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Analysis of Off-Gas Streams from Small- and Large-Scale Sparging Salt Vessels using Laser-Induced Breakdown Spectroscopy

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September 2025

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Radioisotope Science and Technology Division

ANALYSIS OF OFF-GAS STREAMS FROM SMALL- AND LARGE-SCALE SPARGING SALT VESSELS USING LASER-INDUCED BREAKDOWN SPECTROSCOPY

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ABBREVIATIONS

DEMON	Double Echelle Monochromator
FASTR	Facility to Alleviate Salt Technology Risks
LIBS	laser-induced breakdown spectroscopy
MCS	multichannel spectrometer
MSR	molten salt reactor
ORNL	Oak Ridge National Laboratory
RGA	residual gas analyzer

ABSTRACT

Laser-induced breakdown spectroscopy (LIBS) is being developed as an analytical monitoring tool for molten salt reactor (MSR) systems. A mobile LIBS platform was configured on a small-scale salt vessel (~100 g of salt) and then transported 0.6 km to successfully monitor an engineering-scale pumped salt loop (~100 kg of salt). Several studies on monitoring salt aerosols, hydrogen isotopes, and noble gases were performed on the small-scale system. All these studies were then implemented for monitoring various tests on the engineering-scale loop. Aerosolized salts from both the storage tank and pump bowl were monitored using LIBS to assess bulk salt analyte ratios (Mg, Na, K) and evaluate impurities (H, O, Ca). In addition to monitoring salt aerosols, LIBS was used in tandem with a residual gas analyzer (RGA) and a Raman spectroscopy system to detect hydrogen isotopes as they were sparged through and subsequently depleted from the loop storage tank. Lastly, a similar test with noble gases (100 ppm Kr, 1000 ppm Xe) was performed, in which LIBS demonstrated a faster response than the RGA used in-line. The tests and results are summarized in this report with detailed publications to follow. Key takeaways are listed in the conclusion of the report.

1. INTRODUCTION

Molten salt reactors (MSRs) are an emerging advanced reactor concept that offers benefits over conventional light-water reactors: high thermal efficiency, passive safety features, low operating pressure, and the potential for online generation of radioisotopes.[1-5] However, with these benefits also come challenges, largely related to the use of dissolved nuclear fuel in the liquid salt that circulates through the core. As fission occurs and fission products are generated, these species will either remain in the salt, precipitate onto solid surfaces, or evolve into the gas headspace.[6, 7] The evolution of aerosols, volatiles, and noble gases necessitates an off-gas treatment system to remove them from the cover gas before it is either recirculated through the core or vented to the environment. Key criteria for an off-gas system include the detection of noble gas fission products (e.g. Kr and Xe) at low concentrations, the discrimination of H isotopes, the detection of salt aerosol particles, the ability to quickly detect small changes in concentration (within seconds to minutes), ruggedness against physical disturbances, and the ability to operate continuously for long periods. Understanding the effectiveness of off-gas treatment components is a vital step in the maturity of MSR technology.

Sensor technology for MSRs lags behind other aspects of reactor development. Traditional monitoring techniques are hindered by high temperatures and limited access, high background radiation in the case of gamma spectroscopy, and material compatibility issues with respect to optics and electronics.[8] In the past few years, laser-induced breakdown spectroscopy (LIBS) has been under development for MSR monitoring under the US Department of Energy, Office of Nuclear Energy's MSR Campaign. Preliminary proof-of-concept work has demonstrated the capability of LIBS to monitor aerosolized lanthanides and noble gases and evaluate the effectiveness of off-gas treatment components.[9-12] During these testing efforts focused on off-gas, it was found that aerosol production through sparging also provides a simple means for sampling bulk molten salt compositions.[13] This, in turn, makes LIBS a viable technique for off-gas monitoring as well as a tool for monitoring the salt within various sub-systems. Other sensors are also in development under the campaign, including Raman spectroscopy for molecular gas detection and electrochemical probes for salt redox monitoring.

In fiscal year 2025, the primary goal of the LIBS efforts has been to transition from proof-of-principal studies to demonstrative work on actual molten salt systems with their associated challenges (e.g., temperature, aerosol transport, inert atmospheres). A small-scale salt vessel (~100 g) was set up in a fume hood for initial salt tests. Along with this system, a modified LIBS platform was constructed for monitoring gases and aerosols such that it could be mobilized for deployment in large-scale tests

(~100 kg) at Oak Ridge National Laboratory (ORNL) and at external sites in future years. The final effort of the year focused on transporting this mobile LIBS platform across the ORNL campus to monitor a large-scale pumped Facility to Alleviate Salt Technology Risks (FASTR).[14] This report summarizes the use of LIBS for monitoring MSR-relevant signatures from both small- and large-scale salt systems.

