

**RESEARCH
WEEK2020**

The application of single crystal X-ray diffraction — beyond simple structure characterization

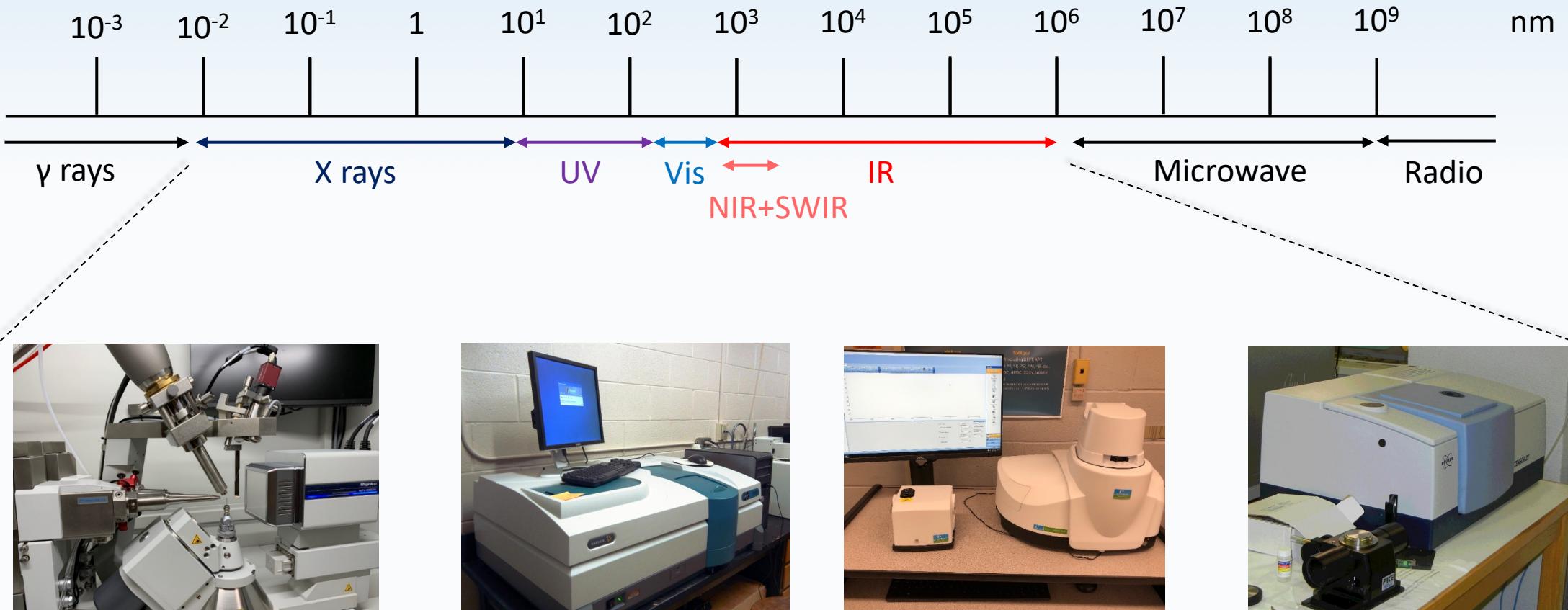
X-ray Crystallography and Materials Characterization

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Electromagnetic Spectrum



X-Rays
(0.071 & 0.154 nm)

UV-Vis-NIR
(175 to 1800 nm)

FT-NIR
(1000 to 2500 nm)

FT-IR
(2500 to 25000 nm)

Facility

Materials Characterization



TA Instruments Q500 TGA
Thermogravimetric analysis

- Temperature range: ambient to 1000°C
- Gases: Air or Nitrogen



Mettler-Toledo DSC1
Differential Scanning Calorimeter

- Temperature range: -90°C to 600°C
- Gases: Air or Nitrogen

Facility

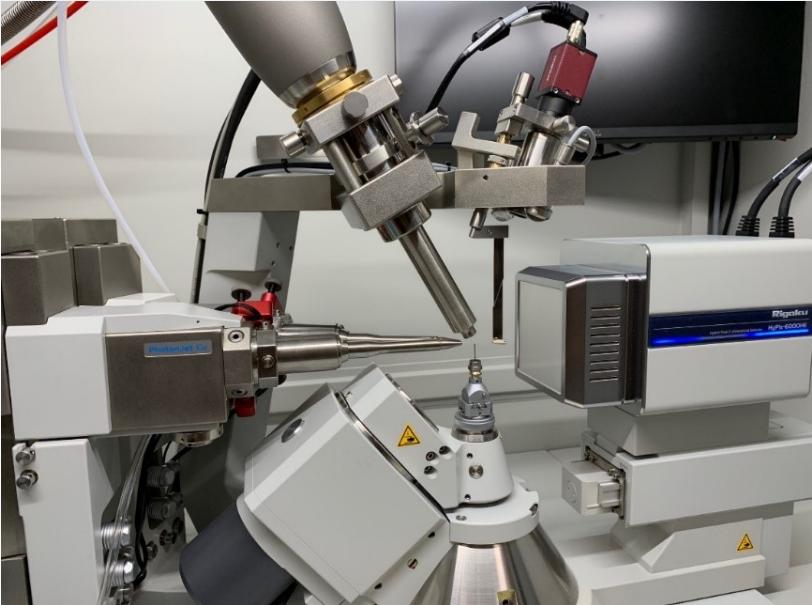
X-ray Crystallography



Rigaku XtaLAB Synergy-S X-ray diffractometer

- 4-circle Kappa goniometer
- Dual PhotonJet sources (Cu and Mo)
- HyPix-6000HE Hybrid Photon Counting (HPC) detector
- Oxford Cryosystems 800 Series Cryostream.

