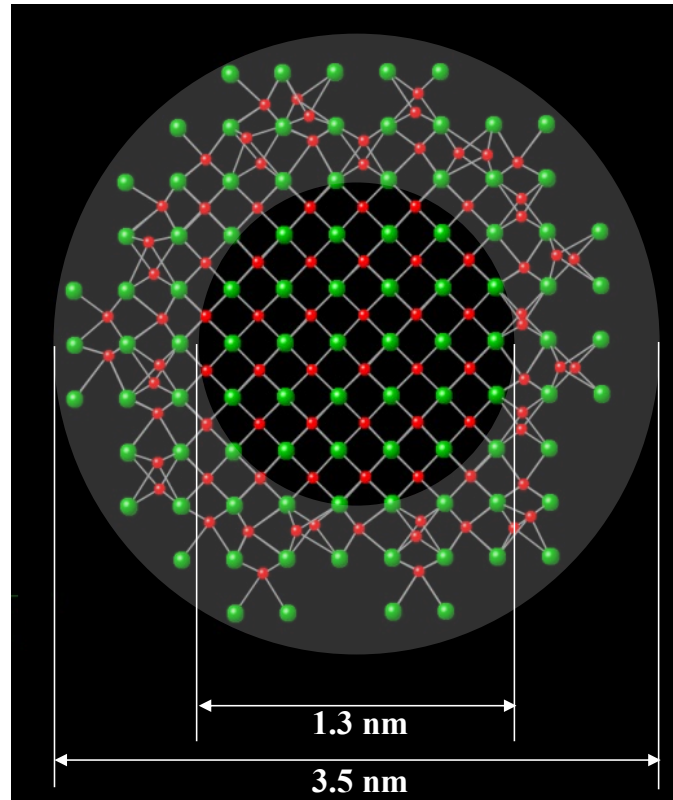


# Nano-Geoscience Capabilities using Synchrotron Radiation



John Bargar

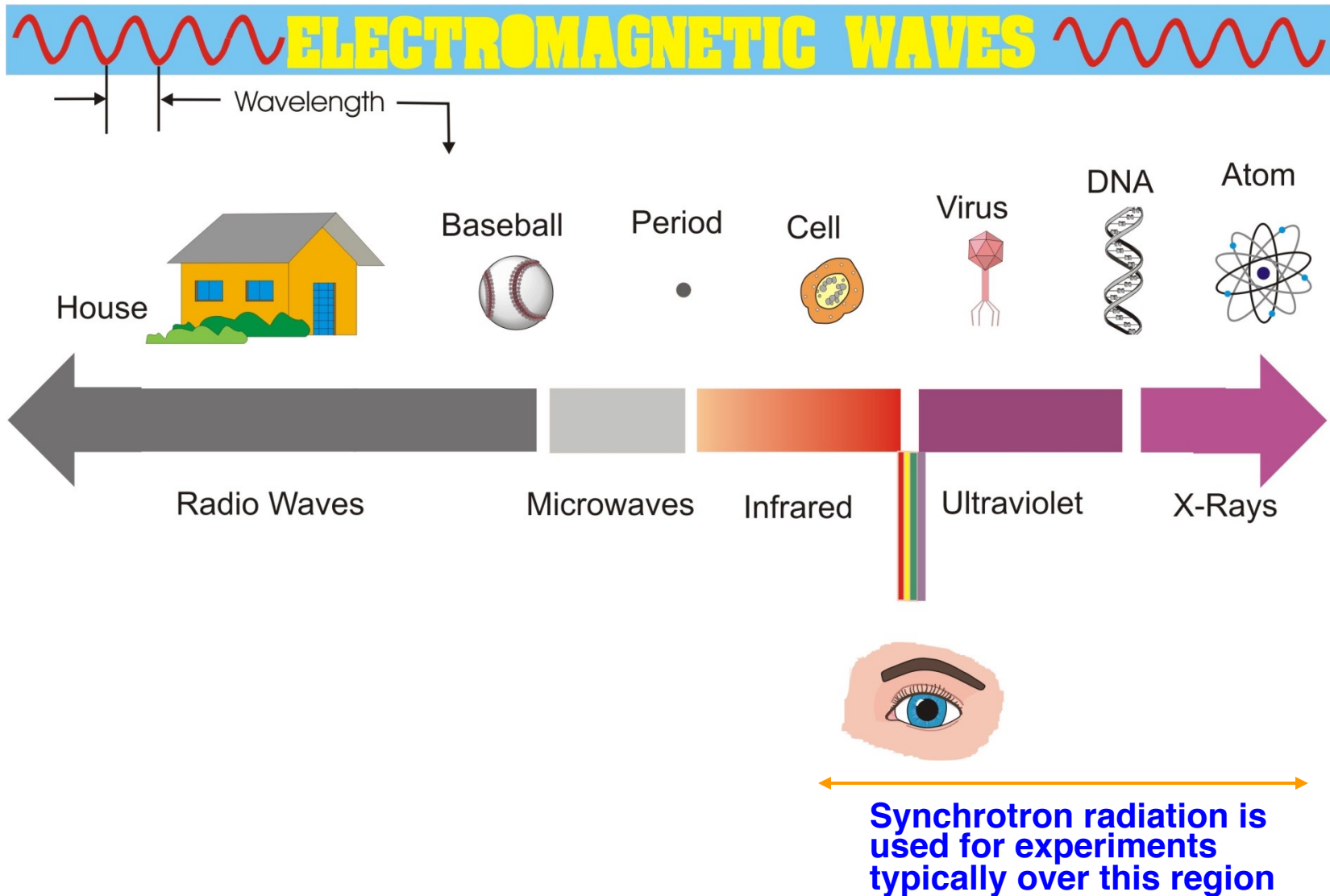
Stanford Synchrotron Radiation Lightsource