2. EXPERIMENTAL

2.1 MOBILE LIBS PLATFORM

The LIBS setup for this study used a mobile LIBS platform with a modular design, allowing various sample stream configurations and spectrometers. In this work, the aerosol/gas stream was fed into the LIBS cell using an aerosol focusing nozzle to concentrate aerosols across the sampling point, which has been demonstrated previously.[13, 15, 16] The LIBS system used a 1064 nm nanosecond Nd:YAG laser fired at 50–150 mJ per shot at 10 Hz (150 Nano-LG, Litron). The laser pulse was passed through a beam expansion module before being focused through an angled window to the sample stream. The angled window prevented damage to the optics from back reflections and sealed the optics from the sample cell. Two spectrometers were used: a multichannel spectrometer (MCS) with broad elemental coverage (6-channel 4096CL, Avantes) and a high-resolution spectrometer with narrow bandwidth (~2 nm) but capable of resolving isotopic shifts (DEMON, LTB Lasertechnik Berlin).[9] The DEMON spectrometer and mobile LIBS platform were outfitted on separate carts for transportation between laboratories. The two systems were transported nominally 0.6 km between buildings for testing on the large-scale salt system (Figure 1). Generally, spectra were measured on both spectrometers with delay and integration times of 15 µs and 1000 µs, respectively. The measurement cell contained eight collection optics, permitting multiple spectrometers to be used in tandem and synced using a pulse generator. The inlet and outlet of the LIBS cell were plumbed into various salt systems depending on the test, as is further described in what follows. The back of the LIBS cell was outfitted with a purge port to apply either vacuum or an inert gas to the cell as needed.

Figure 1. Transportation of the LIBS platform (left) and DEMON spectrometer (right) for tests on FASTR.

2.2 FACILITY TO ALLEVIATE SALT TECHNOLOGY RISKS

FASTR at the US Department of Energy's ORNL is a pioneering system designed for high-temperature testing of chloride-based molten salts, specifically a NaCl-KCl-$MgCl_2$ mixture.[14] Operating at temperatures up to 725°C and constructed primarily of alloy C-276, FASTR incorporates key components such as a centrifugal pump for salt circulation, an air-based heat exchanger, trace heating to prevent salt freezing, and a suite of instrumentation, making it a unique capability within the US research landscape. The system, which holds 250 kg of salt and has the capability of 465 kW of power, allows technologies to be de-risked in a cost-effective manner without using hazardous beryllium- or uranium-bearing salts. FASTR differentiates itself through its scale, its co-located purification system, and its relatively large operating capacity. For several years, the integration of sensors for testing on FASTR has been planned, making the work documented in this report the first step in executing those efforts.[17, 18]

