

Off-gas Xenon Detection and Management in Support of Molten Salt Reactors

Dr. Hunter Andrews and Dr. Praveen Thallapally Oak Ridge National Laboratory and Pacific Northwest National Laboratory, USA

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Meet the Presenters

Dr. Hunter Andrews is an early career researcher in the Isotope Applications Research Group within the Radioisotopes Science and Technology Division, Oak Ridge National Laboratory. Having been at ORNL for the past 3 years, his research focus revolves around the development of in-situ, online monitoring tools for complex environments. His main expertise lies in optical spectroscopy, particularly laser-induced breakdown spectroscopy (LIBS), a rapid form of spectroscopy capable of elemental analysis regardless of sample form. Other research interests include chemometrics, machine learning, mass spectrometry, spectroelectrochemistry, and neutron imaging. He received his PhD in Mechanical and Nuclear Engineering from Virginia Commonwealth University, College of Engineering, USA.

Dr. PraveenThallapally is a chief scientist at PNNL for the past 17 years. He and his team developed a series of novel and transformational applications of porous organic and hybrid (metal organic frameworks, covalent organic frameworks, etc.) nanomaterials and membranes for separation of volatile radionuclides released from nuclear reprocessing and advanced reactors. He published large number of publications (>150) and several patents (>5) focused on materials and membranes for noble gas separation, carbon capture, iodine removal, adsorption chillers/heat pump and sensing. He is tailoring these advanced functional materials as surface acoustic wave sensors to detect and monitor the toxic gases. Other research interests include the development of "porous liquids" for applications in energy storage, catalysis, extraction of critical minerals from unconventional sources. His work was featured on large number of internal and external press. He received his PhD in Chemistry from the University of Hyderabad, India.







Email: andrewshb@ornl.gov; praveen.thallapally@pnnl.gov



Metal Organic Frameworks for Noble Gas Capture **Praveen Thallapally** Pacific Northwest National Laboratory

Driving Factors

Why

- U.S. EPA 40 CFR 190 and NRC requires volatile radio nuclides (¹⁴C, ³H, ¹³¹I, ¹³³Xe and ⁸⁵Kr) must be captured and sequestered to meet the regulation
- Noble gas capture is the most difficult to capture as they are inert by definition
- Potential economic incentive if captured

Major sources of emissions:

- Regular operation of nuclear power plant
- Advanced reactors
- Reprocessing of spend nuclear fuel, nuclear accidents, medical isotope facilities





Thallapally and co-workers Nature Communications volume 11, Article number: 3103 (2020)

Applications of Noble Gases

Sortune Business Insights reported "The noble gases market size stood at USD 40.34 billion in 2020 and continue to grow

> High purity of Xe

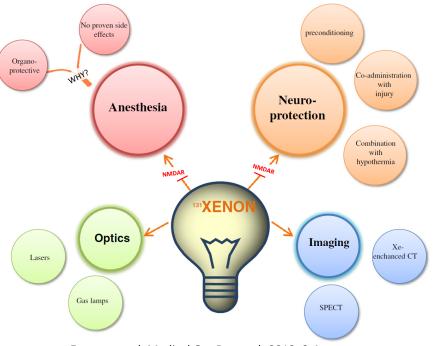
□Space Industry – Propellant

- NASA Xe-ion-thrusters is projected to use approximately 16 metric tones of Xe, for a cost ranging between \$81–100 million at today's market price
- Medical Anesthesia, Imaging
 - Approximately 313.4 million major surgical procedures were performed around the world in each year.
 - Due to the supply issues and cost of Xe makes it prohibitive to use. Could open-up huge market
- Semiconductor Plasmas in deposition and etch
 - Demand for chips increase so as noble gases (~multi billion-dollar industry)
- Building and Automotive lighting

> High purity of Kr

- Buildings Window insulation
- Automotive Head lights, Laser lights
- Geoscience to detect the age of ancient ground water





Esencan et al. Medical Gas Research 2013, 3:4

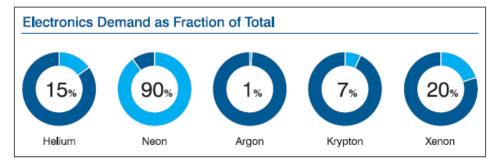


Figure 7. Electronics application demand makes up widely varying fractions of the total market for each of the rare gases.

Advanced Reactors

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□ A gram of actinide material, large MSR produce a kilogram of noble gas per day (~19 L) at reactor operating temperature and atmospheric pressure

Recovery and Better Noble Gas Management

- Provide cleaner gas feed for noble gas recovery
- Enables bulk value-added product
- MSR plant operation enable growing noble gas needs
- Cost of operations and capital reduced with each noble gas recovered
- Sequential removal of volatile gases improves noble gas recovery processes

Xe Kr Ar



http://periodictable.com/Elements/054/index.html



to start commissioning SIPF gas centrifuges (GC) to produce enriched Xenon-129. Xenon-129 is the newest isotope to show its effectiveness in polarized lung imaging; there is no U.S. production capability. This isotope has also garnered the interest of the medical community in monitoring lung function and damage from infectious disease such as COVID-19. The FY 2022

Source: DOE isotope production and research

Sequential Recovery of Noble gases

Current Technologies and Alternatives

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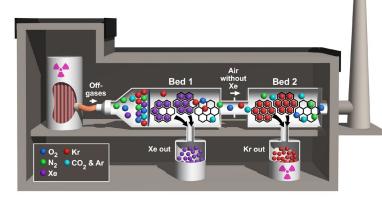
Current Technology

- Cryogenic removal of Xe and Kr
 - Projected to be expensive
 - Potential for O_3 accumulation
 - Hazardous conditions

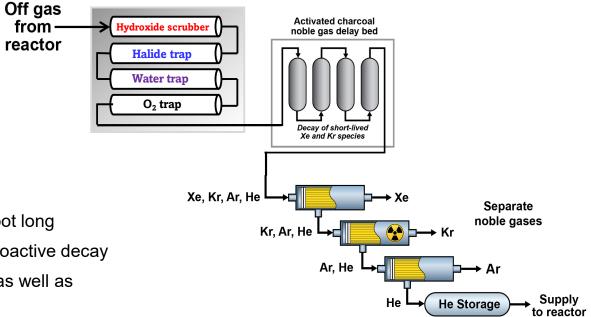
Charcoal delay beds (MSR)

- Requires 4-5 charcoal tanks with 6 9 foot in diameter and 50 foot long
- o Fire hazard: Presence of oxygen and heat production due to radioactive decay
- Oxygen needs to be removed upfront from cryogenic distillation as well as charcoal beds

Xe and Kr







1) Too complex, 2) Large footprint, 3) Costly, 4) Hazardous and safety issues

MOFs as Alternate Technology

- Higher capacity and selectivity represents significant cost reduction compared to cryogenic and charcoal beds
- Smaller size columns, reduced footprint and no fire hazard
- Remove Xe (non-radioactive) and Kr in separate steps at near RT
 - Recover process costs by selling Xe?
- o Remove Kr in single step

Thallapally and co-workers Compact and Modular Integrated Off-Gas System and Sensors." Invention Disclosure e-IDR 18117

Metal Organic Frameworks

Shape

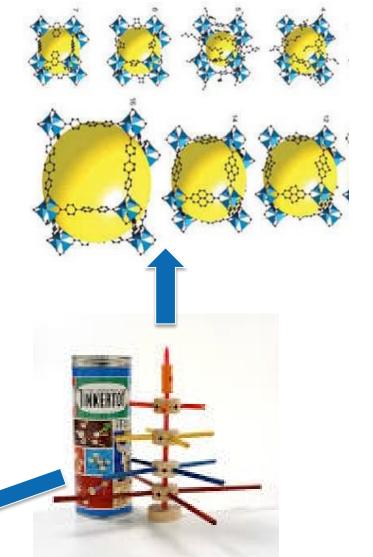
	Zeolites/Charcoal	MOFs	
Safety	Potential bed fires (charcoal)	NA	
Туре	Inorganic/Organic	Hybrid	
Diversity	Limited	Infinite	
Pore Size	Fixed	Fine-tunable 0.3 to 10 nm	
Surface Area	Up to 1000 m ² /g	Up to 8000 m²/g	
Capacity*	Moderate	High	
Selectivity	Need to remove CO_2 , and Water	Not required (CaSDB) Yes for water (for some MOFs)	
Cycle	200	>2000 (PNNL) (water adsorption n desorption)	
Stability	Up to 1 x 10 ⁷ RAD	1.75MGy PNNL and SNL Study Recent literature shows even higher stability	
Cost	Varies	Varies;	



- MOFs with higher adsorption capacity, and selectivity represents significant cost reduction compared to existing technology
- Smaller-size columns and reduced footprint

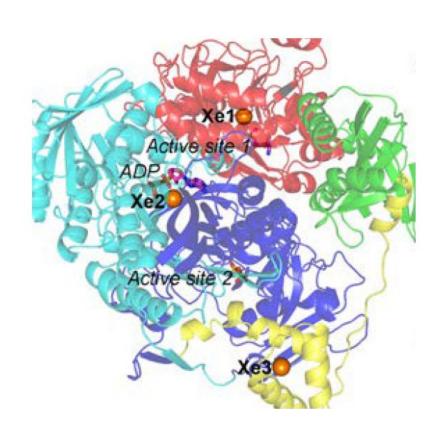
Surface functionality

Dimensions



Inspiration from Proteins and Biological Molecules

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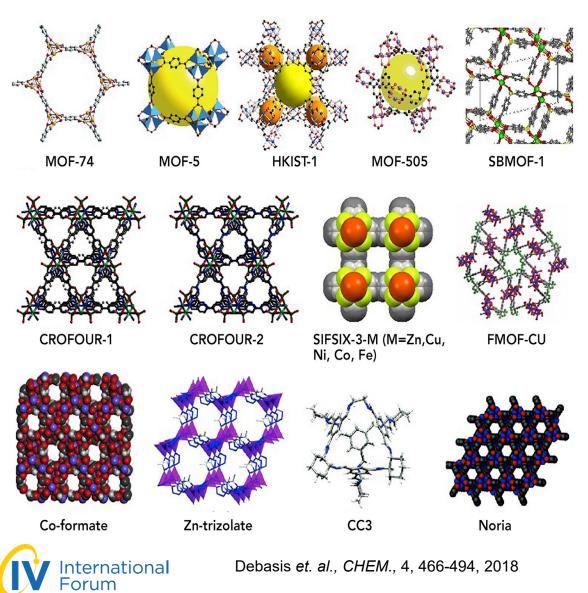
- Structural biology can provide some insight into how and where noble gases bind to proteins.
- In 1965, Shoenborn et. al., reported the x-ray structure of the complex formed between myoglobin and xenon.
- Crystallographic studies under Xe and Kr pressure provided valuable experimental insight on the importance of hydrophobic cavities, channels, and other structural voids.
- Among many adsorption sites, the prominent cites for Xe and Kr are hydrophobic cavities with pore size ranging from 0.4 to 0.6 nm in size

