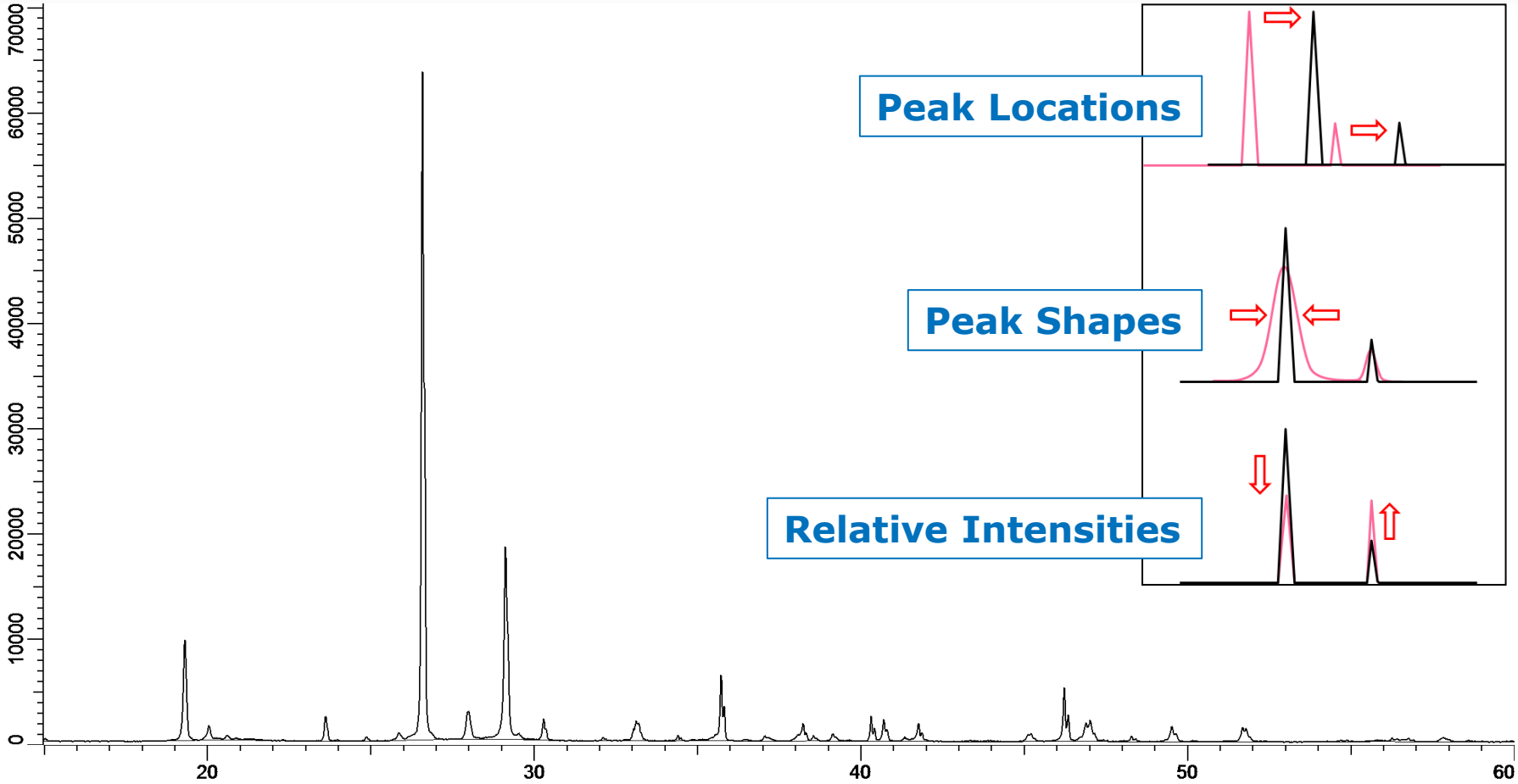
Specimen Preparation for XRPD

Common Sources of Error in Laboratory X-ray Diffraction

Nathan Henderson, Ph.D.
Sr. Applications Scientist - XRD

X-Ray Diffraction

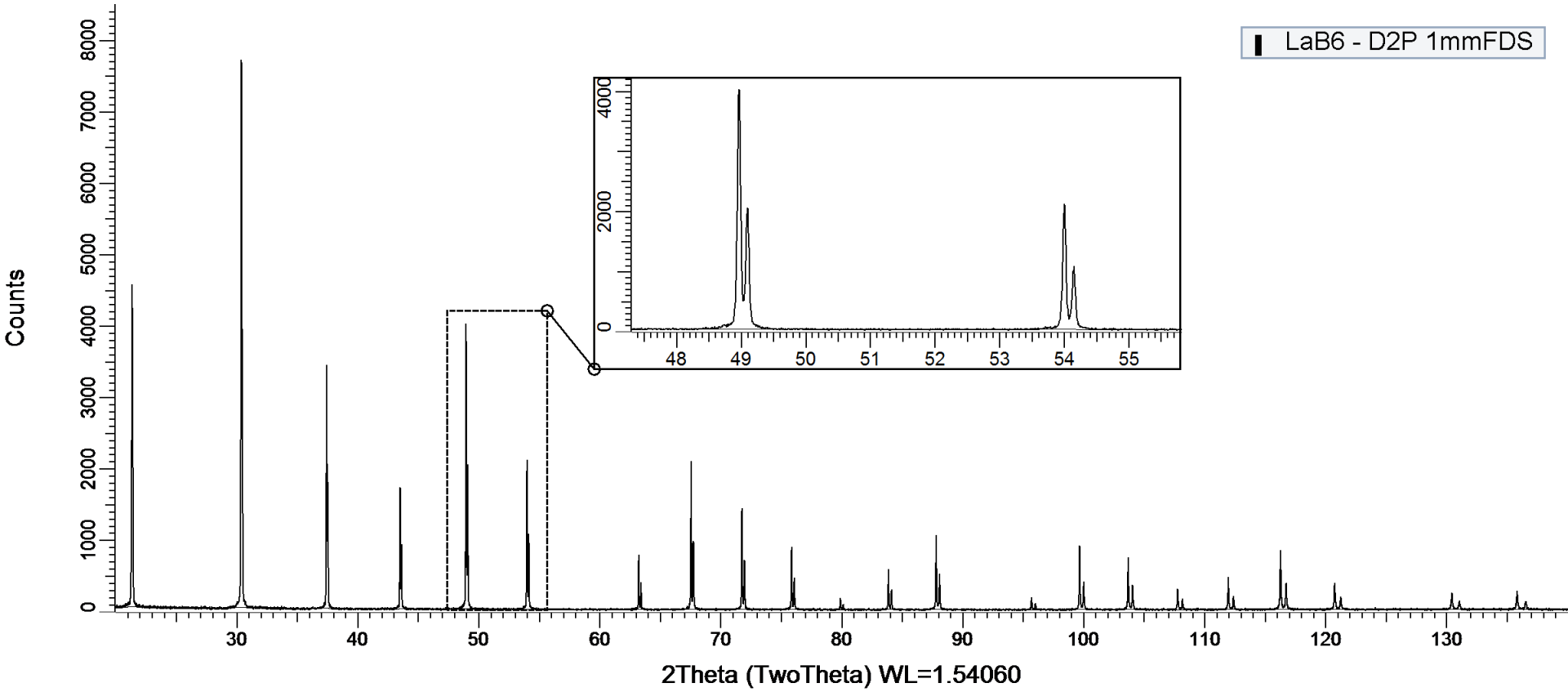
Example Diffraction Pattern



Data Quality

NIST SRM 660b (LaB_6)