NNCI Nanoscience Earth and Environmental Science Research

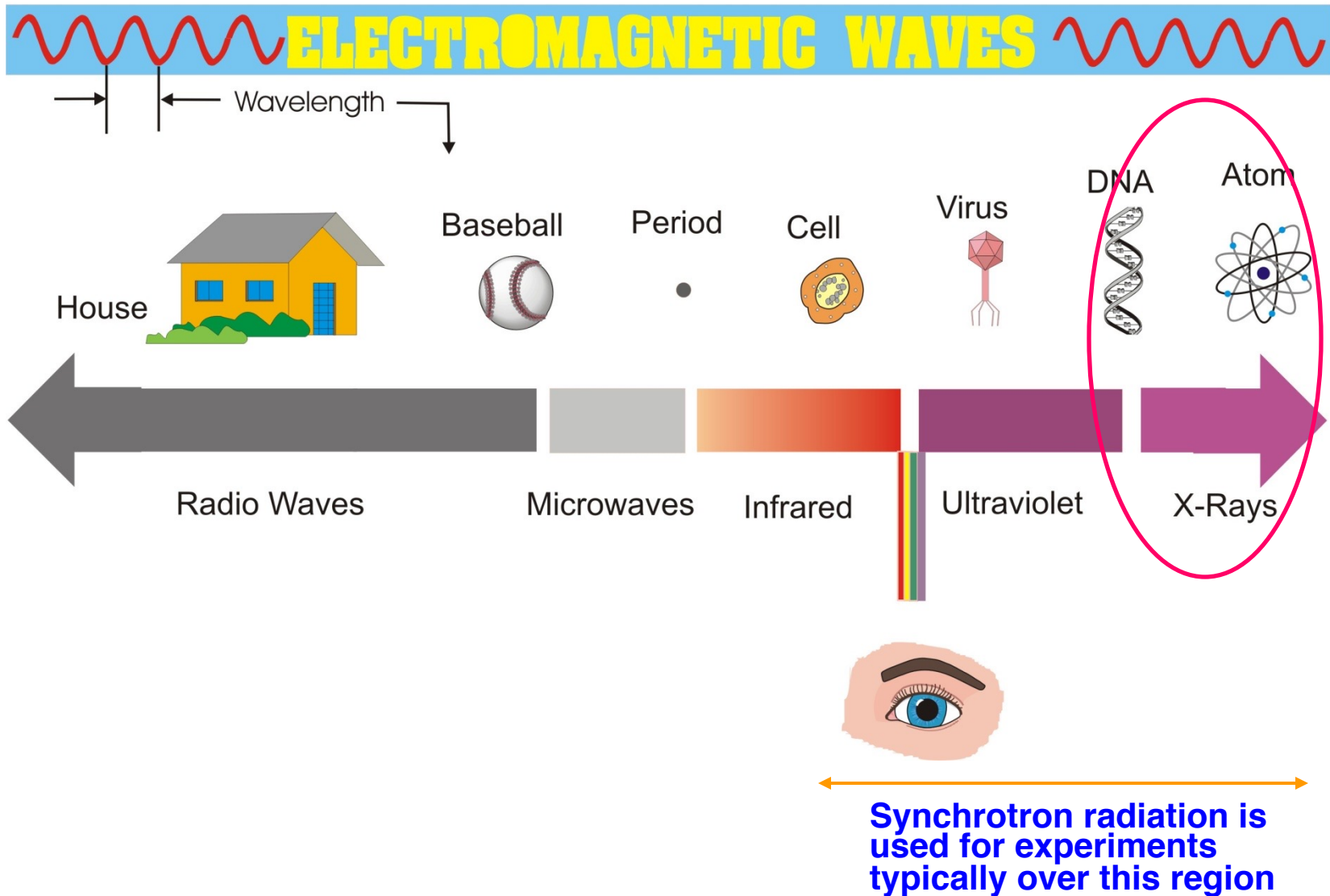
Community Virtual Workshop

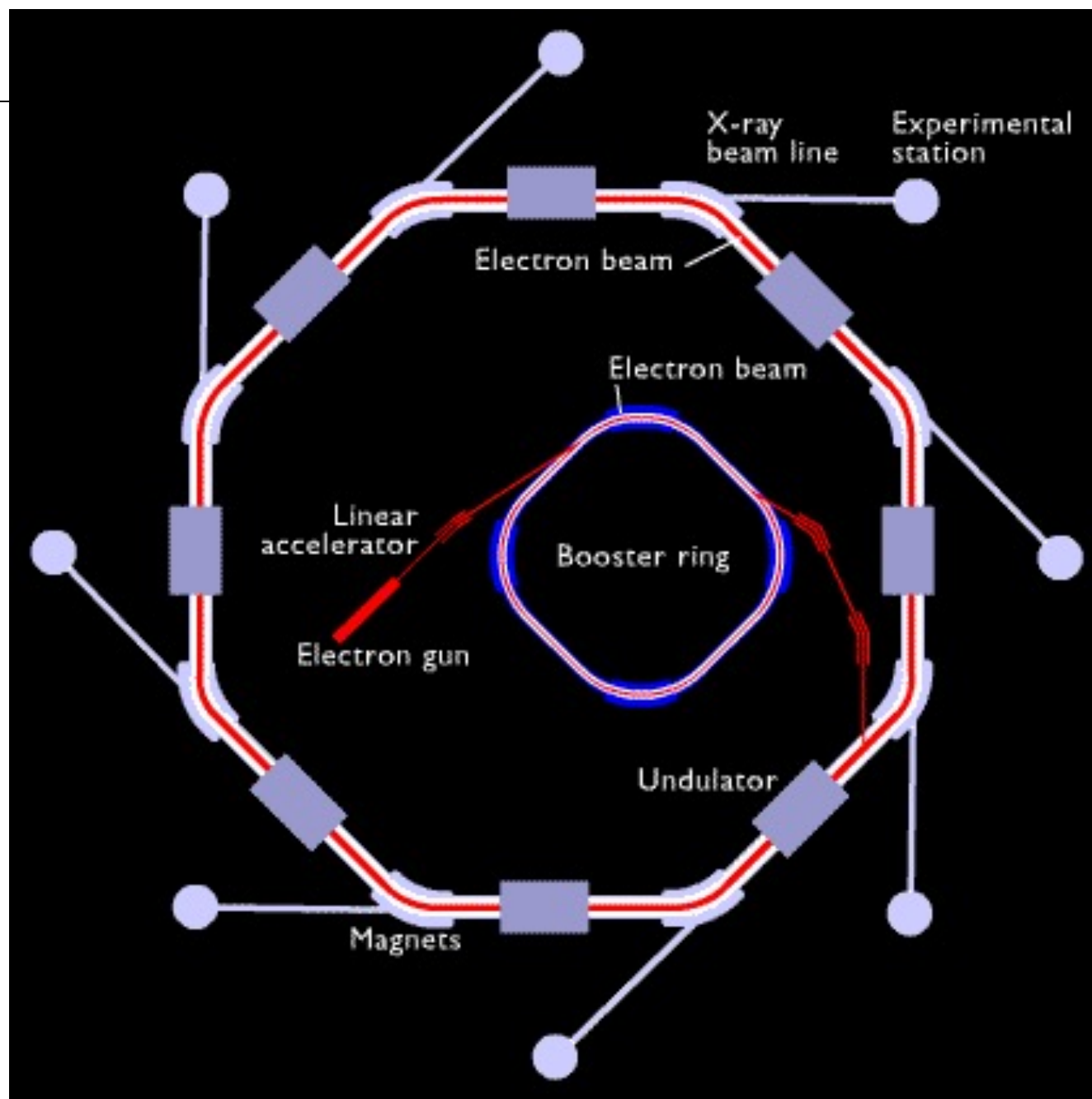
May 25, 2021

# Electromagnetic Radiation - How It Relates to the World We Know



# Electromagnetic Radiation - How It Relates to the World We Know





# What Makes Synchrotron Radiation (SR) so Useful?



## Wide energy spectrum:

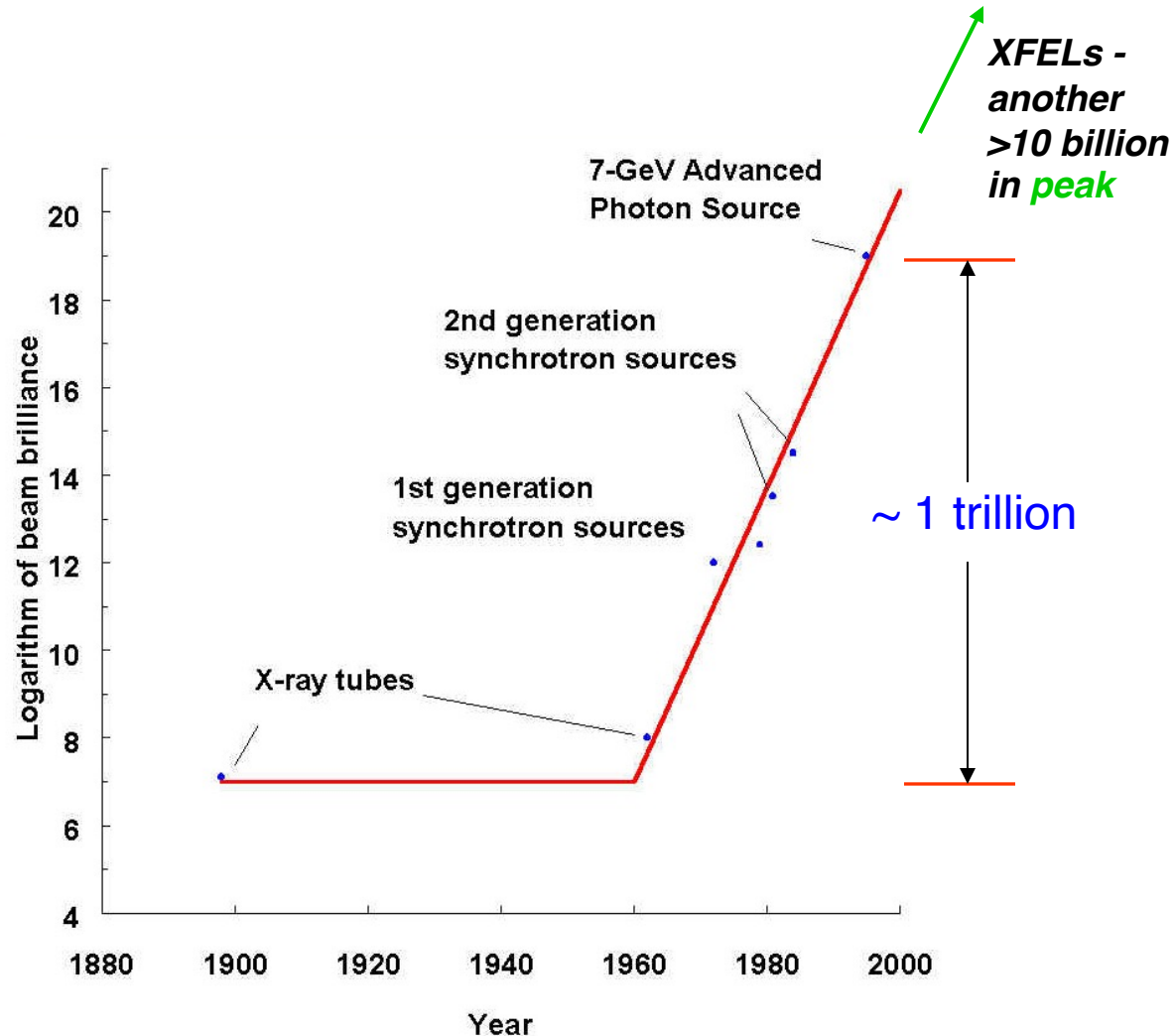
SR is emitted with a wide range of energies

## High brightness:

SR is extremely intense (hundreds of thousands of times higher than conventional x-ray tubes)

## Highly polarized and short pulses:

SR is emitted in very short pulses, typically less than a nano-second (a billionth of a second)



*SR offers many characteristics of visible lasers but into the x-ray regime!*

