NSF's ChemMatCARS: A Synchrotron X-ray National Facility for Chemistry and Materials Research

NSF's ChemMatCARS
UCHICAGO

Science Highlights in Advanced Crystallography

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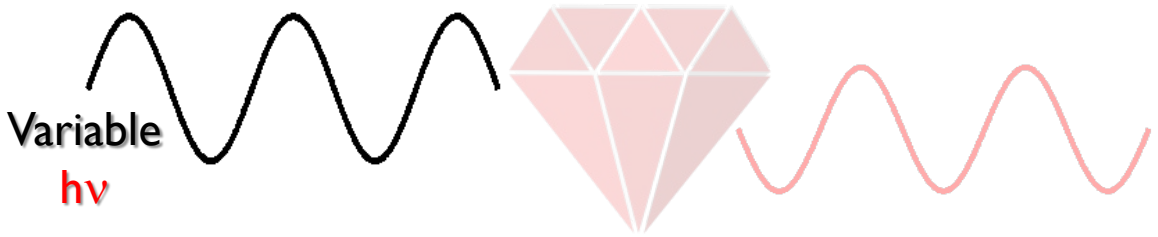
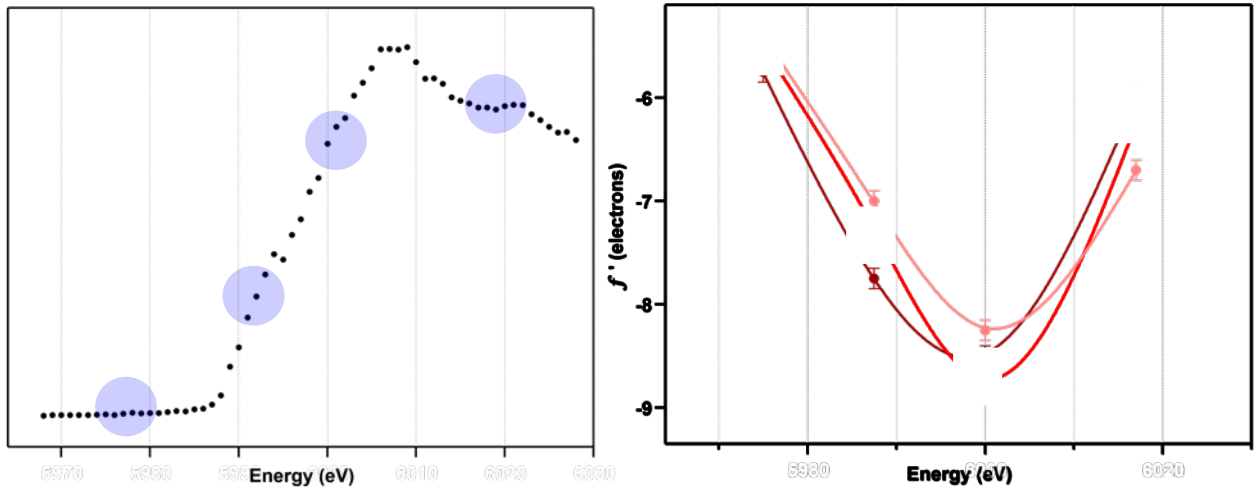
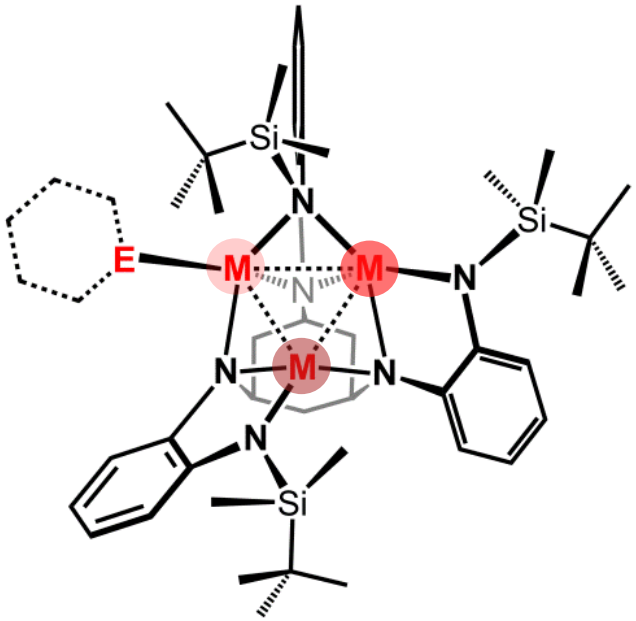
Resonant Diffraction Anomalous Fine Structure (DAFS)

Elemental ***identification*** at given crystallographic sites
Only at ***synchrotron*** light sources

