UiO : Department of Chemistry
University of Oslo

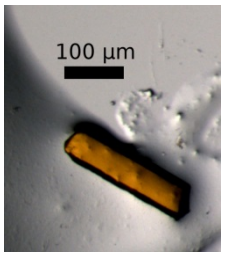
Single crystal XRD facilities

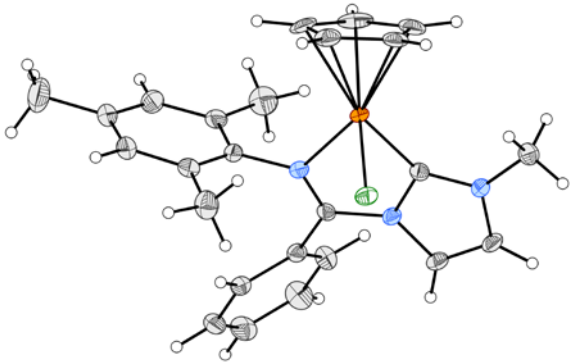
D8 Venture and APEXII at UiO

Sigurd Øien

Overview

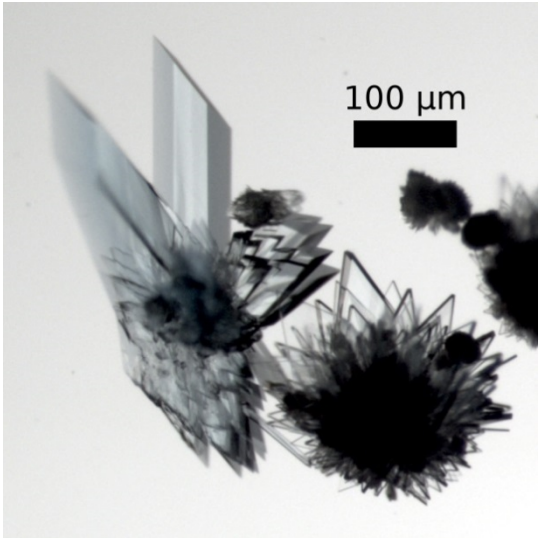
- Has the ability to locate every atom in a crystallized compound
- Widely used in small molecule and biomolecule characterization, pharmacy.

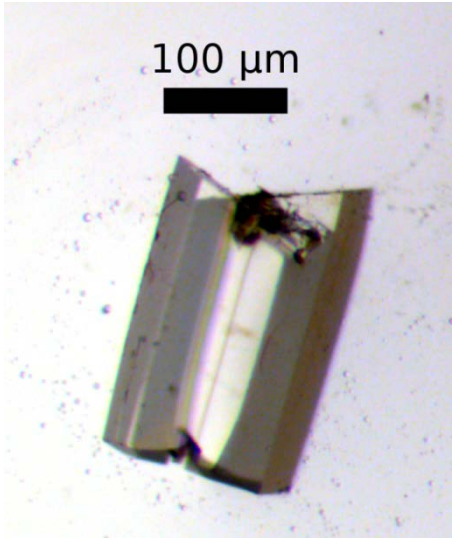


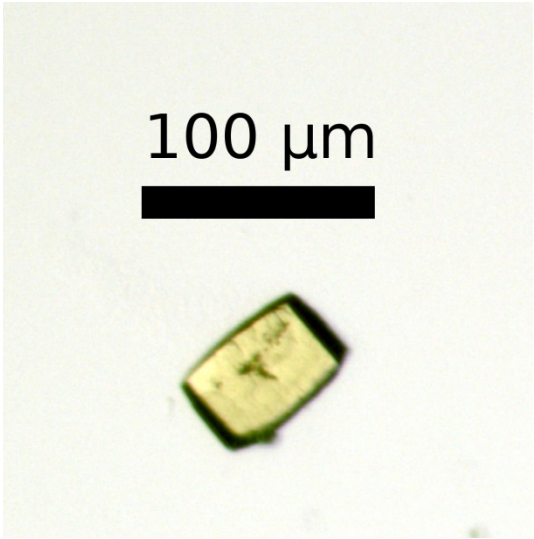


Procedure - crystallization

- Sample has to be a single crystal
- Cofactors, solvates, counterions etc.
- Beam radius at sample ~50 µm







