Brookhaven National Laboratory

U.S. DEPARTMENT OF ENERGY

Data reduction and background removal

Akhil Tayal

03-19-2024

@BrookhavenLab

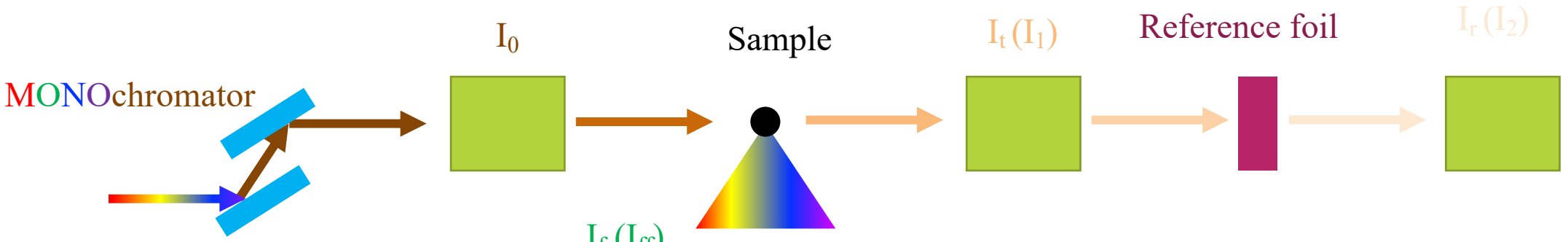
XAFS books

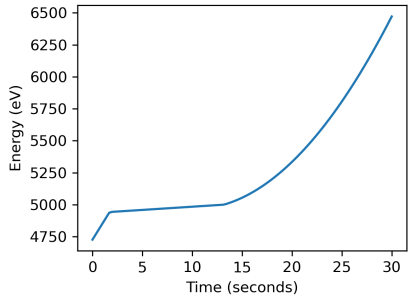
Introduction to XAFS: A Practical Guide to X-ray Absorption Fine Structure Spectroscopy
Grant Bunker

EXAFS: Basic Principles and Data Analysis
Dr. Boon K. Teo

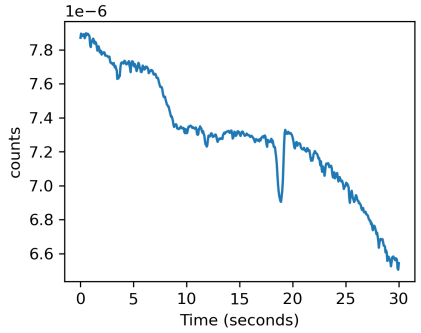
XAFS for Everyone
Scott Calvin

Data collection

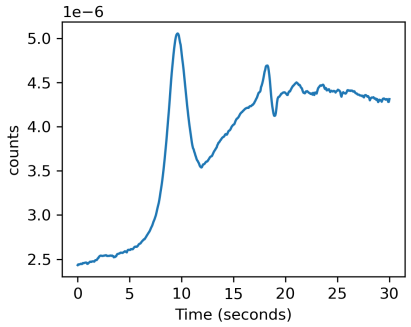




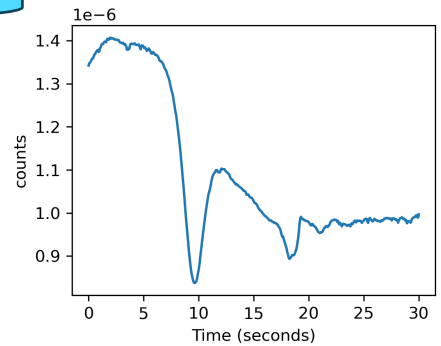
MONOchromator



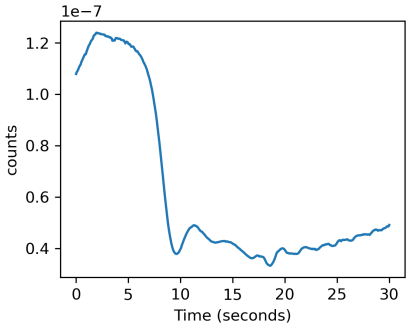
I0



If

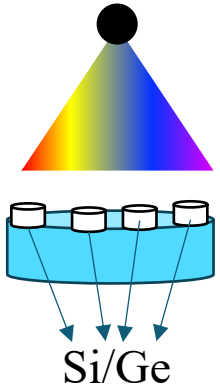


It (I1)

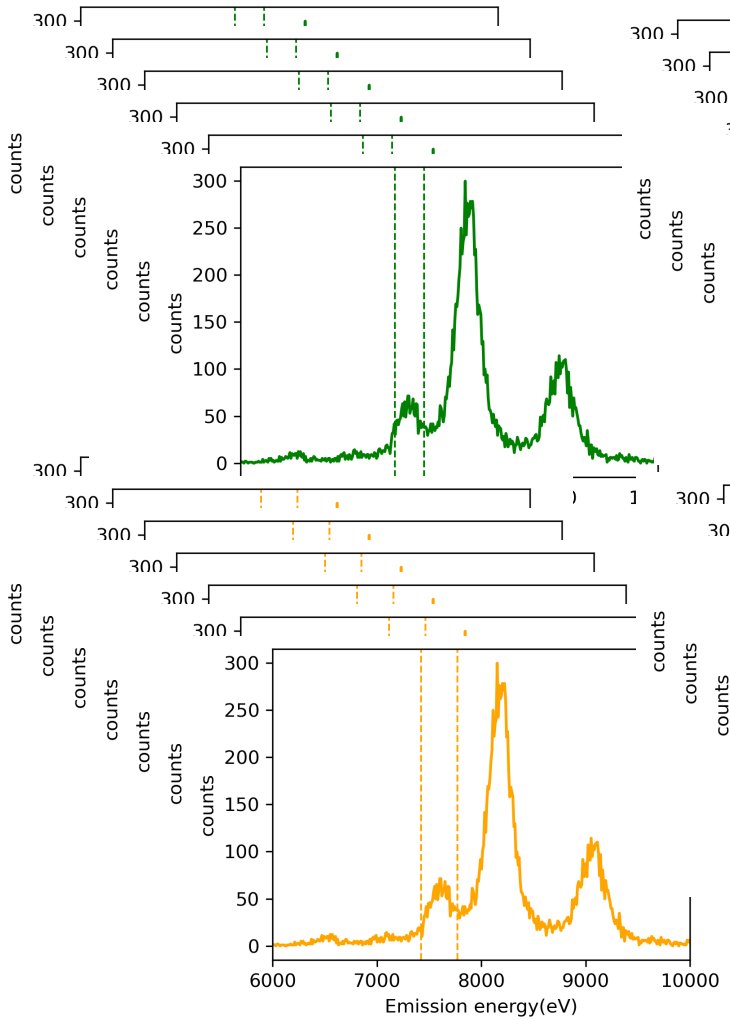


Ir (I2)

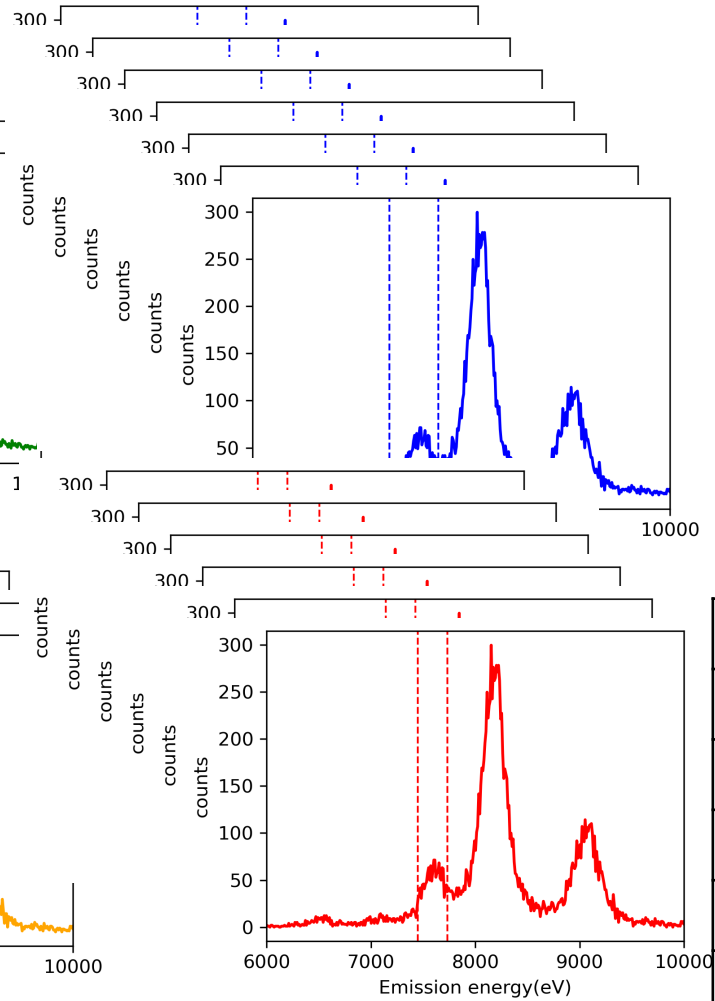
Si/Ge detector data



Channel 1



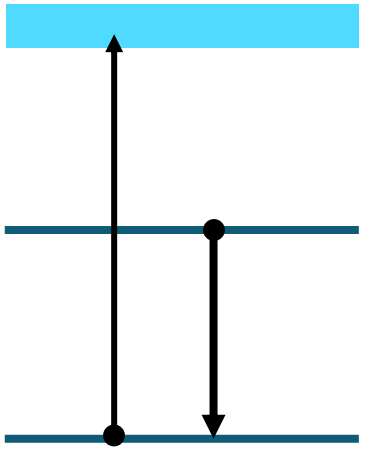
Channel 2



Channel 3

Channel 4

Energy Level



Energy	Ch1	Ch2	Ch3	Ch4
E1	45	44	45	46
E2	42	41	40	42
E3	20	23	25	20
E4	21	21	21	21
E5	23	20	21	22