Sharp Reflections (small FWHM)
Good signal-to-noise (smooth background)



Common Types of Error

Instrumental Error

- Flat Specimen Error (Location and Shape)
- Axial Divergence (Location and Shape)

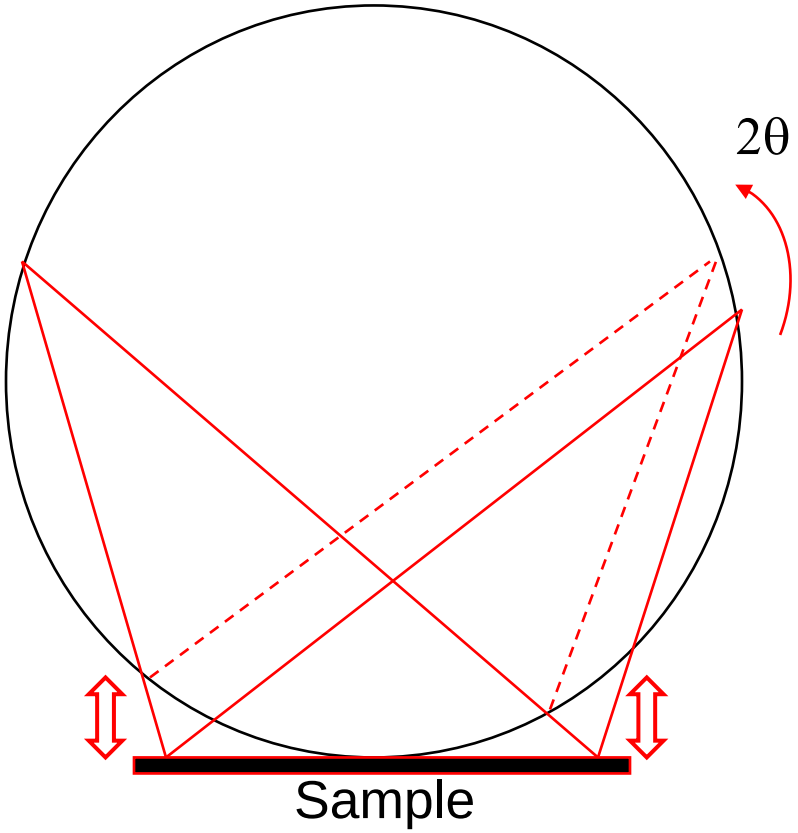
Specimen Error

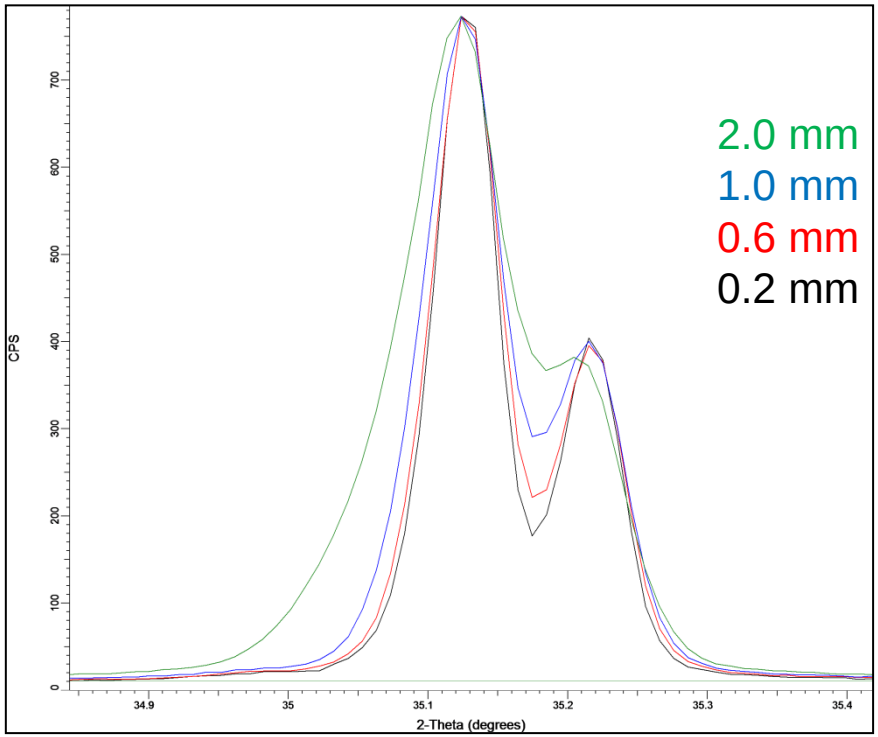
- Displacement (Location)
- Preferred Orientation (Relative Intensities)
- Transparency (Location and Shape)
- Particle Statistics (Relative Intensities)
- Inhomogeneity (Relative Intensities and Shape)

Instrumental Error

Flat Specimen Error

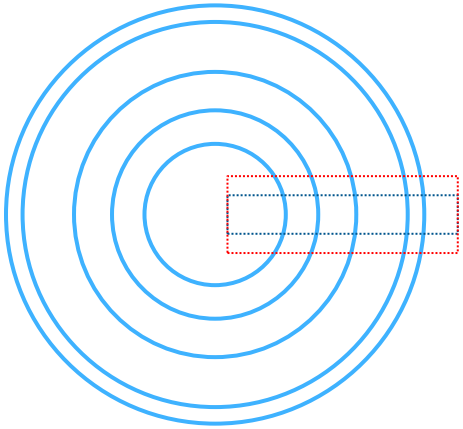
Peak broadening (lower resolution)
Shift to lower angles (lower accuracy)



