Amplifying science insights - synergistic partnerships between EMSL and Light Sources

Ken Kemner, APS

Douglas Mans, EMSL



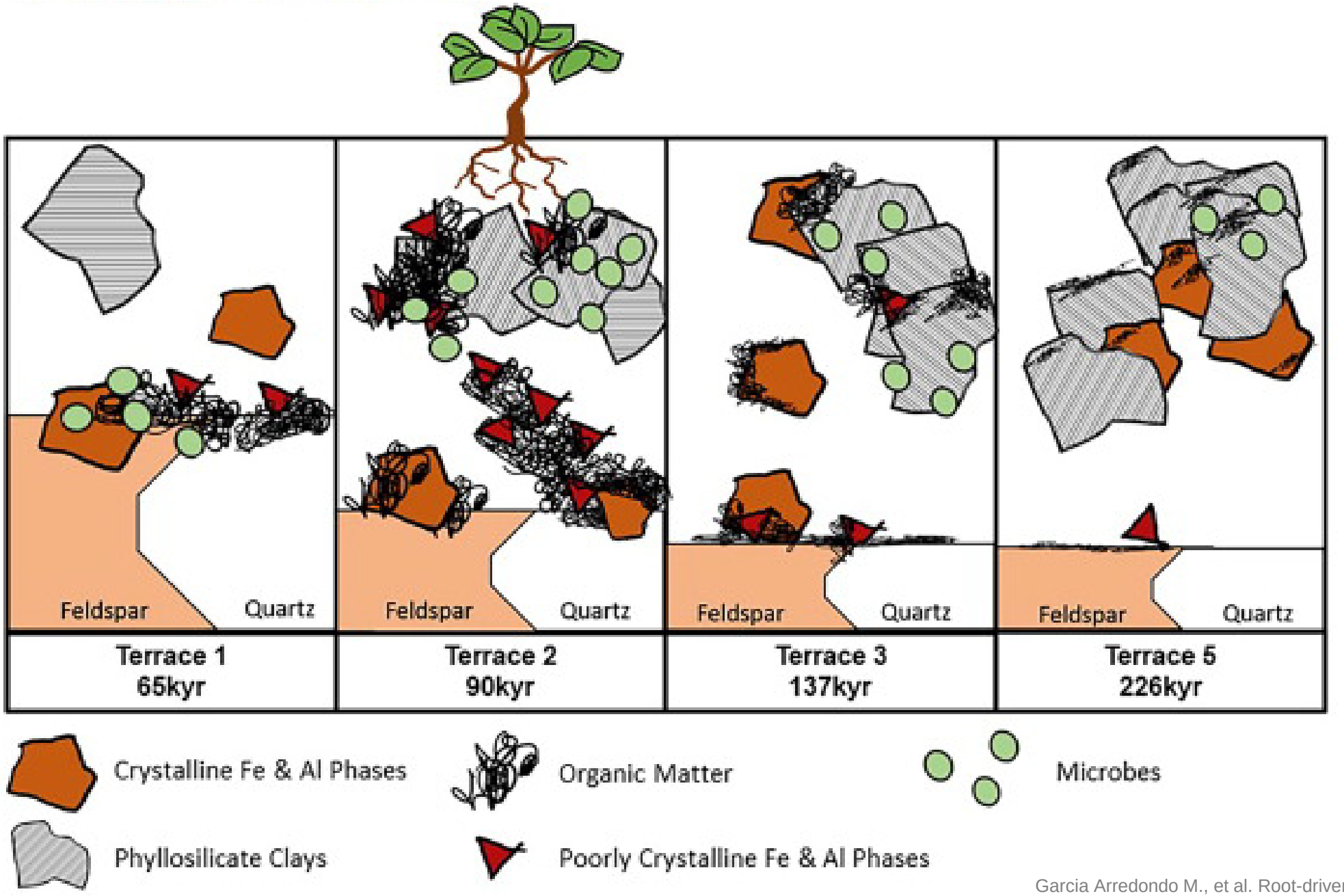
SSRL

NSLS-II
National Synchrotron Light Source II

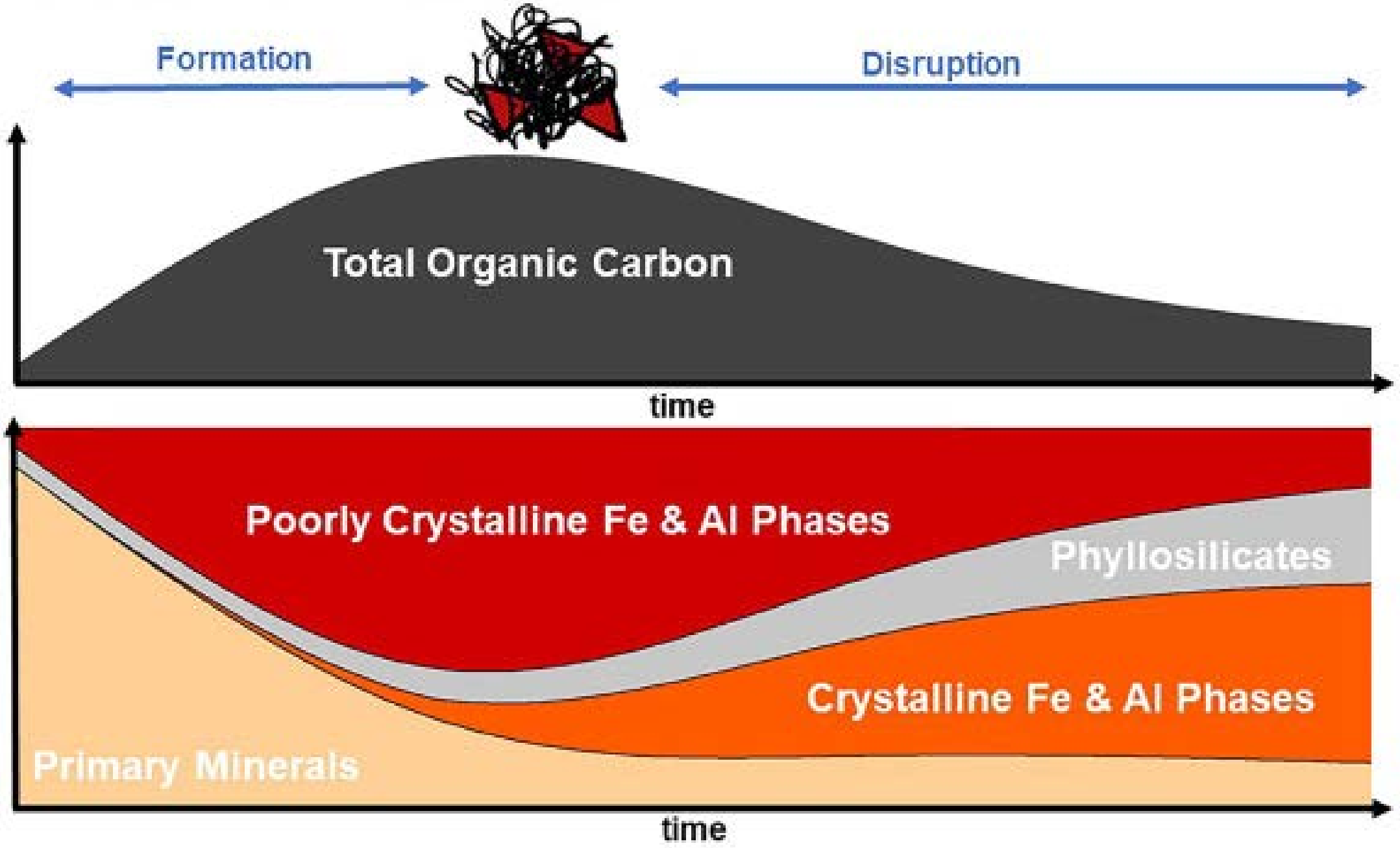
EMSL

Advanced Photon Source

