Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

SAVANNAH RIVER NATIONAL LABORATORY
OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

September 4, 2019 SRNL-L3310-2019-00011

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Attached is the report on "SRNL Report on Solicitation Vendor Instrumentation Demonstration and Selective Ion-Electrode." This report summaries the market survey of online instrumentation used to monitor process stream composition using commercial off-the-shelf instrumentation that could be implemented into the Hanford Tank Waste Treatment and Immobilization Plant.

Attachments:
Attachment A. SRNL Report on Solicitation Vendor Instrumentation Demonstration and Selective Ion-Electrode Testing
Attachment B. ERCo. LIBS Analysis of Melter Feed Materials

Cc: C. C. Herman, A. D. Cozzi, J. Manna

Attachment A:
SRNL Report on Vendor Solicitation and Selective Ion-Electrode Testing

Introduction

Effective operation of the Hanford Tank Waste Treatment and Immobilization Plant (WTP) will require information collected from sampling and/or monitoring to maintain environmental compliance, product quality, and operating efficiency. The U.S. Department of Energy (DOE) – Office of River Protection (ORP) recognizes the potential long-term benefit of developing real-time, in-line monitoring (RTIM) at the WTP to improve the current strategy of collecting tens of thousands of samples and analyzing them. In-line monitoring can decrease processing time associated with waiting for sample analysis, reduce workers' exposure to radiation and chemical hazards, decrease secondary waste volumes, potentially improve product quality, and provide production support and enhance overall operational control.

The Assistant Manager, WTP chartered an Integrated Technical Team (ITT), which includes personnel from the Savannah River National Laboratory (SRNL), Bechtel National, Inc., and DOE-ORP, to evaluate the capability and potential benefits of replacing routine sample collection requirements with real-time, in-line monitoring[1,2]. The initial focus of the ITT is the Direct Feed Low Activity Waste (DFLAW) Program. This work scope is being performed in multiple phases. Phase 1 is Requirements and Opportunities Analysis, Phase 2 is Applications and Benefits Determination, and Phase 3 is Qualification and Demonstration.

Phase 1 included 6 tasks[3] and was completed by SRNL in 2017. The tasks in Phase 1 were: (1) determining the functional requirements for determining analytes and properties of streams, (2) determining the technical basis for process control, (3) determining the sampling points within the Low Activity Waste (LAW) facility and Effluent Management Facility (EMF), (4) data quality and management, (5) process control challenges, and (6) preparing a Preliminary Analysis Plan[4-7]. DOE-ORP was briefed on the findings from the Phase 1 tasks and requested that Phase 2 work focus on establishing a basis for a materials balance approach that minimized process sampling[8].

The Phase 2 work scope contains tasks to evaluate a materials balance only approach as well as continuing scoping evaluations for instruments that could be used for RTIM[3]. The Phase 2 work scope was completed by both SRNL and Pacific Northwest National Laboratory (PNNL). The tasks performed in Phase 2 were:

1. Overall program administration and technical oversight
2. Evaluation of a Material Balance Approach
    a. Evaluation of uncertainty of this approach compared to uncertainty of current methods
    b. Evaluation of automating the material balance within the framework of existing control software
    c. Development of the methodology to obtain and evaluate data during cold commissioning to support a material balance approach
    d. Development of a framework to utilize during presentation of the revised approach to stakeholders and regulators
3. Screening Evaluations of Instruments to Supplement the Material Balance Approach

   a. AP-105 melter condensate evaluations
   b. Screening testing with simulants
4. Evaluation of the current DFLAW feed qualification program to identify analytes that could be eliminated
   a. Inorganic analytes
   b. Radionuclides
   c. Organic analytes

This report describes the results of Task 3b, Screening testing with simulants.
The Savannah River National Laboratory requested demonstrations of commercially available analytical instruments by the vendors or laboratory demonstration. The techniques tested included: Raman spectroscopy, Infrared spectroscopy (IR), laser induced breakdown spectroscopy (LIBS), X-Ray Fluorescence Spectroscopy (XRF), and ion-selective electrochemical probes. Additionally, the instrumentation would need to be durable enough to withstand the environment of the plant, preferably with little to no maintenance. The insertion point of the instrument would be dependent upon the instrumentation.