Data File

# energy	i0	it	ir	iff	xs_roi01	xs_ch01_roi01	xs_ch02_roi01	xs_ch03_roi01	xs_ch04_roi01
13073.000000	-3.972144e-06	-1.405205e-07	-1.108702e-08	-2.481562e-07	1.622849e-02	4.025056e-03	4.057912e-03	3.530254e-03	4.615269e-03
13078.000000	-3.987104e-06	-1.411195e-07	-1.117292e-08	-2.494934e-07	1.638280e-02	4.028765e-03	4.117364e-03	3.528756e-03	4.707913e-03
13083.000000	-4.007183e-06	-1.421063e-07	-1.126769e-08	-2.511177e-07	1.649624e-02	4.073405e-03	4.140805e-03	3.540213e-03	4.741813e-03
13088.000000	-3.985857e-06	-1.418912e-07	-1.128669e-08	-2.500882e-07	1.633222e-02	4.058421e-03	4.052907e-03	3.541270e-03	4.679625e-03
13093.000000	-3.987541e-06	-1.424549e-07	-1.134783e-08	-2.504947e-07	1.640753e-02	4.098472e-03	4.056191e-03	3.525928e-03	4.726942e-03
13098.000000	-4.003168e-06	-1.434870e-07	-1.147684e-08	-2.520052e-07	1.623098e-02	4.071138e-03	4.027229e-03	3.554777e-03	4.577840e-03

```
# Facility.name: NSLS-II
# Facility.mode: Beam available
# Facility.current: 399.7092291335699
# Facility.current: 3 GeV
# Facility.year: 2023
# Facility.cycle: 3
# Facility.GUP: 313873
# Facility.SAF: 312125
# Experimenter.name: Akhil Tayal
# Beamline.name: ISS (8-ID)
# Beamline.x-ray_source: damping wiggler
# Beamline.collimation_mirror1.material: Si
# Beamline.collimation_mirror2.material: Pt
# Beamline.collimation_mirror2.bender_loading: -259.0
# Beamline.focusing: toroidal mirror
# Beamline.focusing.material: Pt
# Beamline.focusing.bender_loading: -398.0
# Beamline.harmonic_rejection: Rh
# Mono.scan_mode: Si(111)
# Mono.d_spacing: 3.1354951
# Mono.scan_mode: pseudo-channel cut
# Mono.scan_type: fly_scan
# Mono.trajectory_name: 647b56c3-e11a.txt
# Mono.direction: None
# Mono.angle_offset: 0.69726544
# Mono.angle_offset: 39.95 deg
# Mono.encoder_resolution: 48.0 nrad
# Detector.I0: ion chamber
# Detector.I1: ion chamber
# Detector.I2: ion chamber
# Detector.IF: PIPS
# Detector.I0.length: 15 cm
# Detector.I1.length: 28 cm
# Detector.I2.length: 15 cm
# Detector.IF.thickness: 300 um
# Detector.I0.gas.N2: 50.0%
# Detector.I1.gas.N2: 50.0%
# Detector.I2.gas.N2: 50.0%
# Detector.I0.gas.He: 50.0%
# Detector.I1.gas.He: 50.0%
# Detector.I2.gas.He: 50.0%
```

```
# Detector.aux: {'Xspress3': {'config': {}}}
# Element.symbol: Pt
# Element.edge: L2
# Element.line: None
# Scan.transient_id: 395793
# Scan.uid: 47eb3f72-47c2-4132-bcc6-0d293a2b9627
# Scan.edge_energy: 13273.0
# Scan.start_time: 09/23/2023  21:15:46.030720
# Scan.end_time: 09/23/2023  21:17:22.651740
# Scan.name: Pt0p05_rep RT cool Pt-L2 90sec 0002
# Scan.comment:
# Sample.name: Pt0p05_rep
# Sample.comment:
# Sample.position.x: 7.666231008499999
# Sample.position.y: -89.5050982975
# Sample.position.z: -12.988999999999995
# Sample.position.theta: 0.0
# SampleHeater.temperature1.setpoint: 300.0
# SampleHeater.temperature1.readback: 1372.0
# SampleHeater.current.setpoint: 0.0
# SampleHeater.current.readback: 0.0
# SampleHeater.temperature2.setpoint: 25.0
# SampleHeater.temperature2.readback: 33.7
# SampleHeater.voltage.setpoint: 0.0
# SampleHeater.voltage.readback: 0.0
# SampleHeater.PID.P: 0.025
# SampleHeater.PID.I: 0.07
# SampleHeater.PID.D: 0.0
# SampleGasCart.MFC.CH4.setpoint: 0.0
# SampleGasCart.MFC.CH4.readback: 0.0
# SampleGasCart.MFC.CO.setpoint: 0.0
# SampleGasCart.MFC.CO.readback: 0.0
# SampleGasCart.MFC.H2.setpoint: 0.0
# SampleGasCart.MFC.H2.readback: 0.0
# SampleGasCart.MFC.exhaust.setpoint: 100.0
# SampleGasCart.MFC.exhaust.readback: 25.67
# SampleSwitchValve.GHS.readback: 1
# SampleSwitchValve.GasCart.readback: 0
# SampleSwitchValve.Inert.readback: 0
# Potentiostat.Voltage.readback: 0
# Potentiostat.Current.readback: 0
# SampleGasHandlingSystem.gas_a.name: None
# SampleGasHandlingSystem.gas_b.name: None
# SampleGasHandlingSystem.gas_c.name: Ethylene
# SampleGasHandlingSystem.gas_d.name: None
# SampleGasHandlingSystem.gas_e.name: He
# SampleGasHandlingSystem.MFC1.setpoint: 25.0
# SampleGasHandlingSystem.MFC1.readback: 25.0
# SampleGasHandlingSystem.MFC2.setpoint: 0.0
```

Important terms

Strength of absorption is “cross section” σ (cm^2)

Probability of absorption = $x\rho\sigma_{total} = x\mu$ (μ is linear absorption coefficient)

Bouguer’s Law:

$$I_t = I_0 \ e^{-\mu x}$$

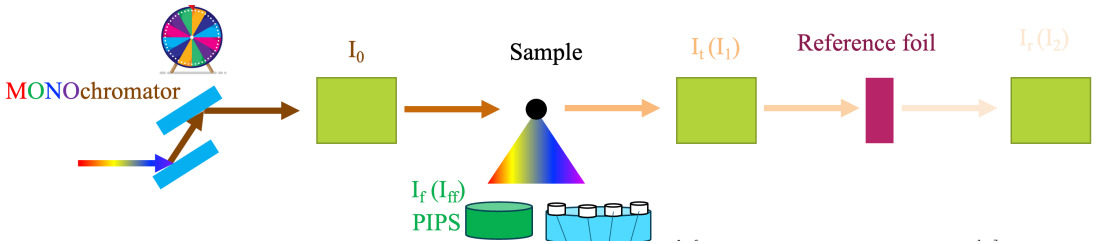
Absorption coefficient for transmission

$$\mu x = log\left(\frac{I_0}{I_t}\right)$$

Absorption coefficient for fluorescence

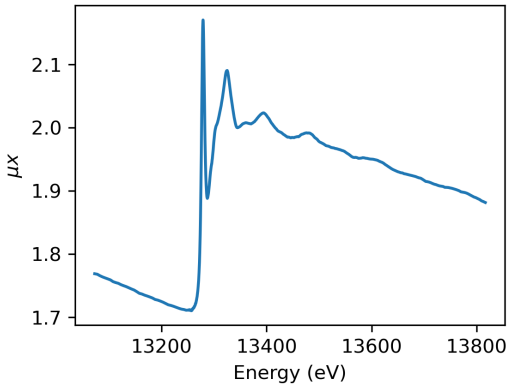
$$\mu x = \left(\frac{I_f}{I_0}\right)$$

Calculation of μ (absorption coefficient)

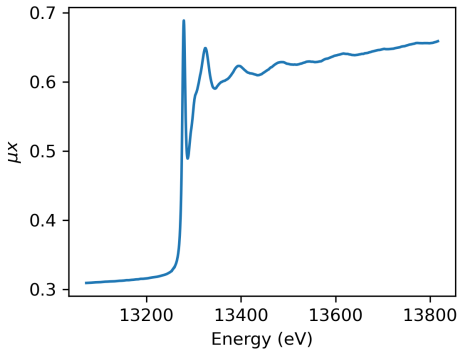



```
# energy          i0             it             ir             iff            xs_roi01   xs_ch01_roi01 xs_ch02_roi01 xs_ch03_roi01 xs_ch04_roi01
13073.000000 -3.972144e-06 -1.405205e-07 -1.108702e-08 -2.481562e-07 1.622849e-02 4.025056e-03 4.057912e-03 3.530254e-03 4.615269e-03
13078.000000 -3.987104e-06 -1.411195e-07 -1.117292e-08 -2.494934e-07 1.638280e-02 4.028765e-03 4.117364e-03 3.528756e-03 4.707913e-03
13083.000000 -4.007183e-06 -1.421063e-07 -1.126769e-08 -2.511177e-07 1.649624e-02 4.073405e-03 4.140805e-03 3.540213e-03 4.741813e-03
13088.000000 -3.985857e-06 -1.418912e-07 -1.128669e-08 -2.500882e-07 1.633222e-02 4.058421e-03 4.052907e-03 3.541270e-03 4.679625e-03
13093.000000 -3.987541e-06 -1.424549e-07 -1.134783e-08 -2.504947e-07 1.640753e-02 4.098472e-03 4.056191e-03 3.525928e-03 4.726942e-03
13098.000000 -4.003168e-06 -1.434870e-07 -1.147684e-08 -2.520052e-07 1.623098e-02 4.071138e-03 4.027229e-03 3.554777e-03 4.577840e-03
```

