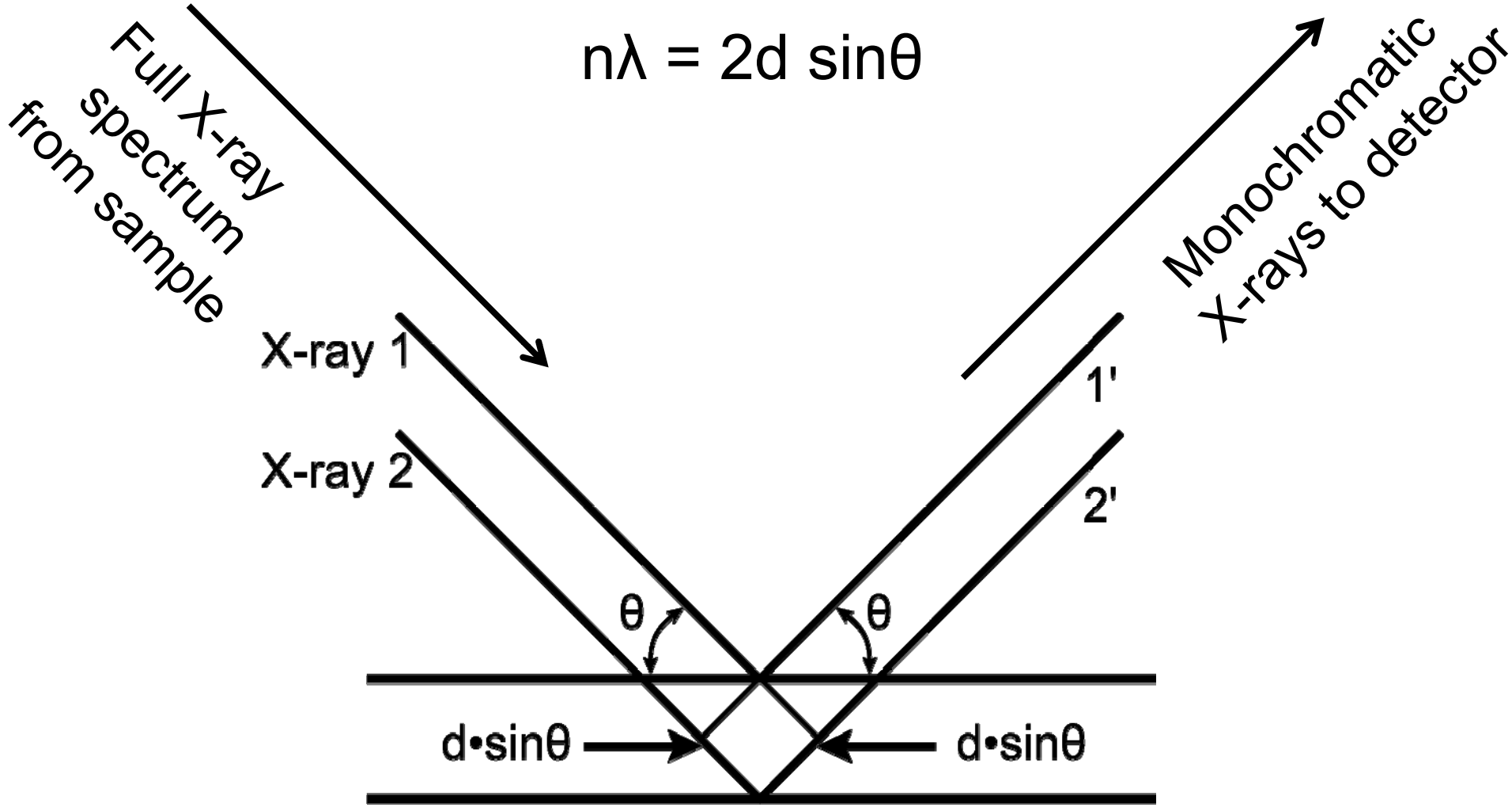
ThermoFisher SCIENTIFIC

WDS spectrometer technologies:
Not all WDS spectrometers are created equally

Steve Seddio

The world leader in serving science

What is WDS?



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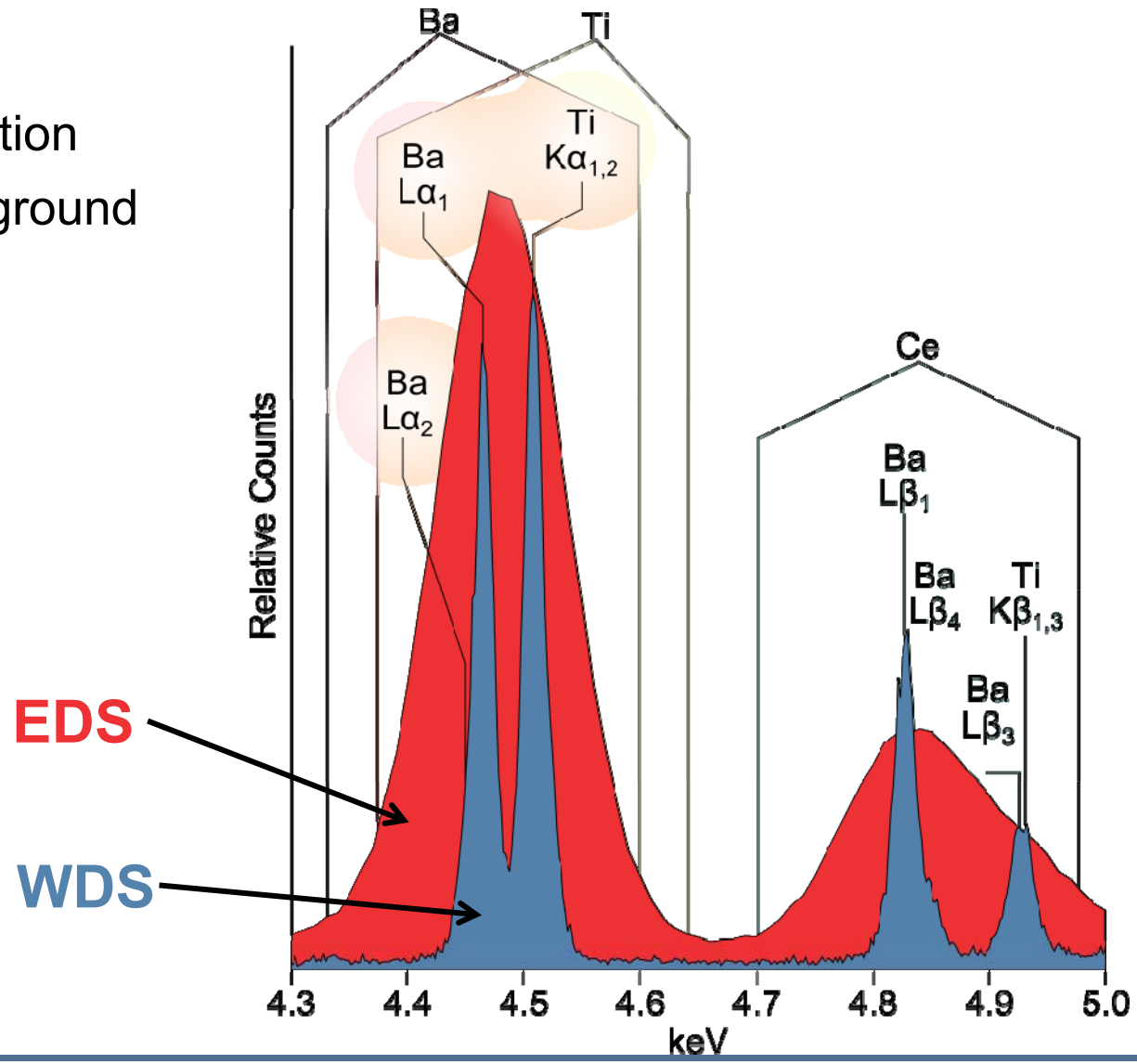
Why WDS?

- The result:
  - Best energy resolution
  - Best peak-to-background



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The Challenge of WDS

- Hypothetical sample in an SEM
- For microanalysis, sample is normal to the electron beam

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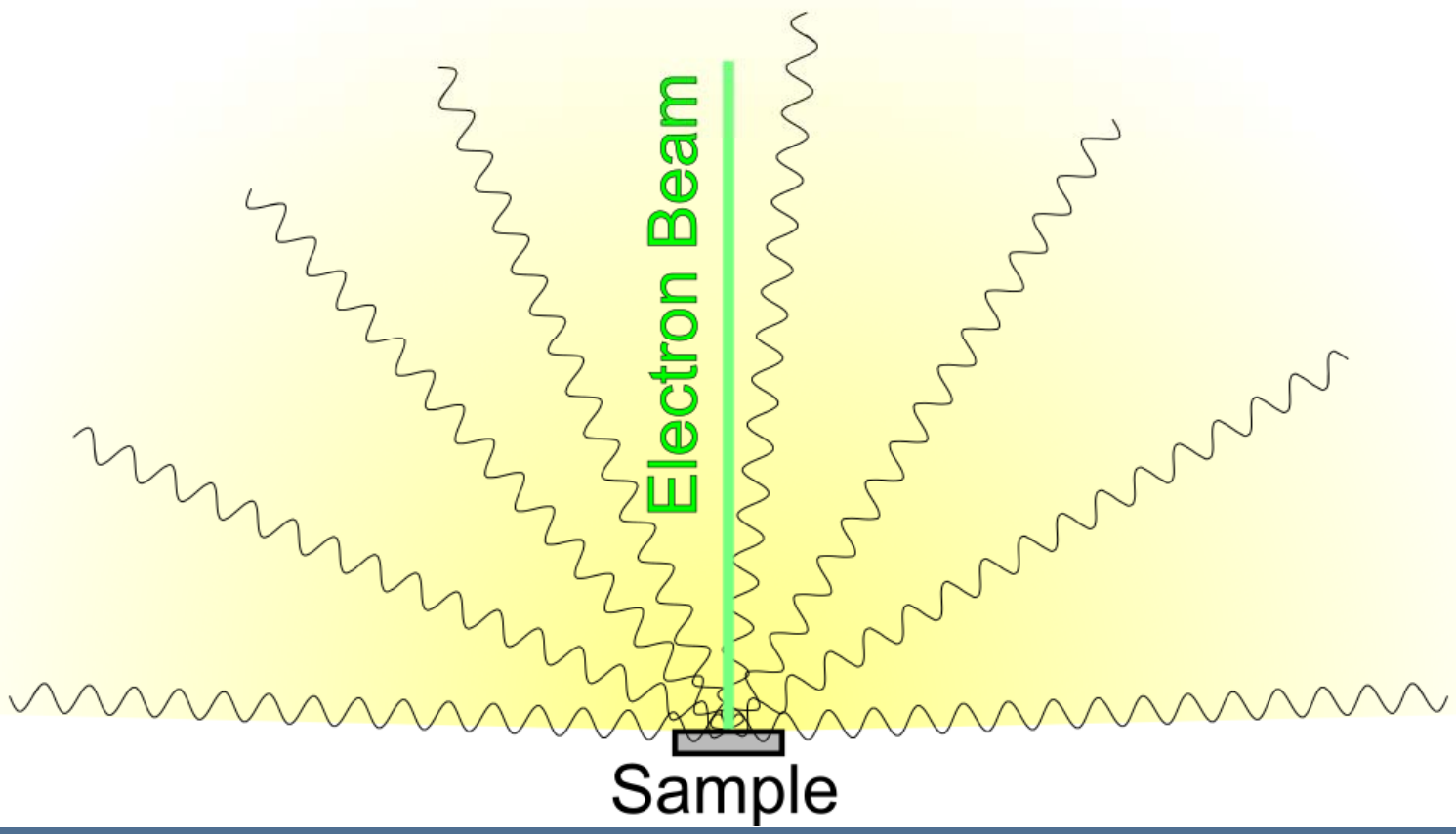
The Challenge of WDS

- X-rays are emitted from the excitation volume in a 3-dimensional hemispherical wavefront



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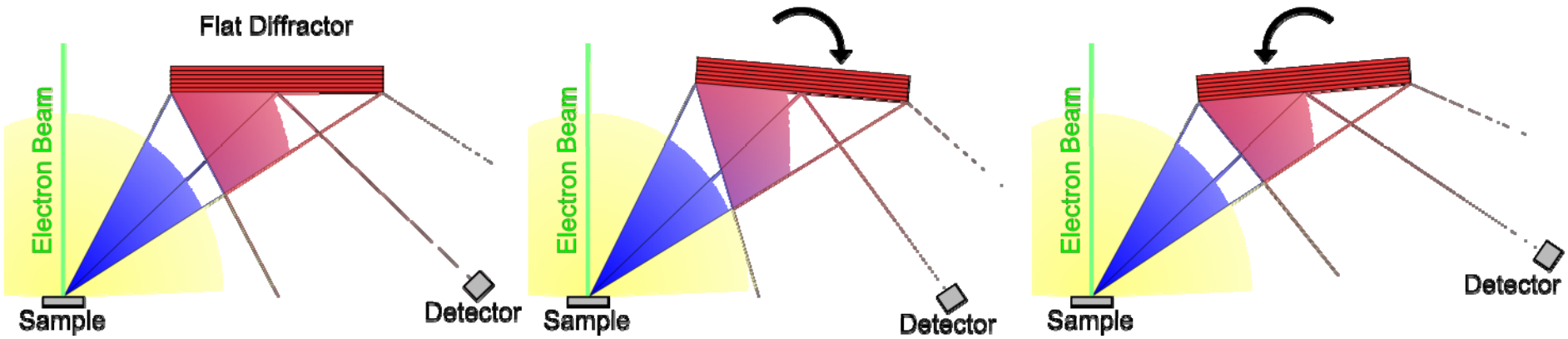
The Challenge of WDS

- Get diverging X-rays to diffract off of a diffractor and then be counted by a detector at a meaningful intensity



- Flat diffractors:
  - Simple geometry
  - X-rays interact with diffractor with different θ
  - X-rays continue to diverge after diffraction → low count rates at detector.

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Meeting the Challenge

- All WDS spectrometers consist of
  - Diffractor
  - Proportional counter (a.k.a., detector)
    - Flowing P10 gas (90% Ar, 10% CH_4)
    - Sealed Xe
- Some WDS spectrometers include an X-ray optic near sample
- There are two types of spectrometers that have been developed to meet the WDS challenge
  - Rowland circle WDS
  - Parallel beam WDS

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Meeting the Challenge: Intensity

- For WDS, the count rate is a function of
  - X-ray generation
    - Accelerating voltage
    - Beam current
    - Vacuum
    - Sample composition
    - Sample preparation
  - Solid Angle and Optic
    - Diffractor size
    - Diffractor distance from sample
    - Size of optic
    - Reflectance and/or transmittance of optic
  - Other
    - Diffractor “reflectance”
    - Type of gas in detector
    - Pressure of gas in detector
    - Transmittance of detector window
    - Ambient temperature
    - Presence / transmittance of spectrometer window

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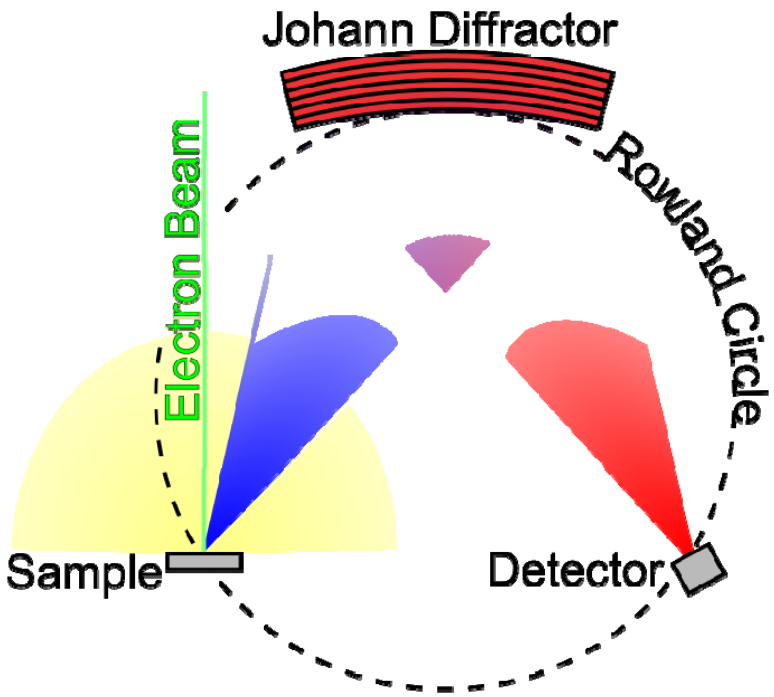
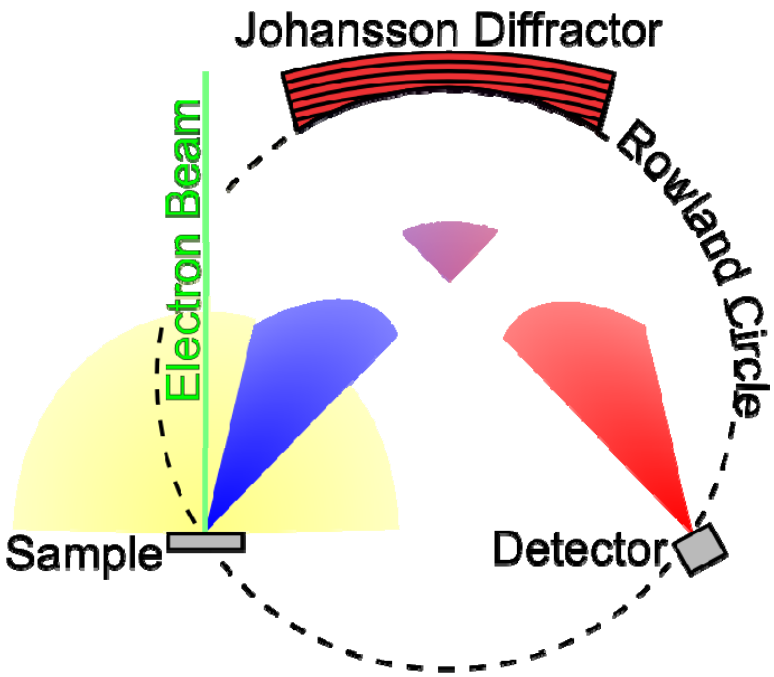
Rowland Circle WDS: The Classic Solution (c. 1882)

- Curved diffractors focus the diffracted X-rays on to detector





- Requires complex motion so that the sample, diffractor, and detector remain on a circle of fixed radius

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Rowland Circle WDS: The Classic Solution

Exciting RC-WDS animation here!

