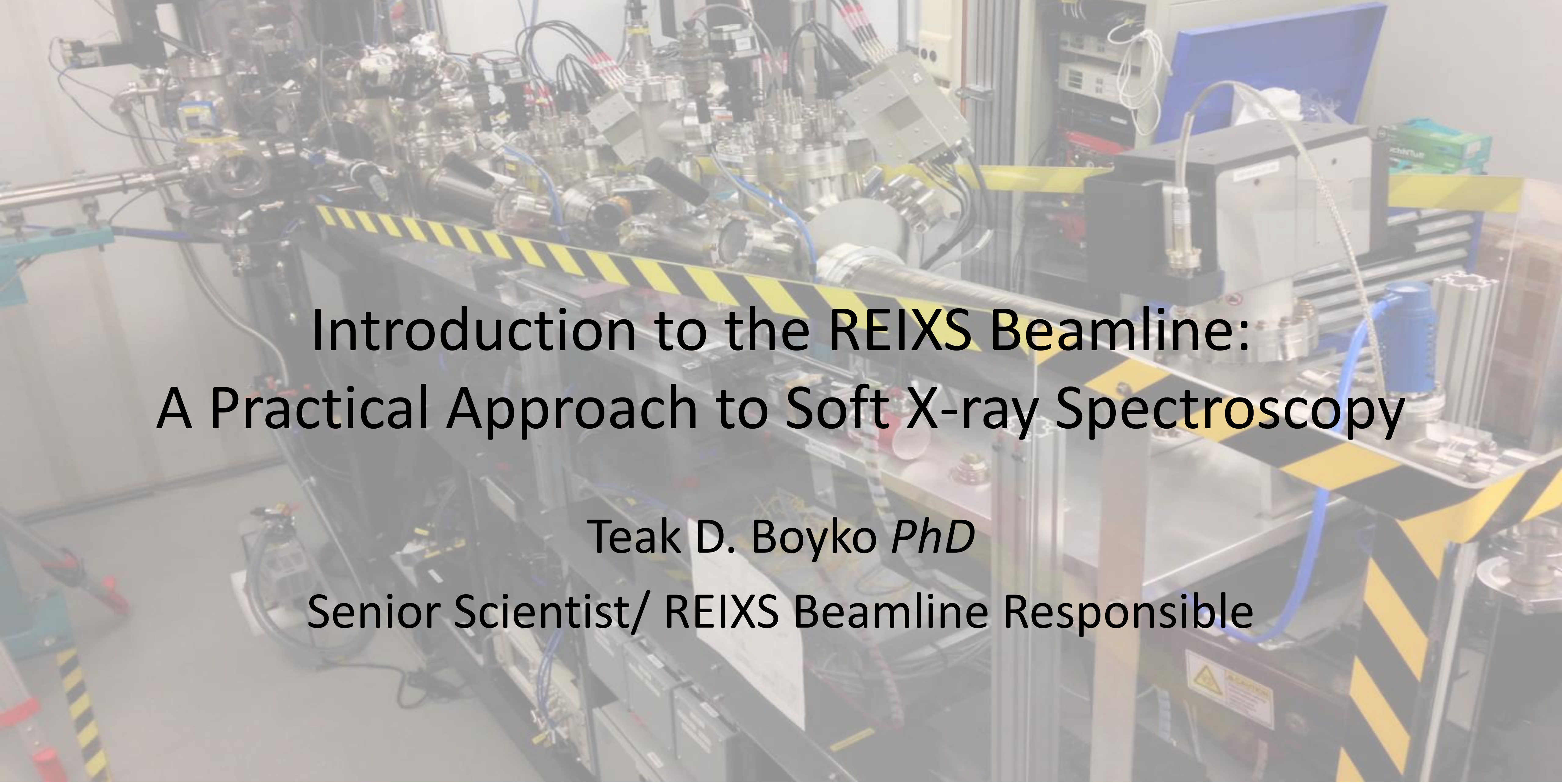

Introduction to the REIXS Beamline:
A Practical Approach to Soft X-ray Spectroscopy

Teak D. Boyko *PhD*

Senior Scientist/ REIXS Beamline Responsible

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Acknowledgements

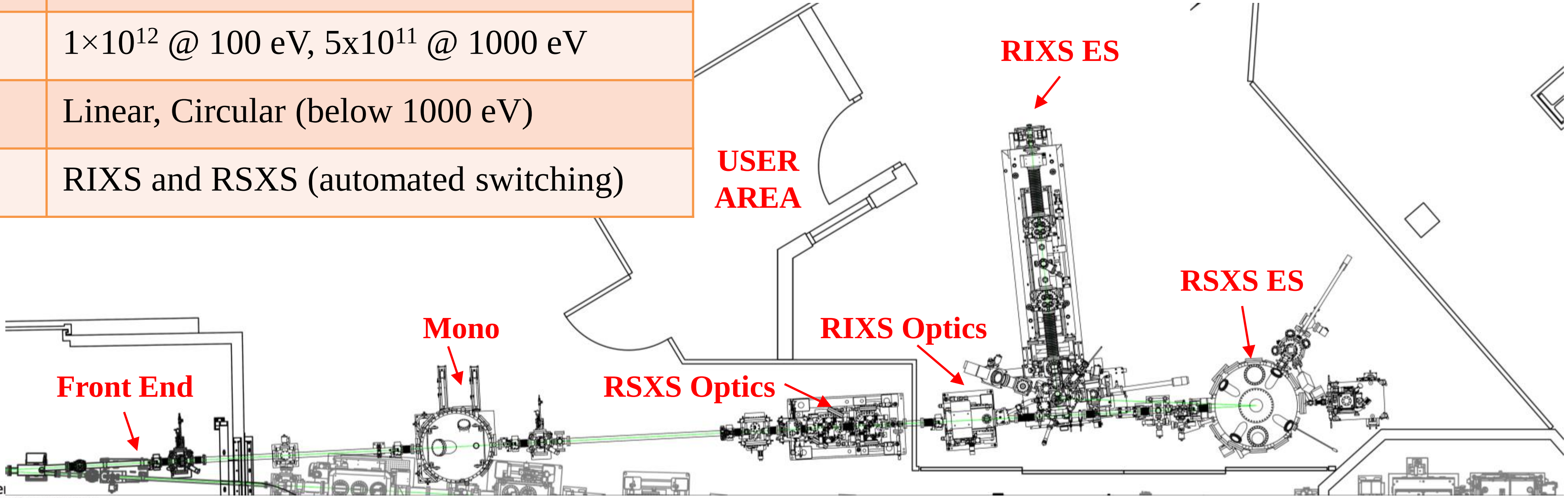
- **Beamline Staff**
  - Feizhou He (Manager, Senior Scientist)
  - Ronny Sutarto (RSXS Specialist, Scientist)
- **Beam Team (RIXS Endstation)**
  - Alexander Moewes
    - Patrick Braun
- **Facility Technical Support**

Outline

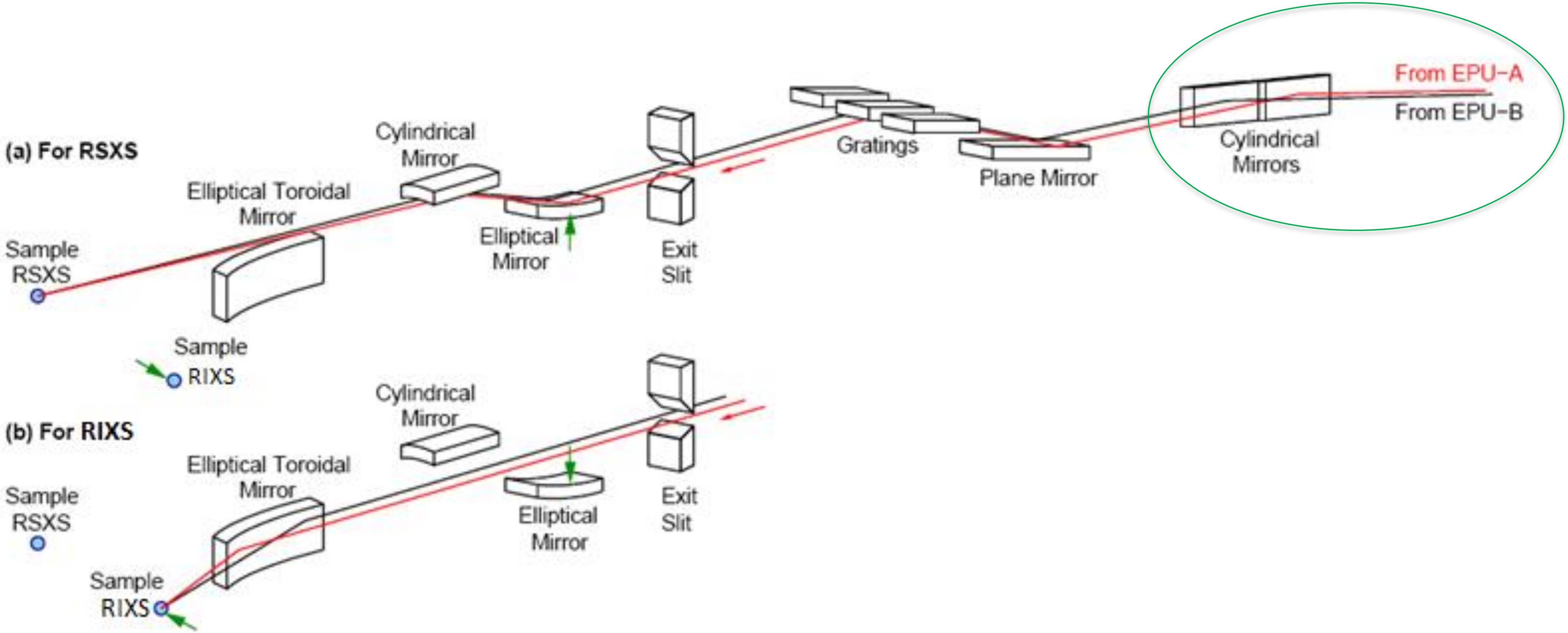
- **What are the best setups for the EPU and Monochromator?**
  - Polarization and harmonics
  - Mirror coatings and gratings
- **How are energy resolution and beam size correlated?**
  - Exit slit gap
  - Attenuating the beam
- **What samples can we measure?**
  - Beam size on the sample
  - Sample form factors
  - Cooling
  - Magnetic fields
- **How to deal with multiple detectors?**
  - Current and V2F convertors
  - Silicon drift detectors
  - Grating x-ray spectrometers
- **What are some tips and tricks to get good data?**
  - Sample damage
  - Electron yield issues

Resonant Elastic and Inelastic X-ray Scattering Beamline (REIXS) Layout

Source	APPLE II type EPU
Monochromator	VLS-PGM - 3 gratings (Au LEG, Au HEG, Ni LEG) - 4 mirror coatings (C, Au, Ni, Si)
Energy Range	95 – 2000 eV (2500 eV for diffraction)
Resolution	5×10^{-5} @ 100 eV, 1.3×10^{-4} @ 1000 eV
Photon Flux	1×10^{12} @ 100 eV, 5×10^{11} @ 1000 eV
Polarization	Linear, Circular (below 1000 eV)
Endstations	RIXS and RSXS (automated switching)



Where to start? EPU



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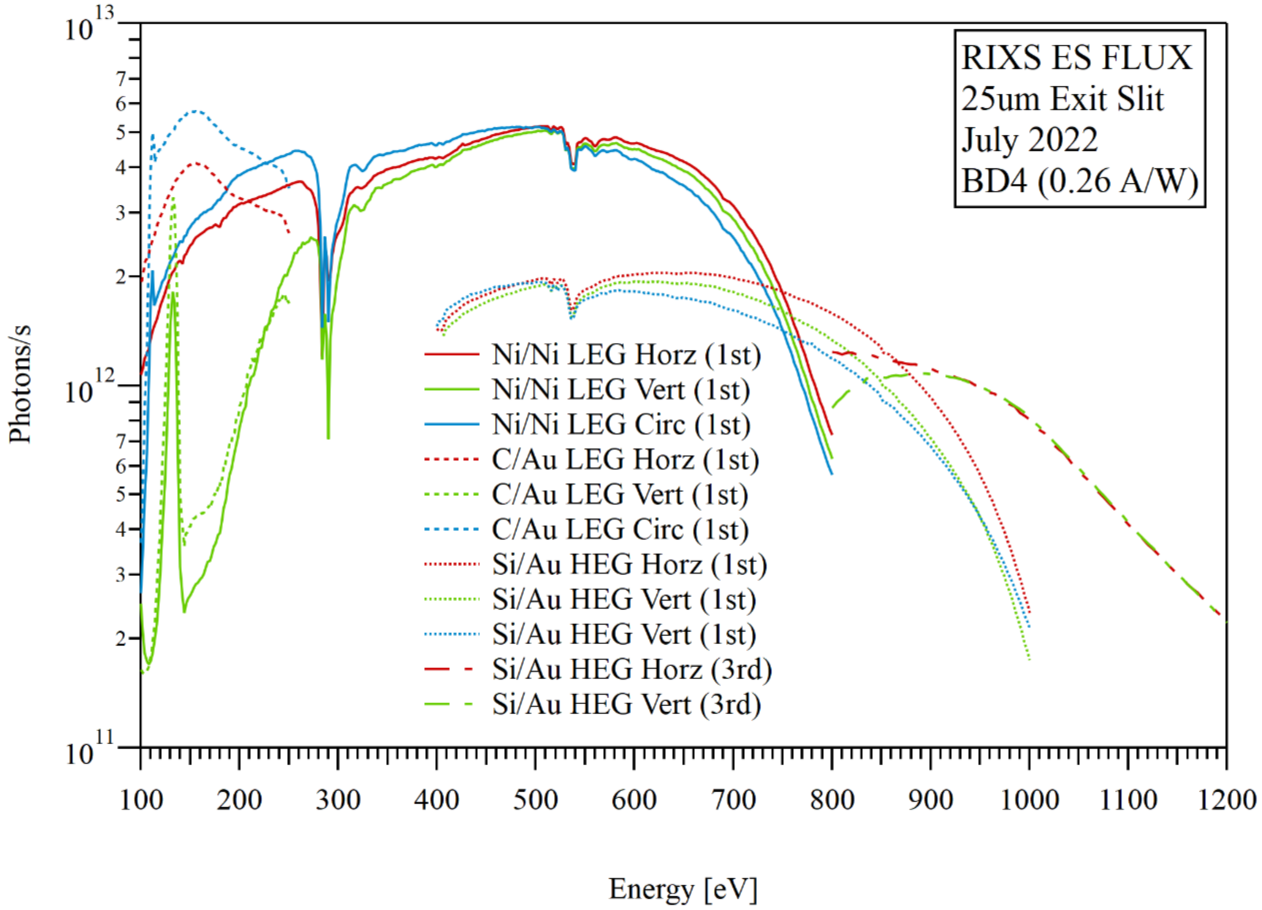
How to choose polarization and harmonics?

- Polarizations Available
  - **Horizontal**
    - Full energy range
  - **Vertical +/-**
    - Somewhat full range, but limited at minimum gap
  - **Inclined**
    - Polarization from Vert+ to Vert-, limited at minimum gap
  - **Circular left**
    - Limited energy range
- Harmonics
  - Extends the energy working range.
  - Gap becomes too large: weak field
  - How important is adding a gap offset?
    - Typically no gap offset needed since width becomes larger at higher energies.



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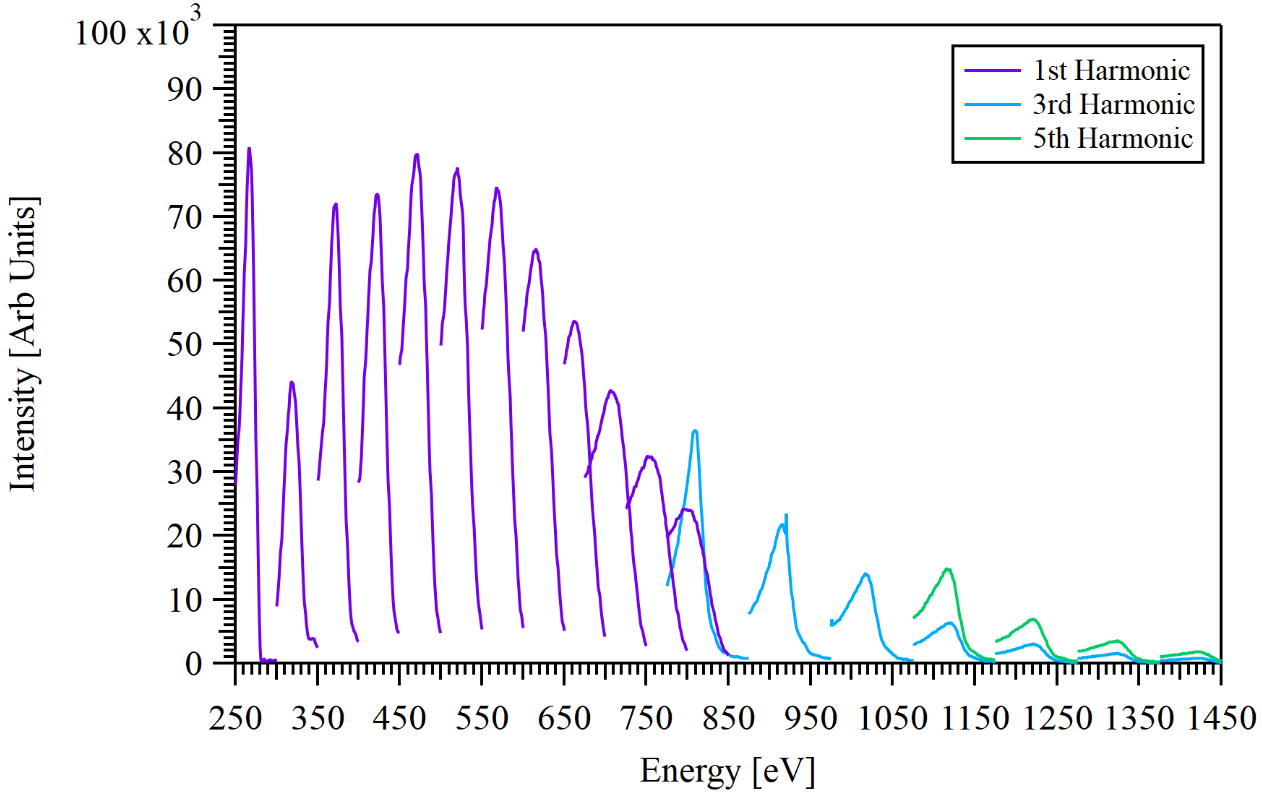
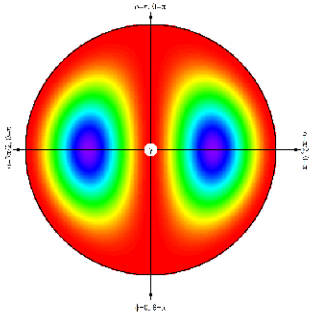