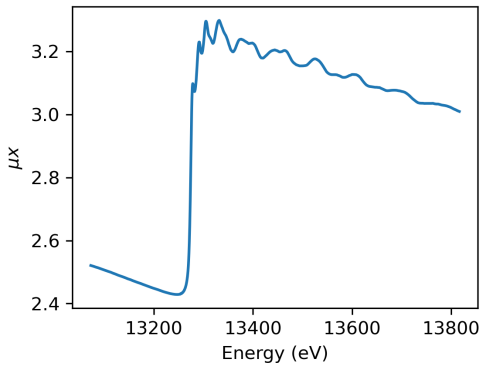
Absorption coefficient for sample transmission: $\mu x = log\left(\frac{i_0}{i_t}\right)$



Absorption coefficient for sample fluorescence: $\mu x = \left(\frac{i_{ff}}{i_0}\right)$; $\mu x = \left(\frac{xs\_roi1}{i_0}\right)$



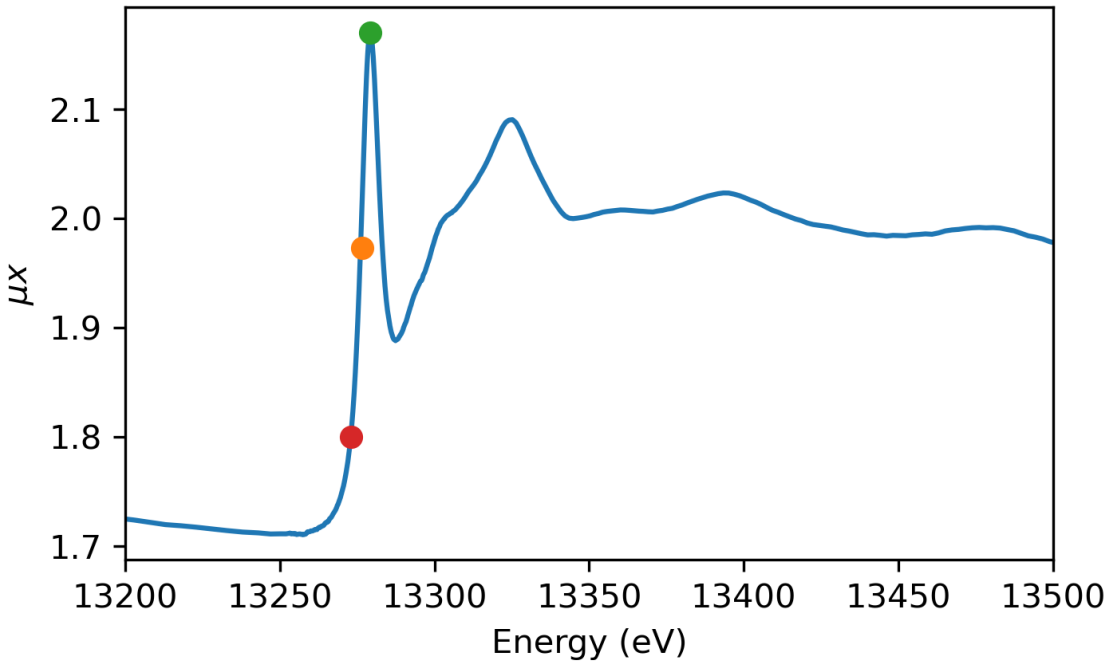
Absorption coefficient for reference transmission: $\mu x = log\left(\frac{i_r}{i_t}\right)$



Before processing some common steps

- Rebinning
- Energy alignment
- Merging

E_0



E_0 = White line

E_0 = first inflection point

E_0 = Tabulated value

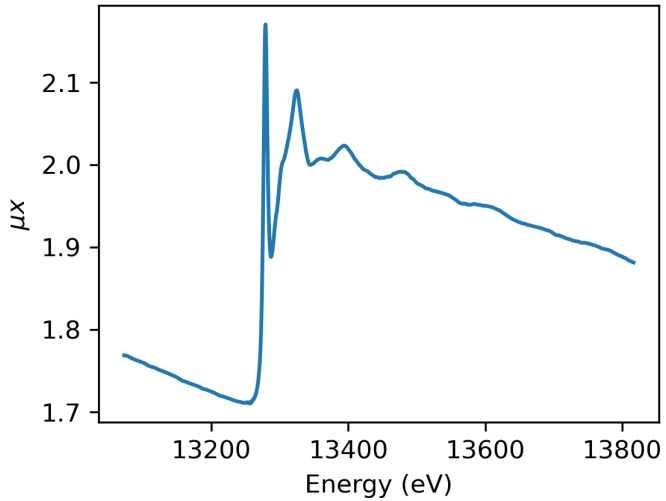
Some background

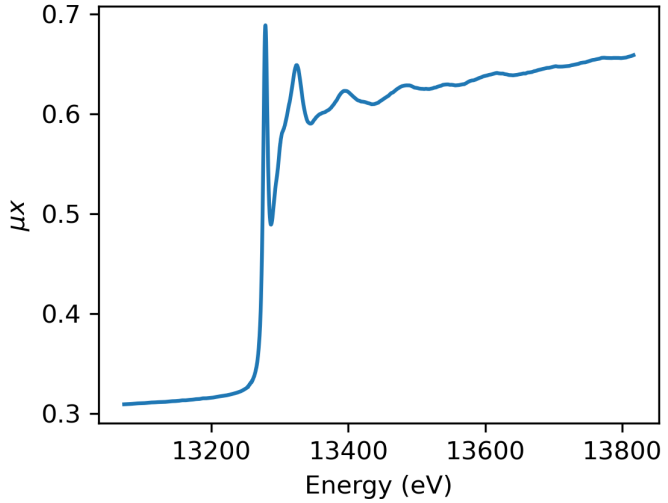
XAFS analysis based on comparison

- Fingerprinting
- Linear Combination Analysis
- Curve Fitting with Theoretical standards

$$\mu_{measured} = \mu_{extrinsic} + \mu_{intrinsic}\,(1 + \chi)$$

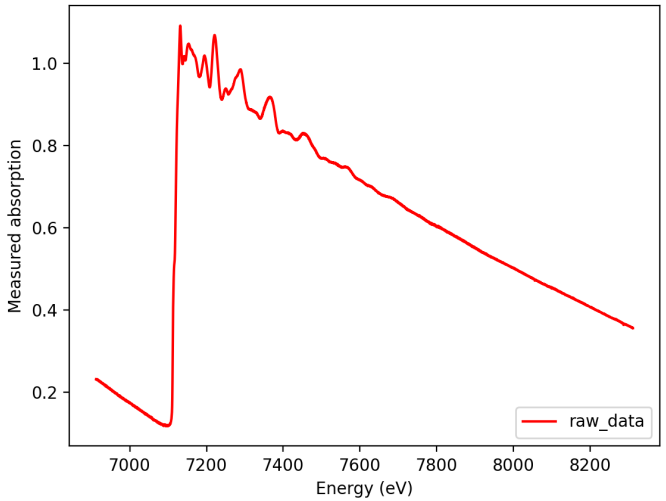
$$\frac{\mu_{measured}}{edge\ jump} = \frac{\mu_{extrinsic}}{edge\ jump} + \frac{\mu_{intrinsic}}{edge\ jump}\,(1 + \chi)$$