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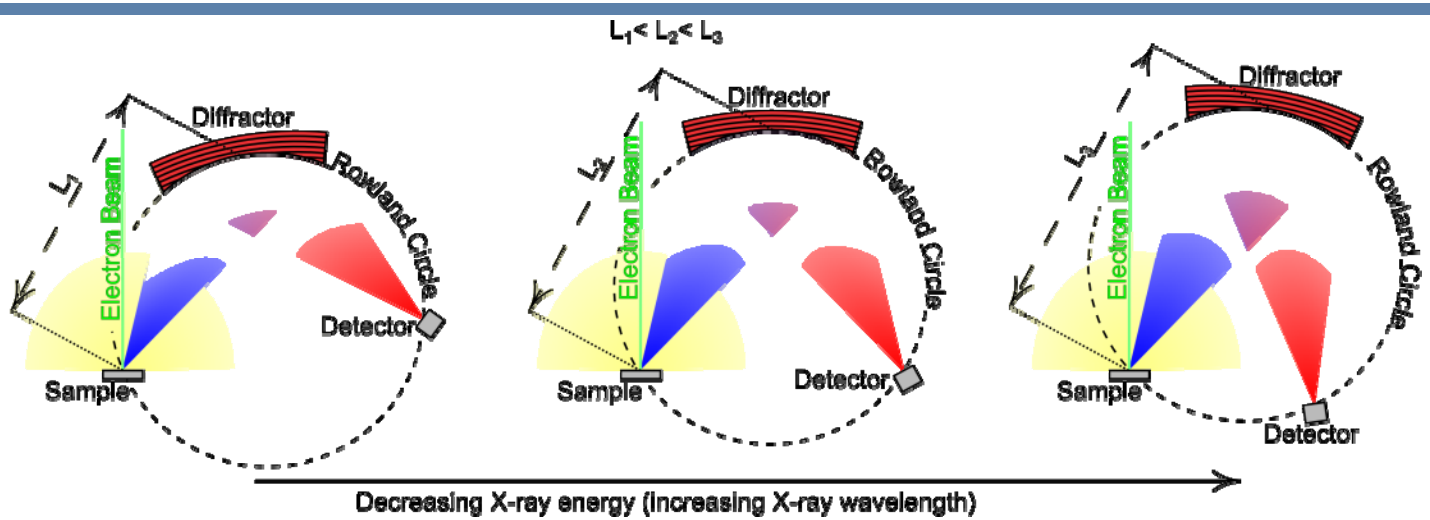
Rowland Circle WDS: The Classic Solution



- To maintain RC geometry, diffractor must move away from sample for low-energy X-rays

$$L = 2R\,sin\theta = \frac{R\lambda}{d}$$

- The result is a decrease in solid angle → low intensity (c/(s × nA)) for low energy X-rays

$$\Omega = \frac{A}{L^2}$$

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Rowland Circle WDS: The Classic Solution

- Diffractor rotation affects solid angle

$$\Omega = \frac{A\sin\theta}{L^2} = \frac{A\sin\theta}{(2R\sin\theta)^2} = \frac{A}{4R^2\sin\theta}$$

$$\Omega = \frac{A\frac{\lambda}{2d}}{L^2} = \frac{A\frac{\lambda}{2d}}{\left(\frac{R\lambda}{d}\right)^2} = \frac{Ad}{2R^2\lambda}$$

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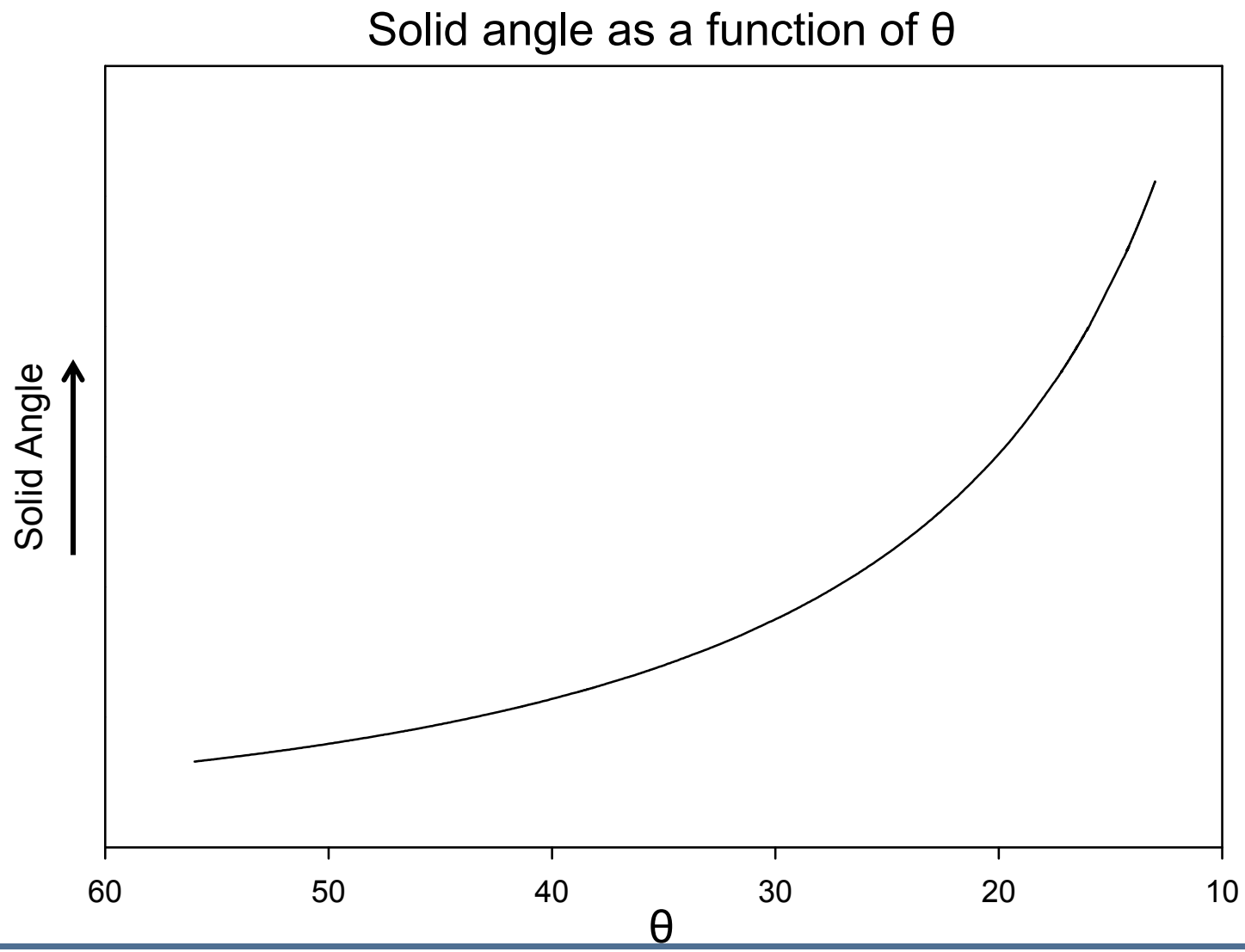
Rowland Circle WDS: Solid Angle



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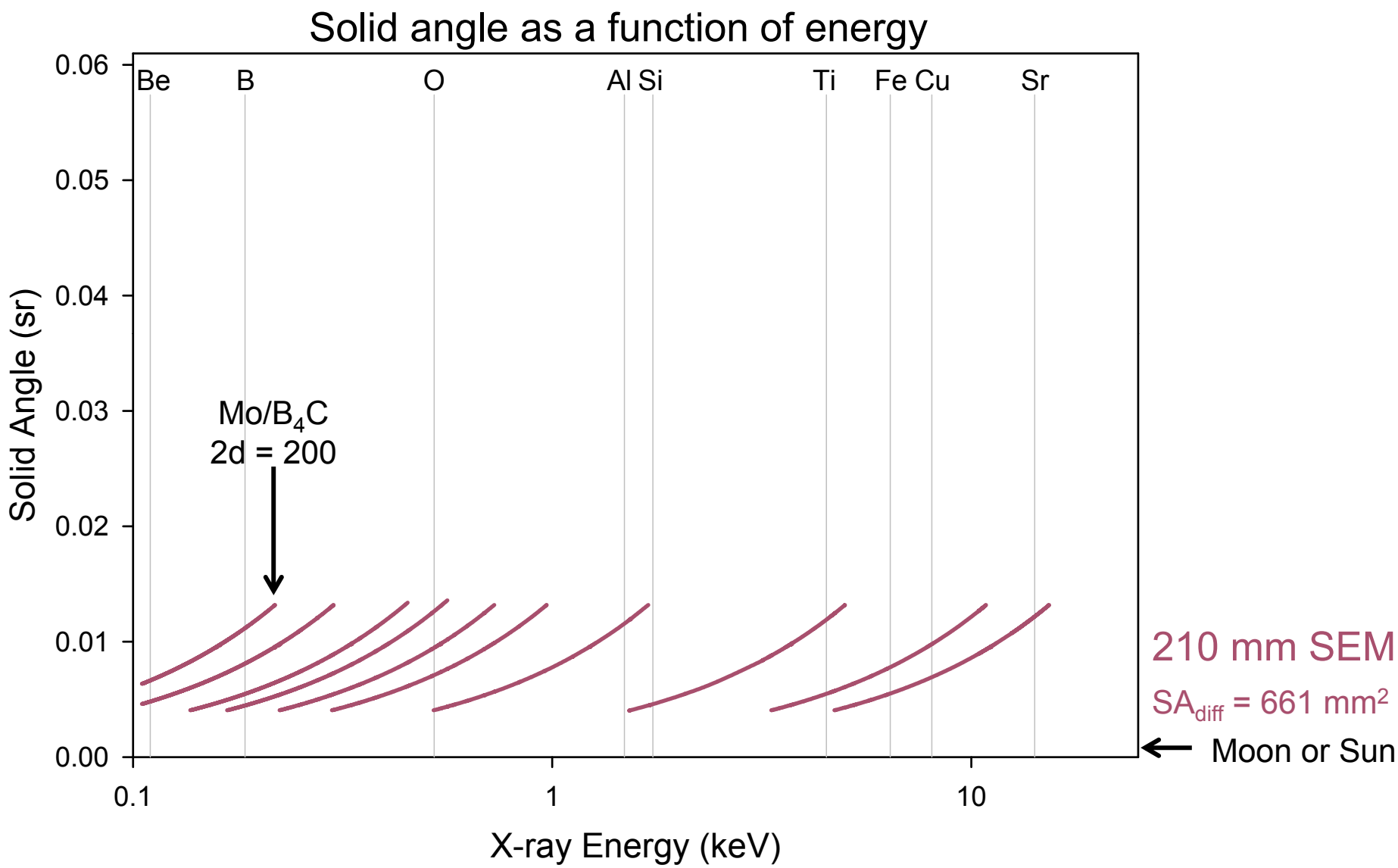
Rowland Circle WDS: SEM Solid Angle



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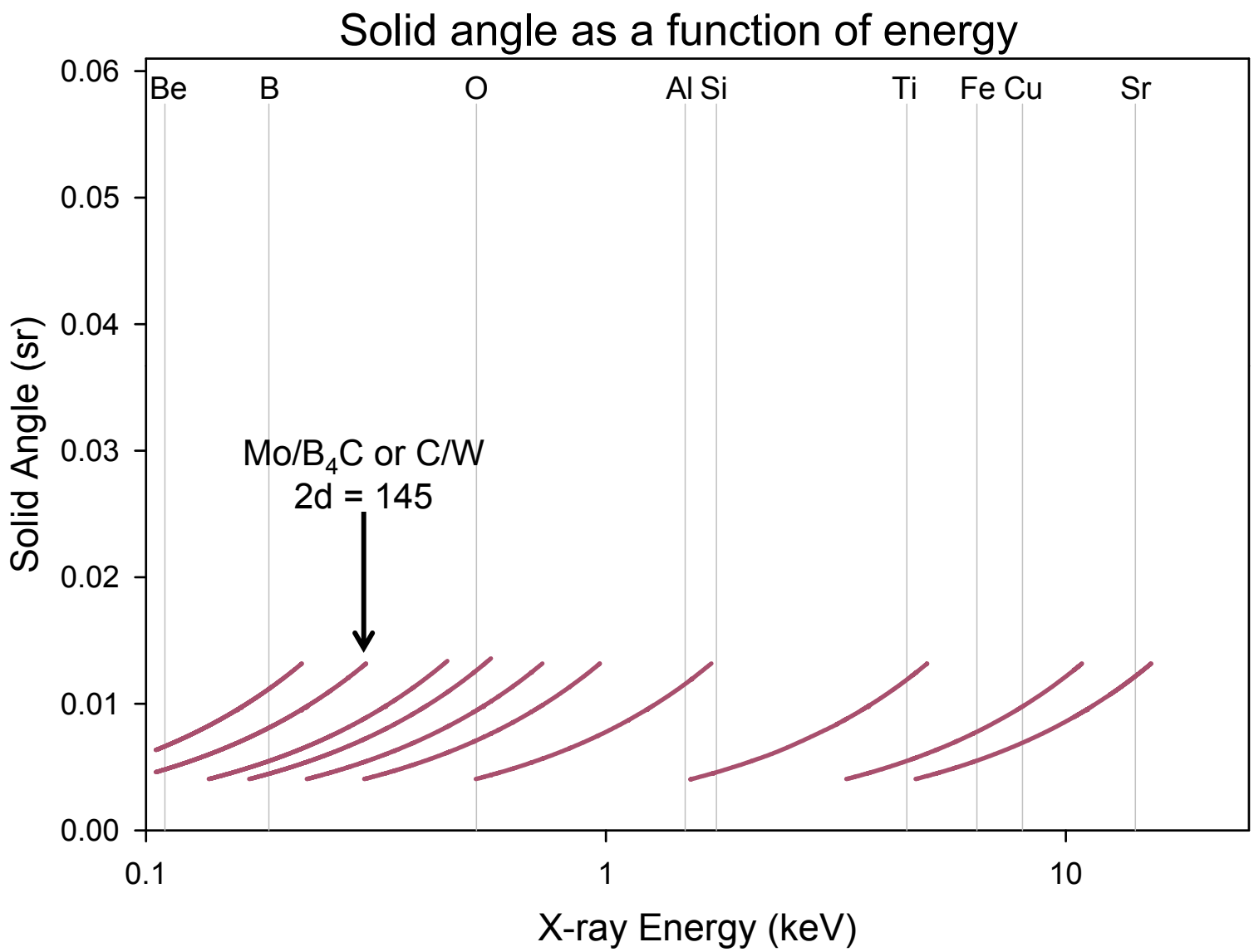
Rowland Circle WDS: SEM Solid Angle



