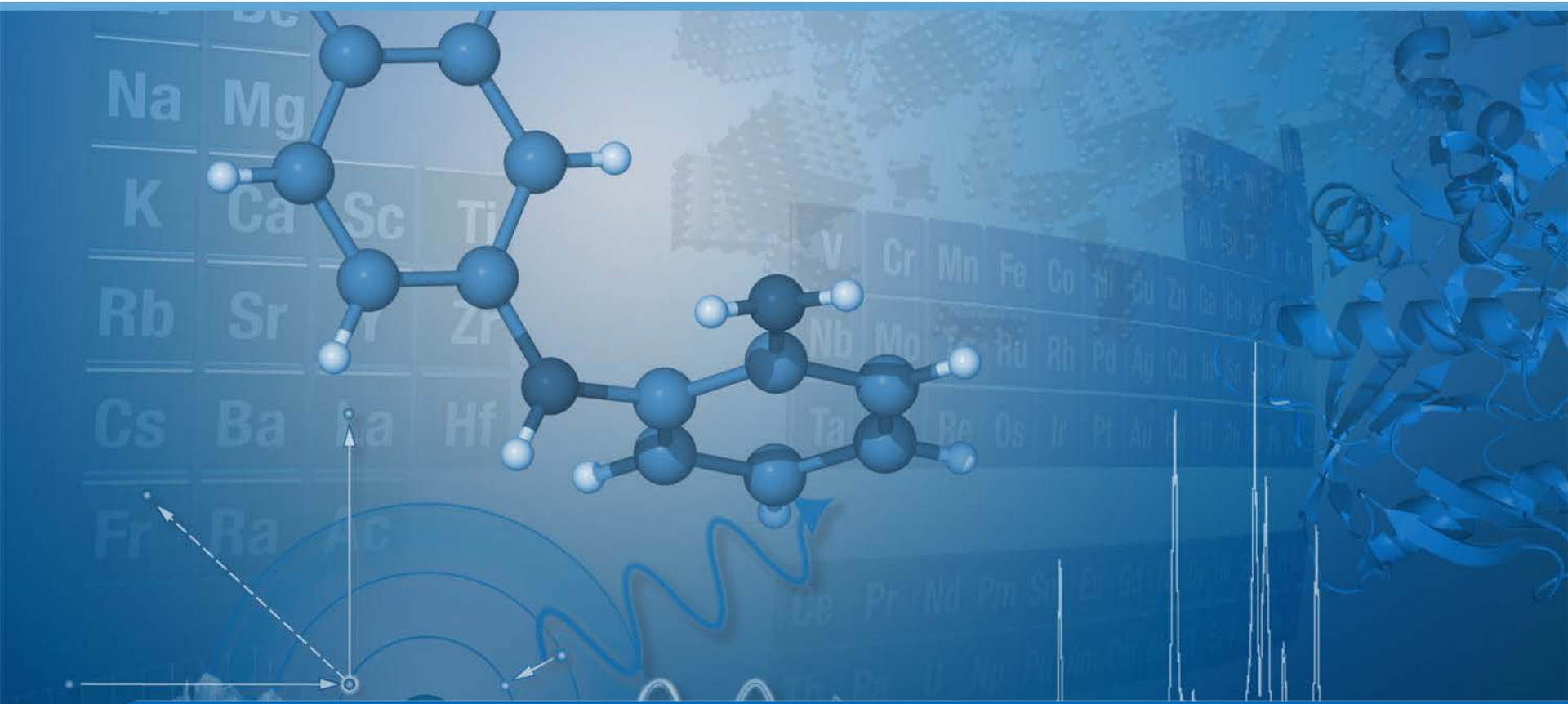
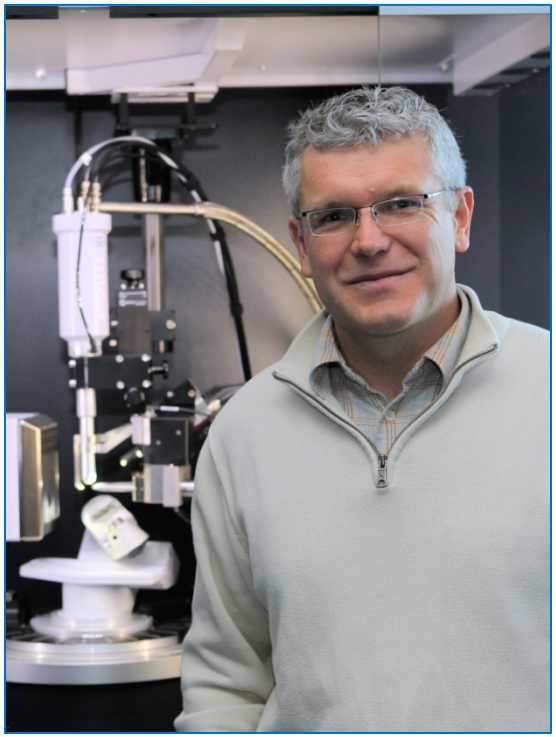
Fast, Intuitive Structure Determination I: Crystal Mounting



Welcome



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Overview

- Properly sized and mounted crystals improve data quality.
  - Minimize absorption effects.
  - Reduce background scatter.
  - Improve strategy optimization.

Overview

- Small-molecule crystals
- Tools and equipment
- Crystal supports
- Adhesives
- Selecting crystals
- Cutting crystals
- Mounting crystals

Growing Crystals

- *Growing and Mounting Single Crystals Your Diffractometer Will Treasure*
- www.bruker.com/service/education-training/webinars/sc-xrd.html