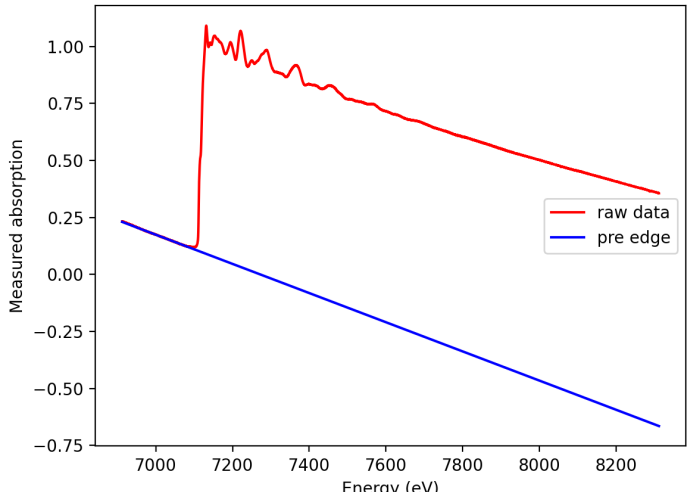


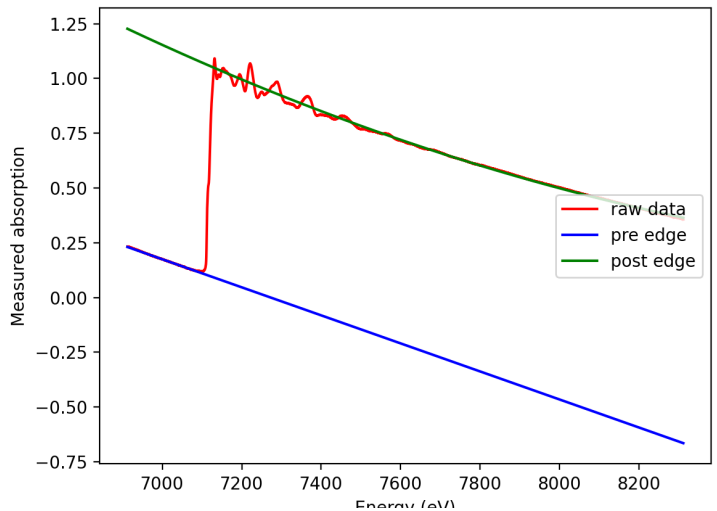


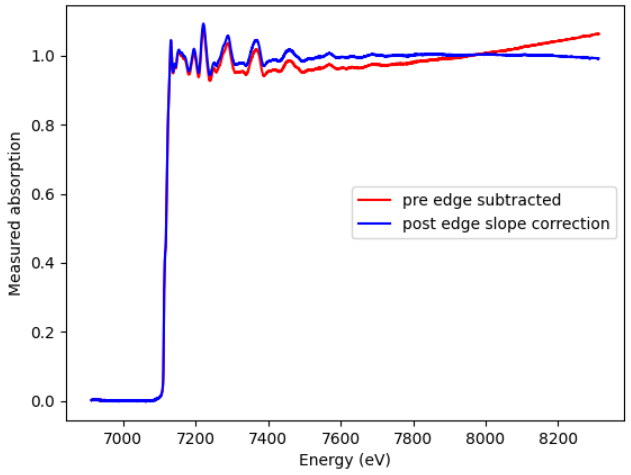
Pre and Post-edge background subtraction

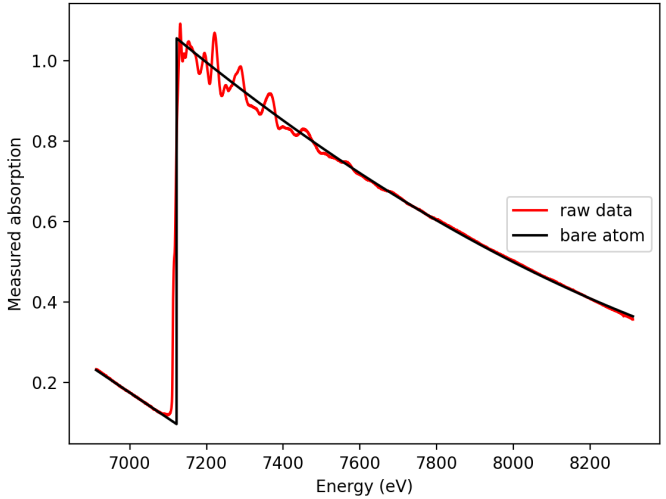
$$\mu = CE^3 - DE^4 \ (Victoreen\ Equation\ imperical)$$

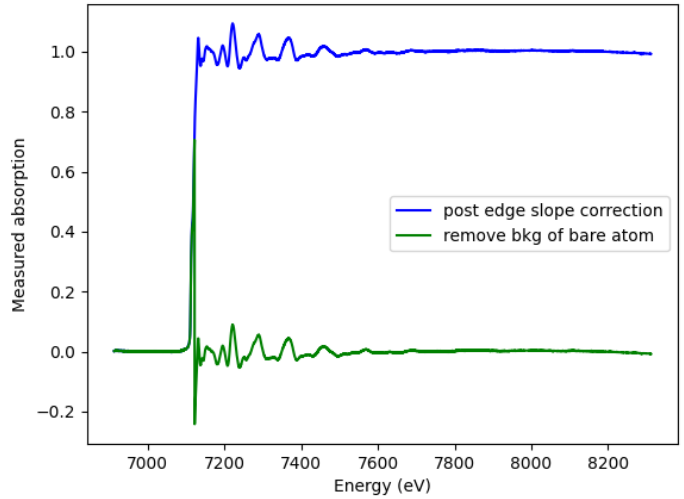








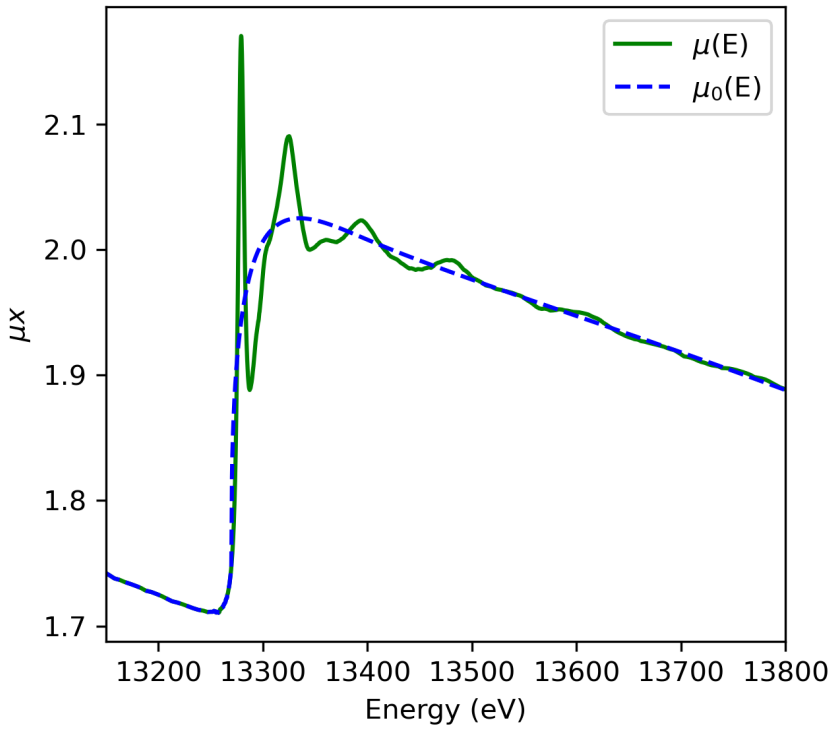


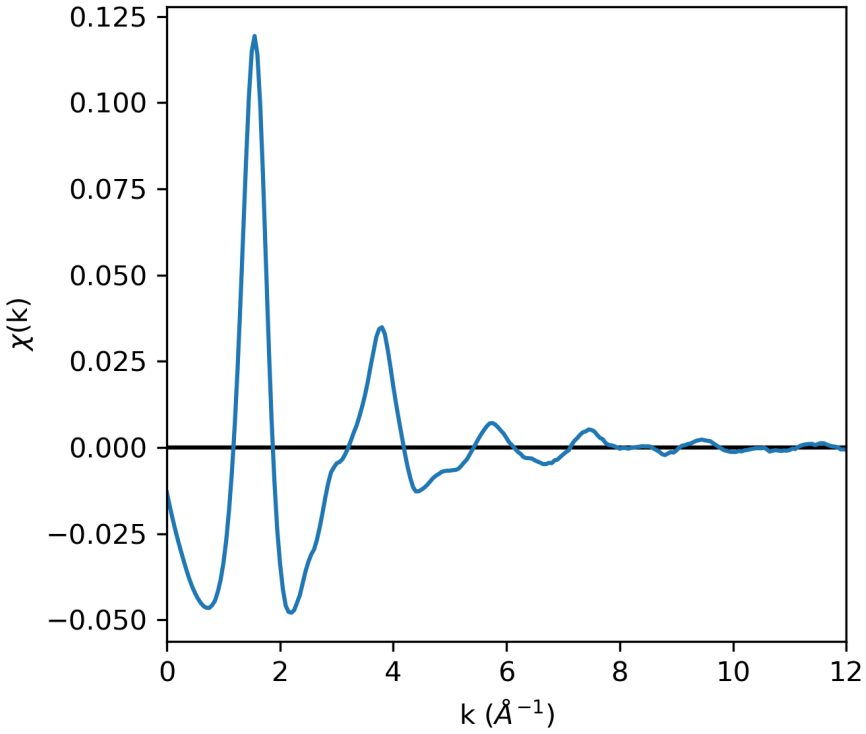


Conversion of E to k

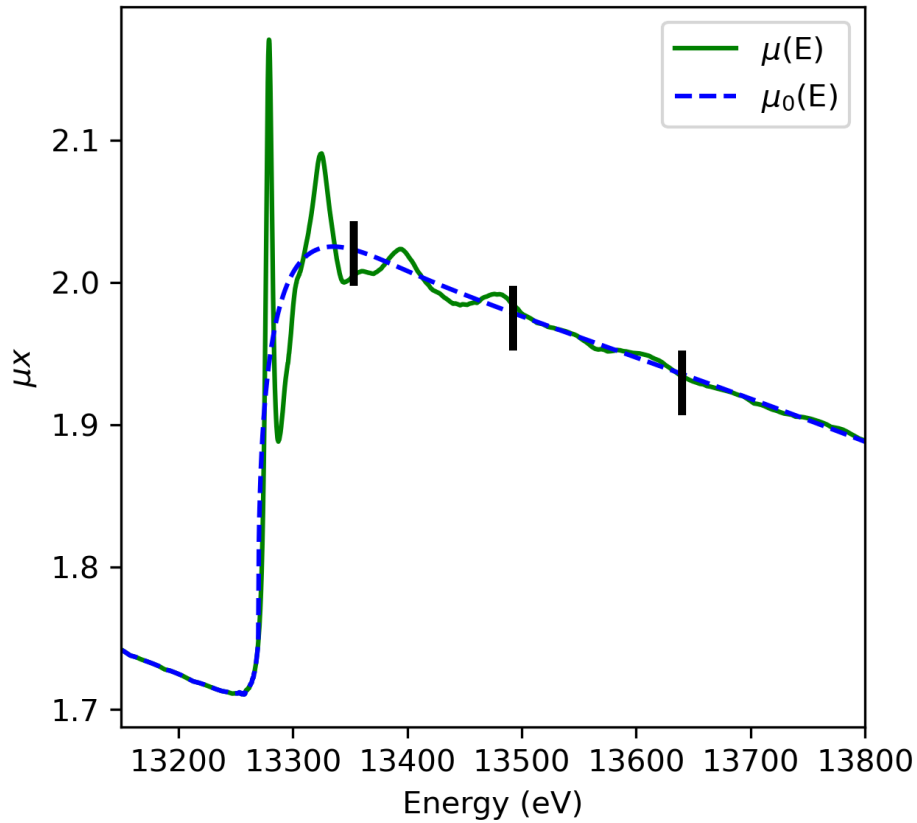
$$k = \sqrt{\frac{2m\left(E - E_0^{exp}\right)}{\hbar^2}}$$