210 mm SEM

SA_{diff} = 661 mm^2

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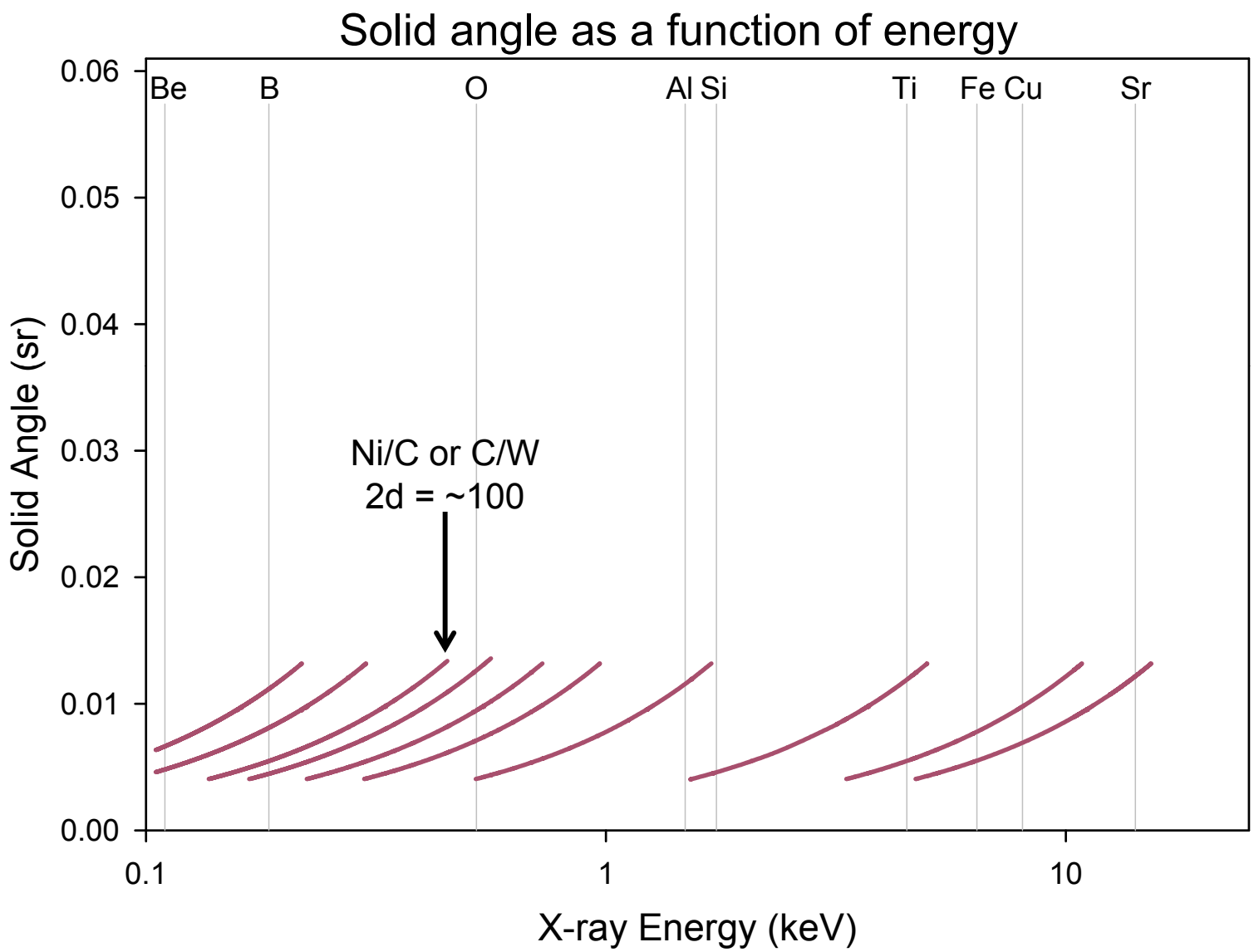
Rowland Circle WDS: SEM Solid Angle



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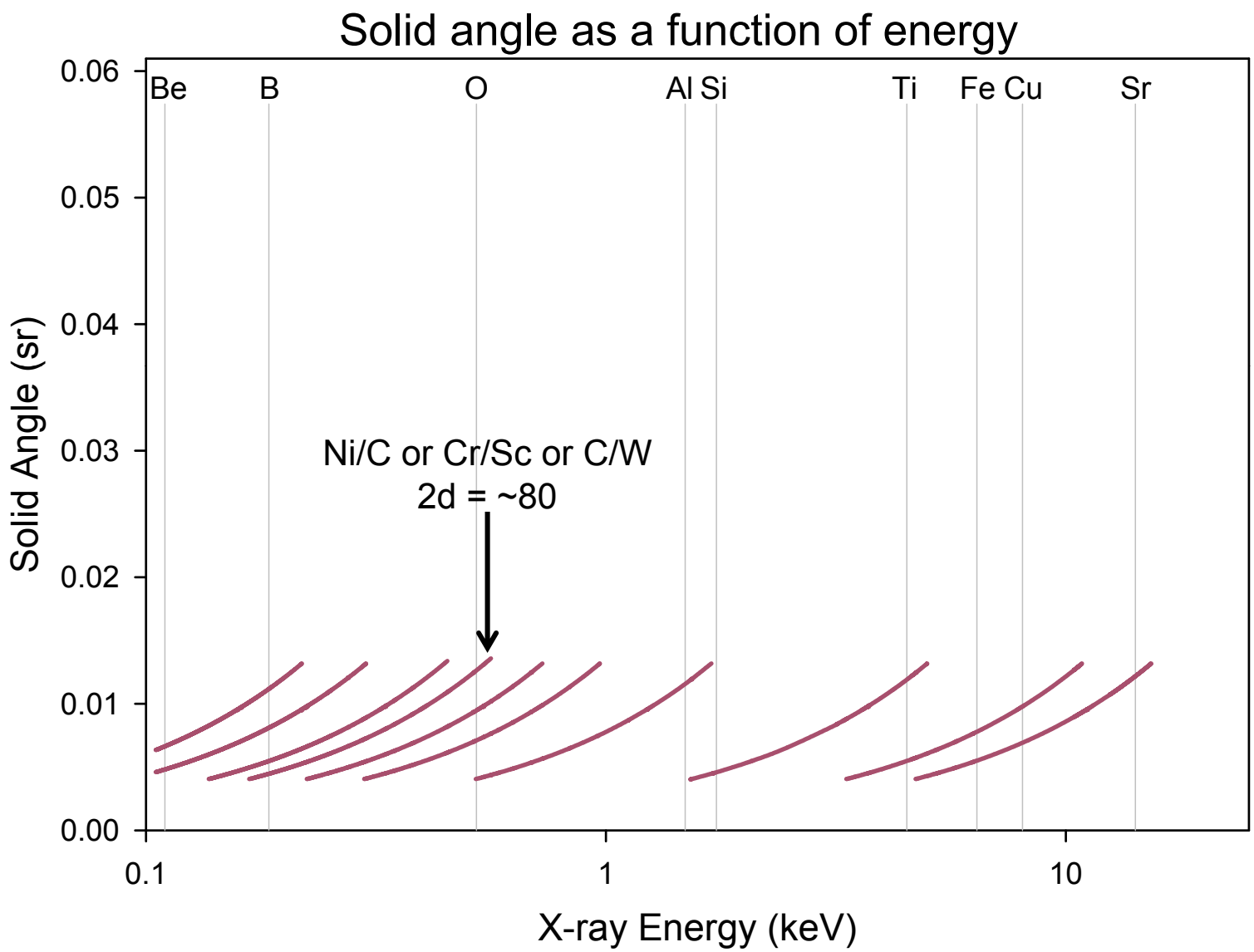

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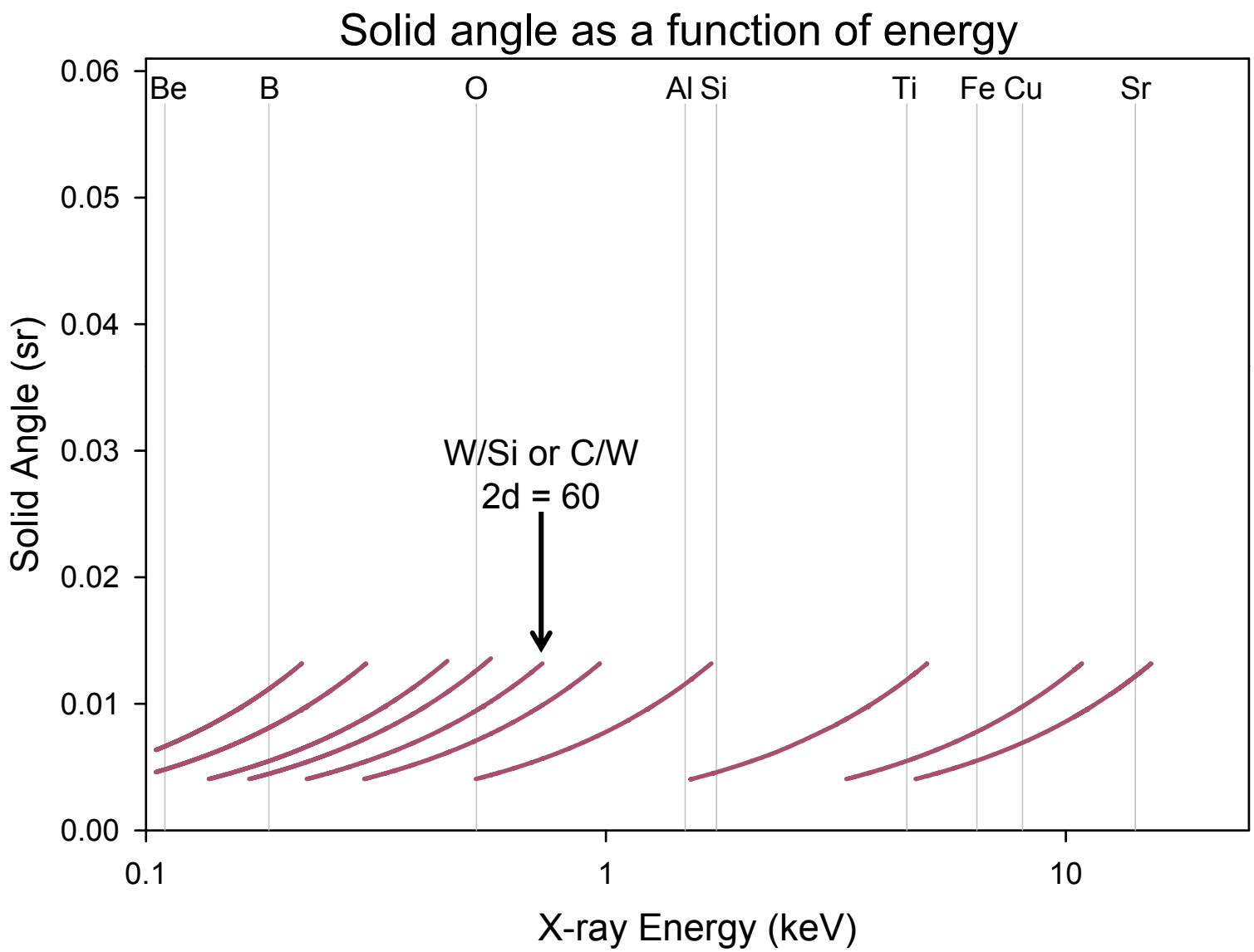

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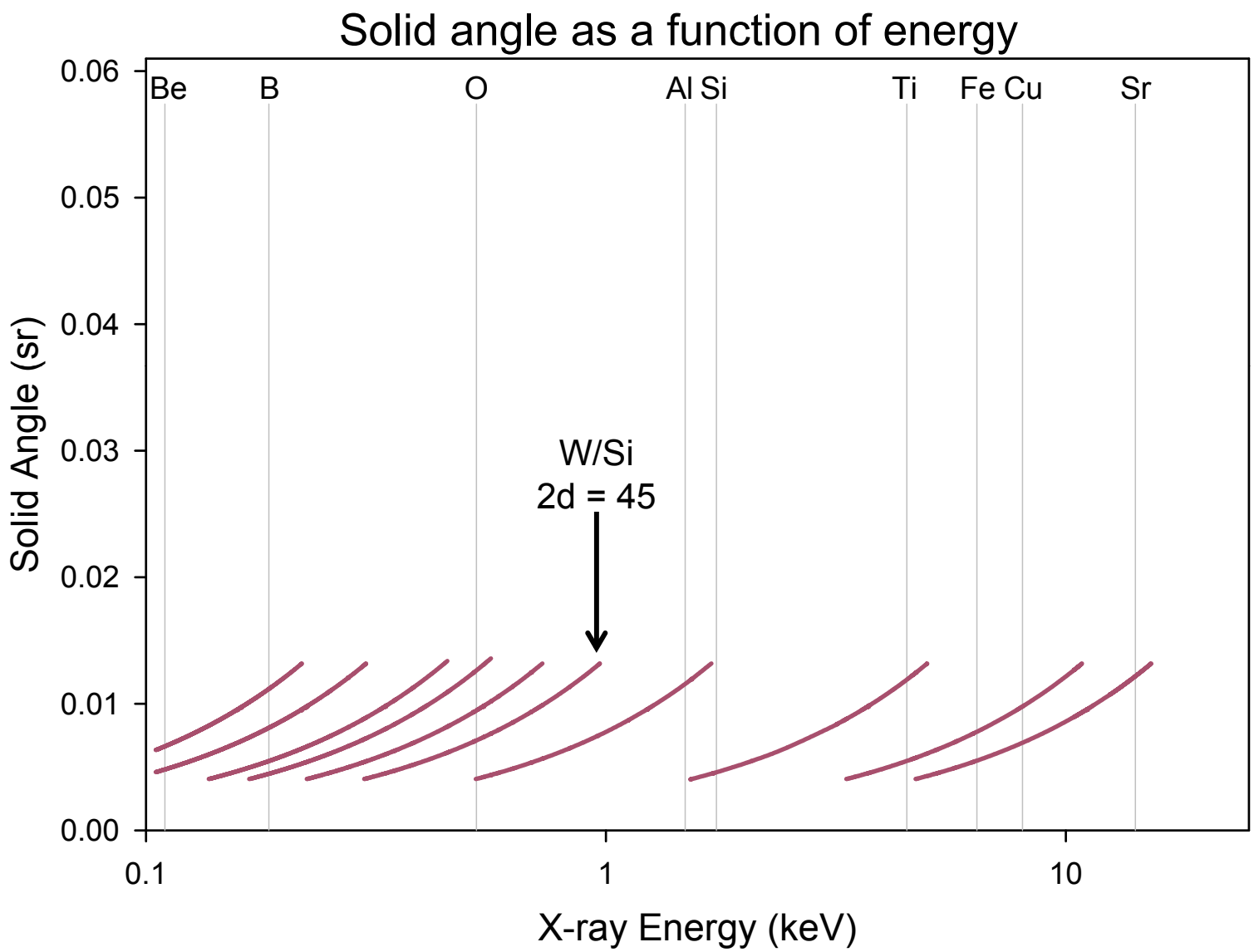
Rowland Circle WDS: SEM Solid Angle



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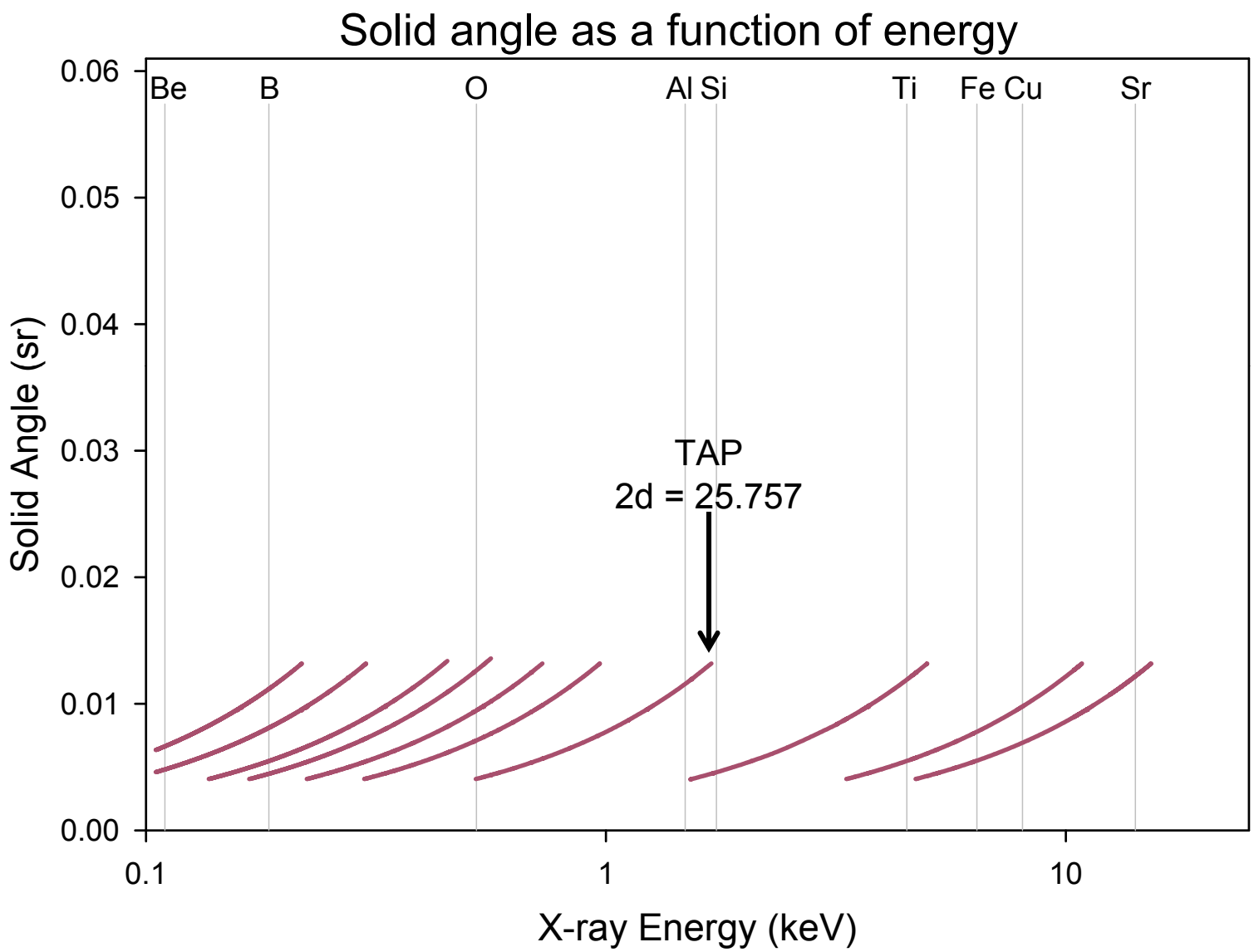