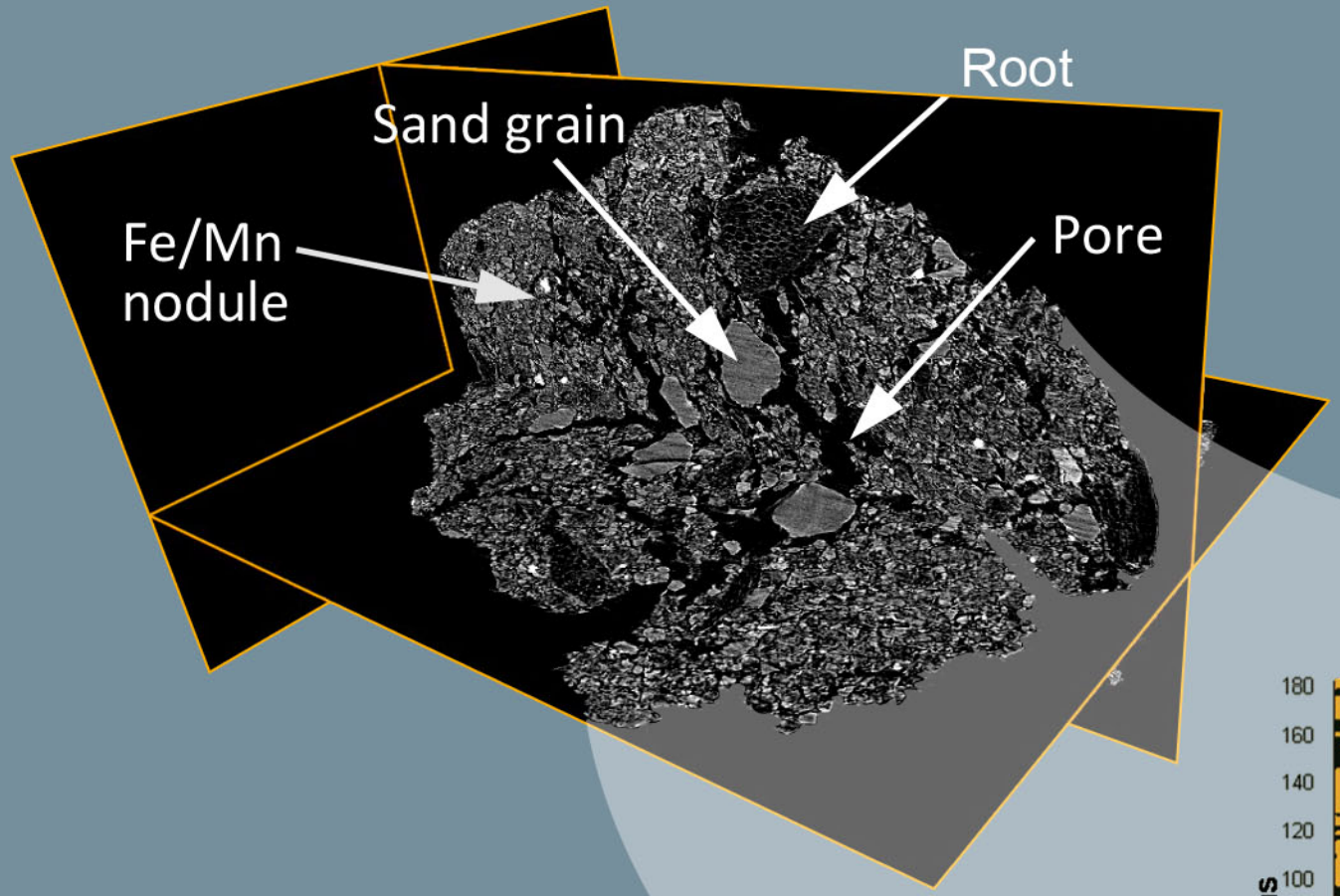
ALS
ADVANCED LIGHT SOURCE

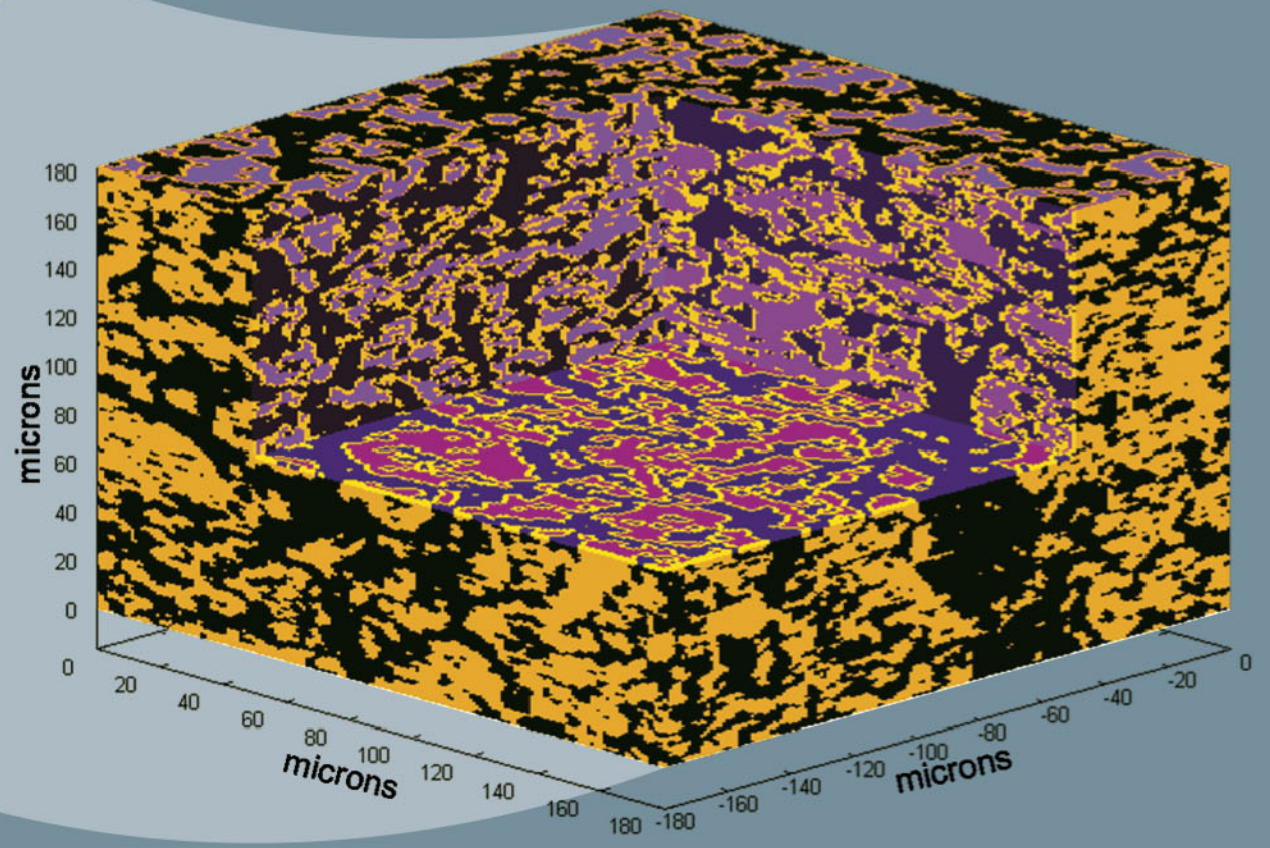


Garcia Arredondo M., et al. Root-driven weathering impacts on mineral-organic associations in deep soils over pedogenic time scales. *Geochim. Cosmochim. Acta* (2019), DOI:10.1016/j.gca.2019.07.030