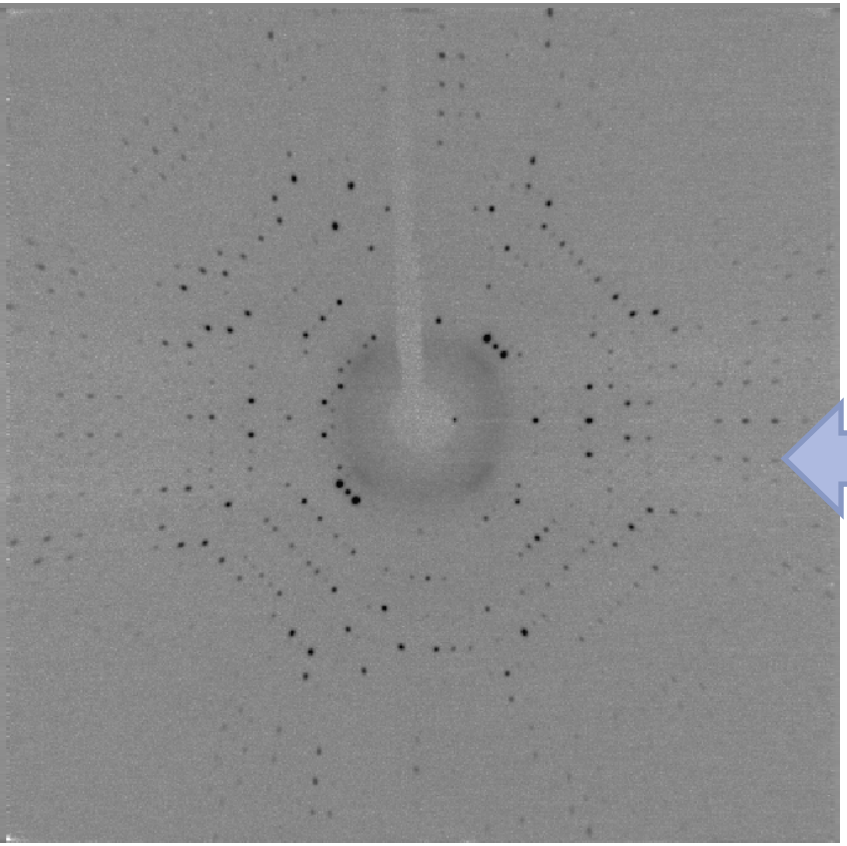
Our instruments

- Bruker Apex II
- Mo Kα source
- 512x512 area detector
- Cryosystem 80 – 400 K

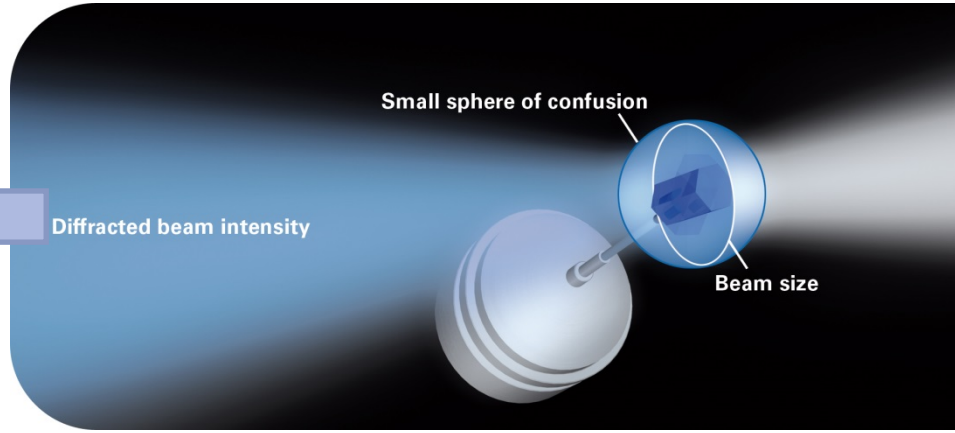
Our instruments

- Bruker D8 Venture
- Dual Source
  - Mo and Cu Kα
- 1024x1024 area detector
- Cryosystem 80 – 400 K