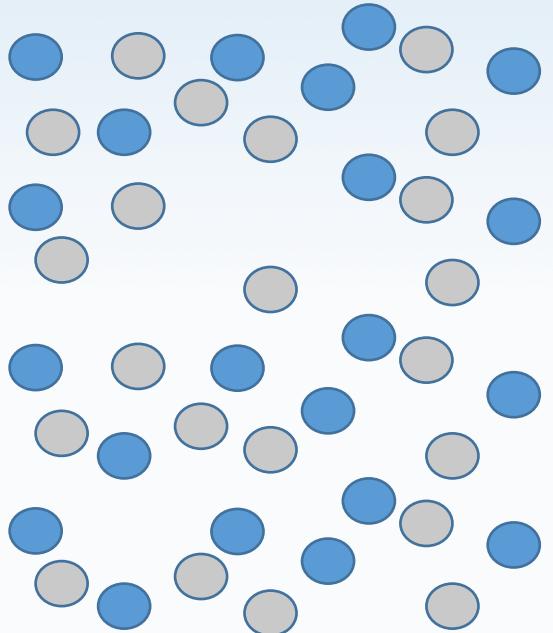
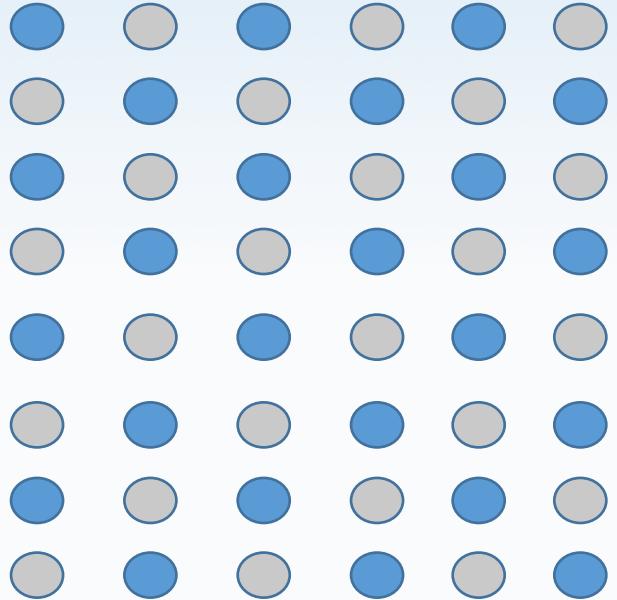
X-ray Crystallography



Rigaku XtaLAB Synergy-S X-ray diffractometer

- Collection temperature from 80 to 400 K.
- **Single crystal** diffraction (crystal size as small as 20 μm)
- Transmission-mode (in capillaries or Kapton loops) **powder** diffraction