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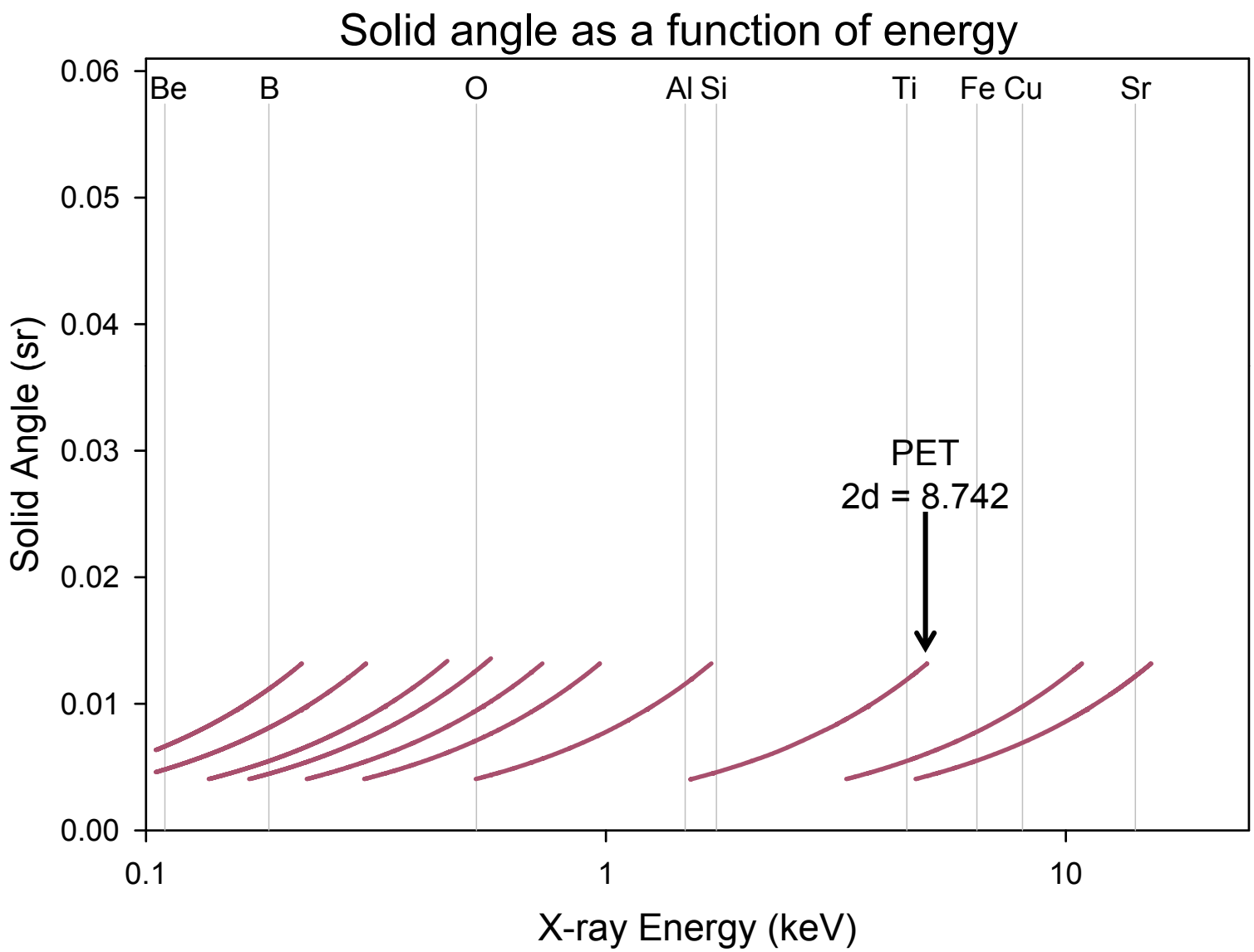
Rowland Circle WDS: SEM Solid Angle



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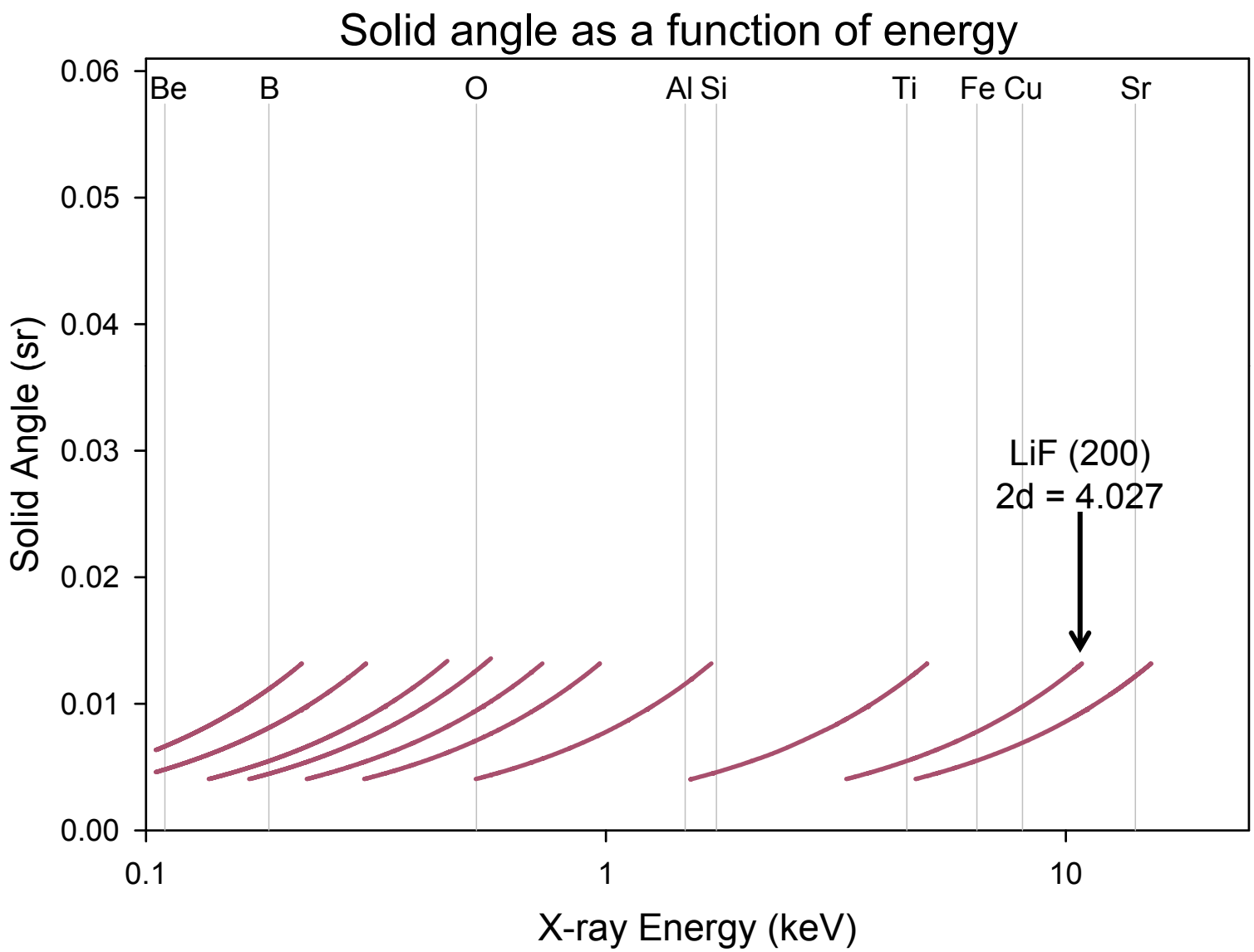
Rowland Circle WDS: SEM Solid Angle



210 mm SEM

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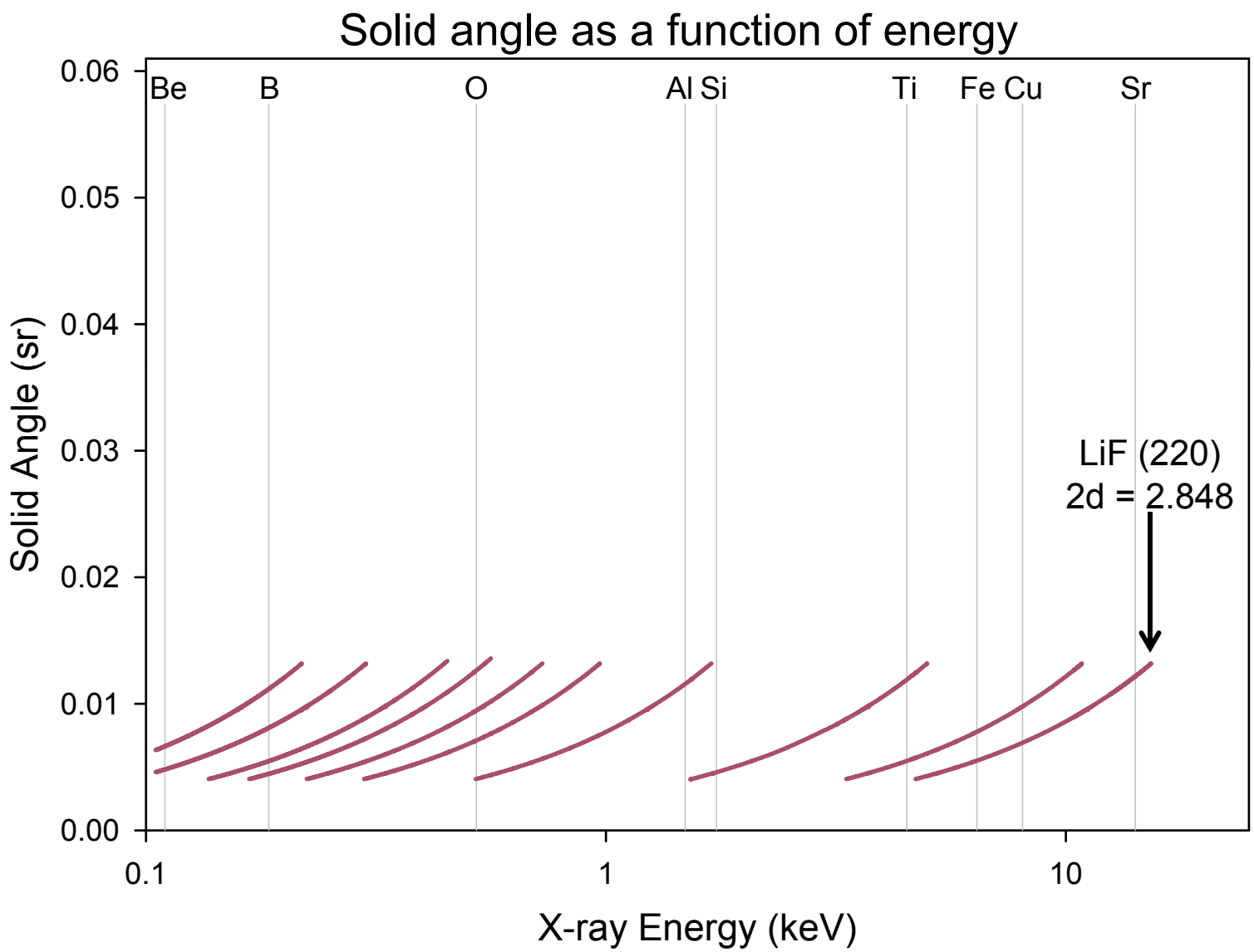
Rowland Circle WDS: SEM solid angle



210 mm SEM

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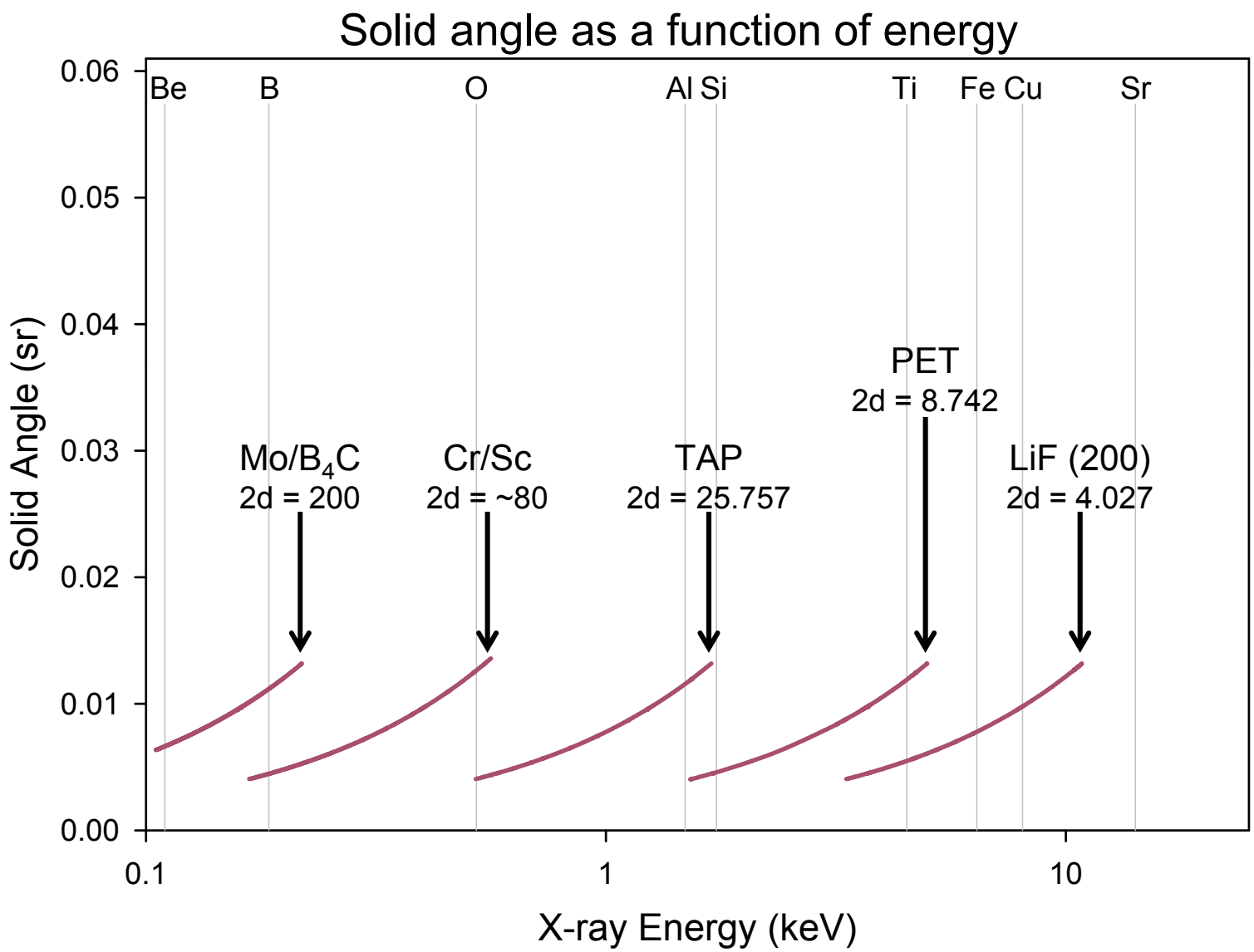
Rowland Circle WDS: SEM Solid Angle



210 mm SEM

SA_{diff} = 661 mm^2

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Rowland Circle WDS: The Electron Microprobe (EPMA)