# Synchrotron methods for nanoparticle studies

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- High-resolution X-ray powder diffraction (SR-PD)
- Extended X-ray Absorption Fine Structure (EXAFS) Spectroscopy
- High-energy X-ray total scattering & PDF analysis (X-PDF)
- Small-angle X-ray scattering (SAXS)

# Synchrotron methods for nanoparticle studies

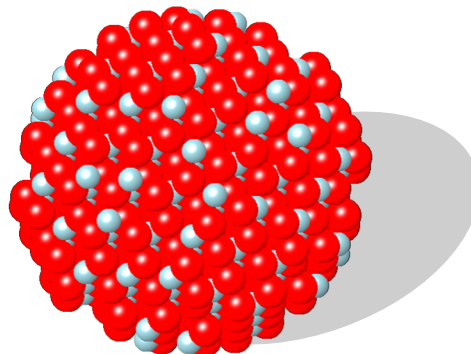
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- High-resolution X-ray powder diffraction (SR-PD)
- Extended X-ray Absorption Fine Structure (EXAFS) Spectroscopy
- High-energy X-ray total scattering & PDF analysis (X-PDF)
- Small-angle X-ray scattering (SAXS)

# What is the structure of biogenic nano-uraninite?

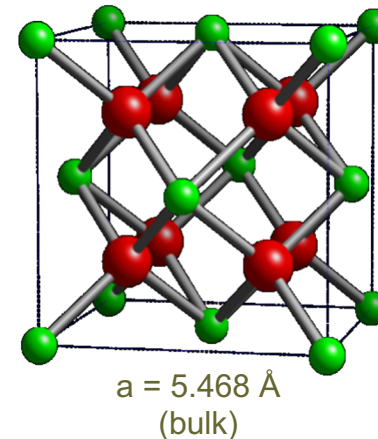
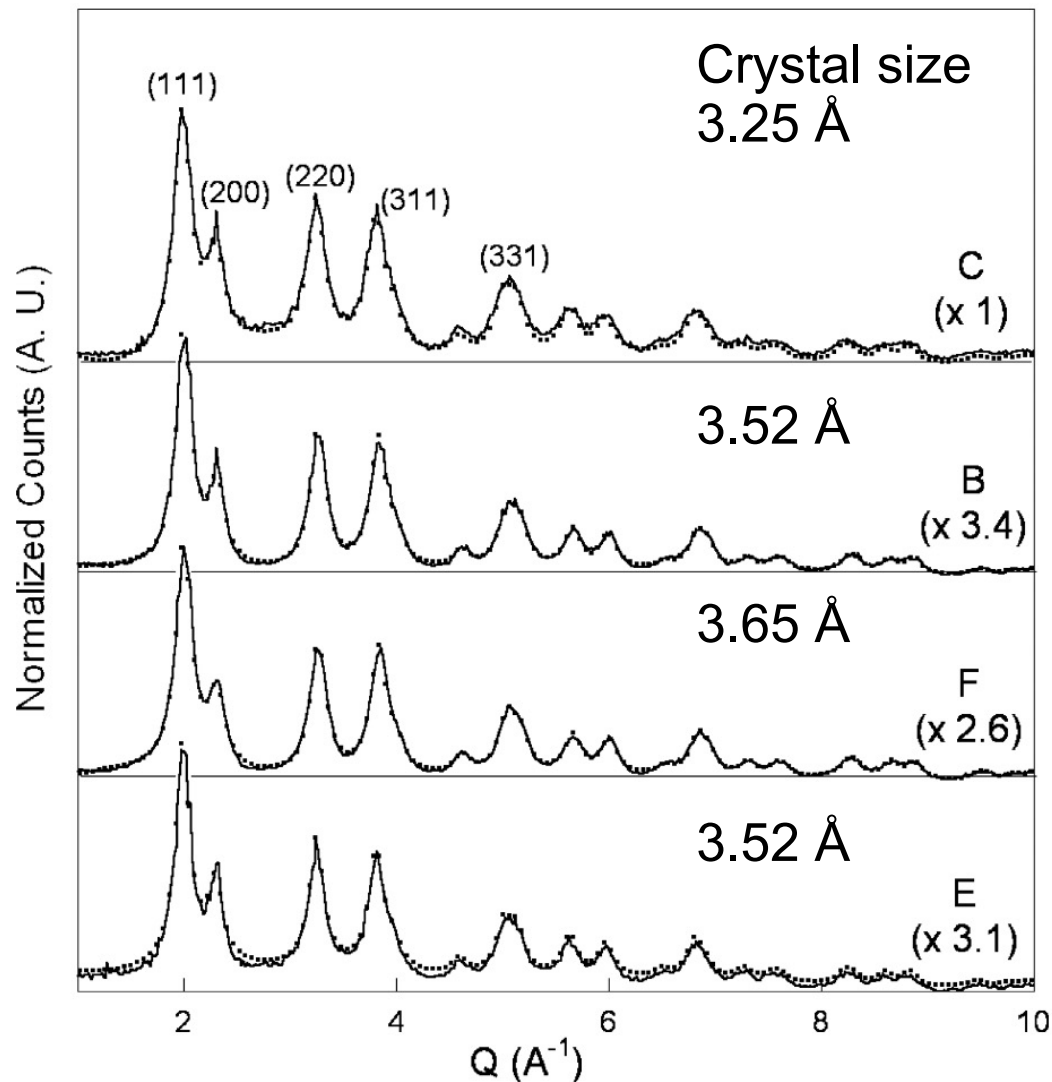
Hypothesis: Will be non-stoichiometric, nano-sized & highly defected due strain