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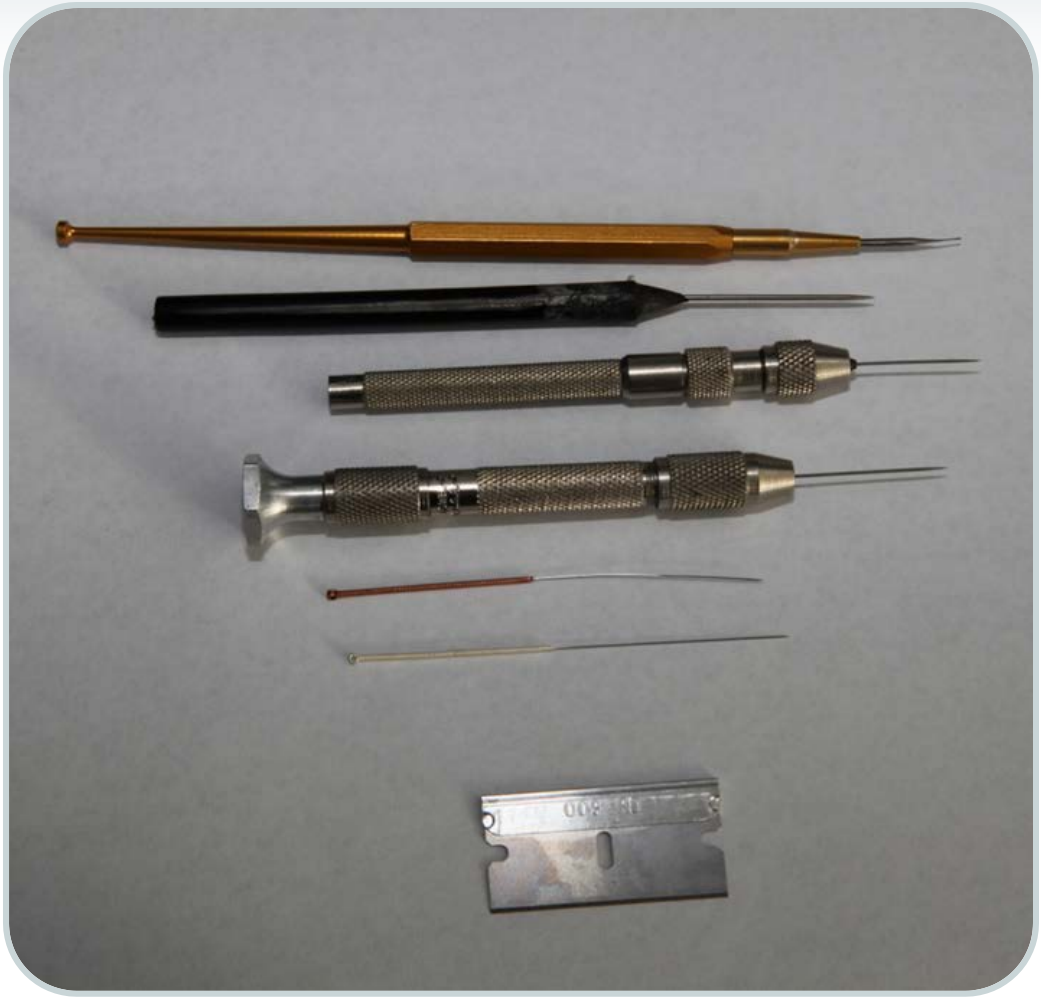
The microscope

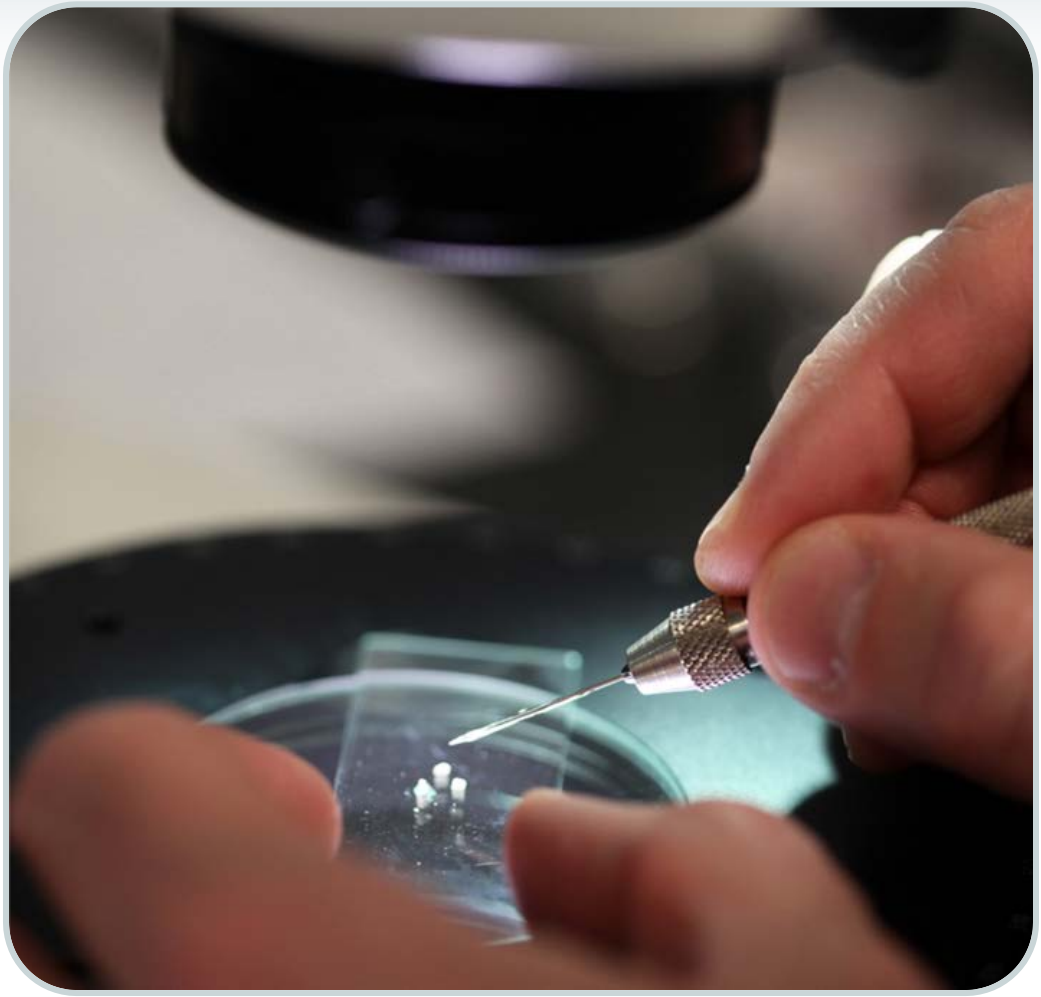
- Microscope requirements:
  - Good quality optics
  - 60-100x magnification
  - Polarizing stage
  - Good working height
  - Good light

Tools



- Few tools needed
  - Manipulation
    - Micro-tools
    - Dissecting needles
    - Pin vise/sewing needles
    - Acupuncture needles
  - Crystal cutting
    - Single-edge razor blade
    - Scalpel
    - X-acto knife