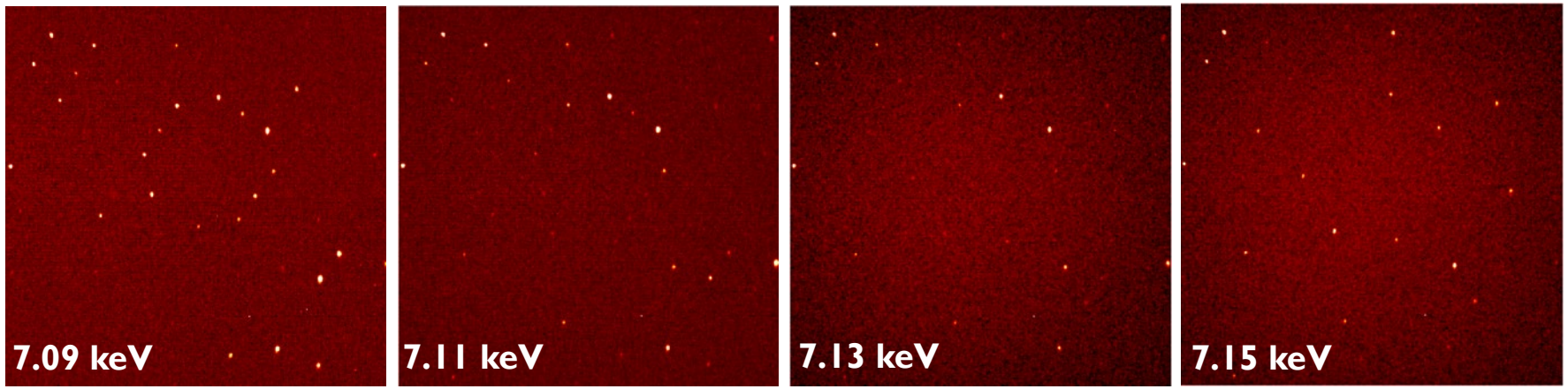






$$f = f^0 + f' + if''$$

Permits *site-specific* element ID and oxidation state determination



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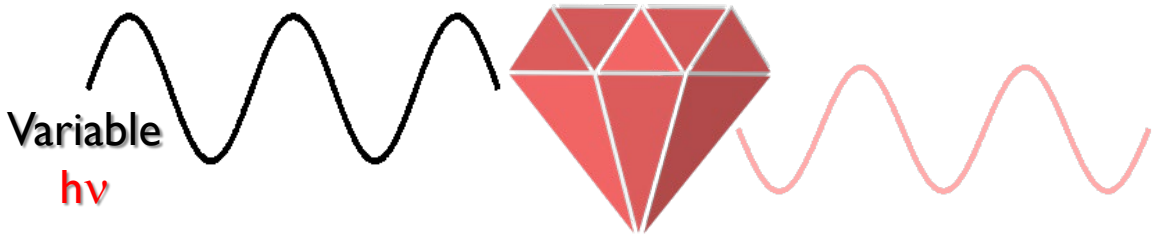

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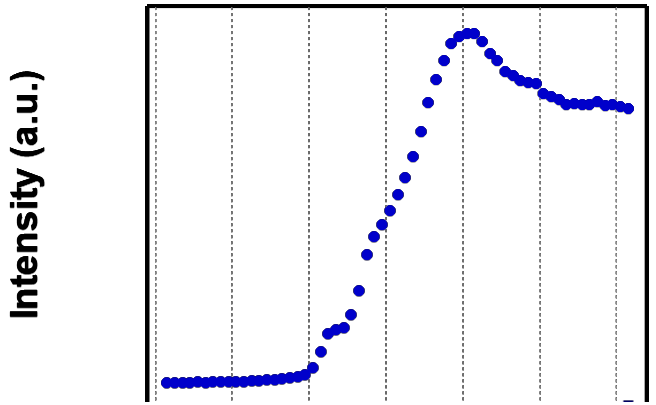
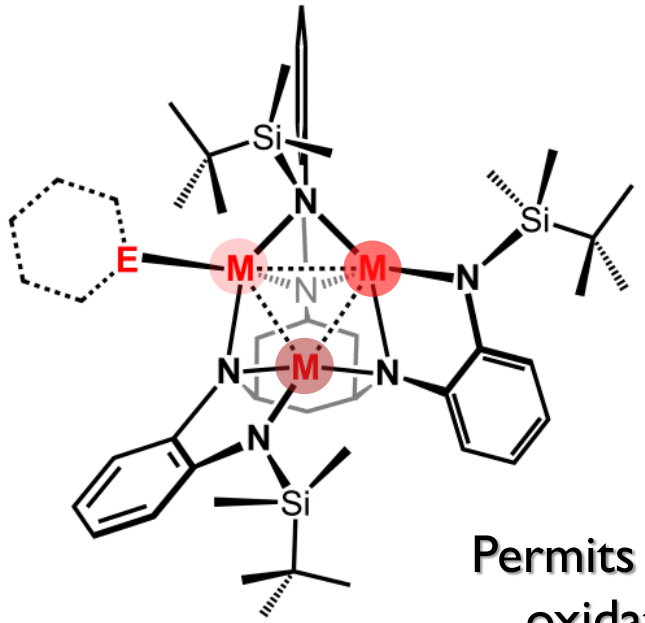
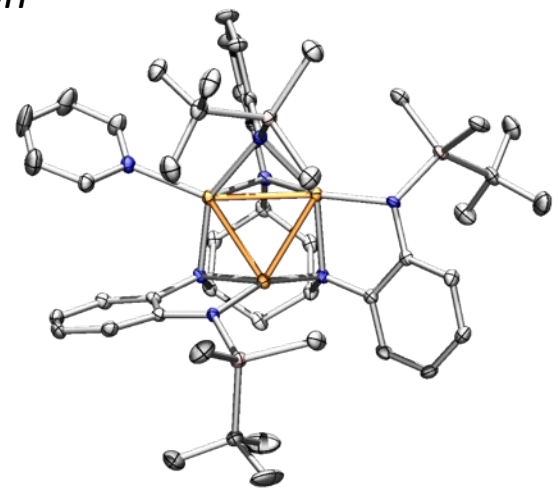


Fe K-edge: photoionization of a Fe(1s) electron





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Bartholomew, Teesdale, Hernandez Sanchez, Malbrecht, Juda…YS Chen, TAB *PNAS* **2019**, *116*, 15836