- Electron microscope with typically 5 RC-WDS spectrometers
  - Spectrometers can concurrently analyze the same or different elements
  - 2 or 4 diffractors in each spectrometer

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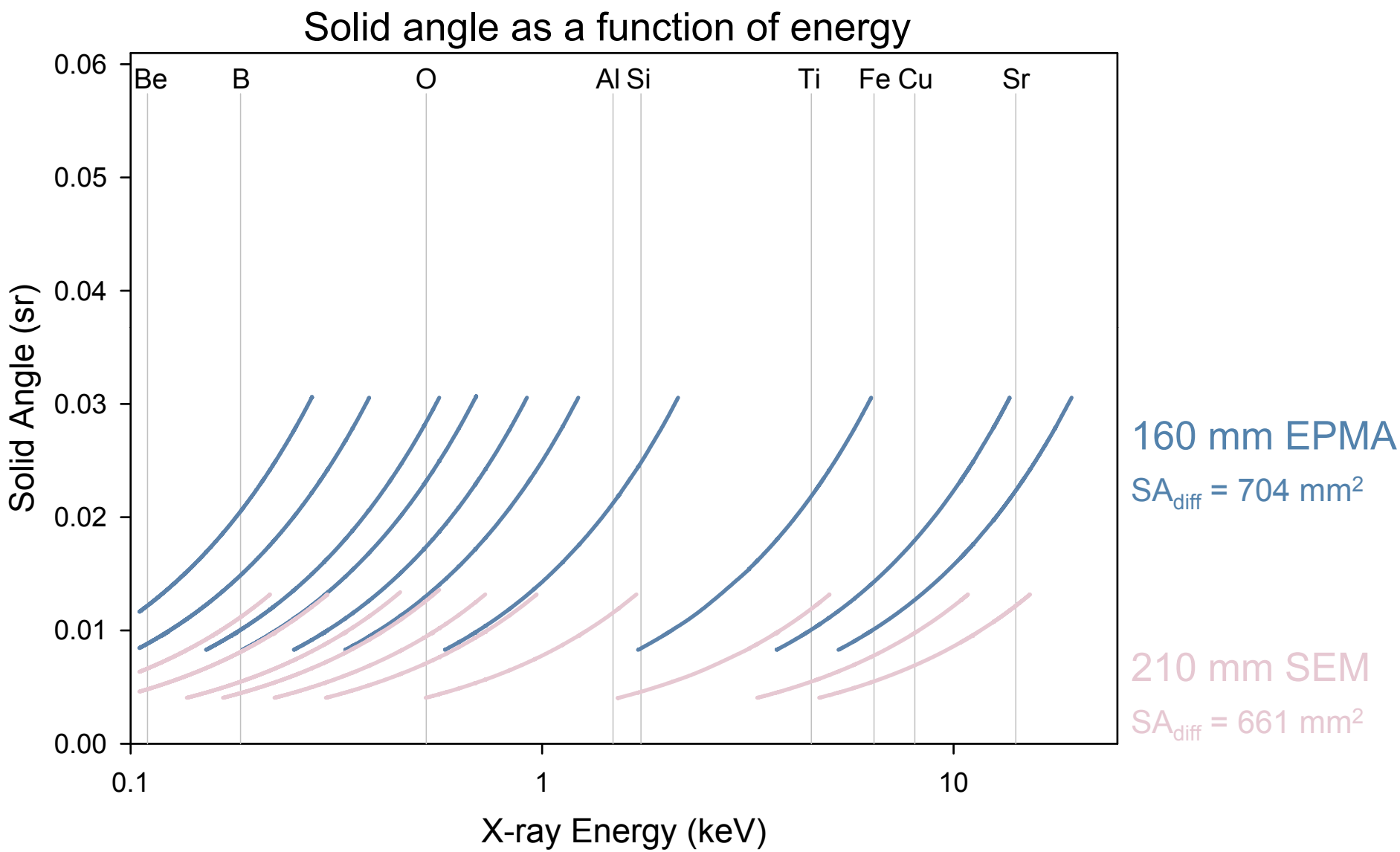
Rowland Circle WDS: EPMA Solid Angle



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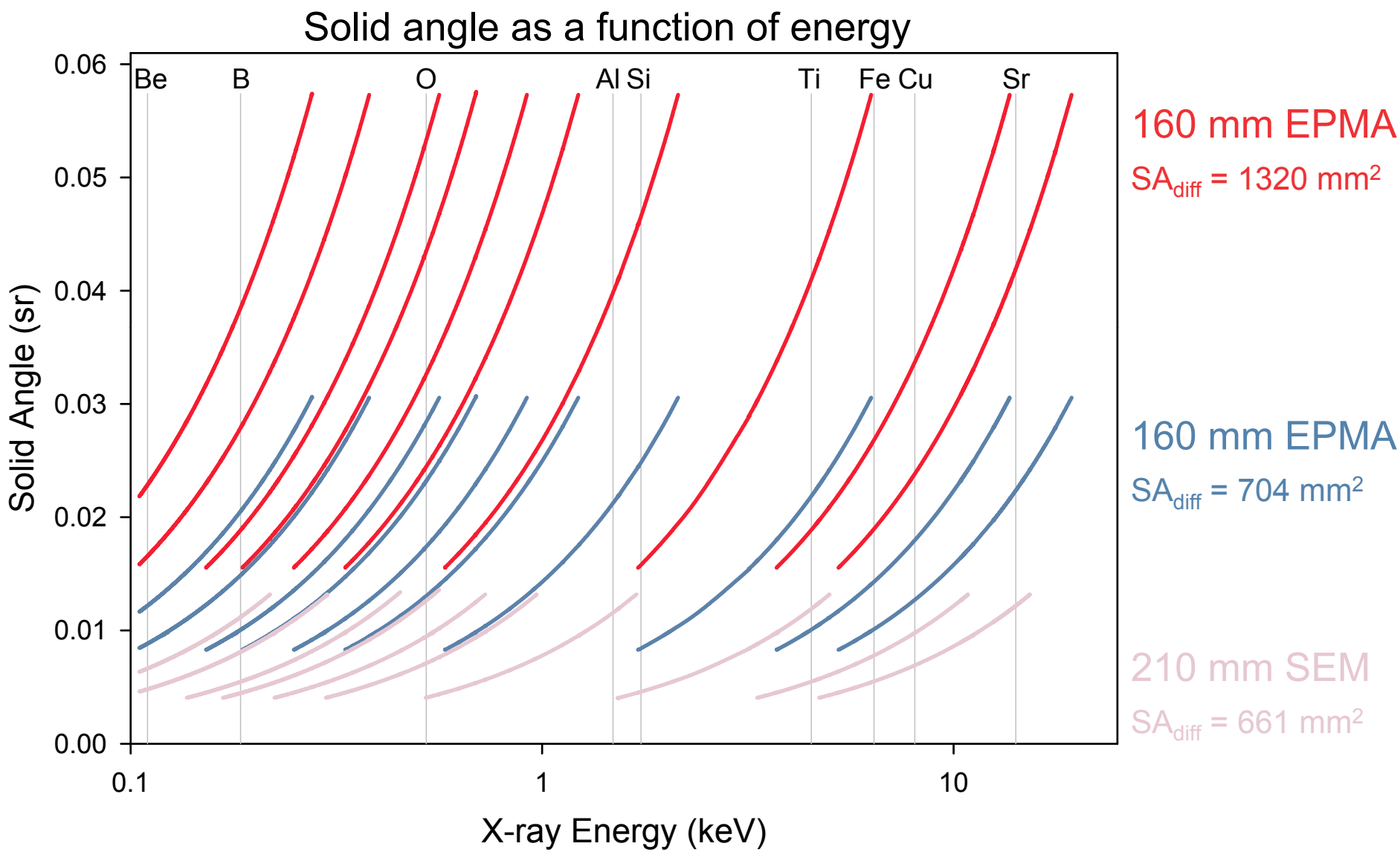
Rowland Circle WDS: EPMA Solid Angle



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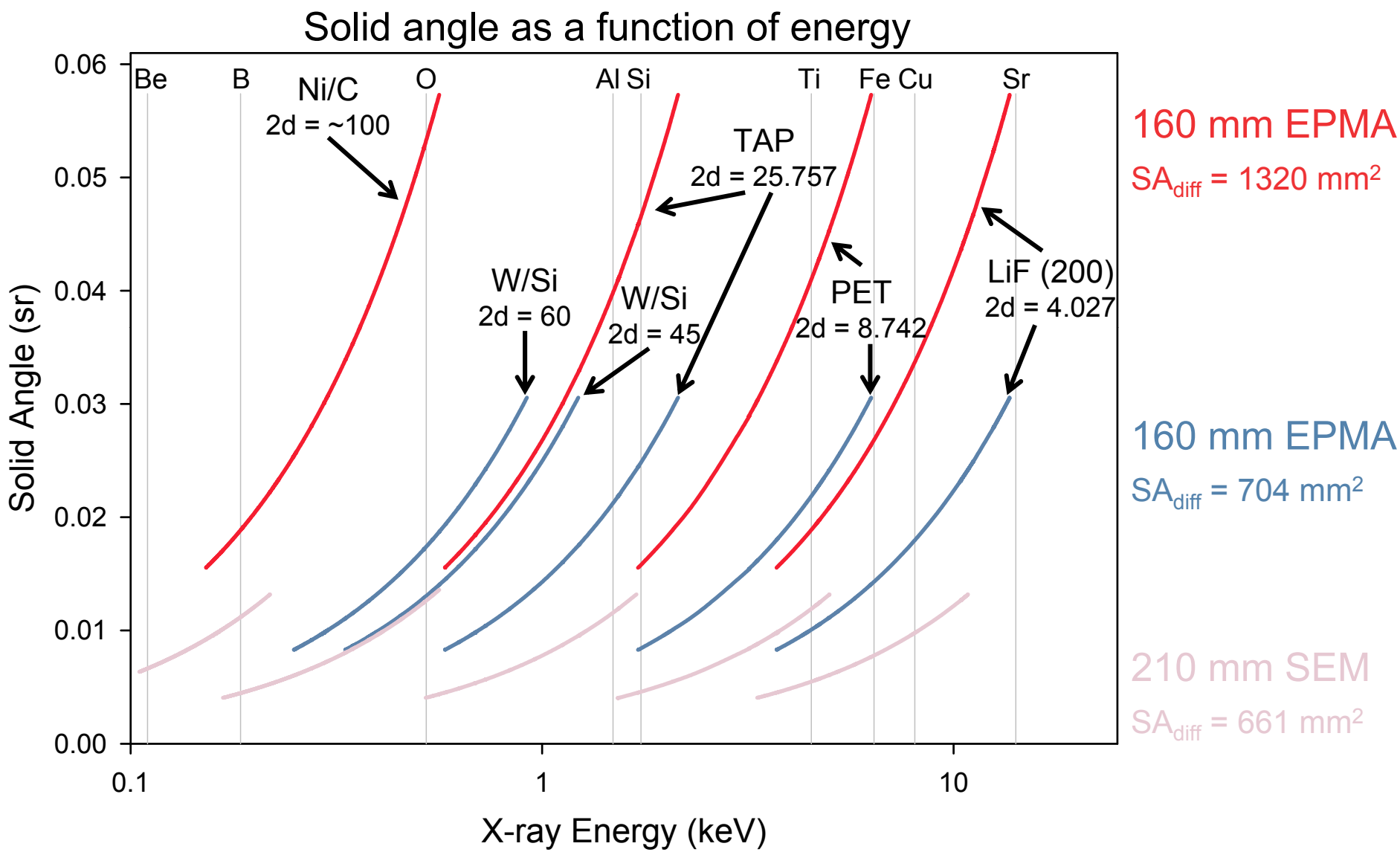
Rowland Circle WDS: EPMA Solid Angle



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Rowland Circle WDS: The Classic Solution

- Pros
  - Excellent energy resolution
  - Excellent peak-to-background
- Cons
  - Complicated spectrometer geometry
    - Best results when the chamber is designed for the spectrometer.
    - X-rays measured with large θ have low intensities
    - Typically relies on an optical microscope to ensure sample is at proper working distance
      - Not commonly available to the SEM user.
      - Requires a horizontal geometry

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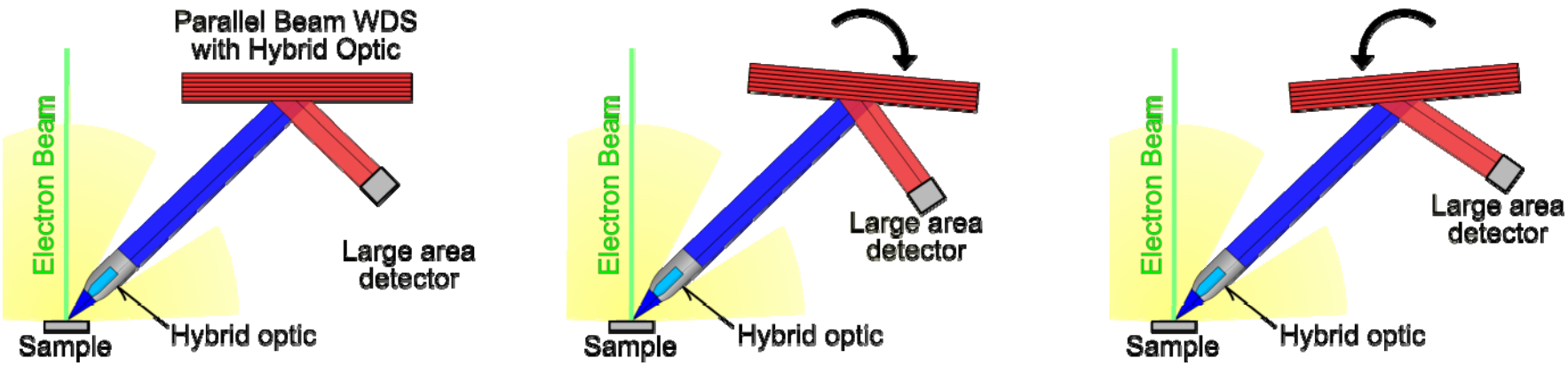
Parallel Beam WDS: A Modern Approach



- Parallel beam WDS spectrometer
  - Collimating optic is located near (~ 20 mm) from sample
    - Grazing incidence
    - Polycapillary
    - Hybrid
  - Parallel X-ray beam incident on diffractor
    - No Rowland circle geometry needed
    - Diffractor is flat

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Parallel Beam WDS: A Modern Approach

Exciting PB-WDS animation here!