Crystalline or Amorphous



Single-crystal vs. Polycrystals

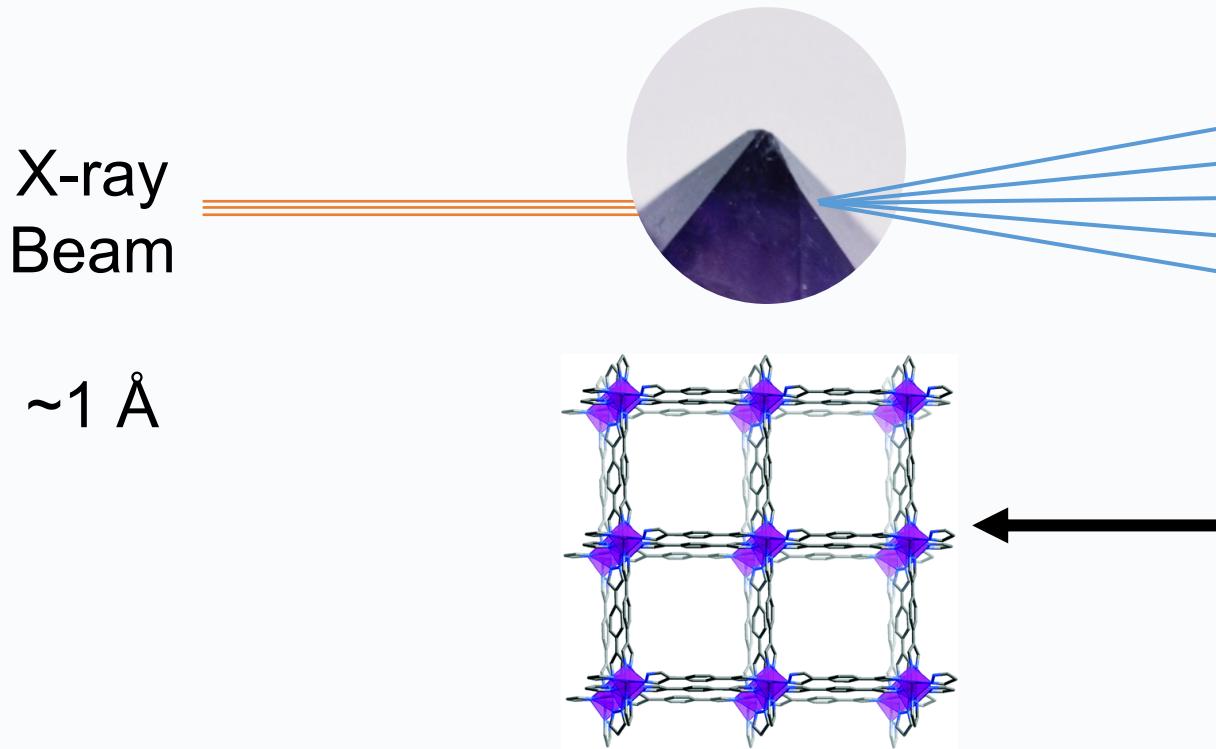


continuous and unbroken

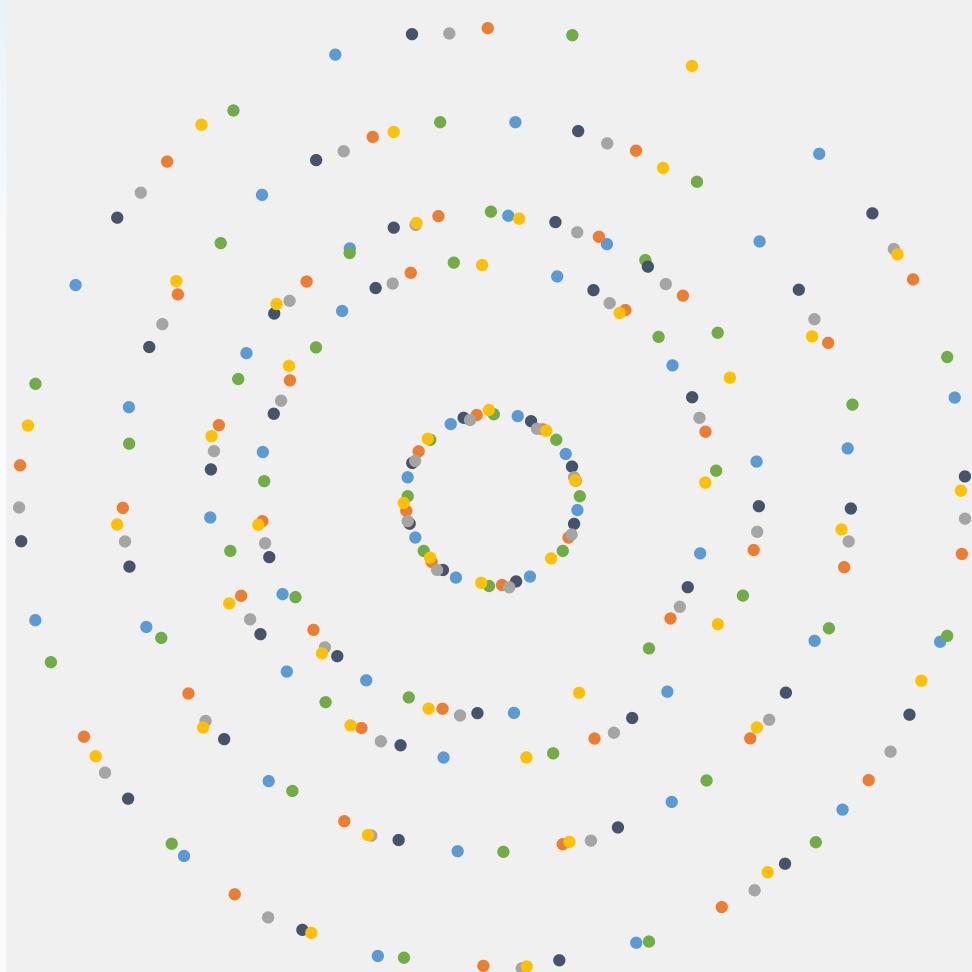