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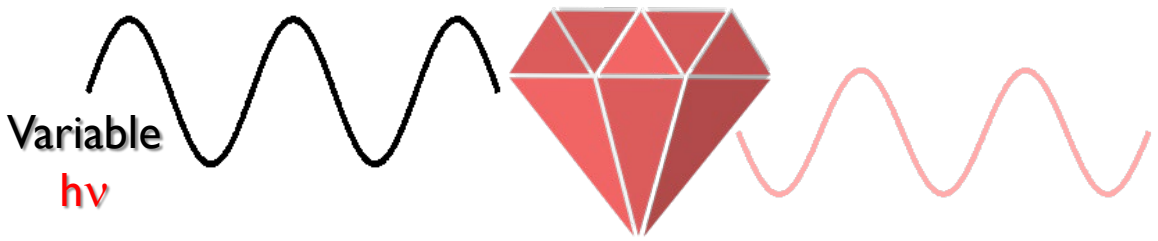

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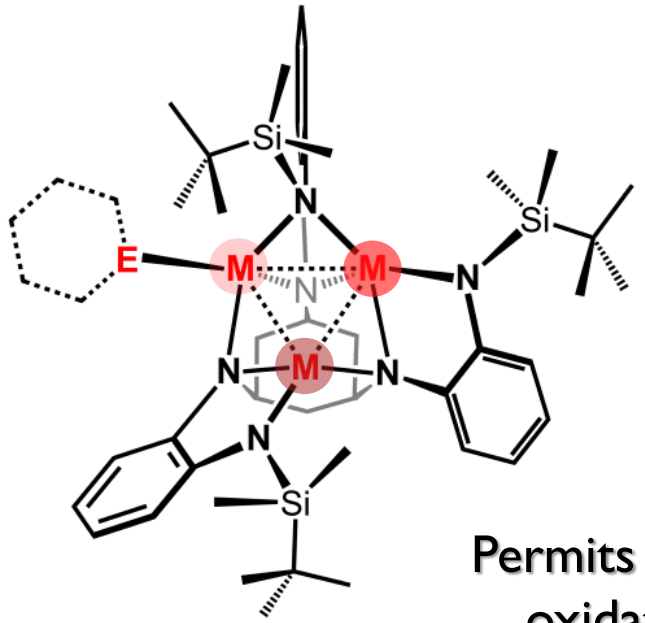

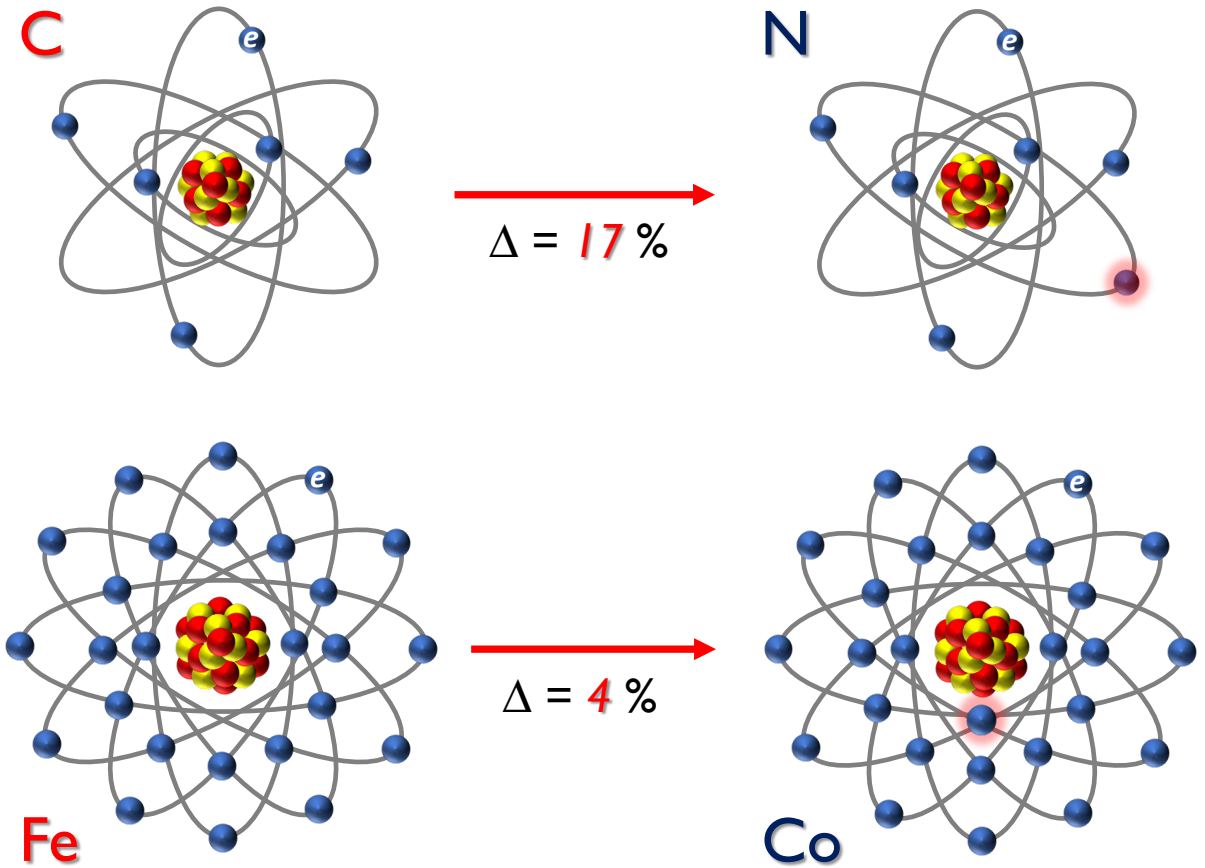

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Powers, et al. JACS ***2013****, 135, 12289*