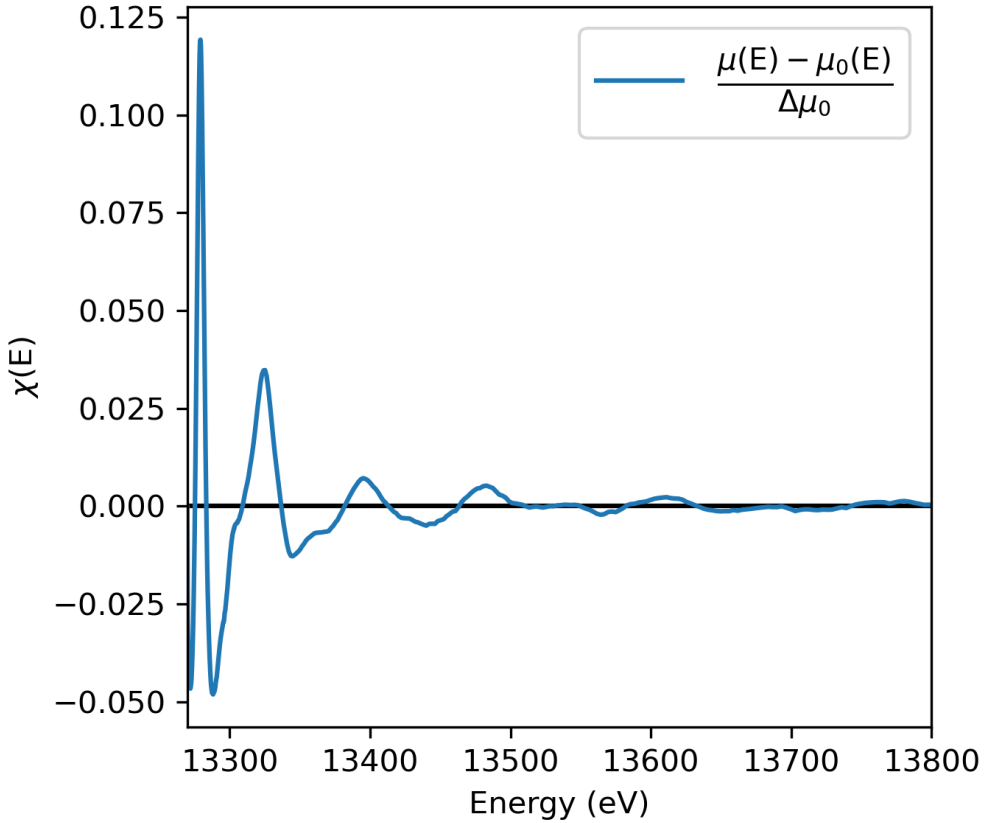
$$k = \sqrt{0.2625\left(E - E_0^{exp}\right)}$$