aggregate of crystals

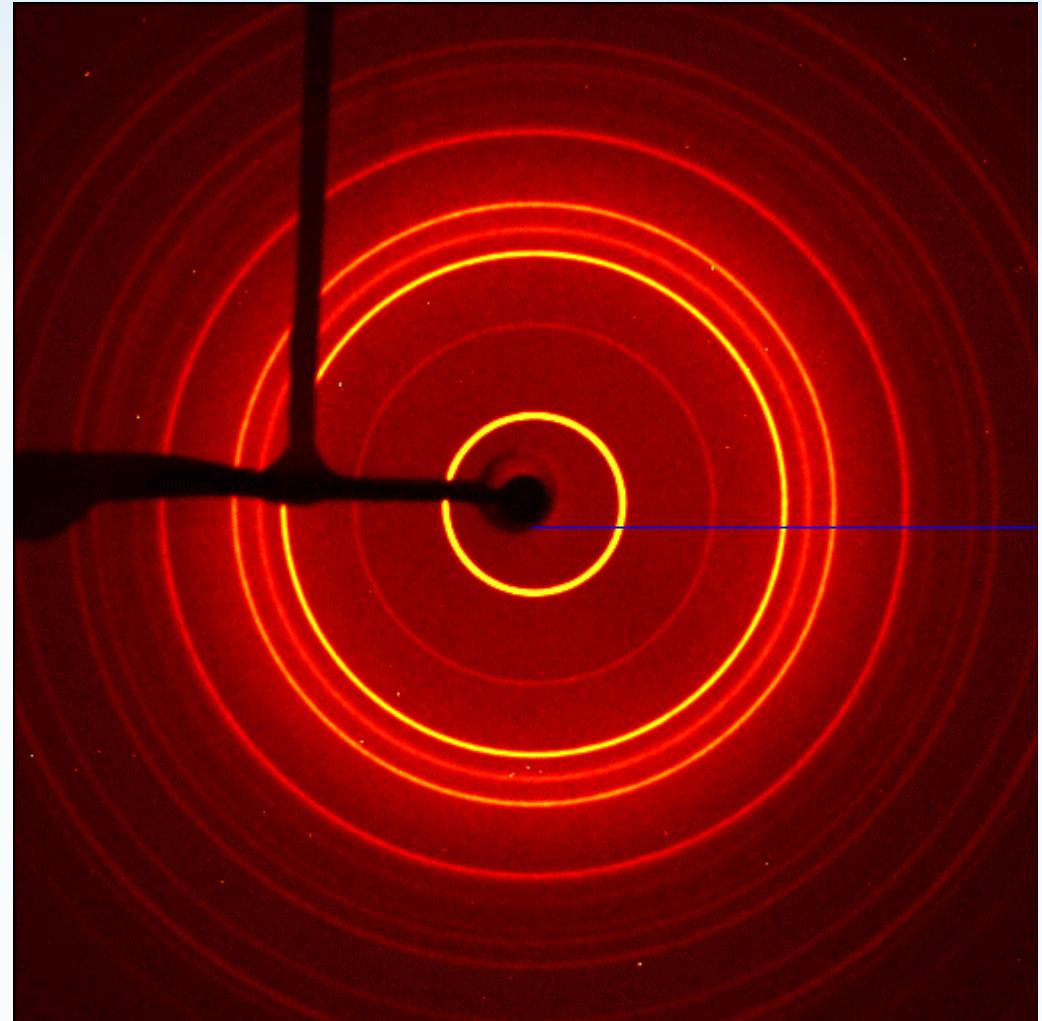
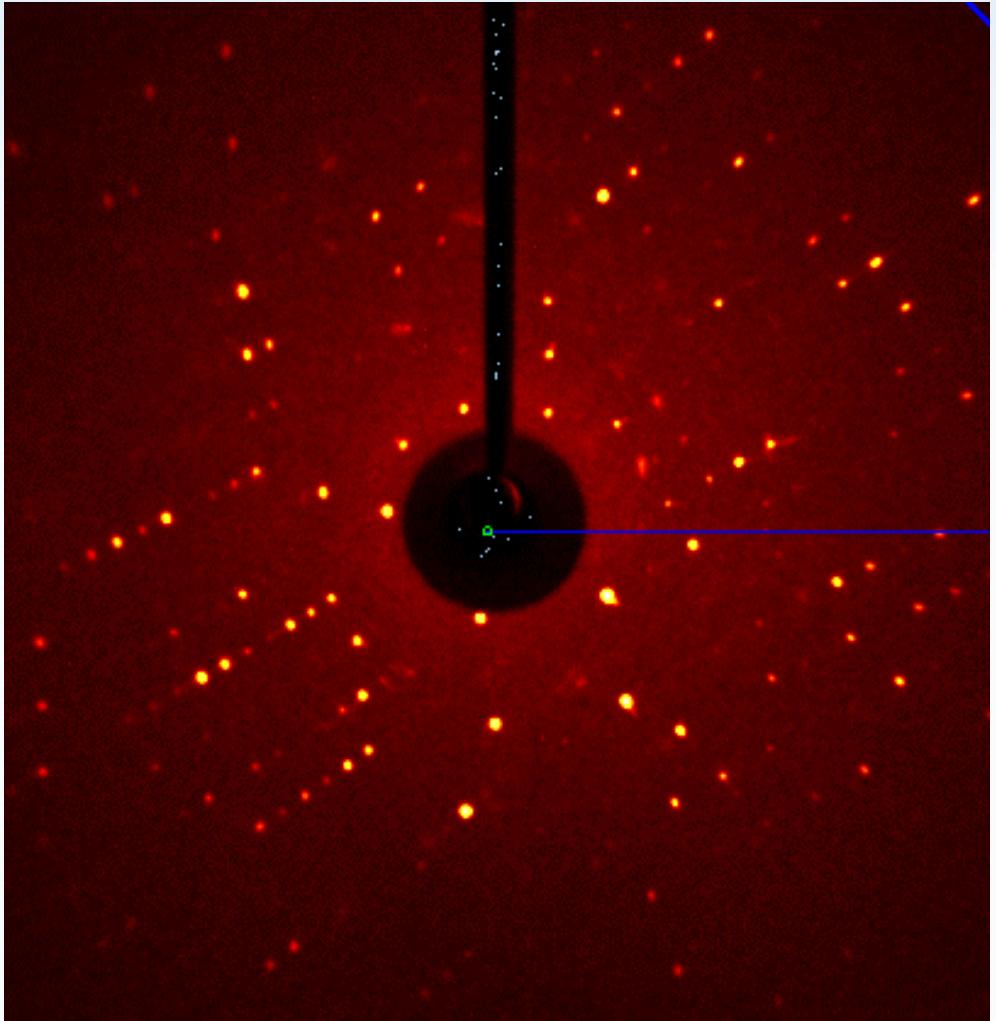
Single-crystal X-ray diffraction (XRD)