Design and develop synthetic hosts

Adsorbents Studied

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Expertise | Collaboration | Excellence



- Pore diameter close to the KD of Xe/Kr
 - Chen et. al., *Nat. Mat.,* 2014
 - Debasis et. al., Nat. Comm., 2016
 - Elsaidi et. al., Chem. Eur J., 2017
 - Elsaidi et. al., Nat. Comm., 2020

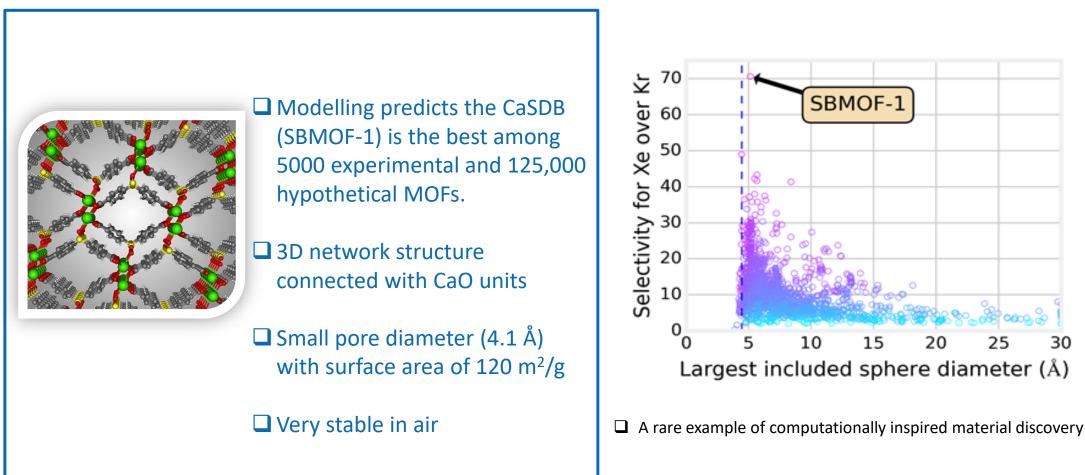
High density of open metal sites

- Thallapally et. al., Chem. Commun, 2012
- Liu et. al., Chem. Commun, 2014
- Ghose et. al., J. Phys. Chem C., 2016

Polar functional groups within pore surface

- Chen et. al., *J. Am. Chem. Soc.*, 2015
- Elsaidi et. al., Angew Chem. Int. Ed., 2016
- Temperature switching selectivity
 - Fernandez et al., J. Am. Chem. Soc., 2012
- High surface area and Impregnation with silver Nanoparticles
 - Liu et. al., Chem. Commun., 2013
 - Feng et. al., J. Am. Chem. Soc. 2016
 - Elsaidi et. al., Chem. Sci., 2017

Rare example of computationally inspired material discovery

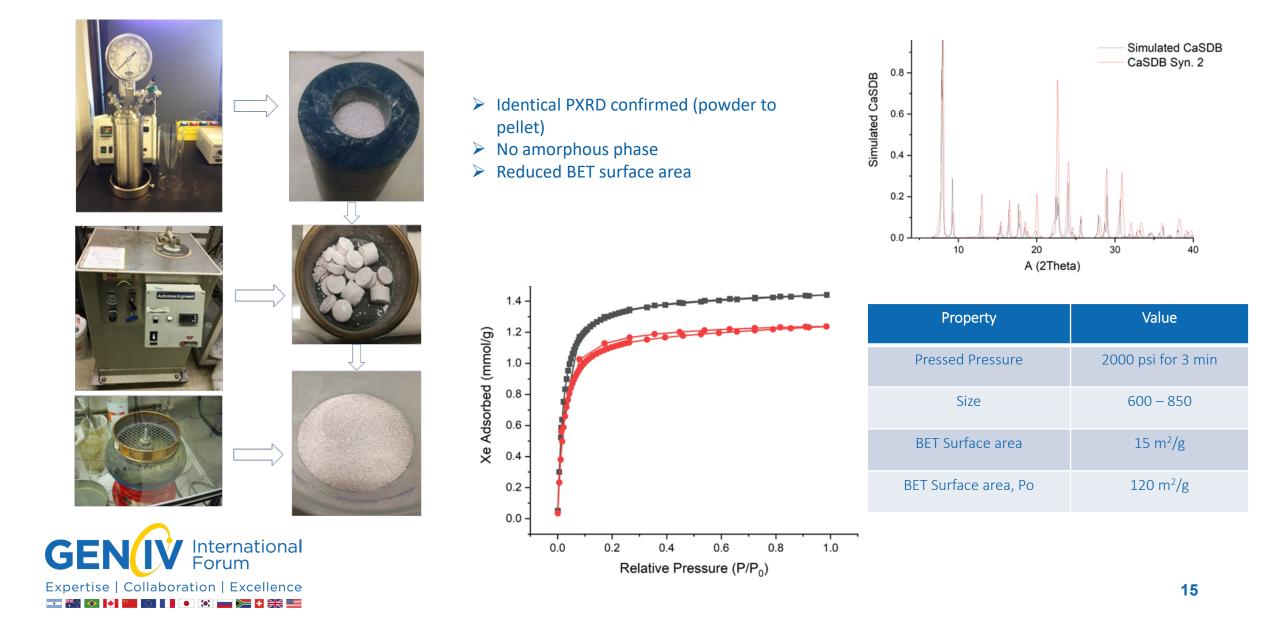


Thallapally, PK., Vienna et. al., USPTO WO/2017/218346A1

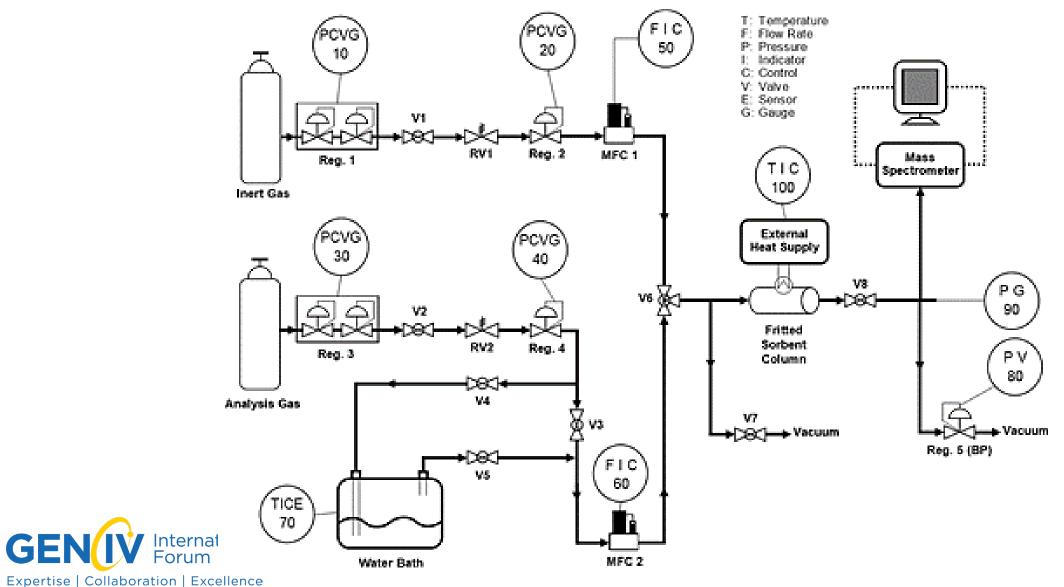


Thallapally, Ali Z. Riley, BJ., Paviet, P., Matyas, J., Vienna, J., Compact and Modular Integrated Off-Gas System and Sensors." Invention Disclosure e-IDR 18117 Banerjee, D, Thallapally, PK, Kunapuli R., McGrail, BP, Liu J et al., Surface acoustic wave sensors for refrigerant leak detection., USPTO WO2021/041359 A1

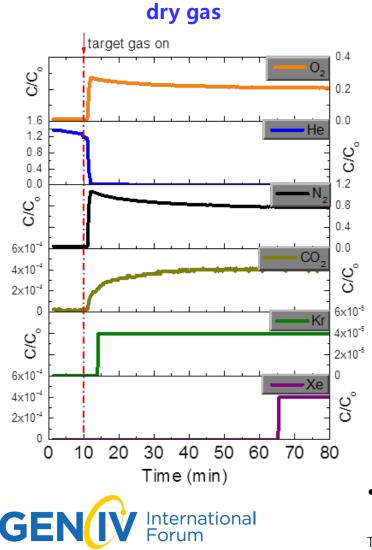
MOF Synthesized at PNNL



Breakthrough Measurements Apparatus



Single Column Breakthrough Experiments



ise | Collaboration | Excellence

Conditions

- Air = 78% N₂, 21% O₂, 0.9% Ar, 0.03% CO₂, 1300 ppm Xe, 130 ppm Kr
- Flow rate = 20 cm³/min
- T = 25 °C (298K)
- MOF = CaSDB

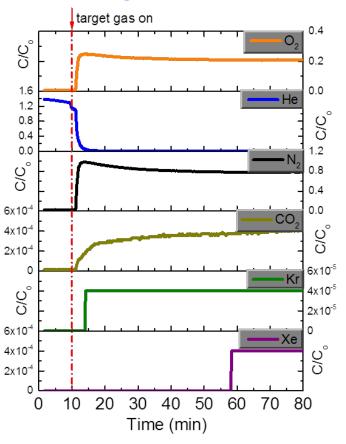
➢ Results

- Xe capacity = 30 mmol/kg vs 8 mmol/kg (NiMOF) and 22 mmol/kg (CC3)
- >95% of the Xe captured from air
- Xe/Kr (selectivity) = 15

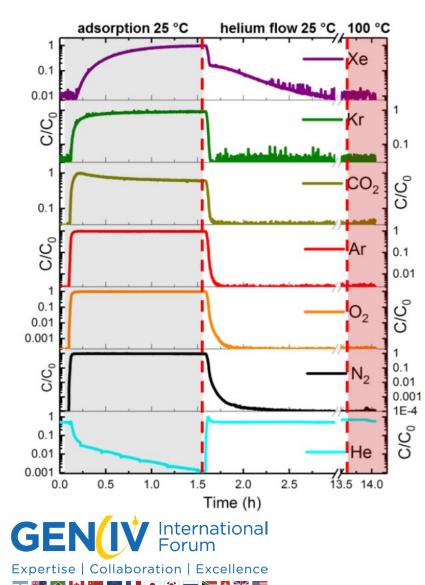
CaSDB falls in the optimal pore size and shape, making it stand out among other MOFs