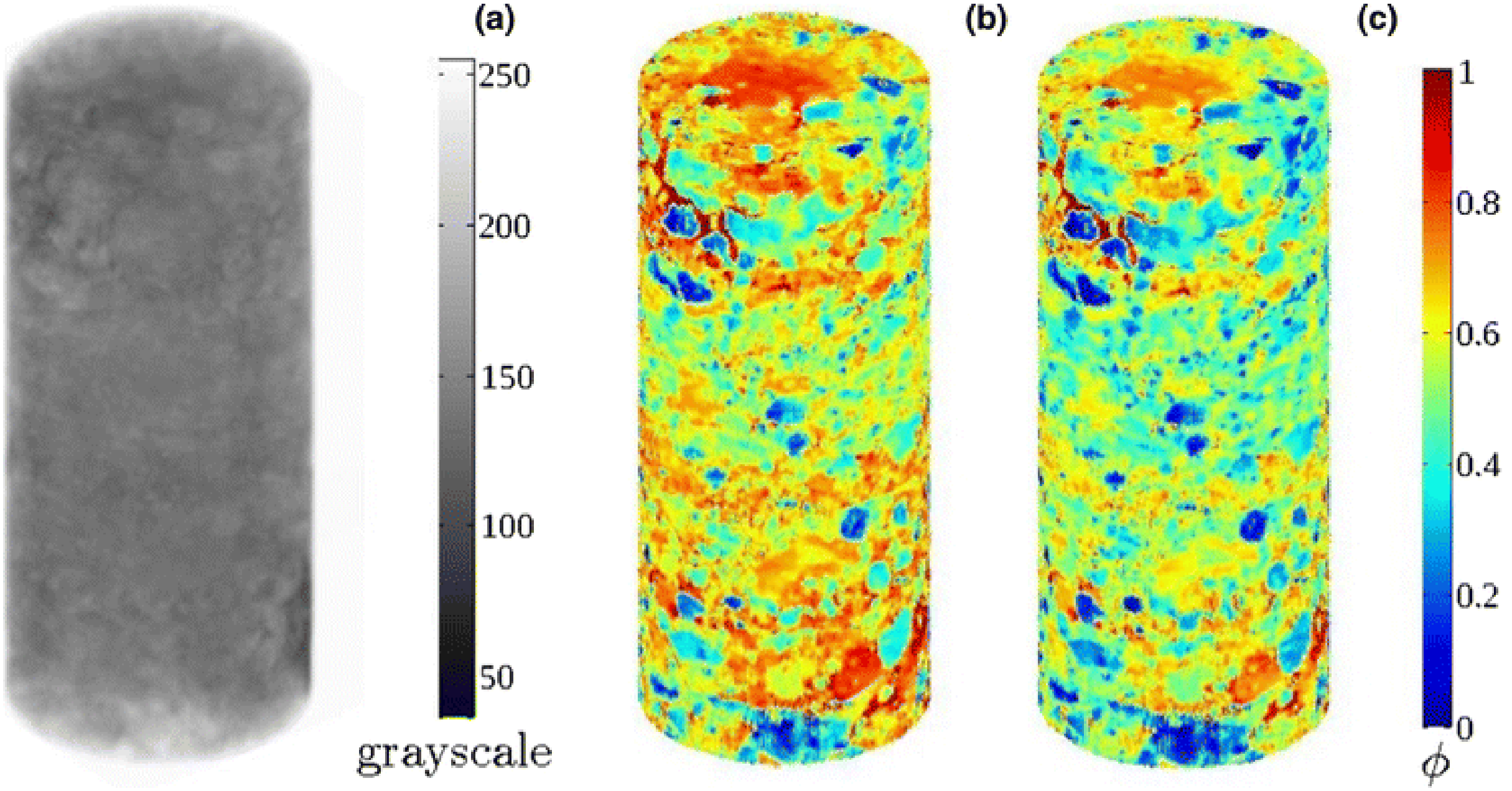


Garcia Arredondo M., et al. Root-driven weathering impacts on mineral-organic associations in deep soils over pedogenic time scales. *Geochim. Cosmochim. Acta* (2019), DOI:10.1016/j.gca.2019.07.030