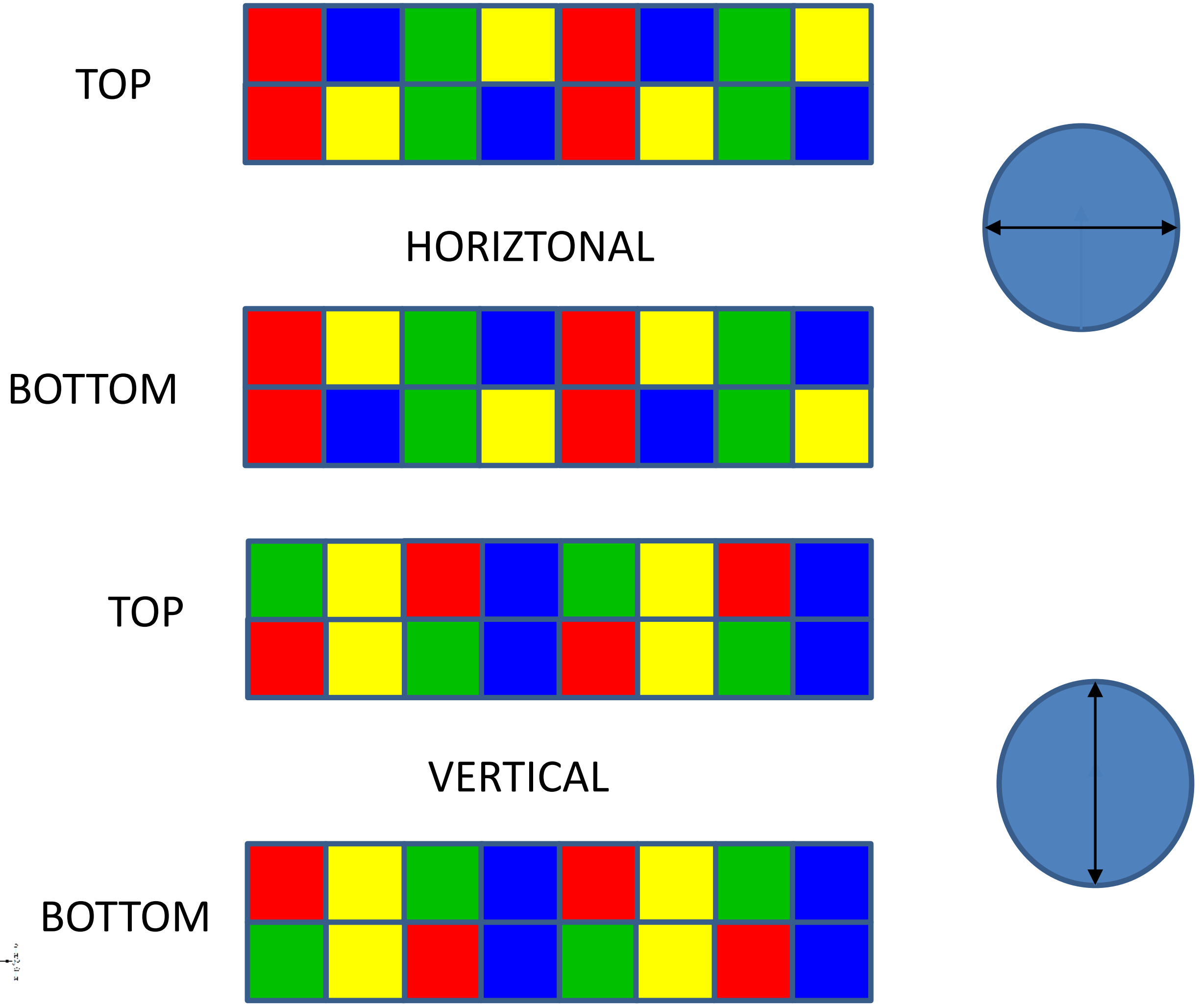
Measured with charge scattering on SDD
Not precisely calibrated, qualitative only!

Considerations for changing/selecting polarization?

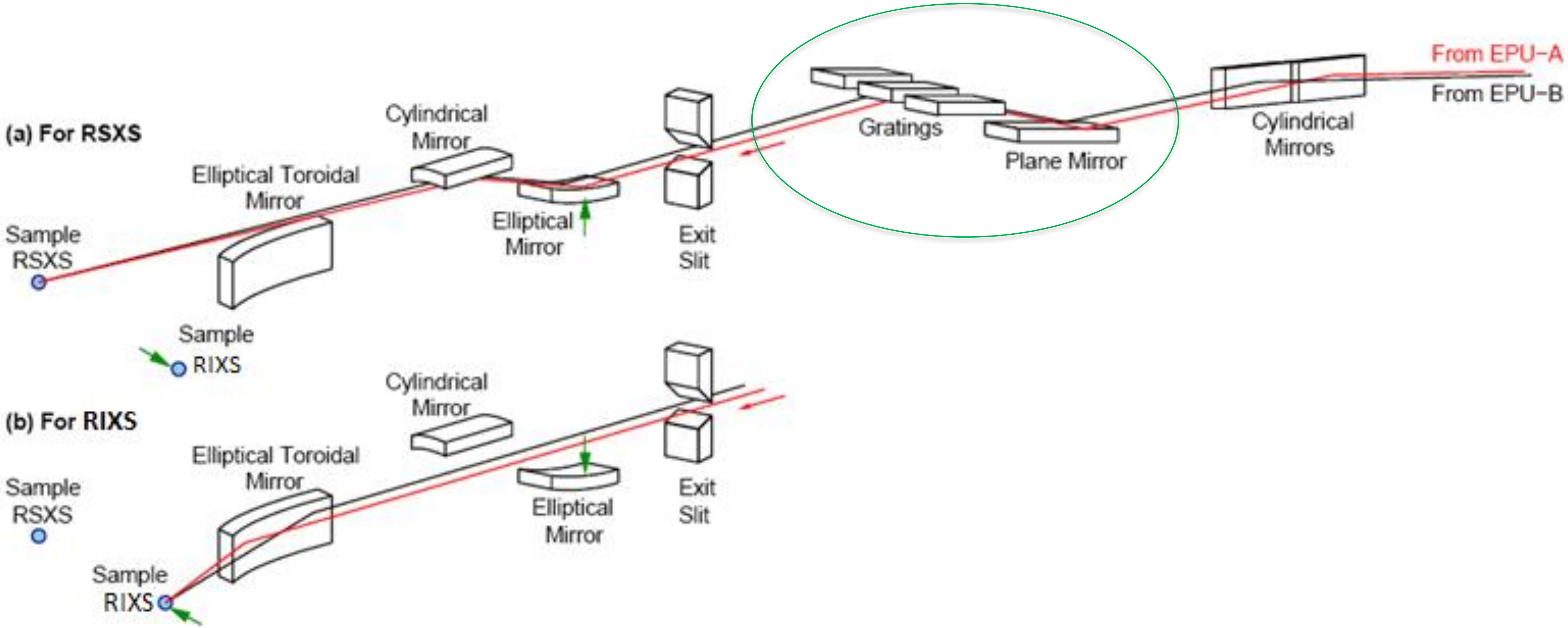
- **Horizontal**
  - Stray BM light will be horizontal
  - Generally more flux than Vert
  - Best for XRF experiments: no charge scattering.
- **Vertical +/-**
  - Gap is always smaller than Horz, gap change required when switch to Vert +/- from Horz
  - Low Vert flux can be contaminated with Horz
- **Inclined**
  - Gap and girder motion when scanning angle
- **Circular left**
  - Highest flux
  - Vertical component increases charge scattering
  - No gap change from Circ Left to Circ Right

$$\sigma_R = \pi r_e^2 \int_{-1}^{1} |f(\theta)|^2 (1 + cos^2\theta)\, d(cos\theta)$$



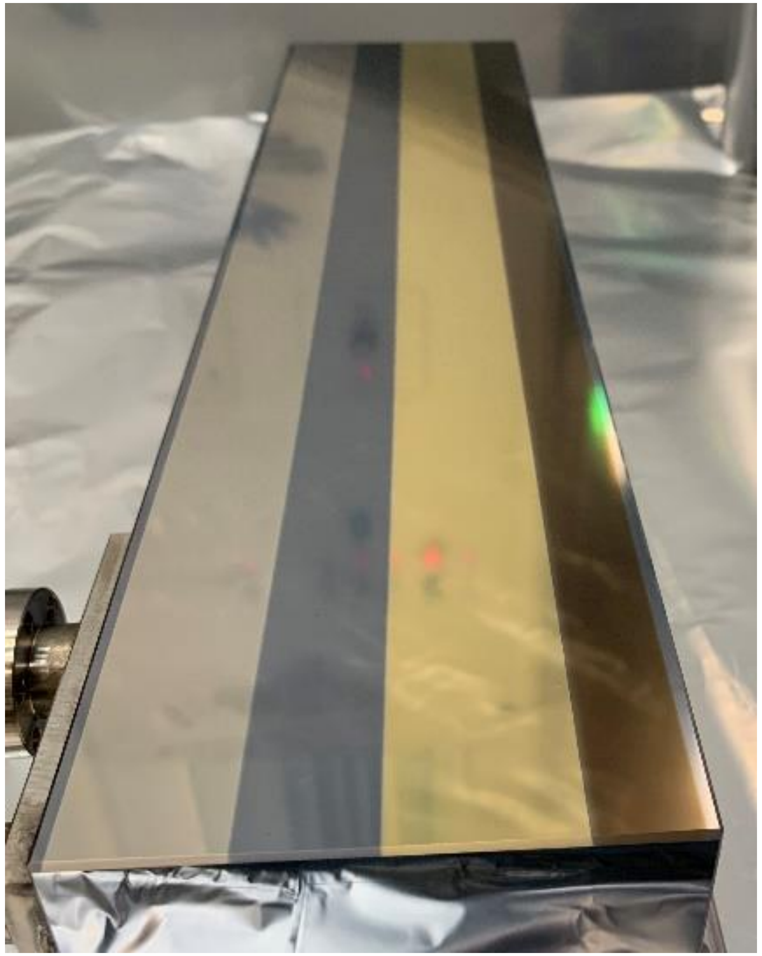


Where to next? VLS-PGM Monochromator



How to choose mirror stripes and gratings?

- 4 Mirror Stripes
  - Ni, Si (SiO2), Au, C
  - Reduce higher order content
  - Improve efficiency
- 3 VLS Gratings
  - Au LEG: < 250 eV
  - Ni LEG: 250 – 750 eV
  - Au HEG: > 750 eV
- VLS-PGM Focus
  - Fixed on exit, no exit slit motion.
  - Beam is horizontal in and out.



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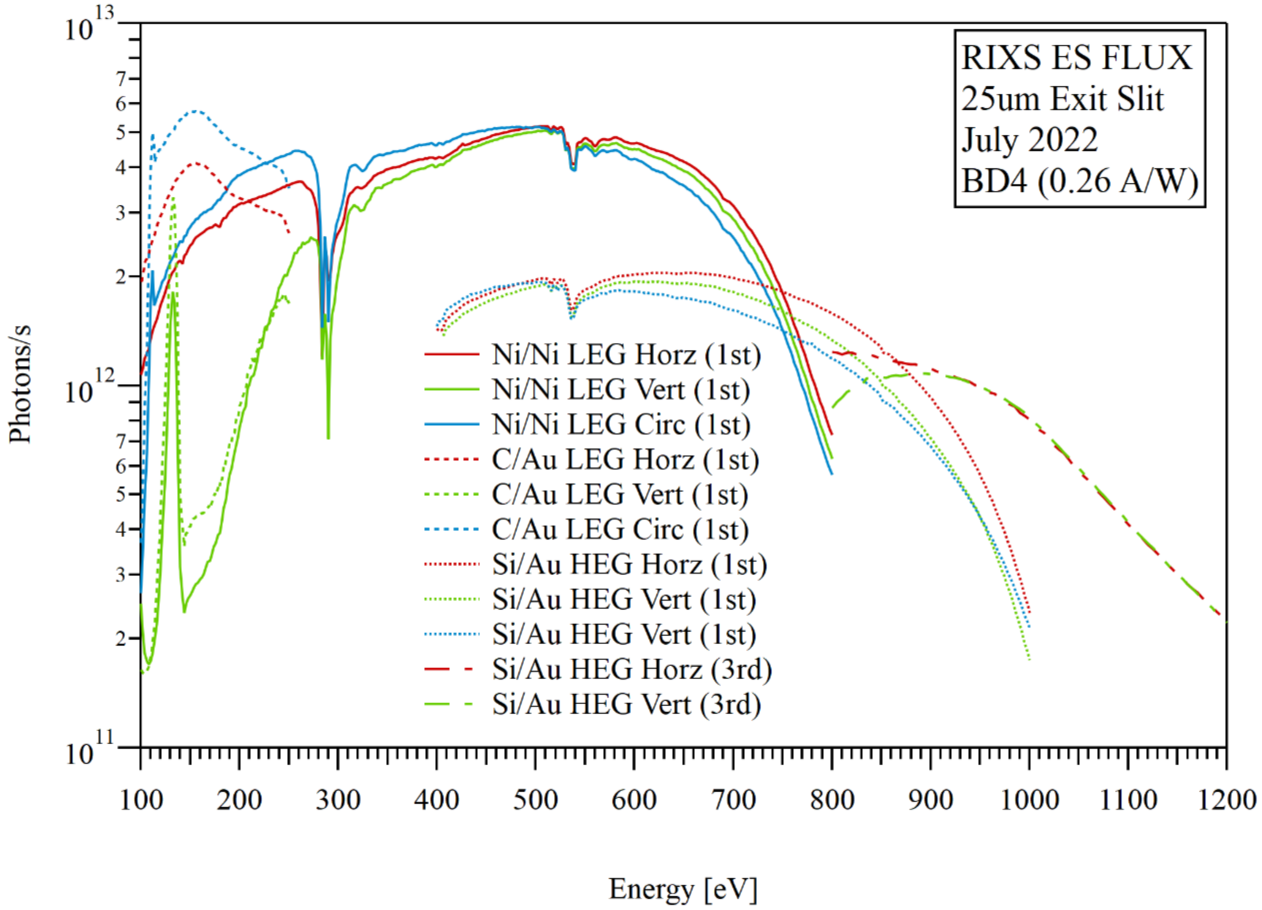