Procedure – data acquisition

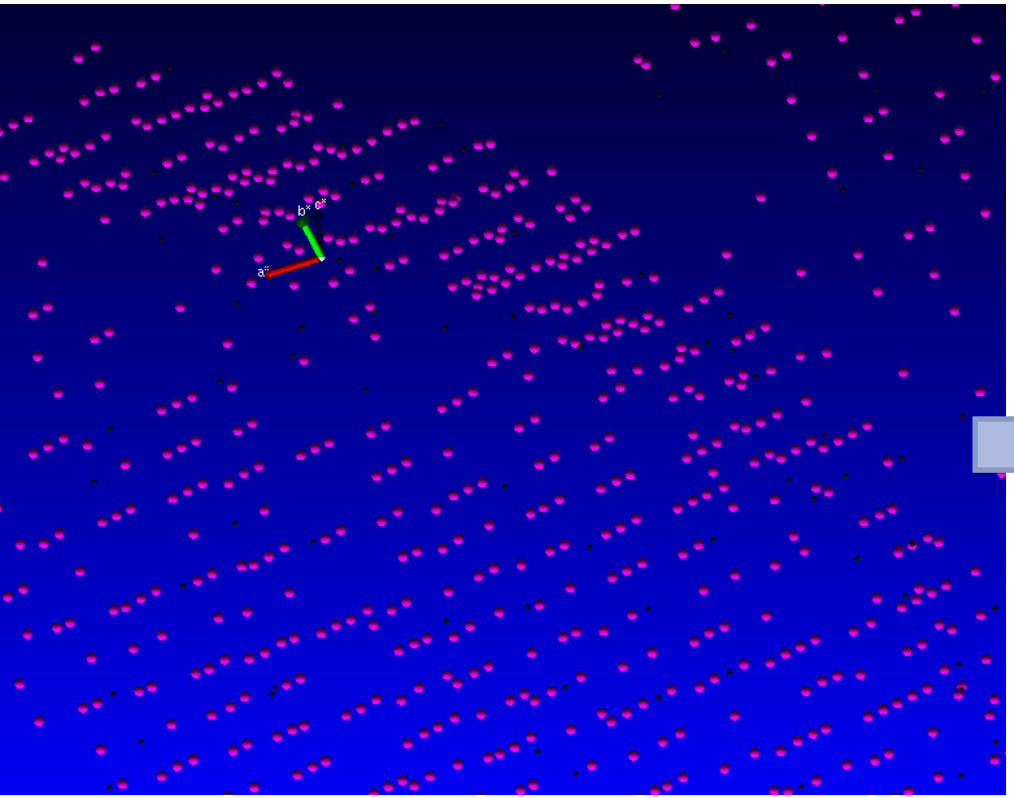


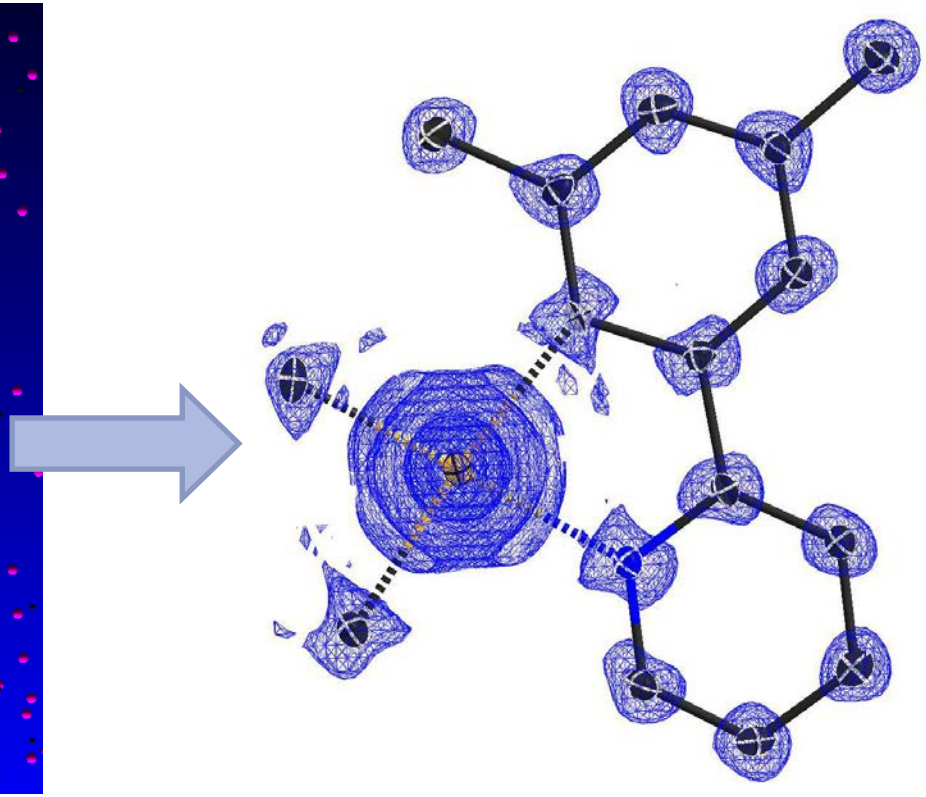
Depending on crystal quality,
20 minutes to 3+ days