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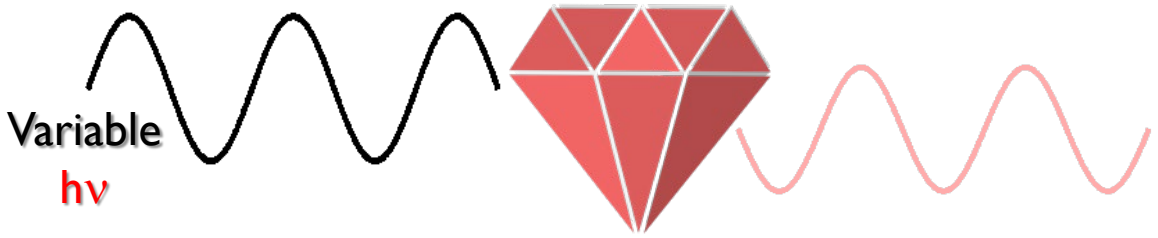

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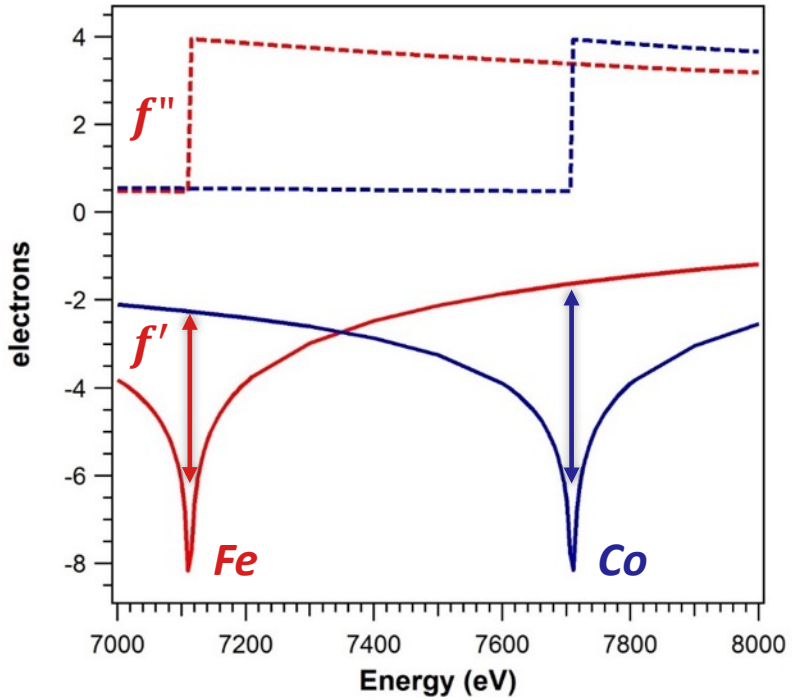