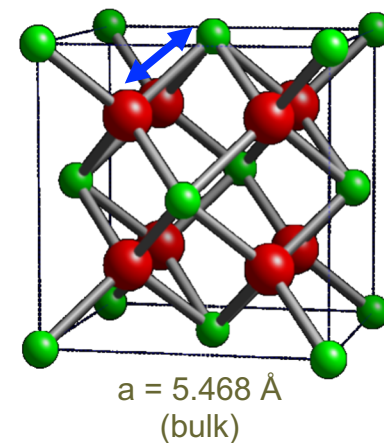
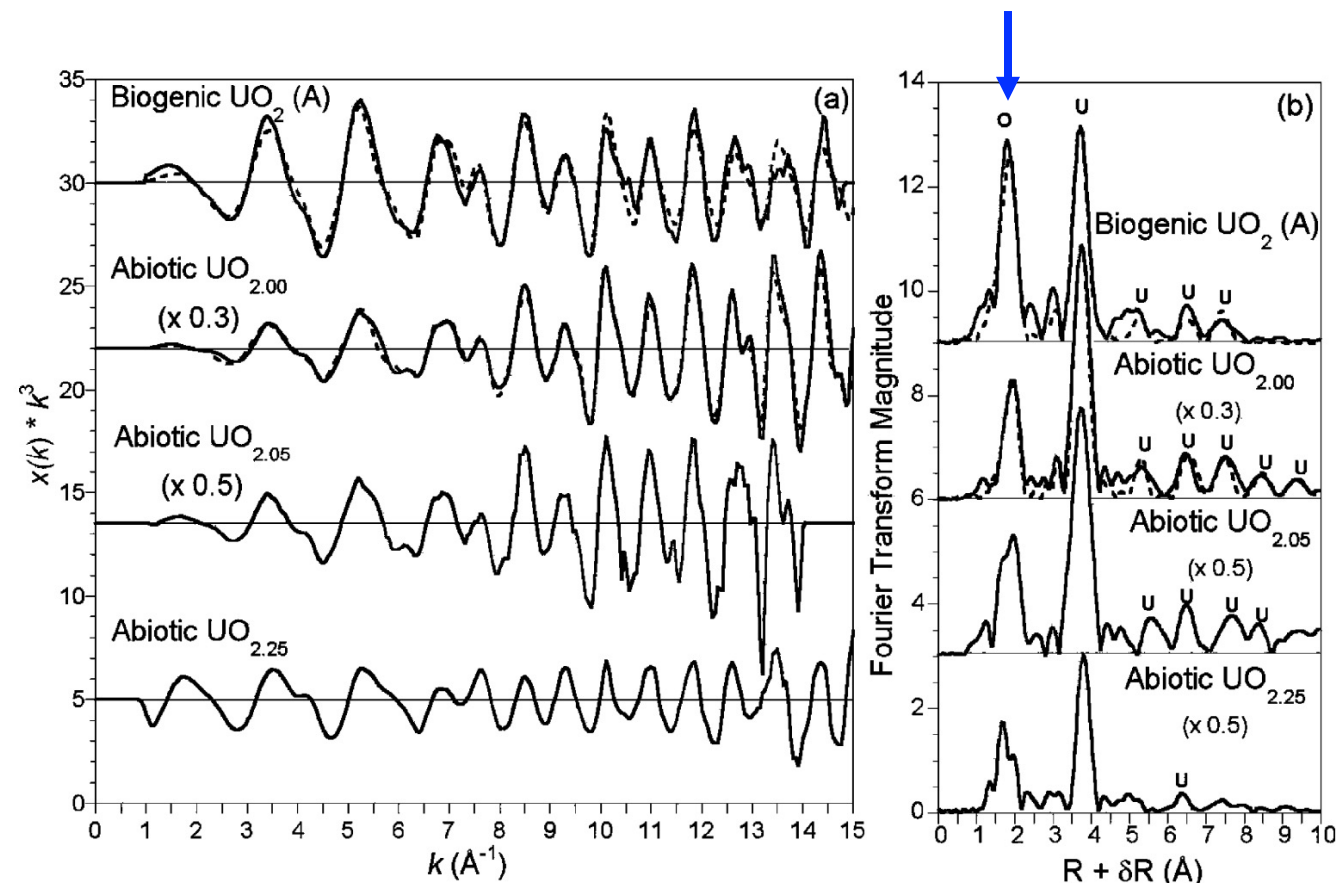
# Powder Diffraction