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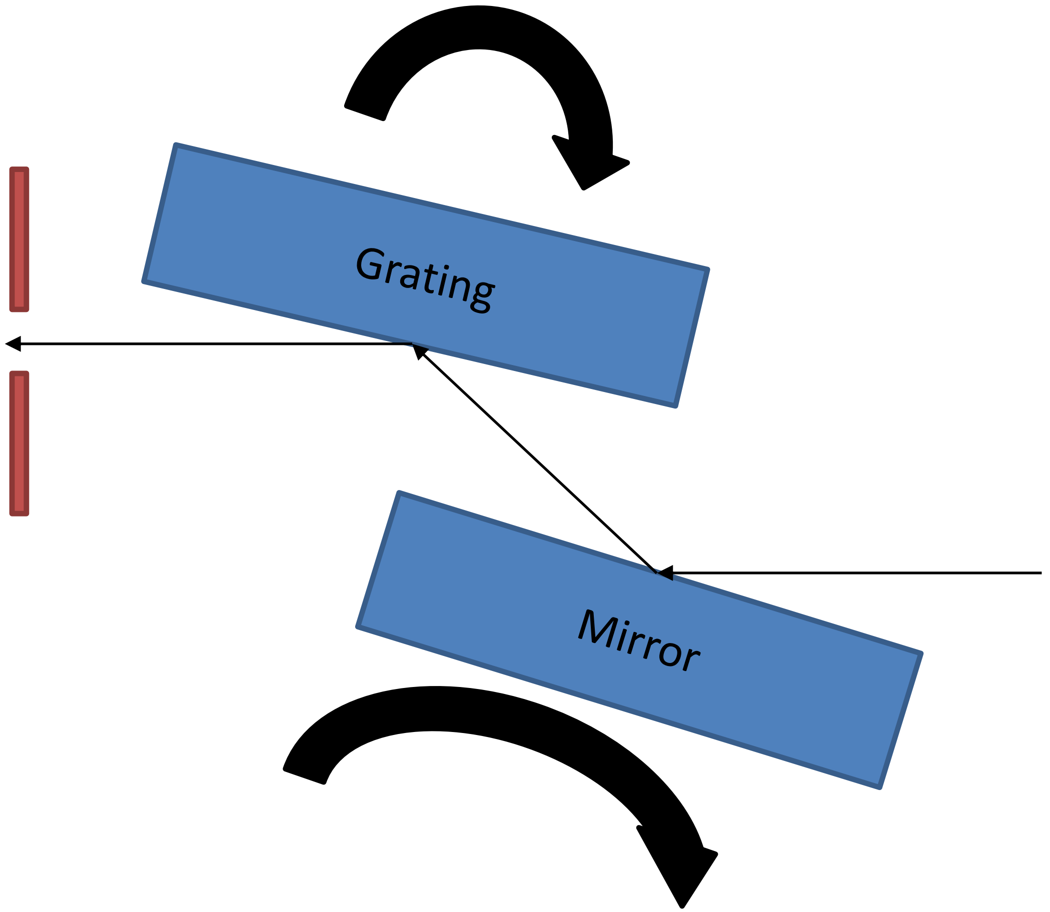
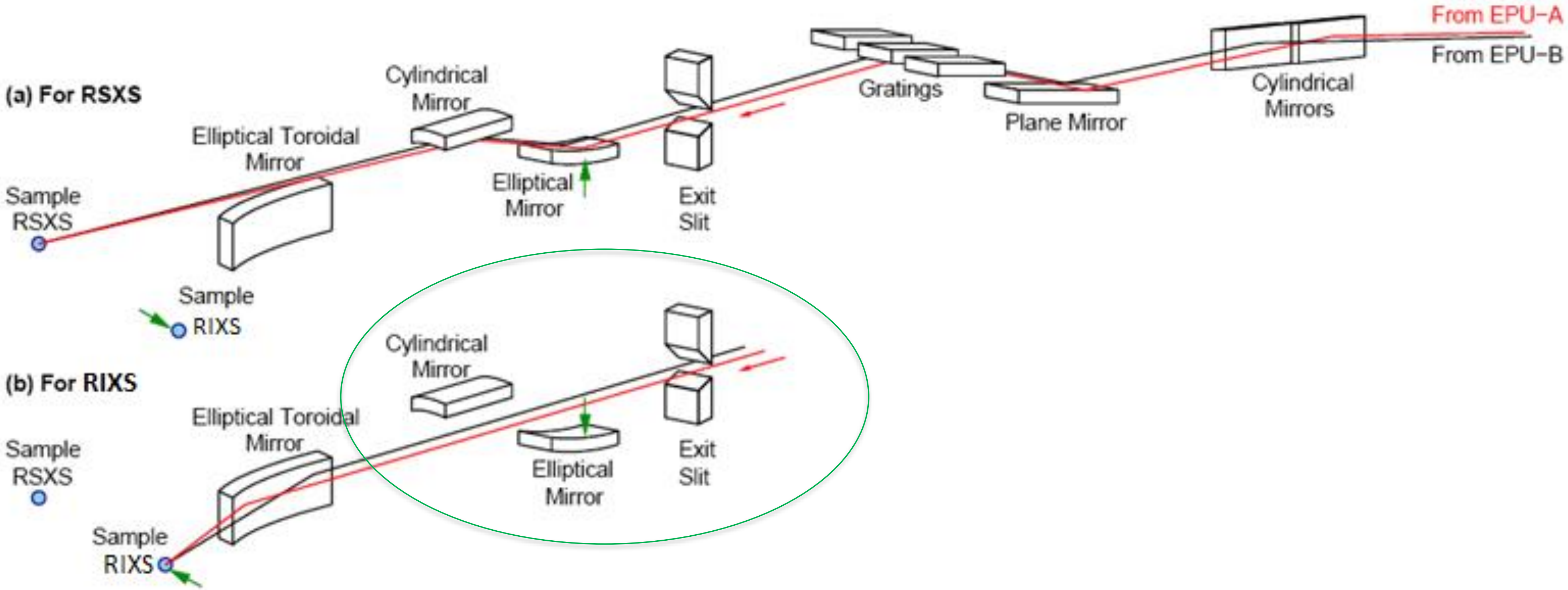
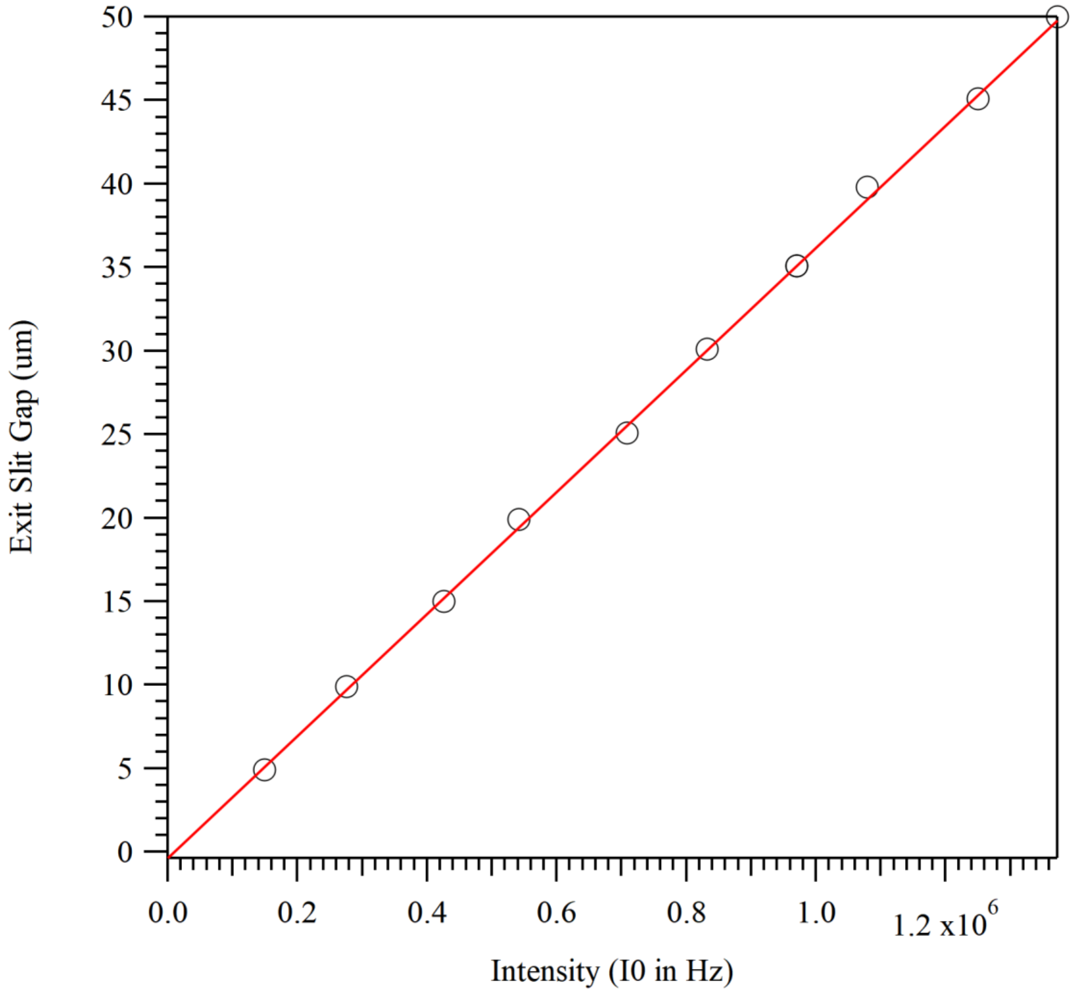


Where to next? Exit Slit and Apertures



Getting less flux from the beamline?

- Exit Slit
  - Historically used to attenuate the x-ray beam
  - Changes beam size and incoming energy resolution
  - Calibrated to be linear in intensity.
  - Typical value is 25um.
- 4 Jaw #2
  - Sits between Mono and Exit Slit
  - Previously unused
    - Calibrated to precisely attenuate the x-ray beam
    - 10 – 100% of nominal flux
  - Exit Slit would need to be 2.5um to attenuate 90%.
- Exit can be opened to 100 um
  - Beam damage increases
  - Resolution of incoming energy degrades



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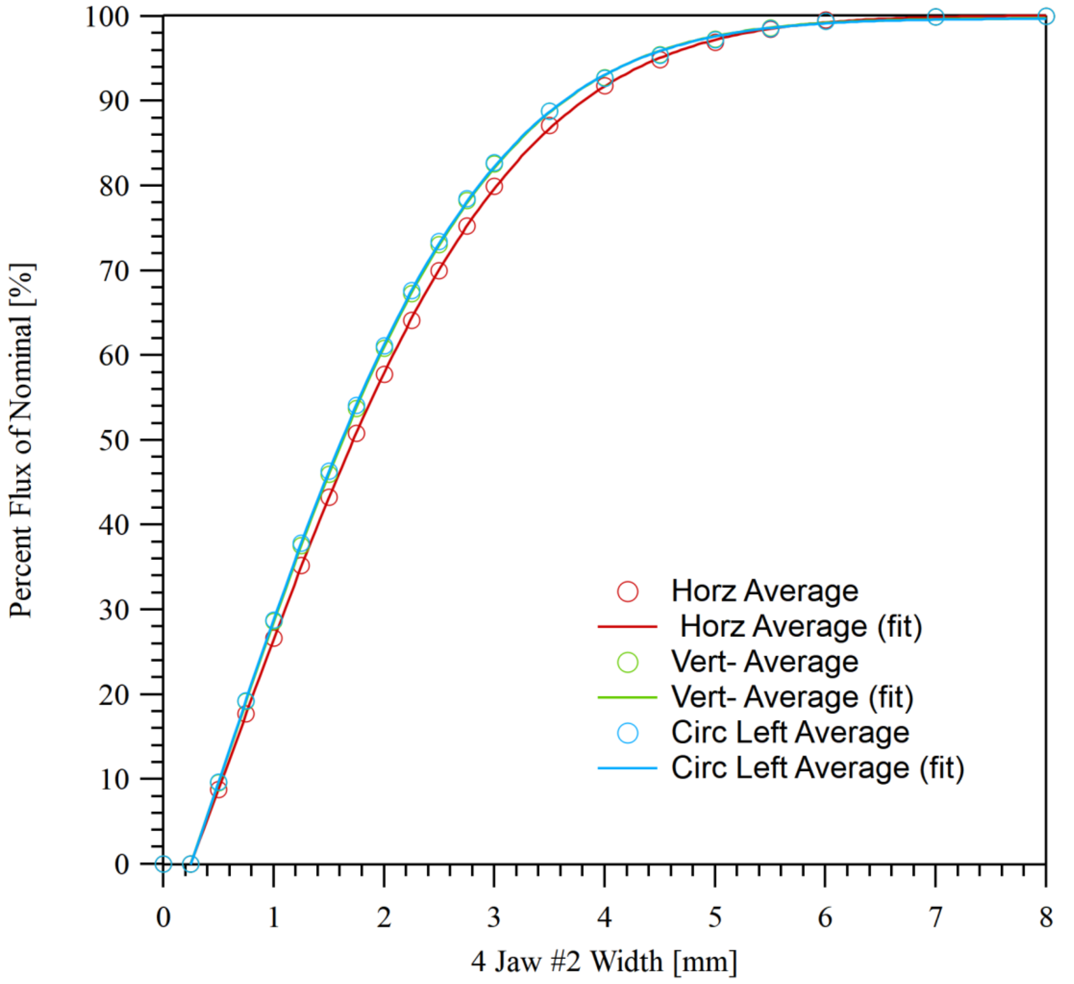
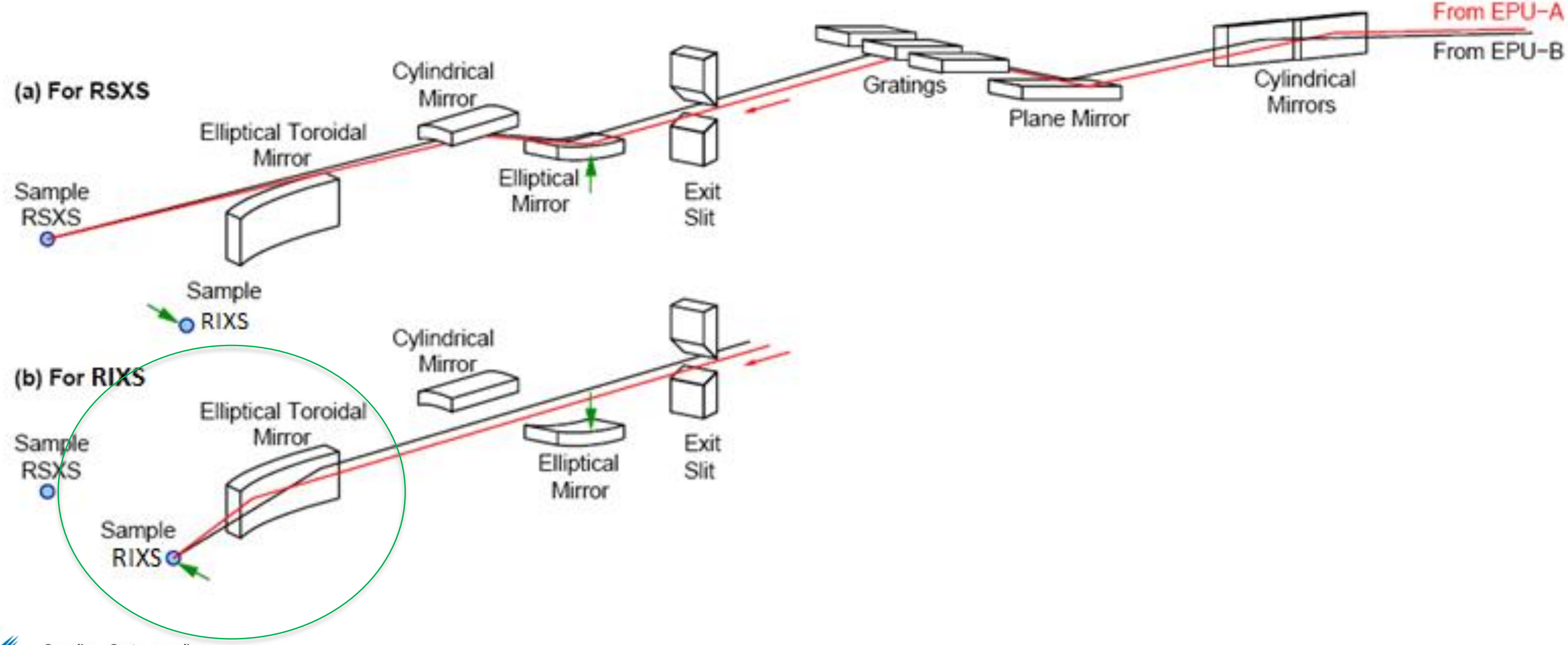


Finally, onto the RIXS Endstation



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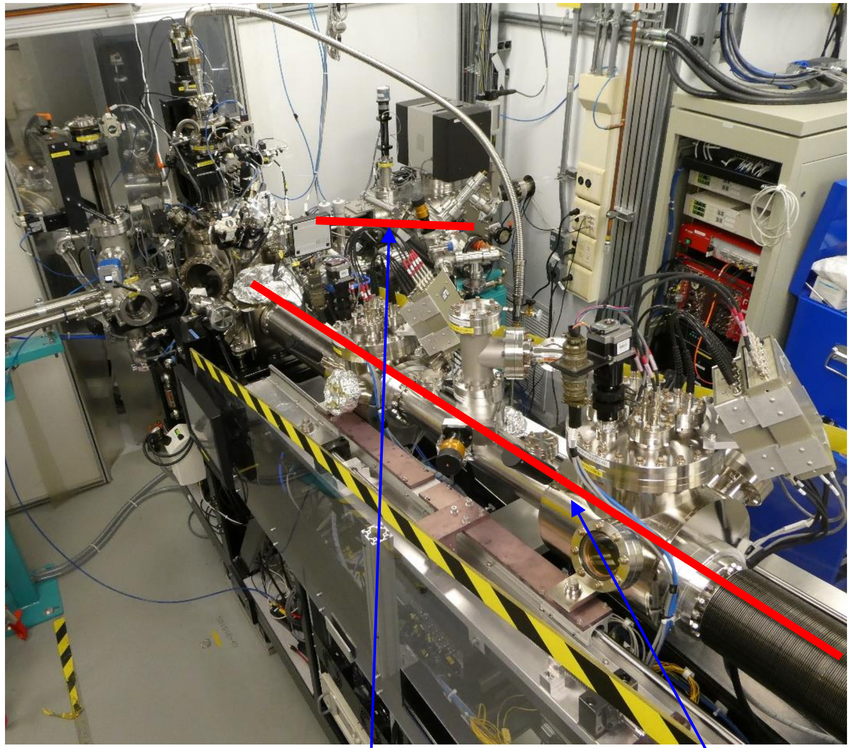
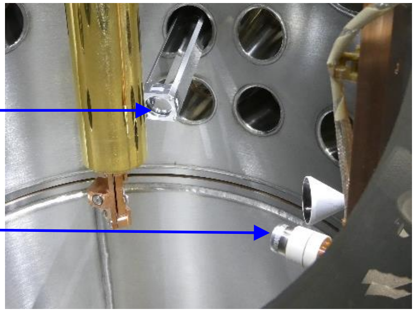
RIXS Endstation (Spectroscopy)

Vacuum	≈ 1 × 10^{-9} Torr
Sample Stage	XYZ Stage w/ Theta Rotation
Sample Temperature	30 – 420 K
Detectors	- Primary Silicon Drift Detector (SDD): 250 – 2500 eV - Secondary SDD: 250 – 2500 eV (Planning) - Rowland Circle Grating Spectrometer: 60 – 1000 eV - VLS Grating Spectrometer: 70 – 600 eV (Construction) - Optical Spectrometer: 190 – 1100 nm
Sample Environment	- Static Magnetic Field
Techniques	- X-ray Emission Spectroscopy (XES) - X-ray Absorption Spectroscopy (XAS) - X-ray Magnetic Circular Dichroism (XMCD) - X-ray Excited Optical Luminescence (XEOL)



Compact VLS Grating Spectrometer
(Under Construction)

Rowland Circle Grating Spectrometer
(Main High Resolution Spectrometer)



Optical Spectrometer Feedthrough

Silicon Drift Detector

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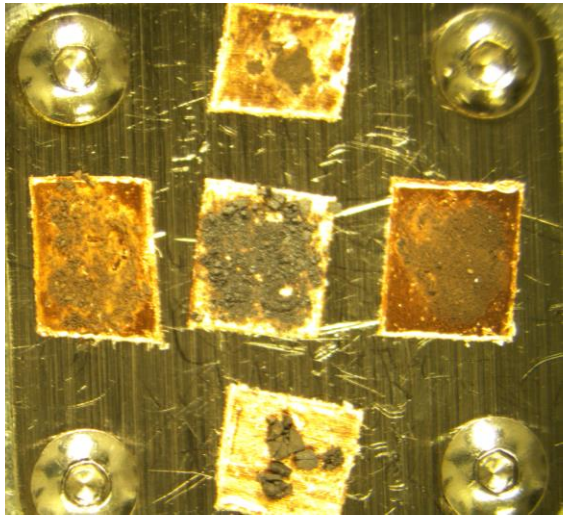
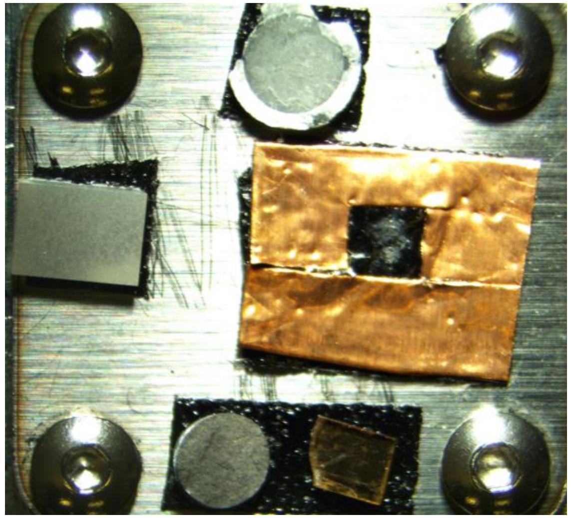
What kind of samples can you measure on REIXS?