Thallapally, Vienna et. al., USPTO WO/2017/218346A1 Banerjee *et. al., Nature Communications*, 2016

Wet gas (RH 48%)



Optimization and Desorption

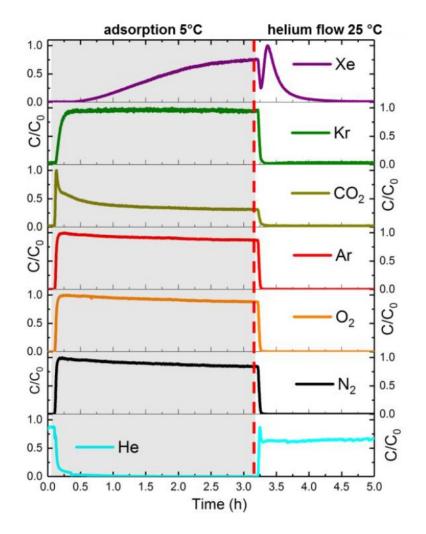


• Adsorption at 5 C

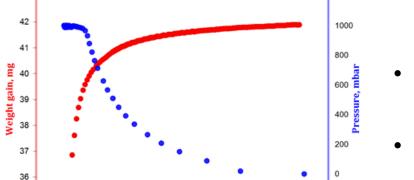
- 220 mmol/kg of Xe at
- 5 C, 8 times higher

than at RT.

- Desorption at 25 C by He purge
- Desorb within 2 hr with He purge



Kinetics and Cycle Experiments at Room Temperature



40

50

60

70

1200

- Faster kinetics, 80% of Xe adsorbed within 10 minutes.
- Cycling study indicate no loss of capacity even after 20 cycles.

Chemical & Engineering News., 94, 26, June 27, 2016

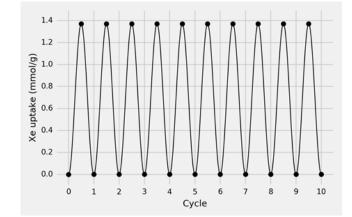
METAL-ORGANIC FRAMEWORKS Selective sorbent

traps xenon and krypton

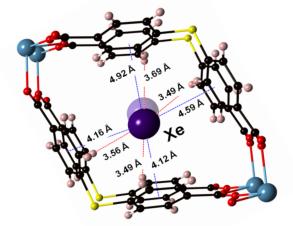
By using computational methods, a multi-institutional research team has analyzed chemical and physical properties of 125,000 porous metal-organic framework (MOF) materials This calciumand found that one based MOF of them is excepselectively traps tionally good at and separates separating xenon xenon and krypton; and krypton from green = Ca, yellow gas mixtures. = S, red = 0, gray = The team then C. white = H. confirmed that

2016, DOI: 10.1038/ncomms11831). Xenor and krypton, along with oxygen, nitrogen, carbon dioxide, and other gases, are evolved when spent nuclear fuel is reprocessed to extract valuable fissile material Reprocessing facilities trap and separate the gases, which include radioactive isotopes, via cryogenic distillation. But that approach is energy-intensive and expensive. Looking for a better option, Praveer K. Thallapally of Pacific Northwest National Laboratory and coworkers searche for sorbents that could selectively trap and separate xenon and krypton during fuel reprocessing. Nonradioactive xenor could be used for commercial lighting, imaging, and other applications, wherea the recovered krypton contains long-live isotopes and must be sequestered. The team identified SBMOF-1, a MOF made from calcium ions and sulfonyldibenzoa linkers, as the best candidate. The team found that SBMOF-1 exhibits the highes xenon adsorption capacity for a MOF an an exceptional ability to separate xenon and krypton from each other and from the other gases by size exclusion.-MITCH JACOBY

prediction experimentally (Nat. Commun







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43

35

0

10

20

30

Time, Min

Two-Column Breakthrough and Co-Adsorption at RT

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- A two-bed technique to remove and separate
 - Bed 1 remove Xe from air
 - Bed 2 remove Kr
 - Yields air without Xe and Kr
 - Off-gas can be released

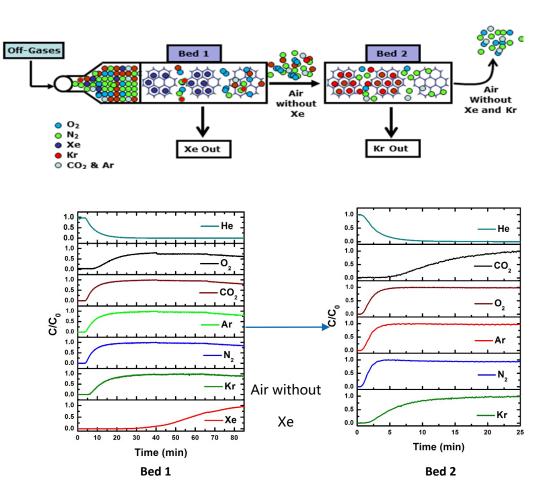
Results:

•	Bed	-	1
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Gas	Breakthrough Time (min)	Capacity (mmol/kg)	Selectivity of X
Xe	18	16 (33.8) ^a	
Kr	1	$0.11(0.75)^{a}$	14
CO_2	5	1.2	3
N_2	0.08	47	209
Ar	0.08	5.28	210
O_2	0.08	12	206
^a Capacity at equilibrium			206

• Bed - 2

Gas	Breakthrough Time (min)	Capacity (mmol/kg)	Selectivity of Kr
Kr	2.5	0.13	
CO ₂	7.5	0.90	0.3
N ₂	0.25	80.8	9.9
Ar	0.25	9.09	9.3
O2	0.25	21.2	9.3

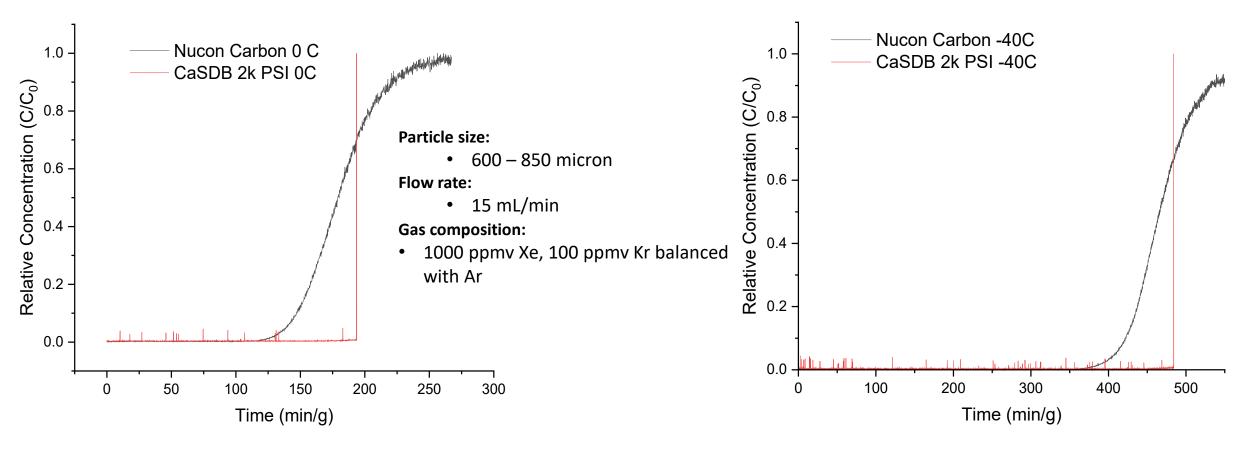




Thallapally, Ali Z. Riley, BJ., Paviet, P., Matyas, J., Vienna, J., Compact and Modular Integrated Off-Gas System and Sensors." Invention Disclosure e-IDR 18117 Thallapally, PK., Vienna et. al., USPTO WO/2017/218346A1

Banerjee, D, Thallapally, PK, Kunapuli R., McGrail, BP, Liu J et al., Surface acoustic wave sensors for refrigerant leak detection., USPTO WO2021/041359 A1

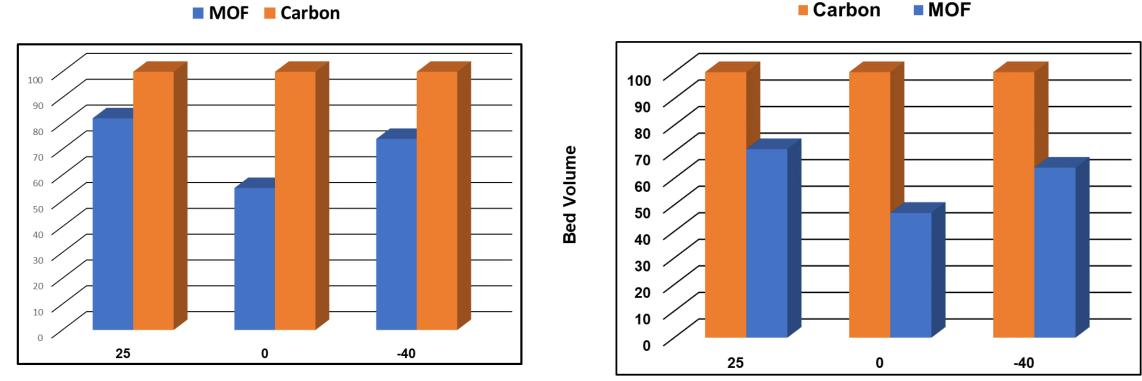
Comparison of MOF vs Carbon



Thallapally, P. K., Robinson, A. J., Zbib, A., Riley, B. J., Chong, S., Liu, J., Murphy, M. K., Okabe, P., Sherrod, R. Noble Gas Management: SBMOF 1 vs. NUCON Carbon; PNNL-33314: The U.S. Department of Energy - Office of Nuclear Energy: GAIN VOUCHER, 2022