Ordered electron density creates diffraction pattern,
as photons are interacting with electrons

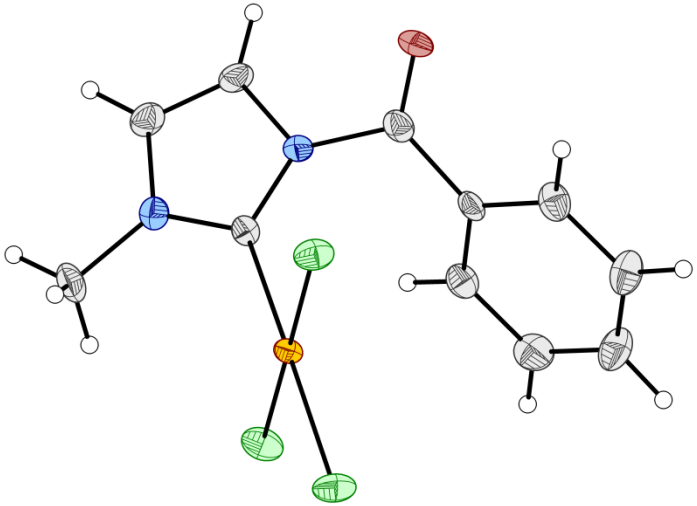
Structure solution

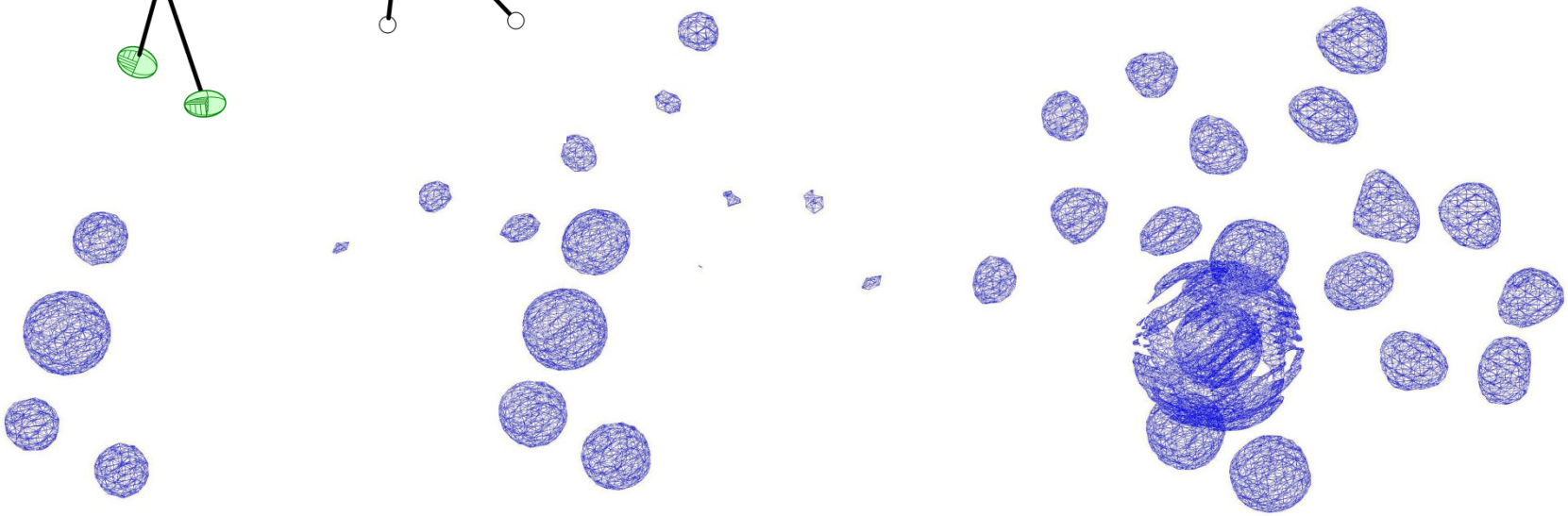




Reflection positions and intensities are recorded,
then Fourier transformed into an electron density map.

