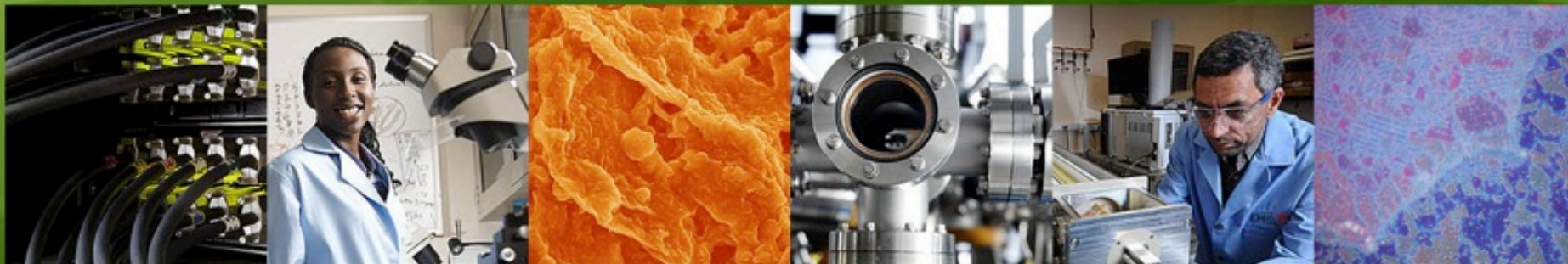


Advanced Magnetic Resonance Capabilities at the Environmental Molecular Sciences Laboratory (EMSL) to Accelerate Studies of Biological, Environmental, and Energy Systems

Karl T. Mueller
Acting Chief Science and Technology Officer
Physical and Chemical Sciences Directorate
Pacific Northwest National Laboratory



EMSL is a national scientific user facility



William R. Wiley's Vision:

An innovative multipurpose user facility providing *“synergism between the physical, mathematical, and life sciences.”*



- **Funded by DOE Office of Science's Office of Biological and Environmental Research (BER)**
- **120 staff members**
- **Opened in 1997 in Richland, WA - Washington state's wine country**

EMSL's mission is to lead molecular-level discoveries for BER and DOE that translate to predictive understanding and accelerated solutions for:



BIOLOGY

Rational design of biological systems

ENVIRONMENT

Understand the role of natural or anthropogenic inputs on climate and subsurface systems

ENERGY

Efficient energy storage and conversion

EMSL provides unique suites of instrumentation to integrate with high-performance computing



