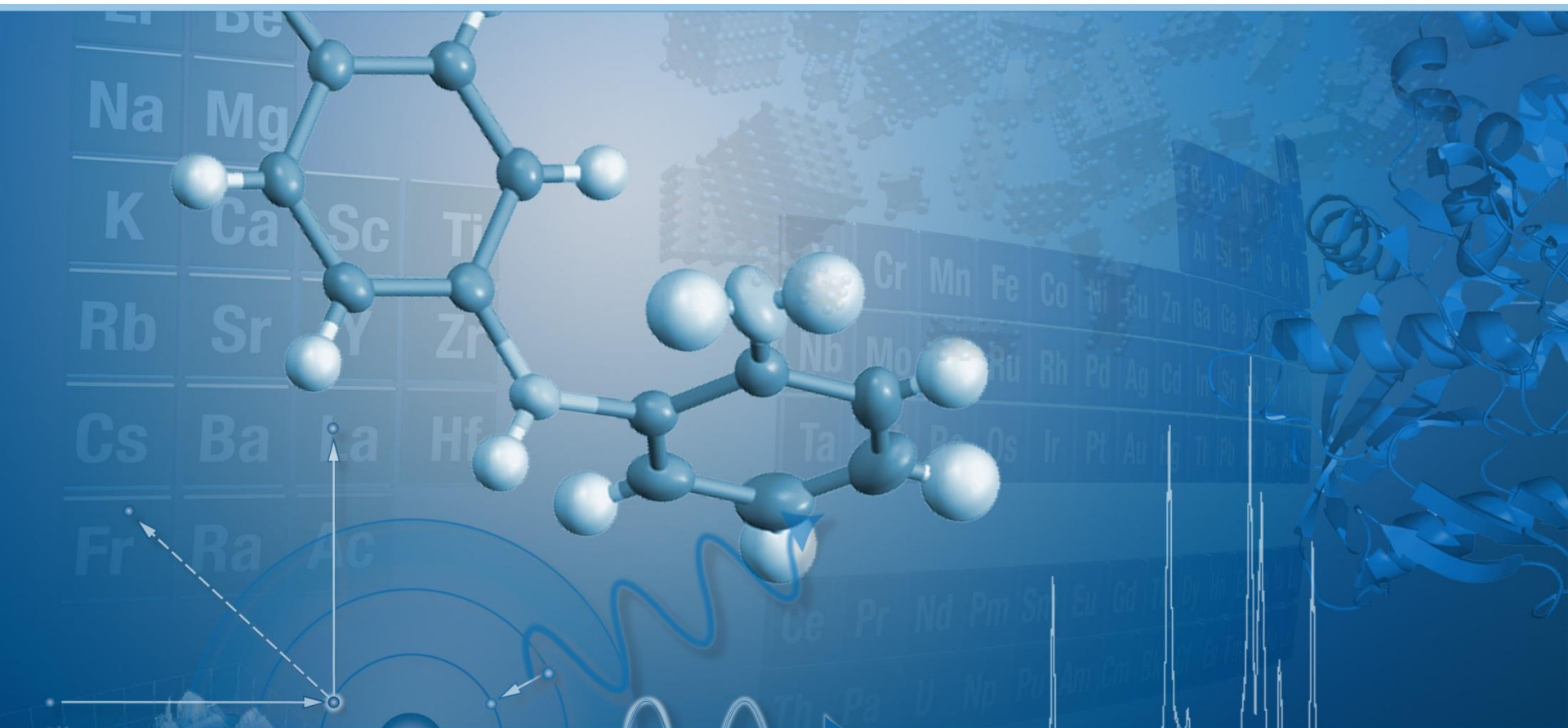# Sample Preparation and Laboratory Instrumentation

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This document was presented at PPXRD - Pharmaceutical Powder X-ray Diffraction Symposium

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# Introduction

- Consider sampling issues

    - Sample-related problems

    - Appropriate sample preparation

        - Adequate particle statistics
        - Random particle orientation

- Think about the sample presentation (instrument) geometry

    - Bragg-Brentano geometry

        - Always focusing
        - Always flat plate reflection

        Highest intensity (LLOD, LLOQ)
        Best resolution

    - Debye-Scherrer geometry

        - Parallel or focusing beam
        - Flat plate reflection (parallel beam)
        - Foil or capillary transmission (parallel and focusing beam)