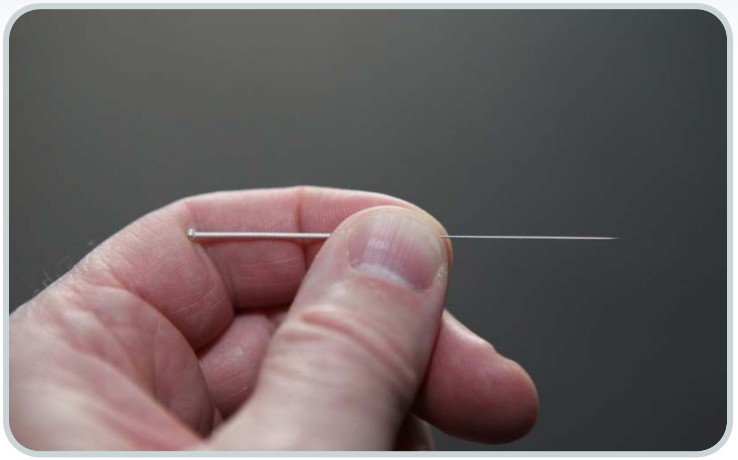
Choosing tools



- Comfortable to grip
- Easy to handle
- Matched to sample
  - Smaller tools for small specimens
  - Larger tools for large specimens

Handling tools

- Pin vise needs just a light grip
- Mass reduces vibration



- Acupuncture needle gripped more tightly
- Easy to flex with fingertips

Cutting tools

- Easy to handle
- Easy to position
- Precise cutting

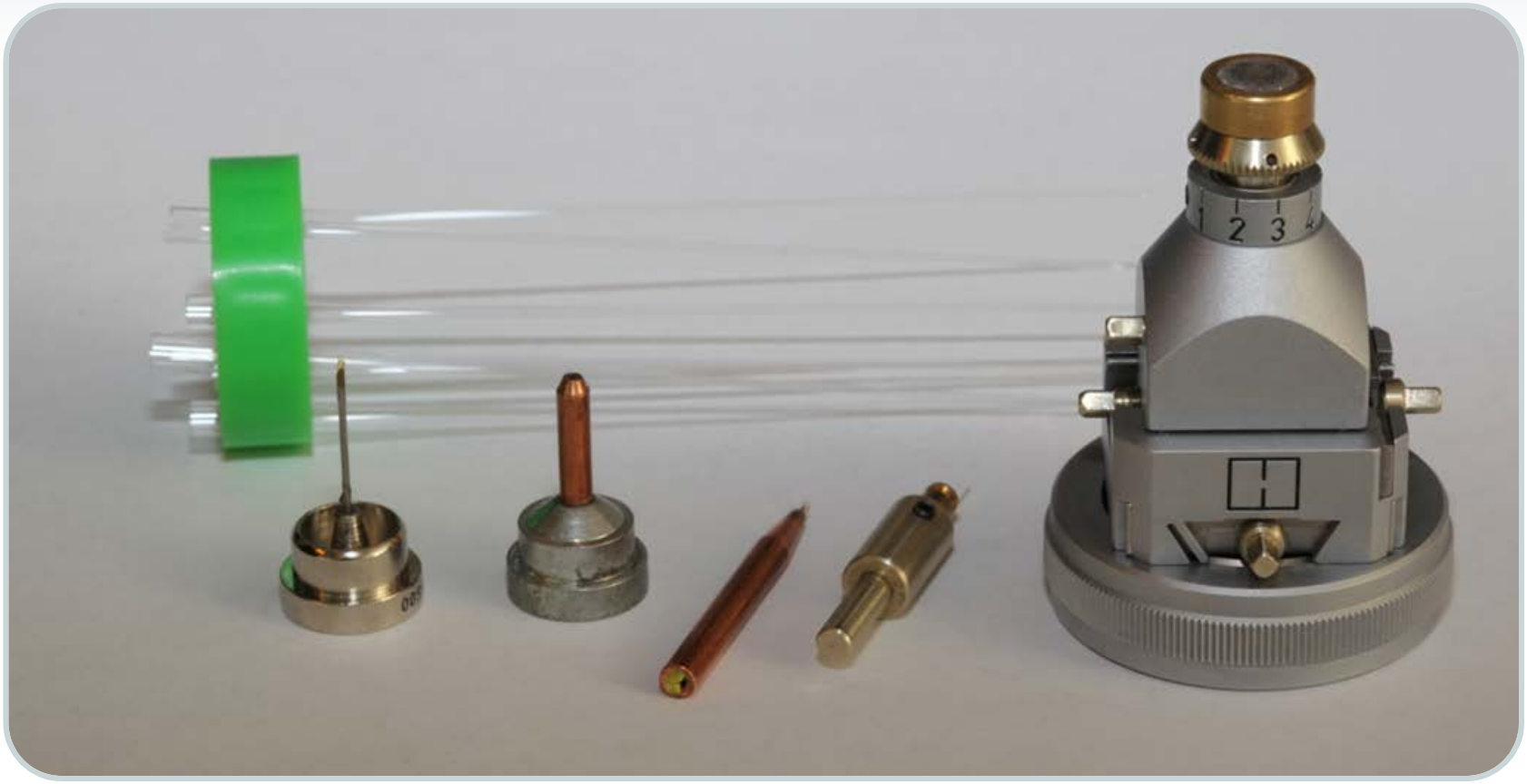
Mounting crystals

- Choice of adhesive affects background scatter and ease of handling.
- For room temperature:
  - Epoxy
  - Cyanoacrylate (Super Glue)
  - UV-curable adhesives