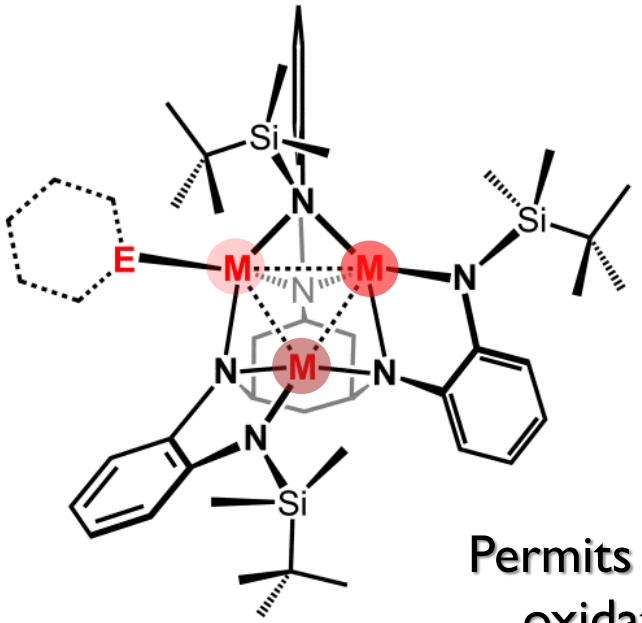

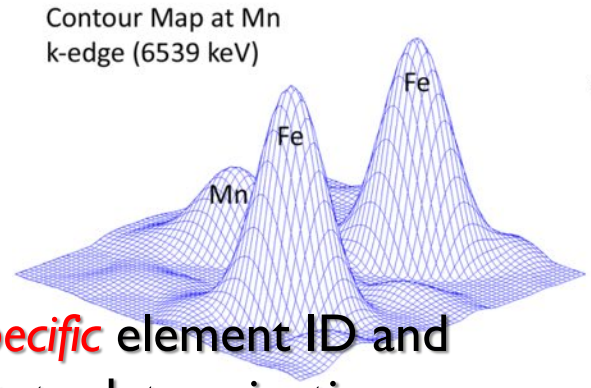

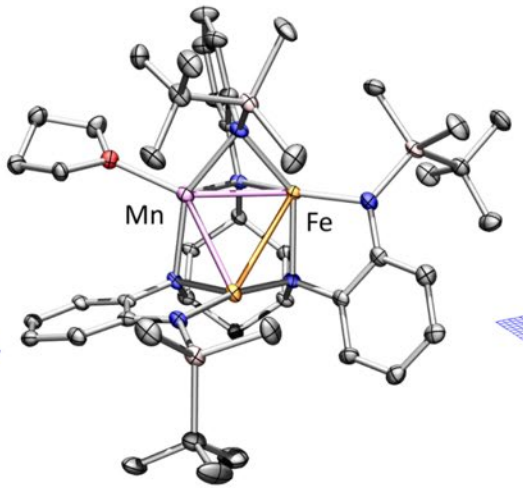

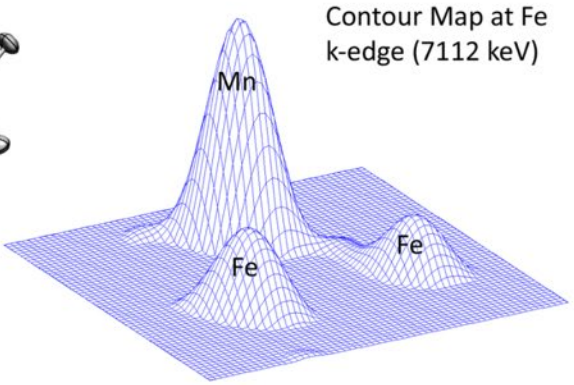

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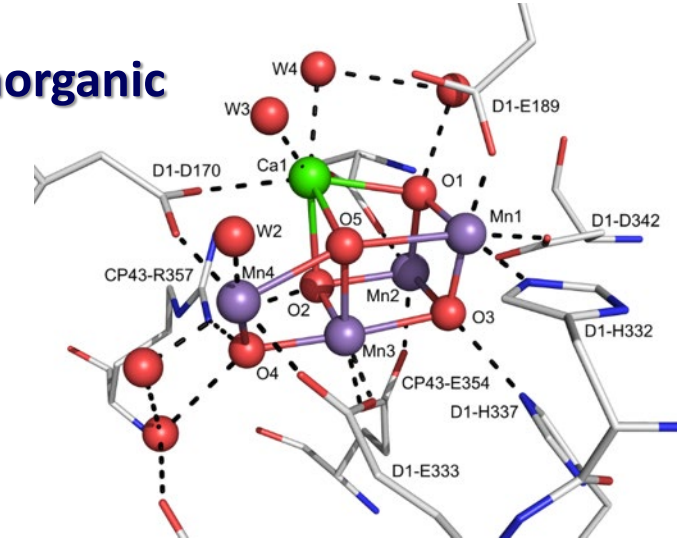
Resonant Diffraction/Diffraction Anomalous Fine Structure (DAFS)

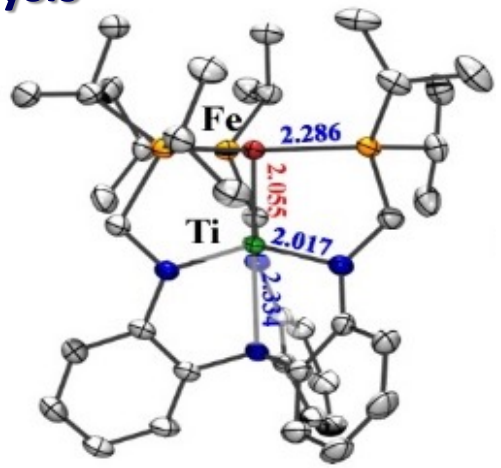
- **Identification of specific elements at given crystallographic sites**
- **Proposed the canted beamline would extend the energy range down to 3 keV, providing access to the entire series of 3d → 4d transition metals, including Sc, Ti, V, K, and Ca, which are not accessible on the existing beamline**