## Biogenic $\text{UO}_2$ nanoparticles

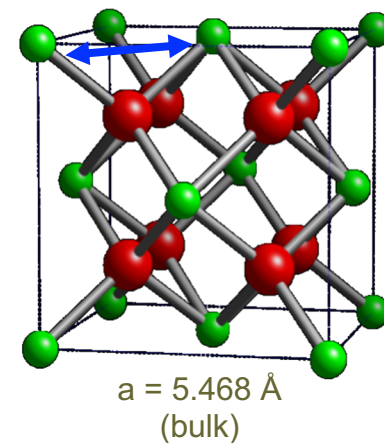
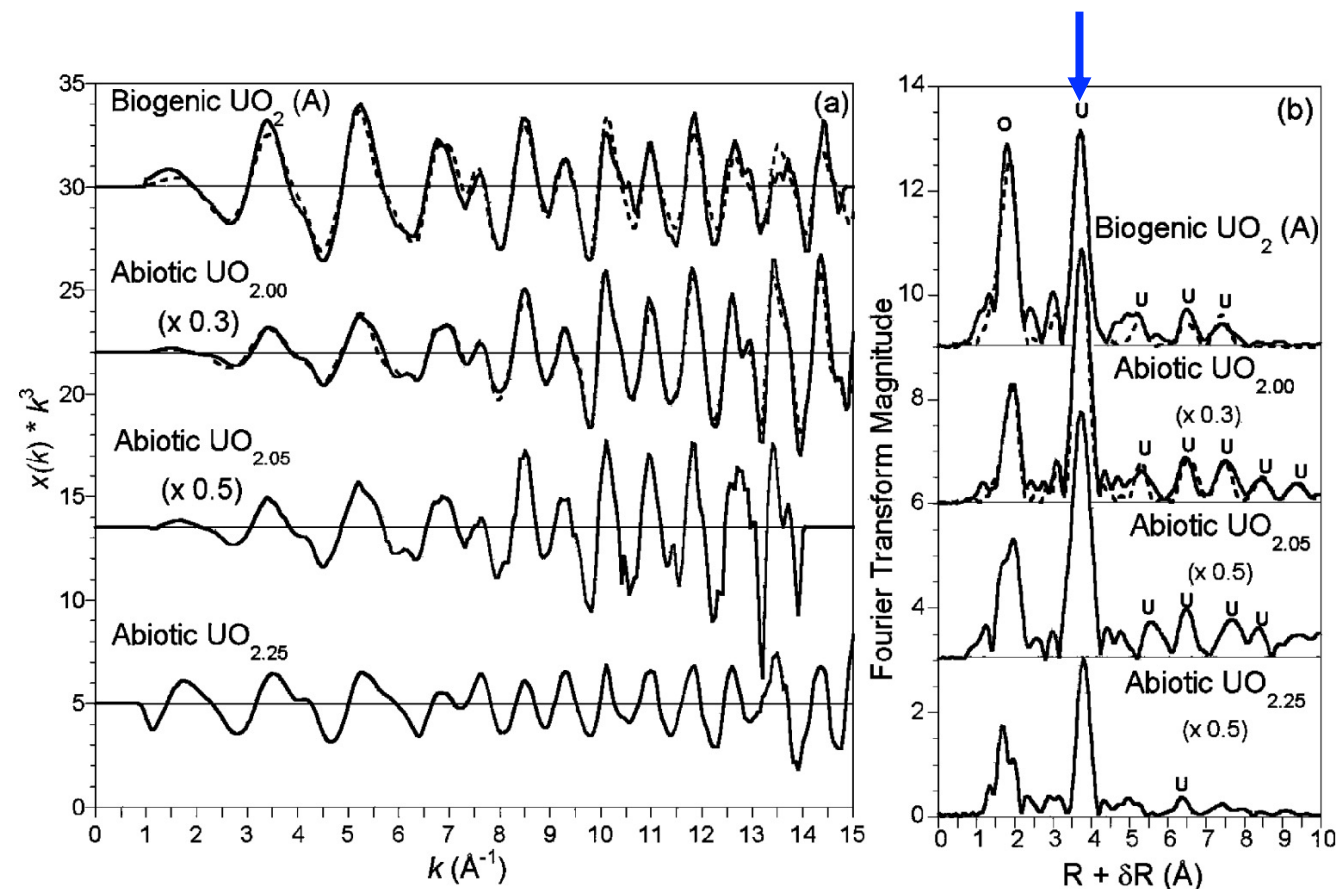


- Particle size from peak broadening
- Lattice parameter showed that there was very little strain!
- Unit cell: stoichiometric  $\text{UO}_{2.0}$
- Other useful information:
  - Atomic positions

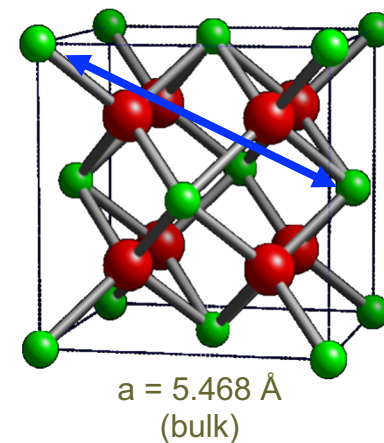
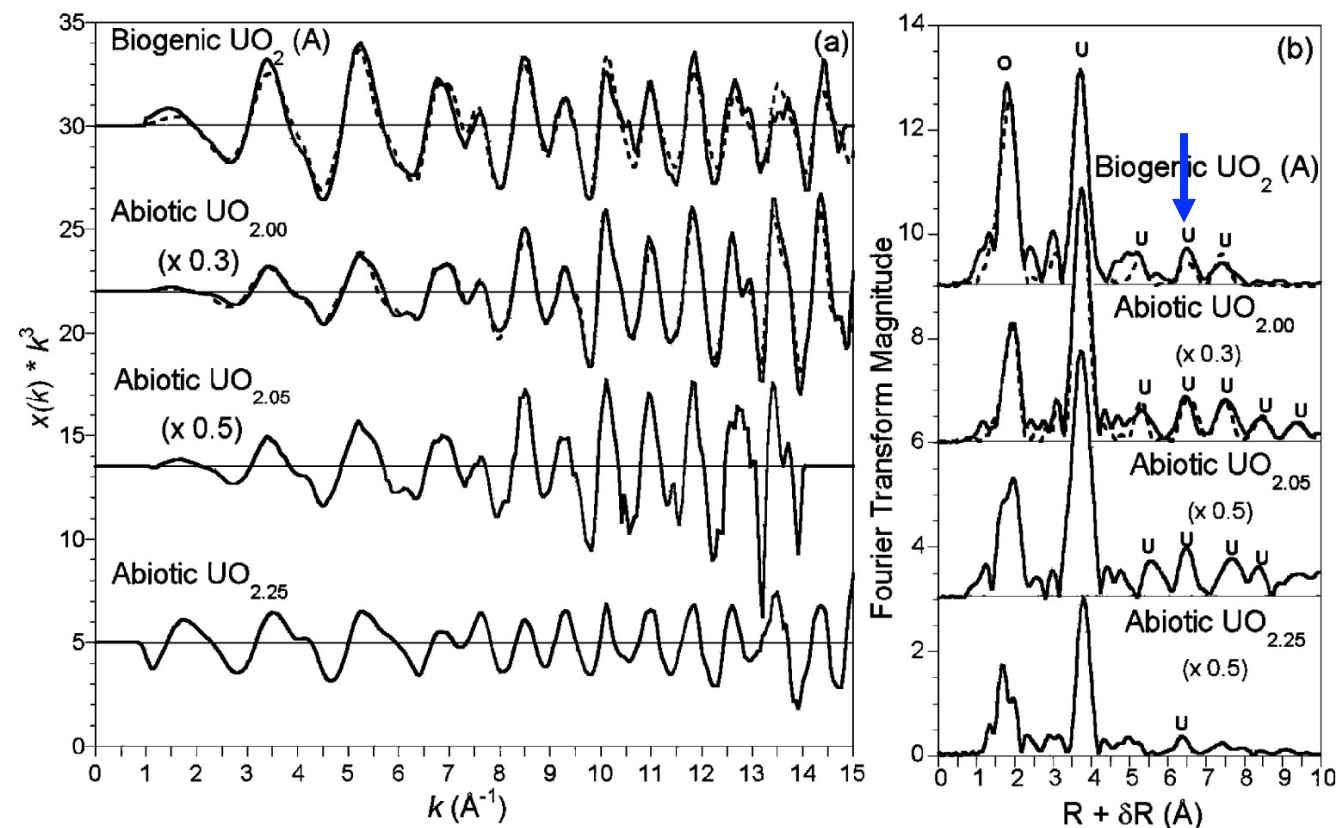
# EXAFS spectroscopy: Biogenic $\text{UO}_2$ nanoparticles