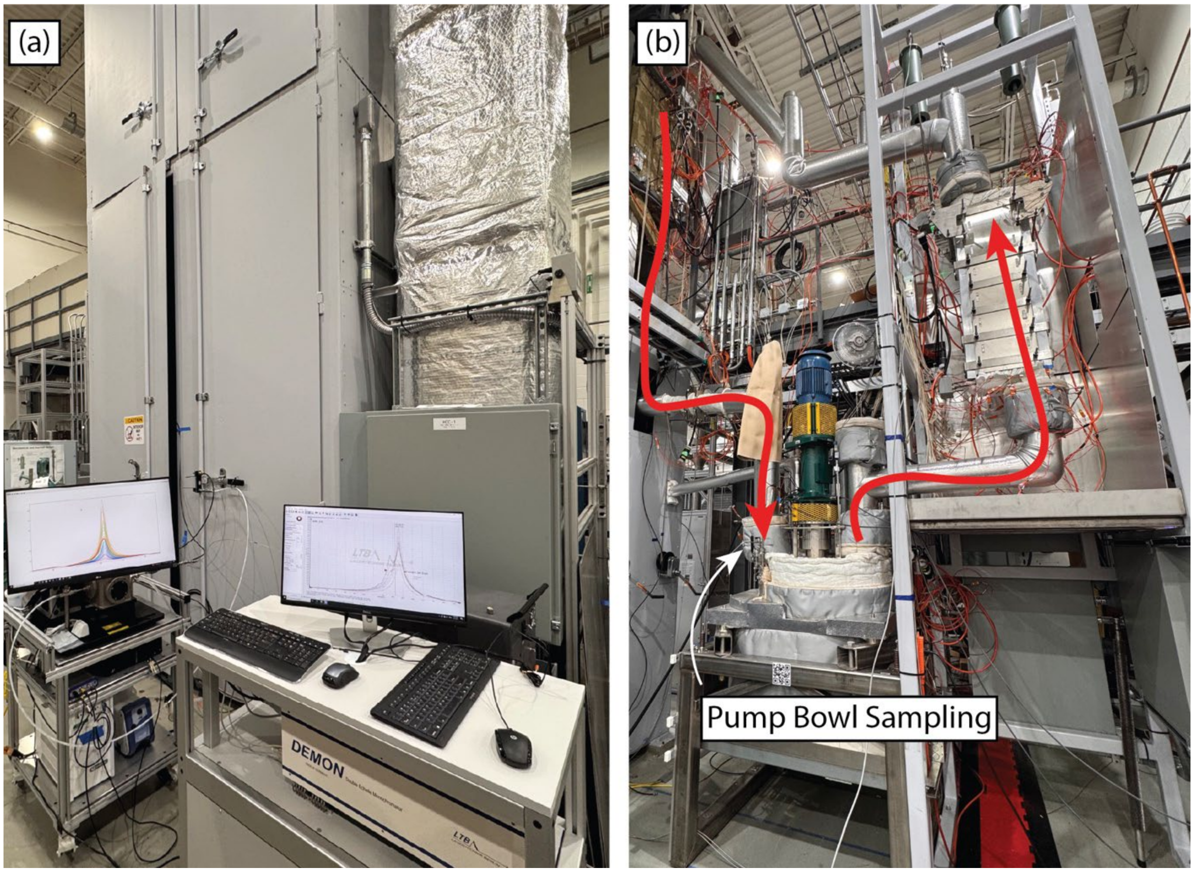

Figure 2. (a) FASTR storage tank with LIBS cart in the front left and DEMON spectrometer in the front right. (b) FASTR loop with the salt direction shown in red and the pump bowl sampling location indicated.

2.2.1 Salt Storage Tank Arrangement

FASTR also includes a salt storage tank in which the salt is kept while the loop is not operating. This tank is equipped with heaters to allow the salt to be frozen and remelted. The tank includes automatically controlled inlet and outlet valves to maintain the gas pressure in the head space. A gas bubbler tube submerged in the salt allows gas to be added by bubbling through the salt. A gas sampling line is located at the top of the tank for sampling the headspace.

2.3 SMALL SCALE SALT POT

Small-scale testing was conducted using a custom-fabricated stainless steel cylindrical vessel approximately 8 cm in diameter and 20 cm tall. The vessel was sealed with a copper gasket and a flange equipped with a 6 mm gas inlet for sweeping the headspace and through which a tube could be inserted into the salt for sparging, a 3 mm inlet through which a thermocouple was inserted into the salt, and a 12 mm outlet for the aerosol-bearing off-gas stream. A gas manifold allowed for separate gases to be flowed through the sweep and sparge lines, both with flow rate monitored by digital mass flow meters (Aalborg, GFM17). The aerosol-bearing gas stream was transported to the mobile LIBS unit for analysis through nylon antistatic flexible tubing. The salt (~100 g) was contained within the vessel inside a glassy carbon crucible. The vessel was heated using a benchtop programmable furnace, with a heating trace on the exposed top flange to minimize temperature gradient. This setup was thoroughly tested, as described in Section 3.1. Initial tests were carried out with nitrate salts ($NaNO_3$-KNO_3 eutectic) at 260–300 °C, followed by tests with chloride salts (LiCl-KCl eutectic) at 400–500 °C. The results of these tests were used to determine the optimal LIBS sampling parameters.

2.4 OTHER SENSOR AND MEASUREMENT DEVICES

An eight-stage cascade impactor was used to collect and measure aerosols produced from the storage tank and FASTR pump bowl, with and without sparging. The cascade impactor separates aerosols based on their aerodynamic particle size.[21] Here, a particle above the cut-off point for a given stage will impact the stage substrate and be collected; meanwhile, smaller particles will accelerate through the stage orifices onto the next stage. The use of a cascade of progressively smaller orifices enables particles to be effectively binned by their particle size. The collected salts were dissolved from the substrates and analyzed using inductively coupled plasma – optical emission spectroscopy to determine concentration and composition. Comparing the concentrations on each stage leads to particle size distributions, and by factoring in the test duration, the aerosol concentration in the gas stream can be determined.

A residual gas analyzer (RGA) mass spectrometer by Stanford Research Systems (SRS) was used for identifying and analyzing gases sampled from the head space of FASTR through a capillary tube feeding into a vacuum chamber. The chamber is evacuated to ultra-high vacuum (UHV) $<10^{-5}$ Torr using roughing and turbomolecular pumps. The RGA operates by ionizing gas molecules into positive ions, separating these ions based on their mass-to-charge ratios, and measuring the ion currents for each mass. The RGA can conduct both qualitative and quantitative analyses of gases in a system. Whereas obtaining mass spectra is straightforward, interpreting the spectra to derive meaningful insights about the vacuum system requires additional effort such as calibration and analysis. The RGA provides complementary data to the LIBS and Raman systems to aid in the understanding of the species being sampled from FASTR. The RGA has also been described previously, when it was attached to the gases being sparged through the Large Scale Fluoride Test Facility (LSTL).[18]

Figure 3. RGA system used for measuring off-gas from FASTR.

A multitrack Raman instrument from Spectra Solutions was sent from Pacific Northwest National Laboratory (PNNL). Raman spectroscopy is capable of remote monitoring through fiber optic delivery of laser light to probes and fiber routing of scattered light back to a spectrometer.[19, 20] The multitrack instrument was equipped with the ability to monitor six Raman probes (locations) simultaneously, with an integrated 405 nm laser. In addition to the standard Raman probe barrels, a custom double-pass Raman flow cell was placed in-line with the mobile LIBS instrument for comparative analysis. The Raman instrument and probes will be sent back to PNNL for characterization and assessment of any damage that they might have sustained as a result of the high temperatures and corrosive environment of the salt loop. Additional data analysis and comparisons with RGA and LIBS are ongoing, but, overall, Raman was successfully deployed on the FASTR loop, and the results are consistent with those observed by the RGA and LIBS instruments.