➢ *Only at synchrotron light sources*

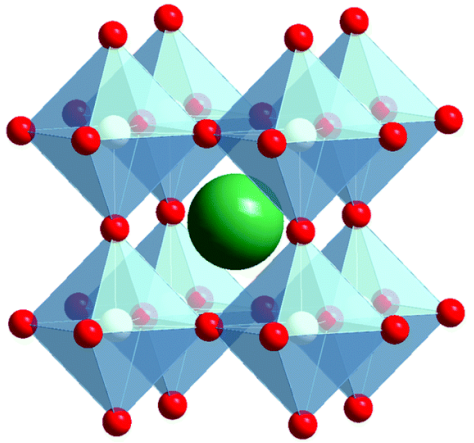
Bio-inorganic



Catalysis



Material Science



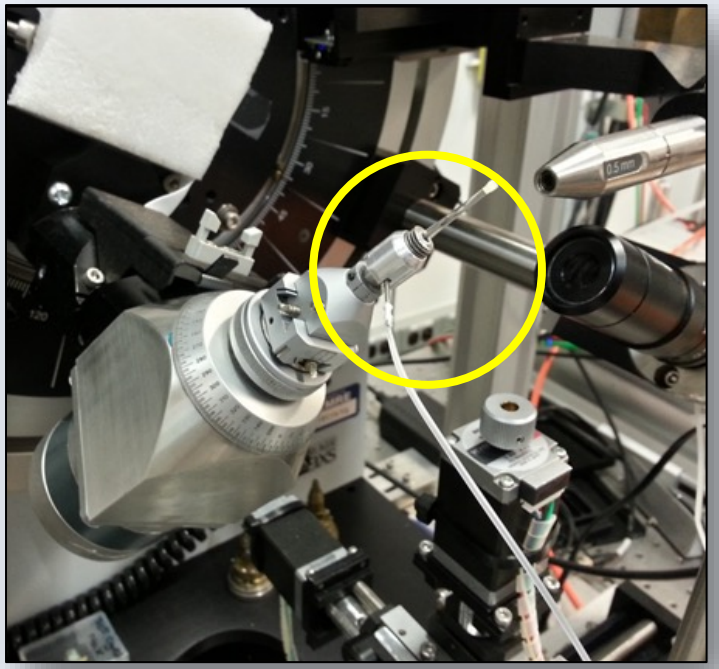
Structural Dynamics Today

In-situ crystal diffraction

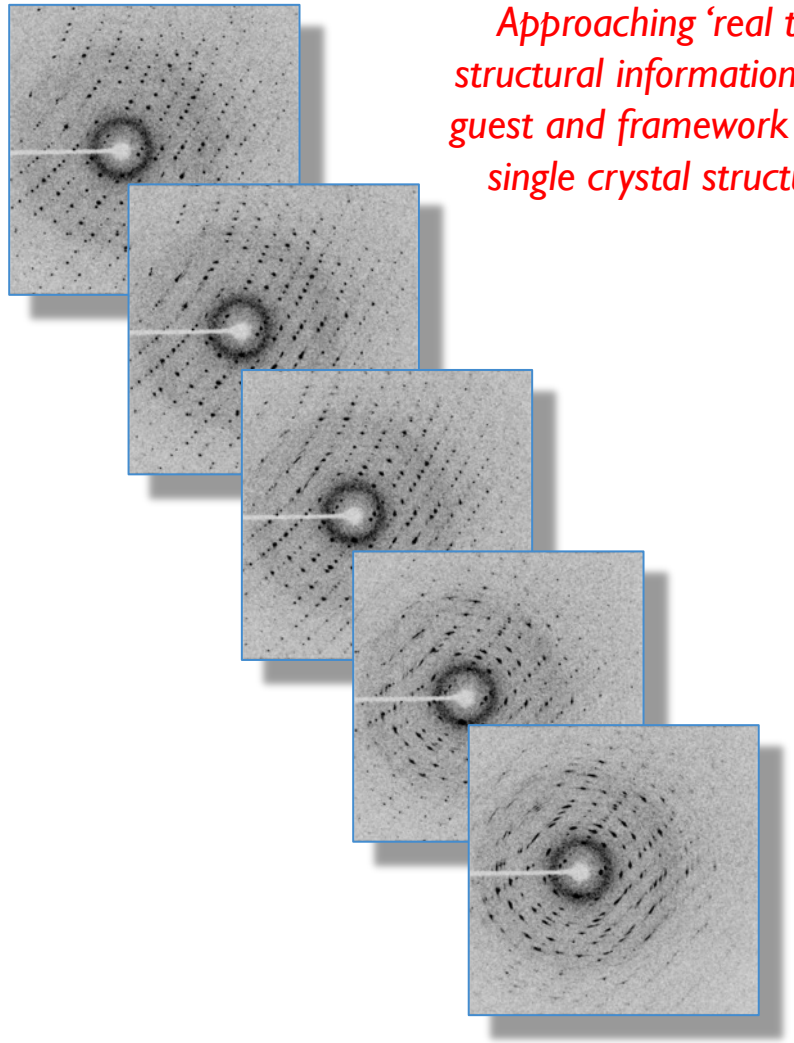
Environmental Control Cell (ECC)

- ***Vacuum, gas, solution and humidity***

Benedict Group and ChemMatCARS
J. Appl. Cryst.(2015), 48, 578-581

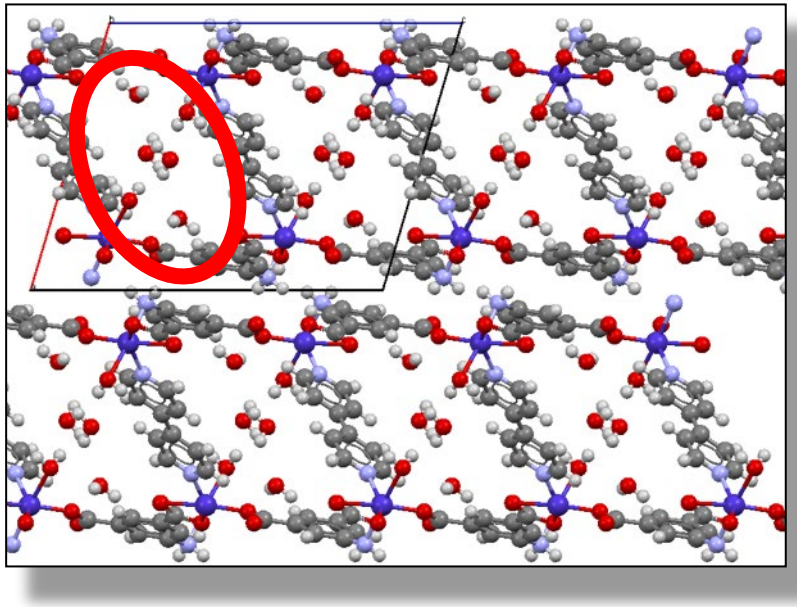


Environmental control cells (ECCs)
Study crystalline nanoporous materials under 'real world' conditions