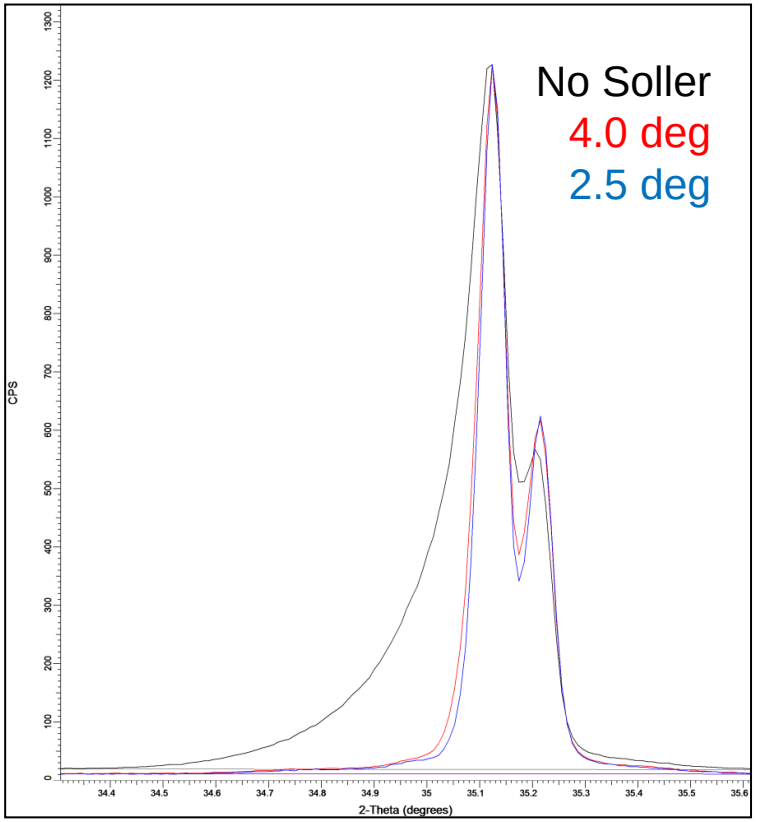


Instrumental Error
Axial Divergence

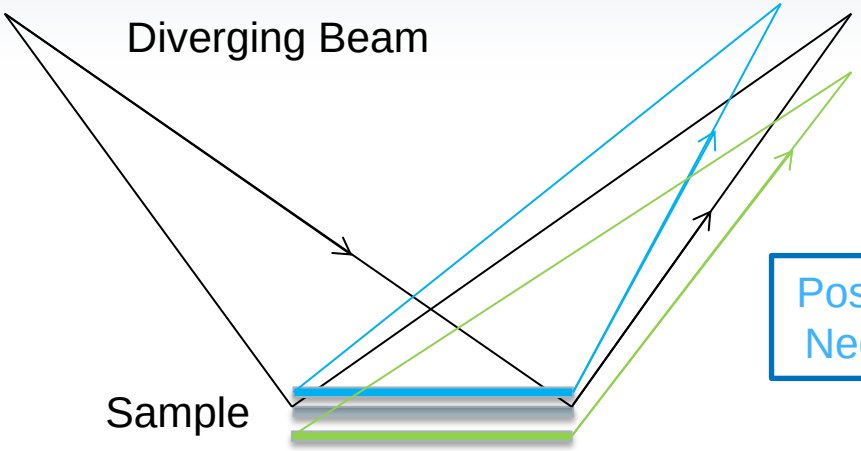
Debye Rings



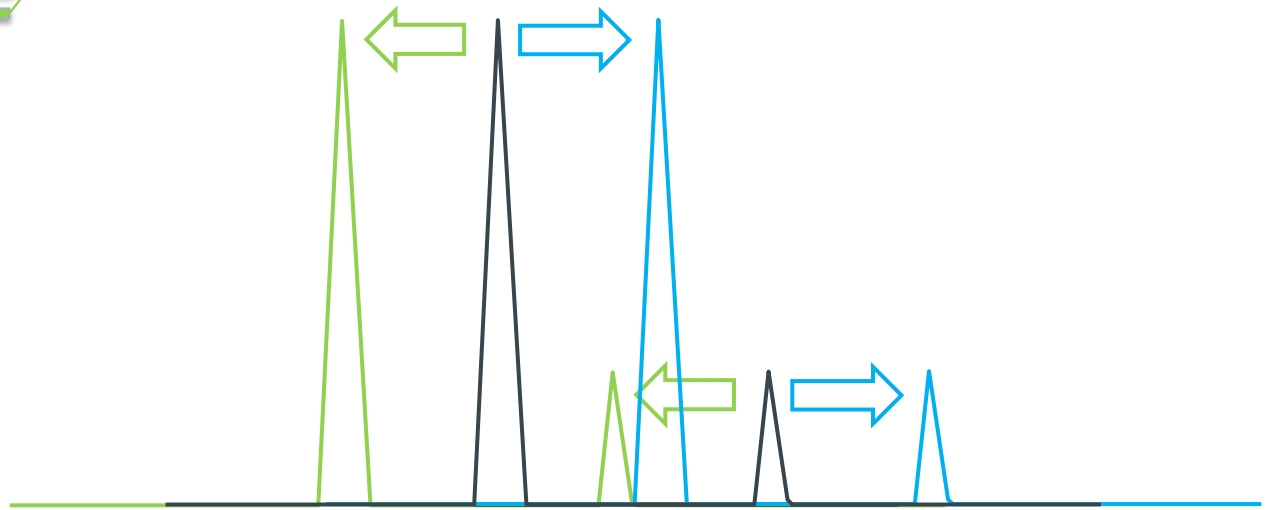
Peak Asymmetry
Lower resolution
Difficult to Integrate Peak Areas



Specimen Error
Displacement



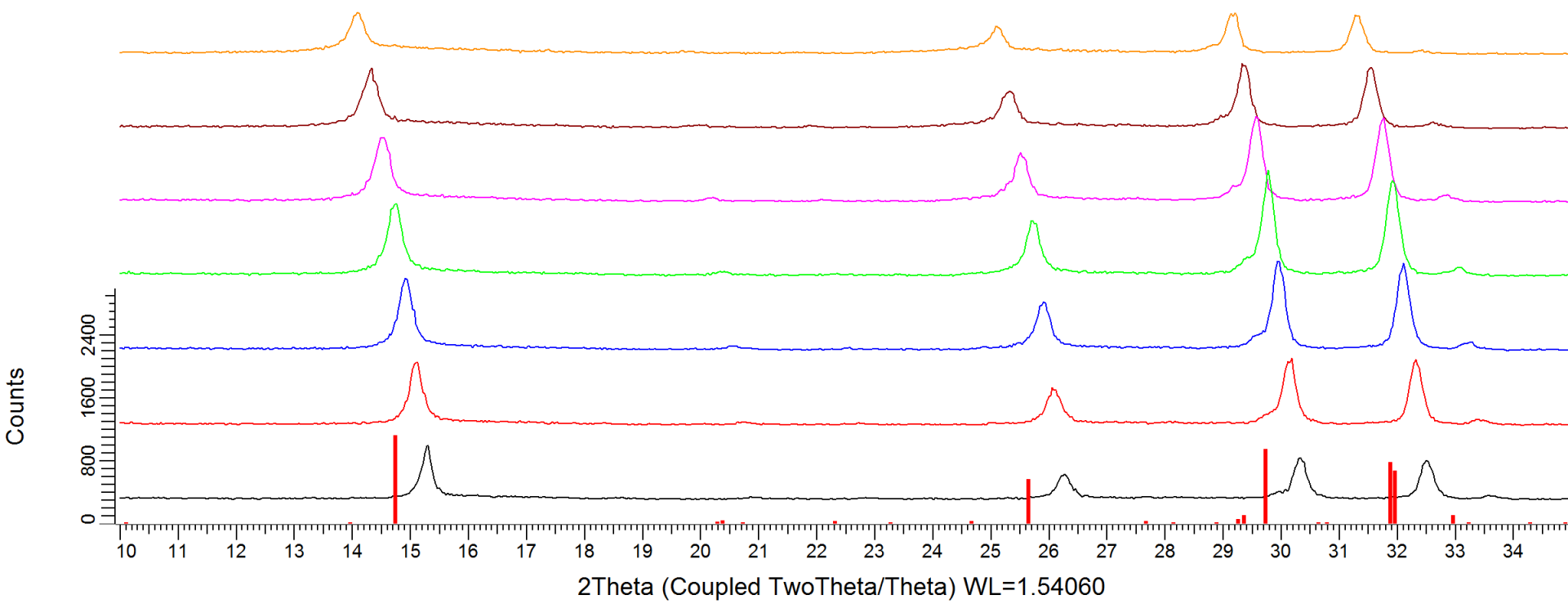
Positive Displacement – Shift to Higher Angles (right)
Negative Displacement – Shift to Lower Angles (left)