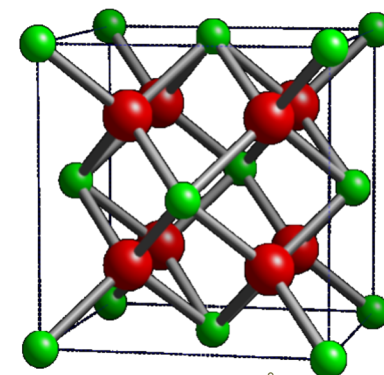
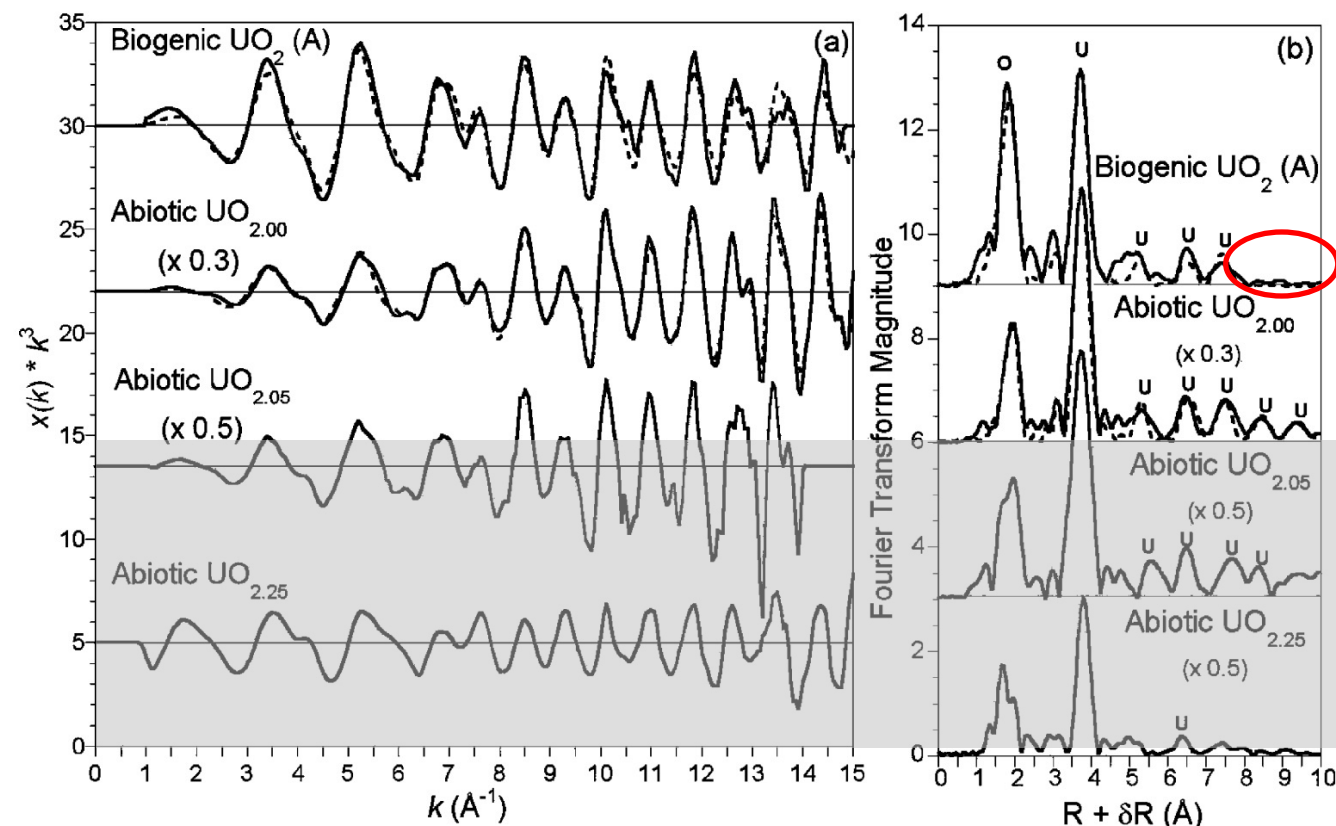
# EXAFS spectroscopy: Biogenic $\text{UO}_2$ nanoparticles



# EXAFS spectroscopy: Biogenic $\text{UO}_2$ nanoparticles

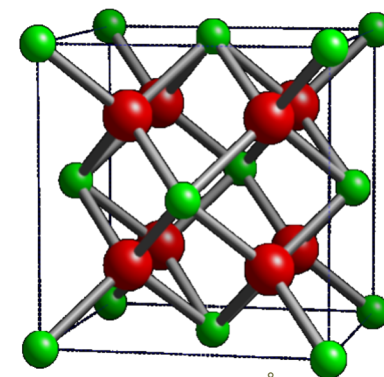
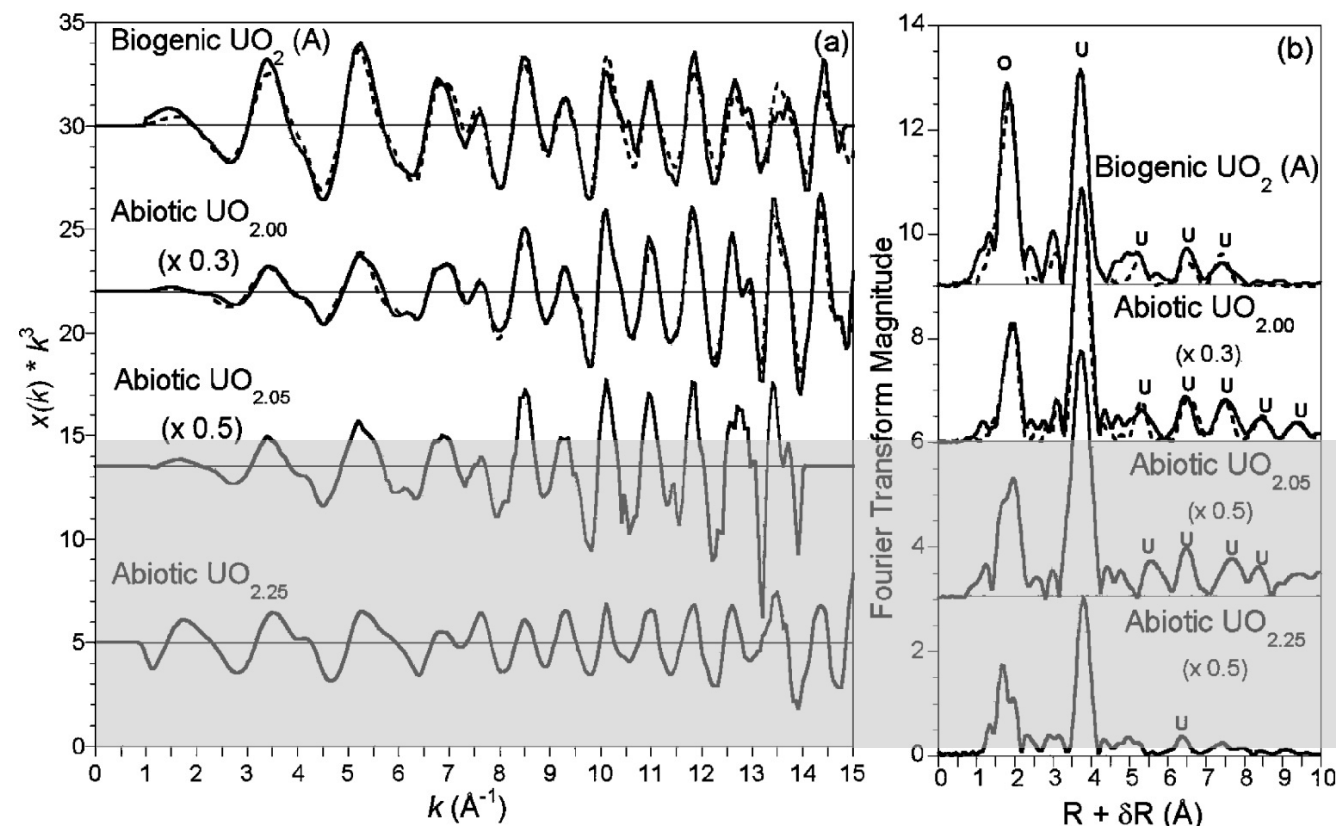