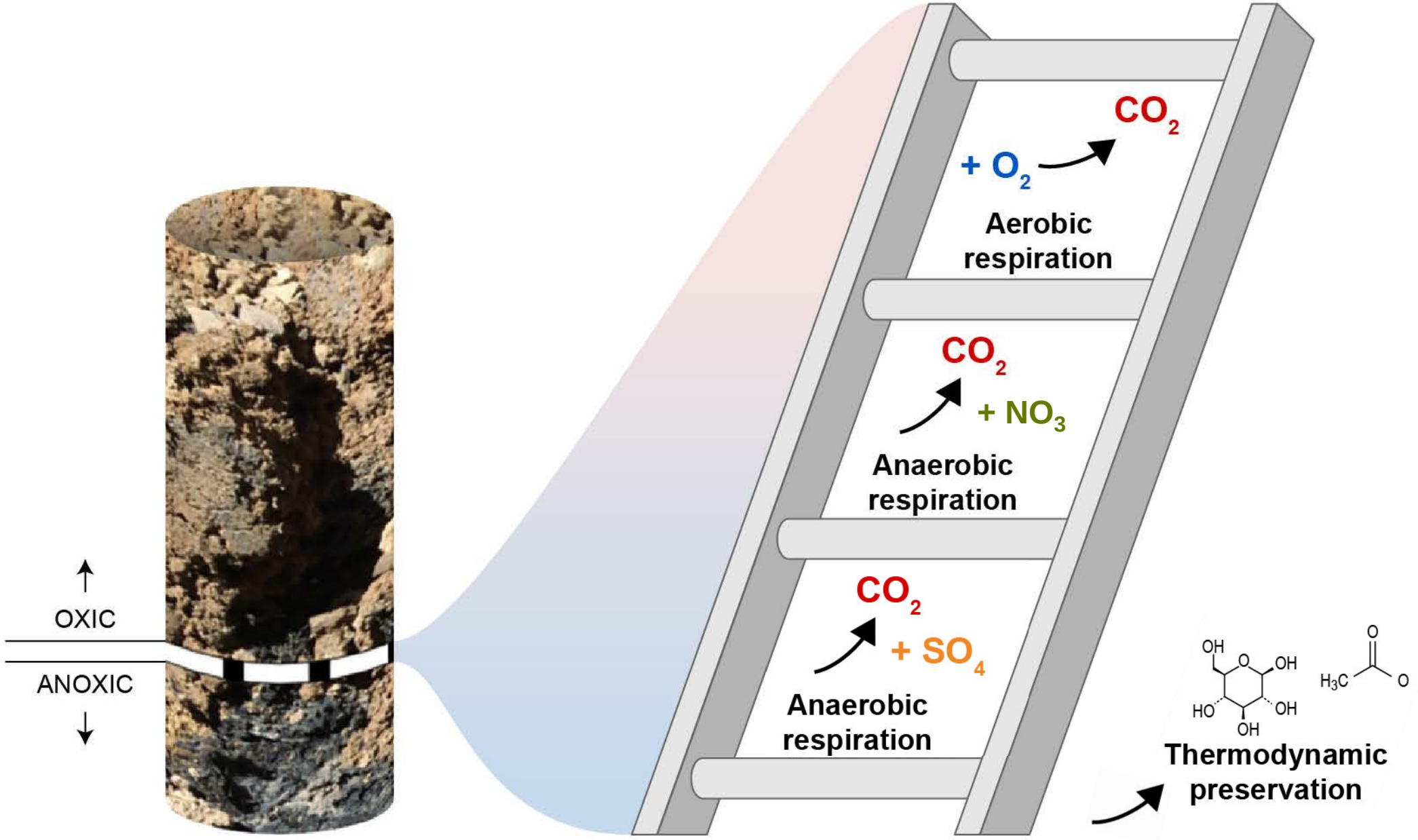


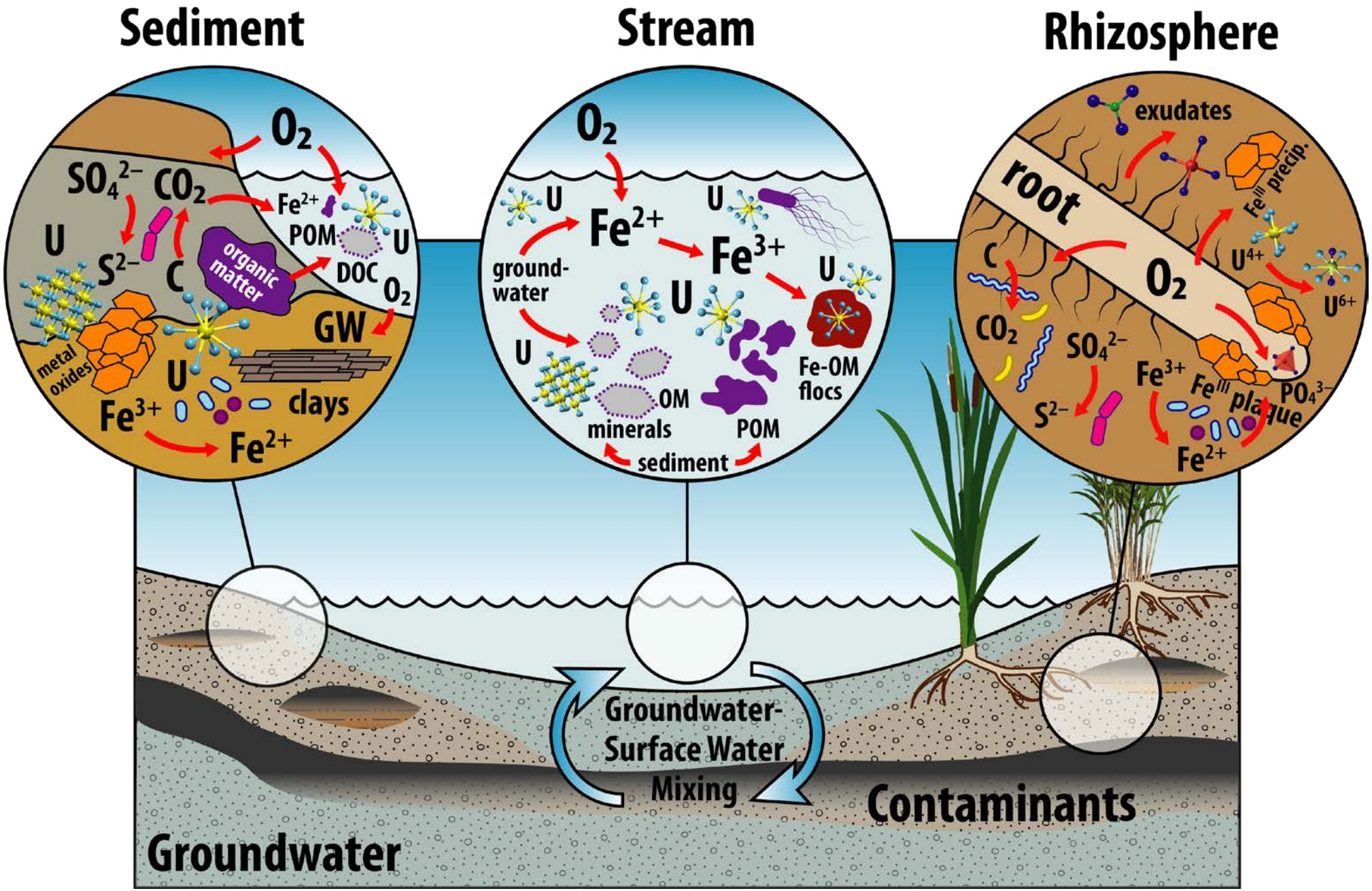
Bailey, V. L., et al. Pore-scale investigation on the response of heterotrophic respiration to moisture conditions in heterogeneous soils. *Biochemistry* (2016) 131:121-134 DOI:10.1007/s10533-016-0270-0



Bailey, V. L., et al. Pore-scale investigation on the response of heterotrophic respiration to moisture conditions in heterogeneous soils. *Biochemistry* (2016) 131:121-134 DOI:10.1007/s10533-016-0270-0

Boye, K., et al. Thermodynamically controlled preservation of organic carbon in floodplains. *Nature Geoscience* (2017), DOI:10.1038/NGEO2940



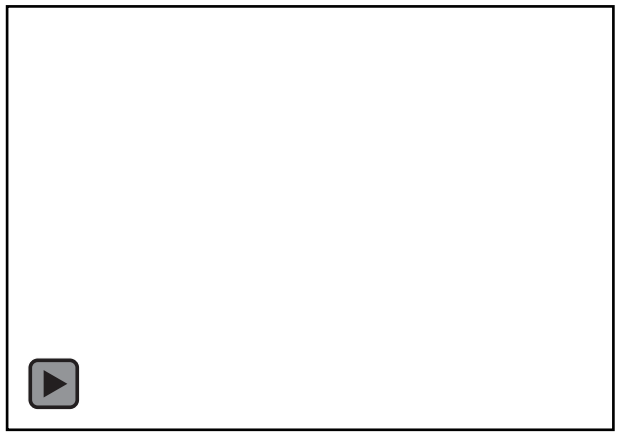

Sediment
Stream
Rhizosphere
O_2
SO_4^{2-}
CO_2
Fe^{2+}
POM
U
DOC
O_2
S^{2-}
C
organic matter
GW
metal oxides
clays
Fe^{3+}
Fe^{2+}
ground-water
Fe^{3+}
Fe-OM flocs
OM
minerals
POM
sediment
exudates
root
Fe^{III} precip.
U^{4+}
U^{6+}
PO_4^{3-}
Fe^{III} plaque
Groundwater-
Surface Water
Mixing
Contaminants
Groundwater