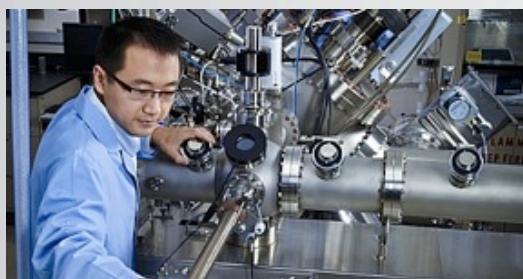
Mass Spectrometry



Cell Isolation & Systems Analysis



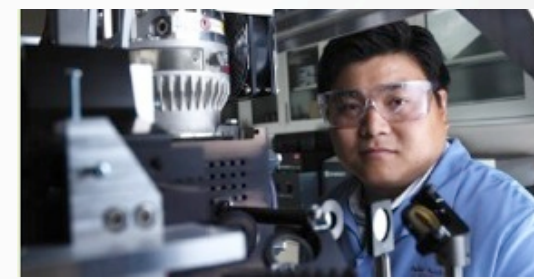
Molecular Science Computing



Deposition & Microfabrication



Spectroscopy & Diffraction



Instrument Development Lab



Subsurface Flow & Transport



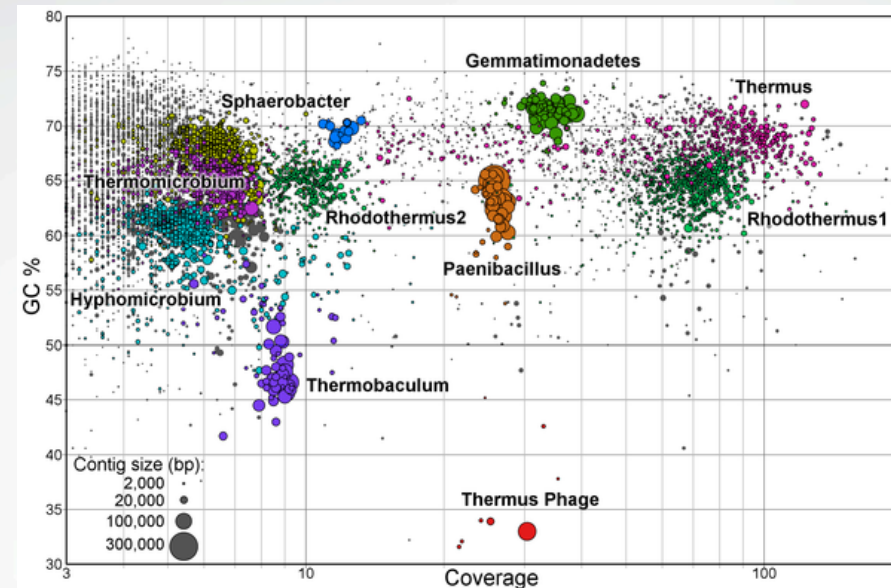
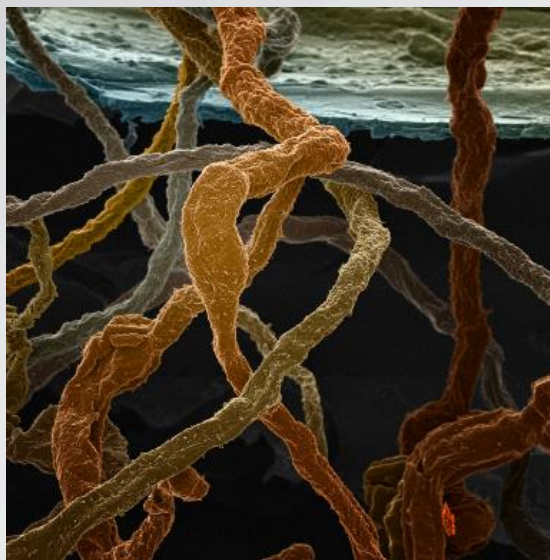
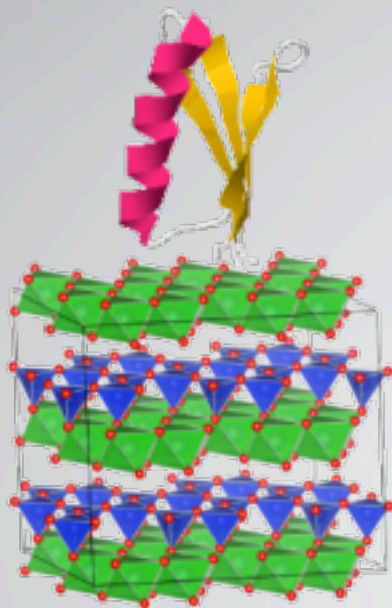
NMR & EPR



Microscopy

EMSL has a wide range of experience with DOE project management

EMSL delivers impact for users



FY	Users	On Site	Publications	Proposals*
			(as of Oct.)	
2013	750	515	411	359
2014	726	406	461	379
2015	715	430	446	379

*Proposal acceptance rate is 38-40%

Magnetic resonance at EMSL

- Suite of low to ultra-high field NMR spectrometers (100 – 850 MHz)
 - ▶ Ultra-low temperature capabilities
 - ▶ Imaging and bioreactor capabilities
 - ▶ Cryoprobes
 - ▶ Liquids operation
 - ▶ Metabolomics
 - ▶ Solid-state NMR
- Two EPR systems
- **Uniqueness tied to ability to develop specialized probes**
 - ▶ *In situ* NMR for batteries and catalysts
 - Continuous Flow MAS
 - High Pressure and Temperature
 - ▶ Radioactive Sample Containment