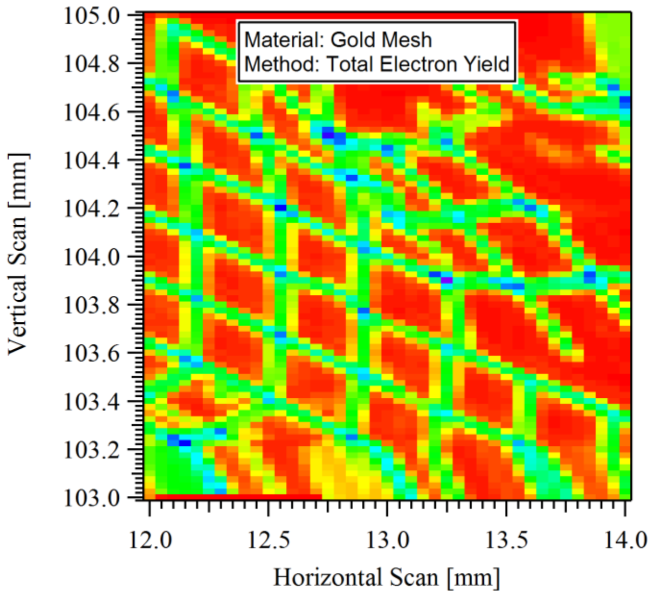
- Samples need to be UHV compatible
  - Outgassing needs to be such that vacuum is ≈5 X 10^{-09} Torr
  - Stable in vacuum
  - Not decompose under beam exposure
- Possible form factors are:
  - Powder
  - Sintered chunks or amalgamated powder
  - Thin films
  - Single crystals
- Adhering Samples
  - Carbon tape for rough samples
  - Copper tape for powders
  - Silver paint when using cryogenic cooling or heating
  - Pressing into indium foil or other malleable foils (UHV)

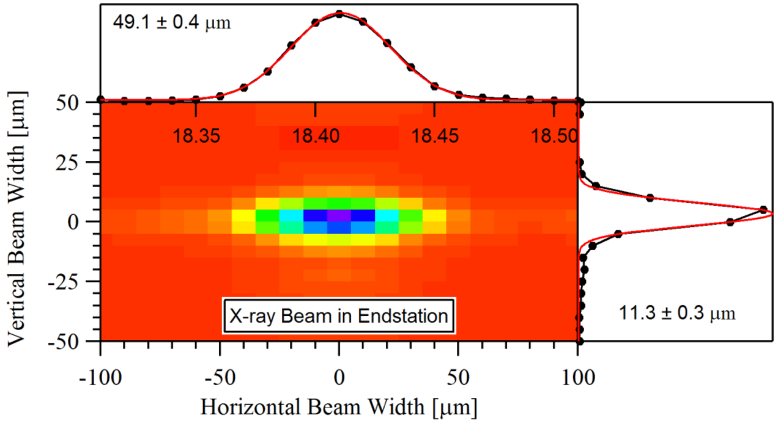




Why we need UHV in the sample chamber?

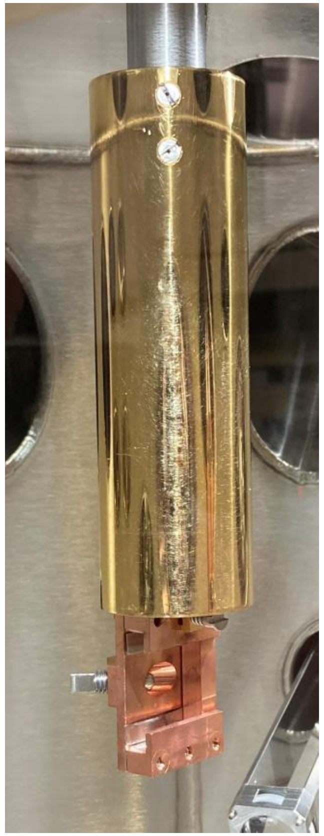
- Optics are very close to the sample chamber.
  - Last refocussing mirror, M5: 1100 mm
  - XES spectrometer gratings: 360 mm
    - Beam induced deposition of materials related to vacuum
    - Need to maintain < 1 X 10^{-09} Torr
  - We have had incidents in the past with Vanadium…
- Why are M5 and the gratings so close?
  - Small beam size: ≈ 11 µm
    - Determines overall resolution of the spectrometer
  - Stable beam height and position
    - Related beam energy stability
  - Improved solid angle
    - Best efficiency for spectrometer





Can we control the sample temperature?

- LN2/LHe Flow Cryostat
  - LN2 is freely available
  - LHe requires ordering, now $1000/day, $3000 minimum
- Practical limits
  - 30K minimum with LHe, 80K with LN2
  - 375K, 425K possible, but vacuum becomes an issue
- Sample/Operational Restrictions
  - Random fluctuations of sample position at low temperature
    - Exhaust gas temperature is not controlled
    - 0.5m long cryostat
  - Samples need to be uniform on the > 100 um length scale
  - Sample position needs realignment at every temperature
- Overnight automated operation is not guaranteed
  - Possible at temperatures above 90K for LN2



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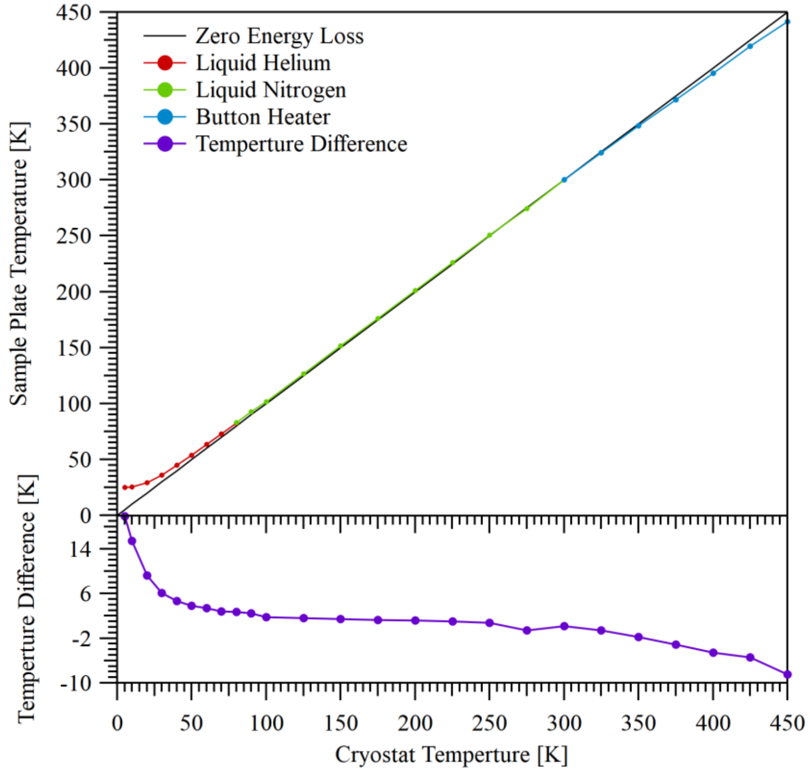
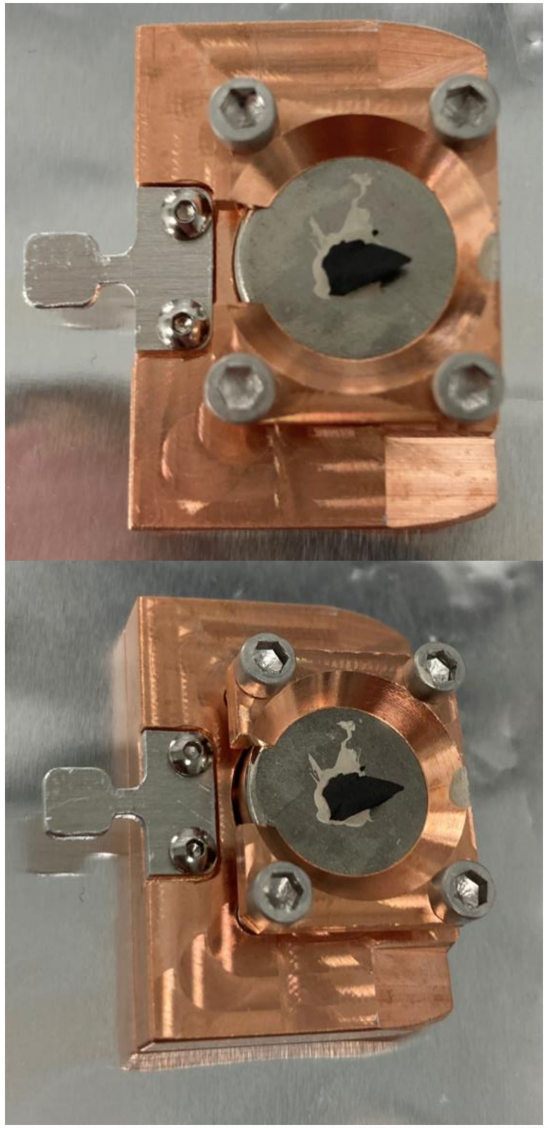


What magnetic fields are available?



- Out of plane
  - 0.35T single or 0.45T for double stack
- In-plane
  - 0.1-0.2T
- Cooling and heating are possible
  - Mounted with silver paint
  - Typically 1-2 samples per setup
- Magnets can be used for XCMD or XMLD experiments
  - Not yet often used, magnet holding plates only made available June 2023
- Please contact beamline staff before planning an experiment that requires an magnetic field.

How to optimize setup on several detectors?

- Current Amplifiers
  - Used to measure photocurrents
  - Dynamic range needs to be optimized

  - XAS: TEY, I0

- Silicon Drift Detector
  - 250 to 2500 eV
  - ≈ 80 eV FWHM

  - XRF
  - XAS: PFY

- Grating Emission Spectrometer
  - 75 to 1050 eV
  - 0.1 to 1 eV FWHM

  - XES
  - XAS: PFY

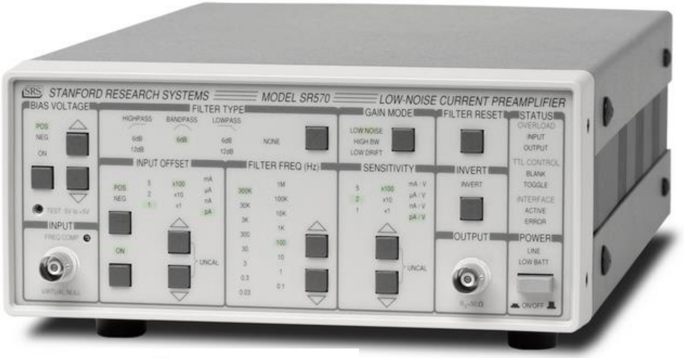
Sample and Mesh Current

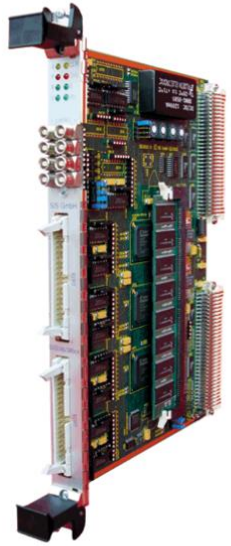
https://www.thinksrs.com/products/sr570.html

https://www.struck.de/sis3820.htmSIS

https://www.kromek.com/product/vtf-voltage-to-frequency-converter-module/

- Sample holder is floating electrically.
  - Thermal heat-sink with sapphire wafer.
- *n*A - *p*A current is converted to 3 – 4 V for transmission over longer distance.
  - ≈ 100 *p*A is the detection limit.
- V – F convertor digitizes the signal.
  - Convert 0 – 5 V to 1 MHz
- Pulse counting with “Master” Scaler/Counter.
  - Synchronizes all data acquisition in time.
- **Things to consider:**
  - Current can not be changed, but output voltage can be adjusted with the amplifier, keep it in the mid-range.
  - If the sensitivity is too high, peaks may clip.

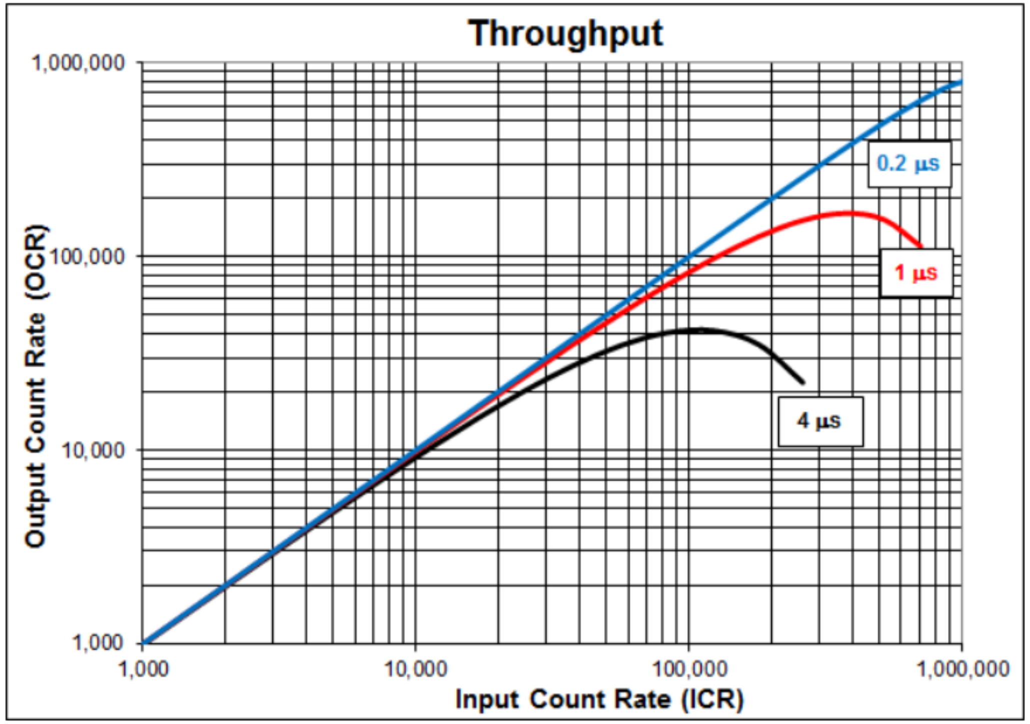




Silicon Drift Detector

FAST SDD with C Series Window

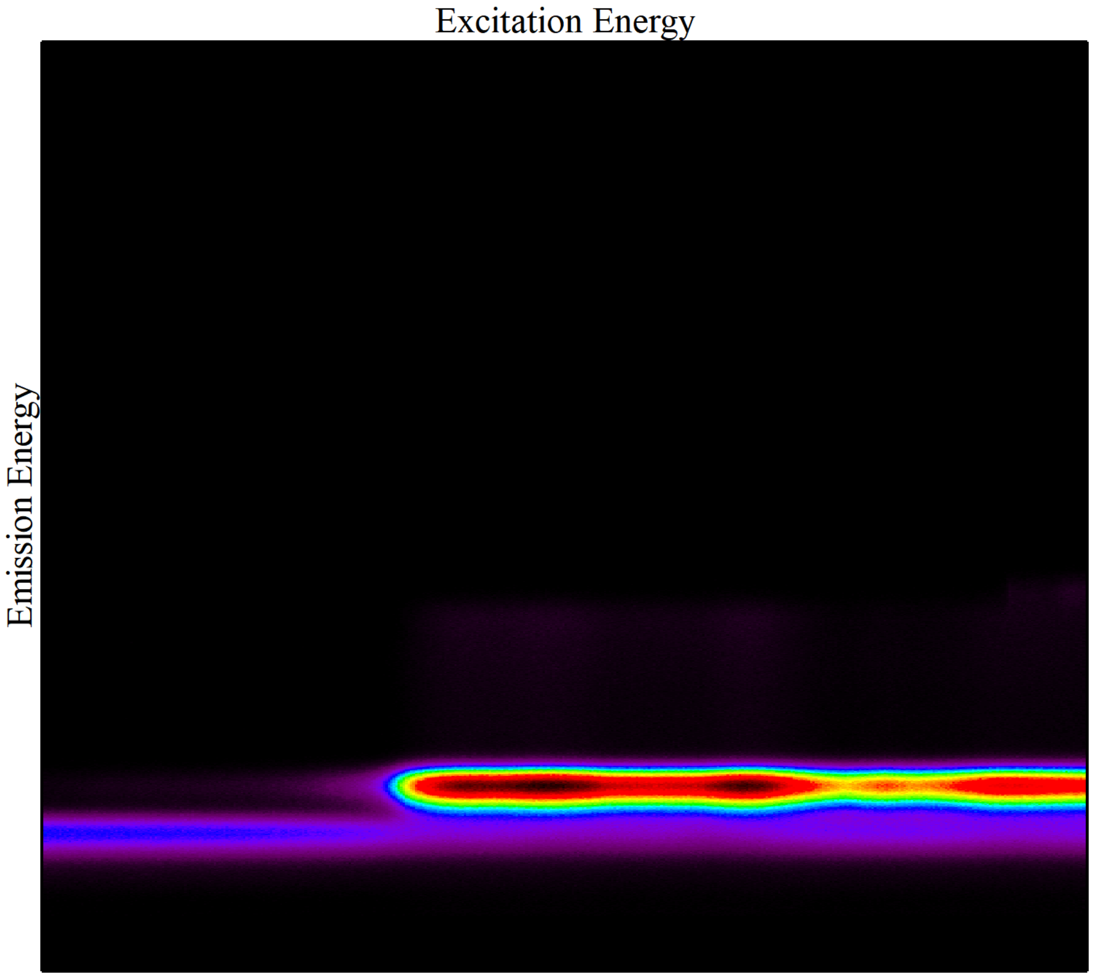
- Pulse Counting Device
  - X-rays generate pulses proportional to the energy
    - Low energy x-rays are more difficult due to electrical noise
    - Low energy cut off, high energy is limited due to QE of silicon sensor
  - Pulse shaping needed to determine energy, but pulse timing causes pile-up
    - Dead time: non linear response
  - Resolution typically 80 eV FWHM
- Advantages
  - Wide energy range: all x-rays can be detected simultaneously
  - Large solid angle: efficient
  - Provides guidance for x-ray emission experiments
    - Photons need to be visible on the SDD to be likely detected on the grating spectrometer



https://www.amptek.com/products/x-ray-detectors/fastsdd-x-ray-detectors-for-xrf-eds/fastsdd-silicon-drift-detector

Silicon Drift Detector: Cont’d

- Optimizing Input
  - Typically we reduce photon intensity.
    - Keep max rate < 100 kcps
    - Always adjust flux for SDD first is 4-jaw
- Data Output
  - MCA (1D array) read out at every data point.
- Data Reduction
  - Sum along data collection axis
    - XRF
  - Sum along emission energy axis
    - XAS, XRF maps, etc.
    - PFY, IPFY
- Data is collected at all times
  - No setup of the detector is required