Analytical Methods and Commercial Instrumentation

X-Ray Fluorescence Spectroscopy (XRF)

X-Ray Fluorescence Spectroscopy (XRF) is a non-destructive analytical technique that can be used to determine the elemental composition of a material. The technique excites a sample with an X-Ray then measures secondary X-Rays that are emitted during electronic relaxation; the emitted X-Rays are unique to each element. The excitation X-Ray interacts with the core electrons of the element, so this technique is not limited by speciation of the element, and likewise couldn't be used to determine speciation directly. Heavier elements, which have more core electrons, generate a larger signal and are more readily measured than lighter elements. Most vendors will claim to be able to measure elements as light as boron, but in more complex systems the signal for larger elements interferes with the signal of the lighter elements. Typically, XRF results are given in relative concentration and not absolute concentration, therefore this technique would be useful for monitoring streams for variations in relative composition and wouldn't replace sampling.

Three vendors were found to have viable XRF instruments capable of monitoring solid compositions or slurry compositions. The vendors in Table 1 were approached because they advertise instrumentation that would serve the needs of the RTIM program.

The vendors in Table 1 did not provide a demonstration. For the most part, they provided good communication about their capabilities, however seemed to lose interest when the application was specified. Their sales staff communicated that the market was either too specialized or not large enough to further develop the necessary technology to suit the needs of the program.

Table 1. XRF vendors, instruments, and capabilities.

Vendor/Company	Instrument Line	Capabilities
Hobre	C-Quand	• Liquid streams containing elements from Silicon to Uranium. • PPM level detection • Pass through sample cell • 15 elements with a single analyzer
FL Smith	EDXRF Slurry Analyzer	Elemental analysis in slurry streams.
Thermo	MEP-300 Analyzer	• Elemental Analysis of solid and slurry streams. • Applications in chemical and mining industries.

Laser Induced Breakdown Spectroscopy (LIBS)

Laser induced breakdown spectroscopy (LIBS) is a type of optical emission spectroscopy where the sample is subject to a short laser pulse and the emitted light spectrum is collected. The emitted spectrum is unique to the elemental composition. LIBS can provide elemental compositions to an accurate degree and laboratory LIBS system is planned for the WTP laboratory for elemental analysis. The technique has some advantages such as the laser pulse delivery options and signal detection options which can be performed remotely. Signal analysis algorithms can deconvolute complex spectra from multicomponent systems.

The vendors listed in Table 2 provide commercially available LIBS instrument or are developing LIBS instrumentation that could possibly meet the needs of the program. Samples of glass forming chemicals were shipped to the vendors for demonstration of the instrument's capabilities.

Table 2. Laser Induced Breakdown Spectroscopy Instrumentation Vendors

Vendor/Company	Instrument Line	*In situ* Measurement Capabilities
Energy Research Company (ER Co.)	Customizable Probe	Solids analysis Liquid analysis Slurry analysis
l'Emission		Slurry analysis
Orano		Solids analysis

The demonstration performed by ER Co. in conjunction with Los Alamos National Laboratory has been selected to further develop instrumentation for the WTP. The results from this demonstration can be found in Attachment B of this memo.

The other vendors did not demonstrate technology more mature than the ER Co. nor were they able to demonstrate *in situ* monitoring that would be required for the use in the plant for process control. ER Co. also has extensive experience in adapting LIBS to operate in a variety of environments. Their demonstration repurposed a probe from another project that could be similar to what is designed for use in the WTP. Their demonstration used a repurposed probe coupled with demonstration analysis software to show a proof-of-concept. The data quality of this demonstration easily meets the needs of the program at this phase. The instrument was able to track and identify increases in certain elements with acceptable accuracy.

Raman and Infrared Spectroscopy

The Georgia Institute of Technology School Chemical Engineering (Ga Tech) was funded by the Department of Energy (DOE) through the Consortium for Risk Evaluation and Stakeholder Participation (CRESP) to explore technology related to departments missions. Using this funding Ga Tech purchased a Mettler-Toledo REACT-IR probe and an accompanying Raman probe. Their demonstration of the technology and the chemometric development has been presented elsewhere.[9] The instrument analysis and algorithms used for data analysis have shown the ability accurately make *in situ* measurements of the multi-component systems. This is proving to be a successful program.

The approach of university involvement has demonstrated a very mutually beneficial process as well. Purchasing commercial instrument by a university reduces the start-up costs and schedule associated with procurement and installation of new instrumentation at a DOE facility, i.e. lower cost and shorter schedule. At DOE facilities analytical instrumentation is governed by nuclear safety and quality assurance requirements for installation and operation that often lengthen the schedule and elevate the cost of the instrument for initial demonstration. Additionally, demonstration models are often not considered because vendors are not willing to risk the loss of an instrument if it were contaminated by radioactivity. Even though simulated samples could be used, laboratory space is not always accessible to offsite vendors for onsite demonstration and sending simulated samples to vendors are met with the results discussed earlier. If the instrumentation fails to meet DOE needs and requirements, there may still be a recovery of the investment for a university for future projects. Top tier research universities, like Georgia Tech, have the capability to modify, develop, and demonstrate instrumentation with upgrades as part of their research and development program for applications in other areas. By working with a university, the risk on the investment is lowered and the return on the investment goes beyond financial point of view. The university develops experts through their process in both the students and researchers that install and test the instrumentation. Later, their expertise gained in the project make them excellent candidates for national lab staff and collaborators for other projects.