Magnetic resonance at EMSL delivers impact for users



UC San Diego



Smithsonian



Alfred University



HALLIBURTON



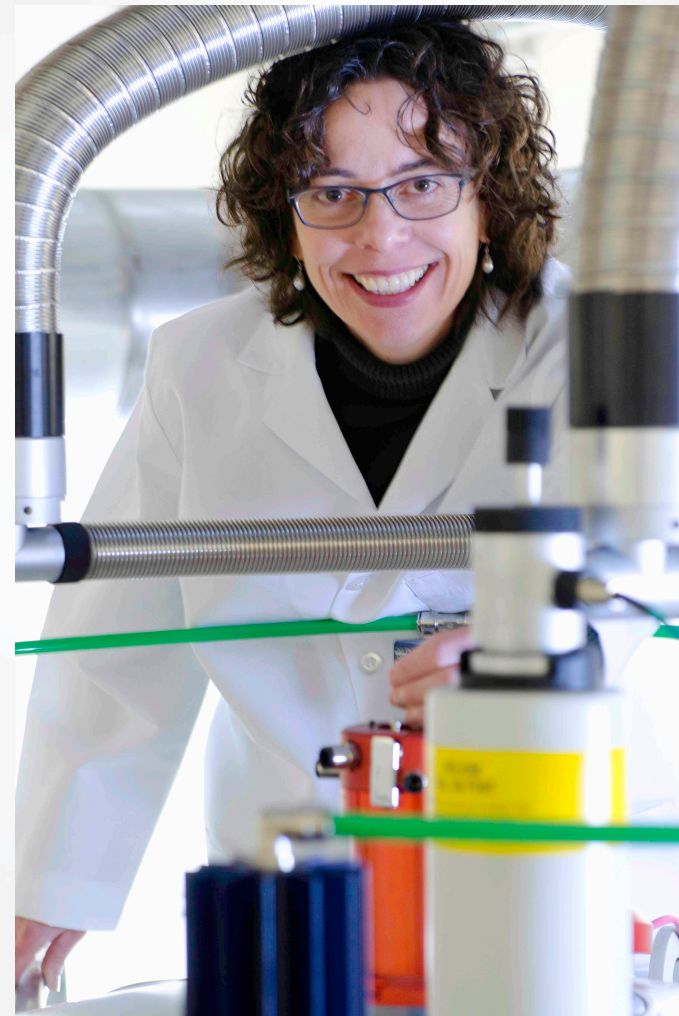
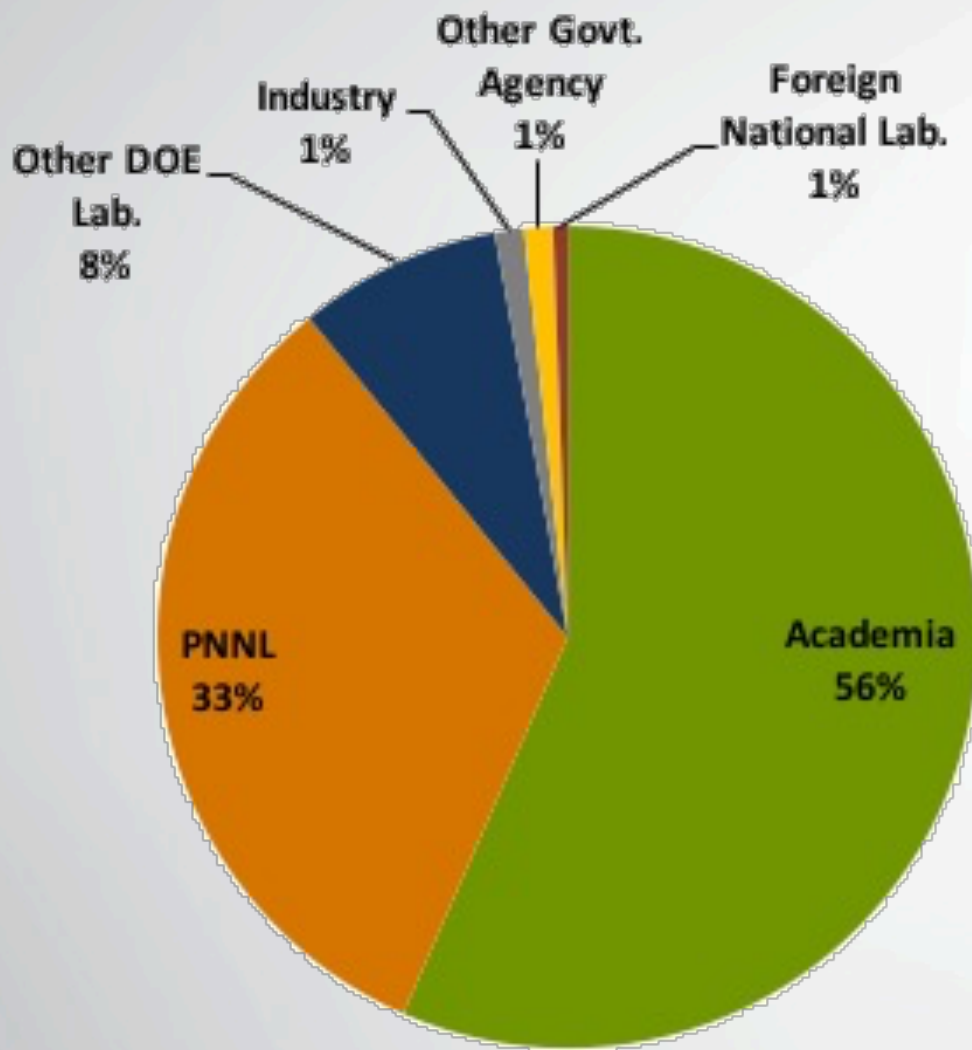
University of Colorado Denver



Magnetic resonance at EMSL delivers impact for users



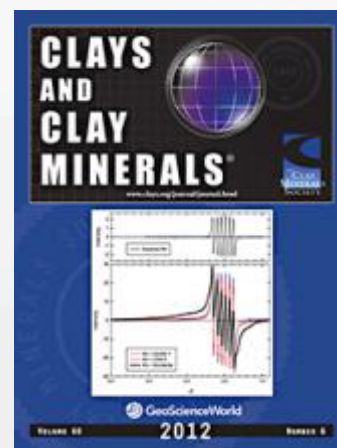
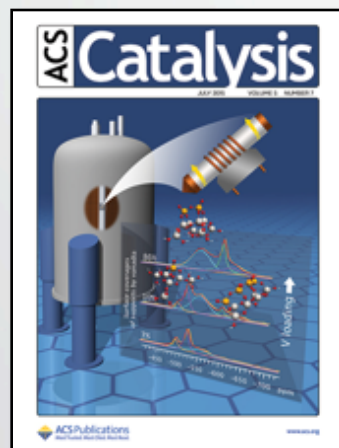
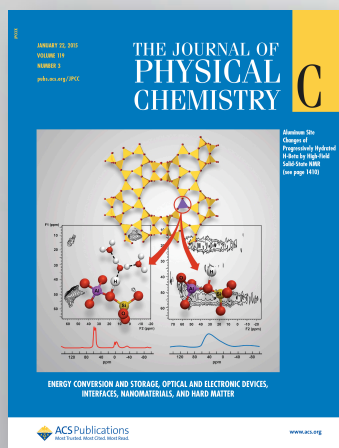
User Institution Types
(65 distinct FY13-15)