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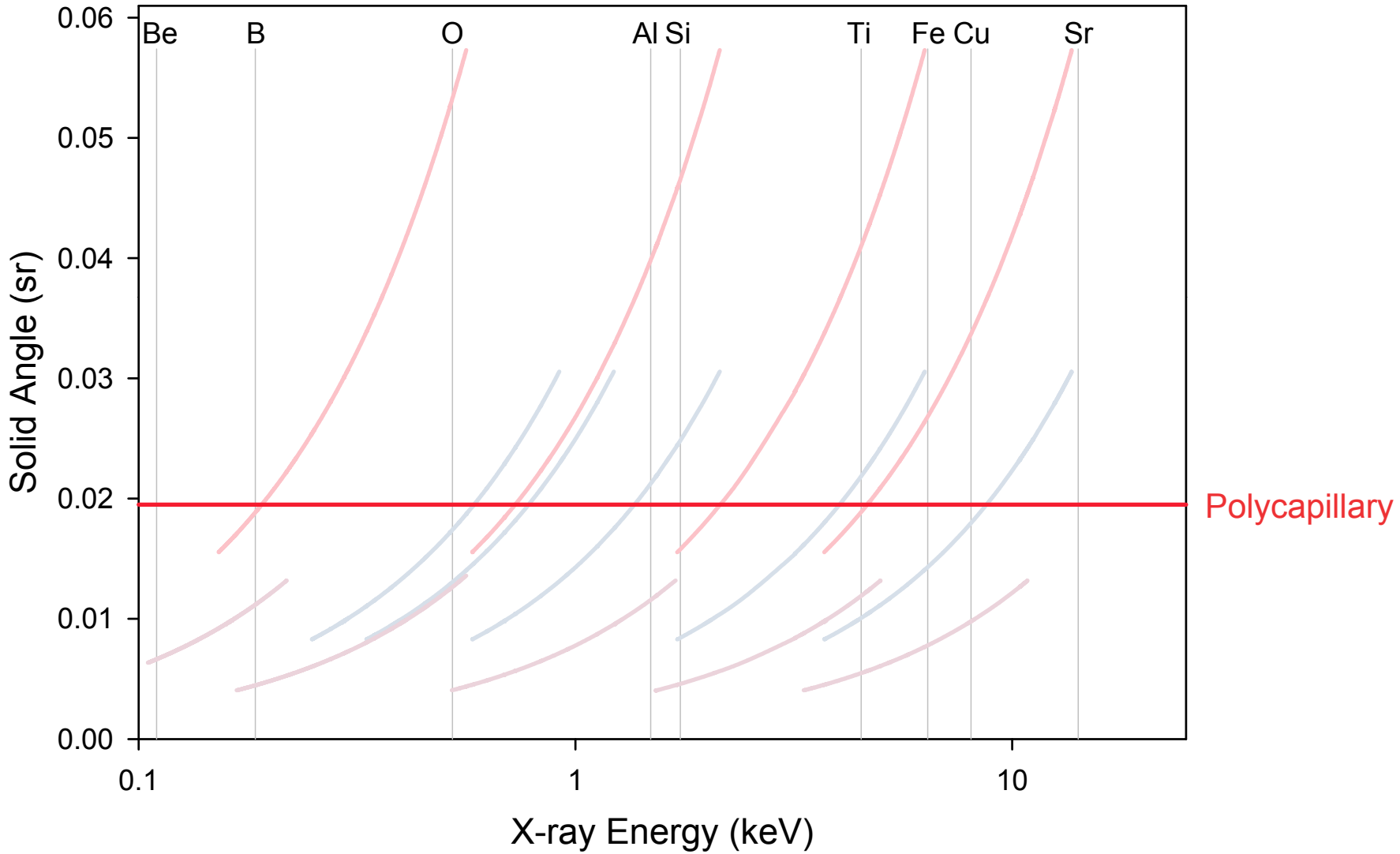
Parallel Beam WDS: A Modern Approach



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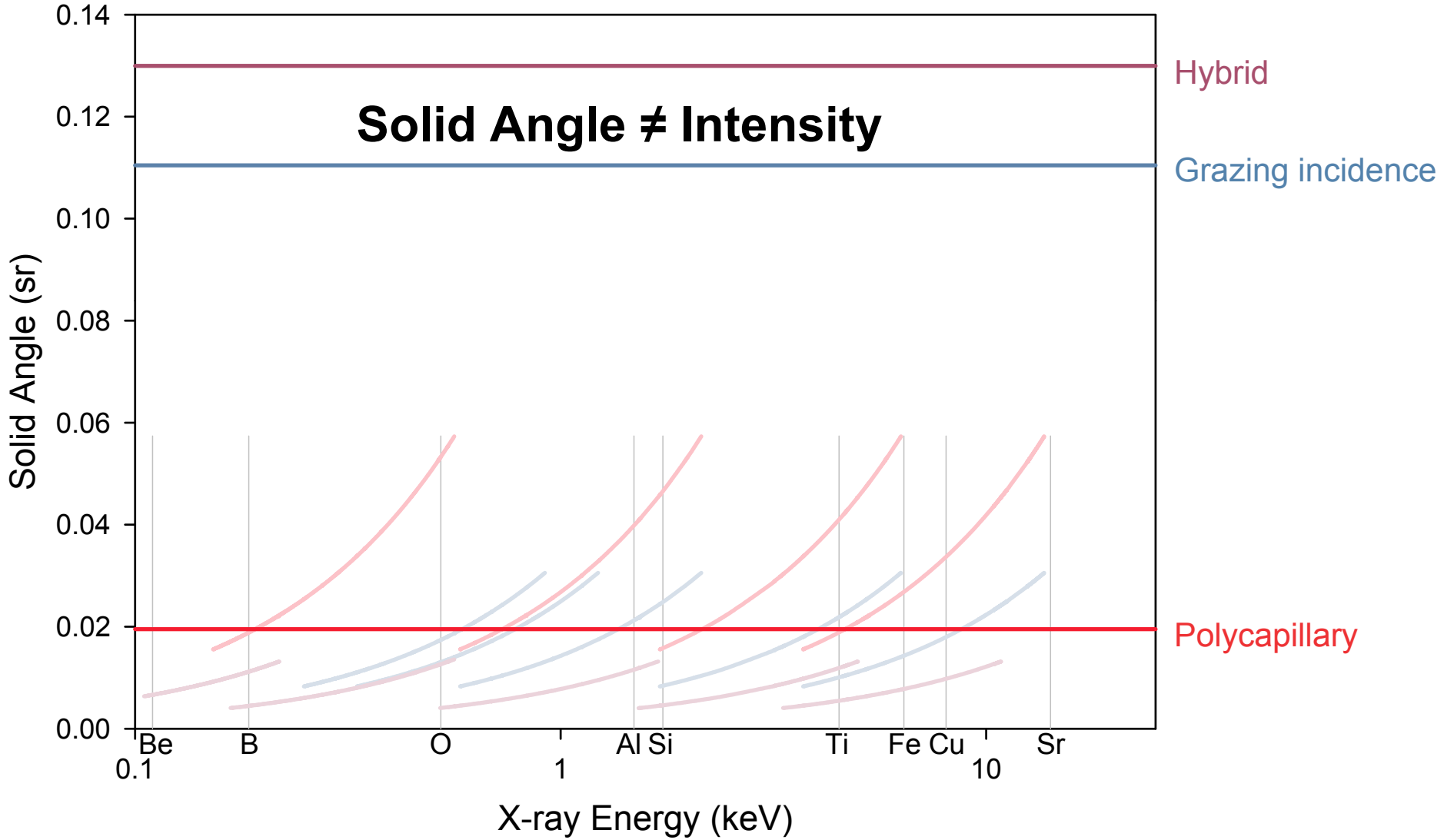
Parallel Beam WDS: A Modern Approach



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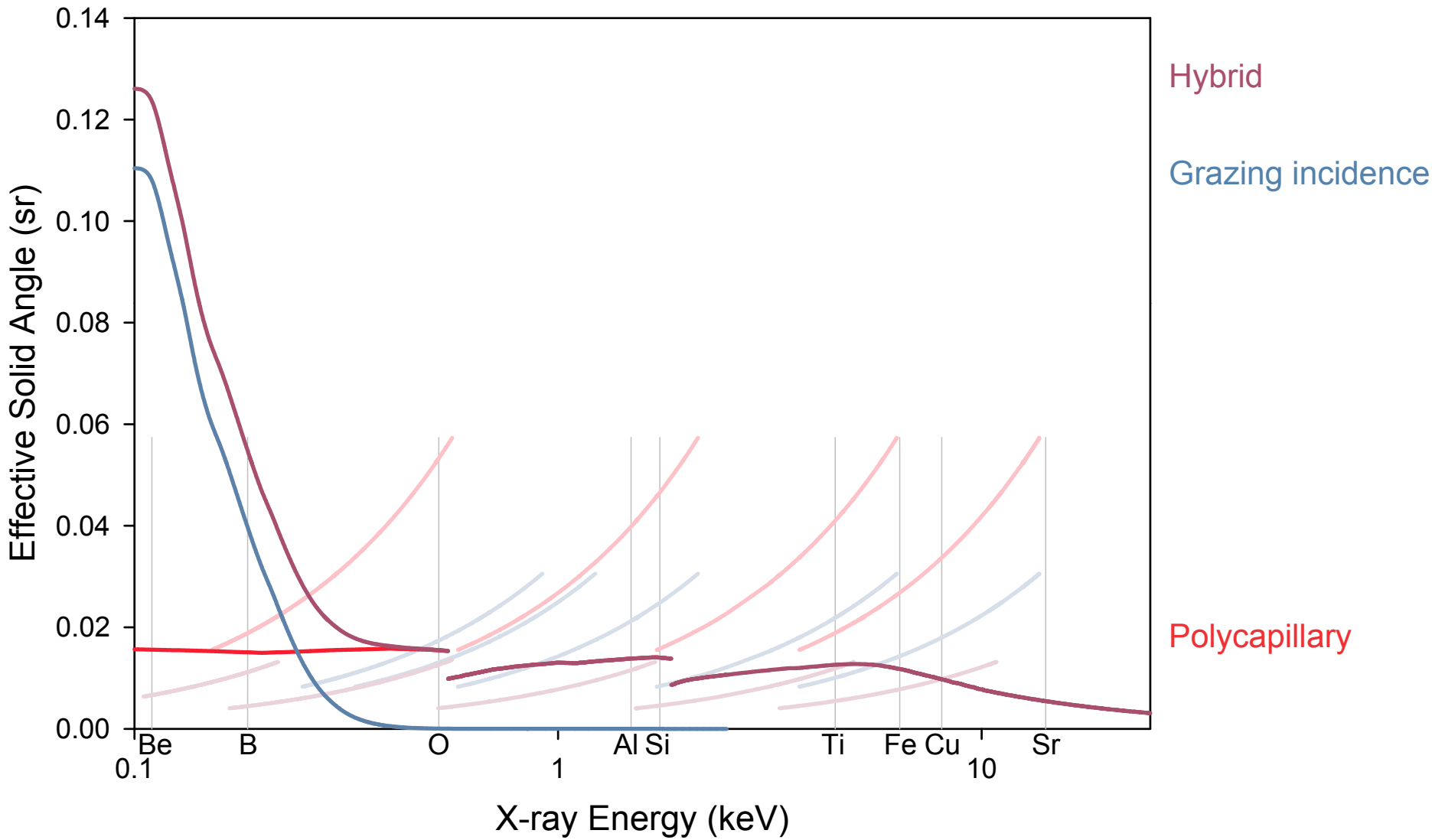
Parallel Beam WDS: A Modern Approach



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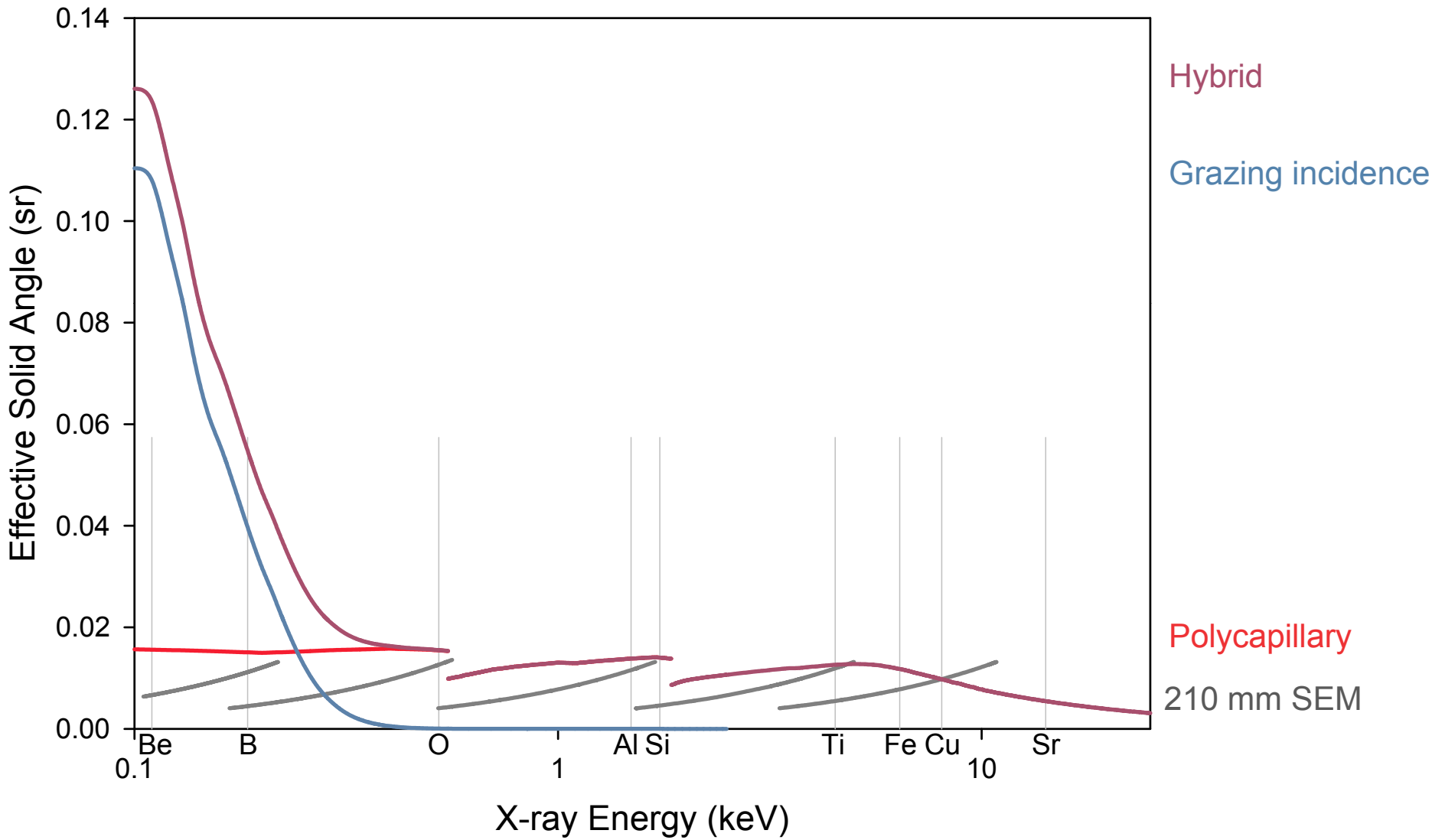

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Parallel Beam WDS: A Modern Approach

- Pros
  - Excellent energy resolution
  - Excellent peak-to-background
  - Excellent intensity
- Cons
  - Cannot accommodate a slit to modestly improve energy resolution with a large intensity cost

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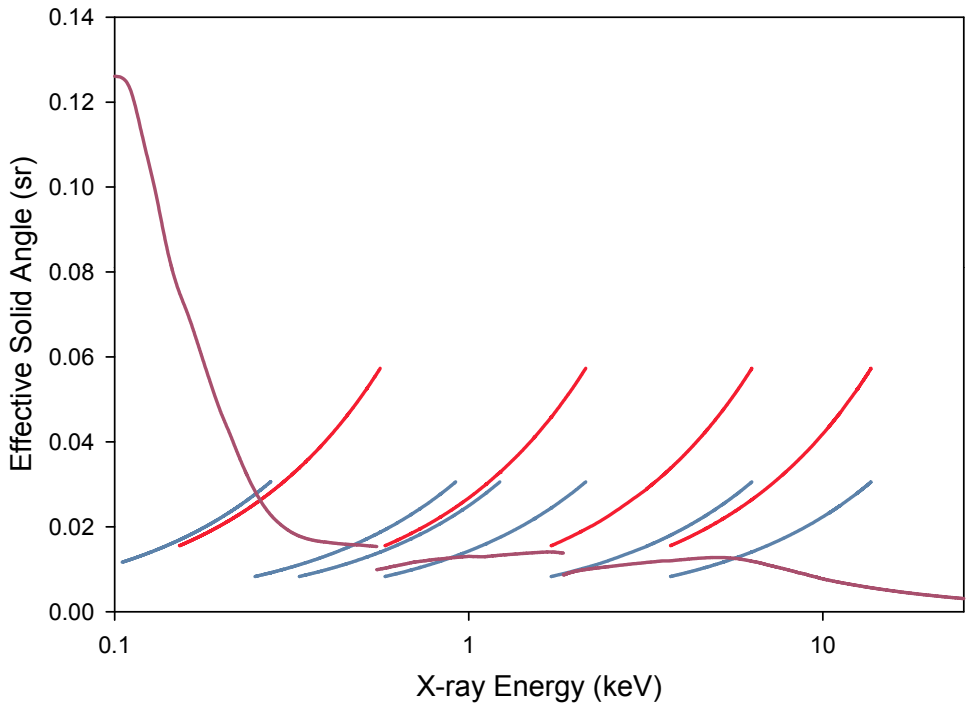
Head-to-Head Comparison: SEM and EMP

- Identical accelerating voltage and beam current

- PB-WDS with hybrid optic
  - 1 spectrometer
  - Sealed Xe detector
  - Flat diffractors

- 160 mm RC-WDS
  - 5 spectrometers
  - 3 detectors with 0.1 atm P10 flow
  - 2 detectors with 1 atm P10 flow
  - Curved diffractors (Johann and Johansson)