Ion-Selective Electrodes

Ion selective electrodes (ISEs) measure the change in electrochemical potential of a specific ion in solution and relates it to the concentration of the ion using a Nernstian relationship. Testing was performed at SRNL to evaluate an ISE for chloride (Cl^-) measurement. The electrode was manufactured by Cole-Parmer and is primarily intended for laboratory use; however, the testing was aimed to determine whether the electrode was robust and stable enough to be developed for measurements in a process.

Chloride specific ISE.

Dissolved Cl^- can potentially be monitored in-line using an Ion Selective Electrode (ISE) but some preliminary proof-of-concept studies must be performed using some appropriate simulant solutions. A description of Ion Selective Electrode or ISE specification is provided in Figure 1 and Table 3.

Figure 1. Image of a Cole-Parmer Ion Selective Electrode.

Table 3. Table of Electrode Specifications from Cole-Parmer Manual (2013).

Variable	Value(s) or Relevant Parameter(s)
Concentration Range	1 M to 5.0 x 10^{-5} M Cl^- (35,500 to 1.8 mg L^{-1} Cl^-)
pH Range	2 to 12
Temperature Range	0° to 80°C
Resistance	<1 Mohm
Reproducibility	± 2%
Solution Composition	For aqueous solutions only; no organic solvents
Size:	11.0 cm length, 1.2 cm diameter and 1 m cable length
Measurement Error	2% for every change in degree Celsius
Interferences	NH_3, NH_4^+, OH^-, S^{2-}, CN^-, I^-, Br^-, and $S_2O_3^{2-}$
	Bi^{3+}, Cd^{2+}, Pb^{2+}, dissolved Mn, Sn and Tl (These cations complex with Cl^- and alter its free ion concentration).
Other	Ionic strength of standard must be reasonably matched with that of the target test solution—particularly at ionic strengths exceeding approximately 1 mMeq L^{-1}.

The Nernst Equation describes the performance of the ISE as shown below:

$E = E_o - S \log X$

where: E is the measured potential; E_o is the reference potential; S is the slope (56 mV decade^{-1}); X is the activity of the Cl in solution.

It should be noted that the activity of the Cl^- is a function of the ionic strength and the free (un-complexed) Cl^- concentration. Therefore, Initial scooping comparisons are best made at a near constant ionic strength.

Results and Discussion:

Use of the electrode began with measurement of a "blank" or background solution of 0.2 M $NaNO_3$ followed until the multimeter indicated the solution measurement had stabilized. This usually took 1 to 2 minutes depending on the stability of the room temperature. The standard solutions were measured, and a calibration curve was generated.

On the first day of using the ISE the calibration results were in variance to the vendor manual but subsequent use of the electrode over a period of a few days resulted in measurements that were much closer to that of the manual. The most important variable that influenced the calibration of the electrode was room temperature. During the time that these measurements were made, the room temperature varied due to a large outdoor diurnal temperature as well as events such as changes in room hood ventilation (Figure 2). New calibrations were performed after 2 hours of use with the effluent management facility (EMF) test solutions as recommended by the vendor. The EMF test solution is listed in Table 4.

Table 4. Composition of the EMF Simulant Solution.

Chemical	Chemical Formula	Target Final Molarity (M)
Potassium fluoride	KF	0.0216
Sodium chloride	NaCl	0.0047
Ammonium nitrate	NH_4NO_3	0.0114
Ammonium sulfate	$(NH_4)_2SO_4$	0.0049
Sodium sulfate	Na_2SO_4	0.0068
Potassium sulfate	K_2SO_4	0.0126
Ammonium chloride	NH_4Cl	0.0438
Boric acid	$B(OH)_3$	0.0849
Zinc nitrate	$Zn(NO_3)_2$	0.0013
Sodium oxalate	$Na_2C_2O_4$	0.0006

Seven measurements of each EMF simulant solution were made at consecutive five-minute time periods. The temperature and absolute mV readings on the multimeter and the time were recorded. The modified EMF solution was measured initially and then known amounts of the modified EMF were diluted with 0.2 M $NaNO_3$ and the seven measurements were repeated etc. [This type of dilution process permitted a wide range of Cl^- levels to be analyzes while keeping the sample matrix somewhat similar although it was diluted.] Differences in the ratio of NH_4^+:Cl^- were examined by spiking the modified EMF solution with known amounts of NaCl. The NaCl-spiked EMF solutions were then diluted with 0.2 M $NaNO_3$ and the various dilutions were analyzed by the ISE for Cl^- content in absolute mV.