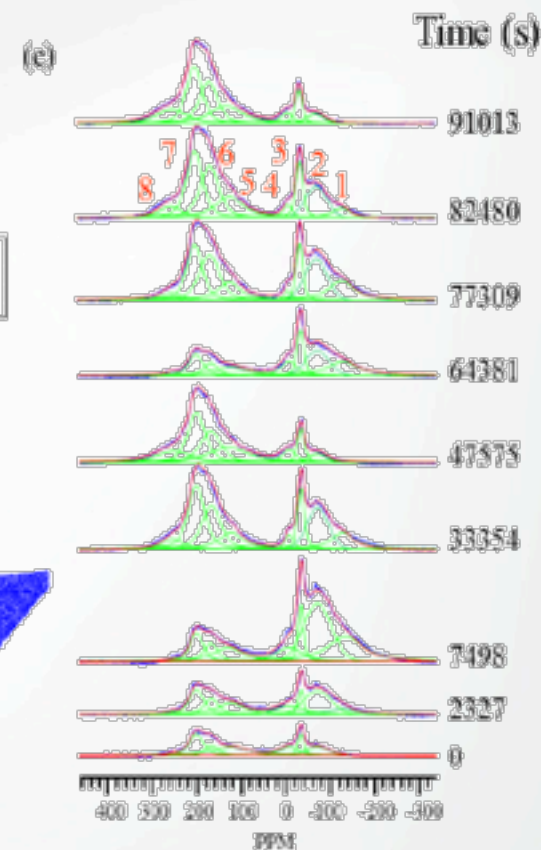
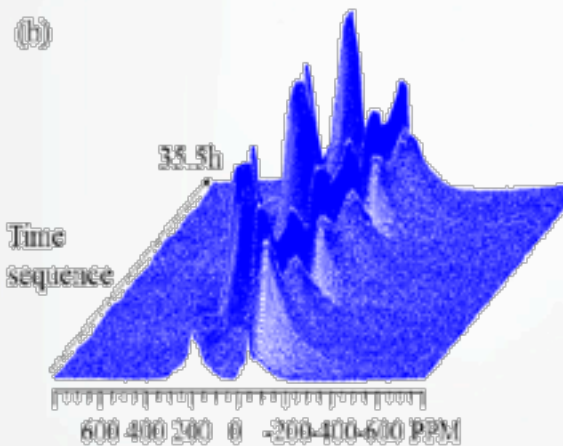
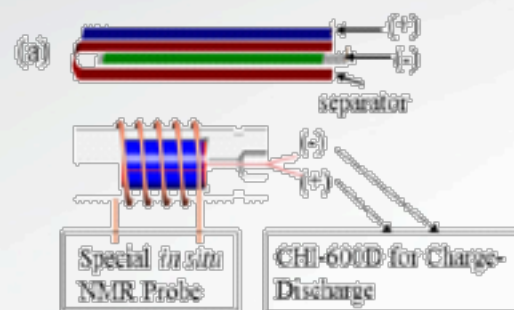
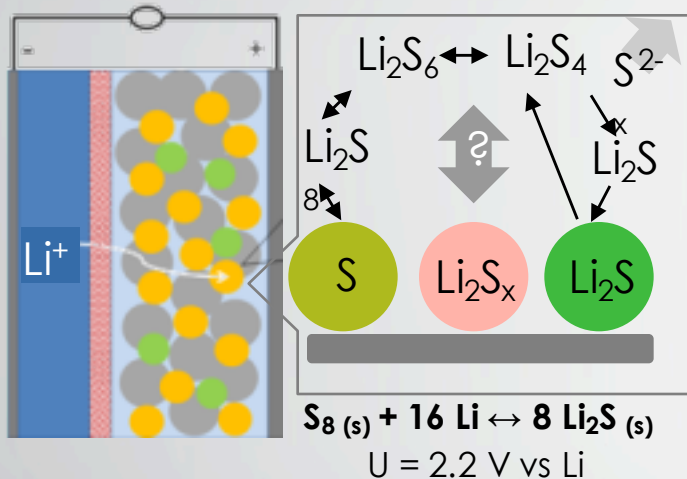
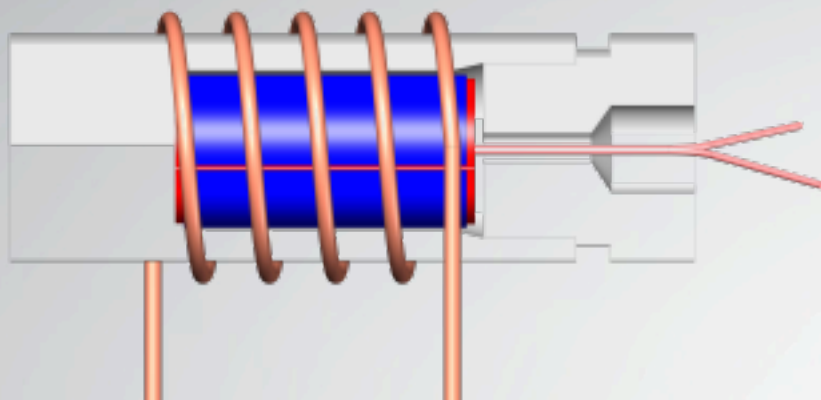
Magnetic resonance at EMSL delivers impact for users