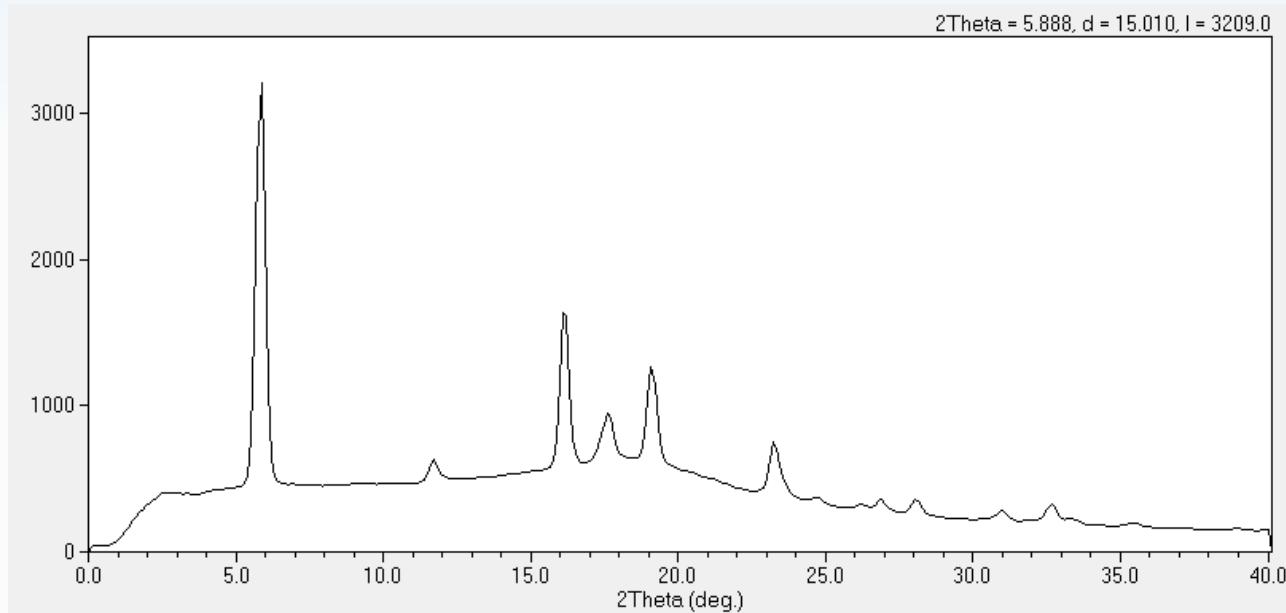
Single-crystal XRD vs. Polycrystals XRD



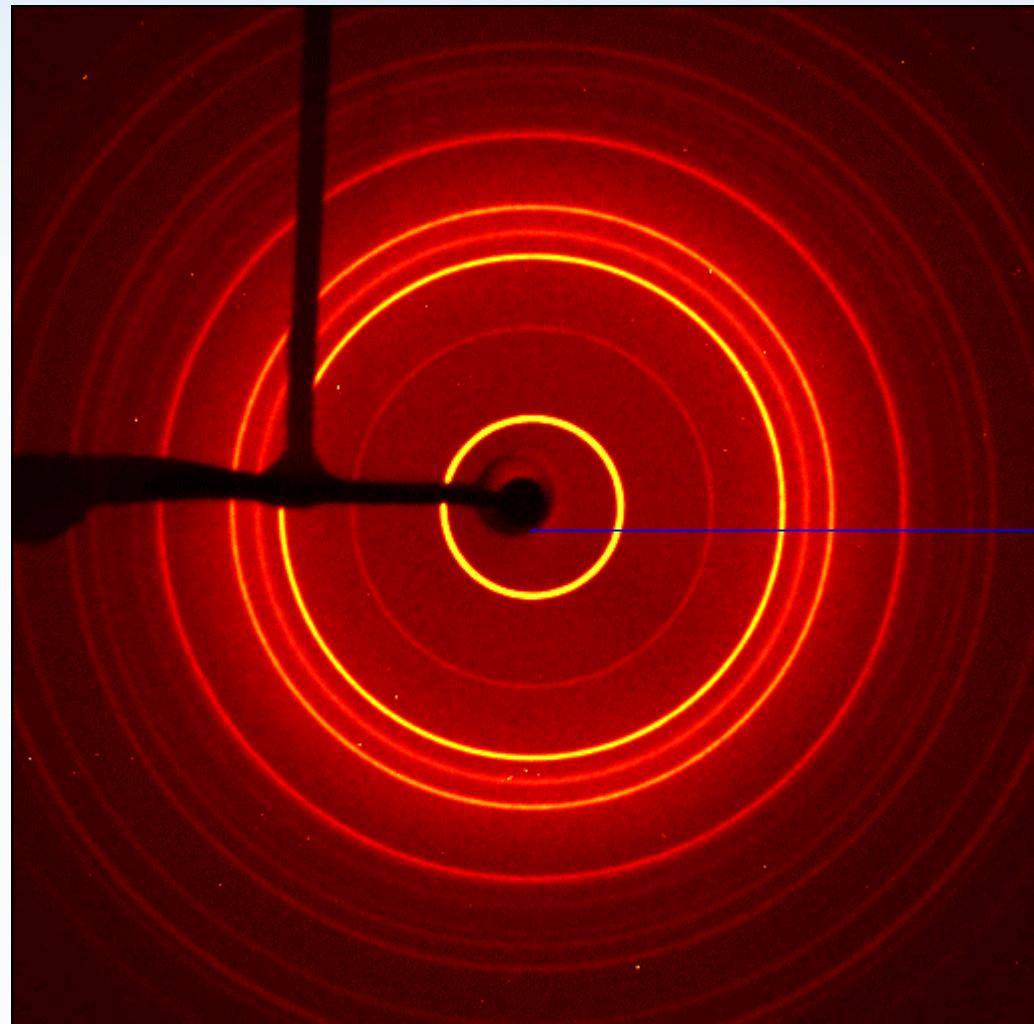
Single-crystal vs. polycrystals XRD