Mounting for low temperature

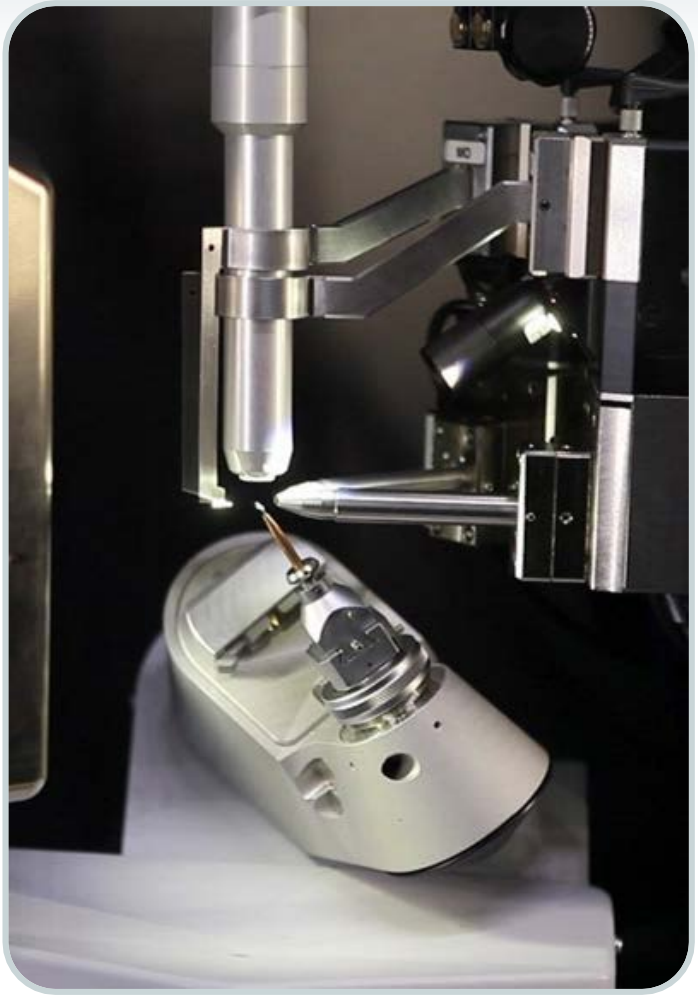
- Cryocrystallography allows for easy handling of routine and sensitive samples
- Material must harden or “glass” at experimental temperature
- Suitable compounds:
  - Hydrocarbon oil
    - Paratone-N
    - STP engine additive
  - Grease
    - High-vacuum grease
    - Apiezon
  - Fluorolube
  - Fomblin
  - Karo syrup

Choosing a mount



There are many options to support the crystal in the instrument.

Choices for mounts

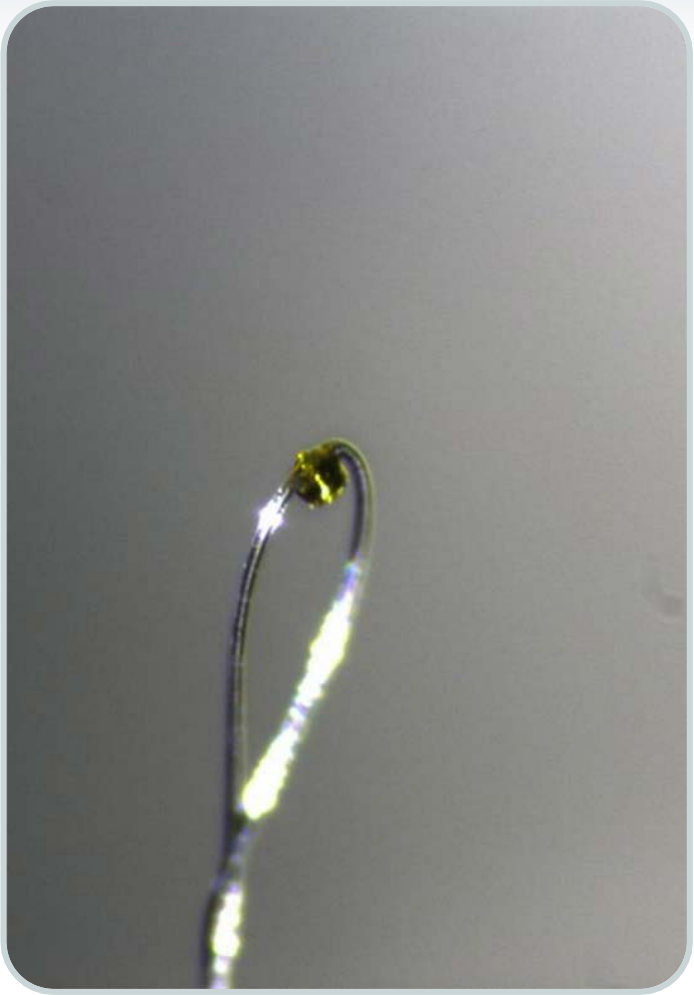


- Different mounts have different properties.
  - Glass fiber
    - Inexpensive
    - Easy to make on demand
    - Fragile
    - Background scatter
  - Monofilament loops
    - $ 1.00 unmounted
    - $ 7.00 on pin
  - Polyimide mounts (MiTeGen)
    - Many sizes and shapes
    - Easy to assemble
    - Easy to mount
    - $ 5.00
  - Capillaries
    - Good for air-sensitive materials
    - Difficult to mount
    - Poor low-temperature performance
    - $ 2.50

Capillary

- Used for air- and moisture-sensitive crystals
- Some background scatter
- May cause ice formation
- Fragile
- Difficult to load
- Sealed by flame or with wax or clay
- Solvent-soaked glass-wool plug to prevent desolvation

Cryoloop



Mounted crystal provided by C. Moore, Univ. Cal. San Diego.

- Typically mounted on end of tapered copper pin
- Place crystal at center of loop or above
- Affix with oil or glue
- Suspend in mother liquor