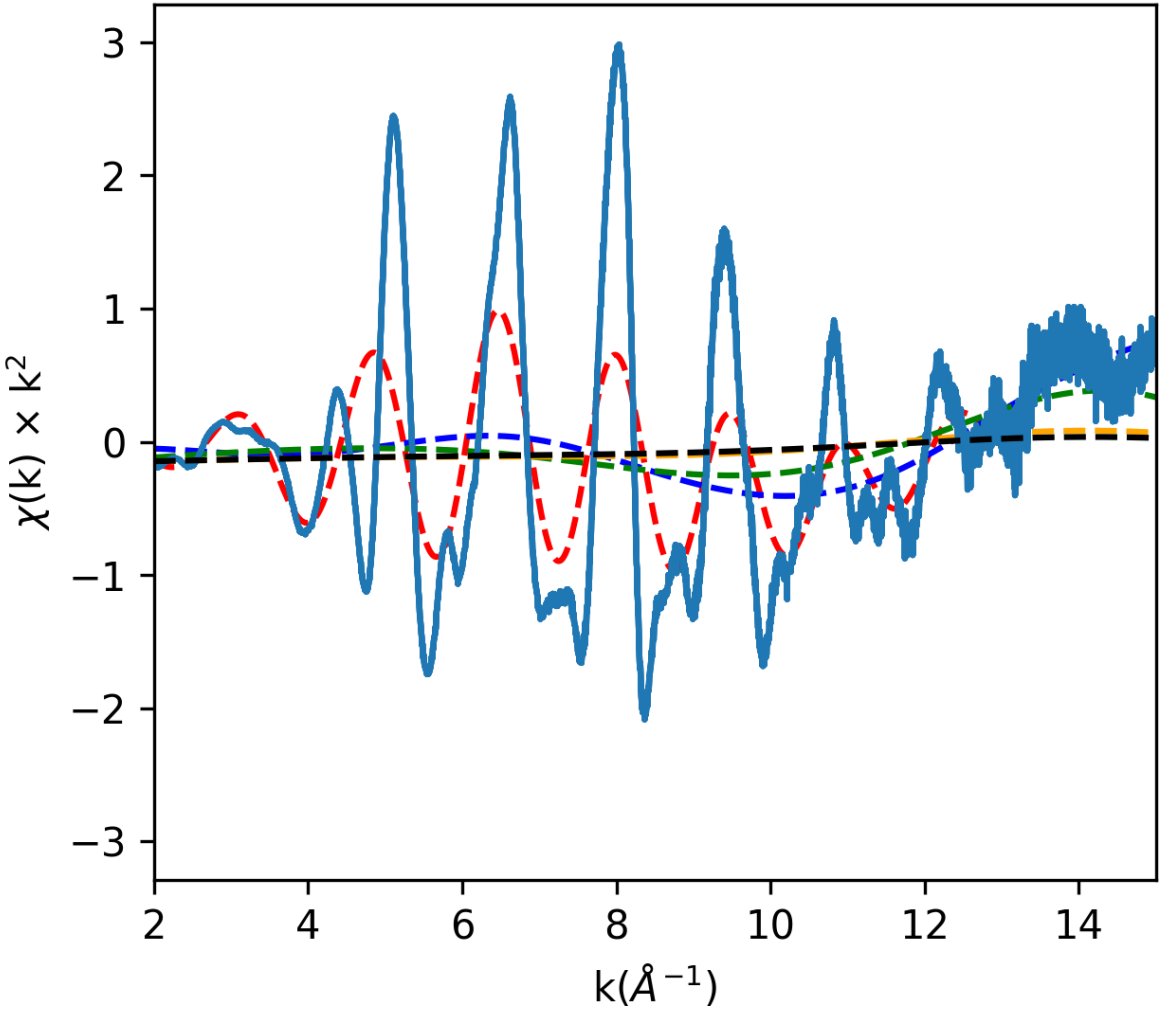


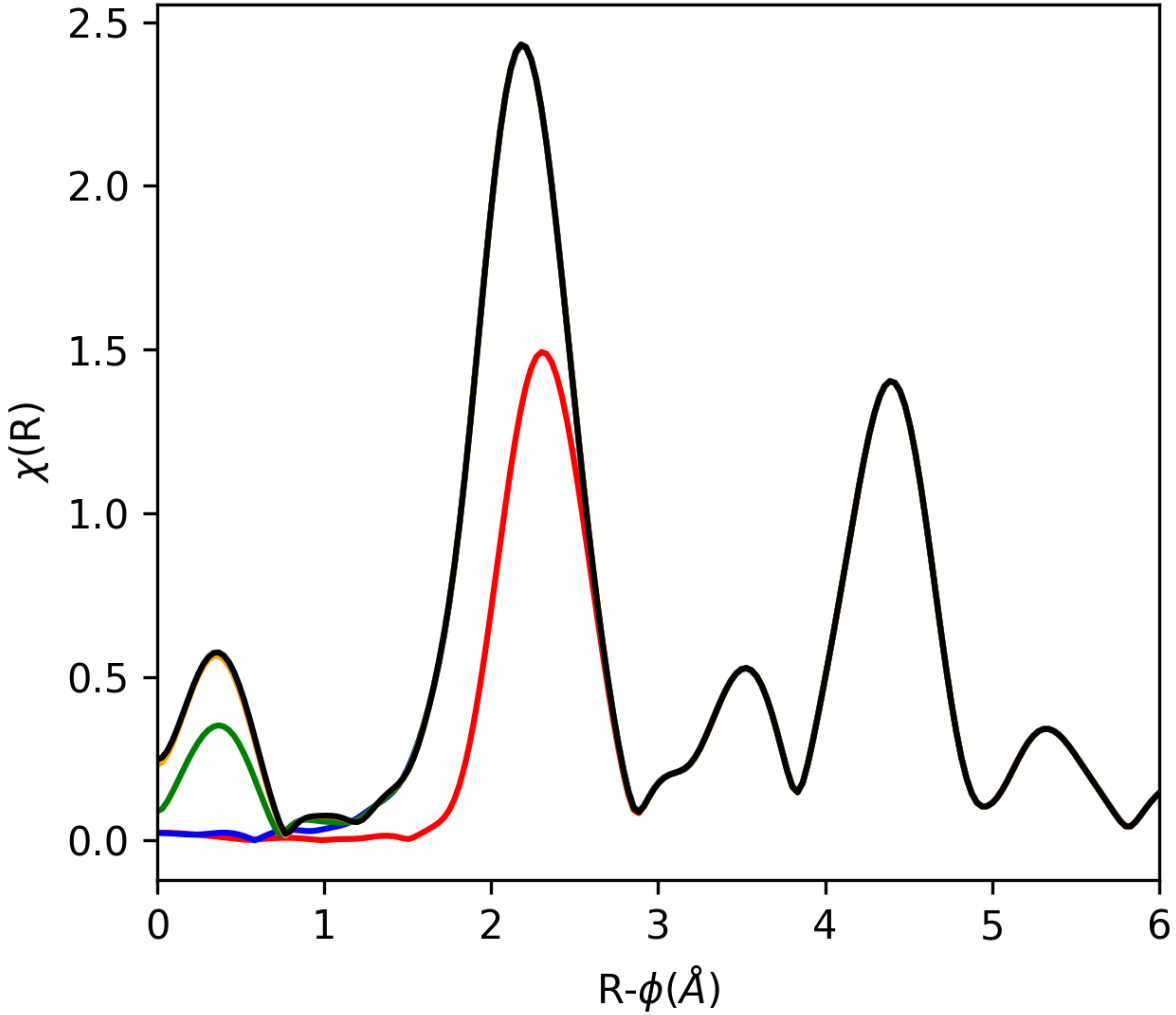
EXAFS extraction



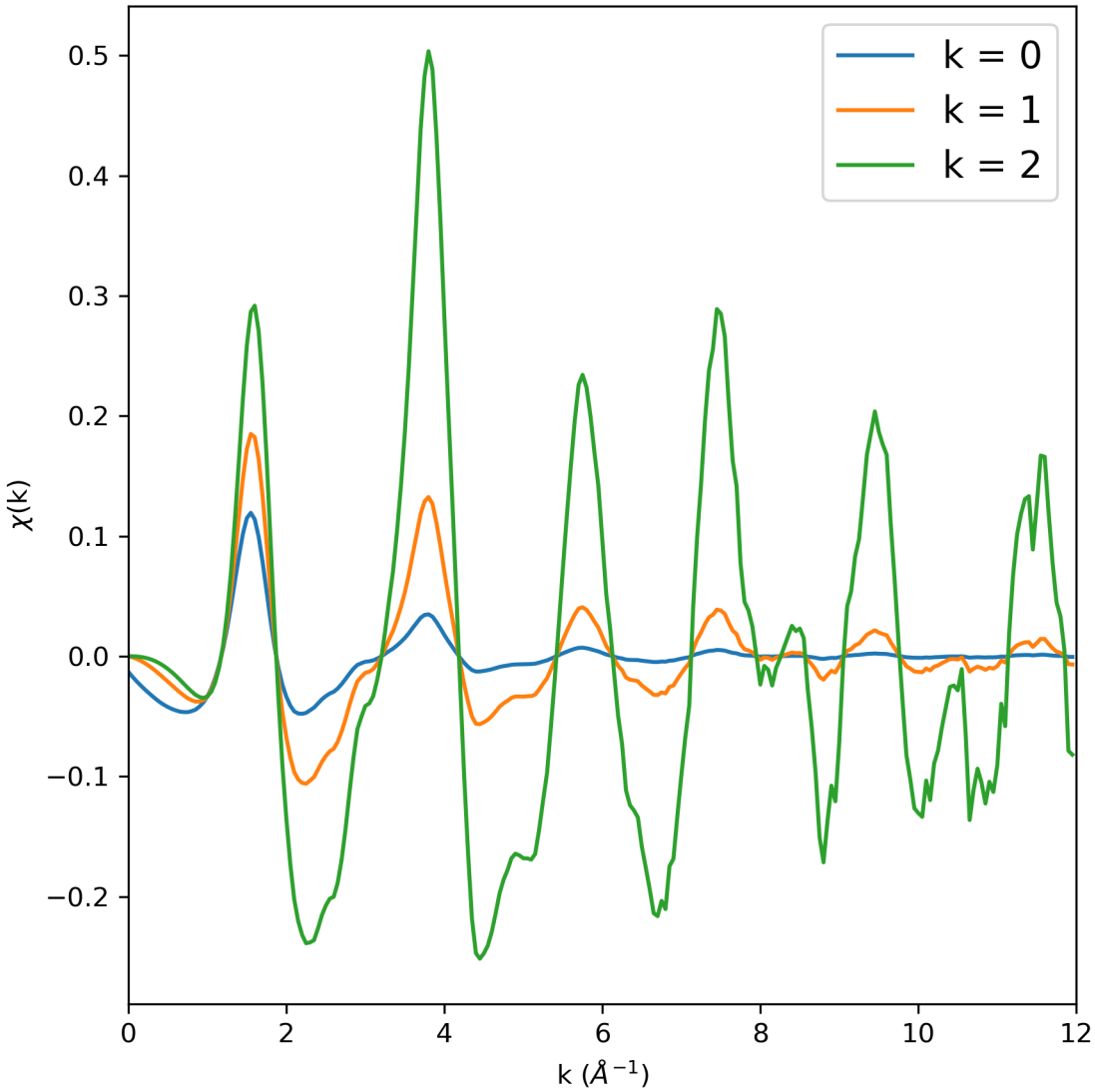


EXAFS extraction





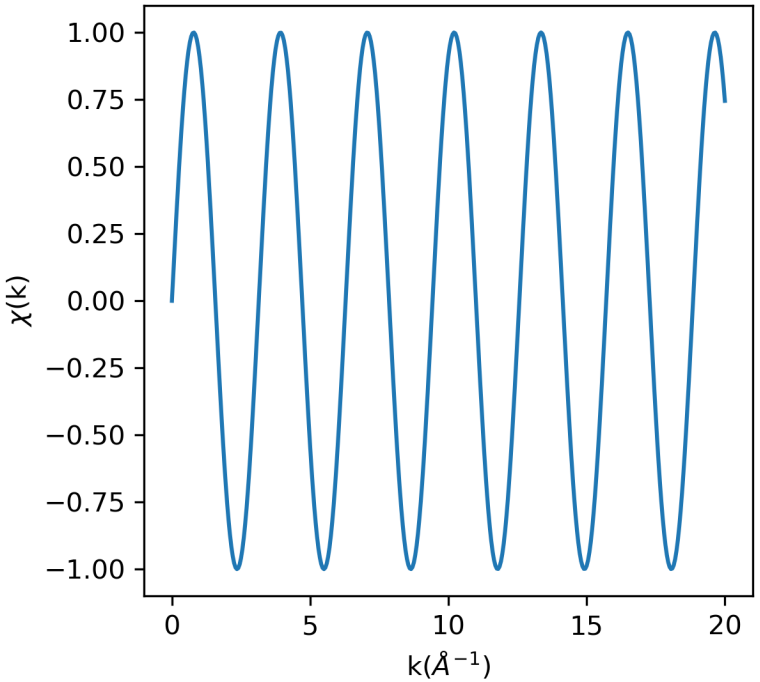
k weighting 1, 2, 3



This procedure is important to prevent the larger amplitude oscillations from dominating the smaller ones in determining interatomic distances, which depends only on the frequency and not the amplitude.