FY	Projects	Users	Publications	"Top Ten"
2013	50	80	111	71
2014	49	77	119	71
2015	65	93	115	56
Distinct/total (over 3 years)	127	175	345	198



In situ NMR for battery systems at EMSL

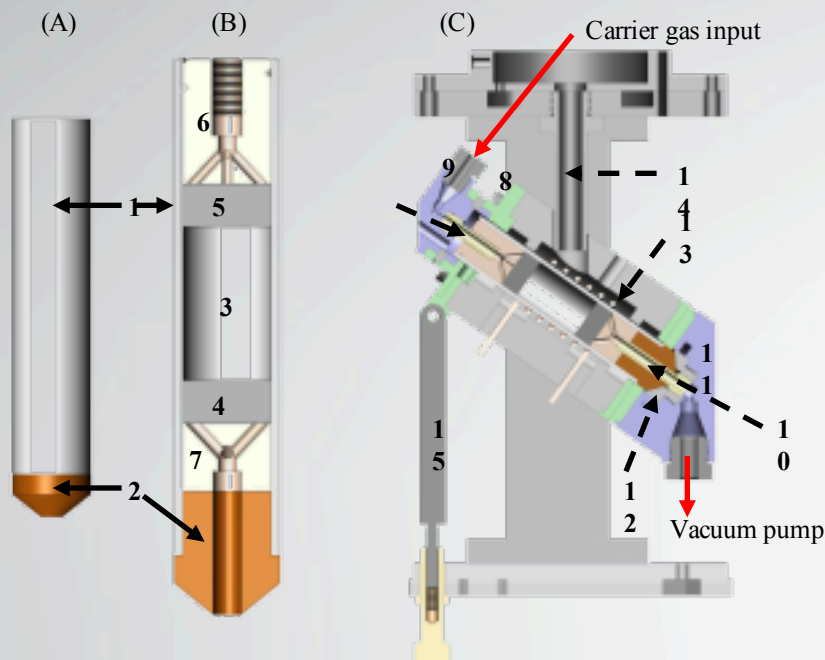


Goals:

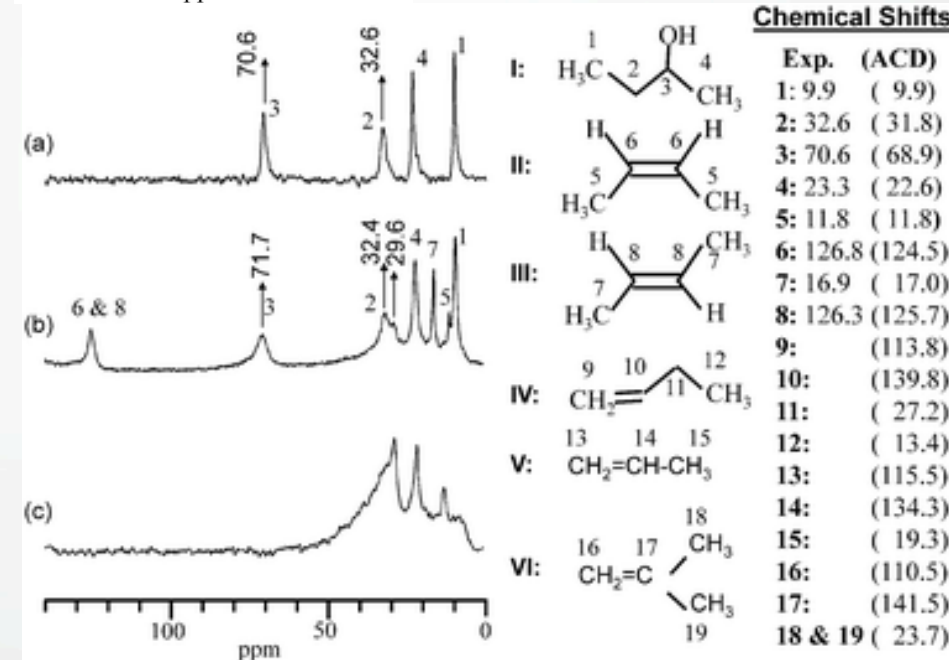
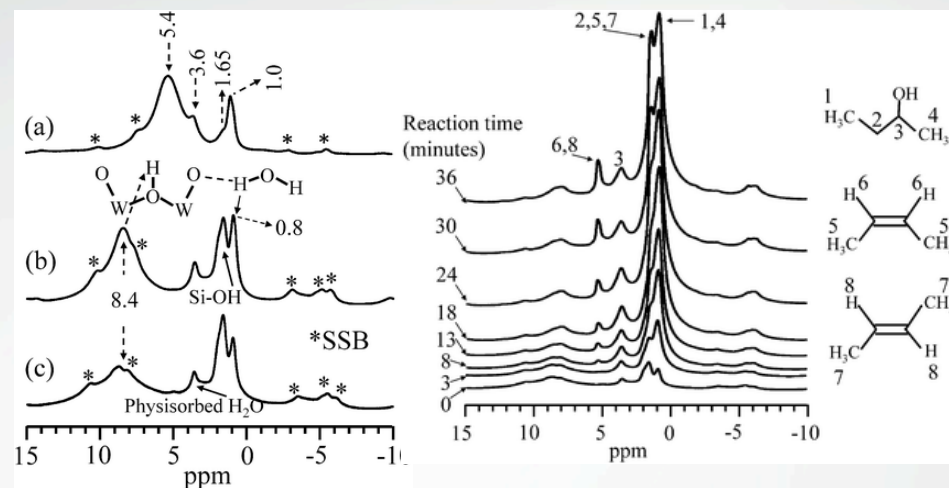
Gain a fundamental understanding of polysulfide dissolution process in organic solvents and electrolytes.