        Minimal sample issues

Bragg-Brentano Geometry
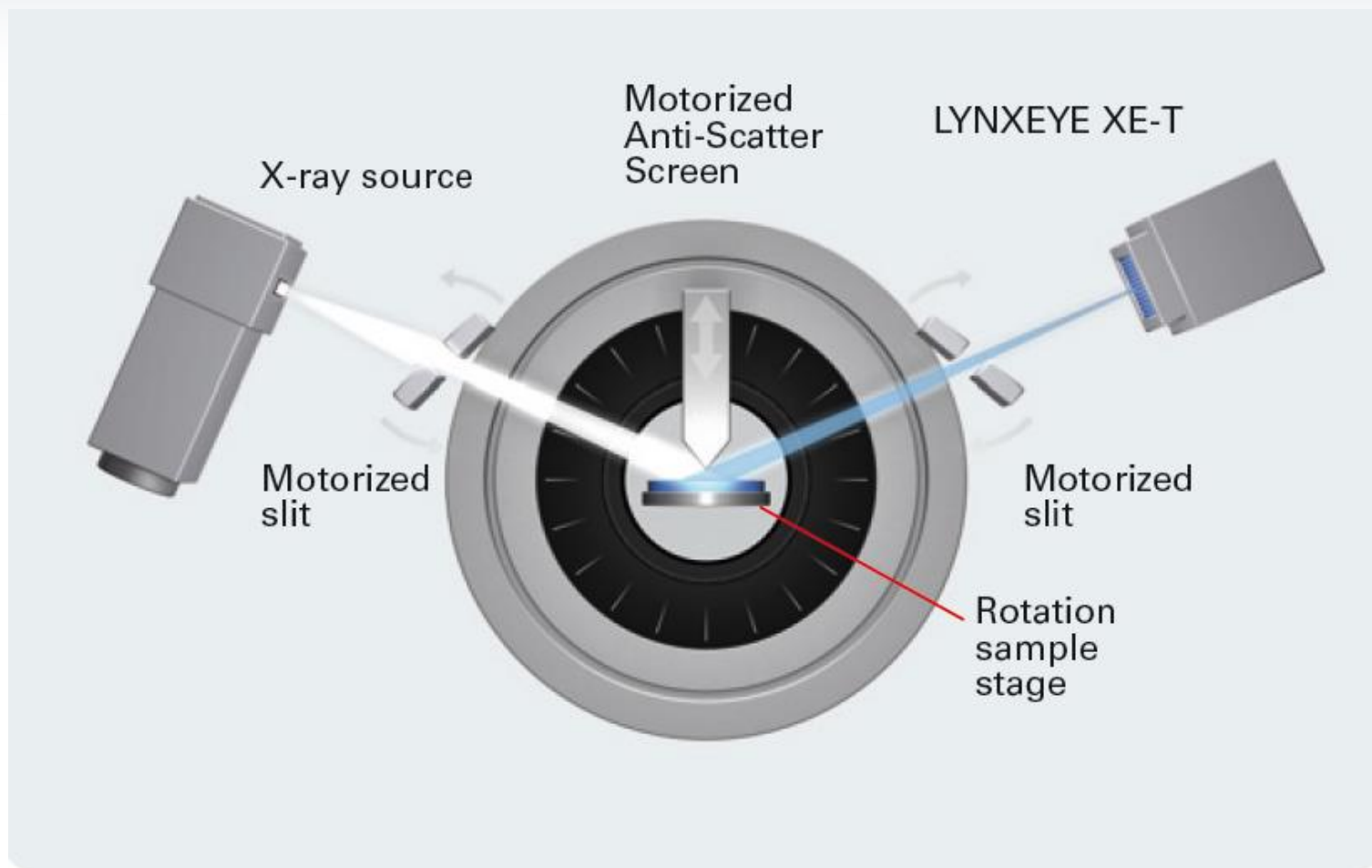
Bragg-Brentano Geometry