Specimen Error
Displacement

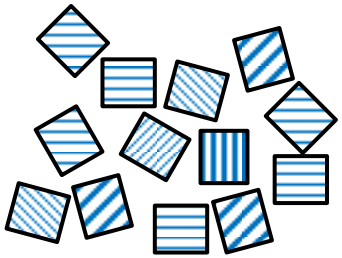
- 1.0 mm
- 0.7 mm
- 0.3 mm
No Displacement
+ 0.3 mm
+ 0.7 mm
+ 1.0 mm

Sample too high → Peaks too high
Sample too low → Peaks too low

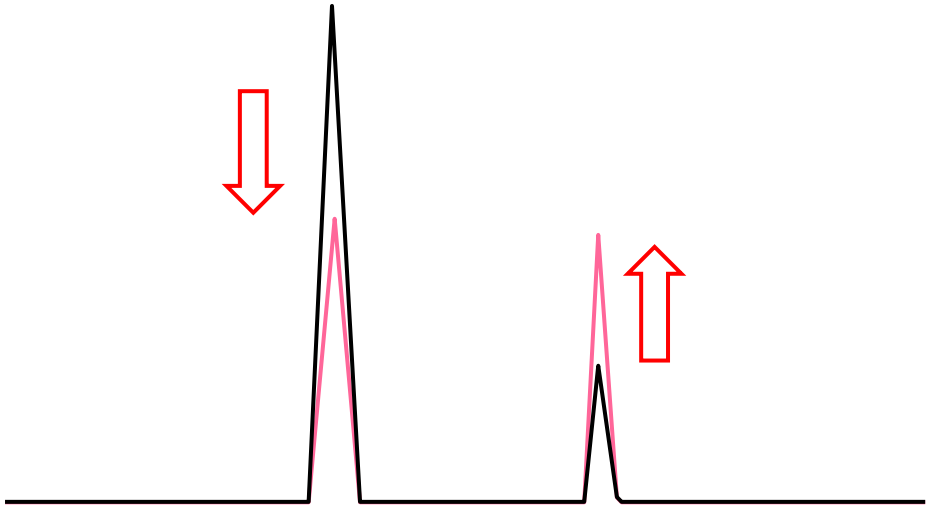


Specimen Error
Preferred Orientation

Random Orientation

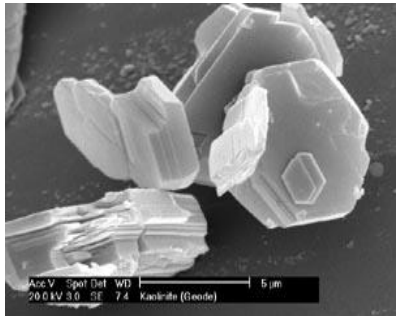


Redistribution of peak intensities