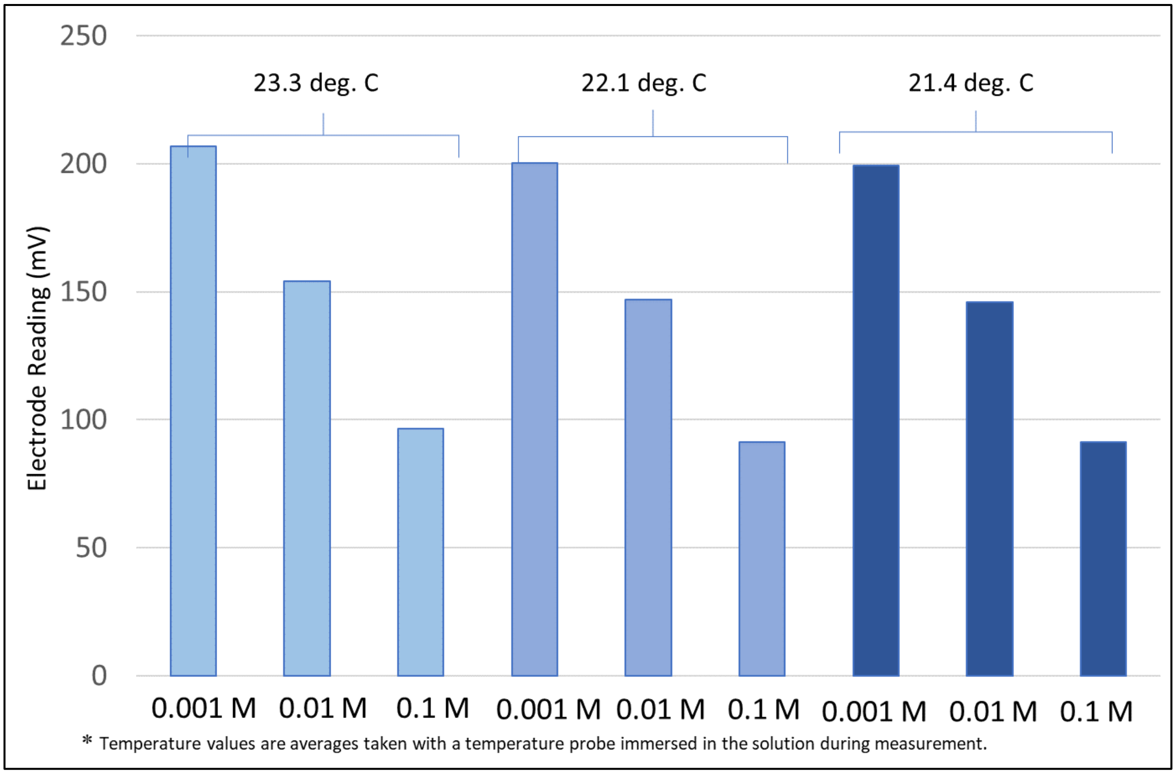

Figure 2. Effect of Solution Temperature on Cl^- Measurement.

On occasion during the sample measurements, there were rapid and pronounced changes in temperature due to alterations in room ventilation (see Figures 3 and 4). In other cases, addition of extra NaCl to the modified EMF was made to test the effect of known levels of NH4+:Cl- on Cl- measurement with the ISE and the make-up of these solutions generated a slight increase in temperature that was detected with the temperature probe. This change in temperature also influenced the mV reading on the multimeter but in all cases, after the various consecutive measurements of the stirred solution, the mV reading for the modified EMF solution became stable providing the temperature was also stable. The influence of a known but varied amount of NH4+:Cl- in the test solutions was insignificant relative to differences in room temperature. This can be observed when the same solution was measured on two different days with the electrode (Figure 5).