# EXAFS spectroscopy: Biogenic $\text{UO}_2$ nanoparticles



- Missing peaks provides information on (very small) particle size

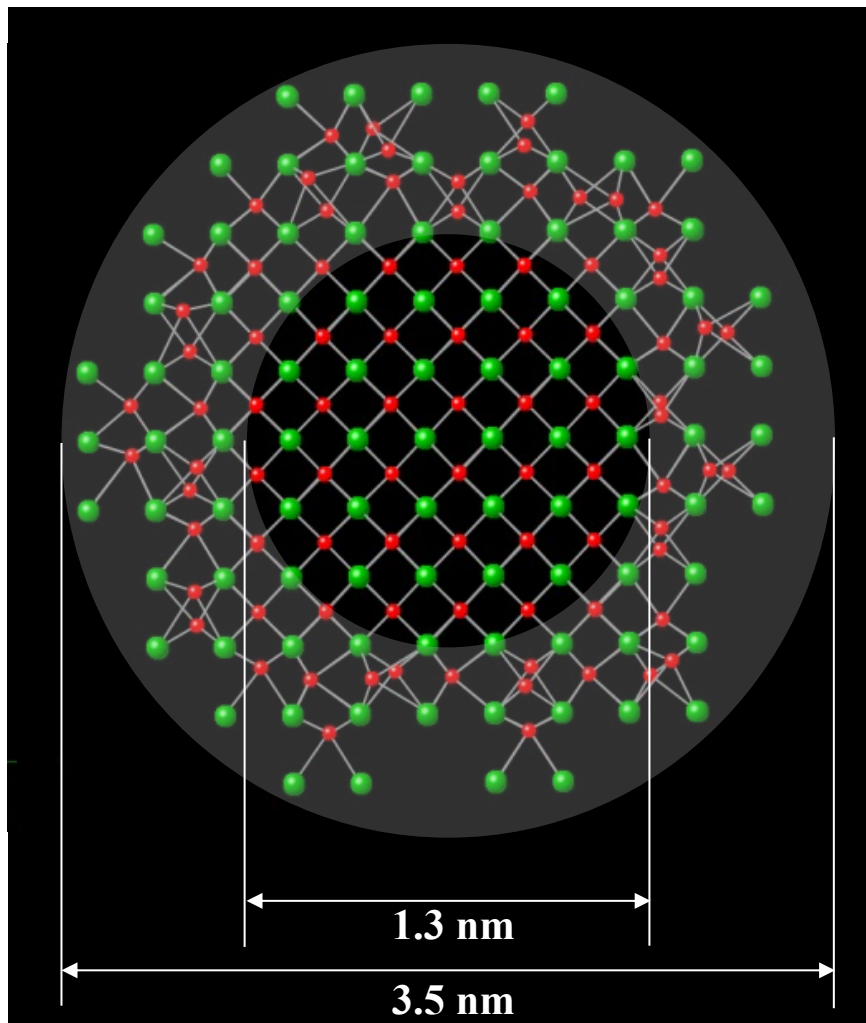
# EXAFS spectroscopy: Biogenic $\text{UO}_2$ nanoparticles



- Structural coherence length of 1.2  $\text{\AA}$
- Smaller than XRD
- What can we learn from this?



# Combining SR-PD & EXAFS spectroscopy: Biogenic $\text{UO}_2$ nanoparticles



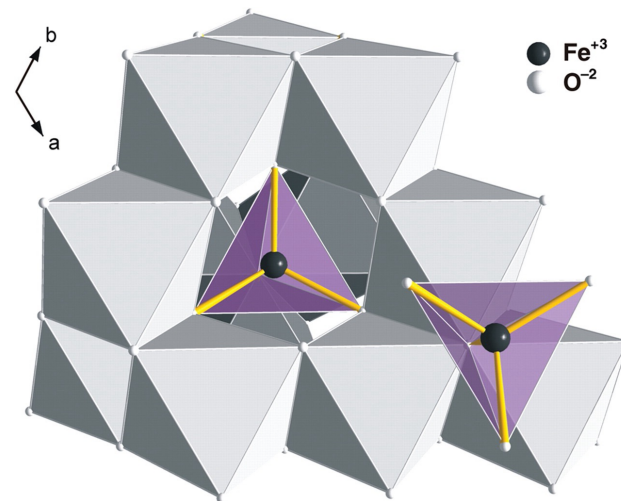
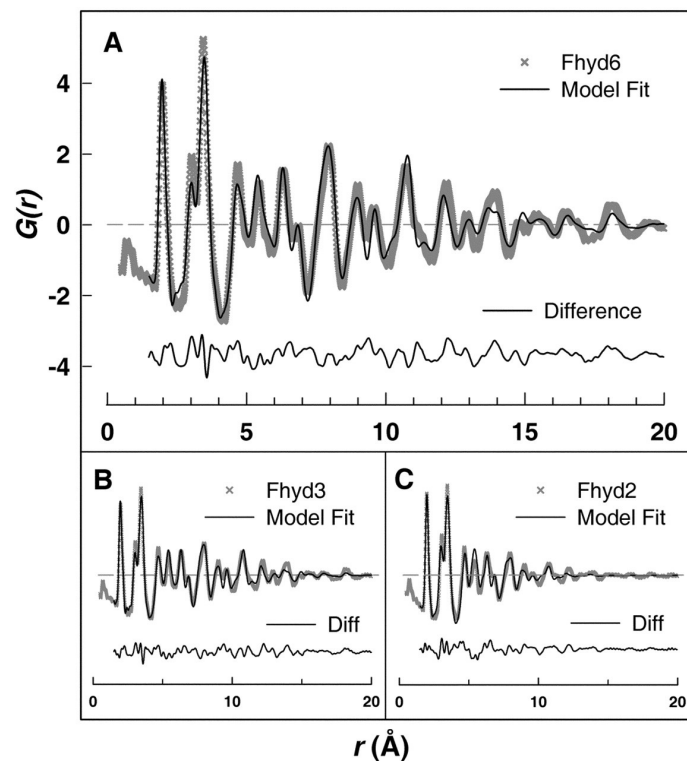
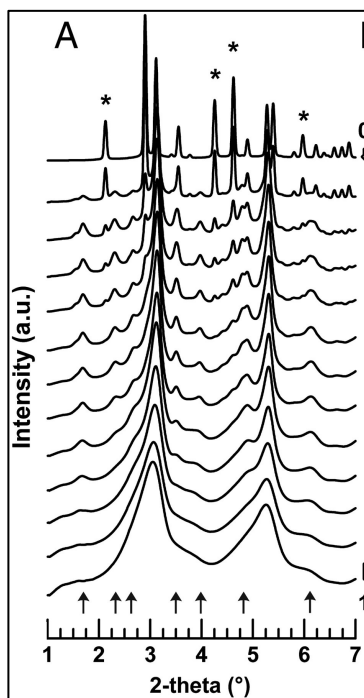
Green = Oxygen; Red = Uranium

- XRD: Diffracting crystal size
- EXAFS: far more sensitive to disorder – gives diameter of highly ordered portion of nanoparticles