APS

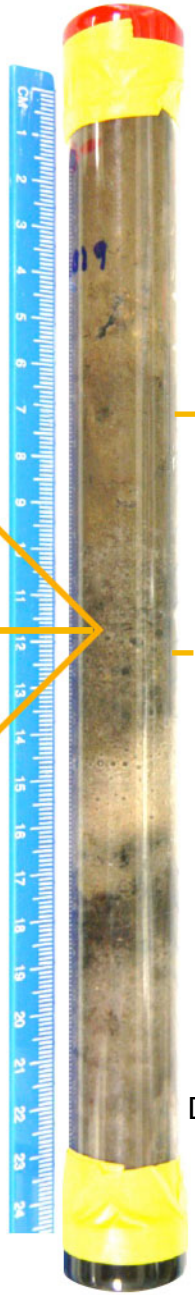


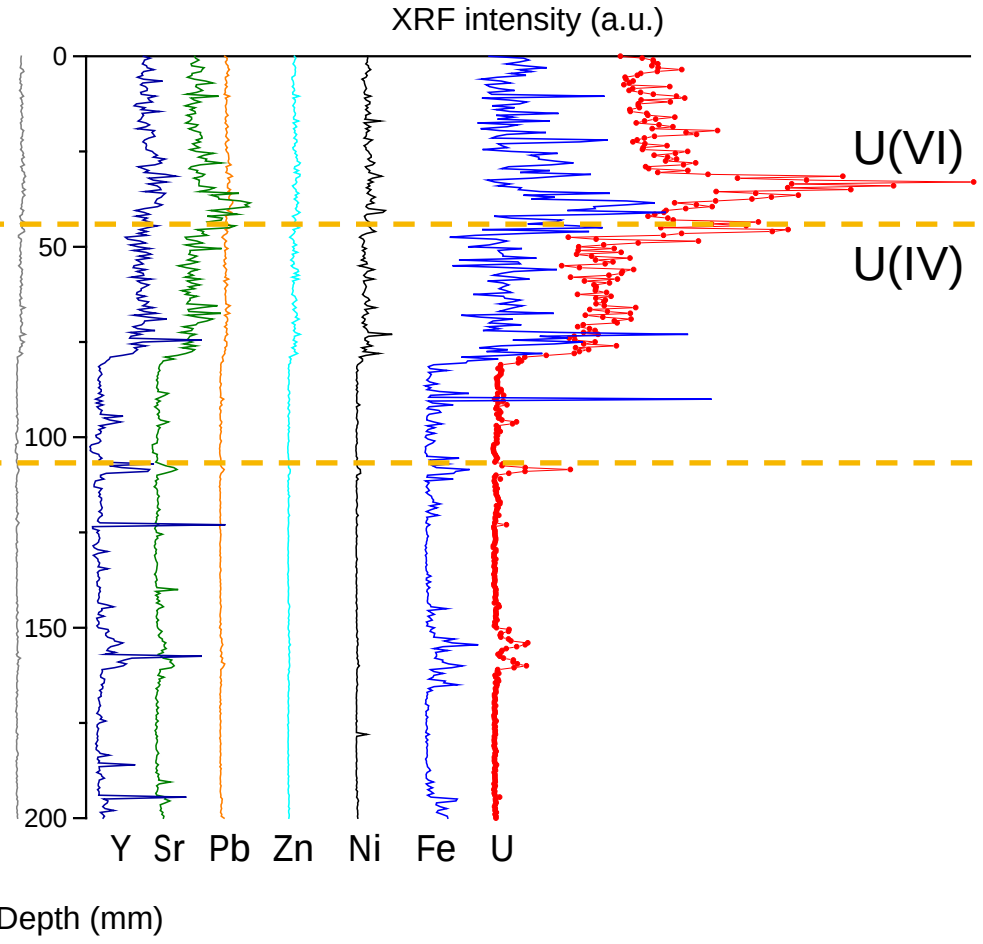
EMSL

C chemistry
Fe chemistry
Particle mineralogy
U associations









U and Fe concentration and chemistry depth profile


FICUS
Facilities Integrating
Collaborations for User Science
SNS
SPALLATION NEUTRON SOURCE
JGI
JOINT GENOME INSTITUTE
DEPARTMENT OF ENERGY
NSLS-II
NATIONAL SYNCHROTRON LIGHT SOURCE II
ALS
ADVANCED LIGHT SOURCE
EMSL
SSRL
Advanced
Photon
Source
NEON
National Ecological Observatory Network

Contact information

DOE, Office of Science, BER: science.osti.gov/ber

DOE, Office of Science, BES: science.osti.gov/bes

APS: Ken Kemner, kemner@anl.gov, aps.anl.gov

ALS: Peter Nico, psnico@lbl.gov, als.lbl.gov/

NSLS II: Ryan Tappero, rtappero@bnl.gov, bnl.gov/ps/

SSRL: John Bargar, bargar@slac.stanford.edu, ssrl.slac.stanford.edu

EMSL: Douglas Mans, douglas.mans@pnnl.gov, emsl.pnnl.gov

Supplemental facility information

At EMSL, we focus on fundamental research to predict molecular and sub-atomic processes underpinning biological and environmental functions.

The Functional and Systems Biology Area focuses on elucidating and harnessing the biochemical pathways that connect gene functions to complex phenotypic responses through a deep understanding of interactions within cells, among cells in communities, and between cellular membrane surfaces and their environment for microbes, fungi, and plants.

The following Integrated Research Platforms enable this science:

Functional Omics—We integrate transcriptomic, proteomic, and metabolomic analyses to uncover biological processes in plant, fungal, and microbial systems.

Molecular Bioimaging—We image the structure and location of proteins and other biomolecules at nanoscale spatial and temporal resolutions.

Cellular Dynamics—We uncover the molecular signals within and between cells as they respond to their environment and other organisms.

The Environmental Transformations and Interactions Area focuses on the mechanistic and predictive understanding of environmental, microbial, plant and ecological processes in above and belowground ecosystems, the atmosphere, and their interfaces.

The following Integrated Research Platforms enable this science:

Biogeochemical Transformation—We investigate how molecular interactions at the Earth's land, water and air interfaces transform and transport nutrients and contaminants within the environment.

Isotope and Chemical Analysis—We track the fate of isotopes and analyze the chemistry of biological and environmental systems.

Molecular Plant Phenotyping—We probe the interaction between genes and the environment to understand, predict and control plant traits.

Douglas Mans
Director of EMSL
douglas.mans@pnnl.gov
(509) 372-4518

Scott Baker
Functional and Systems Biology
scott.baker@pnnl.gov
(509) 372-4759

Nancy Hess
Environmental Transformations and Interactions
nancy.hess@pnnl.gov
(509) 371-6385

EMSL User Services
emsl@pnnl.gov
(509) 371-6003

Opportunities to use EMSL's capabilities are available year-round. Visit emsl.pnnl.gov for more information.

EMSL

www.emsl.pnnl.gov

Advanced Light Source

https://als.lbl.gov/ psnico@lbl.gov hyholman@lbl.gov





The Advanced Light Source (ALS) provides users from around the world access to the brightest beams of soft x-rays, together with hard x-rays and infrared, for scientific research and technology development in a wide range of disciplines including biological and environmental sciences.



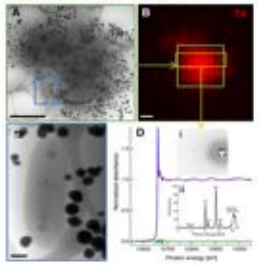
Micro X-Ray Absorption Spectroscopy Beamline 10.3.2

Beamline 10.3.2 provides for the rapid collection of X-ray fluorescence maps and point X-ray absorption and powder diffraction data with ~1-5µm spatial resolution.

Correlative cryogenic spectromicroscopy uncovers selenium bioreduction products



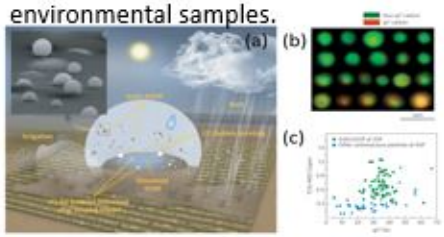
Synchrotron hard X-ray microprobe (SHXM) setup. Cryostage allows correlative cryogenic TEM and microprobe measurements. (A) CLM77K stage. (B) View of the cartridge-grid-holder tongue installed on the mounting frame. (C) View of the stage at beamline 10.3.2 with the sample-transfer tongue inserted. The sample is oriented at 45° to the incident beam, and micro-XRD is performed in transmission mode.



Synchrotron hard X-ray microprobe (SHXM) data sets to precisely determine the distribution, valence state, and structure of selenium in biofilms sampled from a contaminated aquifer near Rifle, CO. Fakra et al. *Environ. Sci. Technol.* **52**, 2018

Scanning Transmission X-ray Microscopy (STXM) Beamlines 5.3.2.2/11.0.2

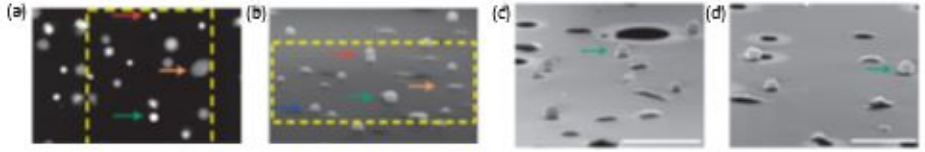
Both beamlines provides for the rapid collection of 3D X-ray absorption data with submicron spatial resolution for quantitative volumetric investigations on chemistry and structure of diverse environmental samples.