Relative Bed Size: MOF Vs Carbon



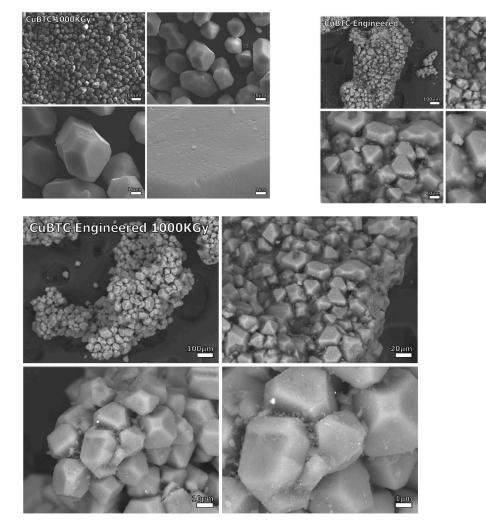
Temperature (°C)

Temperature (°C)

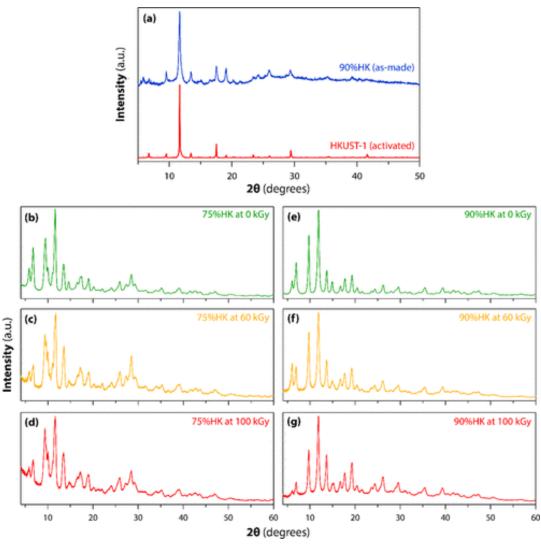
- Thallapally, Ali Z. Riley, BJ., Paviet, P., Matyas, J., Vienna, J., Compact and Modular Integrated Off-Gas System and Sensors." Invention Disclosure e-IDR 18117
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- Thallapally, P. K., Robinson, A. J., Zbib, A., Riley, B. J., Chong, S., Liu, J., Murphy, M. K., Okabe, P., Sherrod, R. *Noble Gas Management: SBMOF 1 vs. NUCON Carbon*; PNNL-33314: The U.S. Department of Energy - Office of Nuclear Energy: GAIN VOUCHER, 2022

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Radiation Stability



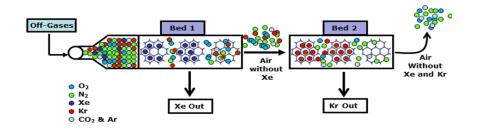




Preliminary radiation stability

Thallapally and co-workers ACS Appl. Mater. Interfaces 2020, 12, 40, 45342–45350 Thallapally and co-workers Nature Communications, 2020

Economic Analysis of Noble Gas Separation



Process	Decontamination Factor	Total Project Capital (\$10 ⁶)	Proposed Annual Consumables ^A (\$)
Cryogenic Distillation	67	8.77	267,000 ^A
Porous material	100 ^B	8.42	78,000

A: Includes compressor/pump utility loads adjusted to capacity factor and \$0.10/kWh, and annual consumables (hydrogen for cryogenic and MOF for the adsorbent process).

B: The MOF DF is theoretical and neither measured nor calculated. The model assumed complete adsorption based on experimental data and performance criteria established in bench scale testing.

DOI: 10.13140/RG.2.1.3431.2725

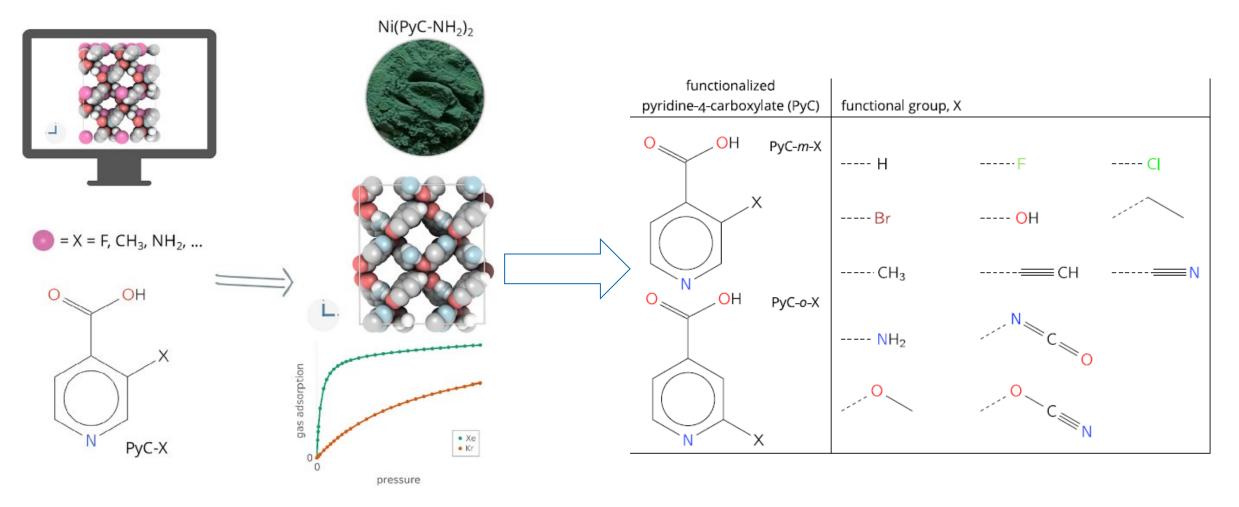
Thallapally, Ali Z. Riley, BJ., Paviet, P., Matyas, J., Vienna, J., Compact and Modular Integrated Off-Gas System and Sensors." Invention Disclosure e-IDR 18117 Thallapally, PK., Vienna et. al., USPTO WO/2017/218346A1



- Techno-economic considerations for noble gas capture from nuclear fuel processing
- > Extrapolated the data from two column approach to 400 m³/h flow rate
 - The economic assessment indicate that improvements in capital outlay, annual operating costs, and improved environmental release profiles with potentially high decontamination factors.
 - Improving the noble gas capacity and selectivity will further improve the economics
 - Assumptions include:
 - > Xe is not recovered for sell
 - Recovery of Xe can further improve the economics

Improved the Xe loading 10X since this report

Computation Informed Optimization

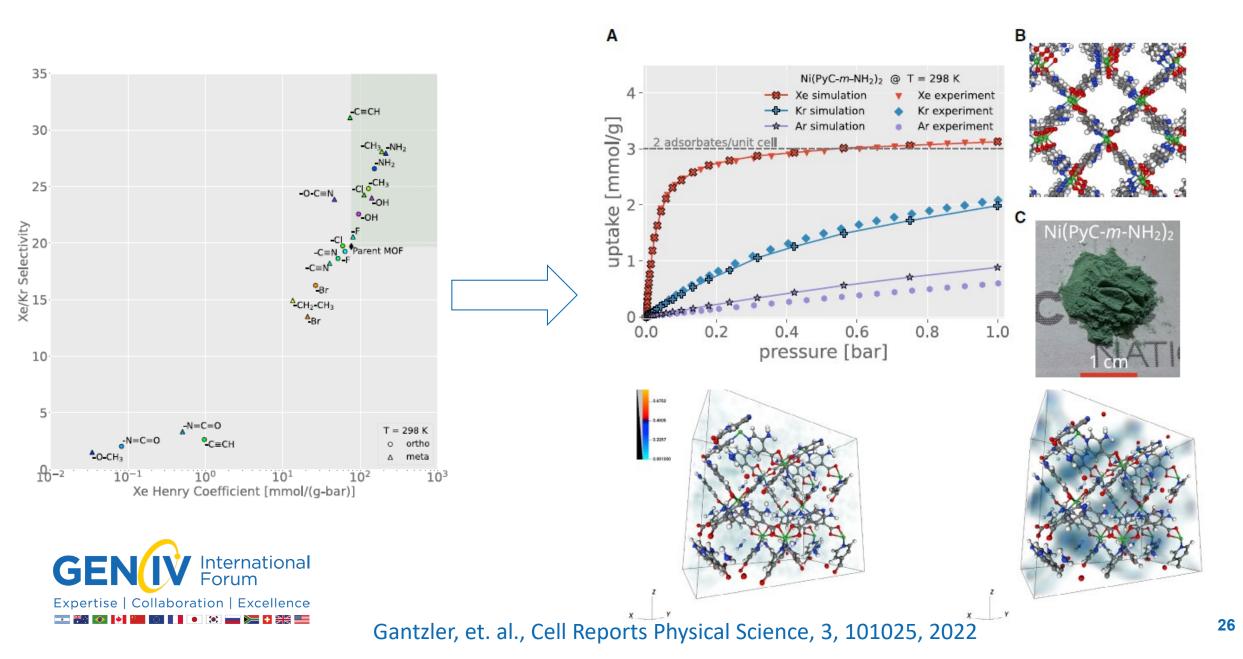


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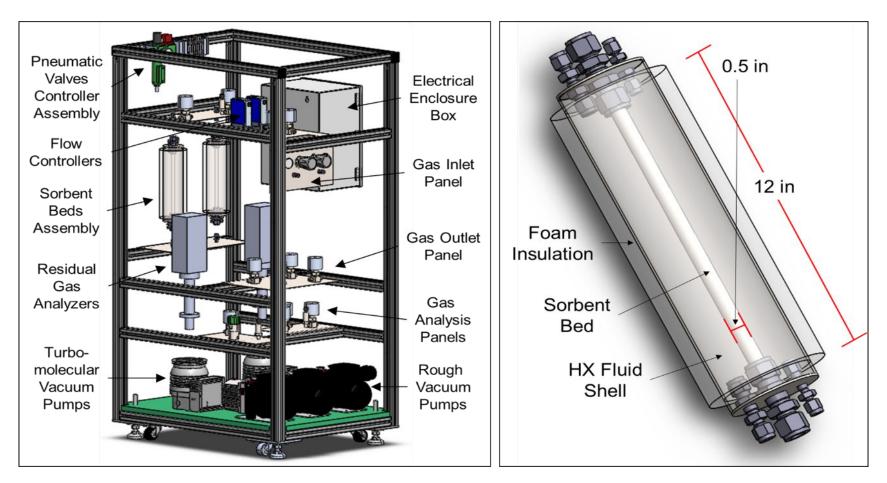
Gantzler, et. al., Cell Reports Physical Science, 3, 101025, 2022

Computation Informed Optimization

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Modular Off-Gas System



Under development as part of ARPA-E program

(Additional beds in sequence to design an integrated off-gas system)

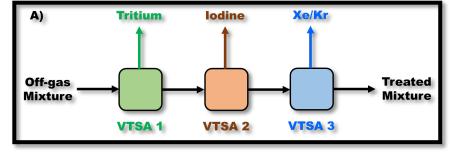


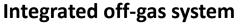
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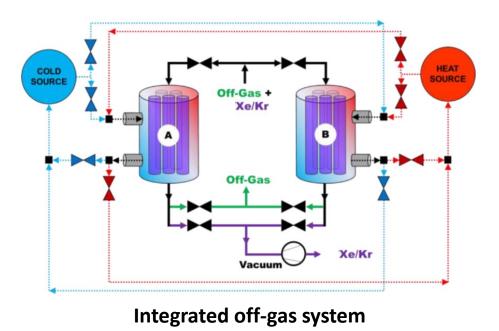
Banerjee, D, Thallapally, PK, Kunapuli R., Mcgrail, BP, Liu J et al., Surface acoustic wave sensors for refrigerant leak detection., USPTO WO2021/041359 A1

What next?