Seddio and Fournelle 2015

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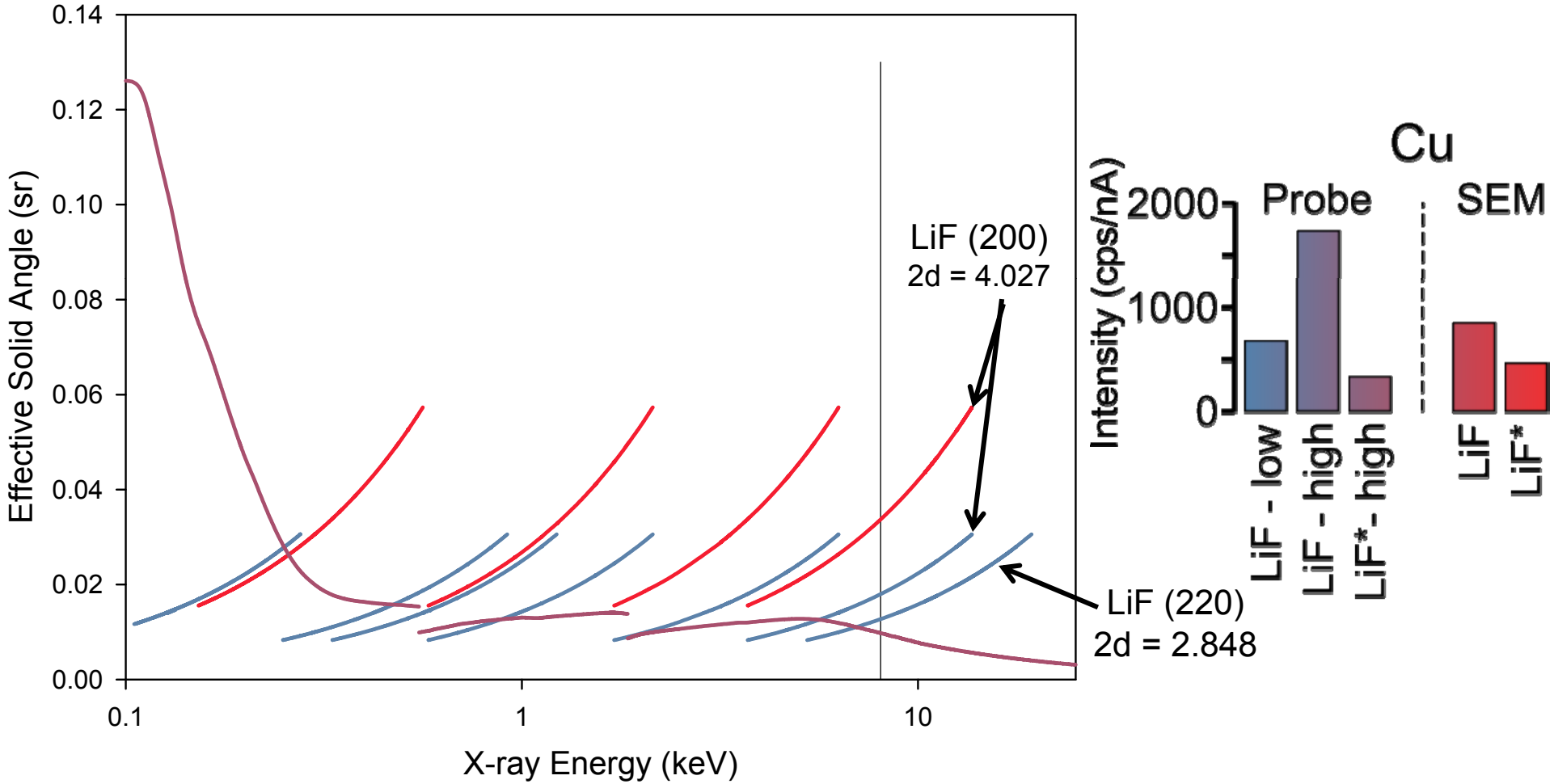
Head-to-Head Comparison: SEM and EMP



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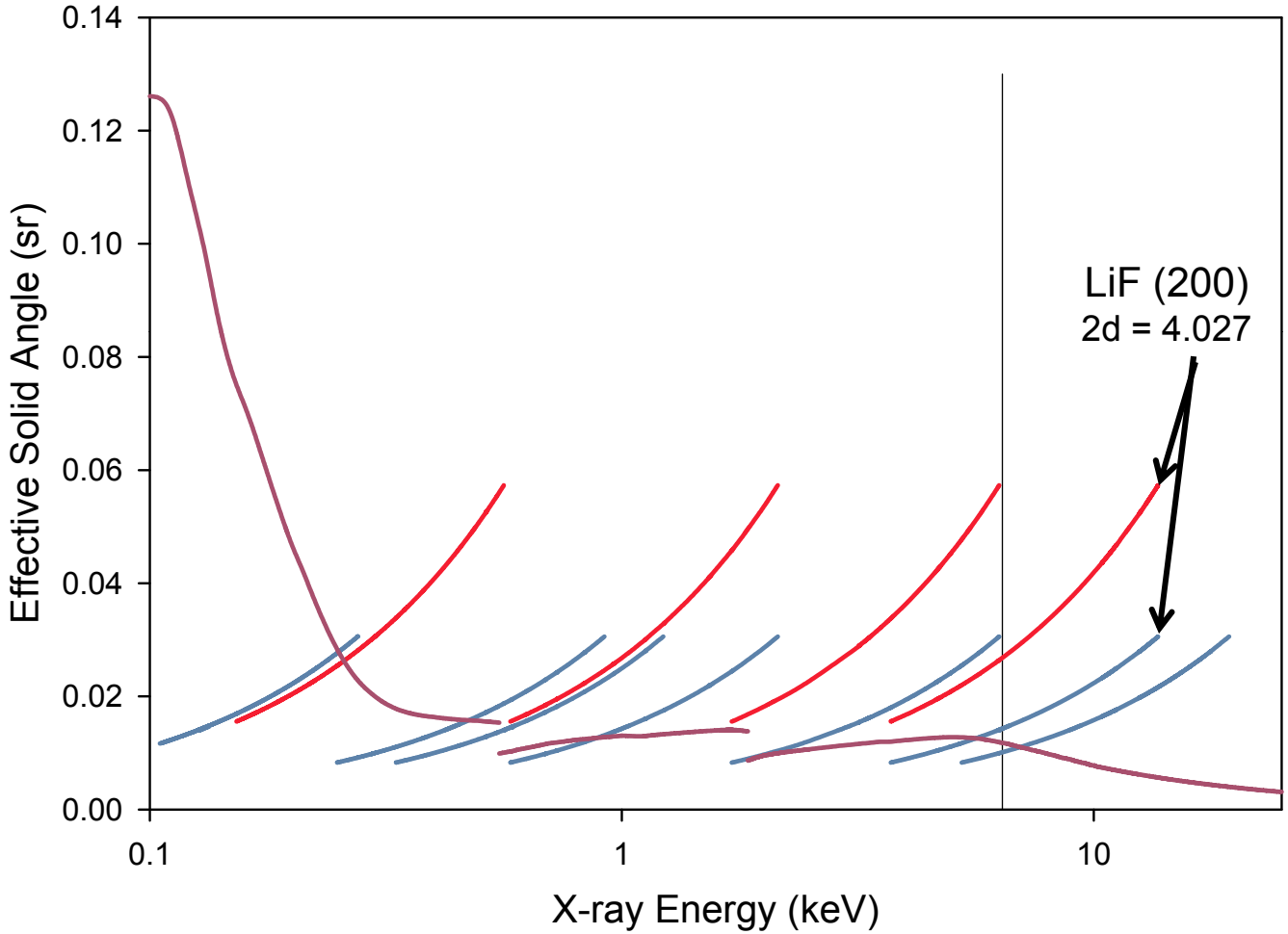

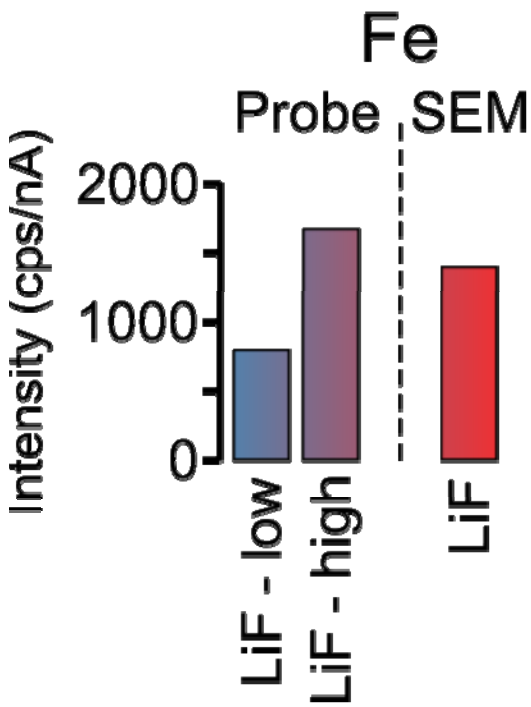

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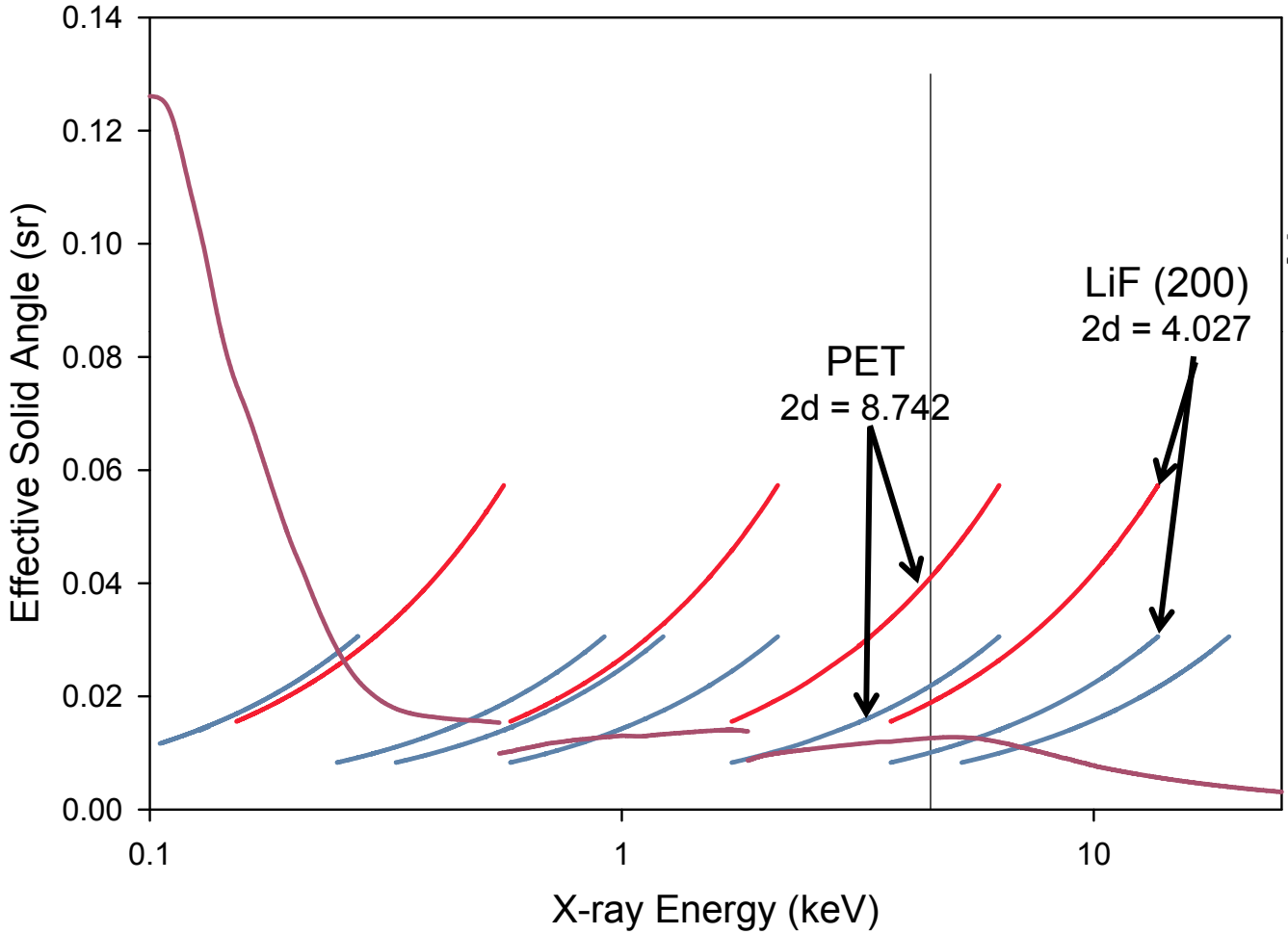

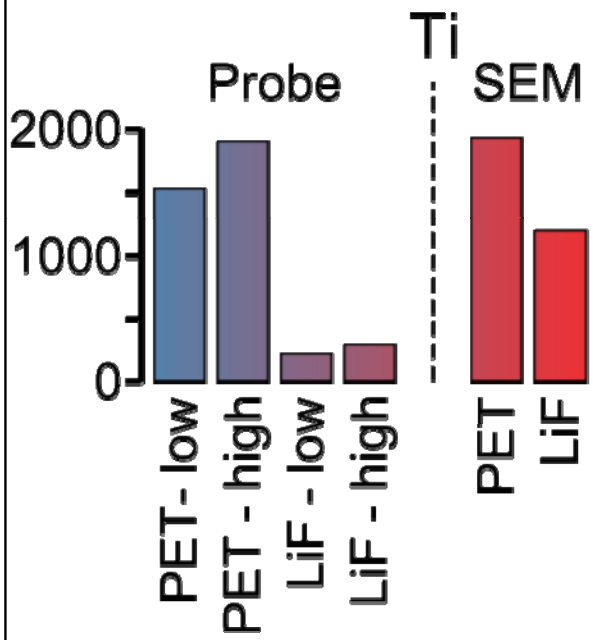

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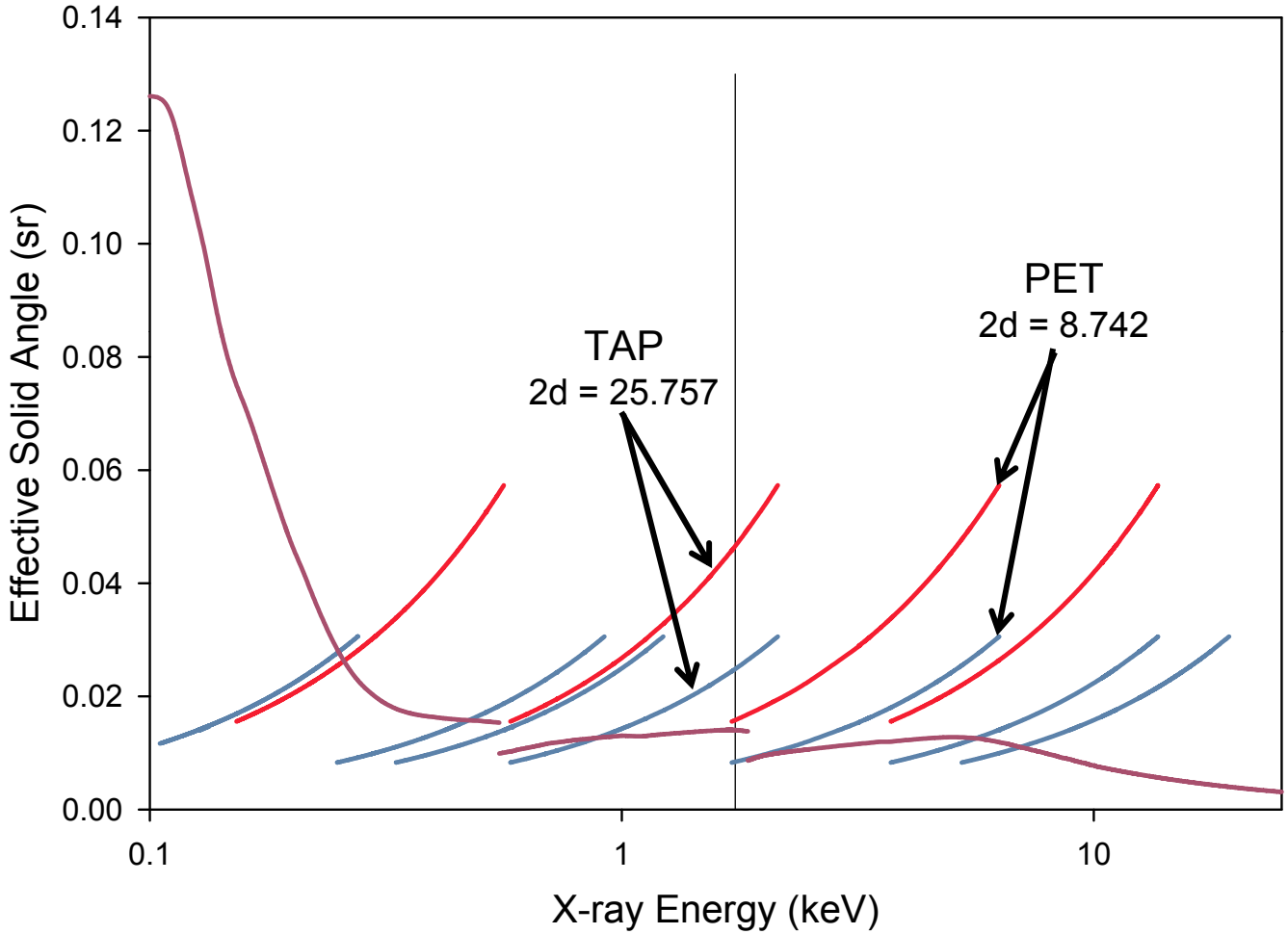
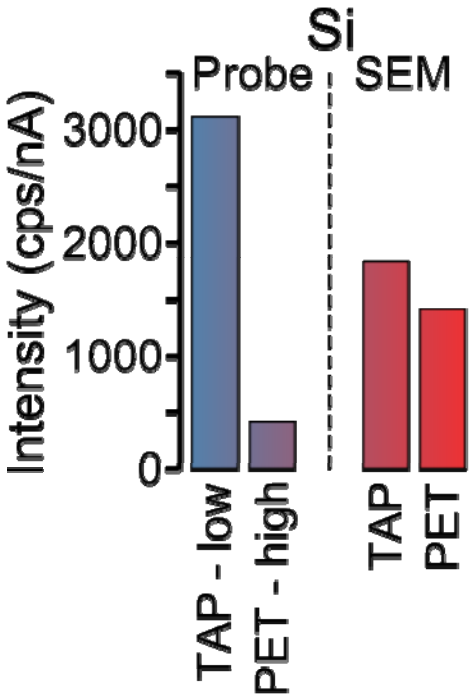
Head-to-Head Comparison: SEM and EMP