Airborne soil organic particles generated by precipitation

a) A schematic illustration of the 'raindrop' mechanism. **b**) Airborne soil organic particles (ASOP) compositional maps with sp2 and non-sp2 carbon shown in red and green, respectively. **c**) Correlation between the total carbon absorption/area equivalent diameter ratio and sp2 carbon content

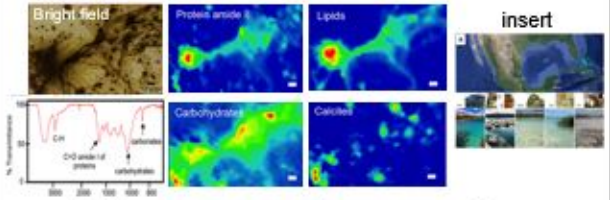
After rain events, sub-micrometer solid particles, with a chemical composition consistent with soil organic matter, contributed up to 60% of atmospheric particles. Chemical imaging and micro-spectroscopy analysis of physico-chemical properties suggest that these Particles may have important impacts on cloud formation and efficiently absorb solar radiation. B. Wang *et al.*, Nat. Geosci. **9**, 433 (2016).



Non-destructive Synchrotron Infrared Spectral imaging

sFTIR Spectromicroscopy at BL 1.4 & BL 5.4

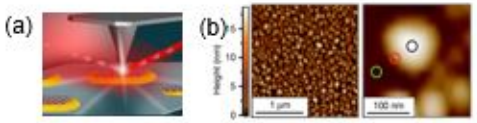
- Spatial/temporal distributions and chemistry of functional groups of molecules
- 2-15 µm focused beam spot & spatial resolution
- Seconds-to-minutes temporal resolution



sFTIR spectromicroscopy images (200 µm by 150 µm) showing the co-localization of microbial biomolecules and minerals in a living microbialite. (insert) sampling locations. Valdespino et al. *Frontier in Microbiology* **9**, 2018.

sFTIR Nano Spectroscopy at BL 2.4 & BL 5.4

- Spatial distributions and chemistry of functional groups of molecules
- 2-10 nm focused beam spot



(a) A schematic of the synchrotron infrared nano-spectroscopy (SINS) setup that combines synchrotron IR with scattering scanning near-field optical microscope (s-SNOM., (b) **SINS can be applied to hard and soft matter, including biominerals, proteins, bacteria, fungi, and other biomaterials to identify and measure local surface chemistry.** (Wu, et al. *Nature* , **541**, 2017)

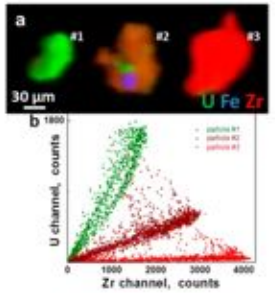
Stanford Synchrotron Radiation Lightsource

http://www-ssrl.slac.stanford.edu





Contact: John Bargar; bargar@slac.stanford.edu

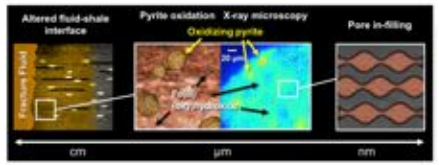


Co-localized U and Zr in Chernobyl particles
Batuk et al. *Environ. Sci. Technol.* **49**, 6474 (2015)

MicroXAS imaging

- Spatial distributions and chemistry of trace and major elements
- 2 µm focused beam spot
- Oxidation states and chemical forms of elements

BL 2-3	2 µm resolution	5.0-24 keV
BL10-2	10-200 µm resolution	5.0-24 keV
BL 14-3	2 µm resolution	2.1- 5 keV

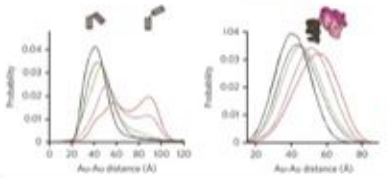


Pyrite oxidation in shale exposed to fracture fluid
Jew et al. *Energy and Fuels* (2017)
Harrison *Appl. Geochem.* (2017)

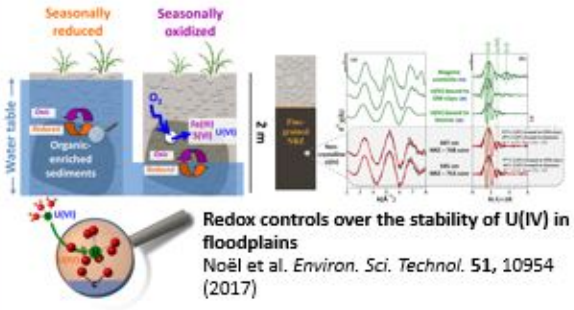
Small angle x-ray scattering (SAXS)

- Size distributions of nanoscale
- 3D details of macromolecules, nanoparticles, surfaces, pores.

BL 1-5	4.6-16.0 keV	2.7-0.8 Å	Material SAXS
BL 4-2	6.0-17.0 keV	2.2-0.7 Å	Bio-SAXS



Structural ensembles of RNA and RNA:Protein complexes
Shi et al. *Nat. Chem. Biol.* **12**, 146 (2016)



Redox controls over the stability of U(IV) in floodplains
Noël et al. *Environ. Sci. Technol.* **51**, 10954 (2017)

X-ray absorption spectroscopy (XAS)

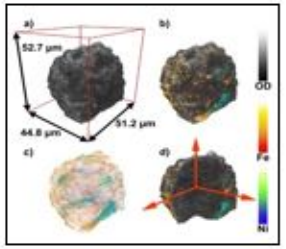
- Electronic structure (oxidation states)
- Molecular structure
- Interfaces, aqueous solutions, biological materials, and amorphous materials.

BL 4-1	5.0 – 38 keV
BL 4-3	2.4 – 14 keV
BL 7-3	5.4 – 32 keV (bioXAS)
BL 9-3	5.0 – 30 keV (bioXAS)
BL 11-2	5.0 – 38 keV
BL14-3	2.1 – 5 keV

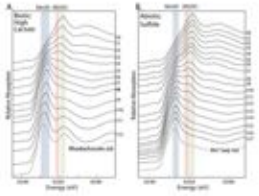
Transmission X-ray microscopy (TXM)

- 2D imaging & 3D nano-tomography
- Soils, soil-microbe aggregates, and rocks
- Energy specific imaging.

BL 2-2	0.6 µm resolution
BL 6-2	30 nm resolution 5-14 keV



Metals Incorporation of a Single Catalyst Particle
Meirer, *et al.*, J. Am. Chem. Soc. **137**, 102 (2015)



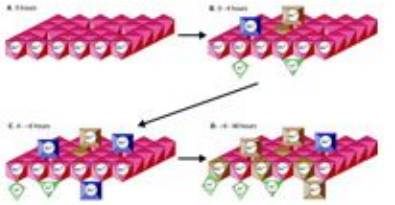
Measuring Real-time Biological and Abiotic Manganese Oxide Reduction
Johnson et al. *Environ. Sci. Technol.* **50**, 8 (2016)

User proposal deadlines:
X-ray techniques: May 1, Aug 1, Nov 1
Macromolecular crystallography: Jul 1, Dec 1, Apr 1

X-ray powder diffraction (XRD)

- Identification of crystalline materials
- Coherent scattering domain size
- Unit cell dimensions

BL 2-1	4-14.5 keV
BL11-3	12.7 keV (1.0 Å)



Lafferty et al. *Environ. Sci. Technol.* **44**, 8467 (2015)

SSRL Structural Molecular Biology program supported by DOE BER and NIH National Institute of General Medical Sciences http://ssrl.slac.stanford.edu/smb/index.html

Advanced Photon Source

Argonne NATIONAL LABORATORY

http://www.aps.anl.gov

kemner@anl.gov





Over 20 beam lines devote some of their scientific effort to Hydrobiogeochemistry.

