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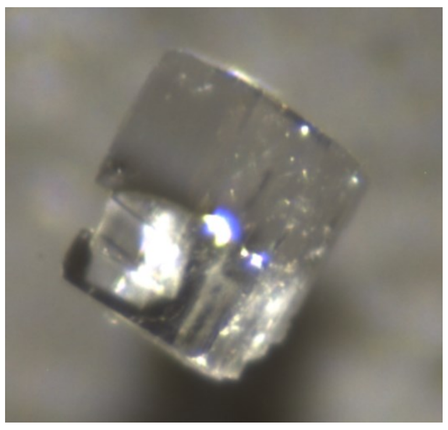
Bernal Institute

# X-ray Diffraction (XRD)
# Bruker D8

## XRD Single Crystal X-ray Diffraction

Single-crystal X-ray Diffraction, SXRD, is a non-destructive analytical technique which provides detailed information about the internal lattice of crystalline substances, including unit cell dimensions, bond-lengths, bond-angles, as well as site-ordering.

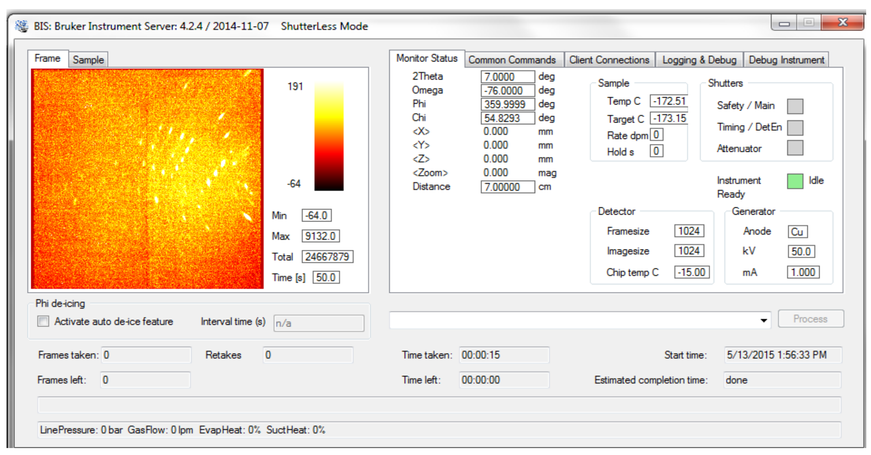
The data generated from the X-ray analysis is interpreted and refined to obtain the crystal structure by single-crystal refinement. X-Rays are either transmitted through the crystal, reflected off the surface, or diffracted by the crystal lattice. Diffracted rays at the correct orientation for the configuration are then collected by the detector. Preferred size of crystals is between 100 nm and 200 nm.



**A Lithium-OX single crystal before mounting on XRD**

## Bruker Single Crystal Diffractometer

- Designed primarily to determine the crystal structure of single crystals. It can also be used for determining crystal orientation
- The diffractometer uses a two-dimensional CMOS detector for fast, high precision transmission diffraction through small single crystals.
- A cryostat is available to control the temperature of the sample between 100 and 400K under a nitrogen stream, which permits more structure determination under varied conditions even for air-sensitive crystals (Oxford Cryostream Cryosystem).




Intensity data was collected on a small crystal of Vitamin C (40 µm x 100 µm x 100 µm) using a D8 Quest with Cu radiation.

## Structural Biology

Crystallography is the most unambiguous method for characterisation of macromolecules, and SXRD provides the information required to understand structure and function of proteins and enzymes.


**Bernal Institute possesses two Bruker D 8 Quest fixed Chi single crystal diffractometers**

1. **Incoatec microfocus CuK α source** (λ= 1.54178 Å) and Photon II detector. Due to the diameter of the incident beam (0.3 mm) the longest dimension of the crystal should be smaller than 0.3 mm. This setup is preferred for small crystals and crystals of compounds containing mostly light atoms (i.e. poorly diffracting organic compounds)
2. **MoK α source** λ= 0.71073 Å and Photon II detector The incident beam diameter (0.5 mm) allows measurement of crystals with the longest dimension of up to 0.5 mm. It is suitable for the study of crystals of compounds containing heavy metal atoms/ions or other strongly absorbing elements, and also to collect data of higher resolution

Samples for single-crystal diffraction should be selected from un-fractured, optically clear crystals. This can be determined by viewing the samples under cross polarized light on a microscope. Crystals can be broken off a larger sample and the best fragment selected. Samples should be between 30 and 300 microns




**Samples are mounted on the tip of a thin glass or polymer fibre attached to a brass mounting pin, and the pin is then inserted into the goniometer head**.

The Bruker Quest D8 has a 3-circle FIXED- X sample stage with open geometry has minimal obstruction and allows easy mounting of additional crystal-conditioning devices. It supports a 360° ϕ drive at the magic X angle of 54.7° and is efficient for data collection, using precise omega scans and “Easy-to-use” geometry.
Apex software package allows structure determination.

Specific applications of single-crystal diffraction include:

- New mineral identification, crystal solution and refinement
- Determination of unit cell, bond-lengths, bond-angles and site-ordering
- Characterization of cation-anion coordination
- Variations in crystal lattice as function of chemical and physical environment

**Contact**
Bernal Institute
E: bernal.institute@ul.ie

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