Elucidate the molecular structure & stability of dissolved polysulfide species.

Continuous flow MAS NMR at EMSL

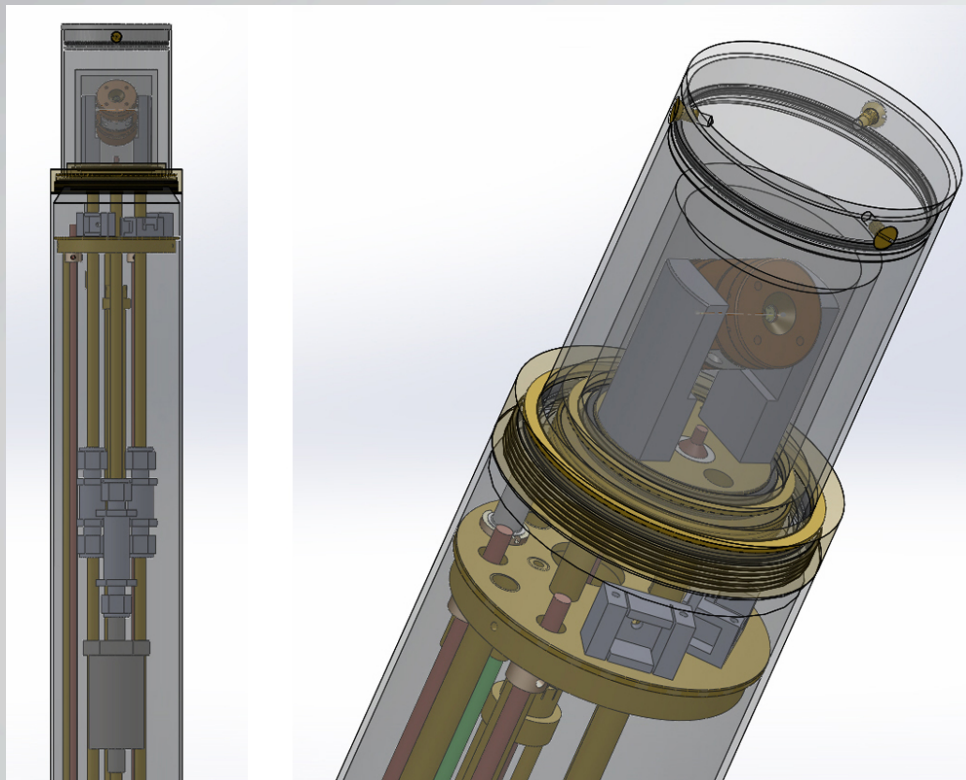


New, high-temp. design (to 650 °C) based on EMSL probe technology.



JZ Hu, JA Sears, HS Mehta, JJ Ford, JH Kwak, K Zhu, Y Wang, J Liu, DW Hoyt, and CHF Peden. 2012. "A Large Sample Volume Magic Angle Spinning Nuclear Magnetic Resonance Probe for In Situ Investigations with Constant Flow of Reactants." *Physical Chemistry Chemical Physics* 14(7):2137-2143.

Magnetic resonance of radionuclides at EMSL



SEALED MAGIC ANGLE SPINNING NUCLEAR MAGNETIC RESONANCE PROBE AND PROCESS FOR SPECTROSCOPY OF HAZARDOUS SAMPLES

Application number: 20140167756

Filed: December 13, 2013

Issued: June 19, 2014

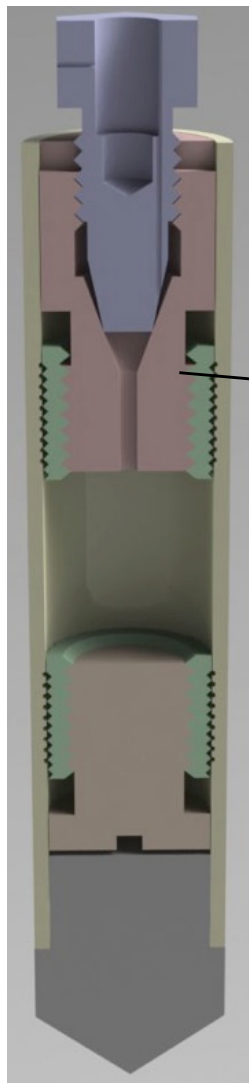
Assignee: BATTELLE MEMORIAL INSTITUTE

Inventors: Herman M. Cho, Nancy M. Washton,
Karl T. Mueller, Jesse A. Sears, JR., Mark R.
Townsend, James R. Ewing