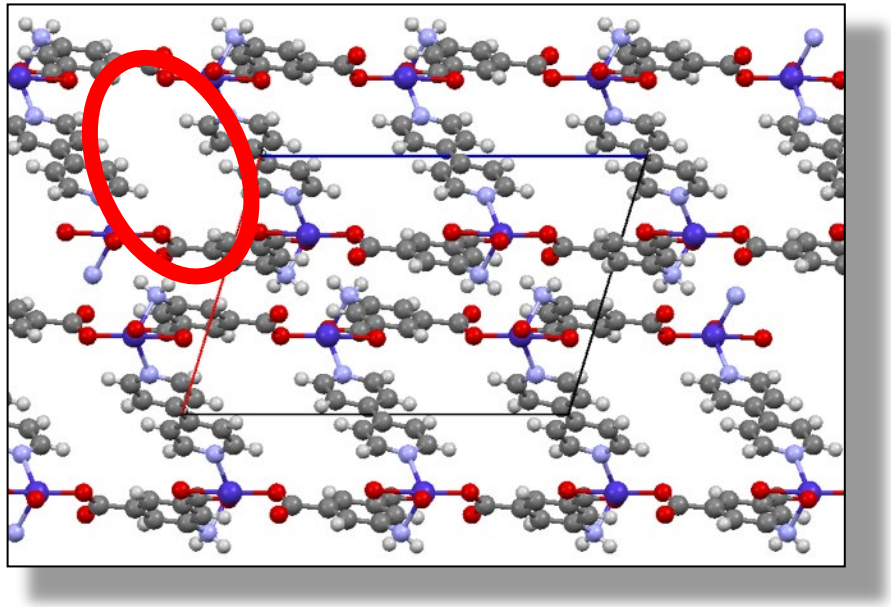
Approaching 'real time' structural information about guest and framework solving single crystal structures!

Time Resolved Structural Dynamics



Trihydrate (one coordinated water and two 'free' water molecules)

Space group $P2_1/c$
a = 12.4381 Å
b = 7.6827 Å
c = 15.8704 Å
β = 106.1466 °

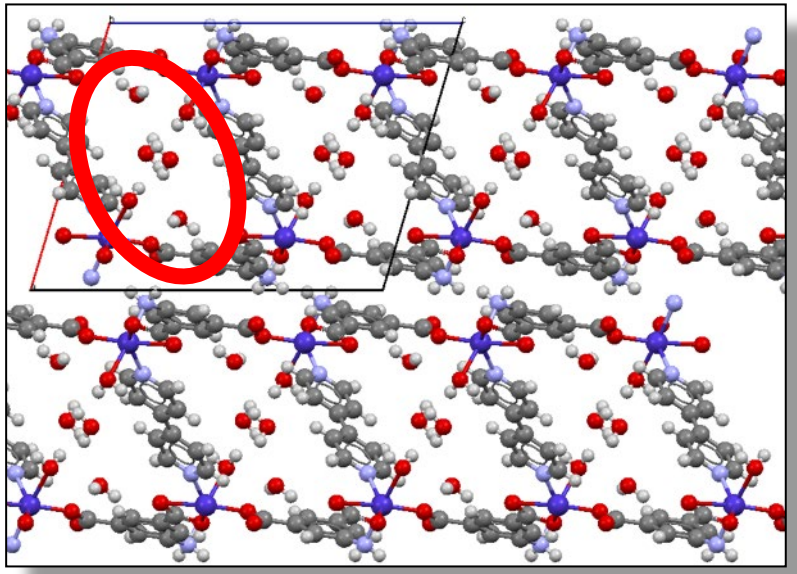


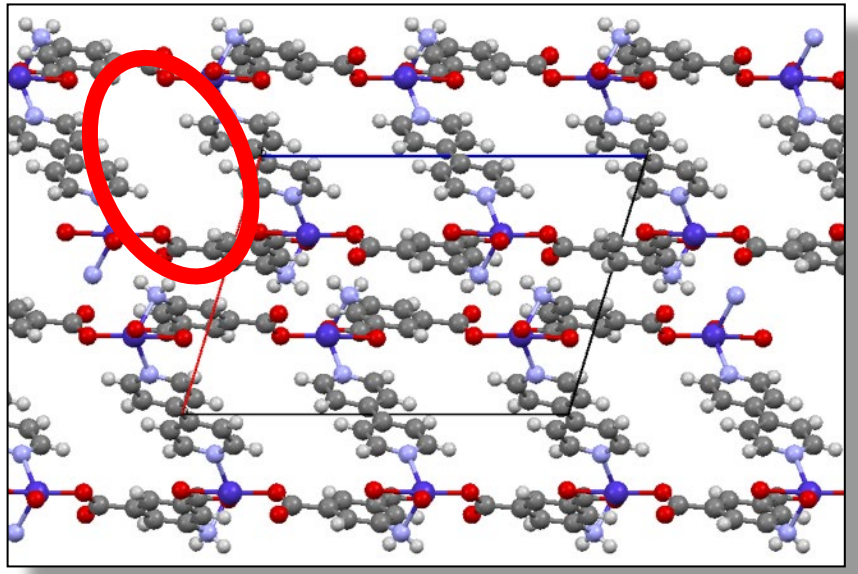
Anhydrous

Space group $P2_1/c$
a = 11.078 Å
b = 7.761 Å
c = 15.945 Å
β = 106.829 °

- How do water molecules leave?
  - Stepwise?
  - Simultaneous?
- What is the relationship between compression of *a*-axis and dehydration?
- *Dynamic in situ X-ray Diffraction* experiments should address both questions!