3. RESULTS AND DISCUSSION

3.1 SUMMARY OF SMALL-SCALE SALT VESSEL STUDIES

Several studies from the small-scale salt vessel have been recently published.[13, 22, 23] These studies are briefly described here. The first study involved a demonstration of using LIBS to monitor hydrogen isotopes and molten salt aerosols simultaneously.[23] This study highlighted the versatility of LIBS for monitoring gases and aerosols in tandem as they evolve from a molten salt. Additionally, multiple spectrometers were used together, which is permitted by the design of the mobile LIBS platform. The high-resolution DEMON spectrometer dwelled on the 656 nm H emission window to monitor the shift from deuterium to protium, and the onboard broadband spectrometer measured salt and gas signatures.

In the aforementioned study, a sparging configuration for aerosol generation was used; however, a previous set of studies using LIBS for molten salt monitoring utilized a Collison nebulizer[11]. This second study compared the aerosols generated from both methods.[13] Aerosol particle size distribution, the impact of transport distance (e.g., the distance between the salt vessel and measurement point), and the effect of flow rate were examined. Overall, the sparging approach was found to use less salt and gas, and it provided an adequate sample for quantification without any heating of gas lines. This approach was used for monitoring various sub-systems on FASTR. This study also included an optimization of the LIBS sampling parameters—including laser energy, delay time, integration time, and number of accumulates—to determine the appropriate settings for the tests on FASTR.

The last of the aforementioned studies investigated the impact of the bulk gas (e.g., Ar or He) on the detection of trace Xe and Kr.[22] While many non-radiological molten salt systems use Ar for economic reasons, an MSR will likely utilize a He cover gas to avoid issues associated with activated ^{40}Ar isotopes. The effect of bulk gas on the LIBS plasma was very apparent, with Ar leading to higher plasma temperatures and electron densities, whereas the He plasma had lower background levels due to its higher activation energy. Fortunately, through multivariate modeling, similar limits of detection for trace Xe and Kr were achieved. Additionally, the transients of Xe and Kr varying from 0 ppm to 600 ppm in both gases were monitored, and relative standard deviations were observed to be less than 5% for over an hour. For more information on these efforts, the reader is directed to the associated journal papers.

3.2 MONITORING GAS FLOW THROUGH THE FASTR SALT STORAGE TANK

Before being attached to the main FASTR salt loop, the LIBS apparatus was attached to the sampling line of the FASTR salt storage tank with a 3 m antistatic nylon tube.

3.2.1 Hydrogen Isotope Monitoring

To demonstrate monitoring H isotopes in the FASTR off-gas, a test was done in which the salt was sparged with 4% H_2 in Ar for periods of 30 min, followed by sparging with Ar for 30 min. This was done at sparge flow rates of 0.3 SLPM, then 0.9 SLPM, then 1.8 SLPM. Then this was repeated with 4% D_2 in Ar, with the final Ar flow rate at 0.3 SLPM, for a total of 11 steps. The flow through the analyzers (LIBS, RGA, and Raman) was maintained at a constant 0.3 SLPM. The pressure in the storage tank was automatically maintained at ~5 PSI by venting gas or adding Ar as needed.

The LIBS was set up to constantly monitor the gas stream; spectra were stored as 100 shot accumulates, collected at 10 Hz, so that each spectrum represents a 10 s average. The laser energy was 50 mJ, and the delay time and integration time were 15 μs and 1 ms, respectively. The flow of gas through the FASTR storage tank was also monitored by residual gas analysis and Raman spectroscopy in series with the LIBS. This provided complementary information to the LIBS by tracking molecular species. These data will be discussed in a separate document.

Figure 4(a) shows the combined LIBS signal for H and D during this test. The sparge gas and flow rate for each of the 11 steps are indicated in the figure. Each segment of the data has been fitted with a linear regression; the slope is indicated in the figure, and the residuals are shown in Figure 4(b). The residuals demonstrate the speed and linearity of the LIBS response. In every case where the sparge gas was changed from Ar to a mixture with 4% H_2 or D_2, resulting in an increase in H or D in the off-gas, the LIBS signal responded almost immediately, with a change of slope completed in under 60 s. This is consistent with the small-scale study, in which the LIBS signal followed gas mixing behavior and changes in gas concentration fast enough to observe oscillations from the dampening effect of the flow controller within 2 min. This highlights the use of LIBS for monitoring changing conditions in MSR-related systems. When the flow rate was changed back to pure Ar, resulting in a decrease in H or D, the signal demonstrated hysteresis and in some cases continued to increase for a short time. Most notably, this was observed during the sixth step (sparging with Ar at 1.8 SLPM). This hysteresis was not observed in small-scale tests without salt present, indicating that the salt retained a significant quantity of H or D that was subsequently removed by the bubbling with Ar, causing the quantity of H or D in the head space and off-gas to remain elevated for a longer period of time.