- MOFs are being developed and tested for noble gas management with success
- Further research is needed to evaluate if MOFs are beneficial to collection of Xe from molten salt reactors
 - □ Impact of acid gases on Xe/Kr
 - Impact of Radiation
 - Build a small-scale integrated system
- Integrate MOF capture technology with molten salt test loop at ORNL
 - □ MOF scale up and fabrication
- Build a lab and bench scale system coupled with selective gas sensors





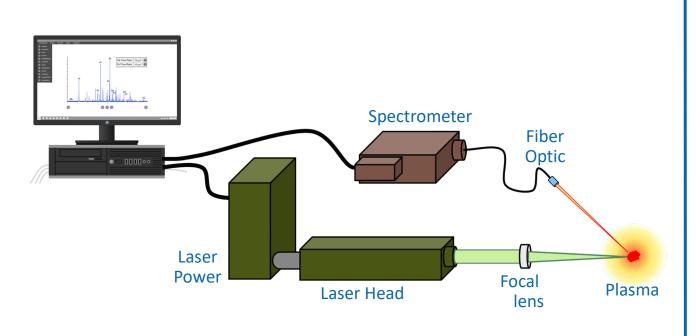




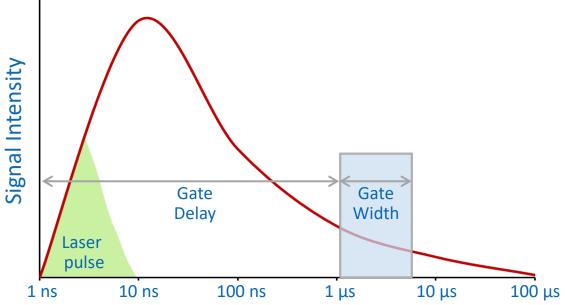
Laser-Induced Breakdown Spectroscopy for off-gas monitoring Hunter Andrews Oak Ridge National Laboratory

Laser-induced breakdown spectroscopy (LIBS) can provide an elemental fingerprint in real-time

A high energy density laser pulse ablates a sample to form a micro plasma at T~10,000 K The plasma light is collected with a gated spectrometer to measure an elemental signature







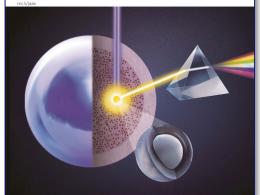
LIBS is being used at ORNL to aid in several areas of nuclear science



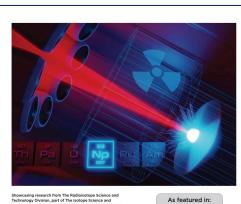


PAPER Kristian G. Myhre, Hunter B. Andrews, Nidia C. Gallego et al. Approach to using 3D laser-induced breakdown spectroscopy (LIBS) data to explore the interaction of 7 Most or all EI-Ba mothers asits with nuclear-grade graphile + OF CHEMISTRY





ROYAL SOCIETY



Showcasing research from The Radioisotope Science and Technology Division, part of The isotope Science and Engineering Directorate at Oak Ridge National Laboratory Oak Ridge, Tennessee, USA.

ROYAL SOCIETY





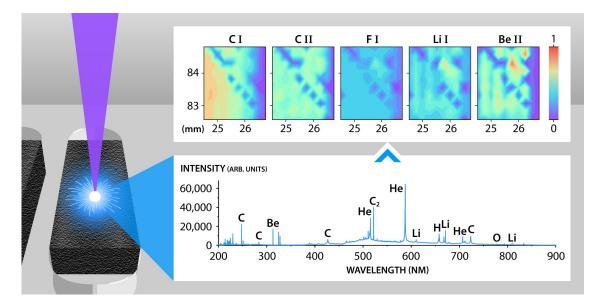
ee H. B. Andrews et al.,





LIBS in advanced reactors

- LIBS being used to evaluate nuclear reactor materials
 - Penetration of salts (FLiBe, FLiNaK) into graphite passively and with positive pressure
 - LIBS can monitor all relevant species (H, Li, Be, C, O, F, Na) in 3D through continued ablation





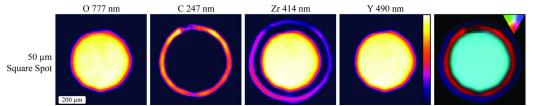
Myhre, Andrews, Gallego et al. Journal of Analytical Atomic Spectrometry 37 (2022): 1629-1641.

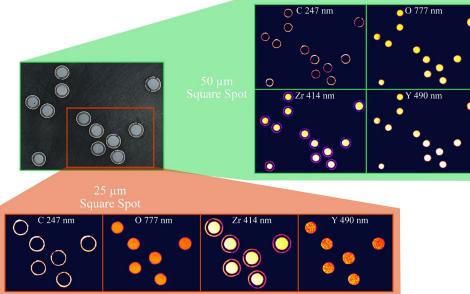




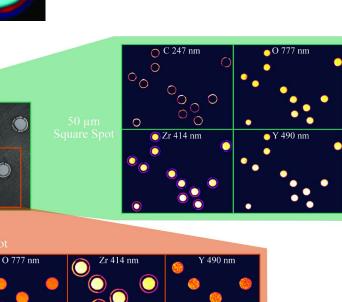
LIBS in advanced reactors

- LIBS being used to evaluate nuclear reactor materials •
 - Elemental mapping of surrogate TRISO particles —
 - Elemental depth profiling to investigate layer thicknesses and _ homogeneity







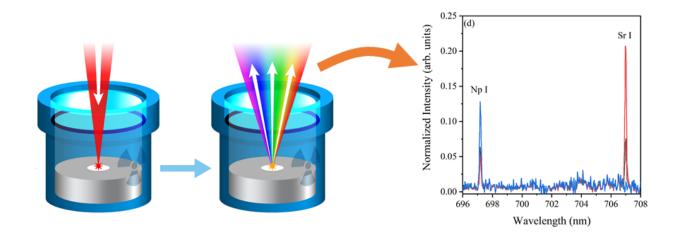


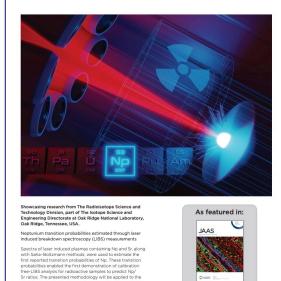


Manard and Andrews et al. Journal of Analytical Atomic Spectrometry 38 (2023): 1412-1420.

LIBS for monitoring reprocessing/chemical separations

- LIBS is being used for impurity analysis in radioactive samples
- First calibration free LIBS measurements of actinides



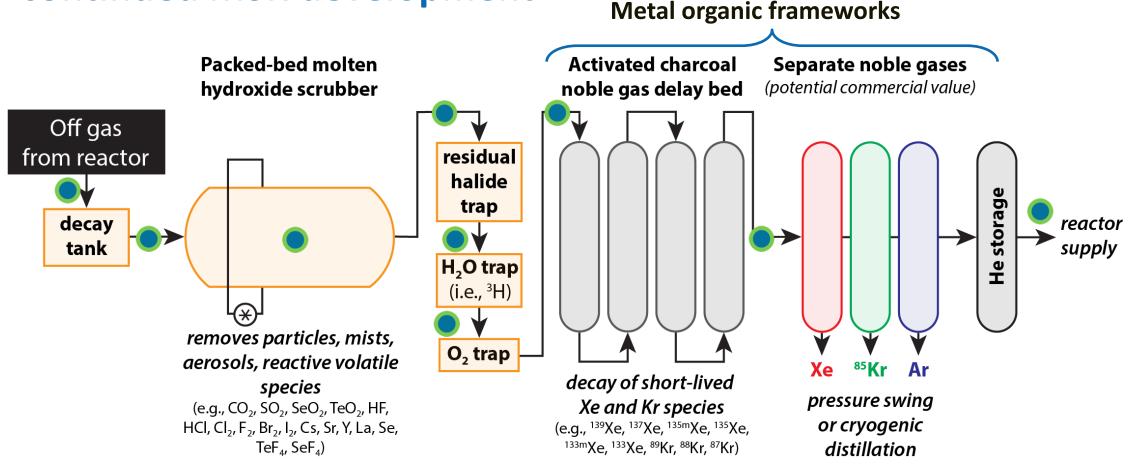






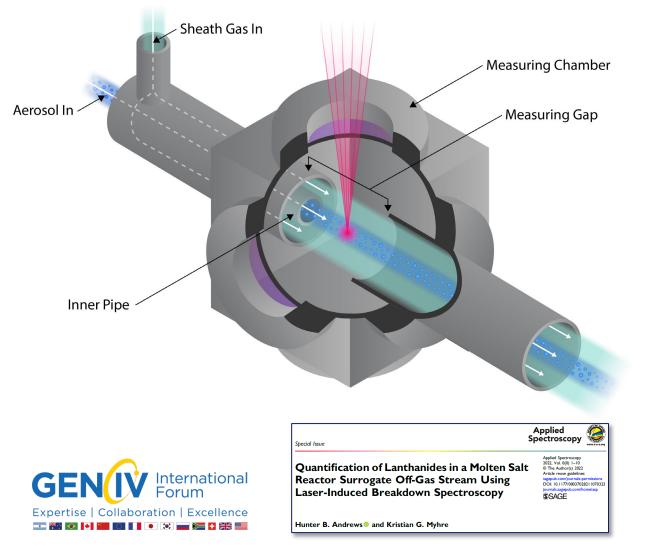
Andrews et al. Journal of Analytical Atomic Spectrometry 37 (2022): 768-774.