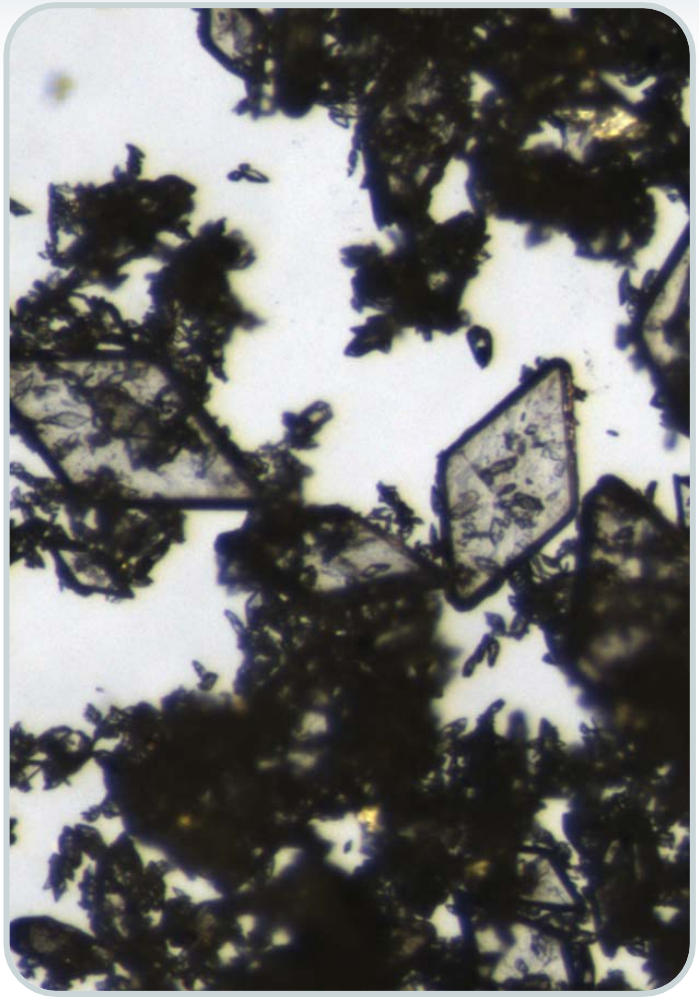
Glass fiber

- Inexpensive
- Pulled from glass cane
- Easy to vary thickness
- Some background scatter
- Mount to tapered copper pin for low-temperature work
- Affix crystal with small amount of oil or grease
- Choose fiber thinner than crystal

MiTeGen MicroMounts

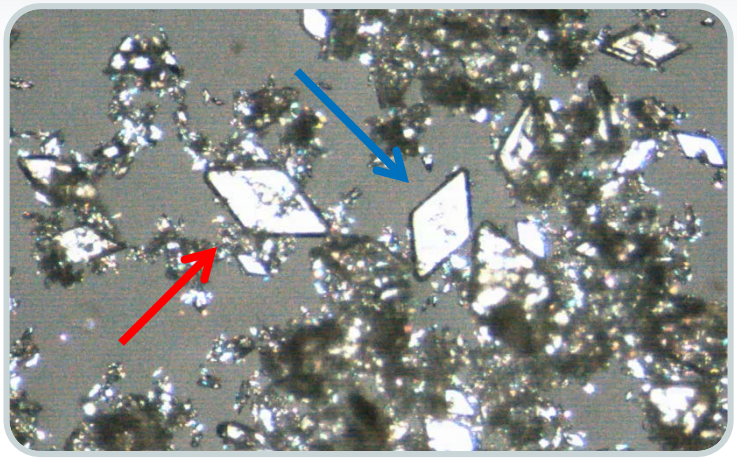
- Almost no background
- Easy to use
- Affix with small amount of oil or glue
- Many different styles and sizes

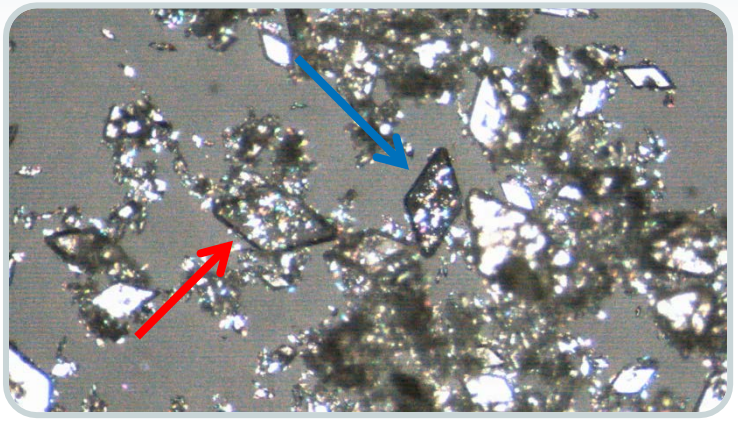
Selecting a crystal



- Choose crystal matched to size of beam
- Free of defects
- Extinguishes plane-polarized light

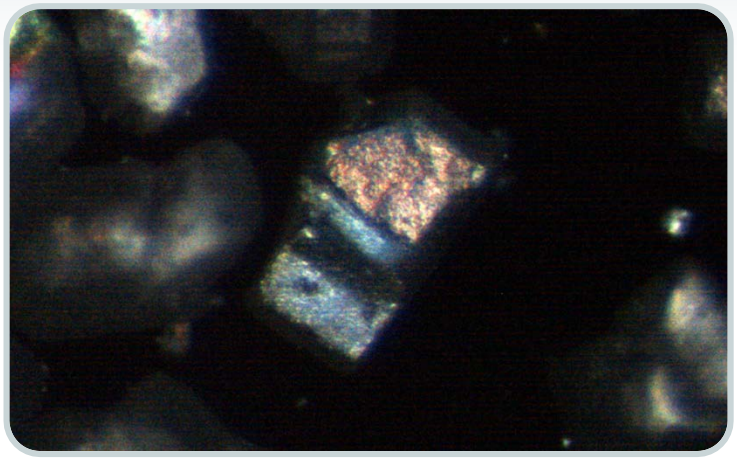
Inspection with crossed polarizers



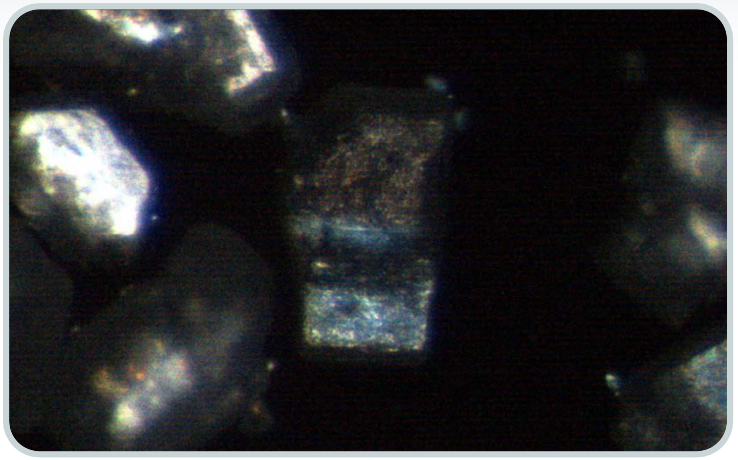


- Crystals are birefringent
- Extinguish plane-polarized light
- On rotation, will pass, then block light