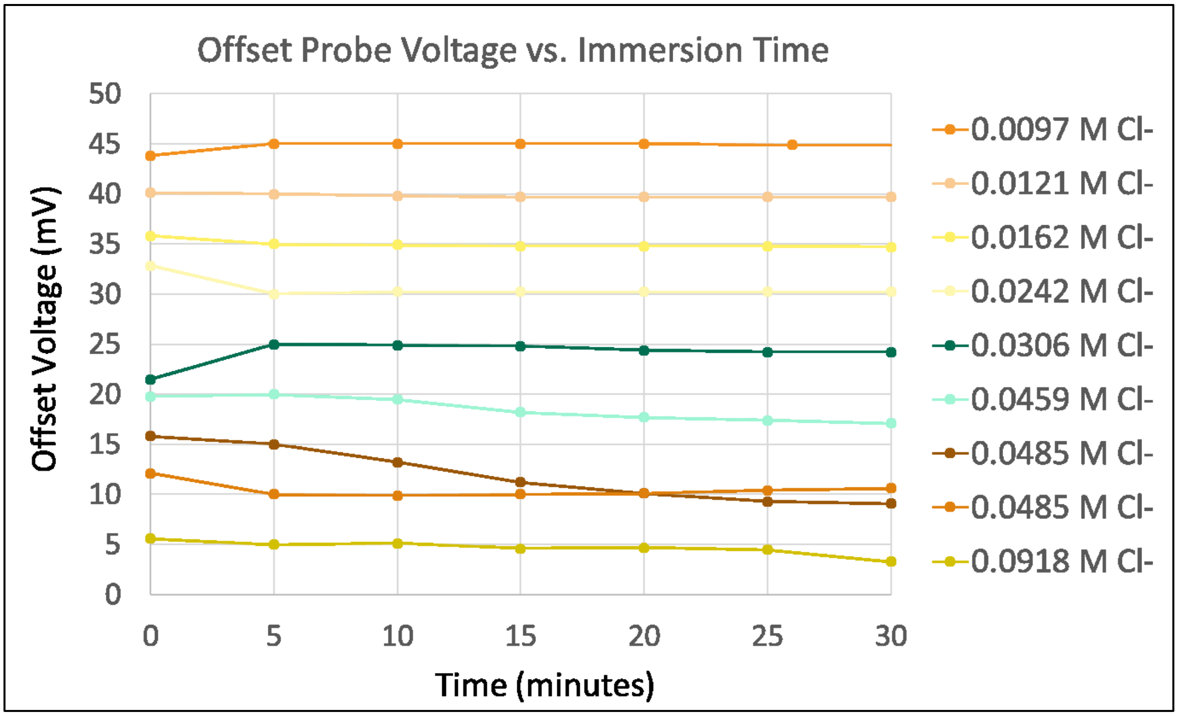

Figure 3. Measurement Temperature with Immersion Time.