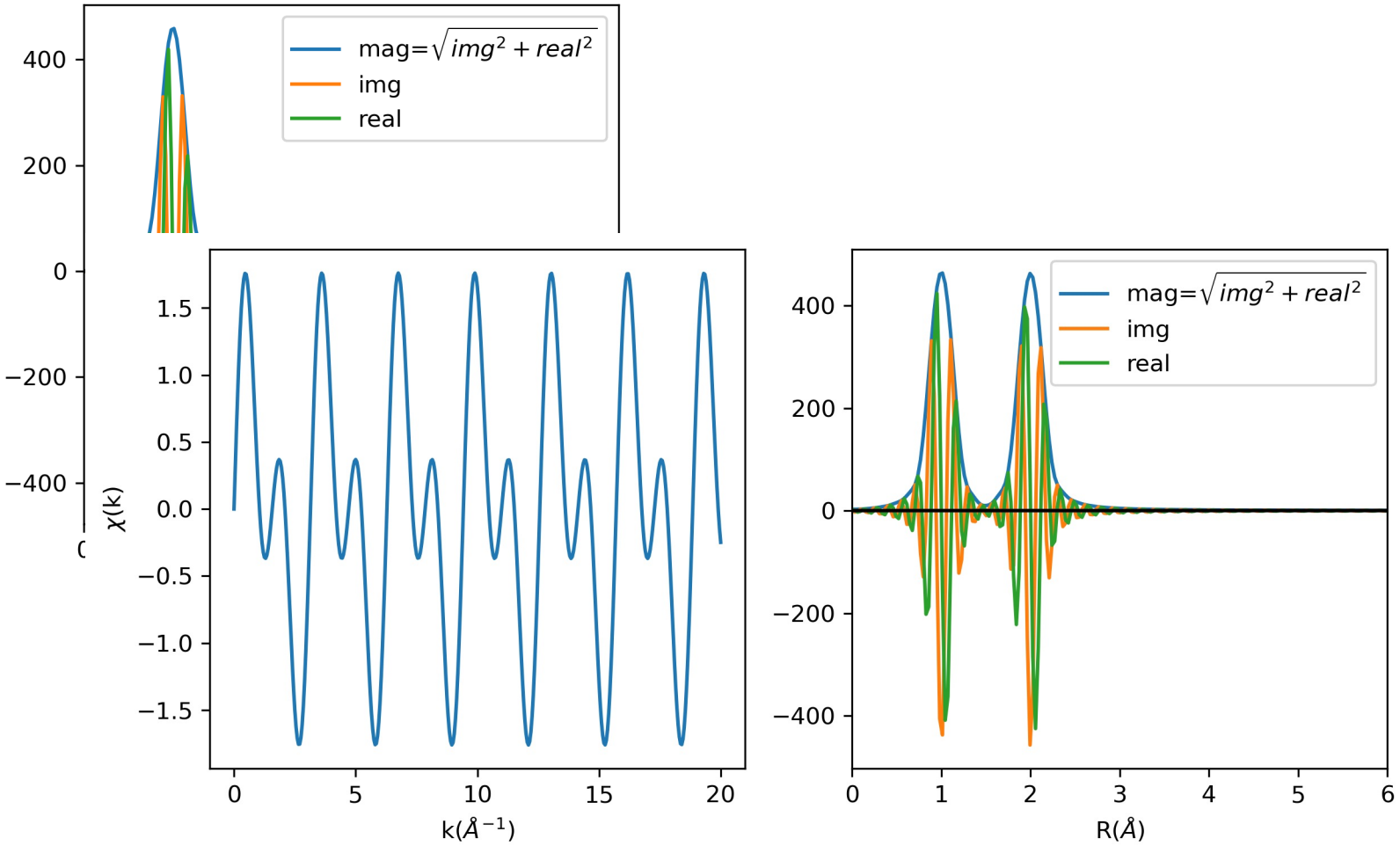
k weight 1, 2, 3 for $Z > 57$, $36 < Z < 57$ and $Z > 36$
Teo and Lee (1979)