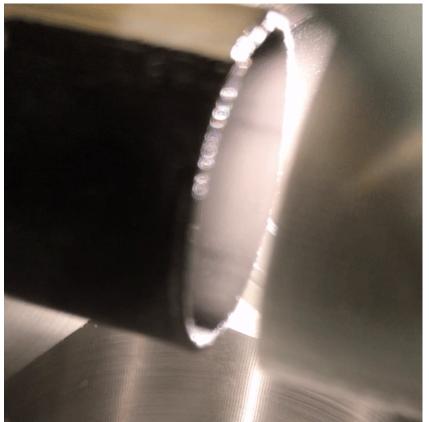
The off-gas treatment system development is critical for continued MSR development





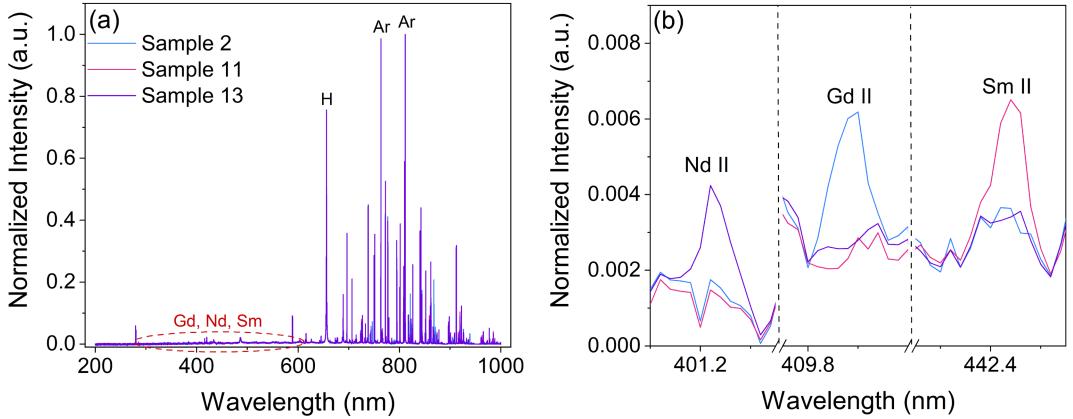
Initial feasibility has been shown on surrogate off-gas streams





Sheath gas is turned on and off repeatedly

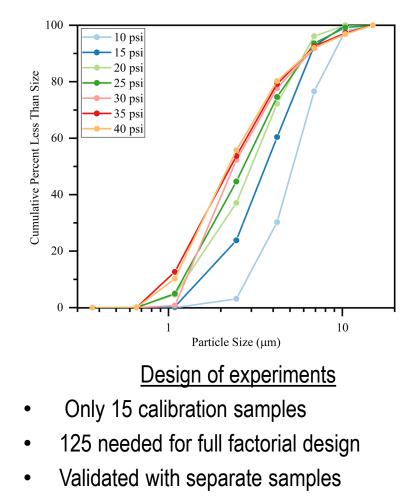
First step: demonstrate quantitative monitoring of lanthanides in aerosol stream

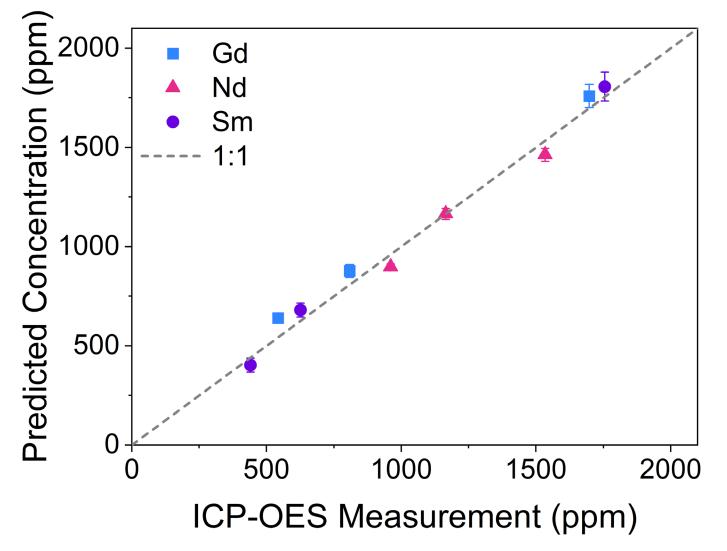


The dilution factor between the lanthanide concentration at the reservoir and measurement point was determined to be 2×10^{-6} , i.e. 2000 ppm in the reservoir ~ 0.0048 ppm in the aerosol stream.



The lanthanide validation samples were predicted with high accuracy

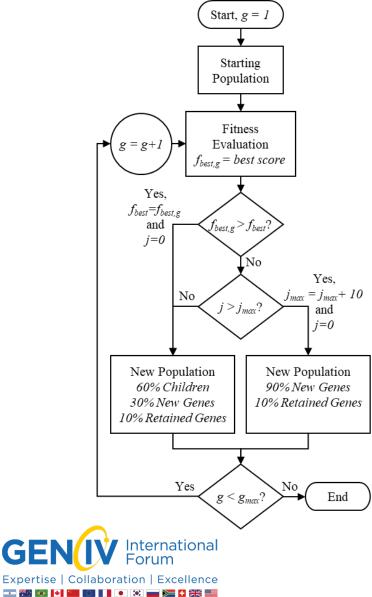


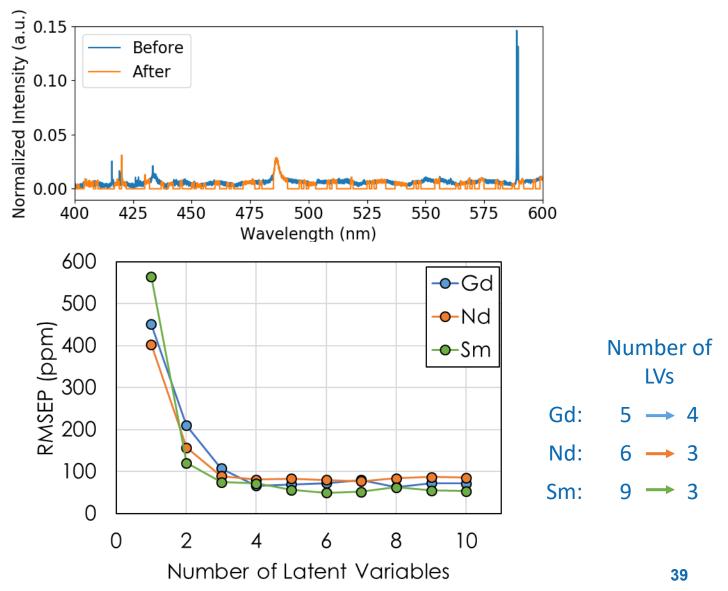




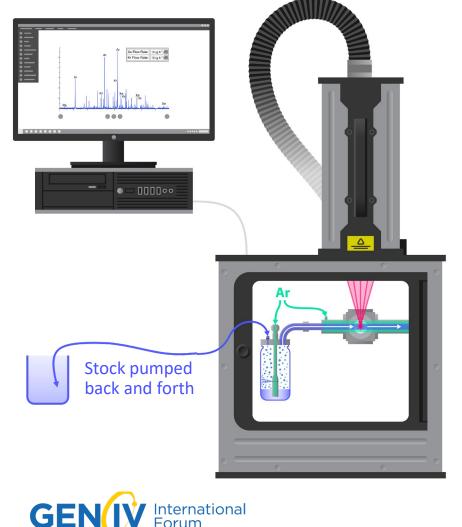
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Genetic algorithm used to refine PLS feature selection

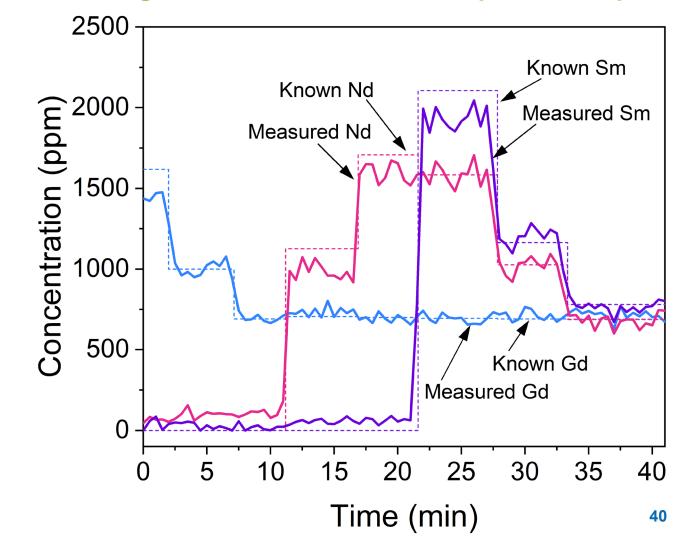




Real-time monitoring demonstrates how LIBS can be deployed in a live system



Genetic algorithm feature selection improved fit up to 73%



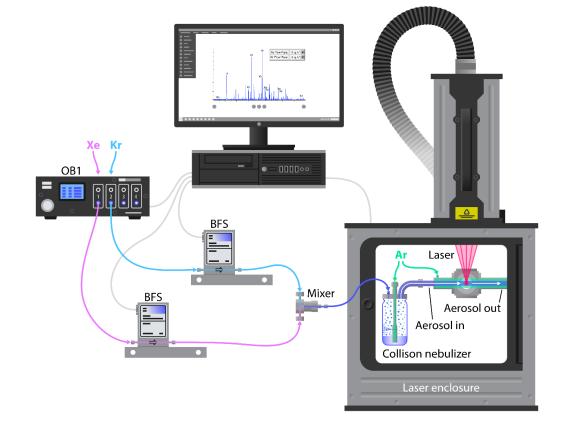
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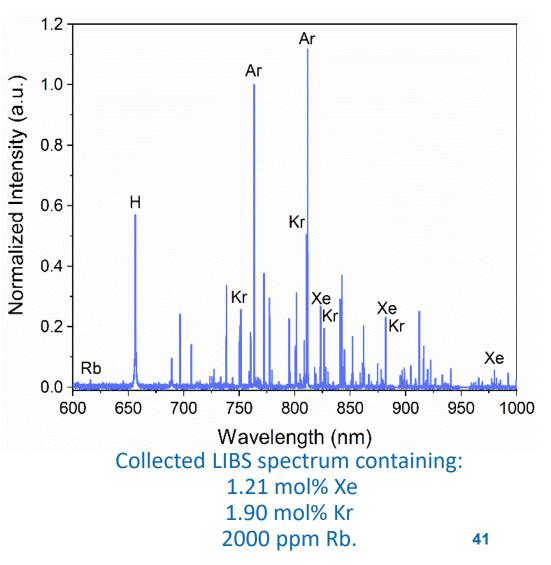
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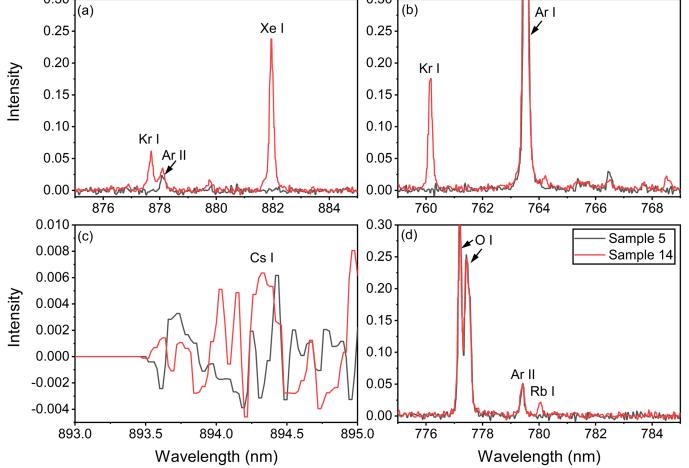
Second step: demonstrate the utility of monitoring noble gases via LIBS