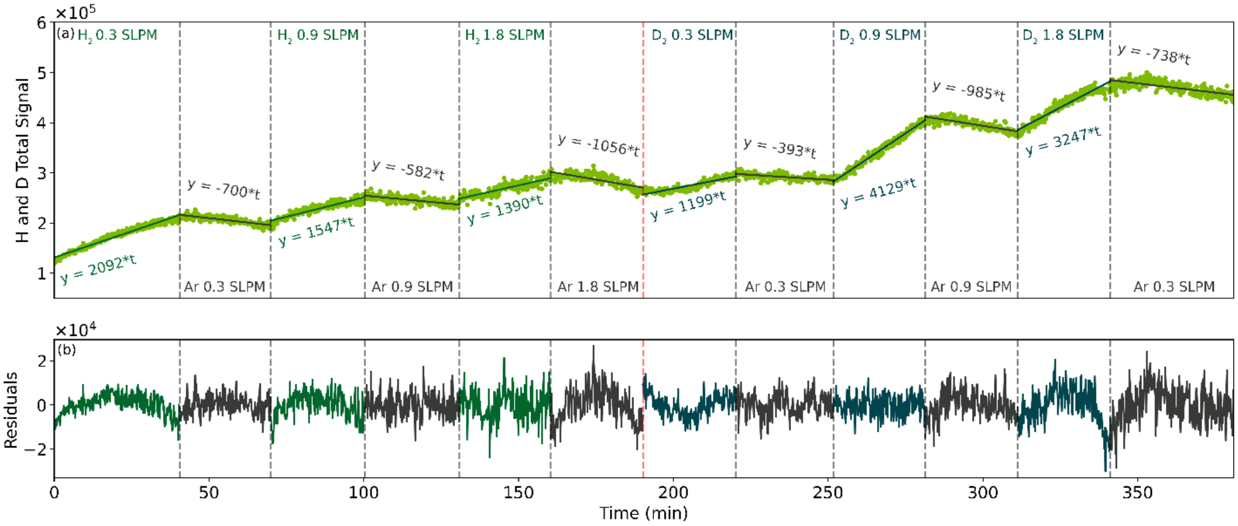

Figure 4. (a) Linear fits and (b) residuals for each period of gas flow in the H isotope test. Vertical lines indicate when sparge gas and flow rate were changed; each section is labeled with the corresponding gas and flow rate. The red line denotes the change from tests with Ar and Ar-H_2 to Ar and Ar-D_2.

Figure 5 shows the LIBS data collected during this test with both the high-resolution DEMON and broadband MCS. The DEMON provided much better resolution of the H and D emission peak shift due to its increased resolving power, although the MCS was still able to detect the shift. This is an important finding as the DEMON spectrometer provides high spectral resolving power and sensitivity at the cost of broad spectral monitoring and a larger overall footprint that are features of the MCS.