Twinned sucrose crystal



- Note split transmission of light
- Seam between domains is clearly visible

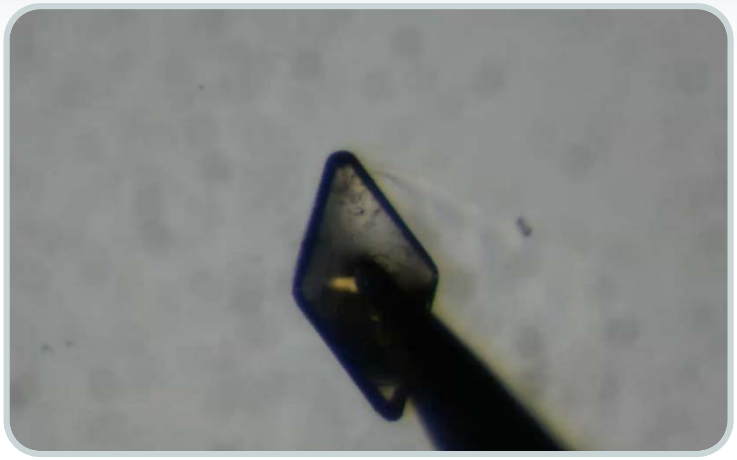


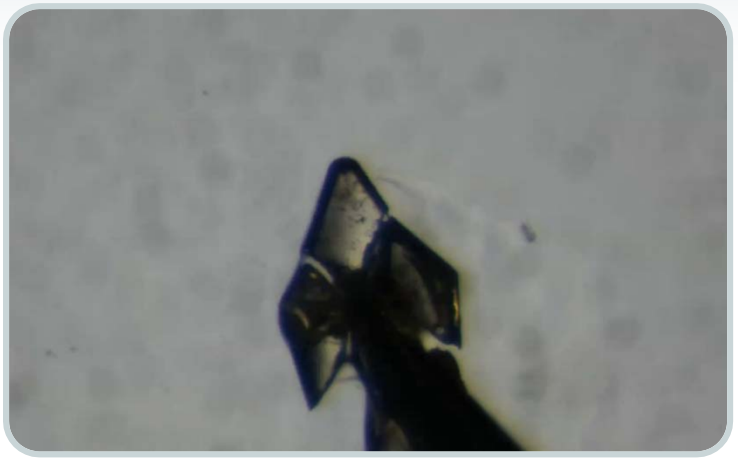
- 2 fragments behave differently on rotation under polarizer

Preparing the sample

By dredging crystal through oil, many small crystallites and other debris can be swept from the crystal.

Don’t crush crystals!

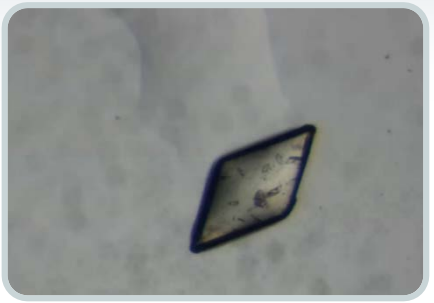


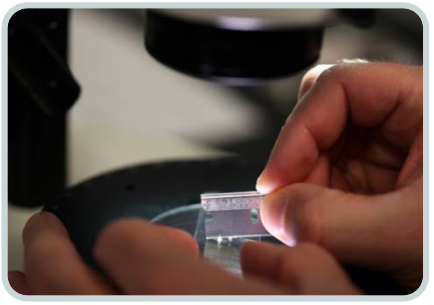


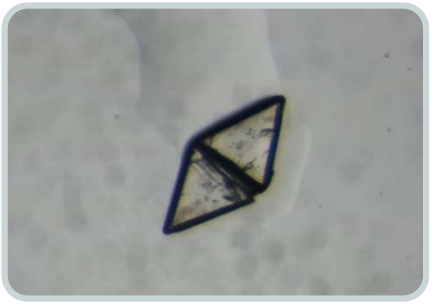
Crushing crystals with a needle or probe results in unpredictable fragmentation. There is much more control if a crystal is cut instead.

Cutting crystals

- Move crystal to open space on slide
- Use razor blade to make cut
- Brace tip of razor on slide to reduce motion
- Gently and smoothly press blade down to cut
- Crystal cleaves cleanly

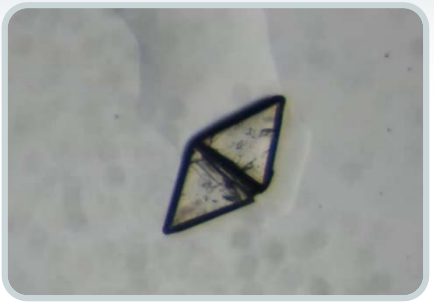


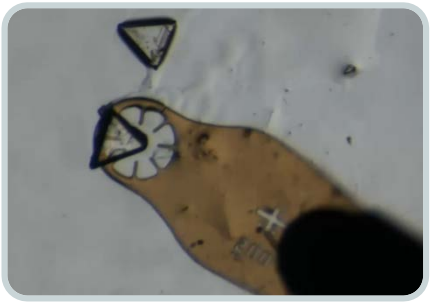


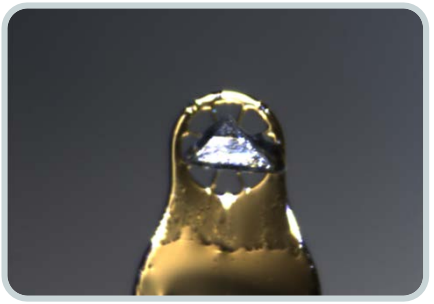


Mounting crystals

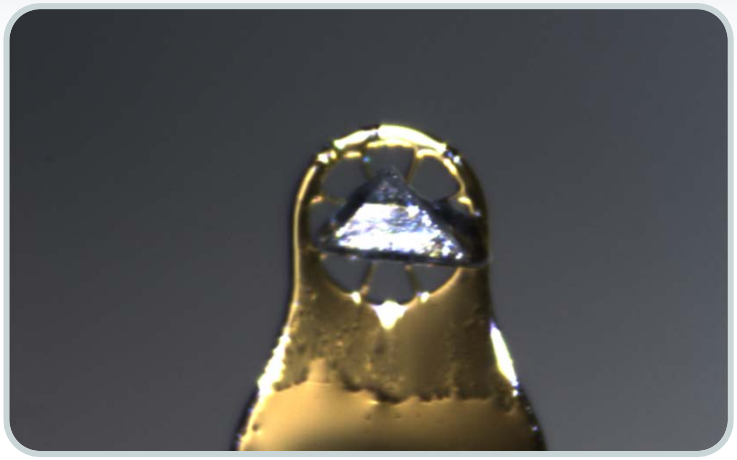
- Select specimen to be mounted
- Slide under crystal with mount
  - Push against crystal when using glass fiber
- Center crystal on mount
- Pick up crystal with minimum amount of adhesive