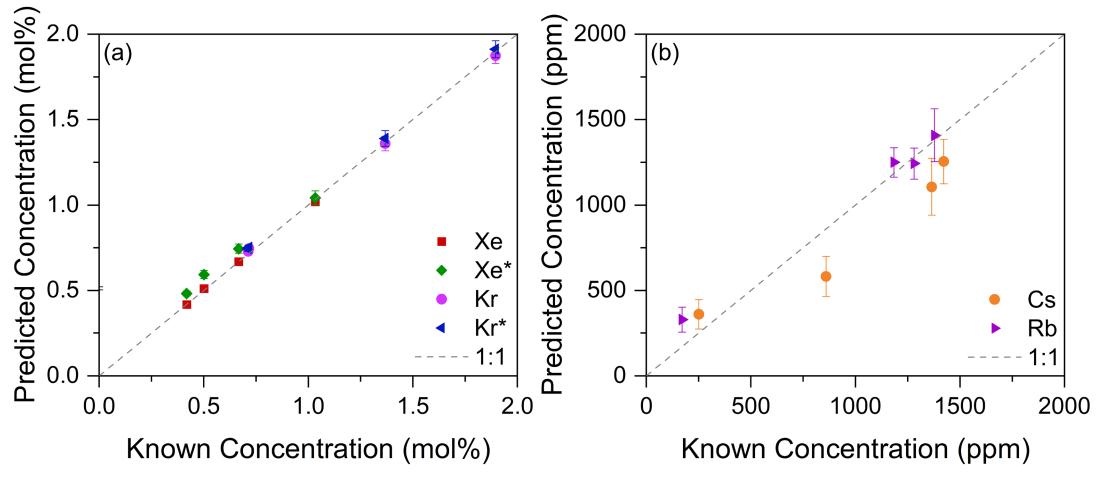
A closer look at collected spectra reveal strong gas peaks in the NIR region



Collected LIBS spectrum of a blank sample (sample 5) and sample 14 containing 1.21 and national 1.90 mol% of Xe and Kr and 2000 and 1800.9 ppm of Cs and Rb.



The noble gas validation samples were predicted with high accuracy

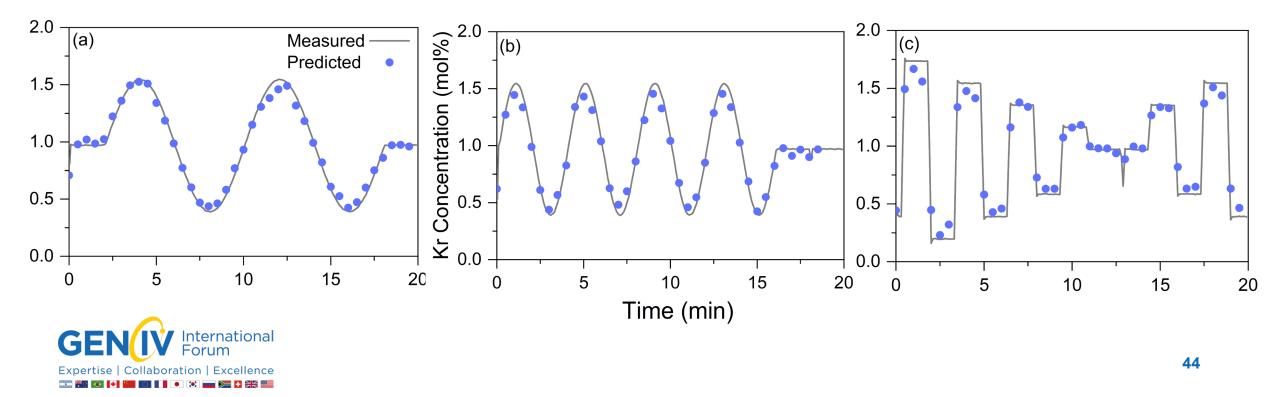


*signifies a univariate regression model

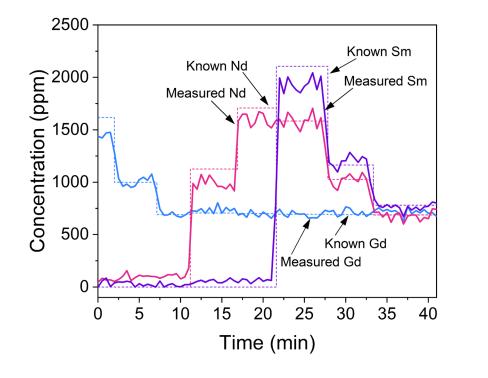


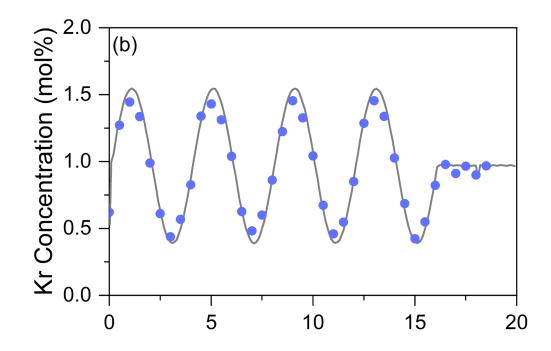
The Kr concentration was successfully predicted in real time

- Real time model successfully estimates the concentration of Kr gas when the gas flow rate is changed.
- Kr signal was collected from a plasma form within aerosol stream.
- Model can cope with entire concentration range and dynamic changes to sample being interrogated.



We have successfully used LIBS to monitor aerosolized lanthanides and Kr in real-time









Coupling LIBS with MOF for Xe breakthrough tests

Open Access Feature Paper Editor's Choice Article

Monitoring Xenon Capture in a Metal Organic Framework Using Laser-Induced Breakdown Spectroscopy

by 😵 Hunter B. Andrews 1,* 🖂 💿, 😵 Praveen K. Thallapally ² and 😣 Alexander J. Robinson ²

¹ Oak Ridge National Laboratory, Oak Ridge, TN 37830, USA

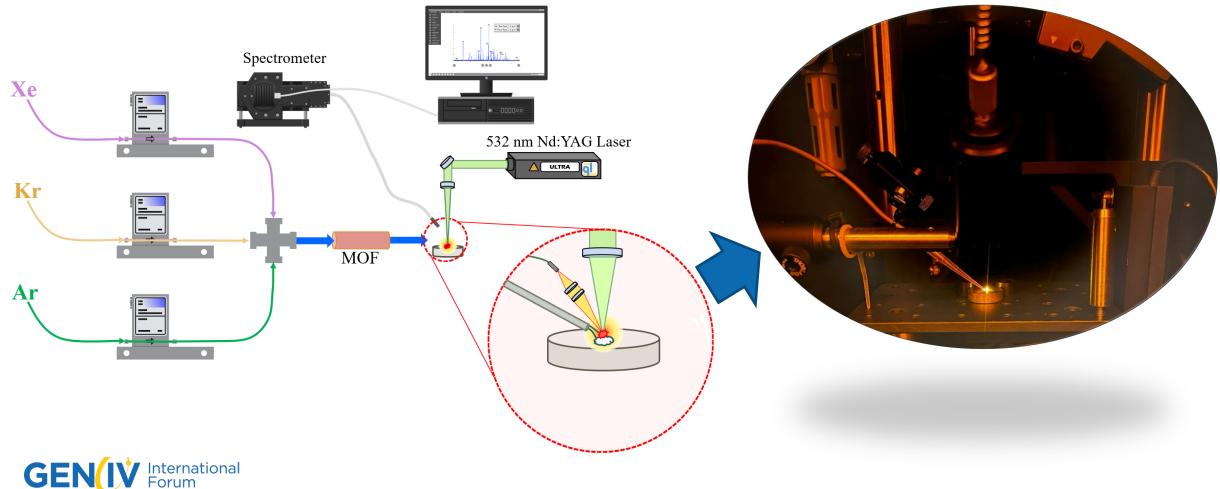
² Pacific Northwest National Laboratory, Richland, WA 99352, USA

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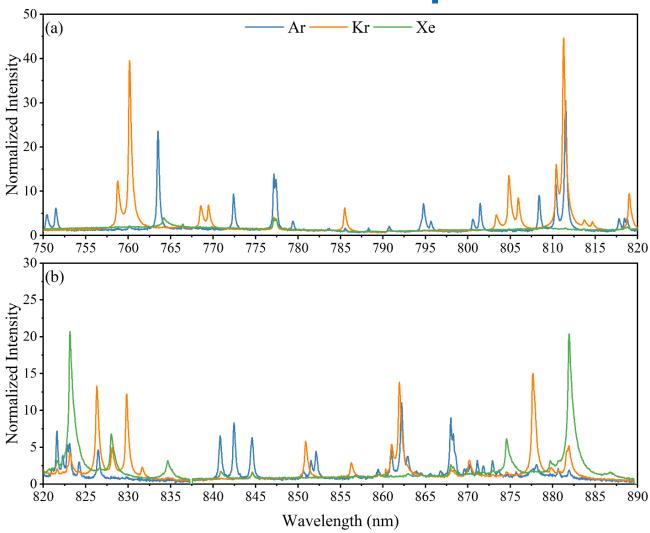
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New LIBS setup was needed to facilitate MOF size and flowrates