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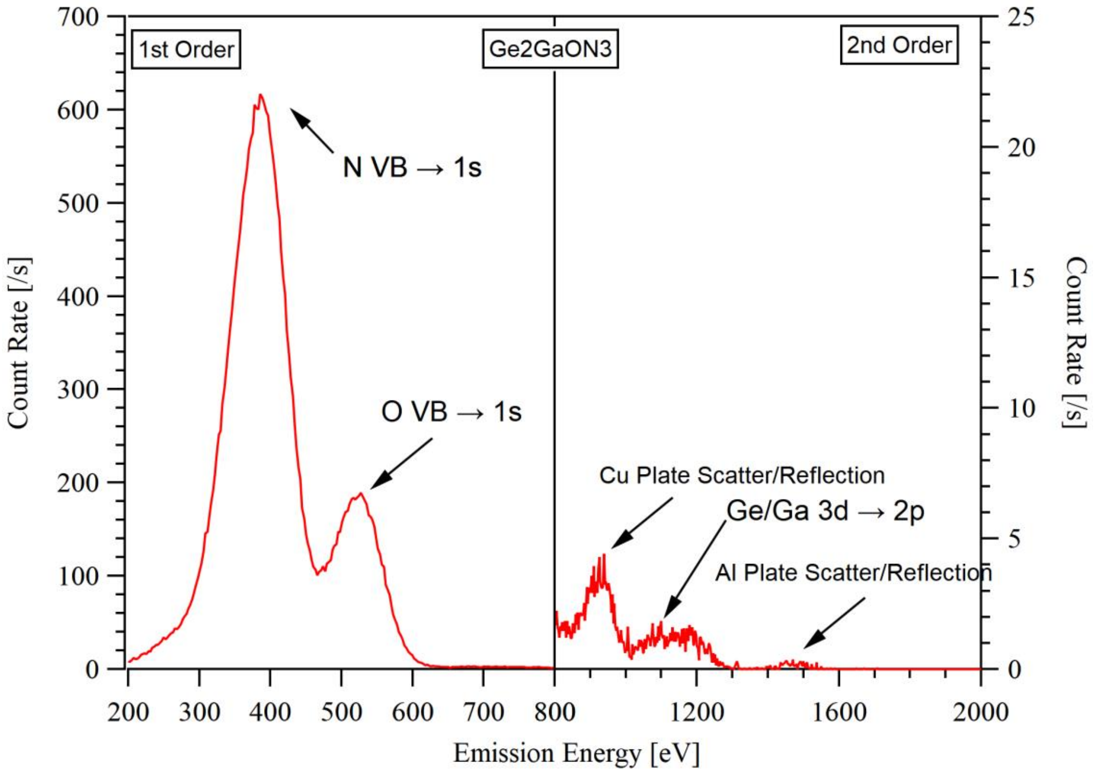

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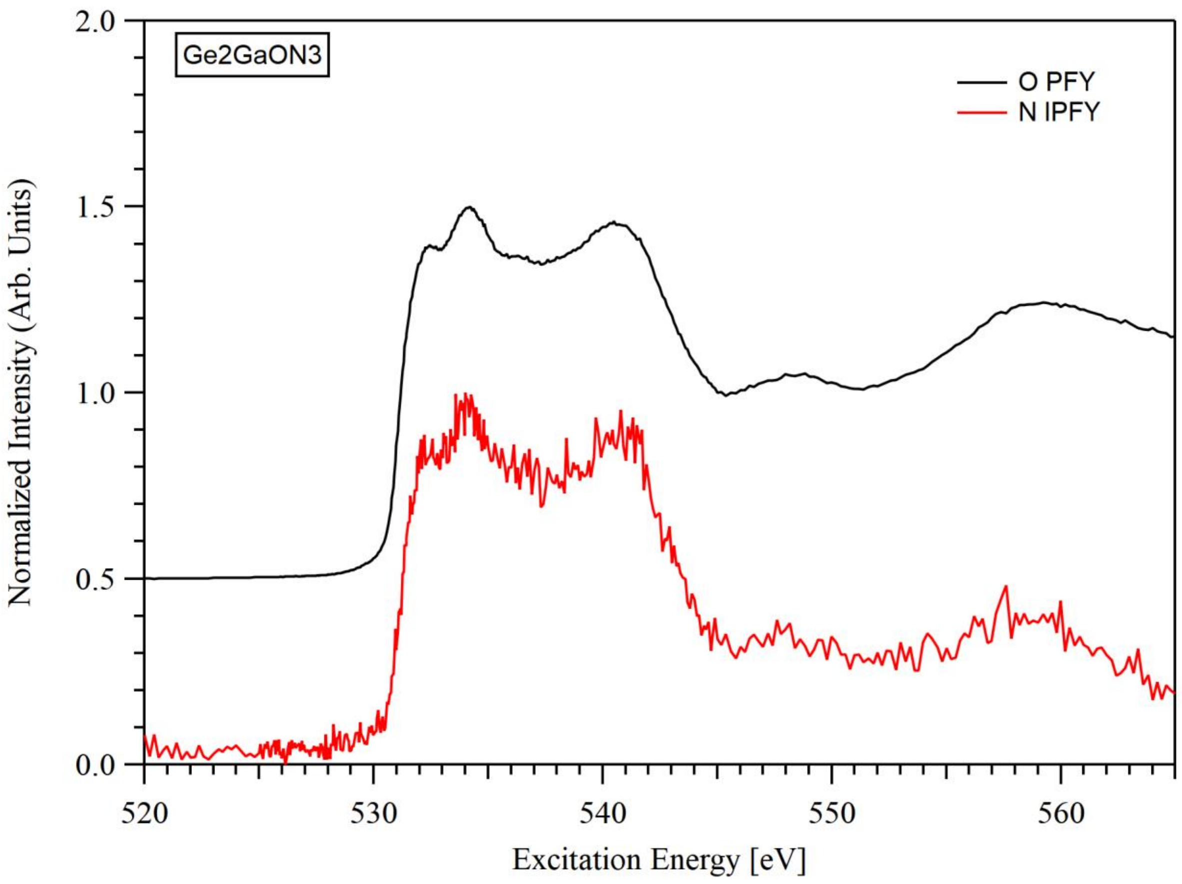


A Closer Look at Photon-In/Photon-Out Spectroscopy

- Partial Fluorescence Yield (PFY)
  - Measures a specific fraction of photons that leave the material.
    - Typically measured with energy dispersive devices:
      - Grating Spectrometers.
      - Solid State Detectors (Silicon Drift Detectors).
    - Self absorption occurs due to the transmission of photons in in the material to interact with a unit volume.

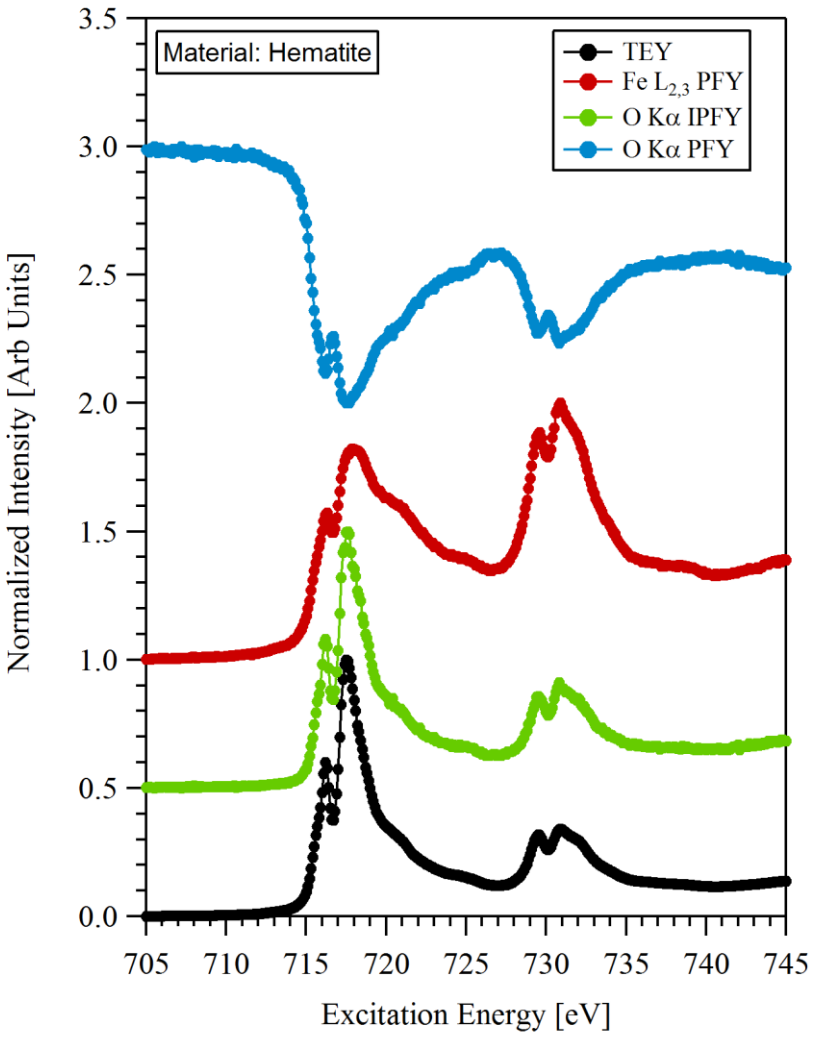
$$XAS_{PFY}[E_{in}] \sim \frac{\sum_{E_{out}} I_{PFY}[E_{in}]}{I_0[E_{in}]}$$

$$I_{PFY}[E_{in}] \sim I_o \sum_{E_{out}} \sum_{Volume} e^{-\mu\,[E_{in}]\vec{r}}\,\mu^x[E_{in}]e^{-\mu^x\,[E_{out}]\vec{r}}C^x_{PFY}$$

If we assume the outgoing photon is fluorescence E_{out} is fixed.

$$I_{PFY}[E_{in}] \sim I_o \sum_{Volume} e^{-\mu\,[E_{in}]\vec{r}}\,\mu^x[E_{in}]C^x_{PFY}$$

$\mu^x = 0$ *before the edge (assuming no 2^nd^ order)*



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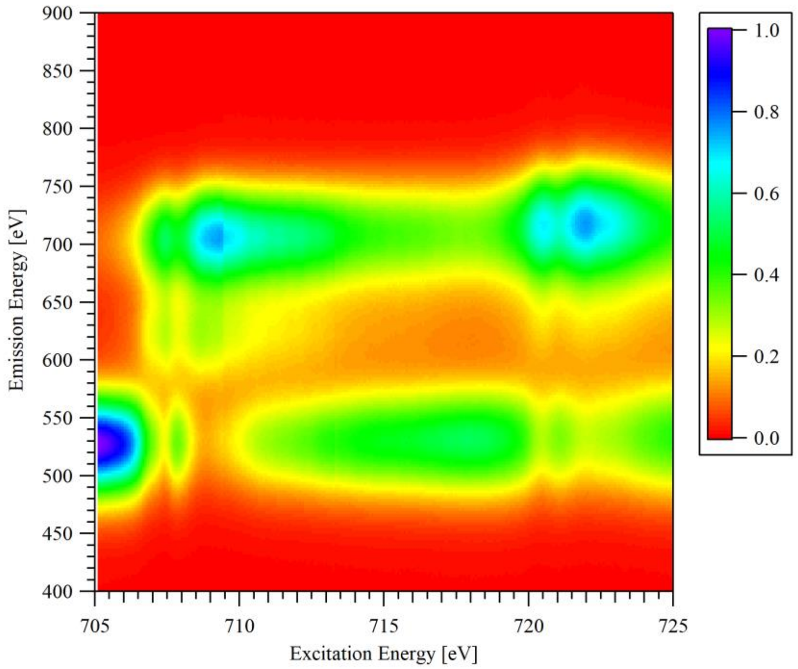
A Closer Look at Photon-In/Photon-Out Spectroscopy

- Inverse Partial Fluorescence Yield: α-Fe_2O_3
  - Assume we are exciting Fe 2p electrons: $E_{in} = 725\ eV$
    - O -> 1s = 543 eV; 2s = 37 eV
    - Fe -> 2p = 720/707 eV; 3s = 91 eV; 3p = 53 eV
  - All core electrons can be photoionized, but since soft x-rays have a small photon momentum, we are limited to dipole $\underline{\Delta l + 1}$
  - 3 lines present: Fe “VB” to Fe 2p, Fe 3s to 2p and O VB to 1s



$$XAS_{PFY}[E_{in}] \sim \frac{\sum_{E_{out}} I_{PFY}[E_{in}]}{I_0[E_{in}]}$$

$$I_{PFY}[E_{in}] \sim I_o \sum\nolimits_{E_{out}} \sum\nolimits_{Volume} e^{-\mu\,[E_{in}]\vec{r}}\, \mu^x[E_{in}] e^{-\mu^x\,[E_{out}]\vec{r}} C^x_{PFY}$$

If we assume the outgoing photon is fluorescence, E_{out} is fixed and $\mu^x =$ const far above the edge.

$$I_{PFY}[E_{in}] \sim I_o \sum\nolimits_{Volume} e^{-\mu\,[E_{in}]\vec{r}}\, C^x_{PFY} \sim \frac{1}{\mu} C^x_{PFY}$$

$$\boldsymbol{I_{IPFY}[E_{in}] \sim \mu C^x_{IPFY}}$$

$$\int e^{-\mu\vec{r}} d\vec{r} = -\frac{1}{\mu} e^{-\mu\vec{r}}\Big|_0^r \approx -\frac{1}{\mu}$$

For large $\vec{r} \to e^{-\mu\vec{r}} = 0$

(soft x-rays)

A Closer Look at Photon-In/Photon-Out Spectroscopy

- Inverse Partial Fluorescence Yield: α-Fe_2O_3
  - Assume we are exciting Fe 2p electrons: $E_{in} = 725\ eV$
    - O -> 1s = 543 eV; 2s = 37 eV
    - Fe -> 2p = 720/707 eV; 3s = 91 eV; 3p = 53 eV
  - All core electrons can be photoionized, but since soft x-rays have a small photon momentum, we are limited to dipole $\underline{\Delta l + 1}$
  - 3 lines present: Fe "VB" to Fe 2p, Fe 3s to 2p and O VB to 1s

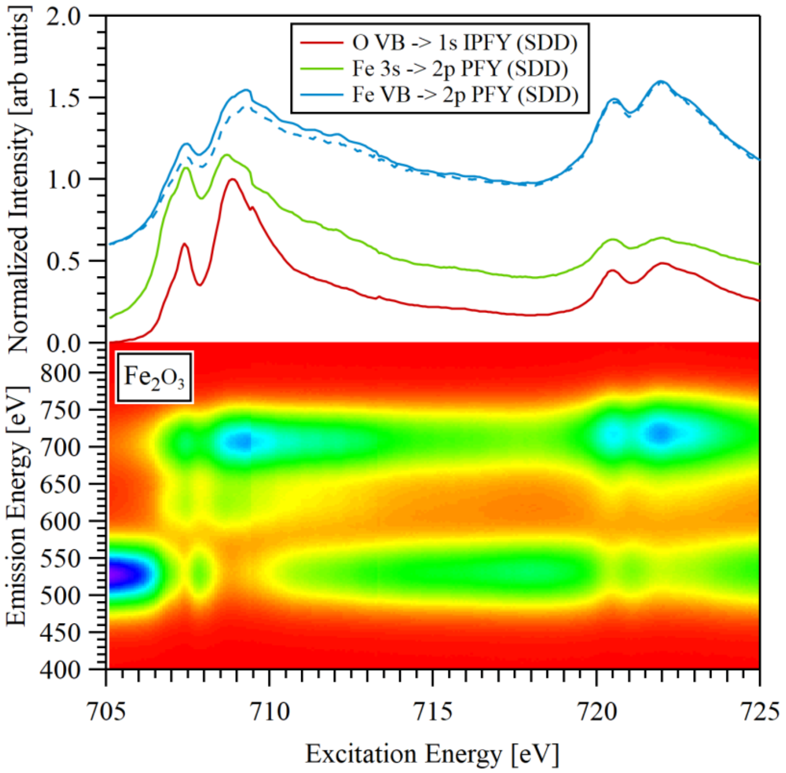
$$XAS_{PFY}[E_{in}] \sim \frac{\sum_{E_{out}} I_{PFY}[E_{in}]}{I_0[E_{in}]}$$

$$I_{PFY}[E_{in}] \sim I_o \sum_{E_{out}} \sum_{Volume} e^{-\mu\,[E_{in}]\vec{r}}\, \mu^x[E_{in}] e^{-\mu^x\,[E_{out}]\vec{r}} C^x_{PFY}$$

If we assume the outgoing photon is fluorescence, E_{out} *is fixed and* $\mu^x =$ *const far above the edge.*

$$I_{PFY}[E_{in}] \sim I_o \sum_{Volume} e^{-\mu\,[E_{in}]\vec{r}}\, C^x_{PFY} \sim \frac{1}{\mu} C^x_{PFY}$$

$$\boldsymbol{I_{IPFY}[E_{in}] \sim \mu C^x_{IPFY}}$$



$$\int e^{-\mu\vec{r}} d\vec{r} = -\frac{1}{\mu} e^{-\mu\vec{r}}\Big|_0^r \approx -\frac{1}{\mu}$$

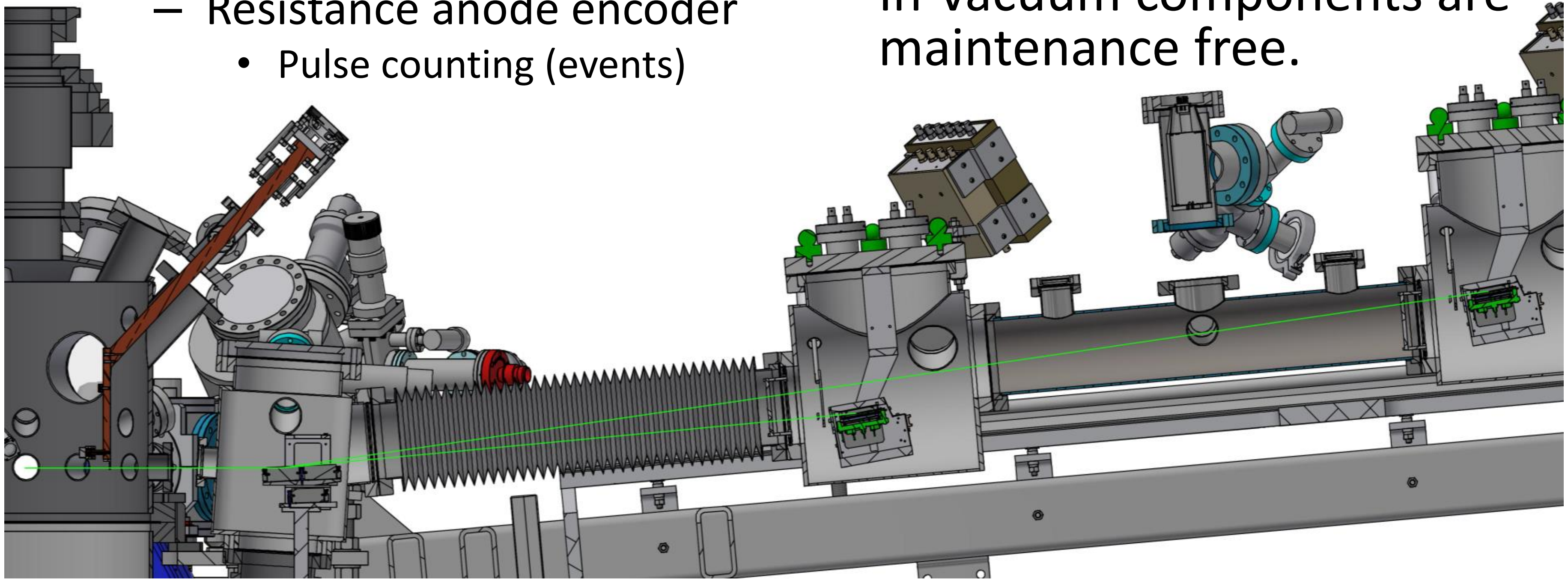
For large $\vec{r} \rightarrow e^{-\mu\vec{r}} = 0$

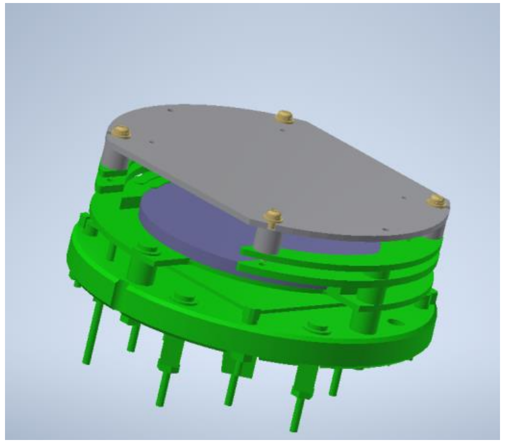
(soft x-rays)

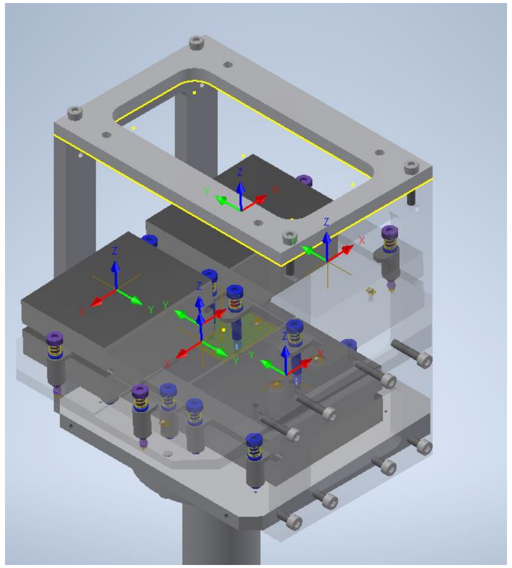
High Resolution Grating Spectrometer: Intro

- Spherical Gratings
  - Rowland Circle
- MCP (Micro-channel Plate)
  - Area Detector
  - Resistance anode encoder
    - Pulse counting (events)

- Six Diffraction Gratings
  - XLEG, LEG, MEG, HEG, HRMEG, HRHEG
  - Positioned using PI Hexapod
- In-vacuum components are maintenance free.