All beamlines can receive actinide samples and sectors 10, 13, and 20 have nearby laboratory facilities.

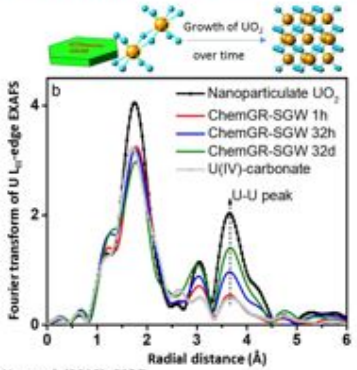
The Advanced Photon Source offers annual courses on synchrotron science open to everyone from students to senior scientists.

- **Sector 2:** sub-micron X-ray microscopy, ptychography, tomography
- **Sector 5:** X-ray powder diffraction (XRD), small angle x-ray scattering (SAXS), X-ray absorption spectroscopy (XAS)
- **Sector 9:** "tender" XAS, hard x-ray nanoprobe- 30 nm resolution (transmission, fluorescence, ptychography)
- **Sector 10: (MRCAT/EnviroCAT):** low-concentration XAS, fluorescence microscopy, spectromicroscopy
- **Sector 11:** Powder XRD
- **Sector 12:** XAS, SAXS
- **Sector 13 (GSECARS):** X-ray absorption spectroscopy, X-ray tomography, microscopy, spectromicroscopy, XRD microscopy
- **Sector 18 (BIO-CAT):** SAXS
- **Sector 20 (PNCCAT):** X-ray absorption spectroscopy, X-ray Raman, fluorescence microscopy, spectromicroscopy
- **Sector 32:** Transmission x-ray microscopy, transmission nanotomography

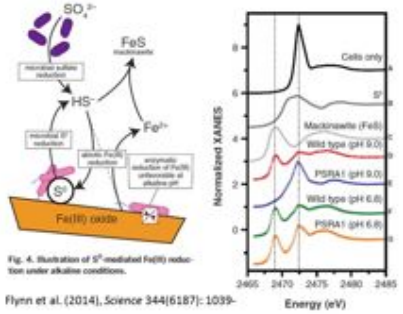
Run Cycle	Run Dates	Proposal Submission Deadline
2019-3	Oct - Dec 2019	July 5, 2019
2020-1	Feb - Apr 2020	Oct 25, 2019
2020-2	June - Aug 2020	Feb 28, 2020

Fully equipped geomicrobiology laboratories available to BER funded Argonne SFA collaborators within half mile of the synchrotron

X-ray absorption spectroscopy (XAS): Element specific local atomic structure

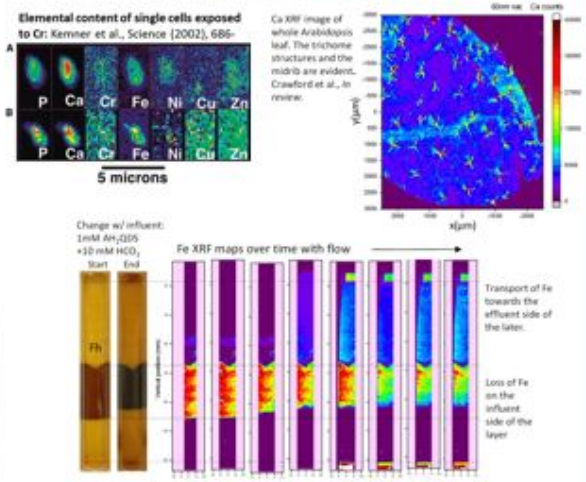


Yan et al. (2018), *ES&T*

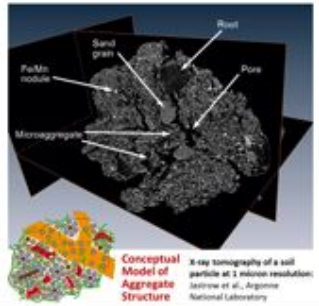


Flynn et al. (2014), *Science* 344(6187): 1039-

X-ray fluorescence microscopy: 2D elemental mapping



X-ray tomography: 3D elemental or pore structure imaging



X-ray tomography of a soil particle at 1 micron resolution: Jastrow et al., Argonne National Laboratory



National Synchrotron Light Source-II (NSLS-II)

Contact: Ryan Tappero; rtappero@bnl.gov



NSLS-II is a state-of-the-art 3 GeV electron storage ring. The facility offers scientific and industrial researchers an array of beamlines with X-ray, ultraviolet, and infrared light to enable breakthroughs in critical areas such as energy security, chemistry and catalysis, biosystems, earth and environment.



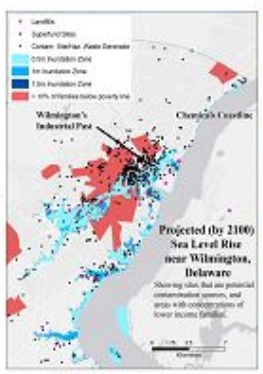
- **3-ID:** Hard X-ray Nanoprobe (X-ray microscopy, ptychography, tomography)
- **4-BM:** X-ray Fluorescence Microprobe (micro-EXAFS, -XRD, -XRF, tomography)
- **5-ID:** Sub-micron Resolution X-ray microscopy
- **6-BM:** "Hard" X-ray absorption spectroscopy
- **7-BM:** Quick X-ray absorption spectroscopy
- **8-BM:** "Tender" X-ray absorption spectroscopy and imaging
- **8-ID:** Inner-shell spectroscopy
- **18-ID:** Transmission X-ray Microscopy
- **28-ID:** High-energy powder diffraction and pair distribution function analysis

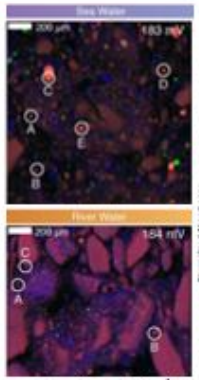
Run Cycle	Run Dates	Proposal Submission Deadline
Fall 2019	Sep - Dec 2019	May 31, 2019
Spring 2020	Jan - Apr 2020	Sep 30, 2019
Summer 2020	May - Aug 2020	Jan 31, 2020

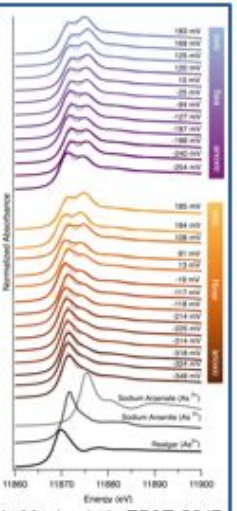
To Become a NSLS-II User: www.bnl.gov/ps/userguide
Beam time is available at no charge to researchers and it is granted through a peer-review proposal process.