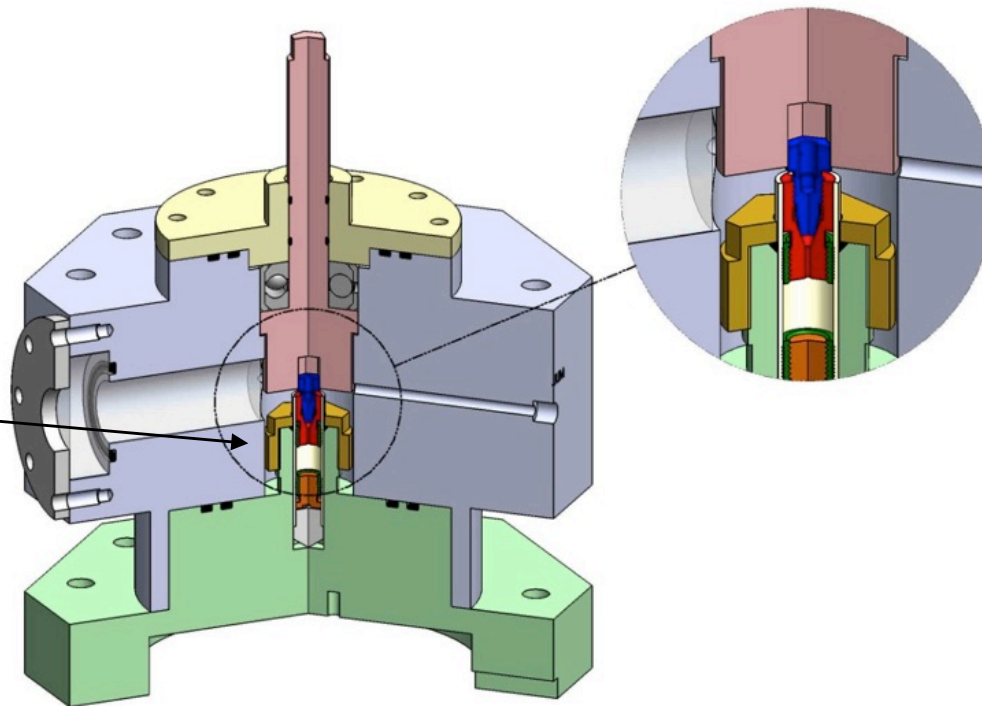
- Probe includes double containment
- Modular design in case of failure
- Hermetically-sealed with full air filtration



EMSL technical accomplishment: *in situ* MAS NMR at high T and P



(A) High pressure sample cell



(B) High pressure sample loading/ reaction chamber

U.S. Patent No. 8,692,548 "Devices and Process for High-Pressure Magic Angle Spinning Nuclear Magnetic Resonance" issued April 8, 2014, **DWHoyt**, JA. Sears, RVF Turcu, KM Rosso, and JZ Hu

D.W. Hoyt, R.V.F. Turcu, J.A. Sears, K.M. Rosso, S.D. Burton, A.R. Felmy, J.Z. Hu, J. Magn. Reson, 212, 378-385, (2011)

R.V.F. Turcu, D.W. Hoyt, K.M. Rosso, J.A. Sears, A.R. Felmy, J.Z. Hu, J. Magn. Reson, 226,64-69,(2012)

Experiments can be carried out *in situ*

- Up to **200 bar**
- Up to **80 °C**
- scCO_2 and scCH_4

Rotor sizes of 7.5 and 6.0 mm

Using standard MAS probes

HP rotor prototypes (underway) will allow
-higher temperatures
-higher magnetic field

EMSL offers users a variety of proposal options to support user community



Users request access via a peer-reviewed proposal process

- Online 4-page proposal
- External peer review

Use of EMSL capabilities *at no cost* for non-proprietary research

Three forms of user projects:

- **Science Theme**

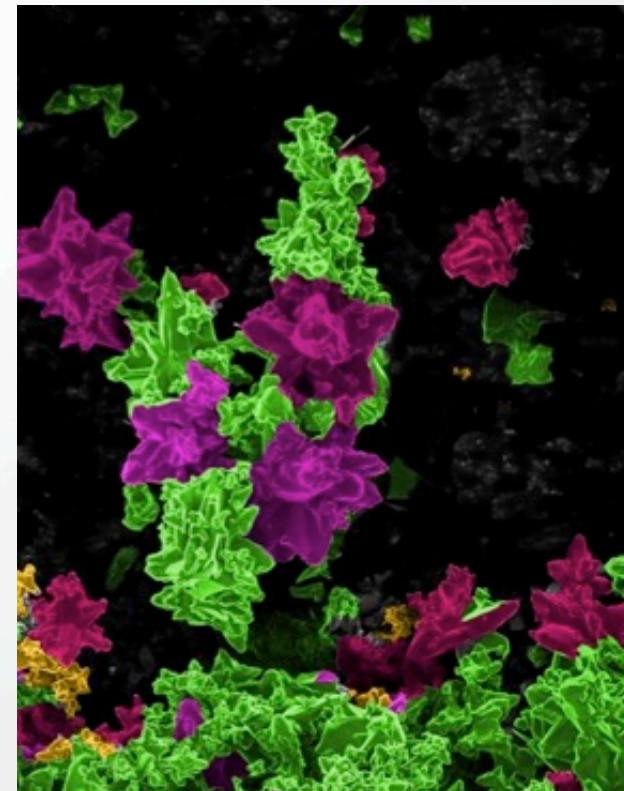
- ▶ Access up to three years (3rd by invitation)
- ▶ Submitted via proposal call

- **General**

- ▶ Access variable (up to 1 year)
- ▶ Submitted at any time

- **Scientific Partner**

- ▶ Cost-share to build capability
- ▶ Access negotiated
- ▶ Can be submitted any time



EMSL management and operations supports the user community



- Dedicated user portal for proposal submission, reporting, scheduling, training, etc.