Biased Orientation

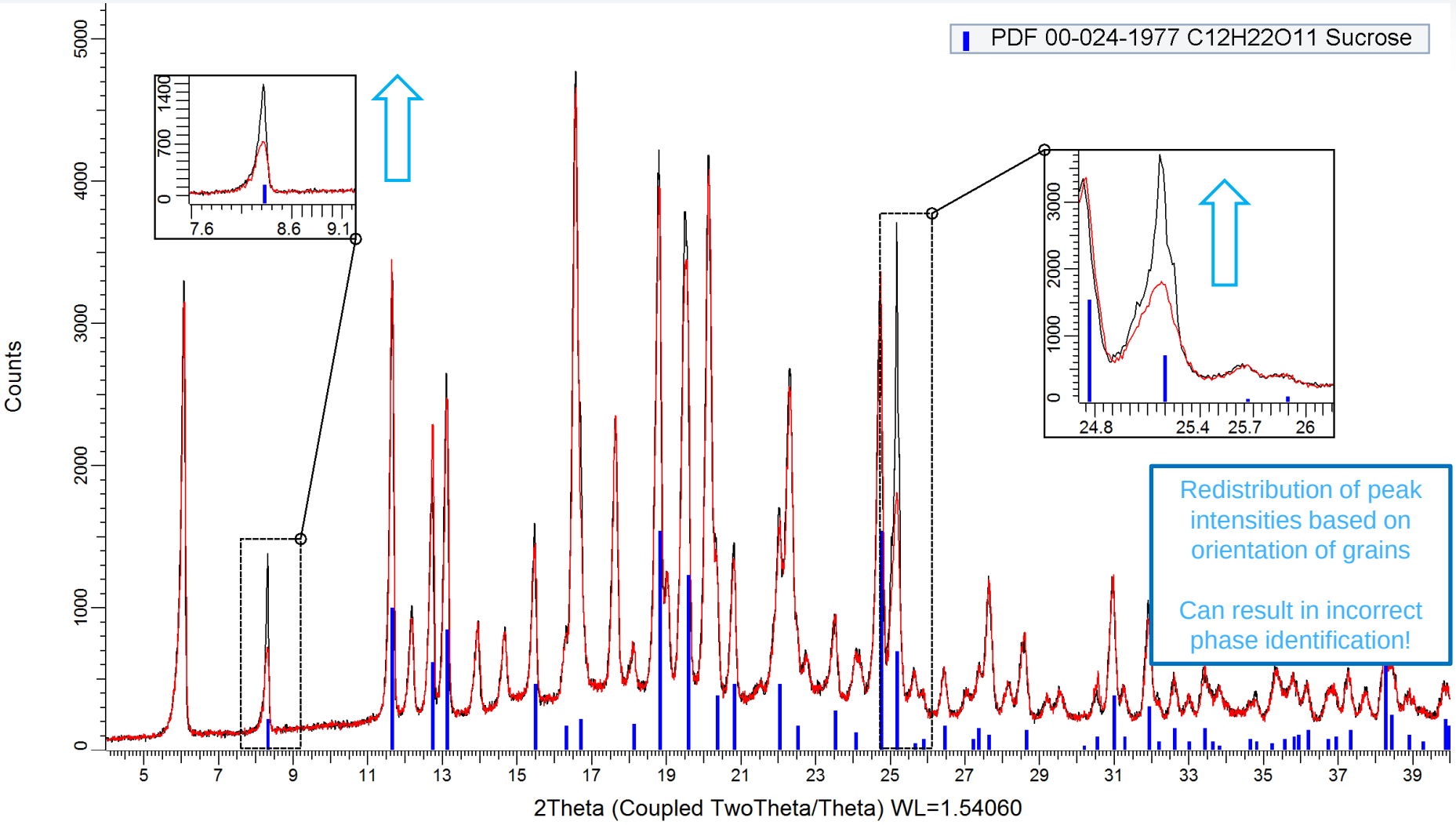
Halloysite



Kaolinite

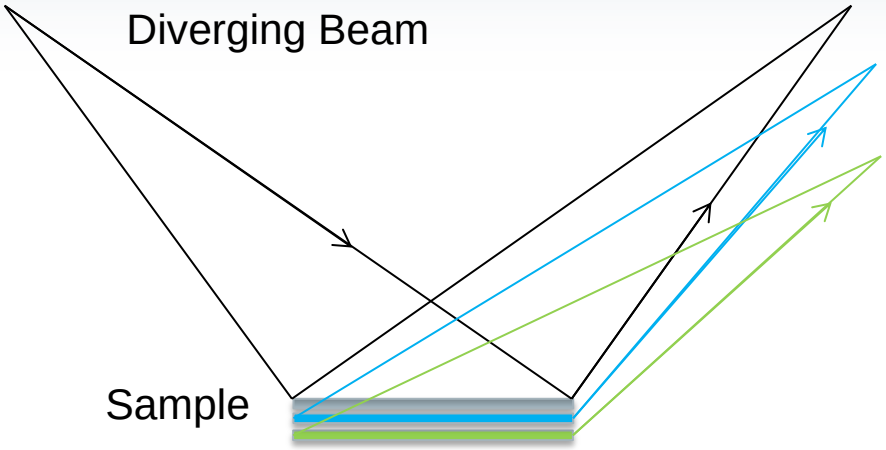
Practical Considerations

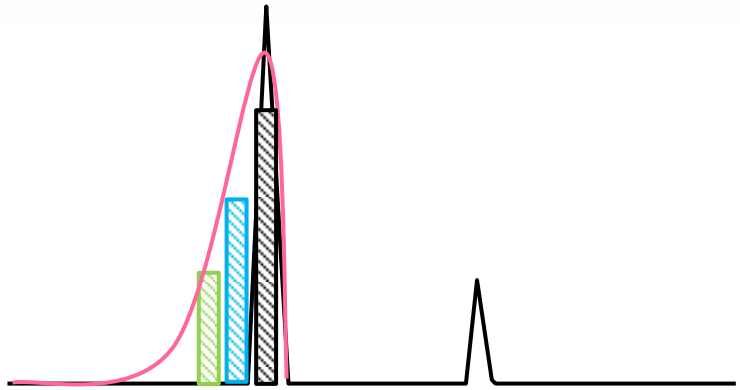
Preferred Orientation



Specimen Error

Specimen Transparency

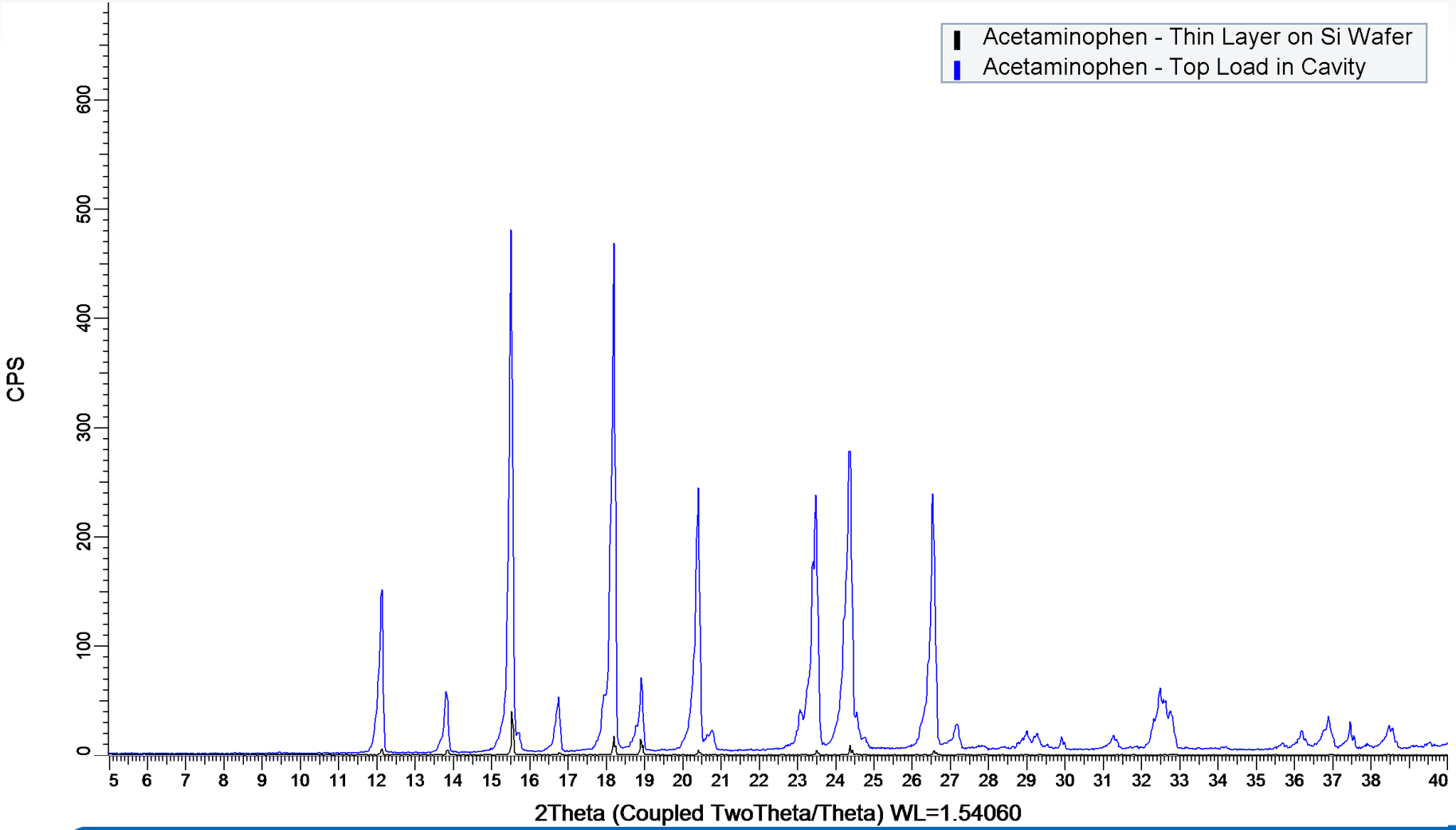




Lighter elements have greater sample penetration
Gradient of diffracting planes
Peak asymmetry