- Focusing geometry, flat plate reflection

- Highest intensity, best resolution

  - Best LLODs and LLOQs

- Requires relatively large sample amounts

- Preferred orientation issues

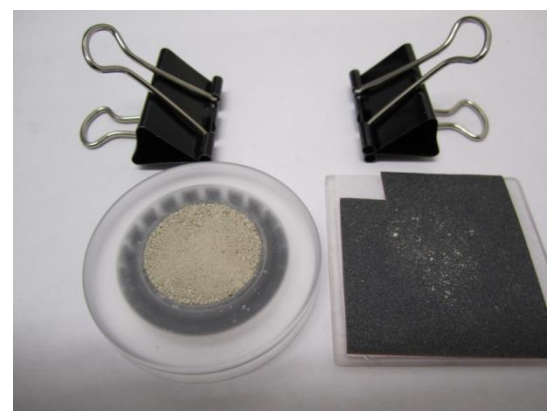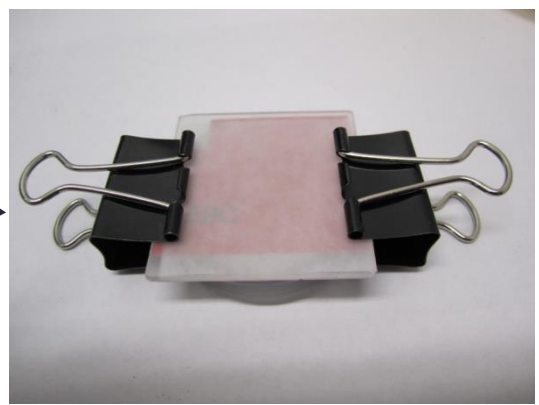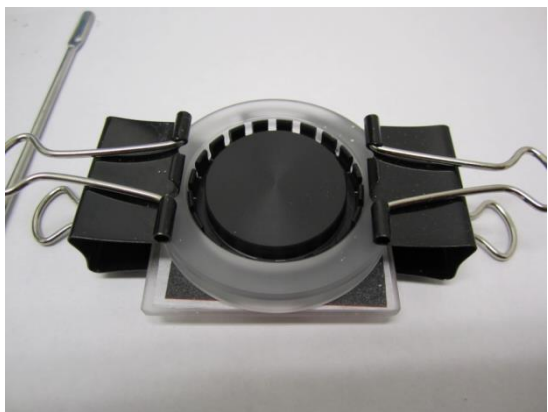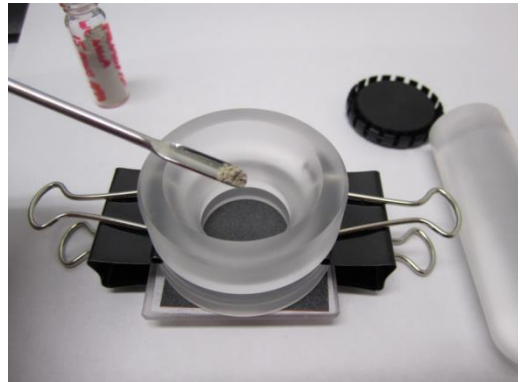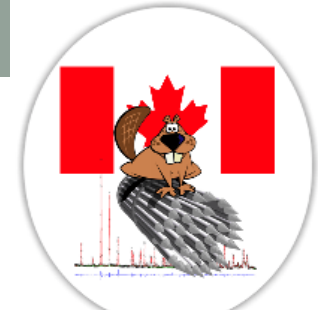- Sample transparency issues (organic materials!)