Time Resolved Structural Dynamics





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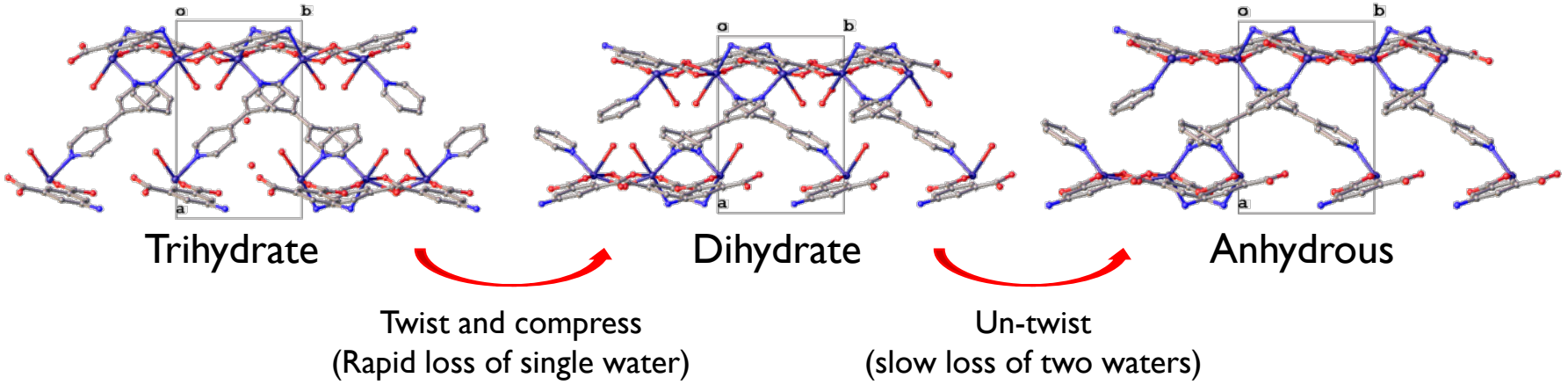
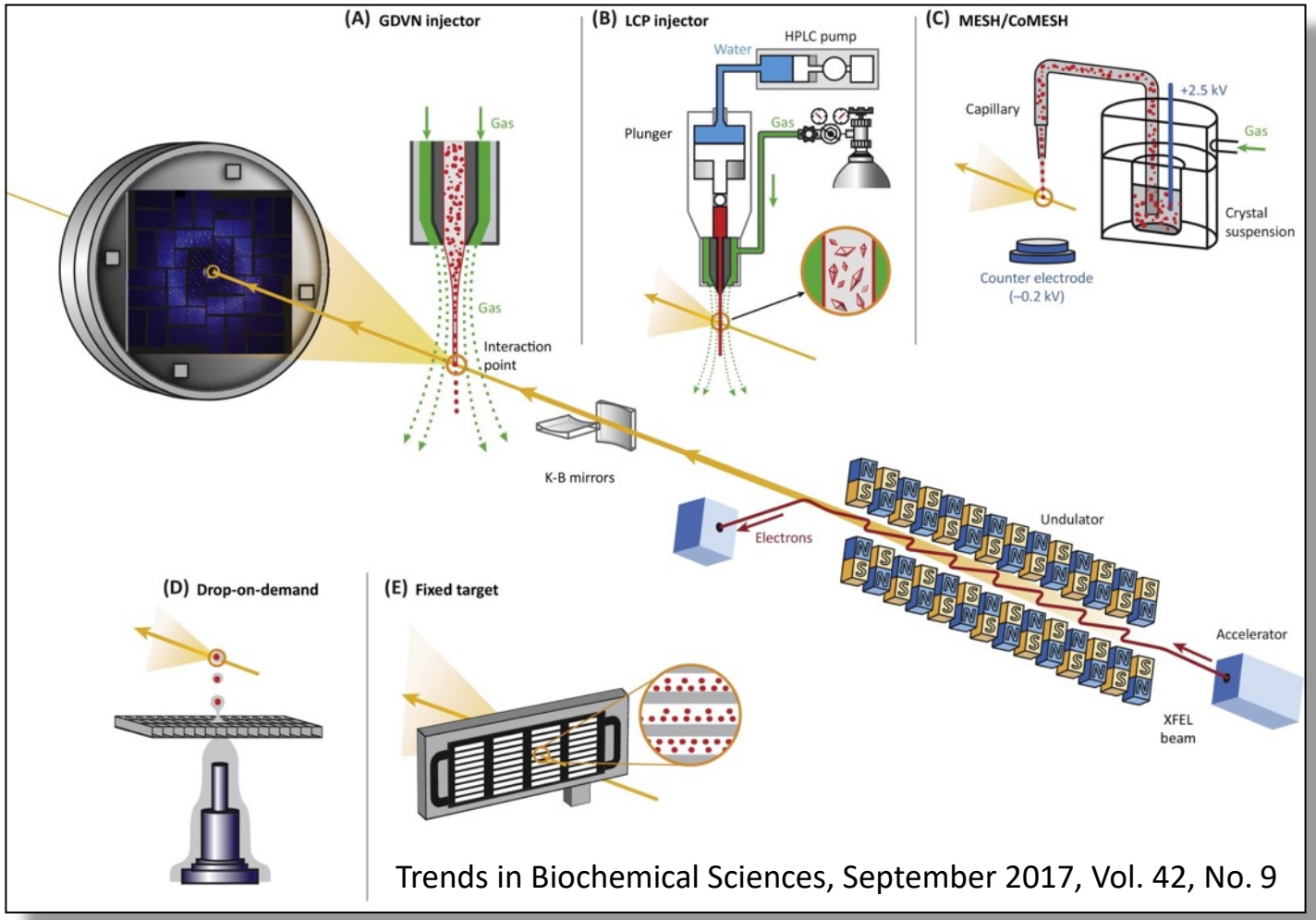


Structural Dynamics Tomorrow: *Small Molecule Serial Crystallography*

- What is it?
  - Technique developed by structural biologists
  - Datasets consist of single images collected from tens or hundreds of thousands of single crystals
  - New sample delivery methods
  - New data analysis methods

- Why would you do this?
- Only method capable of obtaining time-resolved data on:
  - Irreversible processes
  - Applies to process and damage
  - Sub-micron crystals



Trends in Biochemical Sciences, September 2017, Vol. 42, No. 9