Practical Considerations

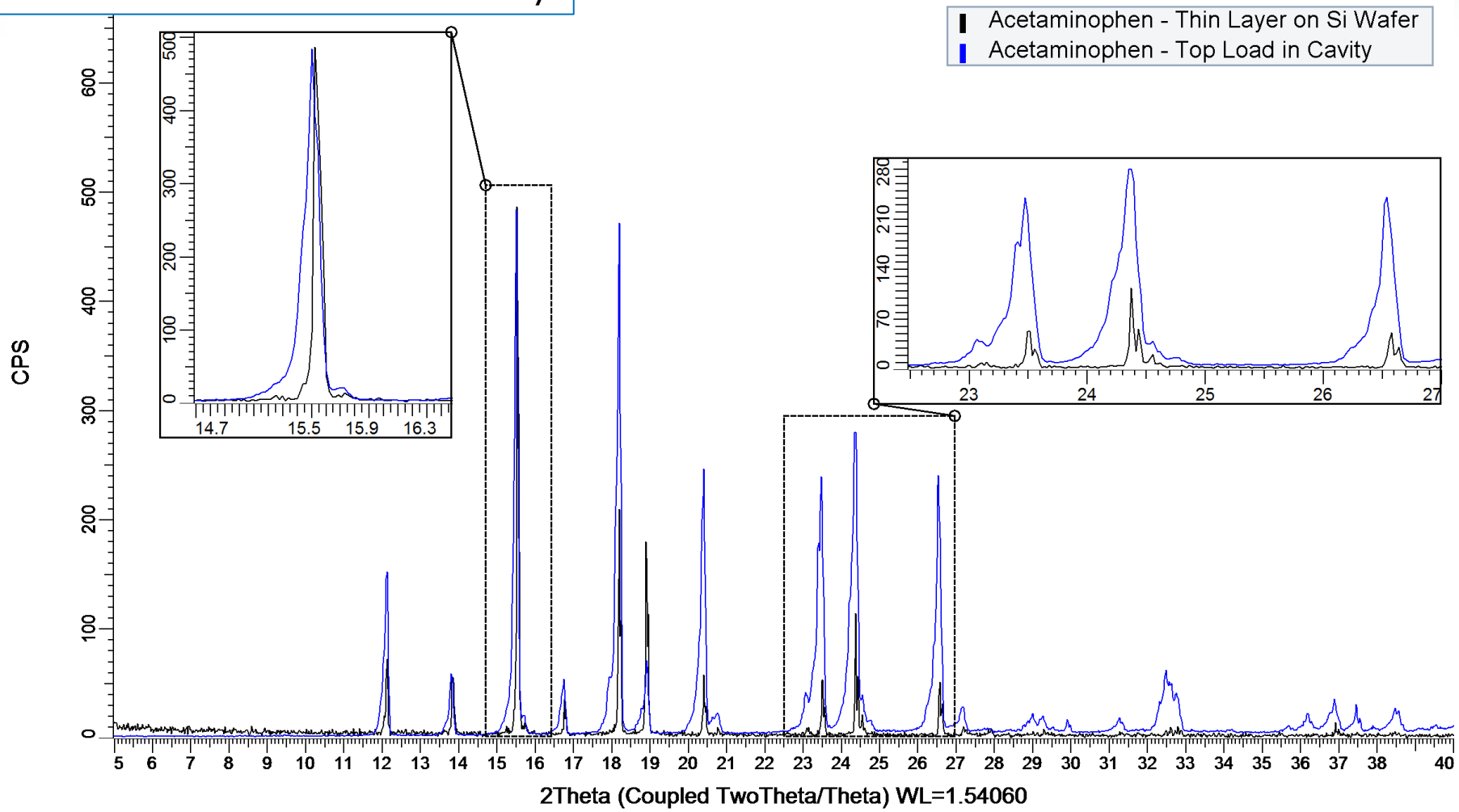
Specimen Transparency



Practical Considerations

Specimen Transparency

Normalized to maximum intensity

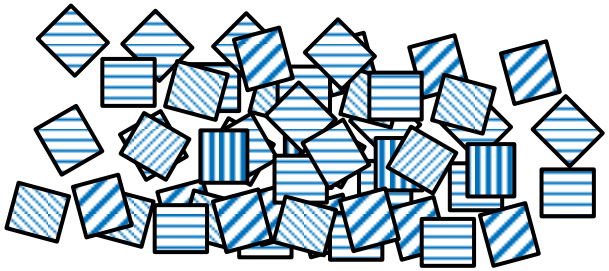


Specimen Error

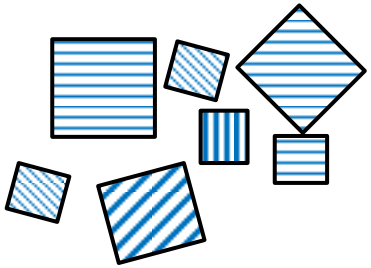
Large Particle Sizes or Limited Statistics

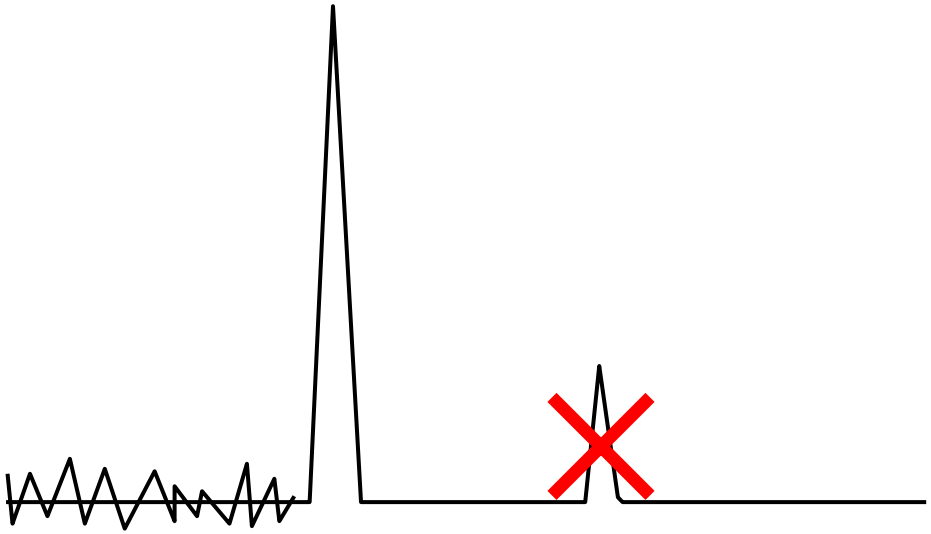
Noisy Background, Missing Peaks, Incorrect Intensities