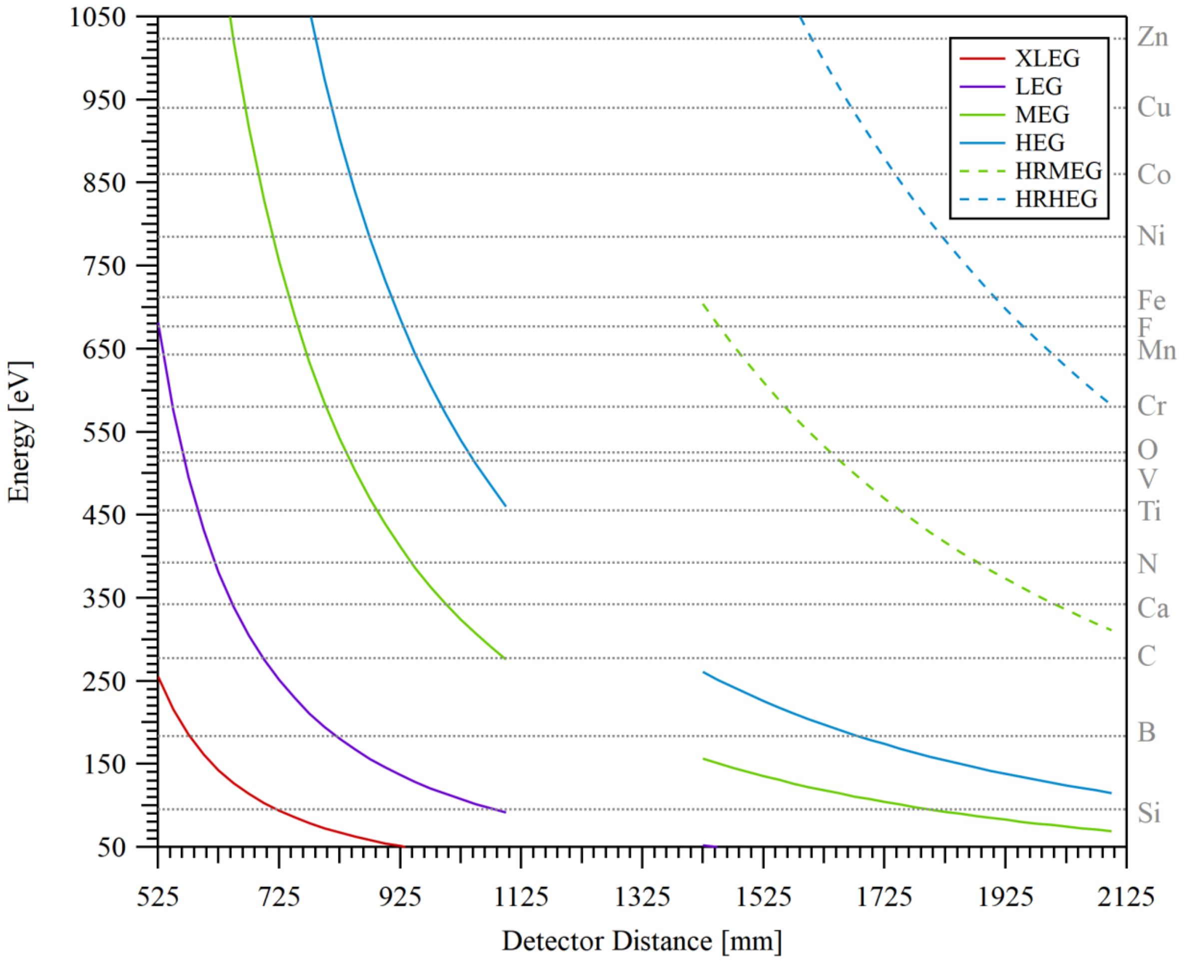






High Resolution Grating Spectrometer

- Differences compared to SDD
  - A specific grating is required for each energy range
  - Needs to be positioned for each energy
  - There is a finite energy window
  - Efficiency is such we are not count rate limited
- Grating Selection
  - Always overlap in energy range
  - Generally, higher energy range means better resolution, but less efficiency
  - Statistics are correlated to resolution
  - Always try to stay in the good range of the gratings for XES
- Practical limit: ≈ 1050 eV
  - XES requires longer counting times
    - Lower solid angle grating
    - Measure spectral density, not intensity
  - Beamline input drops at higher energy as well
    - Precision drops at higher energy, so improved statistics



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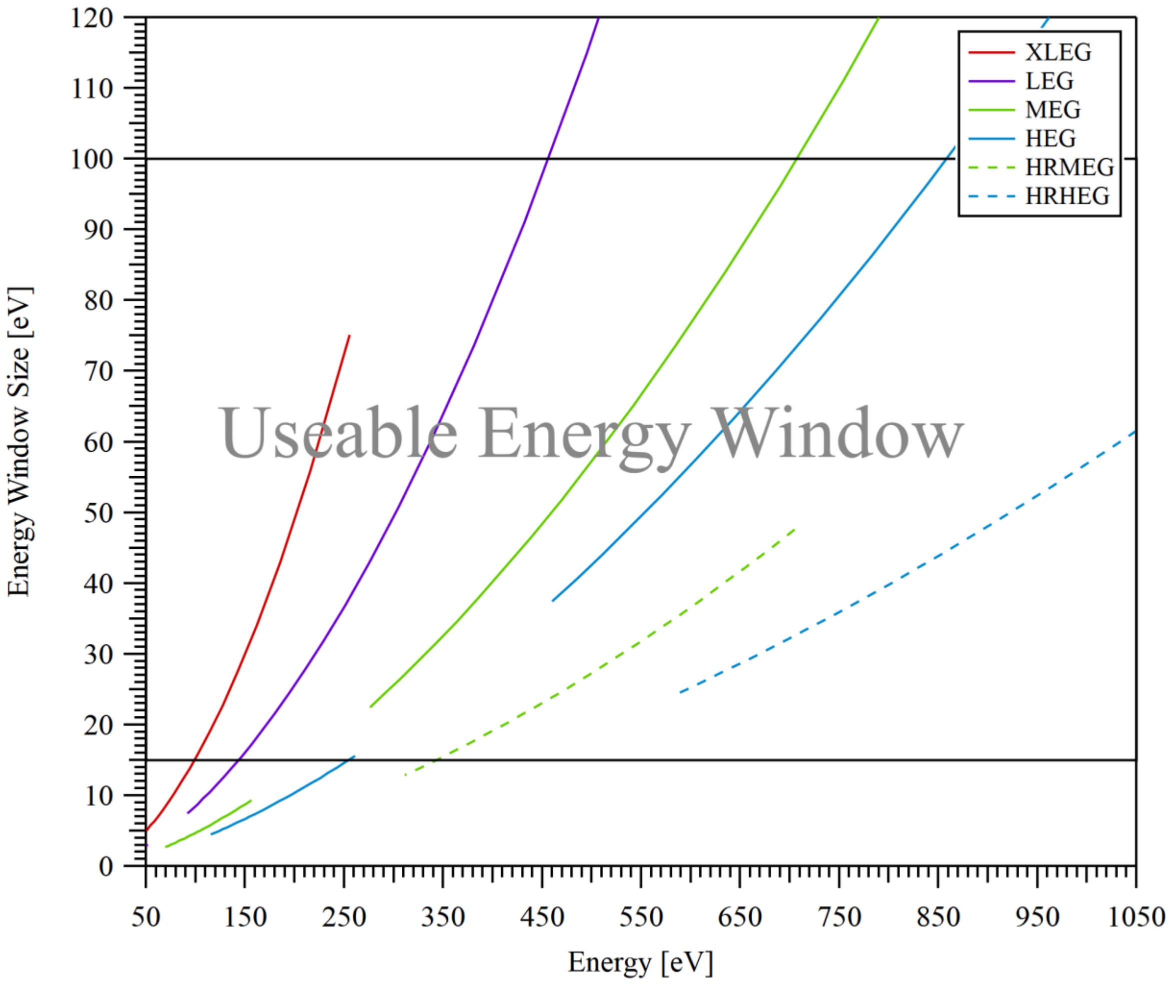

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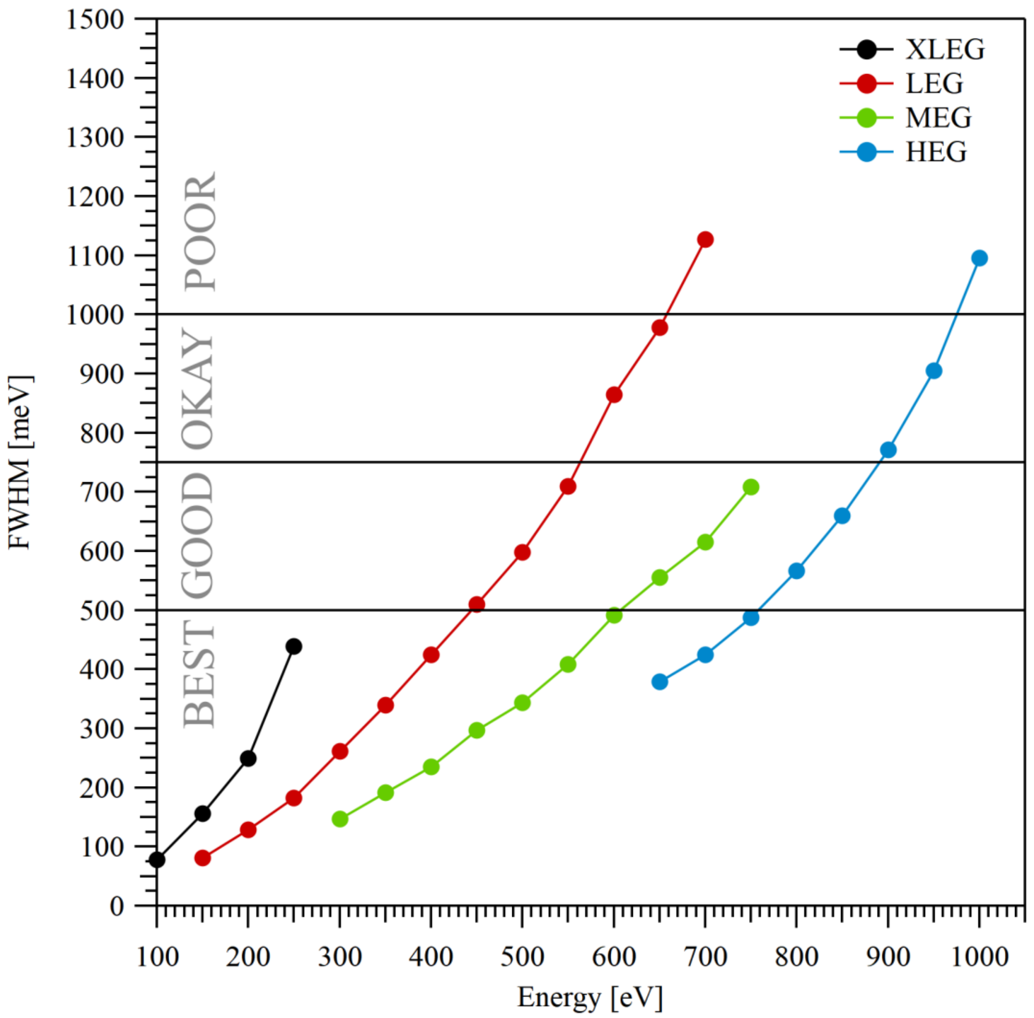

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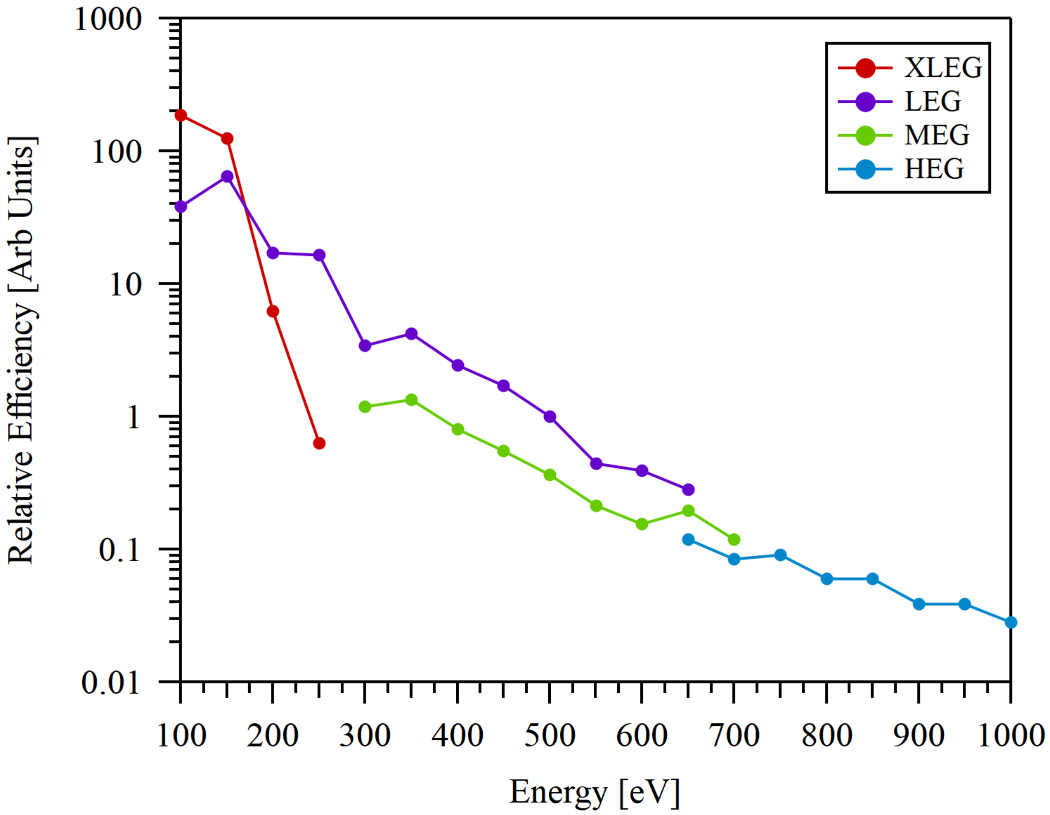