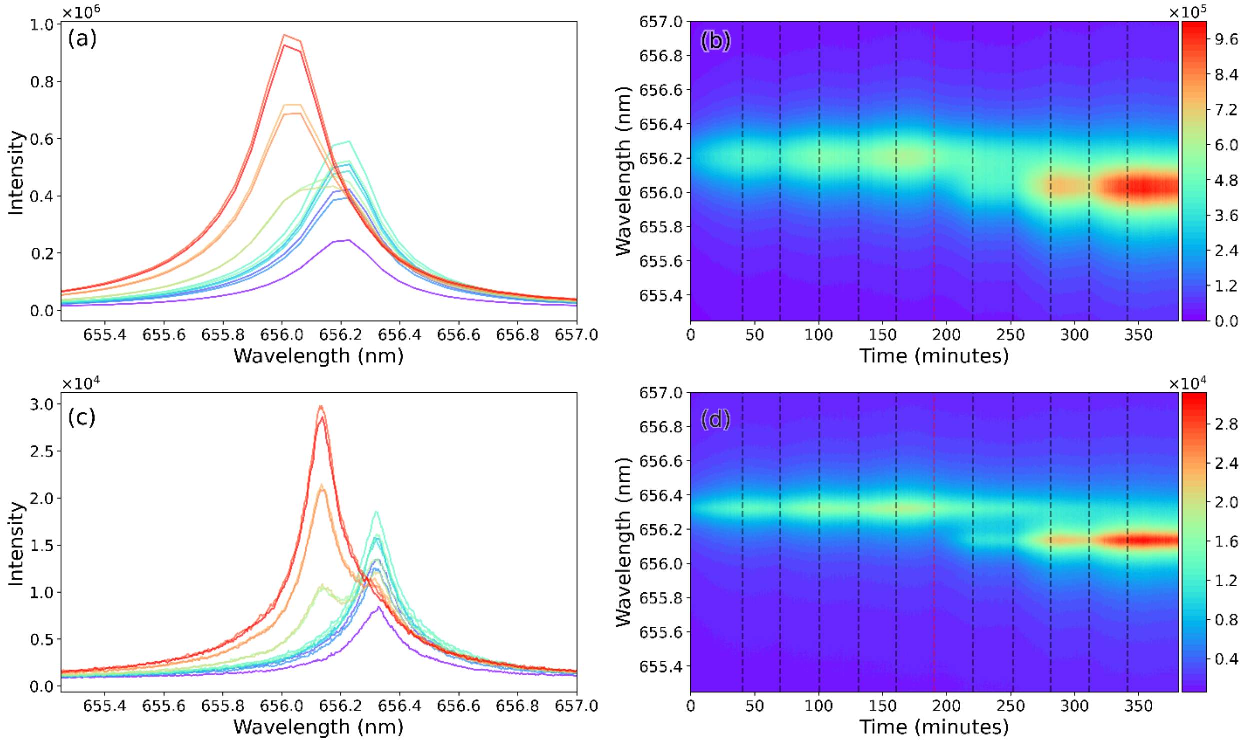

Figure 5. Time evolution of spectra of the H/D alpha line recorded during the first test on the (a,b) broadband MCS and (c,d) high-resolution DEMON. The corresponding surface maps in (b) and (d) show the shift in peak position over time as the composition changed from H to D. The vertical dashed lines in (b) and (d) show when sparge gas and flow rate were changed and correspond to the times that the spectra in (a) and (b) were recorded. The red vertical dashed line denotes the first addition of D_2. Spectra have not been normalized.

In general, emission intensity for a given analyte will increase with increasing concentration, and it is expected that the emission from D will have greater intensity than would an equal concentration of H, as seen in the spectra shown in Figure 5. The increase from 0 to 4% H_2 or D_2 in Ar had a notable effect on the Ar emission intensity due to dilution and changes in matrix. This effect has been described in the literature.[24] One approach to correct for these changes is to normalize the spectra to emission lines from one or more other elements. Normalization is challenging for the DEMON due to the narrow spectral window, as there are no other lines being tracked. Meanwhile, the broadband data of the MCS allow for more robust normalization. Regardless of the non-linearity exhibited in the spectral response, it presents no issue for the direct comparison of the spectra recorded on the two spectrometers. So, while the MCS spectra could be normalized to improve linearity for calibration, no normalization was performed to facilitate comparison between the two spectrometers.

Despite sparging the salt with pure Ar for 30 min and purging the LIBS sample chamber before the test, H was detected in the background measurements before sparging with the H_2 or D_2 mixtures. This is likely related to the trace H_2 impurities in the pure Ar gas stream, which is indicative of LIBS’ high sensitivity to light elements. Tests like the H/D sparging experiment demonstrate the feasibility of using LIBS not only for monitoring molten salt systems, but also for exploring gas–salt interactions and better understanding how gases will permeate through molten salts in a reactor system.

3.2.2 Xenon and Krypton Monitoring

Similarly to the H/D sparging tests, a noble gas mixture was sparged through the storage tank and monitored continuously using LIBS. Here, a pre-mixed gas containing 100 ppm Kr and 1000 ppm Xe in bulk Ar was bubbled through the salt in the storage tank initially at 0.3 SLPM and then raised to 0.9 SLPM at 0.8 h, and the off-gas was monitored by LIBS and RGA in series. The LIBS settings were the same as for the previous test with H isotopes, except a pulse energy of 150 mJ shot^{-1} was used to ensure sensitivity. Transient spectra collected from the noble gas sparging test are shown in Figure 6, along with trends in peak area over time. The trace Xe and Kr signals became distinguishable from noise in the LIBS spectra at approximately 0.2 and 0.5 h, respectively (Figure 6c). This was prior to either gas being detected by the RGA placed in-line and upstream. Additionally, LIBS was able to detect Xe which was beyond the mass range of the RGA. This highlights LIBS' sensitivity to noble gases, which are key signatures for off-gas systems. At 1.5 h, the noble gas injection was terminated and switched to pure Ar. The LIBS was then configured for an overnight run monitoring the noble gas depletion. This lengthy overnight monitoring while the noble gases are purged from the system demonstrates just a small portion of the extended LIBS monitoring performed during this testing campaign.