Good Sampling Statistics



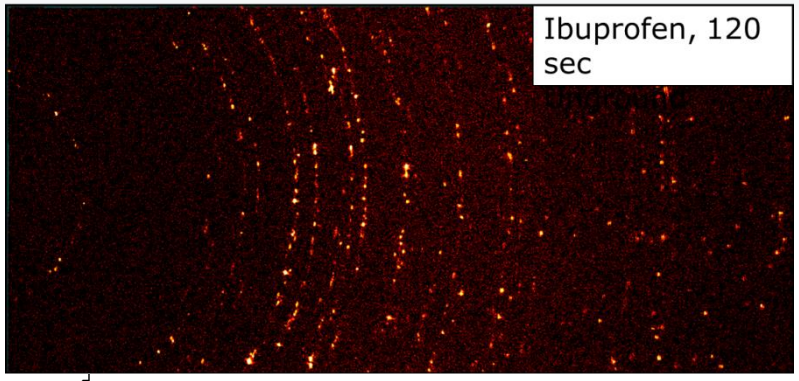
Poor Sampling Statistics

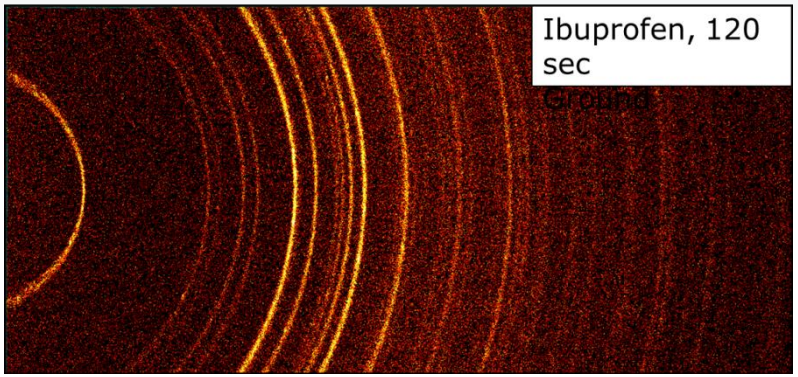


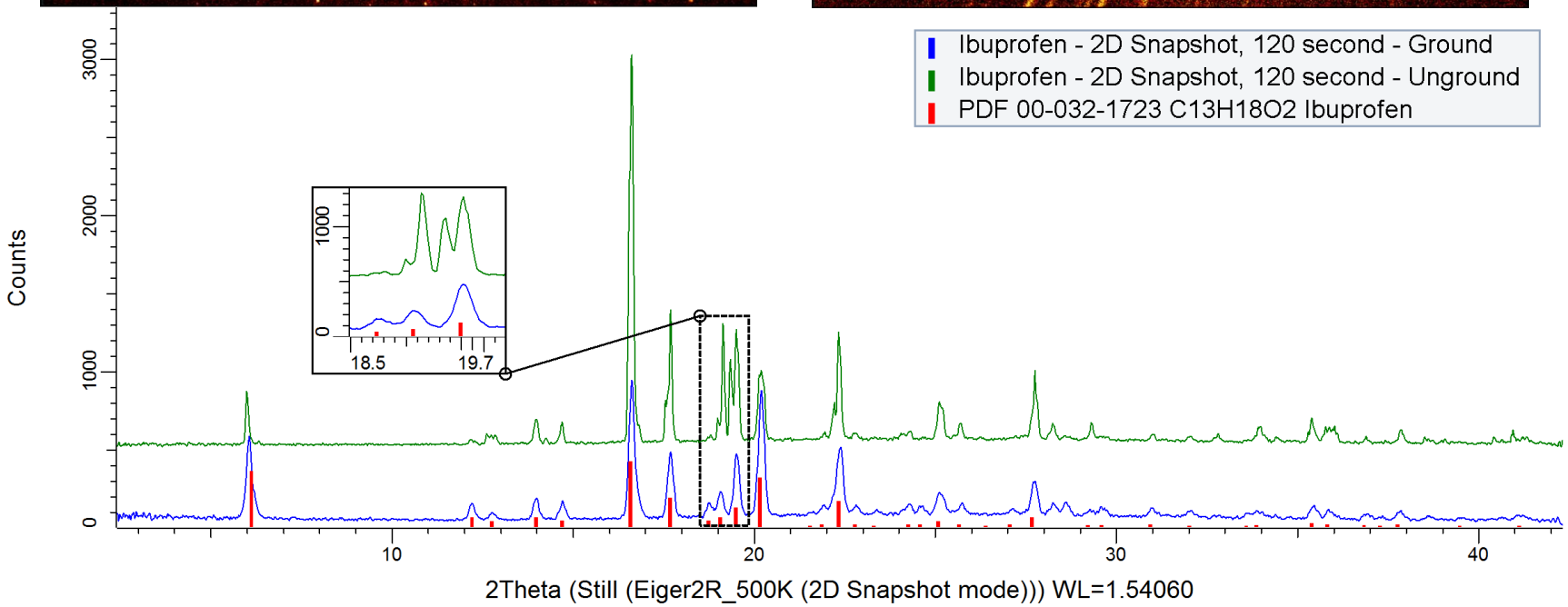


Practical Considerations

Coarse or Grainy Samples





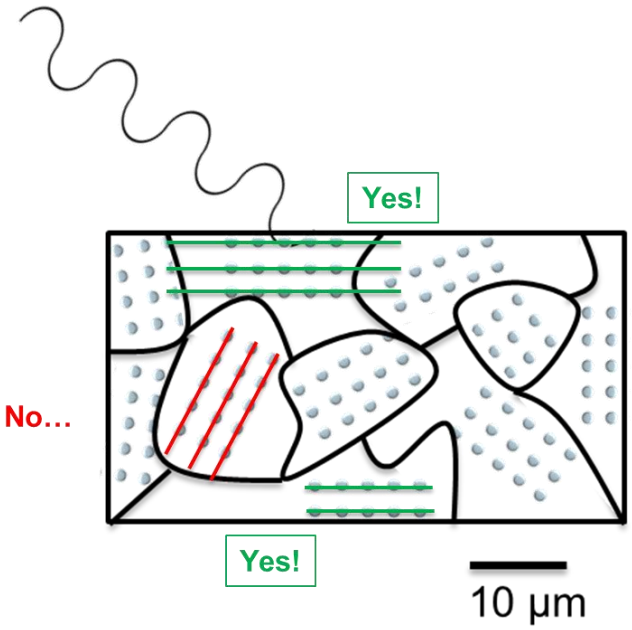


Reproducible Diffraction Intensities

Particle Size and Sample Grinding/Crushing

- Accurate, reproducible diffraction intensities require small crystallite sizes
- Number of diffracting crystallites is directly related to size