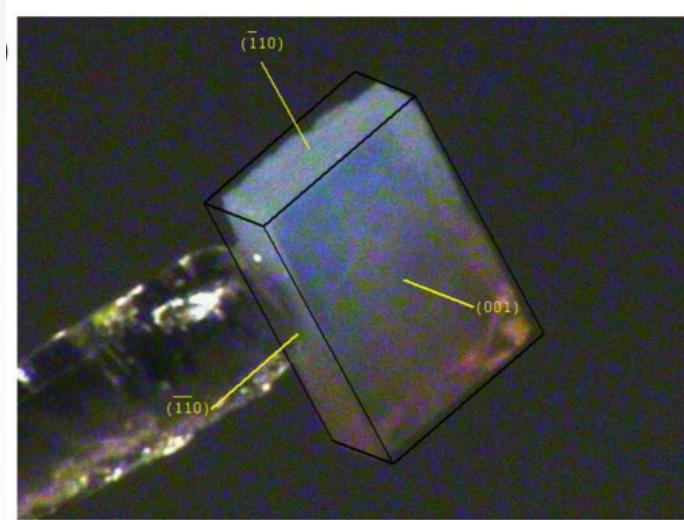
PXRD collected from single-crystal diffractometer



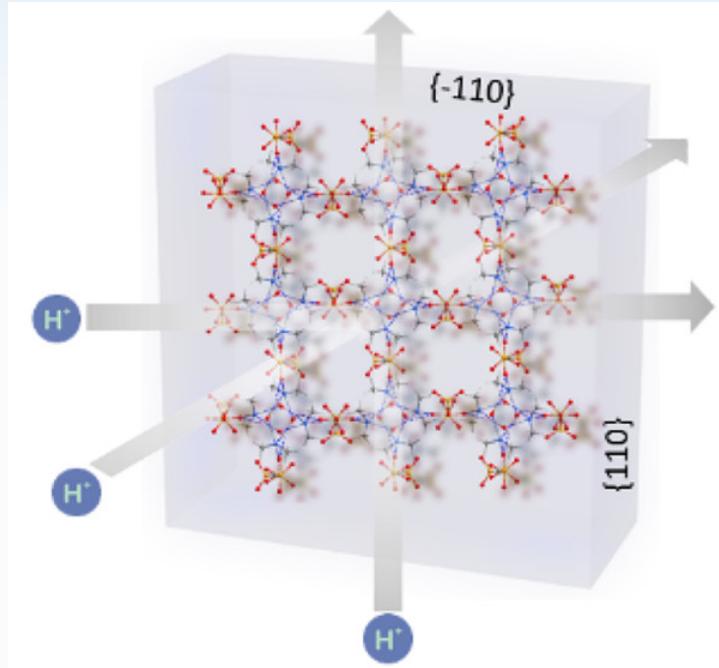
No preferred orientation
Micro-gram sample