Top-loading

- Simplest but most prone to inducing preferential orientation
- Special holders often in this category
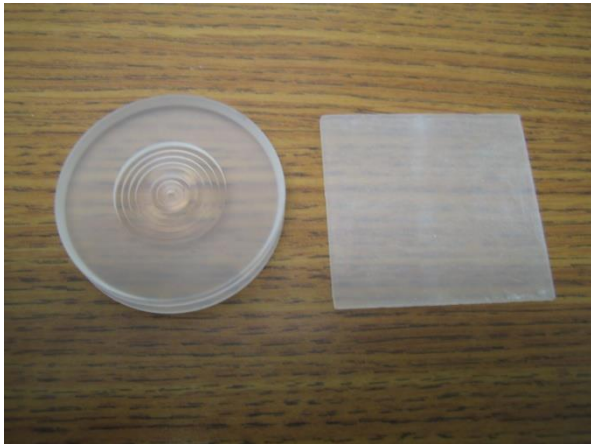
Alternative holders such as cavity zero background silicon or air-sensitive often top-loading as well
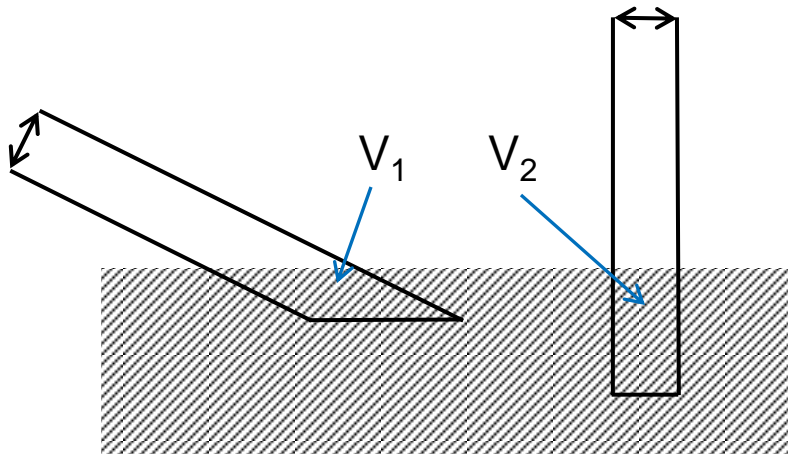
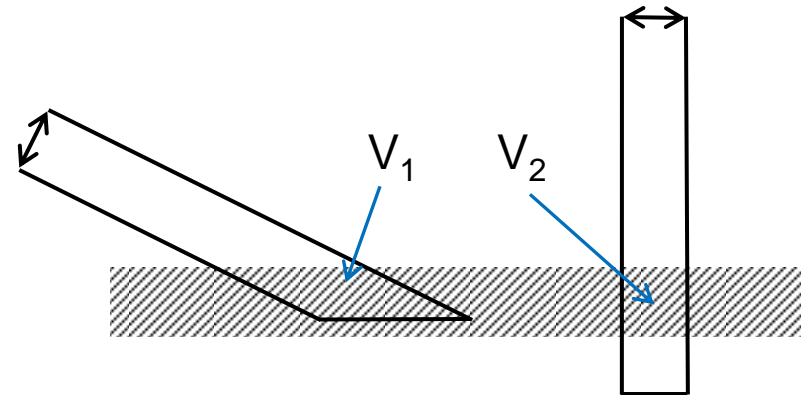# Back-loading

Flat Plate Reflection

For accurate peak intensities

- Use an infinitely thick sample
- High angle intensities reduced if required thickness inadequate
- Beam footprint must not exceed sample length at low angles

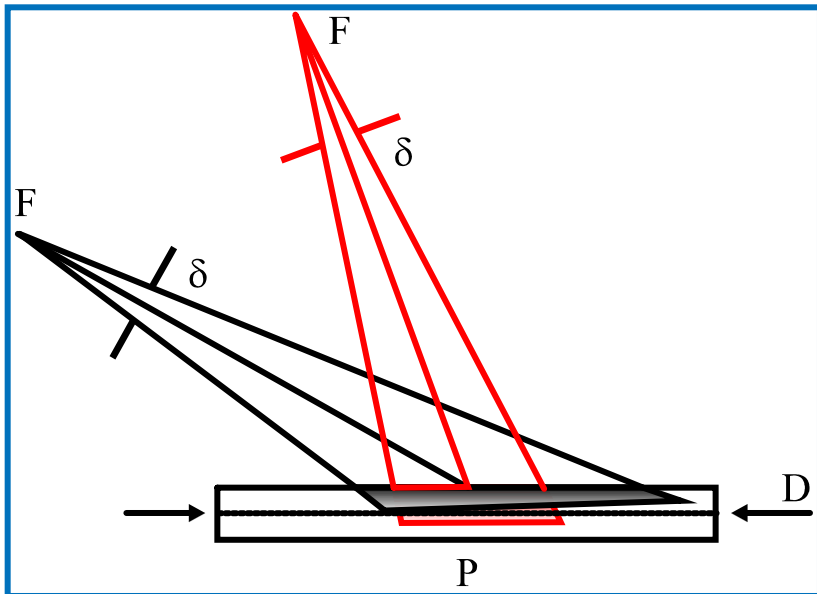**Thick sample:** constant diffraction volume $V_2 = V_1$