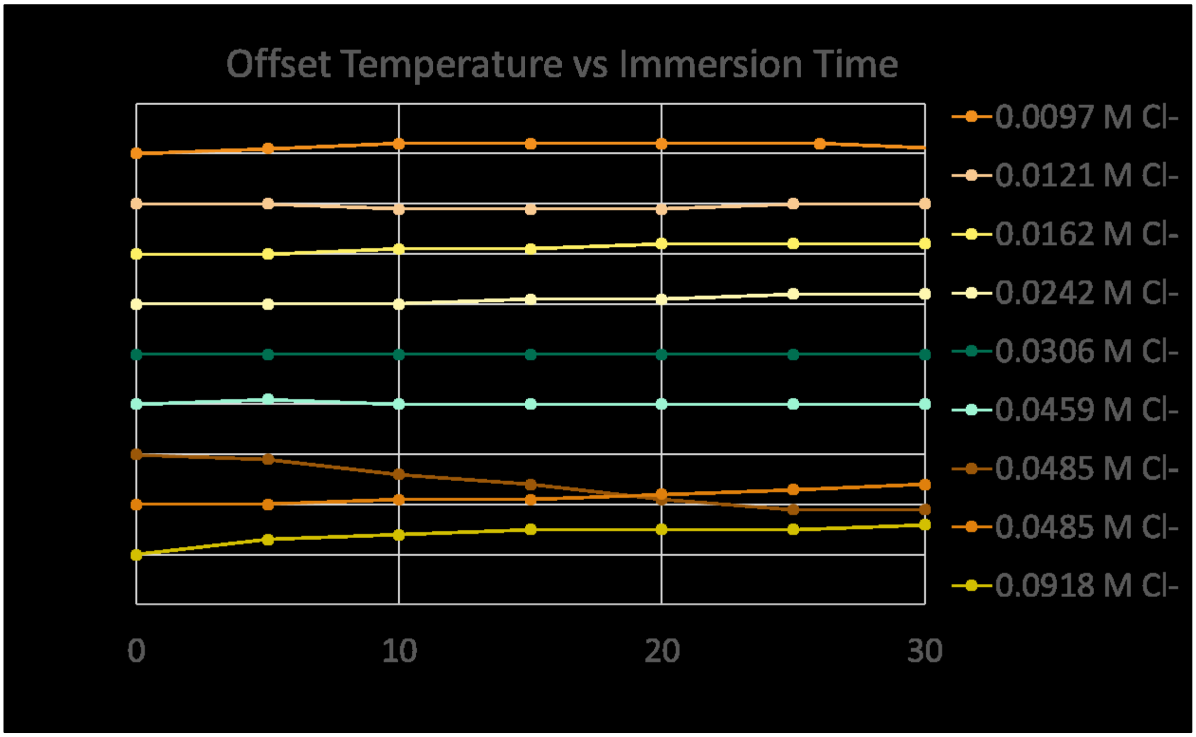

Figure 4. Measurement Stabilization with Immersion Time.

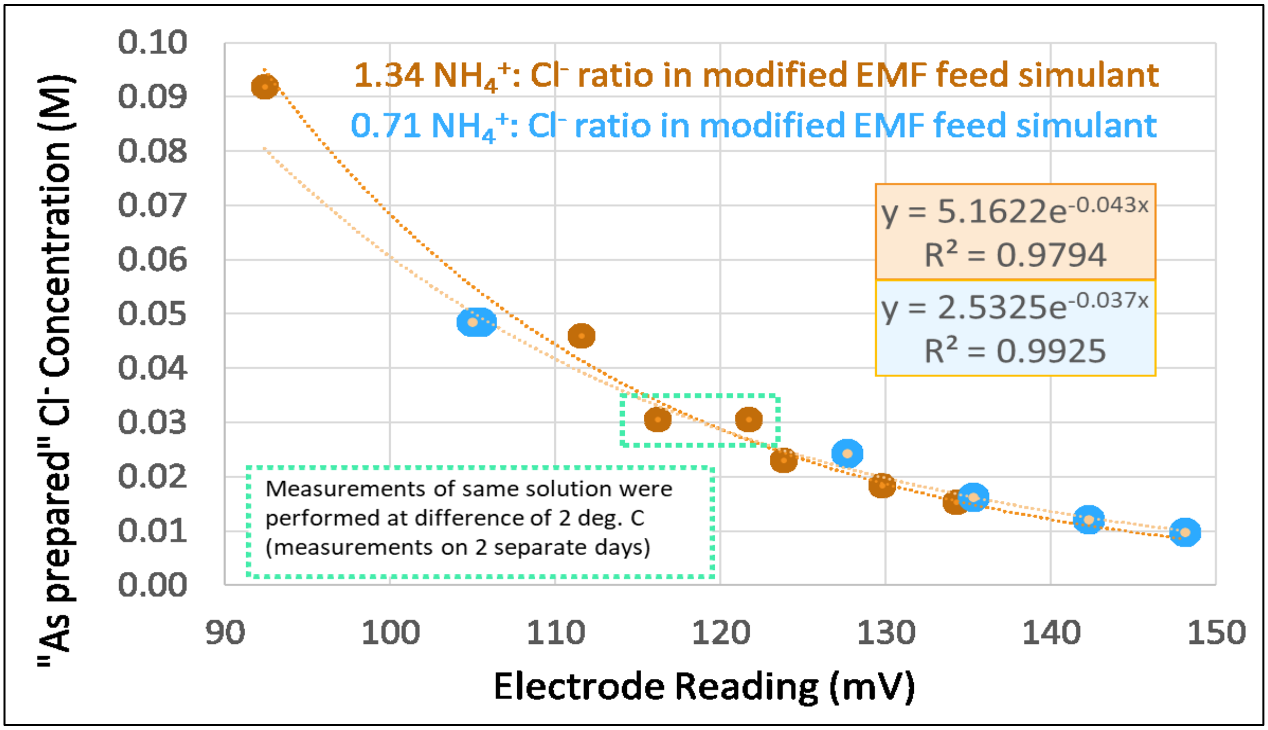

Figure 5. Effect of Solution NH$_4^+$:Cl$^-$ Ratio on Electrode Performance.