Diameter	40 micron	10 micron	1 micron
Crystallites / 20 mm^3	597 000	38 000 000	3 820 000 000
Number Diffracting	12	760	38 000

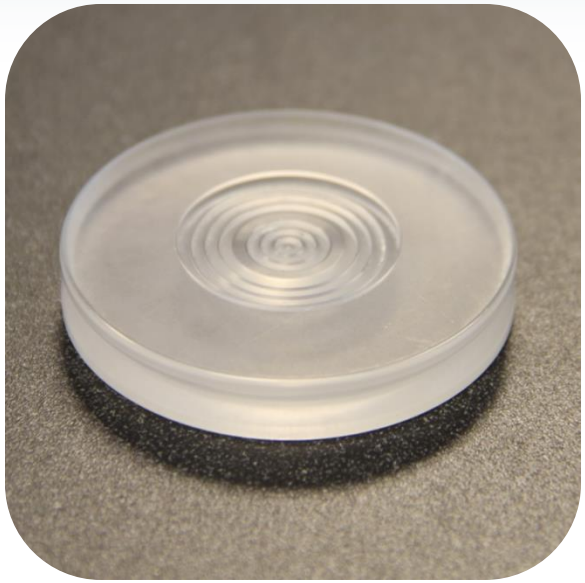


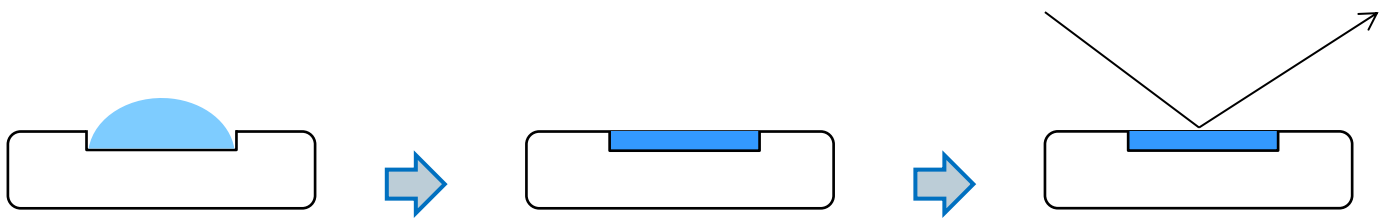
Aim for a smooth powder
Crush (don't grind) organic samples
Rotate or spin to increase particle statistics

Smith, D.K. "Evaluation of the Detectability and Quantification of Respirable Crystalline Silica by X-ray Powder Diffraction Methods" *1992*

Frontload Powder Holder

C79298A3244D82/D84





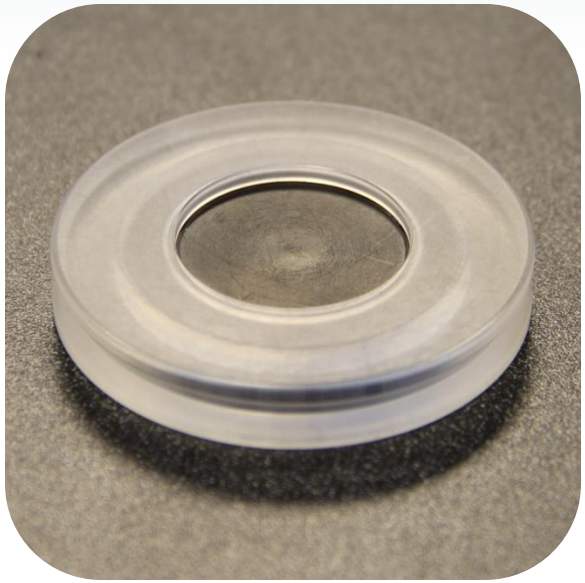
1) Fill

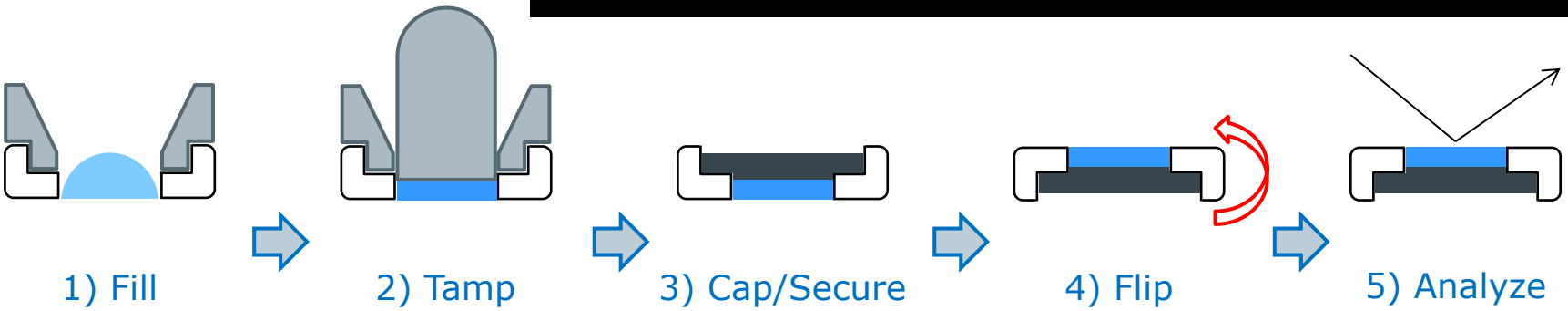
2) Smooth

3) Analyze

Backload Powder Holder

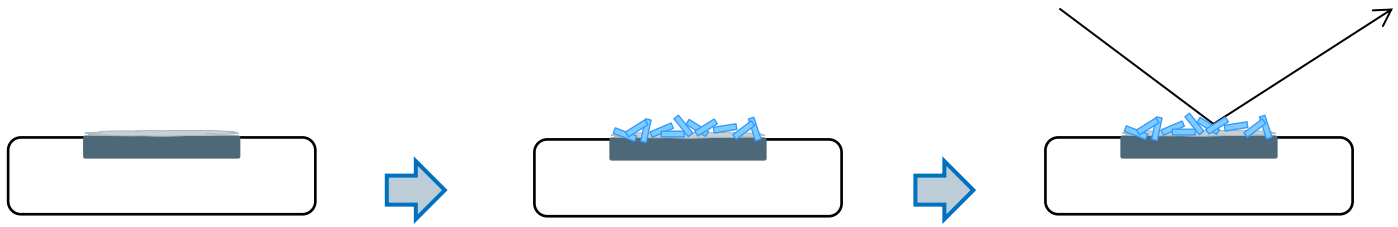
C79298A3244D88/D89





Si Low Background Holder

C79298A3244B249



1) Smear

2) Dust

3) Analyze

Guidelines for XRPD Specimen Preparation

1) Sample height is critical. Take your time and get it right.

2) Make a smooth powder (< 45 micron or -325 mesh).

3) Use as much sample as possible. Cover the sample holder.

4) Use a gentle hand with organic compounds.

5) Practice!