Impacts of sea level rise on arsenic mobility in coastal soils

- Global mean sea levels are expected to rise by 0.8 m by 2100
- Potential impacts to large populations and contaminated sites along coastline
- Investigate soil-As speciation under two inundation scenarios- sea or river water- as a function of Eh







LeMonte et al., ES&T, 2017

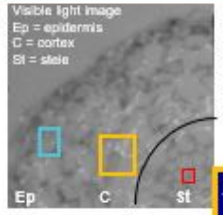
RESULTS:

- Anoxic conditions resulted in reduced As species (more for river water inundation)
- As release into solution was 2x higher for river water than for sea water!
- Ions in sea water (e.g., SO_4) stabilized As-Fe solid phase and salt inhibited DMRB (dissimilatory metal reducing bacteria); solid phase speciation in this soil was dominated by As-bearing Fe oxyhydroxides rather than sorption complexes
- River water did not inhibit reductive dissolution or dissimilatory Fe reduction

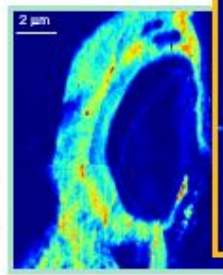
Plant uptake and trophic transfer of engineered nanomaterials (NMs)

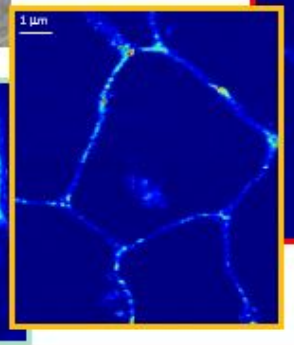
- Agronomic application of NM (phytonanotechnology) has potential to alter conventional plant production systems
- Controlled release of agrochemicals (e.g., fertilizers, pesticides and herbicides) and target-specific delivery of biomolecules (e.g., nucleotides, proteins and activators)
- Investigate effect of surface charge on the uptake and translocation of NM in plants

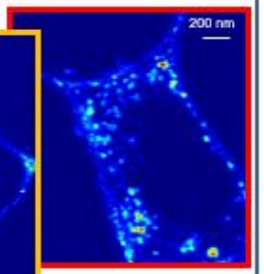
RESULTS (for CeO_2(-) NMs):



Left: CeO_2 NM accumulation at rhizoplane and epidermis
Middle: Apoplastic (between cells) transport of CeO_2 in root cortex!
Right: Endocytosis may be mechanism of CeO_2 uptake into stele from root apoplasm







Li et al., Environ. Sci. Nano, 2019

Upcoming events and announcements



virtual meeting

nsls2cfnusersmeeting.bnl.gov/default.aspx?year=2020



National School on Neutron and X-ray Scattering

June 13 - 27, virtual meeting

Visit the website:

anl.gov/education/national-school-on-neutron-and-xray-scattering



APS/IIT Summer XAFS School

July 12 - 17, virtual meeting

Visit the website:

aps.anl.gov/APS-Reminders/2020-07-12/apsiit-summer-xafs-school

ADVANCED LIGHT SOURCE
2020 User Meeting

ALS
ADVANCED LIGHT SOURCE



August 25 - August 28, virtual meeting

als.lbl.gov/user-meeting/



SSRL EXAFS Summer School

Late August - Early September

Visit the website for updates:

ssrl.slac.stanford.edu/conferences/workshops/exafs2018/index.html

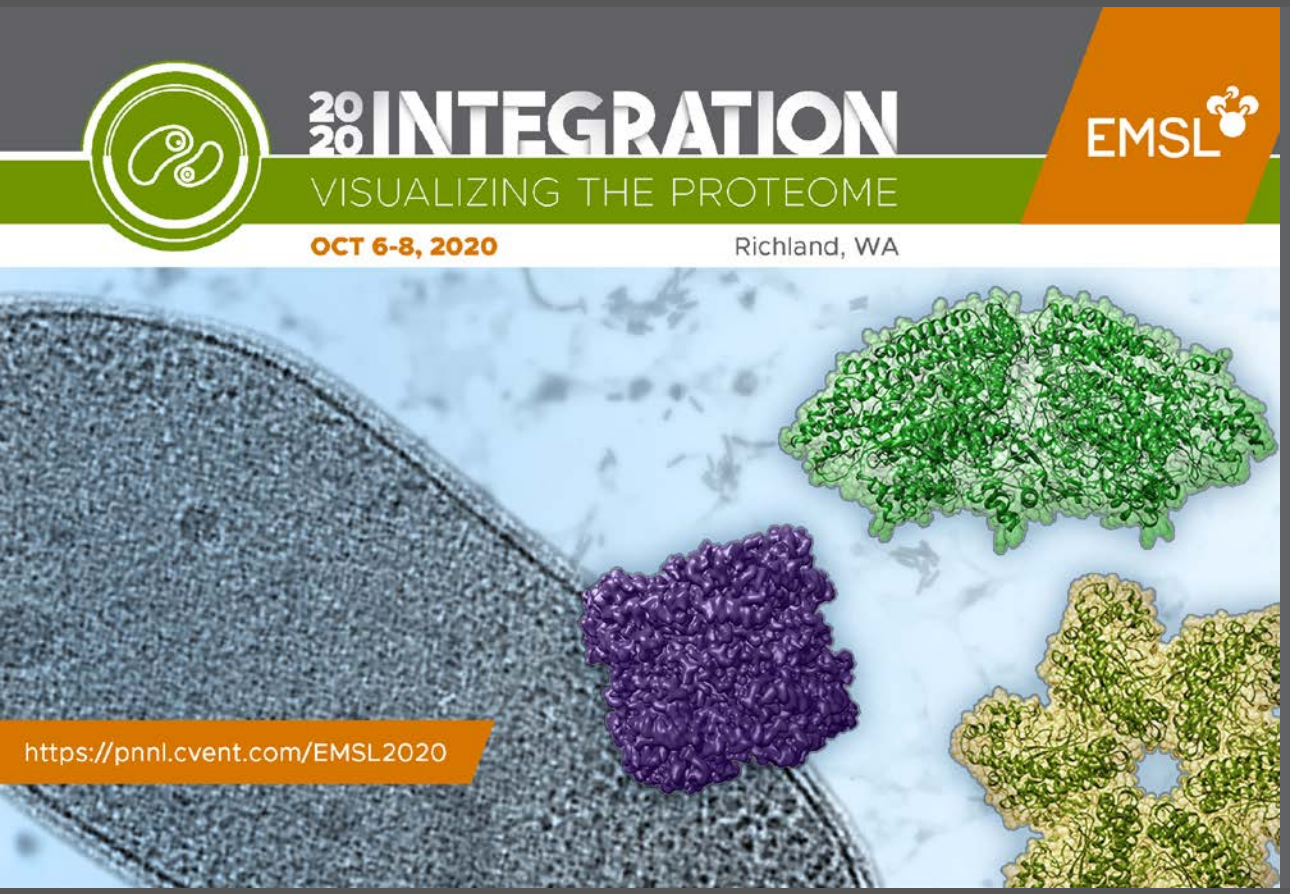


SSRL/LCLS Annual Users' Conference & Workshop

September 30 – October 2, virtual conference

Visit the website:

conf.slac.stanford.edu/ssrl-lcls-2020/



2020 INTEGRATION

Confirmed Speakers

Thomas Braun, Center for Cellular Imaging and Nano Analytics

Joseph Gault, University of Oxford

Albert J.R. Heck, Utrecht University

Ryan Kelly, Brigham Young University

Cheryl Kerfeld, Michigan State University

Dmitry Lyumkis, University of California, San Diego

James Prell, University of Oregon

Steve Reichow, Portland State University

Elizabeth Wright, University of Wisconsin-Madison

Sessions

- Pushing the frontier of structural biology
- Understanding subcellular compartmentalization and ultrastructure
- Annotating protein function and modifications via native and top-down proteomics
- Revealing cellular complexity and dynamics with single to few-cell proteomics
- Advancing cellular models and protein design with high-performance computing

// Questions? emslusrmtg@pnnl.gov

pnnl.cvent.com/EMSL2020





SSRL and ALS plan to host a session at AGU 2020 "Synchrotron Environmental Science-VIII"

JOIN US!

Visit the AGU website for updates:

agu.org/Fall-Meeting







EMSL, ARM, and JGI plan to co-host the AGU 2020 session “Collaborate with a DOE User Facility: Learn About Available Expertise and Resources, Open Call Opportunities, and Important Tips for Submitting Successful Proposals”

JOIN US!

Visit the AGU website for updates:

agu.org/Fall-Meeting



The deadline for this year's summer school has passed but be on the lookout for next year's opportunity. Postdoc and early career researchers are welcome to apply. More information about the 2021 Summer School will be available in December.

pnnl.cvent.com/2020SummerSchool