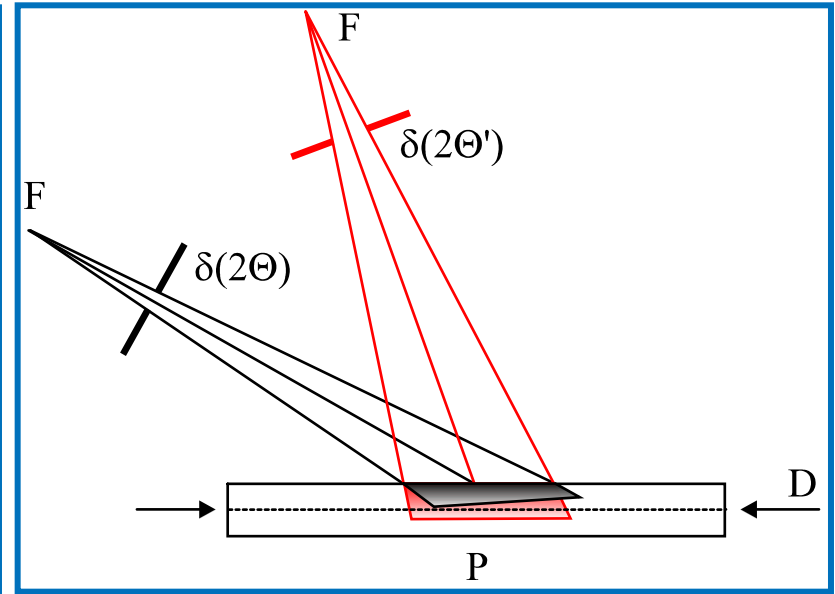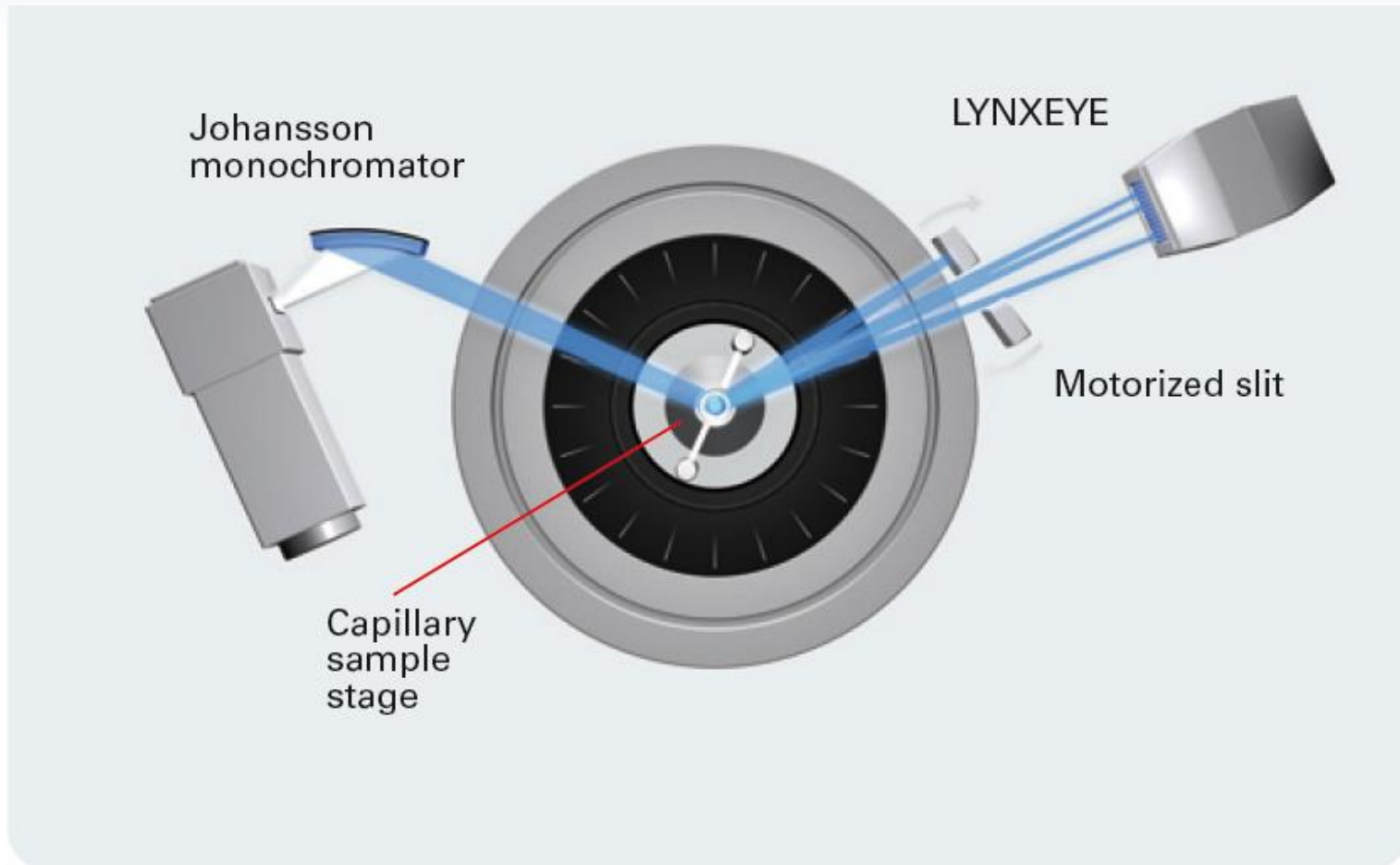**Thin sample:** lower diffraction intensity at higher angles $V_2 < V_1$