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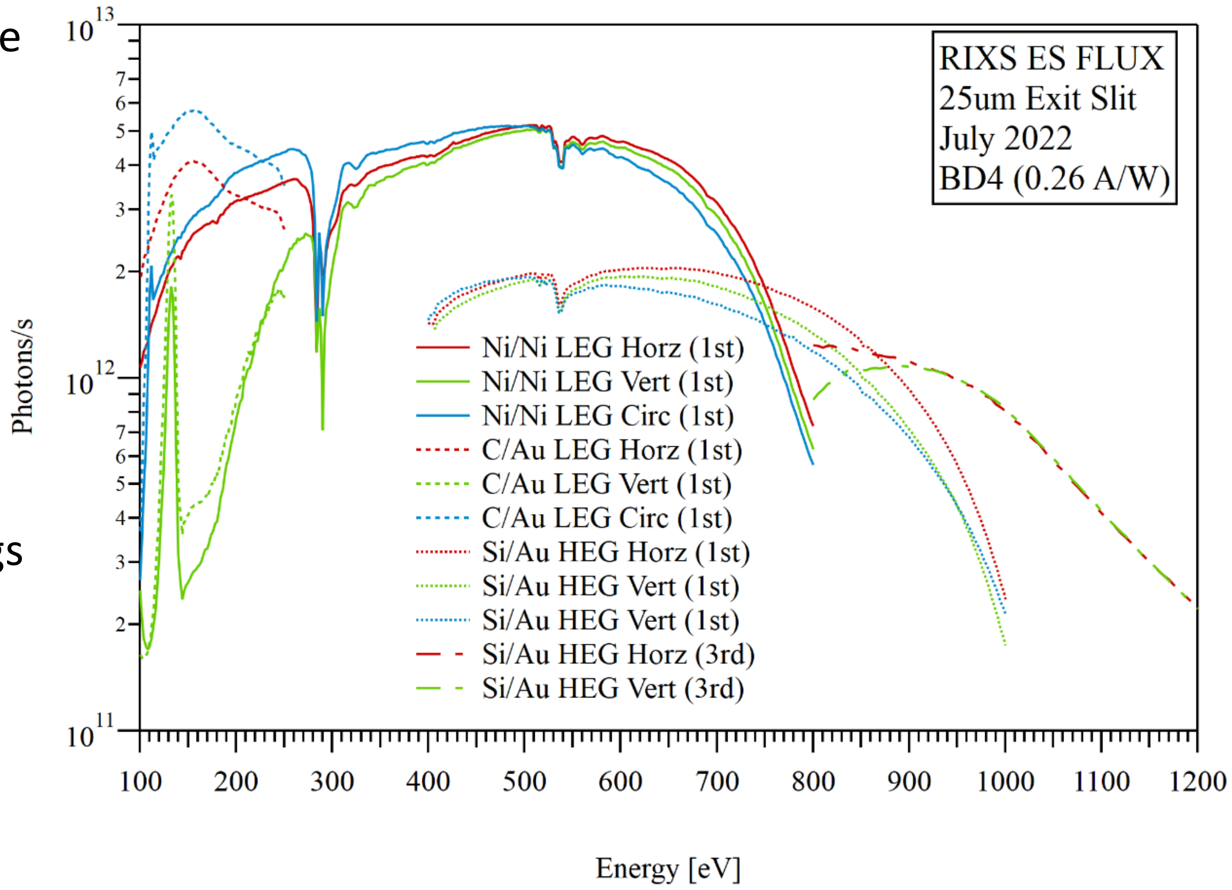
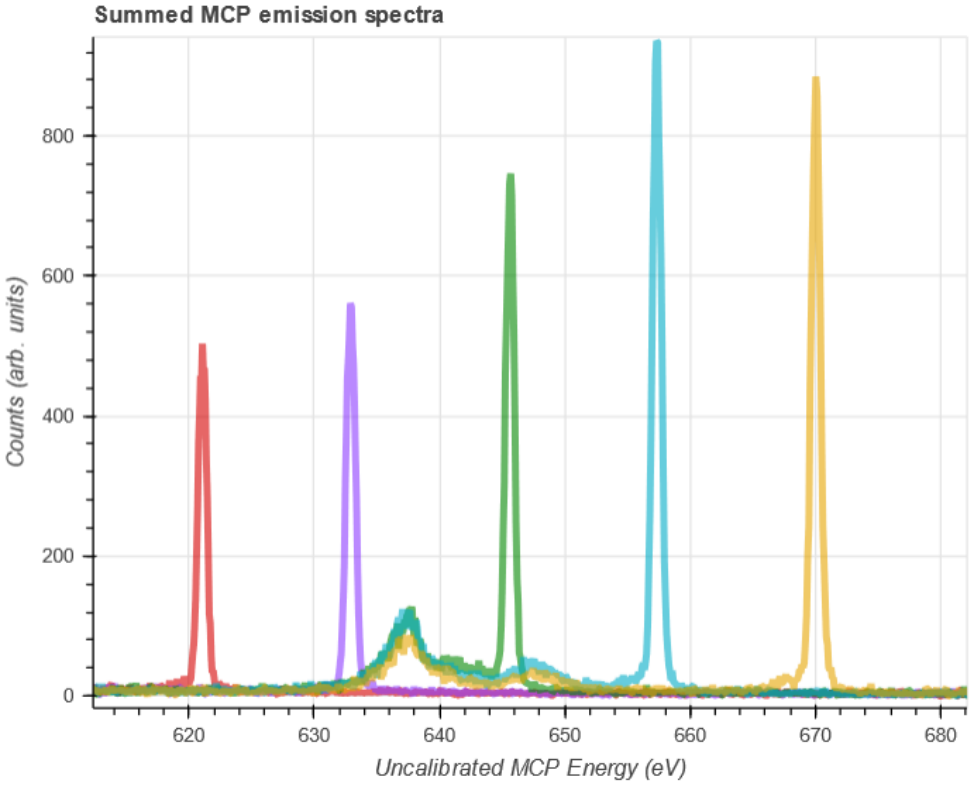


High Resolution Grating Spectrometer: Calibration

- Beamline Energy Calibration
  - Typically only a linear shift.
    - Small XAS range of near edge
  - Grating angle is read directly
    - Motion is repeatable
  - Beamline staff routinely check calibration
    - Repeatable, not absolute
  - Users need to bring standards for repeatable calibration between experiments
    - HOPG for C 1s, *h*-BN for N 1s, BGO for O 1s, etc.
- Grating Spectrometer Calibration
  - We can measure the outgoing energy better than optics/detectors can be positioned
  - Once the spectrometer is in place, **DO NOT MOVE IT!**
  - Requires 2-D calibration
    - Central energy and energy scale
  - Measure charge (coherent) scattering peaks to calibrate relative to the monochromator (beamline) energy
  - Apply similar linear shift that is used for the beamline energy



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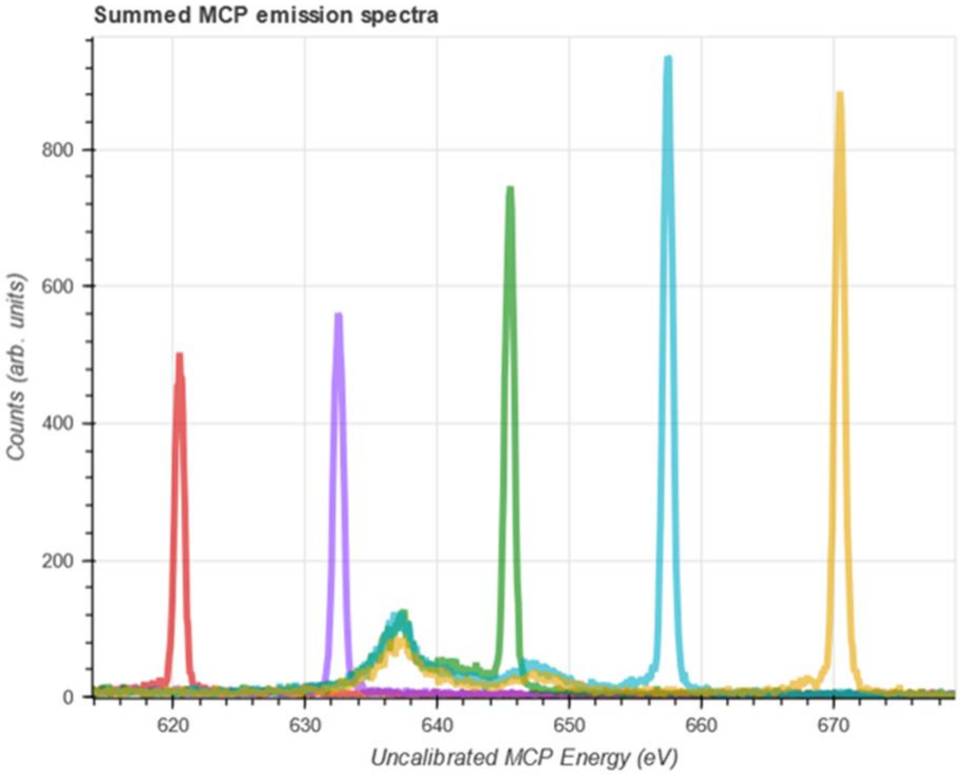
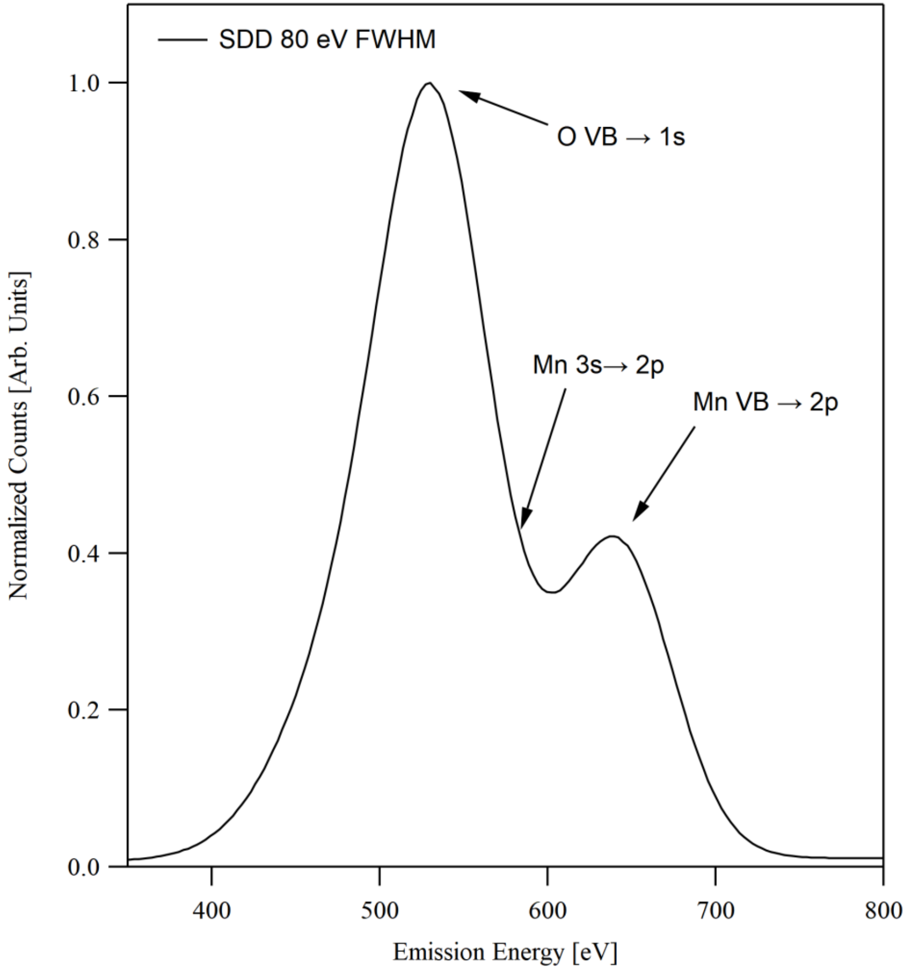


High Resolution Grating Spectrometer: Cont’d

- Why do need < 1 eV FHWM resolution?
  - SDD resolution typically 80 eV FWHM
  - Example: Mn_3O_4
    - Two emission lines visible, 3 actually present
  - Zoom in with < 1 eV resolution?
    - Detailed structure of the emission line.
- Data Collection
  - Similar to SDD, but small energy range.
    - Series of MCAs form an image.
  - Zoom in again with < 1 eV resolution?
    - Photons are not only XRF, but also RIXS
  - Every data point in XAS is a “unique” XES spectrum.
  - Data can summed along the detector energy scale to obtain XAS
    - One can select out all emission lines an even XRF vs. RIXS.
- Images can be manipulated to sum along excitation or emission pathways.
  - Patrick will discuss this in tomorrow’s Jupyter Notebook presentation.



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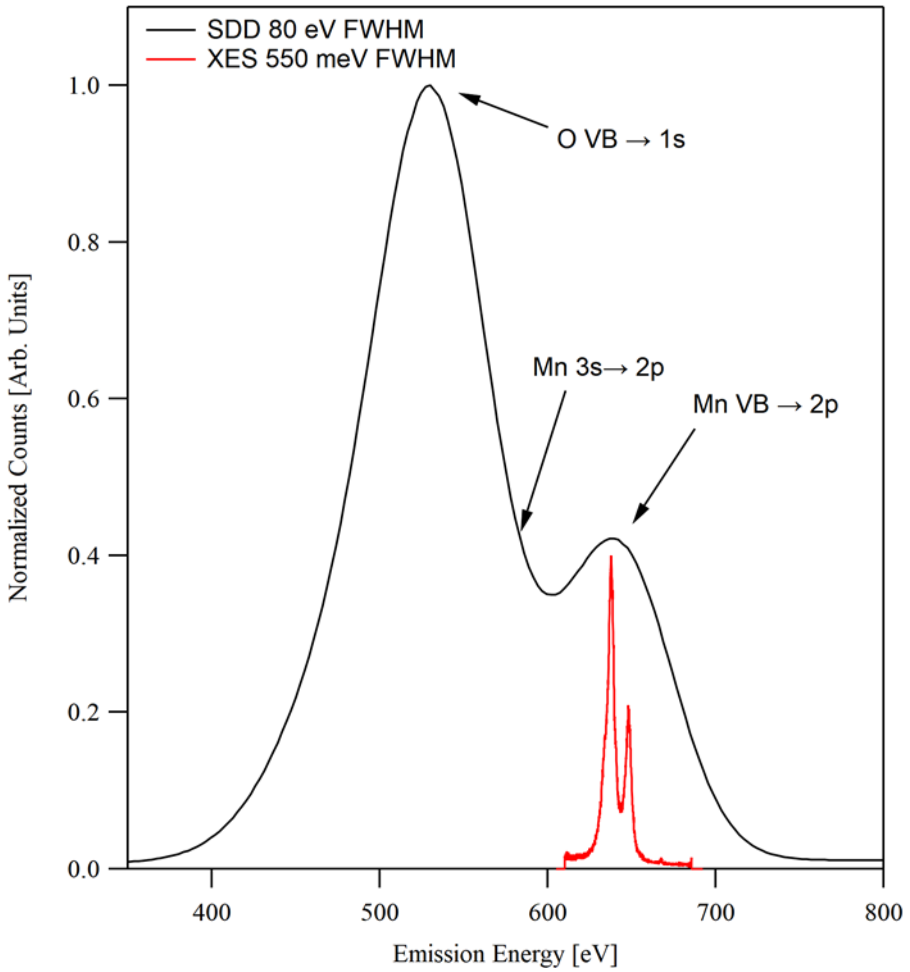

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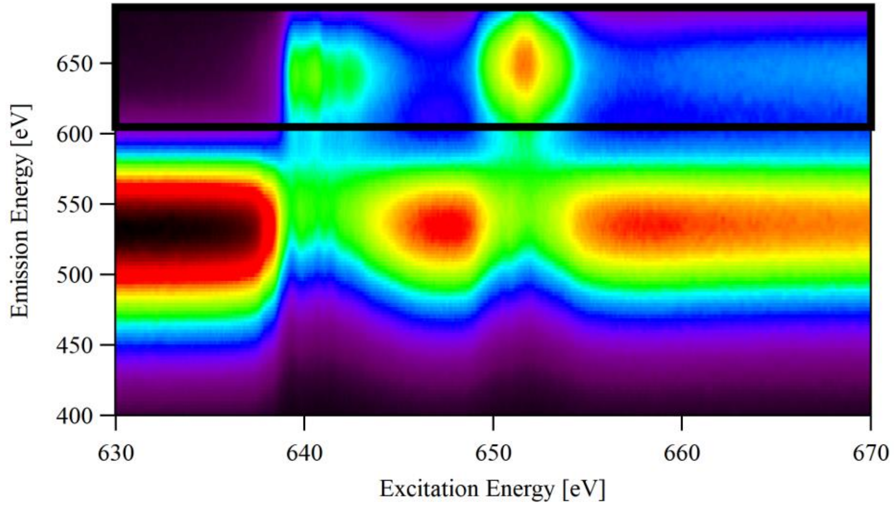

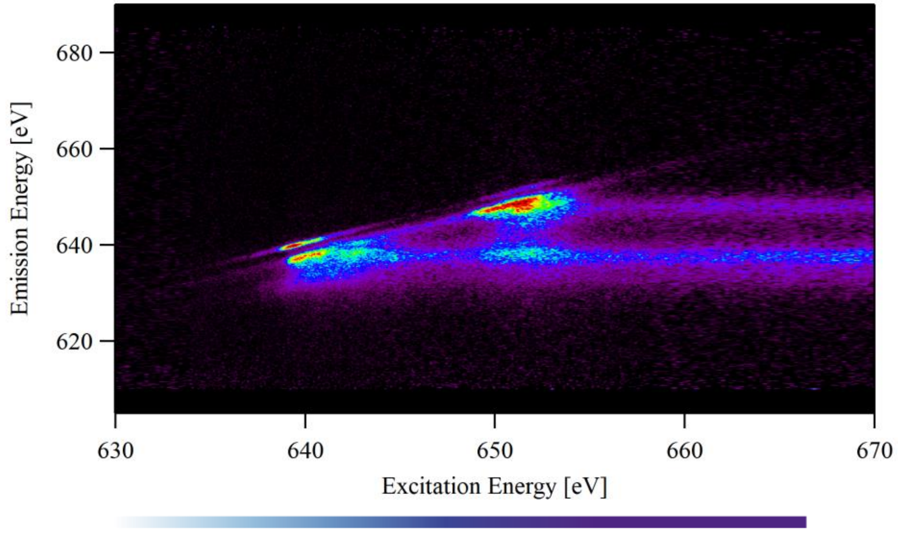

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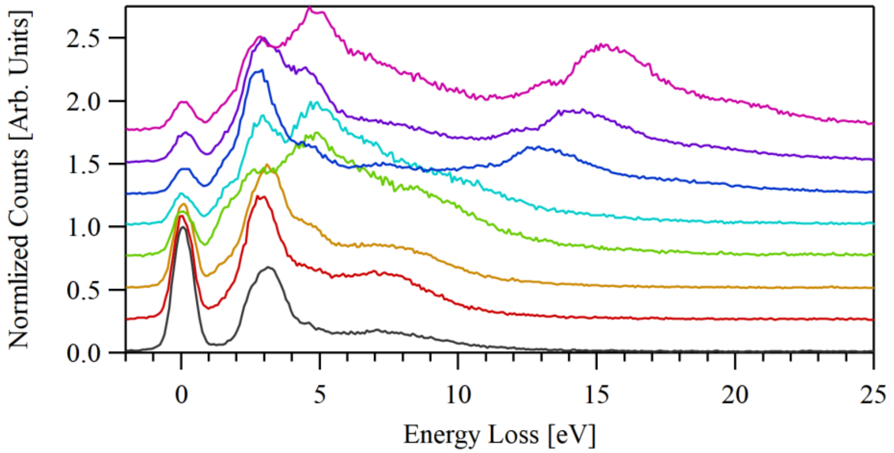