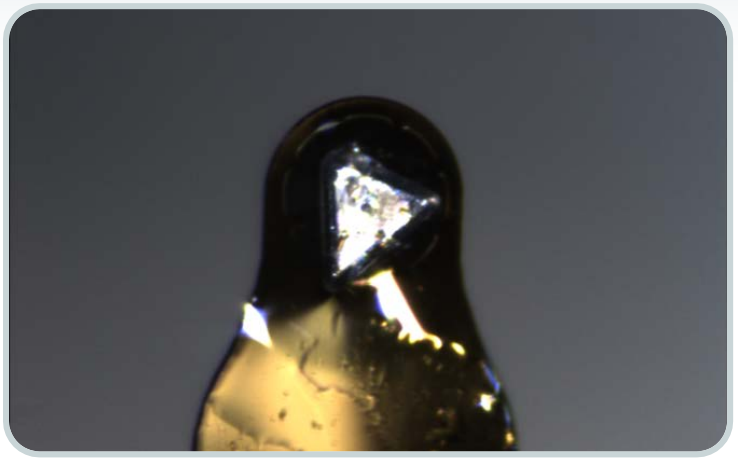




Mounting the crystal

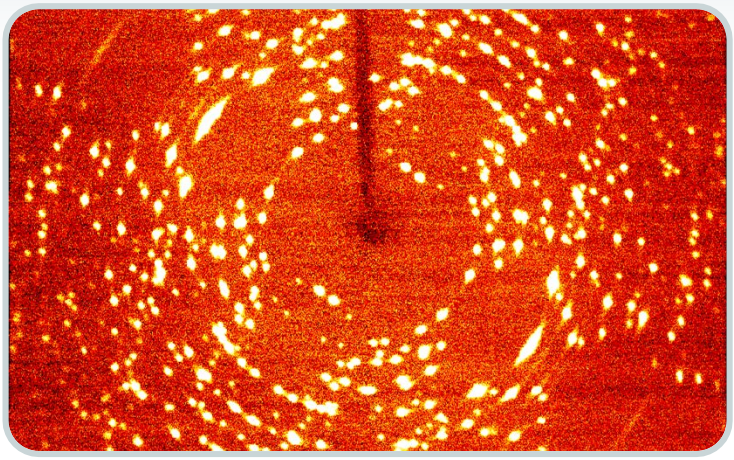


- Minimum amount of oil
- Minimize background scatter
- Easy to visualize edges

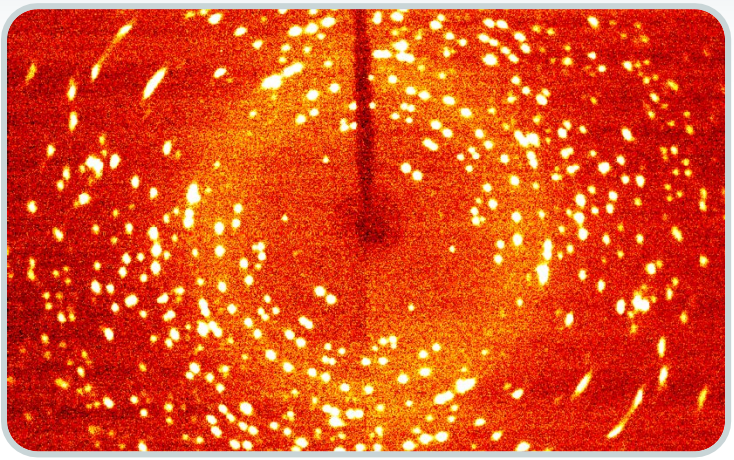


- Too much oil around crystal
- Creates background scatter
- Difficult to visualize crystal
- Crystal may slide in oil

Effects of mounting materials



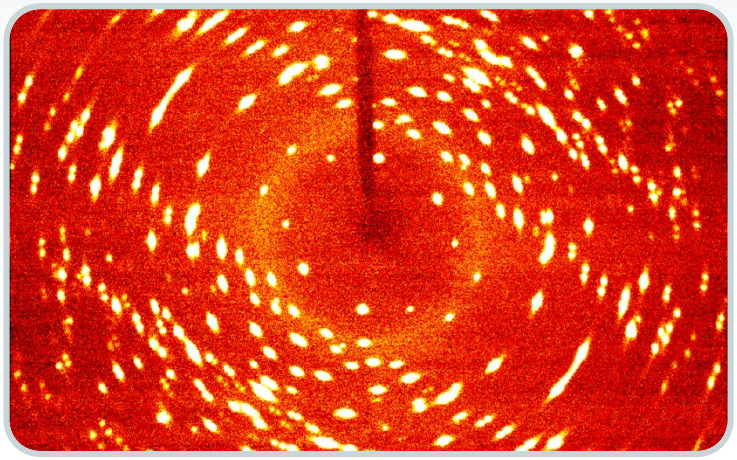
Crystal mounted with minimum oil

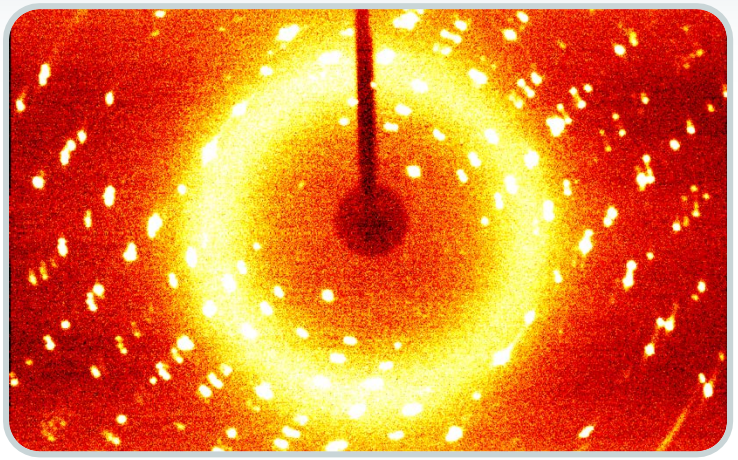


Crystal mounted with excess oil

Excess oil will produce excess background scatter.

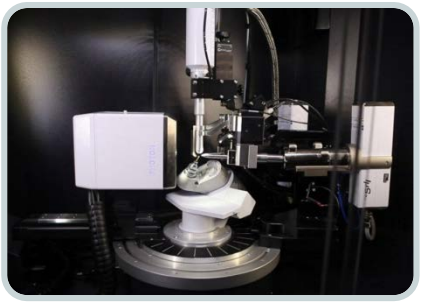
Mounting with a glass fiber





Glass also produces background scatter. Oil exacerbates the problem.