These proof-of-concept studies indicate the ISE appears viable for measurement of Cl$^-$ in the EMF feed. There are additional points of discussion that should be noted regarding its suitability for RTIM with EMF feed:

1) The vendor reports that the ISE is fragile, but no damage occurred upon receipt or during three weeks of intermittent use with the epoxy electrode,
2) The actual calibration frequency would need to be evaluated during any implementation testing,
3) The final calibration protocols would be based on testing in prototypical system,
4) Use of a temperature-controlled cell would be helpful during implementation testing, and
5) The vendor states that electrode life in the lab is 6 months but in-line use expected to a shorter duration of a few months and performance deterioration exhibited by an increased response time and decreasing slope of calibration.

There are only a few methods that present potential for use in in-line Cl$^-$ or Cl quantification. Other potential methods include laser induced breakdown spectroscopy or LIBS.

Conclusions:

There are several analytical methods and instruments available for real-time in-line monitoring the stream contents of Waste Treatment Plant process. Few instruments are technically mature enough for the level of quality required for control in a nuclear processing plant. The ERCo LIBS system has instrumentation and innovative methods that will meet the needs for the RTIM program. The evaluation and chemometric studies of the Mettler-Toledo REACT-IR probe and an accompanying Raman probe performed at Georgia Institute of Technology has shown to have great promise using their data analysis algorithms. This project has also demonstrated how programs like CRESP add to

Department of Energy since their initial involvement was through that program. The conclusion of this phase should be to continue to develop these methods to a final technical readiness level.

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3. M.E. Stone, "Task Technical and Quality Assurance Plan for Hanford DFLAW Real-Time, In-Line Monitoring Program," Savannah River National Laboratory, Aiken, South Carolina, SRNL-RP-2016-00704, Rev 1, 2018.

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6. M.E. Farrar, M.P. Poirier, A.M. Howe, C.C. Diprete, and M.E. Stone, "WTP Real-Time, In-Line Monitoring Program Task 3: LAW and EMF Flow Sheet Sampling Points," Savannah River National Laboratory, Aiken, South Carolina, SRNL-RP-2017-00241, Rev 0, 2017.

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9. S. Kocevska, M. A. Grover, R.W. Rousseau, "Monitoring the Composition of Low-Activity Nuclear Waste using In-Situ Measurements," Waste Management Symposium, 2019, paper 19356, Phoenix, AZ.

Attachment B.

ERCo. LIBS Analysis of Melter Feed Materials

SRNL-L3310-2019-00011

ERCo **ENERGY RESEARCH COMPANY**

LIBS Analysis of Melter Feed Materials
Interim Progress Report

18 September 2018

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Executive Summary

Energy Research Company (ERCo) investigated using its OnSpec probe for measuring the composition of melter feed materials sent by Michael Stone of SRNL. The OnSpec probe uses laser-induced breakdown spectroscopy (LIBS) to determine the concentrations of elements in materials, which can be solids, liquids, or gases. Benefits of OnSpec technology for the melter feed application include standoff measurement capability, so the materials do not need to come into contact with the probe to conduct a measurement, and a ruggedized and safe design for industrial environments.

Preliminary experiments were performed in ERCo's laser spectroscopy laboratory with showed the ability to measure multiple elements in the melter feed powder. These elements included aluminum, boron, calcium, iron, lithium, titanium, zinc, and zirconium. Importantly, all these elements were measured simultaneously, which will allow for frequent and rapid measurements. The next set of experiments will demonstrate the ability of the LIBS system to measure these elements in the melter feed slurry.

1 Introduction

ERCo has developed LIBS-based process controls sensors for industry since 1999. These sensors are used to monitor material composition for process and quality control in a variety of industries, including aluminum and other metals, glass, and coal. The newest model of ERCo's LIBS sensor for molten aluminum is called OnSpec, and is seen in Figure 1. It consists of a sensor head attached to a 5' long tube assembly (only partially shown in the figure) whose forward end is immersed in the molten aluminum. The long tube length allows for installing the probe through a furnace wall, keeping the sensor head safely outside the furnace. The sensor head is attached to a climate controlled instrument cabinet via electrical and fiber optic cables. The system's spectrometer and controlling computer as well as the operator's controls are in the cabinet. The system returns a complete elemental analysis of the molten aluminum approximately every 5 minutes. It is operated via a simple point-and-click interface and requires no knowledge of spectroscopy and no laser safety training or equipment (i.e. it is a Class 1 device).

Figure 1: Rendering of OnSpec LIBS Probe for Molten Aluminum
A ceramic tube is attached to the end of the probe for immersion into molten aluminum.

The OnSpec design is well suited for the monitoring the SRNL melter feed composition. The long standoff distance will allow for mounting the sensor on the outside of the melter feed preparation vessel and/or the melter feed vessel, with only the tube assembly inside the vessel and exposed to the radiation field. The investment in the sensor head and instrument cabinet (the vast majority of the system's cost) is therefore preserved and maintenance is simplified.