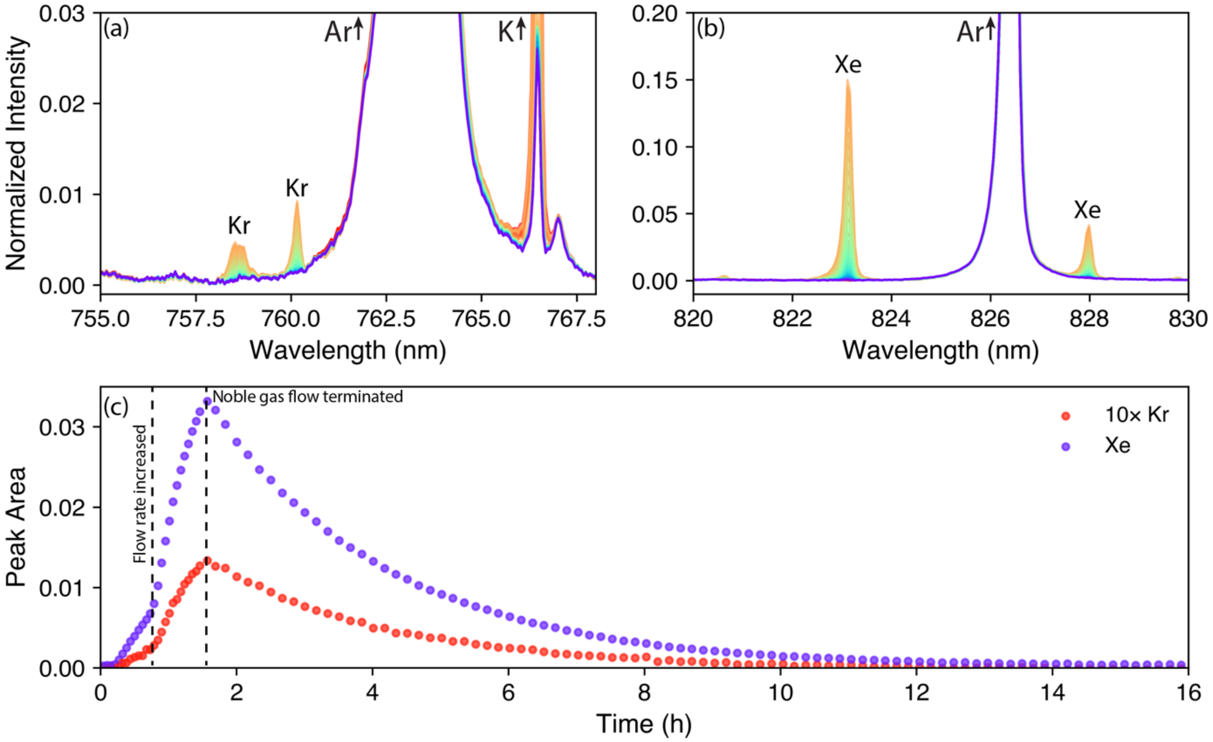

Figure 6. Time evolution of (a) Kr and (b) Xe recorded during the noble gas test on the FASTR storage tank as measured on the broadband MCS. The Xe and Kr peak area trends over time are shown in (c), where the Kr peak area is multiplied by 10 to account for the magnitude of concentration difference.

3.3 MONITORING SALT AEROSOLS FROM FASTR PUMP BOWL BY LIBS

3.3.1 Aerosol Monitoring by LIBS

The mobile LIBS cart was connected to a gas outlet sampling the headspace of the pump bowl on FASTR with a 6.5 m length of antistatic nylon tubing. Aerosols were produced within the pump bowl by agitation of the salt. Gas was added to the pump bowl at 0.15 or 0.3 SLPM through a separate inlet, driving a flow of aerosol-bearing gas through the LIBS via antistatic nylon tubing. The flow through the analyzers was maintained at a constant 0.3 SLPM. The broadband MCS was used to simultaneously monitor gases in the off-gas that are indicative of impurities (H and O) as well as salt aerosol signals corresponding to major salt species Na, K, and Mg, and the trace component Ca (0.4 wt%). The spectral windows used to monitor each of these elements are shown in Figure 7. The DEMON was used for various high-resolution measurements. The successful measurement of salt aerosol species at an extended distance from the sampling point on the salt loop demonstrates that a LIBS system for off-gas monitoring could also be used to track salt composition.