EMSL User Portal

Welcome, Karl Mueller. [LOG OUT](#)

[Home](#) [Proposals](#) [Publications](#) [Reviews](#) [User Info](#) [Training](#) [Schedule Experiments](#)

Scheduling Experiments for Proposal #48846

Proposal Dates: 10/01/2015 - 09/30/2017

* = Read Only Resource

Calendar Legend:
■ Out-of-Service ■ Unavailable ▨ Partially Available
■ My Requests ■ My Bookings

Resources	November 2015																														
	1 Sun	2 Mon	3 Tue	4 Wed	5 Thu	6 Fri	7 Sat	8 Sun	9 Mon	10 Tue	11 Wed	12 Thu	13 Fri	14 Sat	15 Sun	16 Mon	17 Tue	18 Wed	19 Thu	20 Fri	21 Sat	22 Sun	23 Mon	24 Tue	25 Wed	26 Thu	27 Fri	28 Sat	29 Sun	30 Mon	
Electron Spectrometer: XPS High Resolution, Imaging																															
X-ray Diffraction: General Purpose																															
Electron Microscope: Transmission, Environmental																															
* Electron Microscope: Dual FIB/SEM, Environmental (FEI Quanta)																															
* Electron Microscope: Dual FIB/SEM (FEI Helios)																															
Microscope: Helium Ion																															
Electron Microscope: Transmission, Scanning with Electron Energy Loss (EELS)																															

November 2015 | December 2015 | January 2016 | February 2016 | March 2016 | April 2016 | May 2016 | June 2016 | July 2016 | August 2016

To request a resource: click-and-drag to highlight a timeframe, then click the "Request Resource" button.

EMSL Environmental Molecular Sciences Laboratory
Contact: [User Services](#)
[Security & Privacy](#)

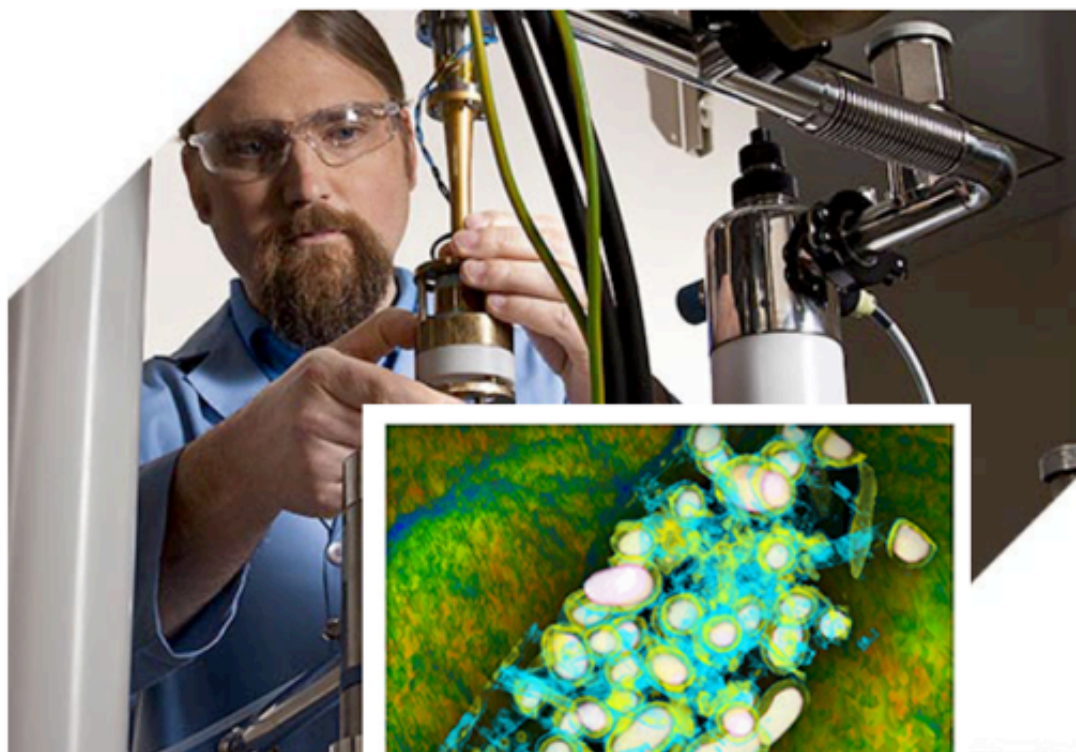
Version: 2.15.1

For more information, visit the EMSL web pages

[▶ FAQ](#)

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Kathryn Mireles, EMSL User
Washington State University

www.emsl.pnnl.gov



Questions?



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