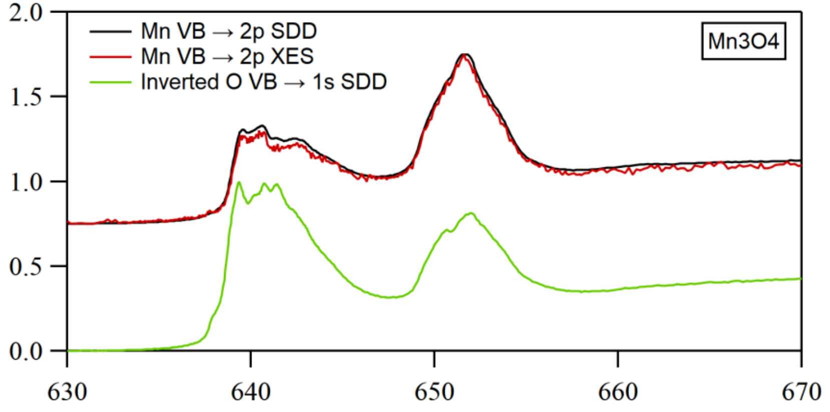

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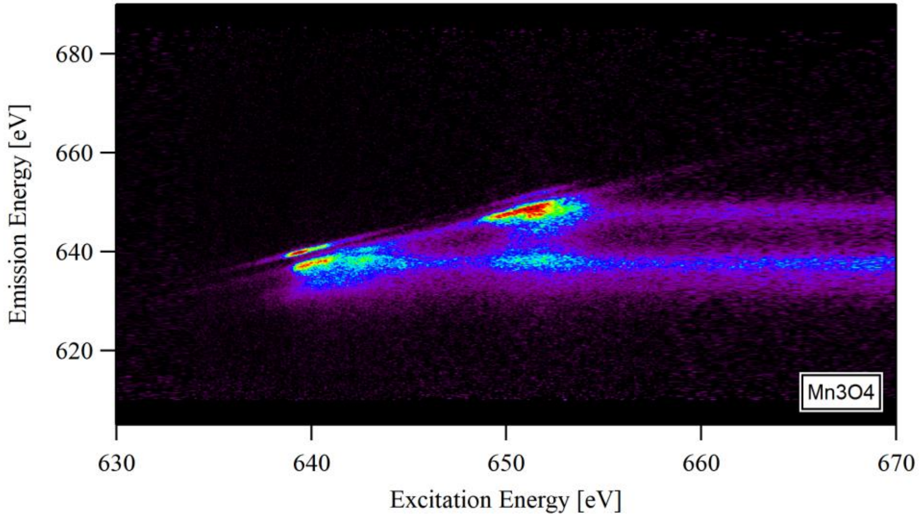
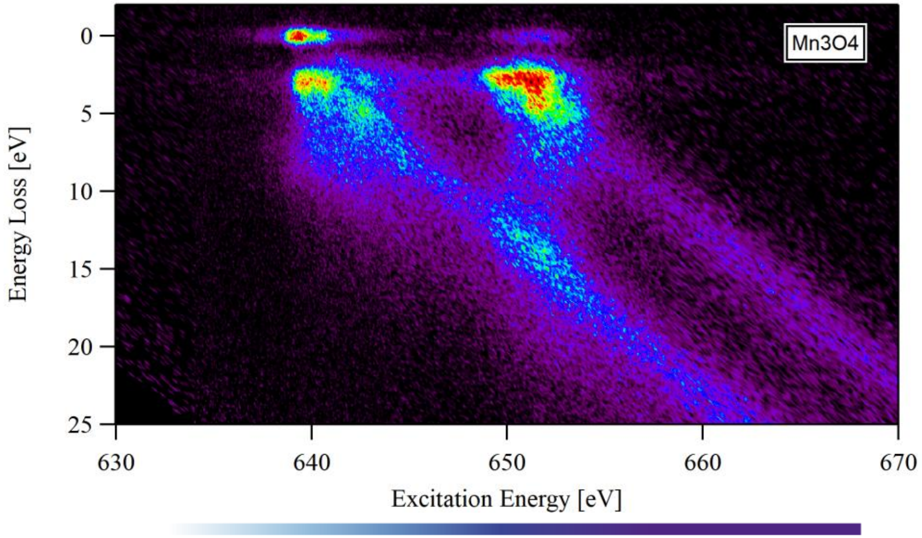
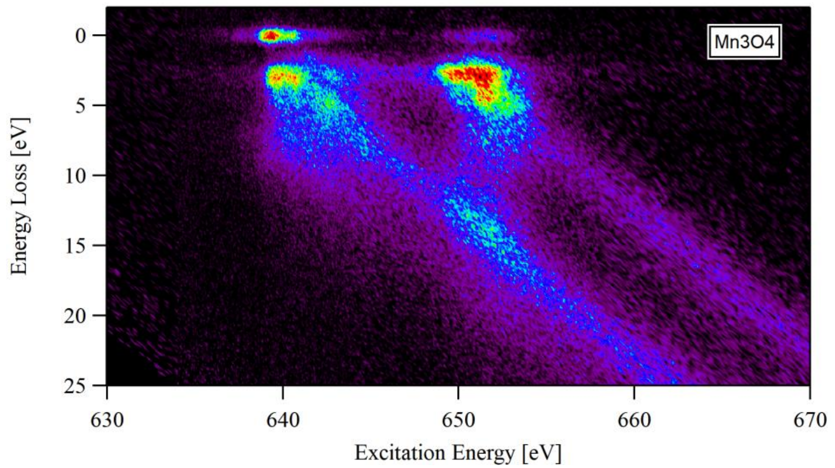
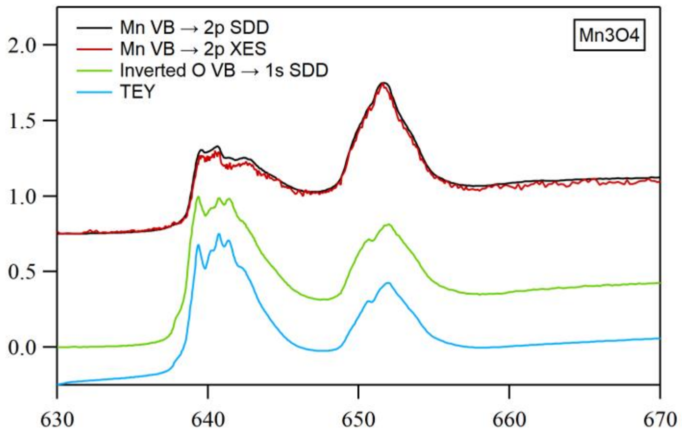


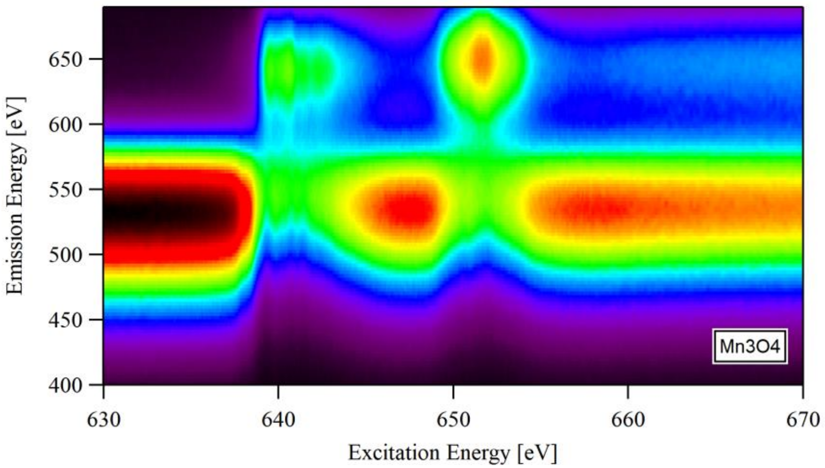


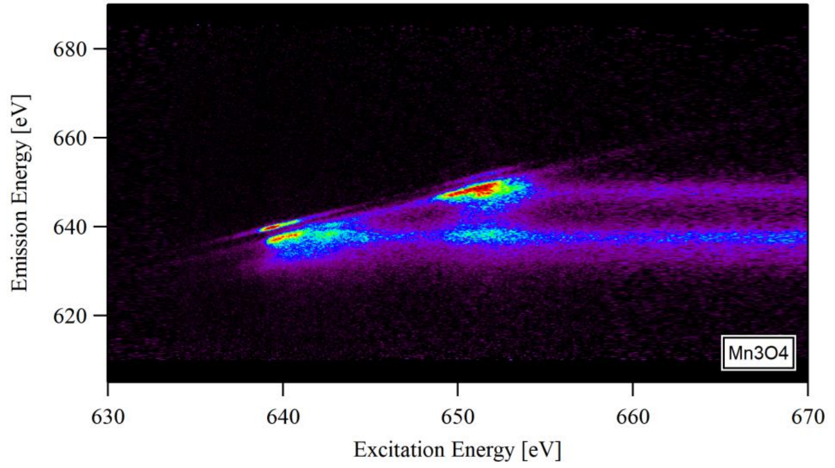
What does this all mean, with regard to my experiment?

- Once the beam is placed on your sample:
  - Angle, temperature, polarization, etc.
- You only need to record data, either moving the energy or another motor: time, position, etc.
- **All data is meaningful, consider it all equally.**
- **Data reduction is equally important!**





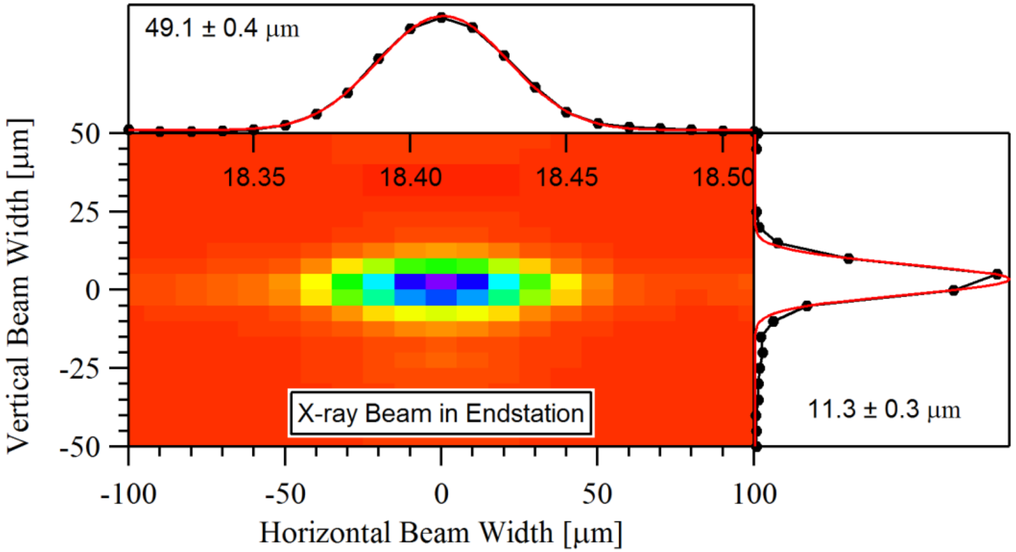




Challenges: Radiation Damage

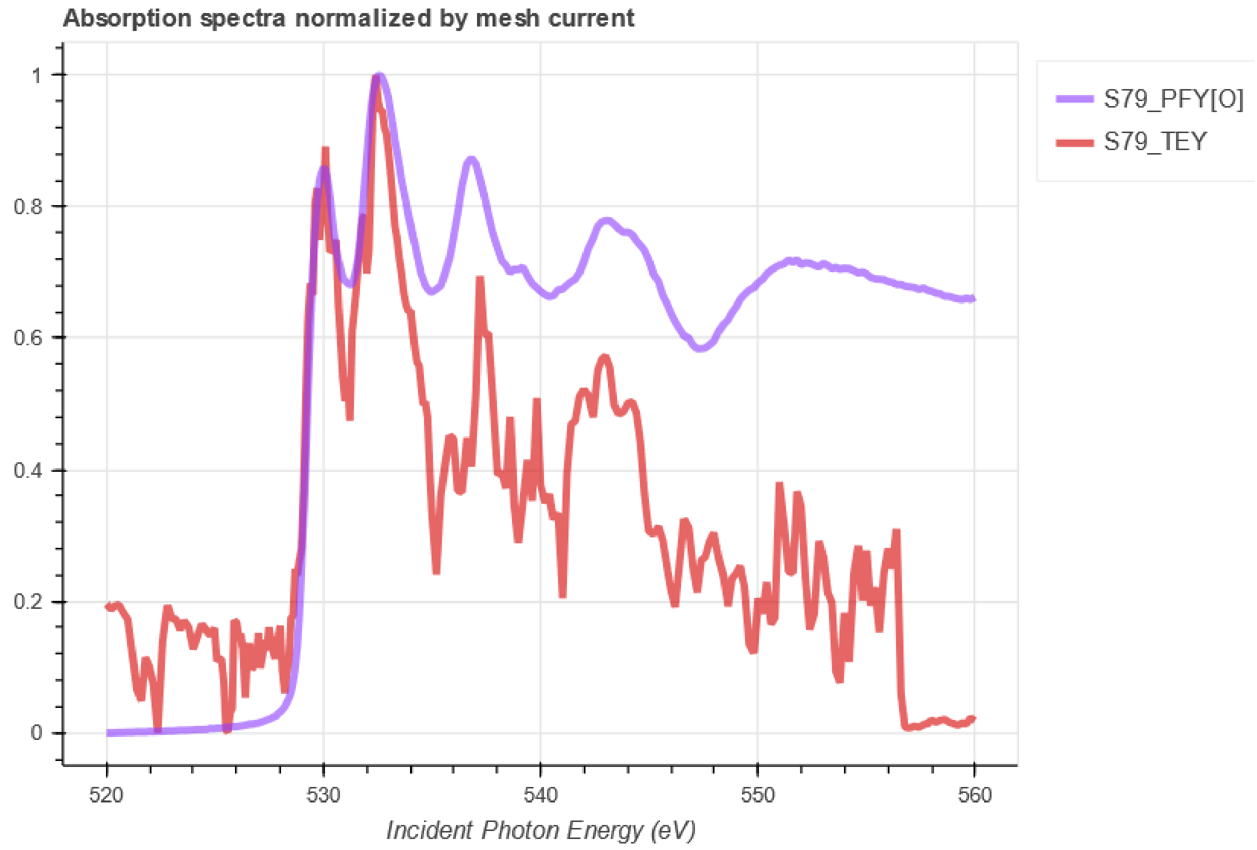
- XES Beamlines
  - Higher flux density
  - Beam is the source for the spectrometer.
  - Sample damage/change is more likely.
- XAS Beamlines
  - 2-3 orders of magnitude less photon density
- Always try to characterize sample damage/changes
  - Do multiple scans, then add them together
  - Do a couple shorter XAS scans, then a longer scan and compare
  - Cooling can alleviate some damage, but not available for powder samples
- Recognizing changes in your sample
  - Change in XAS baseline/onset
  - Change in the overall output with time
  - Change or loss of optical photons emitted

- **SGM**: 1000 μm X 100 μm
  - 1 X 10^5 μm^2
- **PGM**: 500 μm X 500 μm
  - 2.5 10^5 μm^2
- **BL8 (ALS)**: 100 μm X 35 μm (10X more flux)
  - 3.5 X 10^3 μm^2
- **REIXS**: 50 μm X 10 μm
  - 5 X 10^2 μm^2



Challenges: Improving the TEY Signal

- Charging always and issue
  - Insulators/semiconductors
  - Poor conductors
- Signal noise is not downward, but upward
  - Charge cascade due to dielectric breakdown
- Why is my TEY bad and what can I do?
  - Too large of photocurrent density
  - **Too many photons**
- TEY does not need many photons
  - TFY (< 1%): with finite size detector
    - ≈ $10^4 - 10^5$ photons/s
  - TEY (> 90 %)
    - $10^9 - 10^{10}$ electrons/s
- There are other factors that limit TEY
  - Single to background ratio



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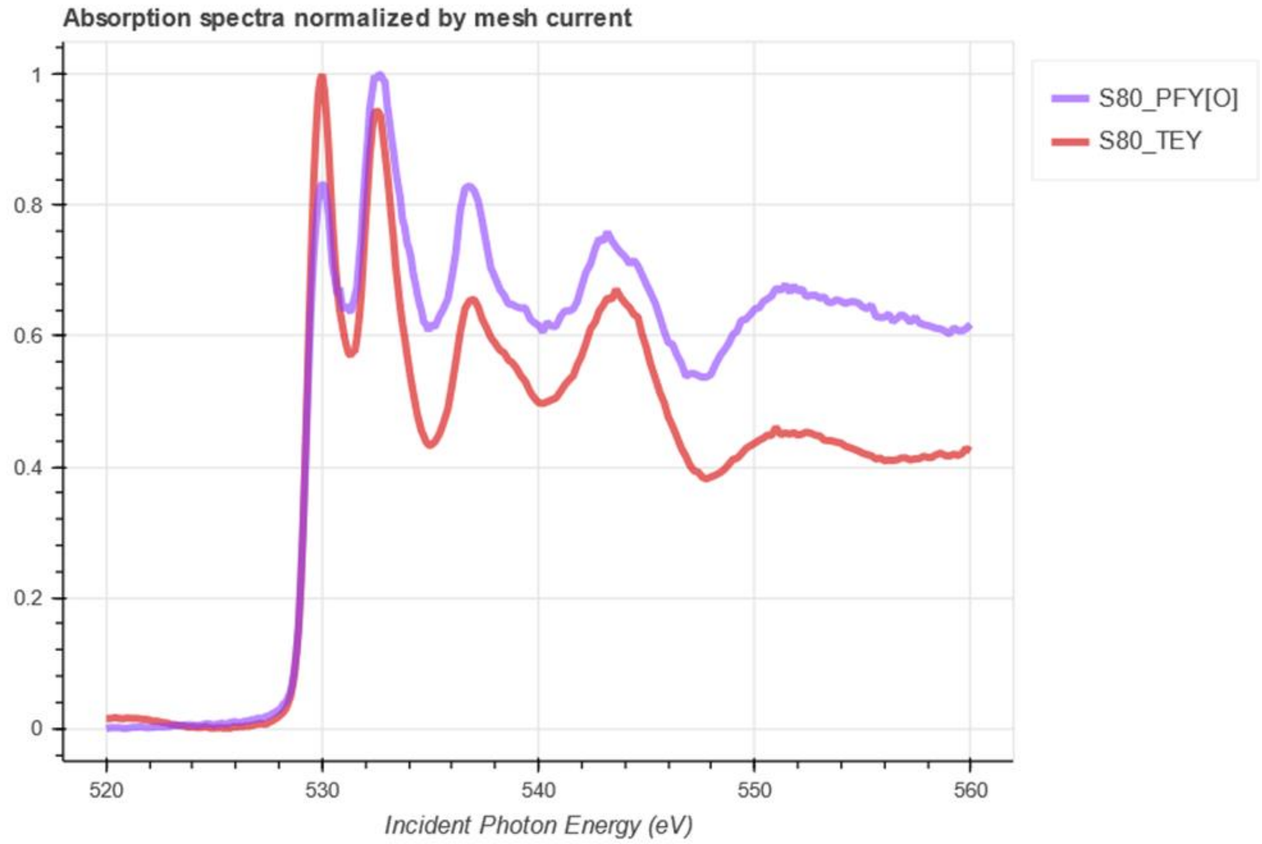

Conclusions

- Plan your experiment considering the practical limitations of the beamline and spectrometer.
  - Always bring reference samples to calibrate your data.
  - Know what resolution you require to achieve your experiment outcome.
- The experiment setup of XES and XAS is quite simple, data reduction during and on the collected data is equally important.
  - Plot your data as you go and keep a log book.
  - Problems in data can be found and corrected before the end of the beamtime.
- Always complete at least two XAS scans to test for sample changes, which will always be worse for longer XES measurements.
  - We can't reduce sample damage if we don't know it is happening.
- Sometime less is more: TEY can be improved by reducing the photon beam intensity.
  - Improves some of the issues, but doesn't solve everything: such as contrast.
- Always consult the beamline staff if not have not carried out a similar experiment on REIXS previously.
  - A well planned experiment is more likely to be successful.