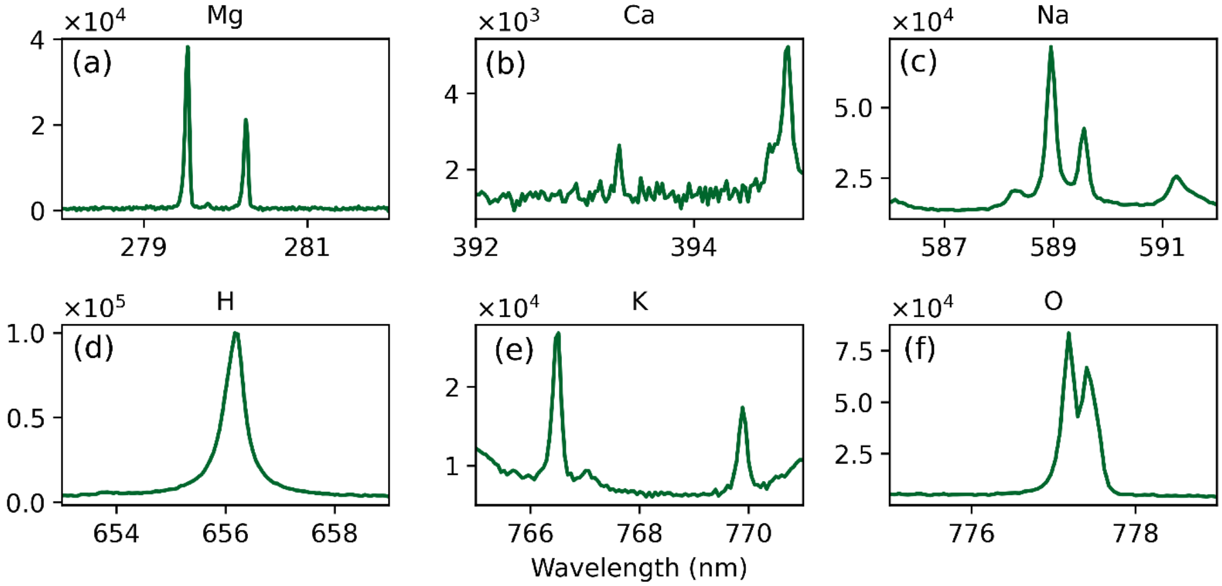

Figure 7. Spectral windows highlighting lines used for detection of (a) Mg, (b) Ca, (c) Na, (d) H, (e) K, and (f) O.

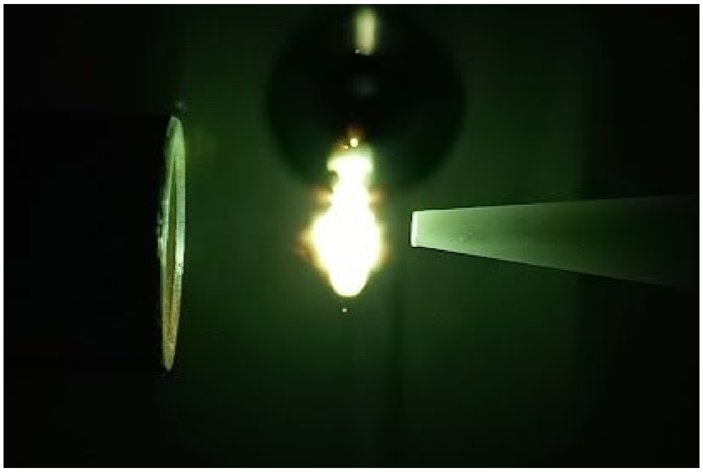

Figure 8. LIBS plasma containing salt aerosols from FASTR in Ar, where aerosols enter from the right through an aerosol focusing nozzle. The bright white emission is the continuum emission of the plasma and emission from Ar. The small orange flares are emissions from salt aerosol particles; the orange color comes from Na. The green tint is due to the color of the laser window on the LIBS chamber through which the image was captured.

4. CONCLUSIONS

Several objectives were achieved during the testing of LIBS apparatus in small-scale and larger-scale systems.

- LIBS has shown that sampling of the salt liquid can be achieved by spectroscopy on the aerosols generated during salt sparging. The quantity and size of the salt aerosols depend on agglomeration and deposition along the walls of the sampling line, a factor that is being considered in follow-on studies.
- LIBS has been shown to be sensitive to the isotopes of H, allowing tracking of H exchange reactions in the salt. Data from the LIBS measurements have been validated by an RGA mounted in series. This is important for understanding tritium transport in MSRs.
- LIBS is sensitive to noble gas detection (Ar, Kr, and Xe). Xe and Kr are important fission gases that will be present in MSR off-gas and need to be managed due to their impact on source terms.
- LIBS was more sensitive than the RGA instrument in detecting Kr. Note that Xe was beyond the mass range of the RGA and could not be compared between instruments.
- The high-resolution DEMON spectrometer offers better sensitivity than the broadband MCS, and the full-spectrum coverage of the MCS allows for simultaneous tracking of multiple species and more robust normalization. Both spectrometers are able to distinguish H isotopes.
- LIBS is a fast-responding technique, reflecting changes in species concentrations within 60 s, which will be vital for detecting changing conditions in an MSR.
- LIBS operated reliably over approximately 2 weeks of testing, including several overnight measurements and two weekend measurements.
- The mobile LIBS proved to be robust, allowing it to be moved from one facility to another. The optics did not require readjustment after the move.
- LIBS was able to detect air ingression into the system during a component swap-out. This demonstrates the ability to detect off-normal events.
- LIBS successfully made measurements at a considerable stand-off distance (e.g., 3–6.5 m) from the salt and gas space source. This result demonstrates the ability to make key measurements from outside the high-temperature and potential high-radiation fields of an MSR.

This study also demonstrated the use of FASTR as a user facility, incorporating instrumentation from PNNL and a small business, as well as accommodating a variety of sensors from ORNL: LIBS, RGA, and a cascade impactor. These measurements can be used to demonstrate the utility and sensitivity of instrumentation in an integrated facility. These tests have provided important information for future considerations when planning testing of LIBS instruments at potential off-site locations in the future (e.g., Abilene Christian University, Idaho National Laboratory, etc.).[25, 26]

5. ACKNOWLEDGMENTS

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