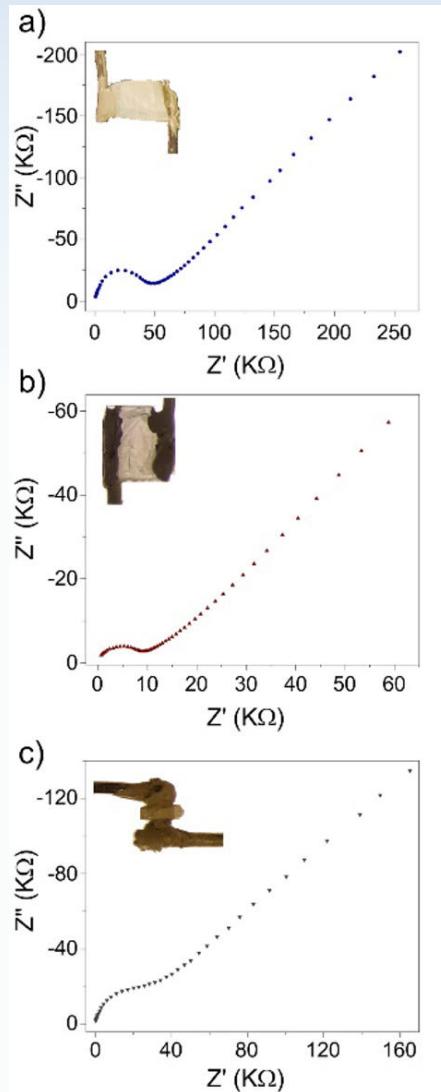
Single-crystal faces index



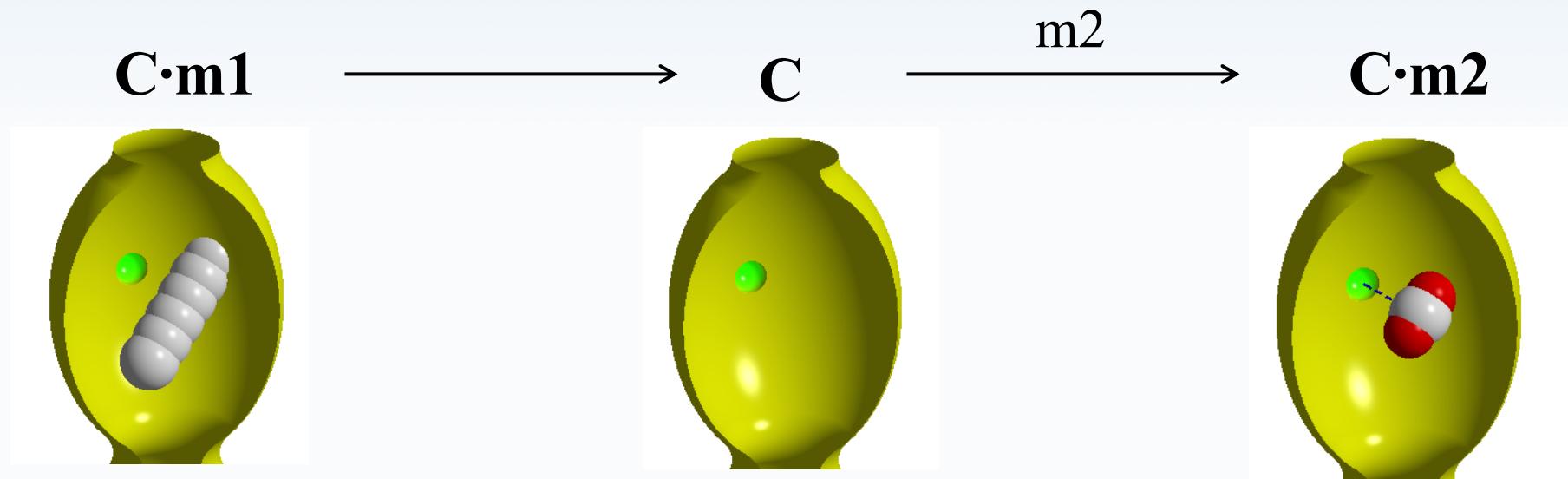
0.1-0.3 mm



**proton, electron, ions conduction
anisotropic magnetism**

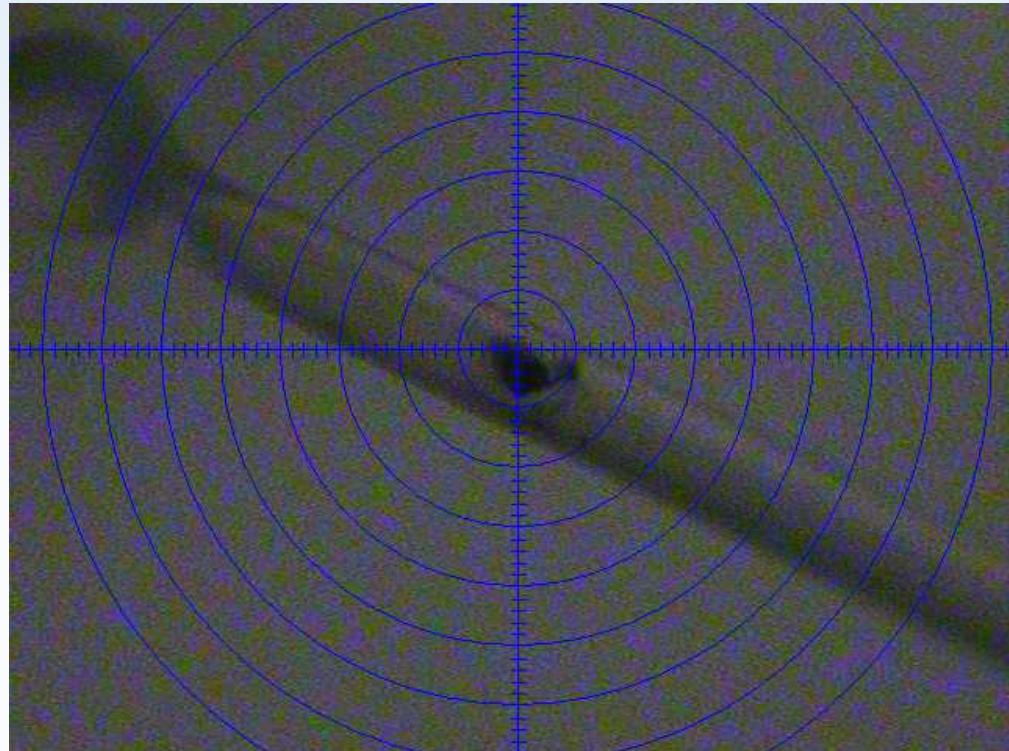


In-situ loading of molecule



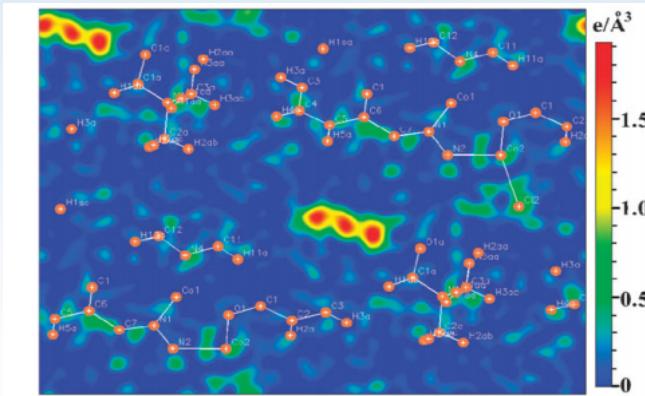
C = compound
 m = molecule

In-situ loading of CO₂ in a single crystal