Fourier Transform



FT of a sine wave with phase 2k

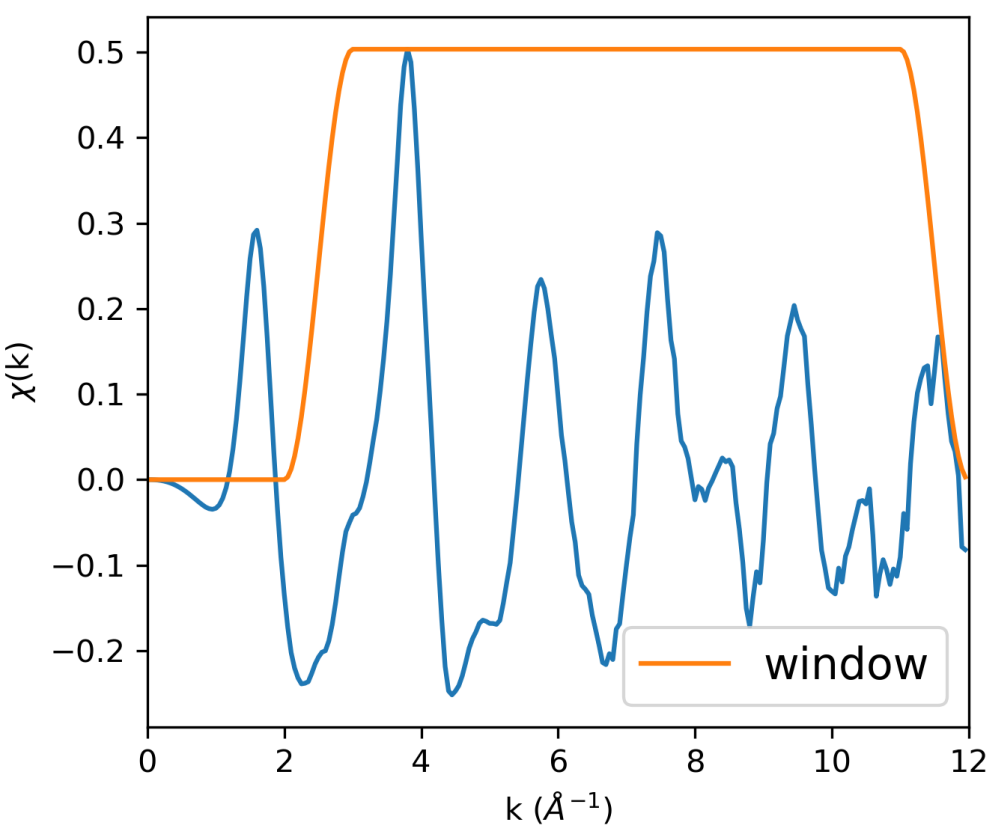
$$\chi(k) = \sin(2k)$$

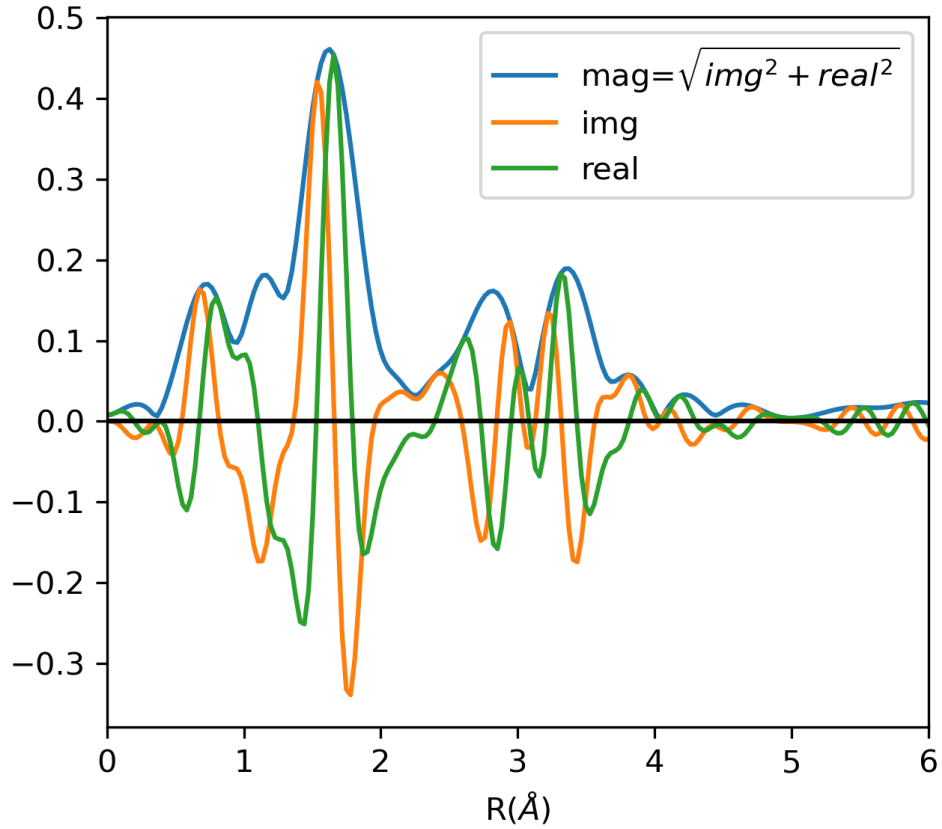


FT of two sine waves

$$\chi(k) = \sin(2k) + \text{sine}(4\text{k})$$

Fourier Transform of PtO_2





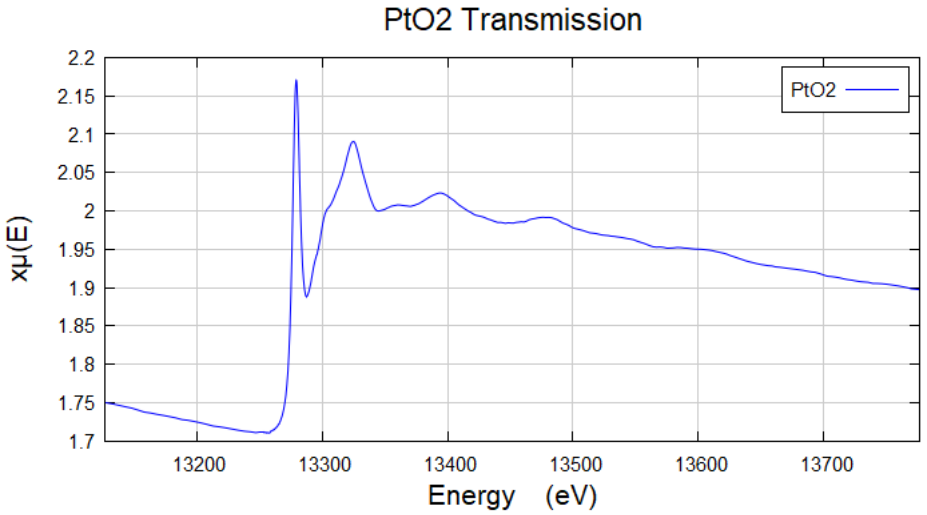
XAFS books

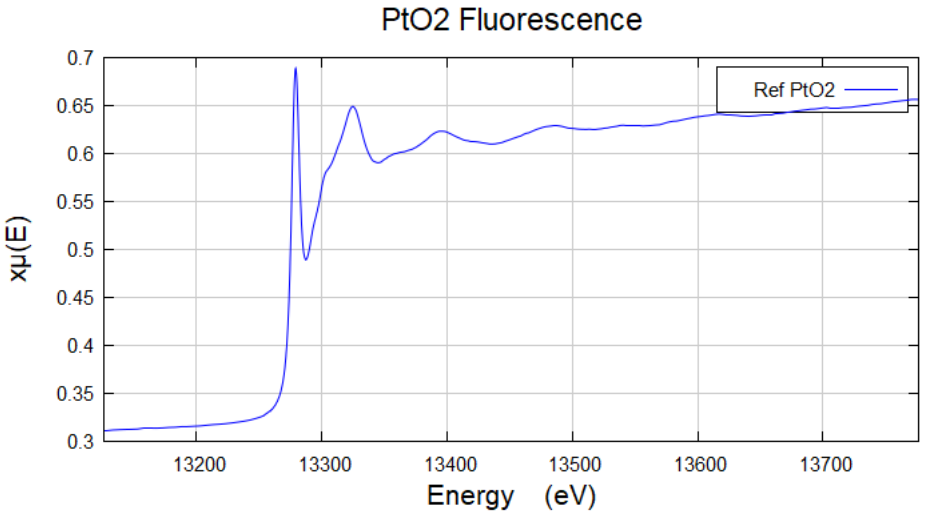
Introduction to XAFS: A Practical Guide to X-ray Absorption Fine Structure Spectroscopy
Grant Bunker

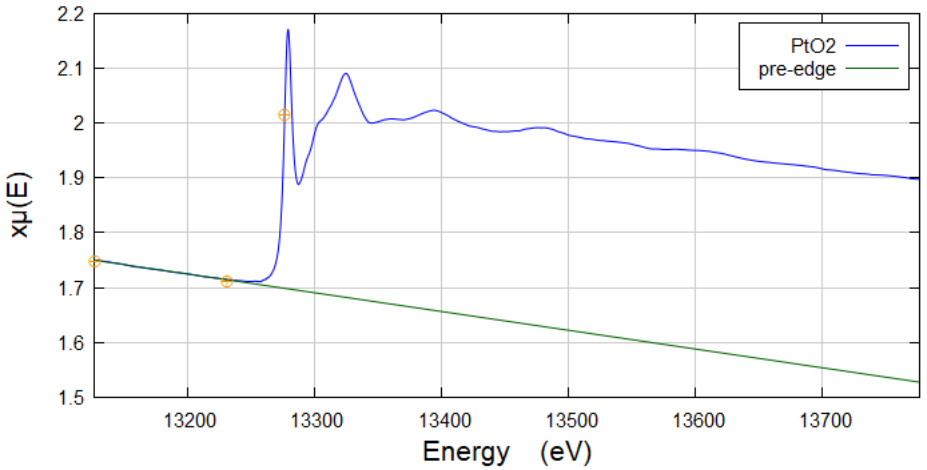
EXAFS: Basic Principles and Data Analysis
Dr. Boon K. Teo

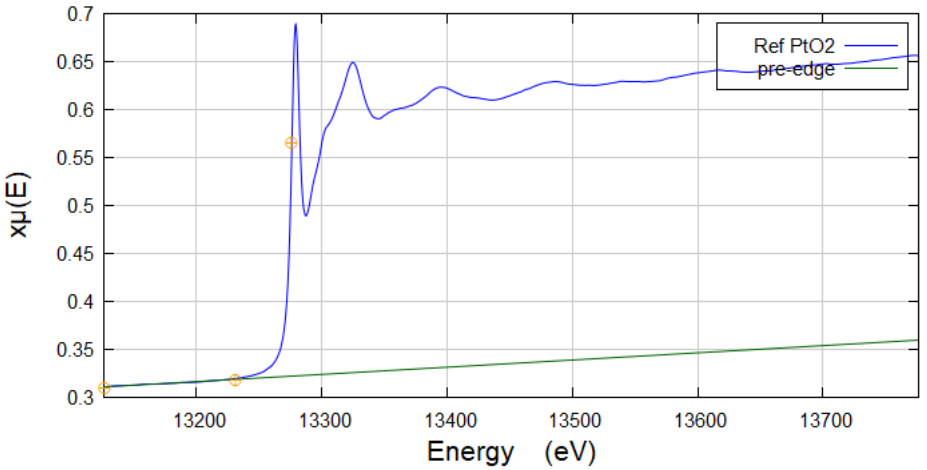
XAFS for Everyone
Scott Calvin

Pre-edge background subtraction



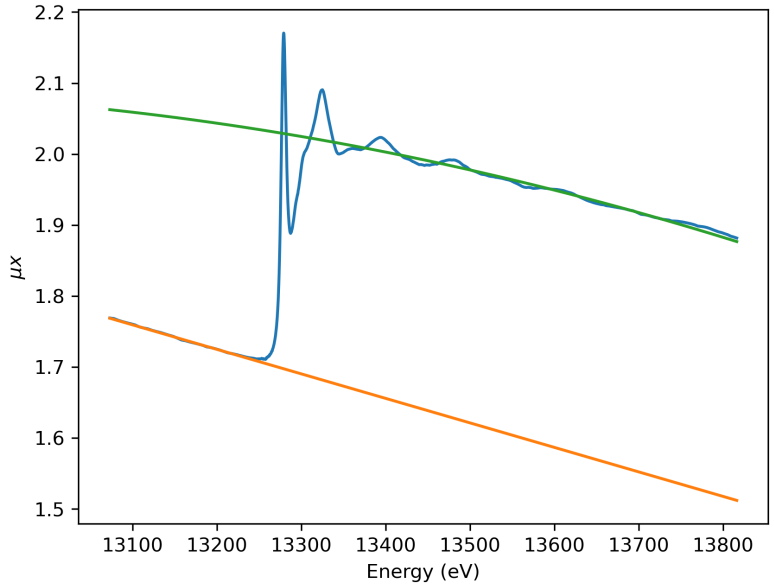


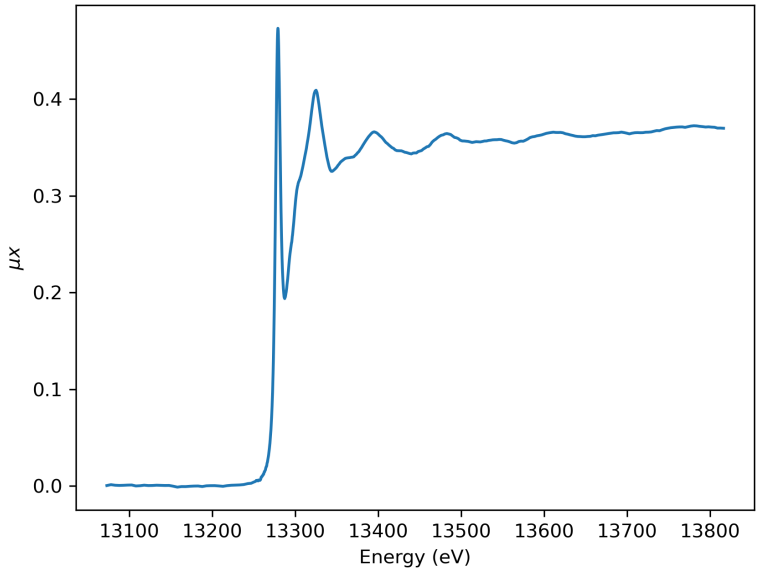


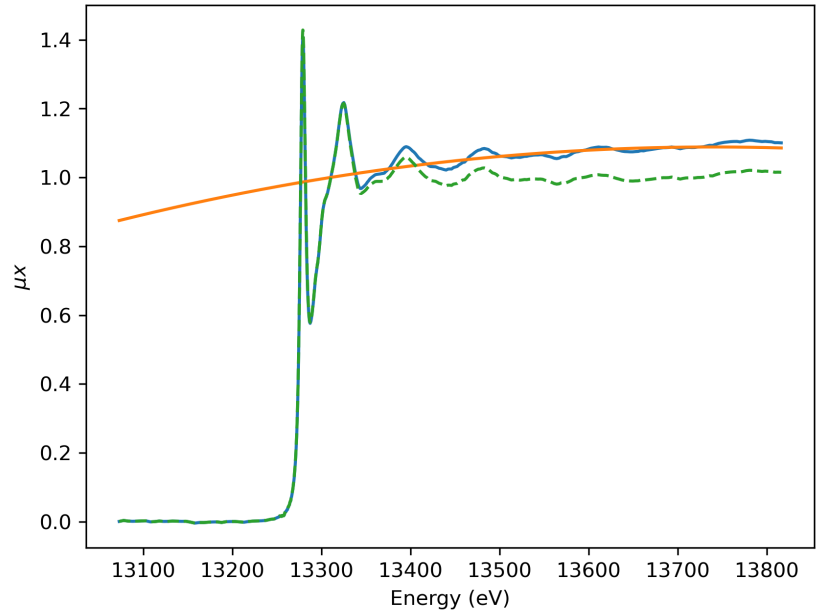


$$\mu = CE^3 - DE^4 \ (Victoreen\ Equation\ imperical)$$

Post-edge background subtraction







Pre-edge background

Post-edge background

Pre-edge subtracted

Post-edge subtracted