Crystal selection and mounting



- Use a quality stereomicroscope with polarizers
- Find good hand tools that are comfortable to use
- Use durable, low-background mounts
- Use a minimum amount of adhesive
- Select a single crystal, sized to beam profile
- Place crystal on mount, well away from support

Conclusion

A little attention to crystal selection can save time on the other side of the experiment.

Questions and Answers

Any questions?

Please type any questions you may have for our speakers in the Q&A panel and click Send.

How did we do?

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Thank you!

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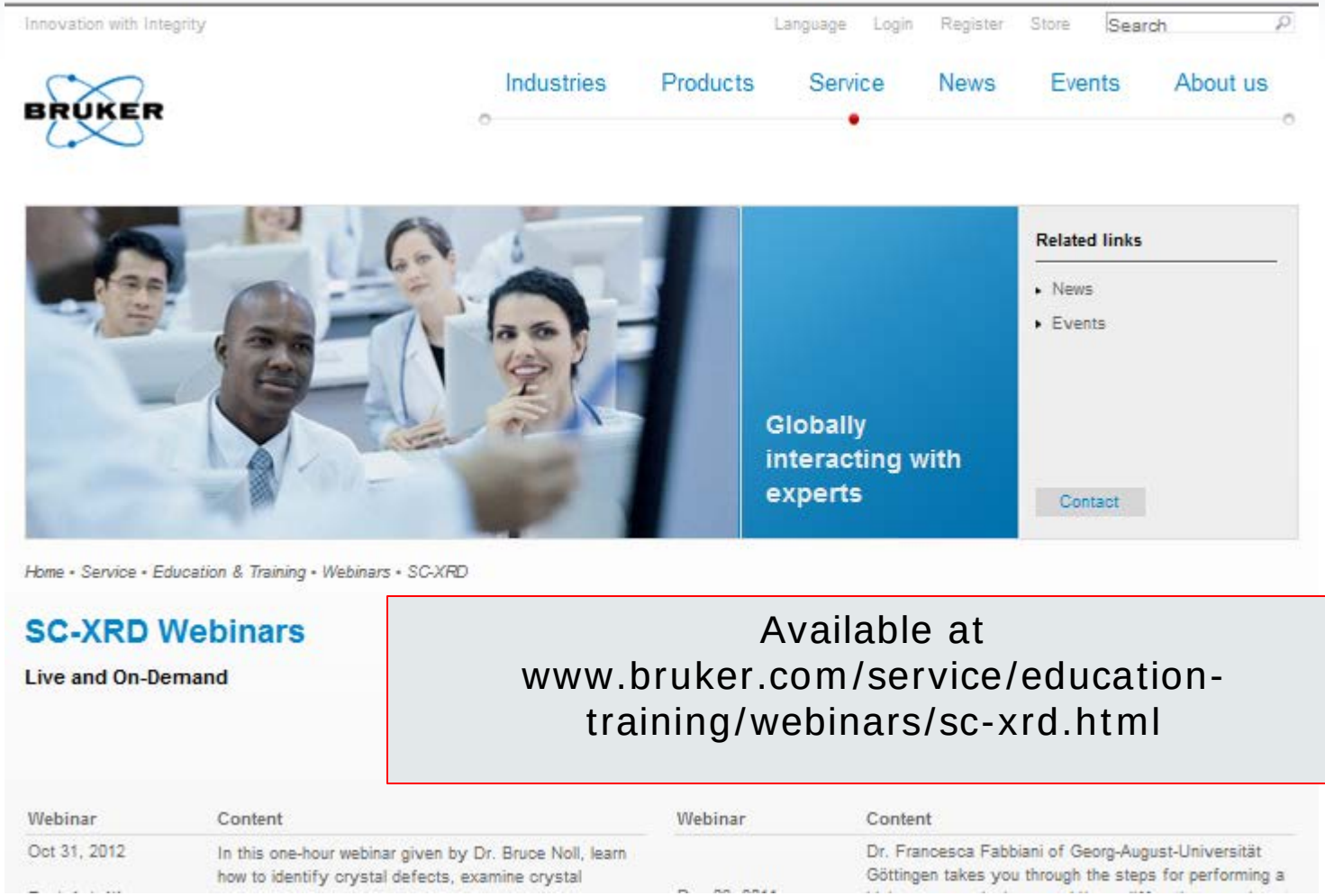

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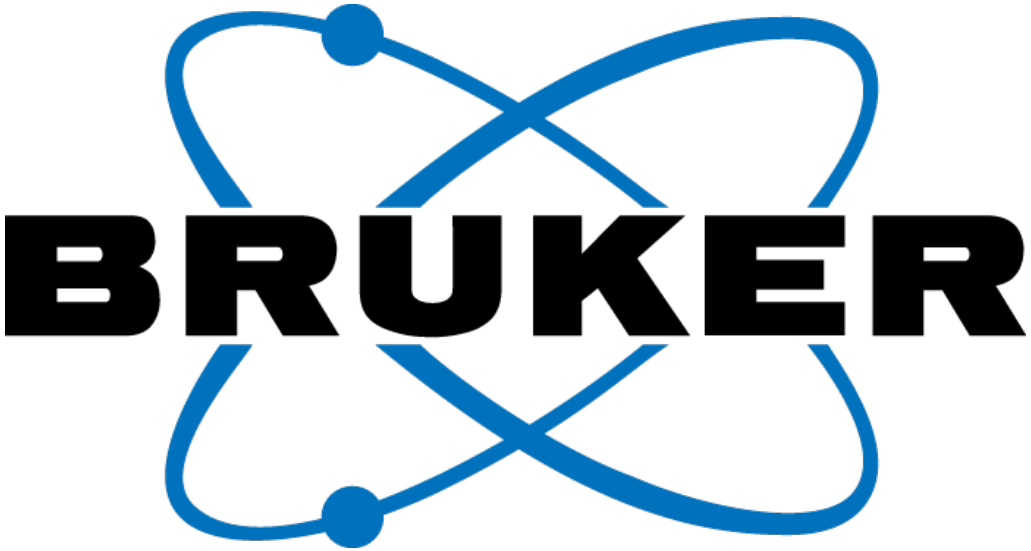

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