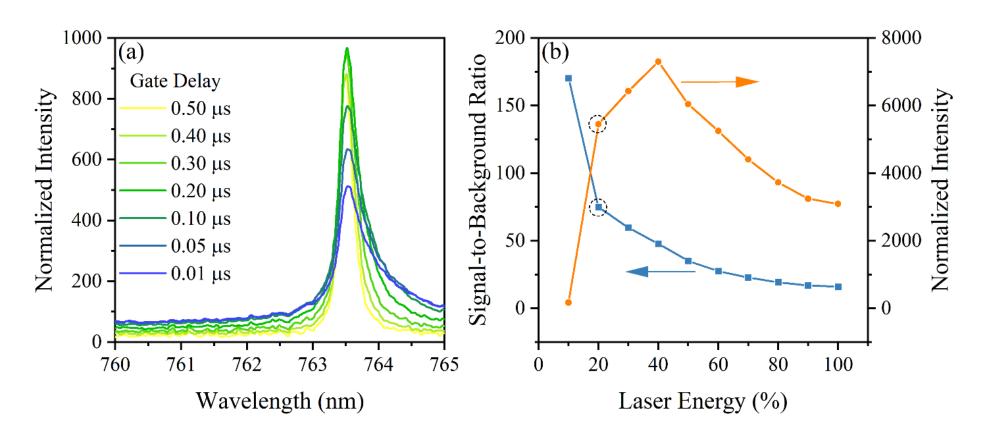
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Pure gases were run to facilitate peak identification





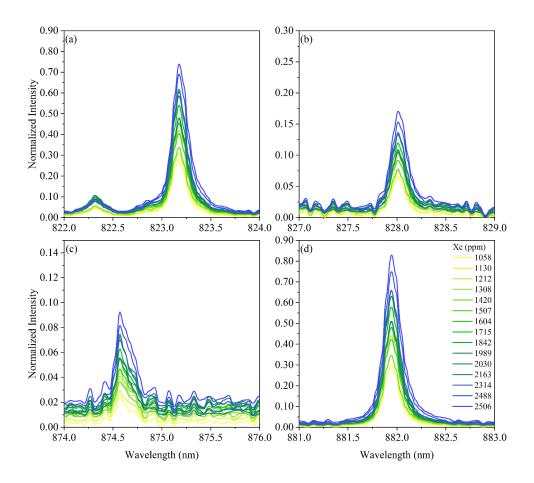
Spectrometer gating and laser energy were optimized prior to data collection

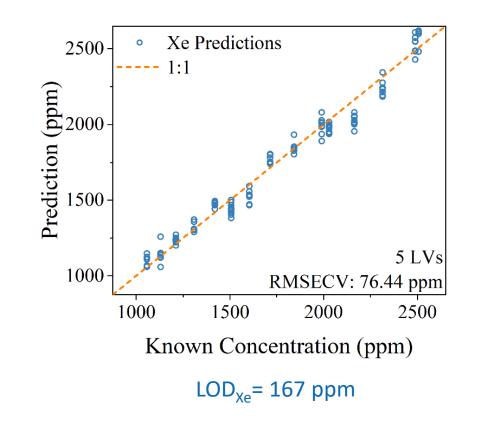


 $SBR = \frac{Peak Intensity - Background Intensity}{Background Intensity}$



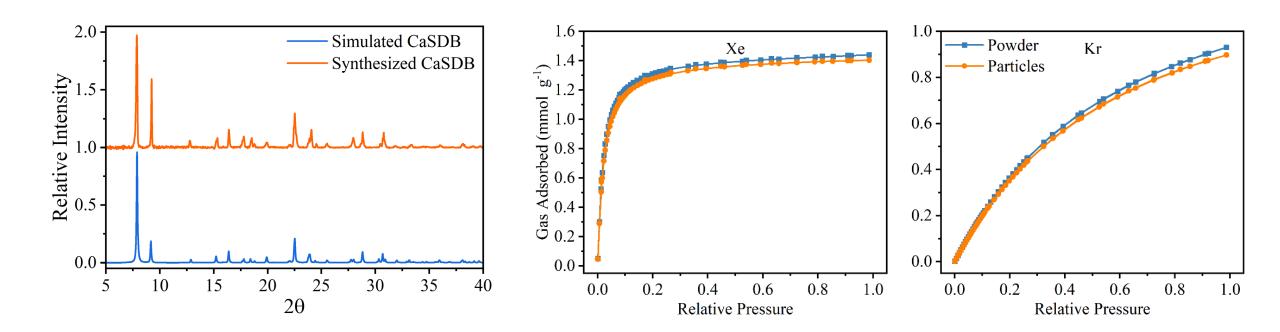
A multivariate model was built for Xe ranging from 1000 – 2500 ppm to estimate limits of detection for the given setup





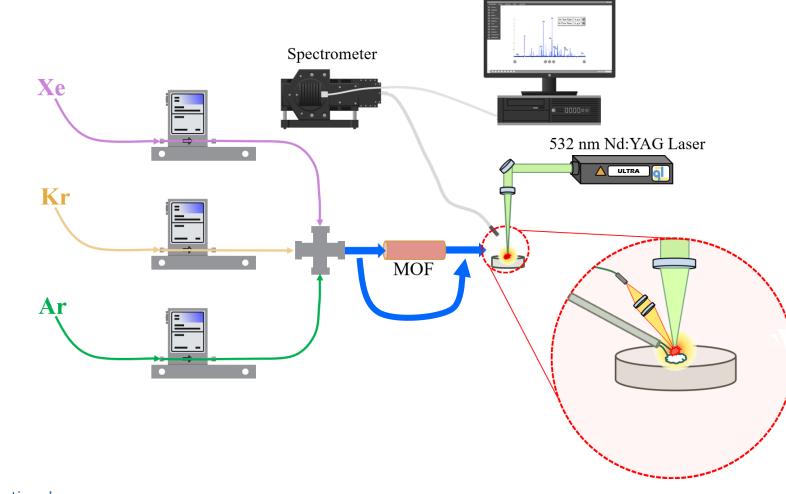


MOF column was synthesized at PNNL and shipped to ORNL for testing



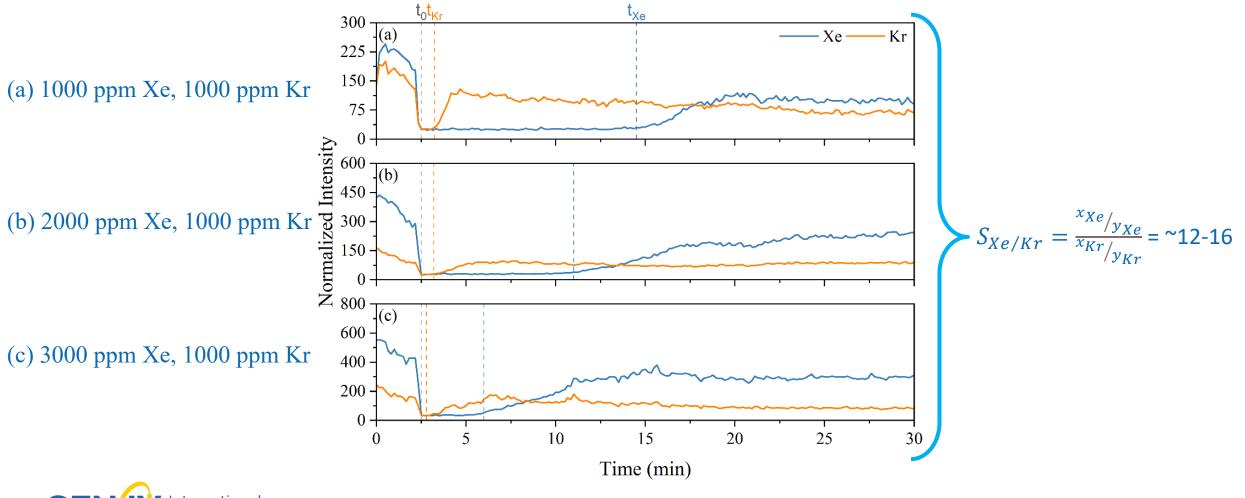


MOF column was activated and loaded into testing system





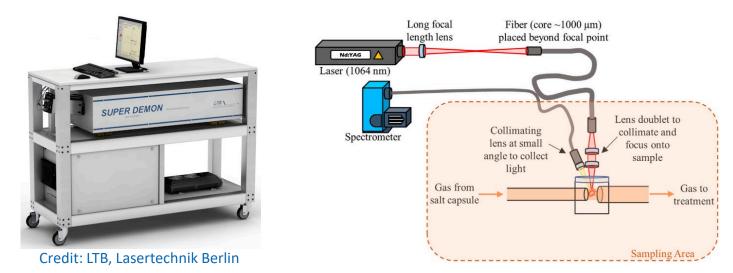
Breakthrough tests were completed on the activated MOF with the LIBS inline for noble gas tracking





Future work will focus on enhancing the LIBS noble gas sensitivity

- Designing new gas cell for in-line LIBS measurements
 - Driving down detection limits will increase the usefulness for such a technique
 - Removal of a sampling substrate will extend the sampling lifetime
- Conversations for deploying LIBS gas sensor on molten salt loops are ongoing
 - Fiber-launched Nd:YAG lasers allow safe integration of LIBS measurement systems onto engineering scale tests
- Isotopic LIBS measurement capabilities are being established for future experiments





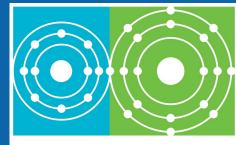




Hunter Andrews, and rewshb@ornl.gov

Praveen Thallapally, praveen.Thallapally@pnnl.gov





Molten Salt Reactor



Upcoming Webinars

Date	Title	Presenter
	Corrosion and Cracking of Supercritical Cooled Water Reactor (SCWR) Materials	Prof. Lefu Zhang, Shanghai Jiao Tong University, China
27 September 2023	EPRI Virtual Reality Training	Robert Eller, EPRI, USA
31 October 2023	The Nuclear Workforce of The Future – Opportunities and Needs for The International Nuclear Sector	Callum Thomas, Thomas THOR, United Kingdom



^{*} 2nd Molten Salt Bootcamp V

What to expect

Three days of lectures, discussions and hands-on activities on multi-disciplinary aspects of molten salt science and technology supported by world experts.

Who should apply

Graduate students, postdocs and professionals with interest in molten salts. Previous experience is not necessary, but selected participants will be required to complete an introductory class ahead of the bootcamp.

Agenda

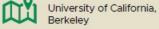
Day 1 - Multiphysics modelling of MSRs Day 2 - Experimental characterization of molten salts Day 3 - Large scale experimental facilities

Participants will work on group projects throughout the bootcamp to explore the concepts learnt.

Contact Us

msrbootcamp@icloud.com



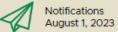




September 6-8, 2023



Application deadline July 28, 2023



Submit your application here