**What is the structure of  
ferrihydrite?**

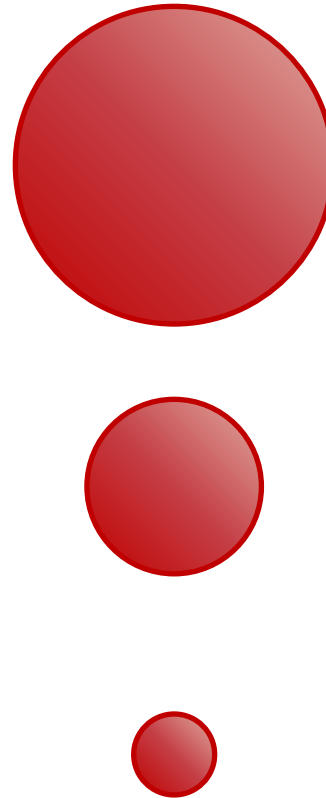
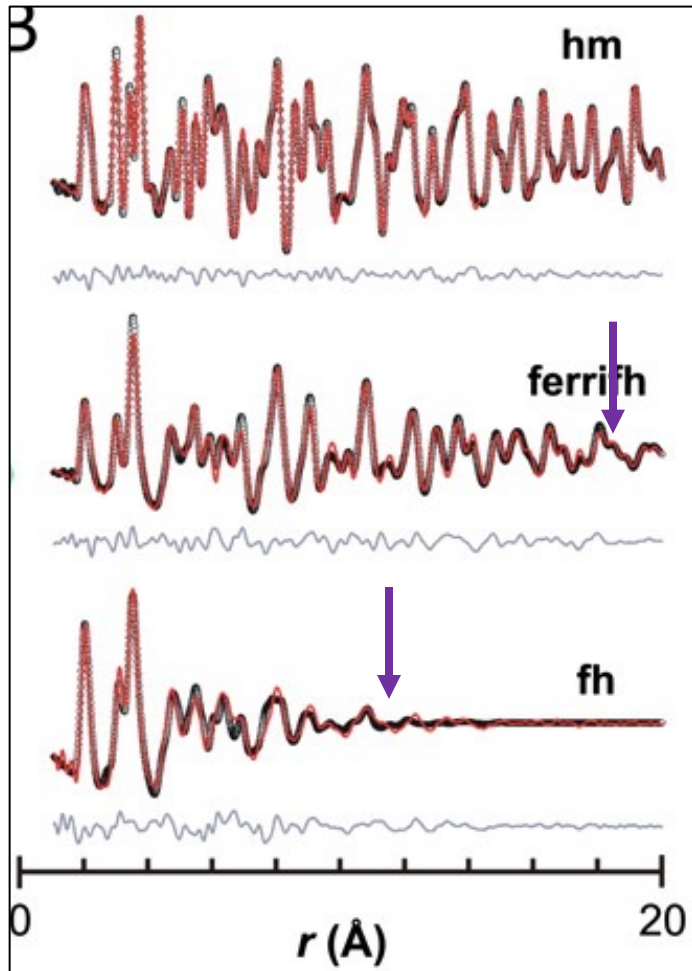


# High Energy X-ray Scattering – PDF Analysis



F. M. Michel *Science* **316** (5832), pp. 1726, 2007

# High Energy X-ray Scattering – PDF Analysis



hematite

↑

ordered  
ferrihydrite

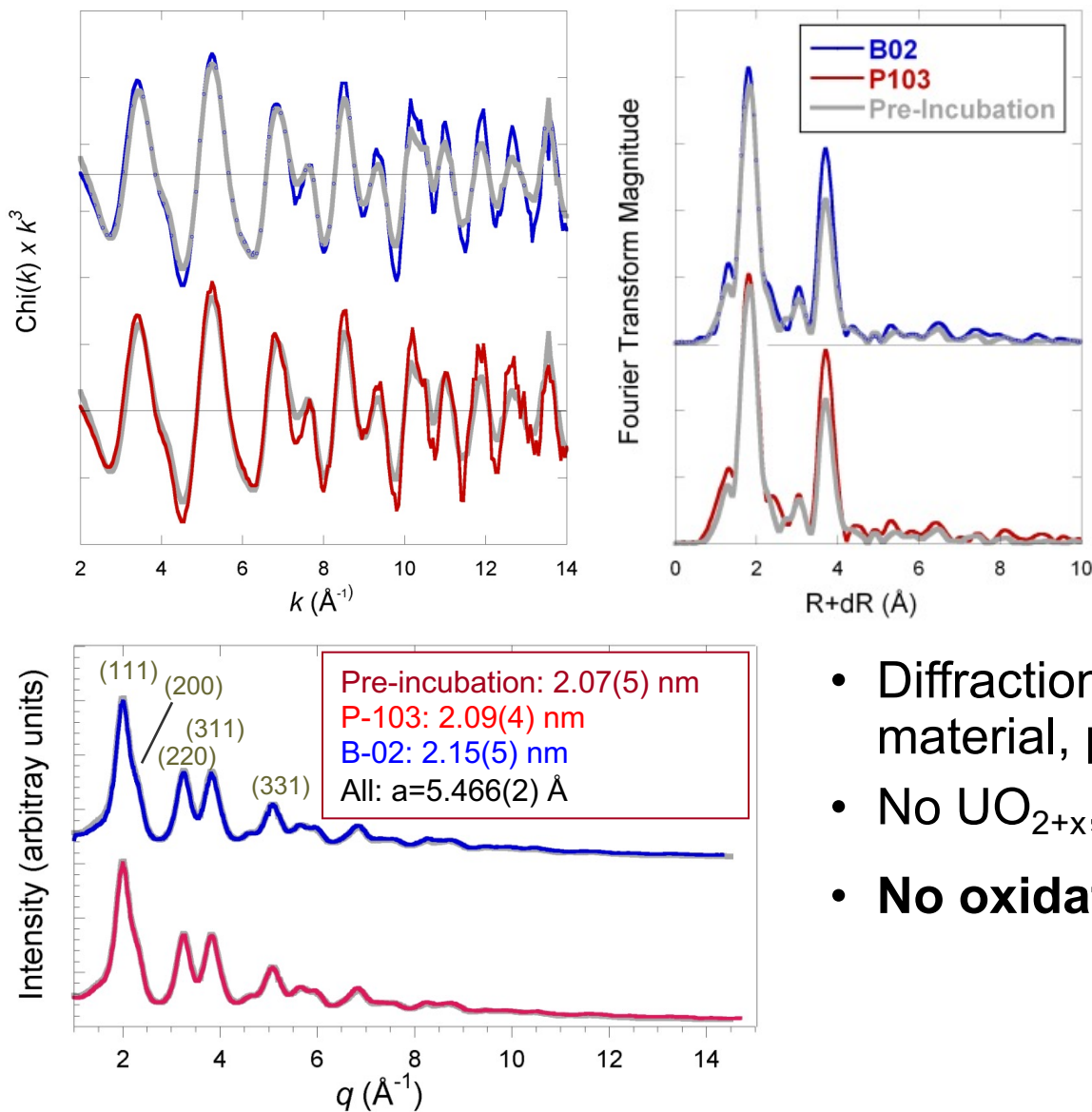
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ferrihydrite

# Thank you!



# Powder Diffraction



- No accumulation of  $\text{UO}_{2+x}$  or U(VI) solids.
- EXAFS: Local structural order similar before/after
- Diffraction: no change in material, particle size.
- No  $\text{UO}_{2+x}$ , calcite, other phases.
- **No oxidation products**