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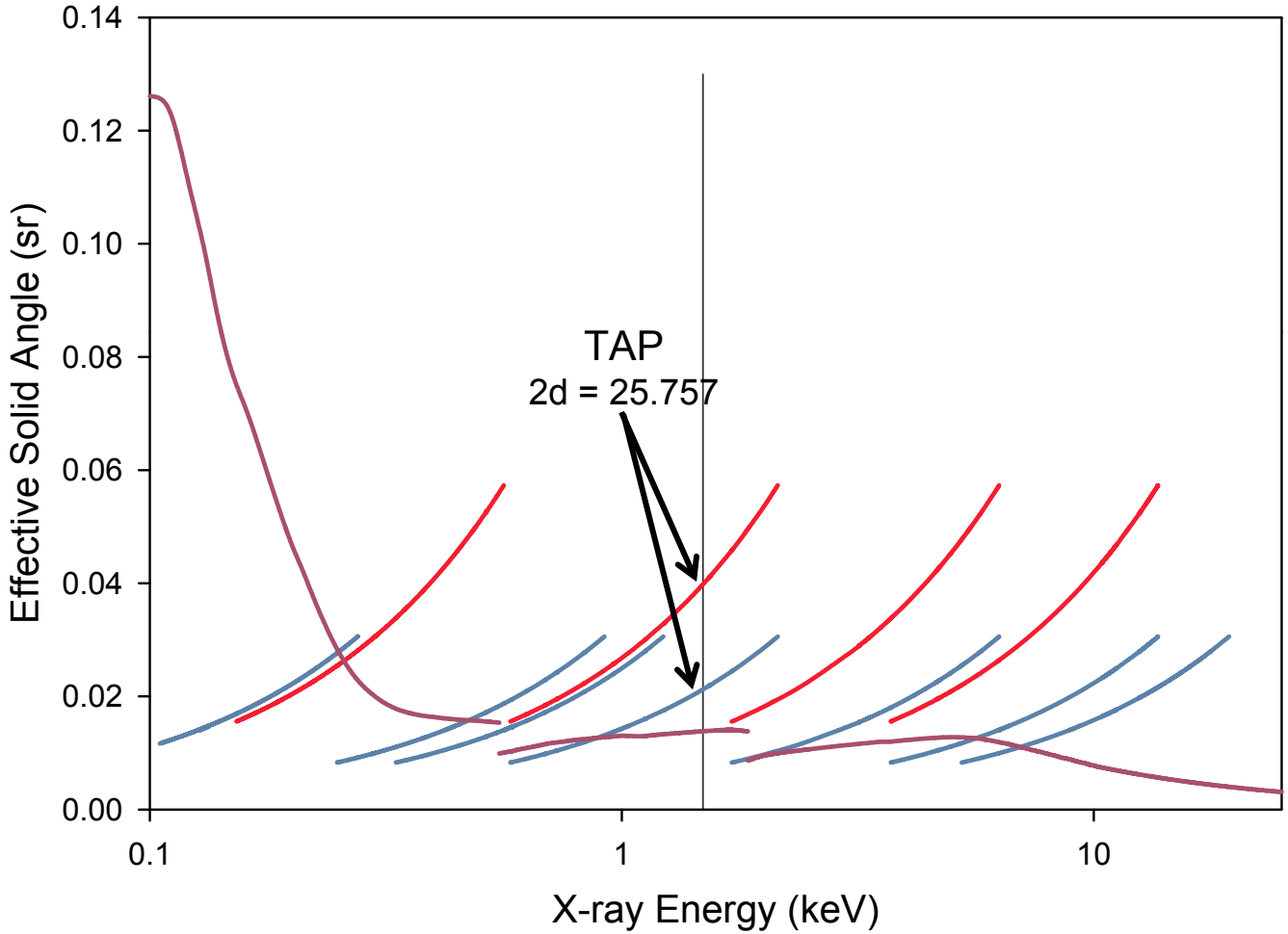
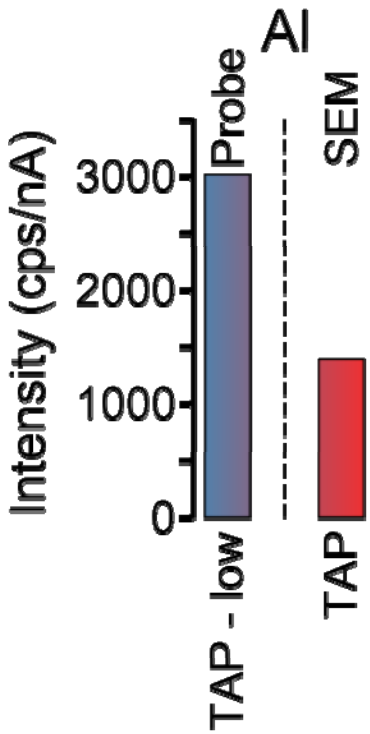
Head-to-Head Comparison: SEM and EMP





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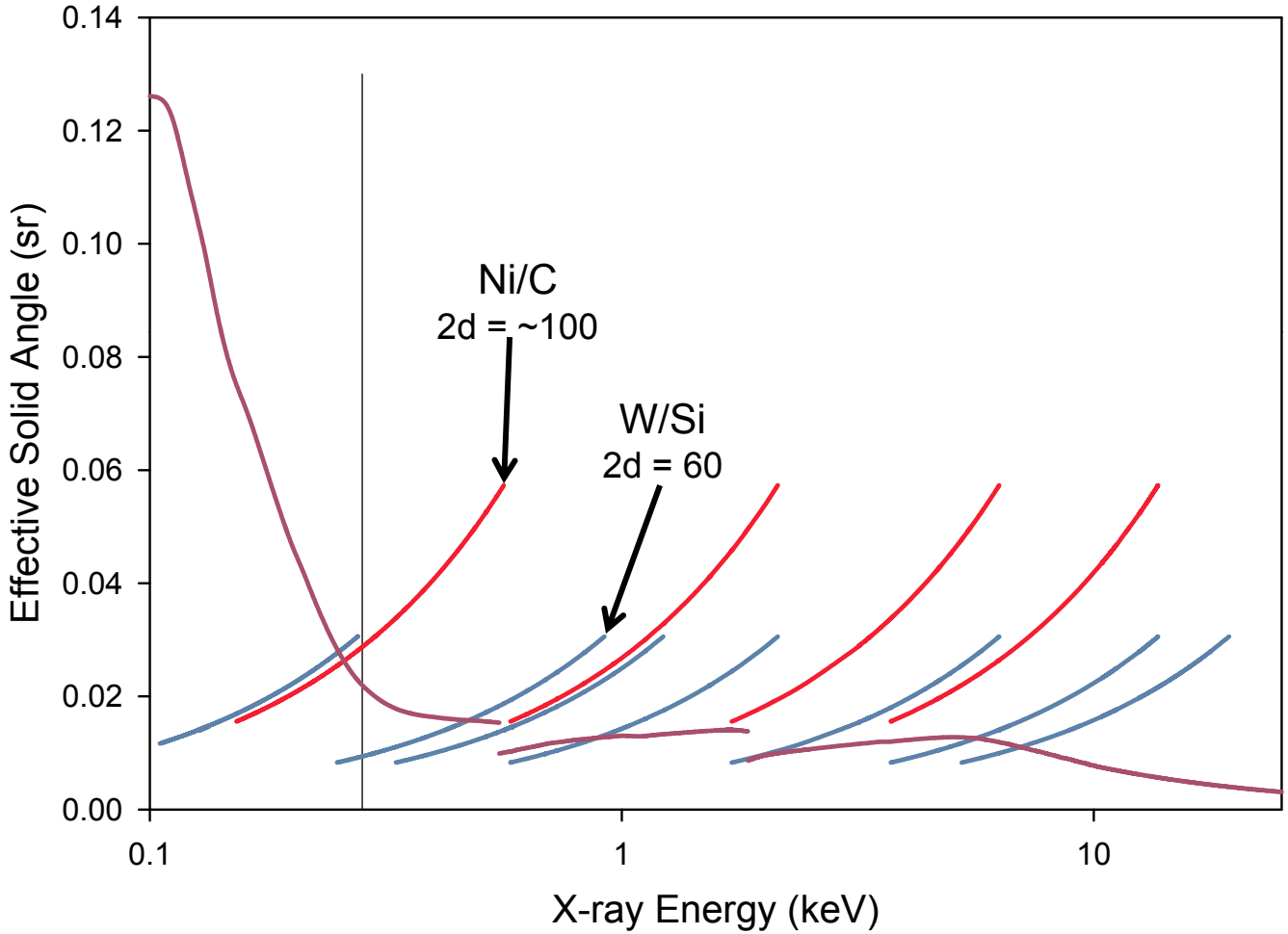

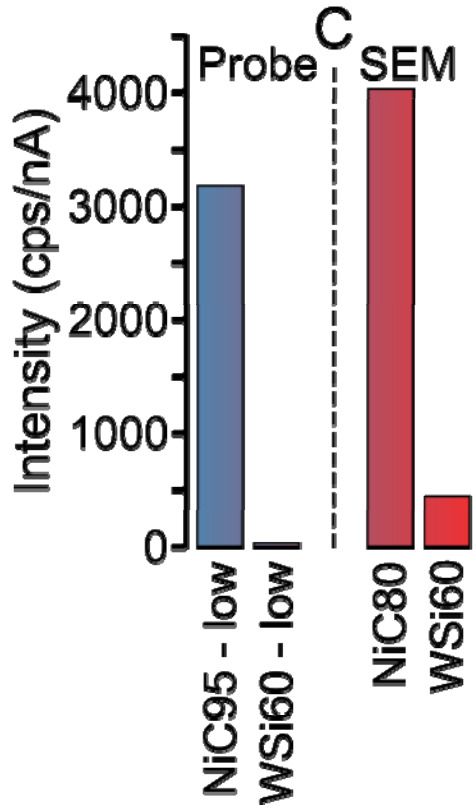

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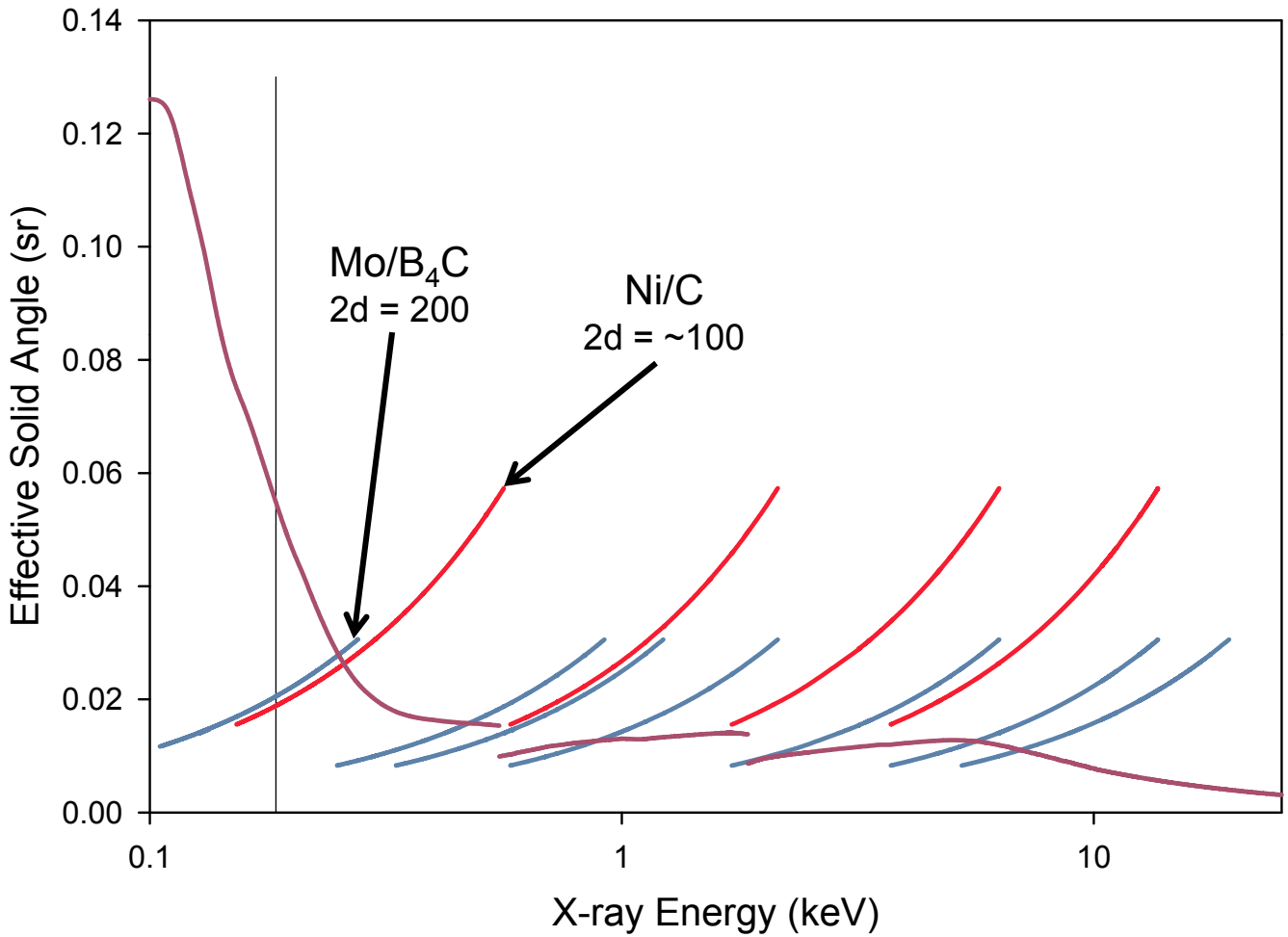
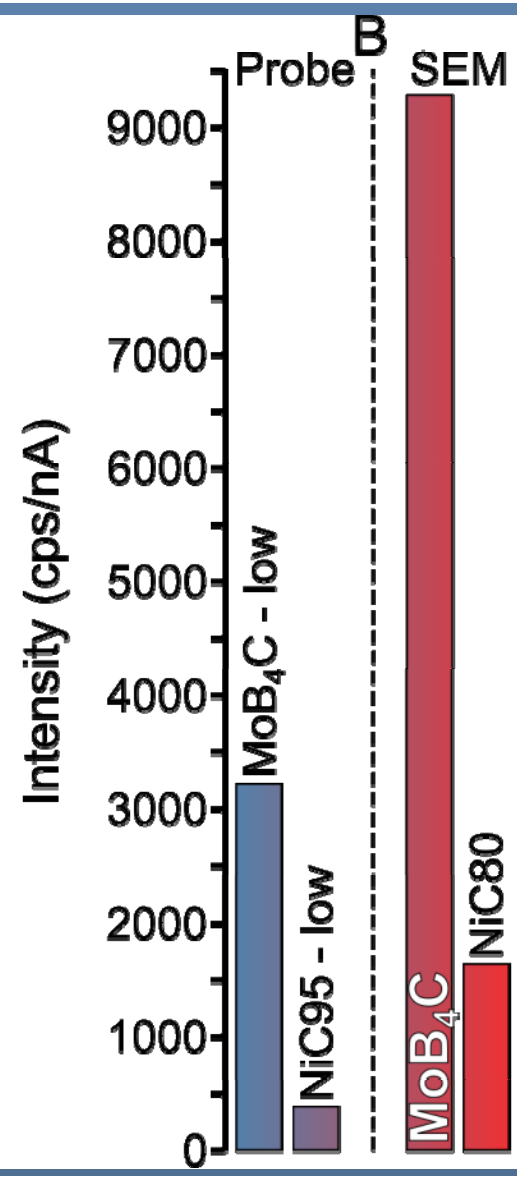
Head-to-Head Comparison: SEM and EMP





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Conclusions

- RC-WDS
  - Excellent solution when microscope is designed primarily for WDS
  - Yields impractically low intensities on SEMs

- PB-WDS
  - Unrivaled low energy X-ray intensity
  - High energy X-ray intensities consistent with the best RC-WDS intensities on an SEM

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