# Reflectance Metric Plot - TiO2 Coupons



# Reflectance Metric Plot - Extrusion Cladding Side

- Time actually starts 9/6/22



# Reflectance Metric Summary and Comparison

- Do sign of R metric slopes agree with average aging rate of different samples?
- TiO<sub>2</sub> coupons:
  - Thin-side:           aging =  $-0.41\% \pm 0.12\%$        R =  $-0.33\% \pm 0.05\%$
  - Thick-side:         aging =  $-0.22\% \pm 0.10\%$        R =  $-0.17\% \pm 0.05\%$
- Extrusion samples:
  - Cladding side:   aging =  $-0.58\% \pm 0.99\%$        R =  $-0.08\% \pm 0.10\%$
- Standards:
  - GoreDRP:         aging =  $+0.16\% \pm 0.14\%$        R =  $-0.12\% \pm 0.10\%$



# Conclusions

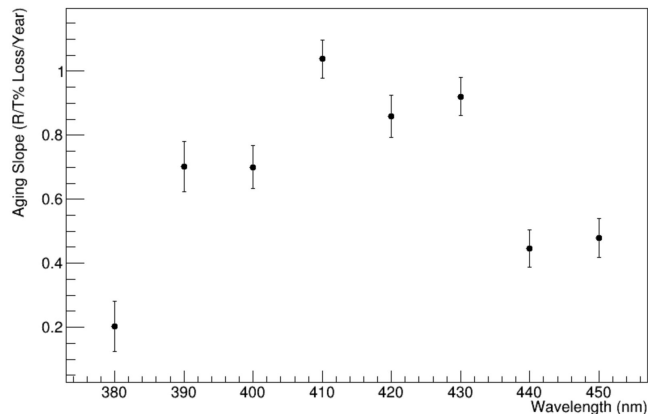
- GoreDRP is a stable standard, the stability of this sample over time proves that the HunterLabs Reflectometer instrument is reliable and has produced quality data for coupons and extrusions
- Coupons and extrusion samples both show very small aging slopes, somewhere around 0.5% or less
- If we put this small aging for cladding reflectivity into MC, does this have a significant impact on the overall light yield of the CRV?

# Backup

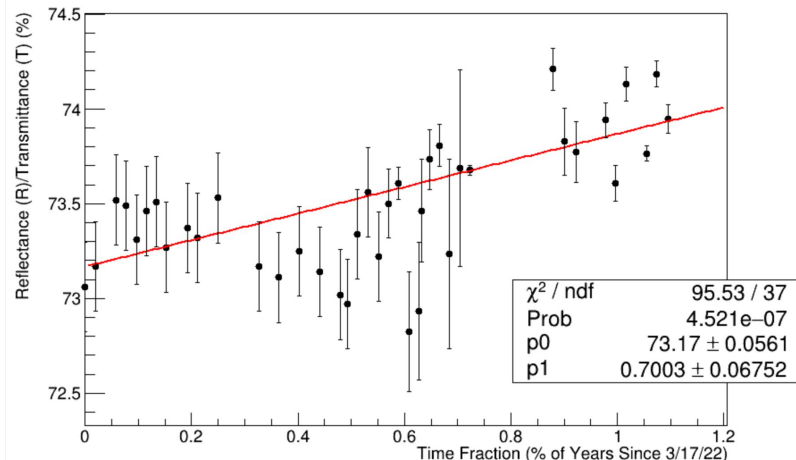
# Disqualified NOvA Standard

Large variation in aging plot slopes, results pointing to unphysical “positive” aging

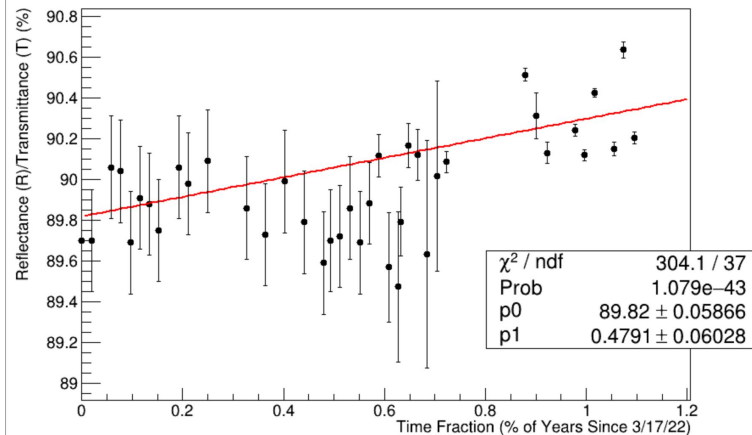
Aging Slopes vs Wavelength, NOvA Standard N-27-09-NC



R/T at 400nm of NOvA Standard vs Time



R/T at 450nm of NOvA Standard vs Time



# Disqualified Polymer-side Extrusions