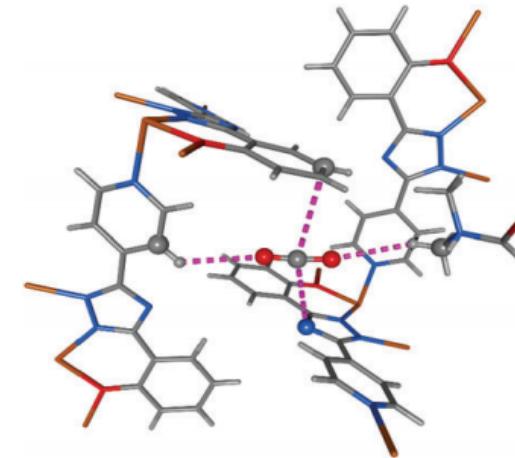


~ 0.1 mm

characterize a catalysis mechanism

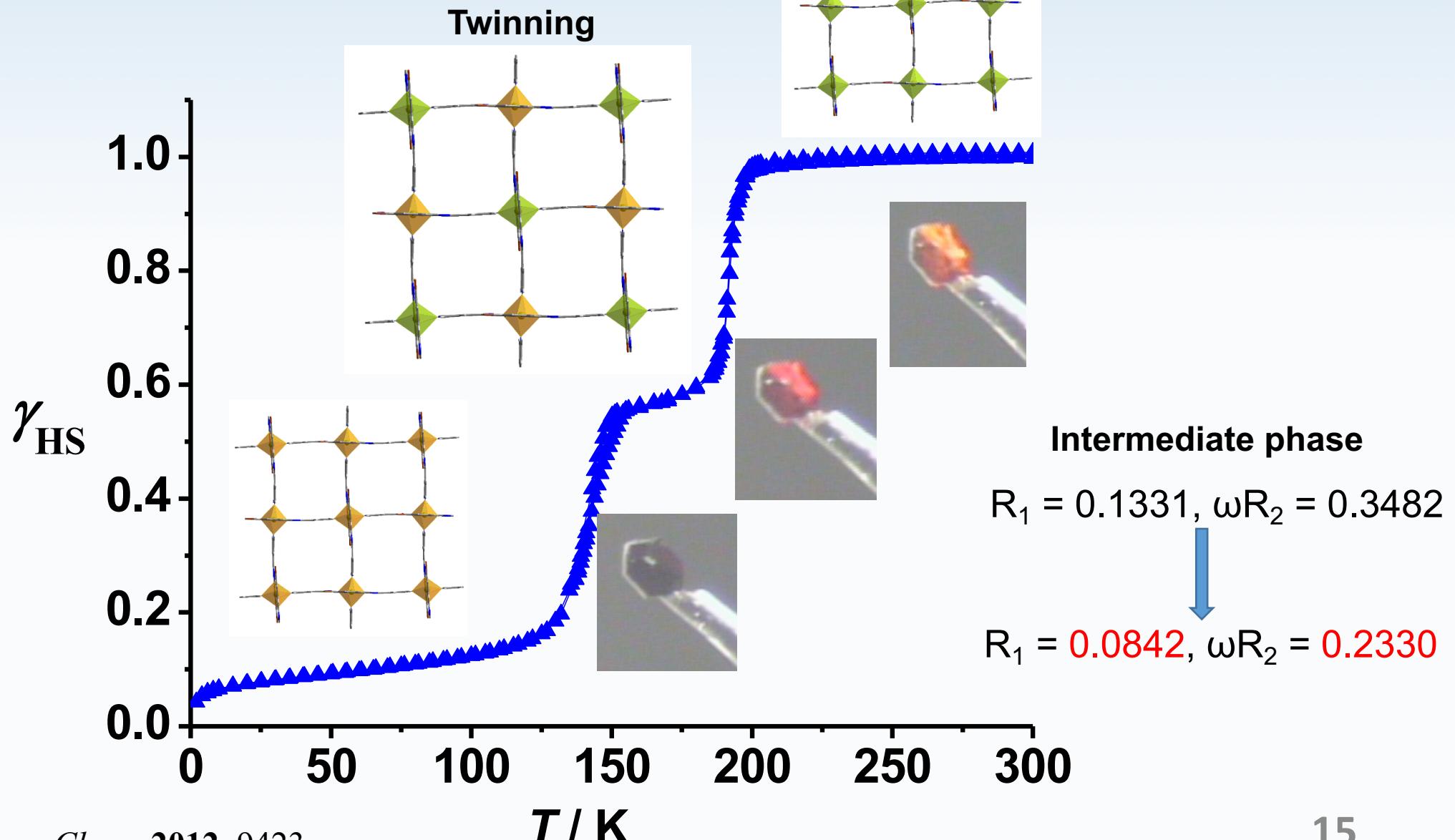


The difference electron density map



The environment of CO₂

Intermediate phase characterization



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Questions?

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