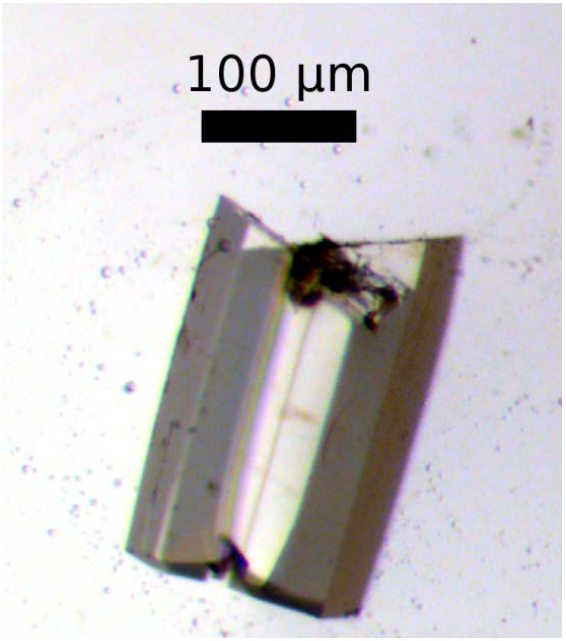
Model and electron density map



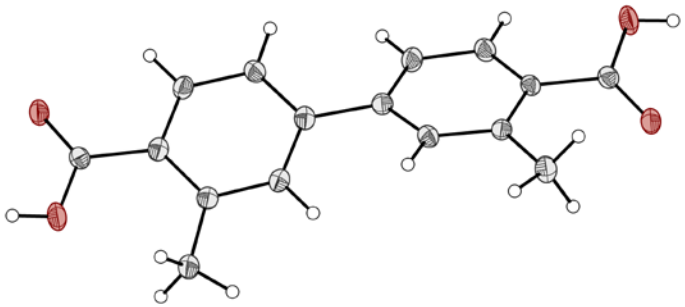


>10 e^{-}Å^{-1} >5 e^{-}Å^{-1} >3 e^{-}Å^{-1}

Twin refinement

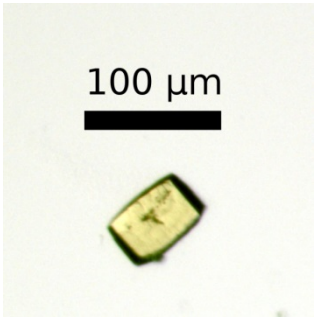


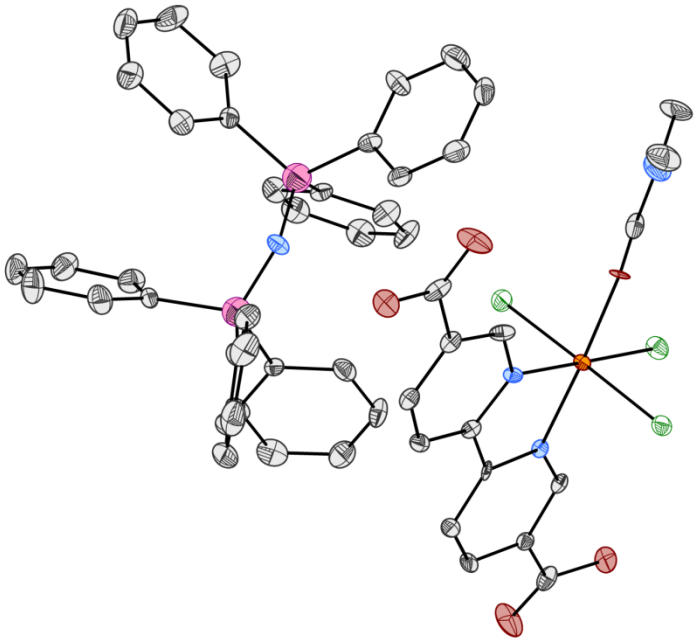
- Reflections are divided into two (or more) groups.
- Difficult, but sometimes unavoidable



Small crystals

- Diffraction strength proportional to crystal volume
- Diffraction weaker at high angles





Summary

- SC-XRD gives the complete composition and structure of your crystal.
- Better crystal, more accurate information