Flat Plate Reflection

- Fixed divergence slits

- Variable divergence slits

- Constant diffraction volume!

- Variable diffraction volume requires SW correction!
- Avoid if possible!

Debye-Scherrer Geometry

Debye-Scherrer Geometry

- Parallel beam - Göbel mirror

  - Modest resolution, good intensity

  - Flat plate reflection, foil or capillary transmission

- Focusing beam - focusing Göbel mirror

  - Intermediate resolution, high intensity

  - Foil or capillary transmission

- Focusing beam - primary monochromator

  - High resolution, poor intensity, pure $K\alpha_1$

  - Foil or capillary transmission

- Requires small sample amounts but tedious sample preparation

- Allows to minimize preferred orientation issues

- XRPD + SAXS (Cu radiation), XRPD and PDF (Mo radiation)

Foil Transmission

- Sample is sprinkled between 2 foils, and X-ray beam shoots through

- Some instruments can mix flat plate reflection and foil transmission sample holders in sample changers

- Ideal for organic and other low absorbing samples

Capillary Transmission

- XRPD + SAXS (Cu radiation), XRPD and PDF (Mo radiation)

- Most effective at reducing preferred orientation effects (except needle-like morphology)

- Made from glass, borosilicate, quartz, Kapton or other low absorbing materials

- Different sizes (0.1 – 1mm diameter) depending on absorption
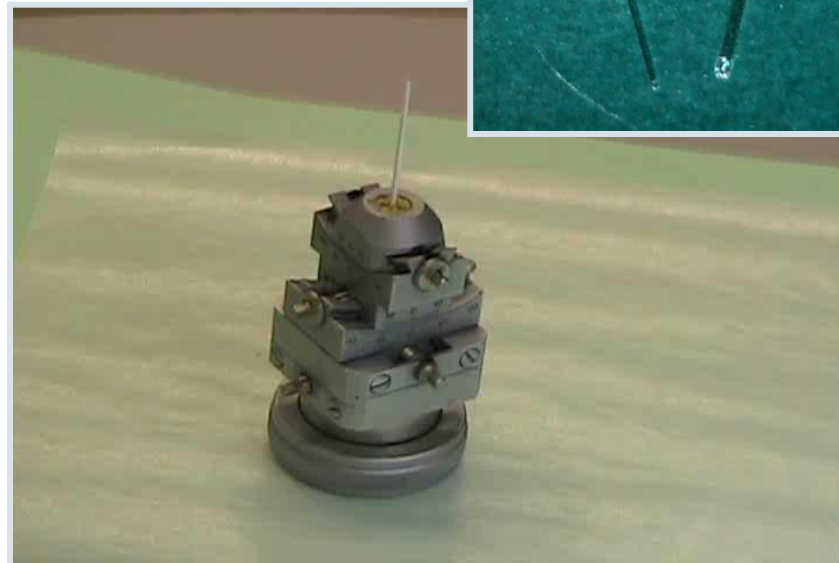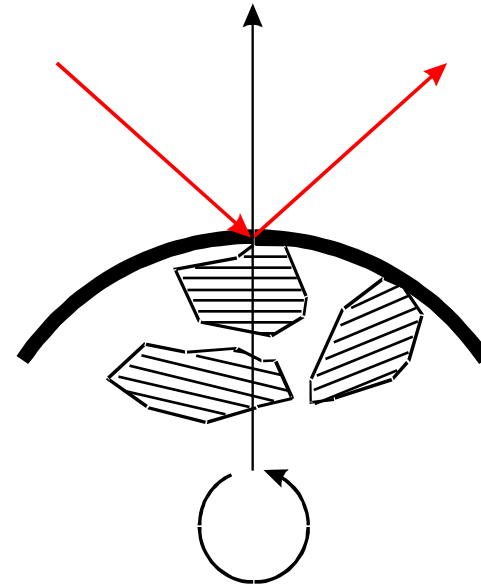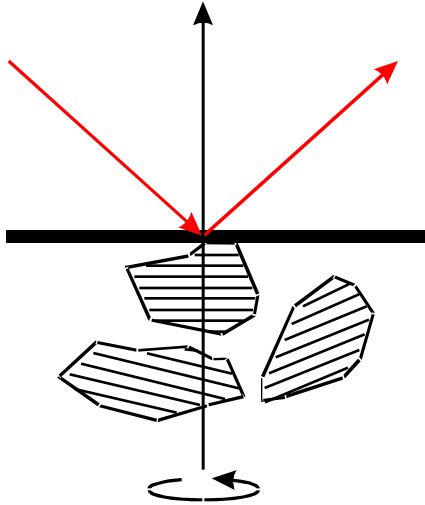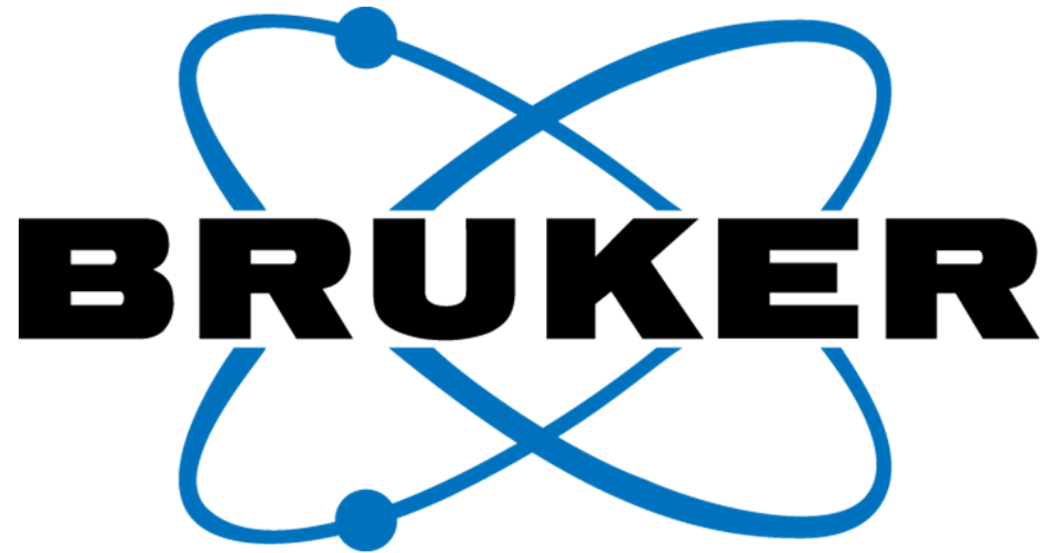
Sources of Error
Preferred Orientation

- Bragg-Brentano geometry

- Parallel or focusing beam Debye-Scherrer geometry using capillaries

Rotation parallel to the scattering vector does not minimize preferred orientation effects!

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