While the forward end of the OnSpec molten aluminum probe is immersed in the molten aluminum during operation, that does not need to be the case for the melter feed application. If the slurry is well mixed so that the surface composition is representative of the bulk, then the tube assembly is not needed and the probe can simply fire through the air.

Similarly, the OnSpec probe can be used to monitor the composition of the powder in the GFC feed hopper. The top surface of the mixture in the hopper can be analyzed, or a custom tip can be engineered for the tube assembly to allow for subsurface measurements.

In order to demonstrate the suitability of the OnSpec design for the melter feed application, LIBS experiments were performed on a sample of the GFC powder with the OnSpec probe components. The next set of experiments will be done on the slurry sample that was also provided to ERCo. These experiments are designed to show that the concentrations of elements in the GFC powder can be measured with the OnSpec probe.

Measuring the composition of the supernatant is also of interest to SRNL in addition to the GFC components. While the molecular composition of the GFC components can be derived from the elemental measurements of elements such as aluminum, titanium, and zinc, this is not the case with the supernatant. Raman spectroscopy, another laser spectroscopy method, is much better suited to measure the molecular composition of the supernatant. Because the LIBS spectrometer can also be used to measure Raman spectra, future work will include developing a design to incorporate Raman measurements of the supernatant into the OnSpec design without adding the significant extra cost of a second spectrometer.

2 Experimental Procedure and Results

The optics, laser, and spectrometer used in the OnSpec probe were mounted on a laboratory optical table and a metal coupon was placed at the focal point of the optics, approximately 5' from the sensor head. Double sided tape was affixed to the coupon, and a thin layer of the GFC powder was deposited uniformly on the side of the tape facing the probe. A total of 100 LIBS spectra were collected from the powder by raster scanning the coupon. These spectra were averaged together and examined for peaks from the elements of interest.

Table 1 contains the composition of the GFC powder provided to ERCo together with the elements that can be used to measure each component's concentration in the mixture. The only exception is glucose, which will need to be measured by Raman spectroscopy. Figure 2 contains plots of peaks of all the elements from a 100 spectra average. As is seen in the plots, all the peaks have very high signal-to-noise and signal-to-background ratios, which facilitate accurate concentration measurements.

In order to measure concentrations of the elements from the LIBS spectra, the peak intensity (either the background-subtracted peak height or peak area) for an element is correlated to the known concentrations of that element from samples with varying concentrations of the element. These data are used to construct a calibration curve that is used to determine the concentration of the element in samples with unknown composition. The only exception to this among the elements in the GFC powder is silicon. Since silicon is present in more than one powder component, a multivariate calibration will need to be used. ERCo has a wealth of experience in this area, applying algorithms such as principal components analysis and partial least squares regression to measure concentrations in complex mixtures.

Table 1: GFC Components and Elements Used to Measure their Concentrations

Glass Forming Chemical Components	Mass (g)	Element
Kyanite Raw (Al2SiO5) 325 Mesh	13.93	**Al**
H3BO3; Boric Acid Technical Grade	30.48	**B**
Wollanstonite NYAD 325 Mesh	12.99	**Ca**
Fe2O3; Iron III Oxide 99.9% Metal Basis	7.82	**Fe**
Lithium Carbonate	5.27	**Li**
SiO2 (Sil-co-Sil Ground Silica Grade 75)	54.59	**Si**
TiO2; Titanium IV Oxide, Rutile, 99%)	2.12	**Ti**
ZnO, ACS 99%	5.27	**Zn**
Zircon ZrSiO4 (Flour) Mesh 325	5.16	**Zr**
Sucrose	8.92	**Raman**

Since only one GFC powder was provided to ERCo, we altered the composition of the powder to demonstrate sensitivity to concentration changes. In the first experiment we added increasing amounts of powdered alumina (Al_2O_3) to the GFC powder and measured changes in an aluminum peak. The results are shown in Figure 3. Each concentration was measured three times. The closely overlapping data points in the plot are evidence of the high repeatability of the measurement. The linear trend in the data will lead to highly accurate concentration measurements from samples with unknown aluminum concentration.

In order to demonstrate that LIBS peaks for the other elements are also similarly sensitive to concentration, we performed an additional experiment involving diluting the powder with increasing amounts of silica. The results are seen in Figure 4 and Figure 5. The highly linear responses show the promise of the OnSpec probe for measuring the concentrations of the GFC components accurately. It should be noted that the OnSpec probe measures all these elements simultaneously, greatly increasing the frequency that measurements can be done.

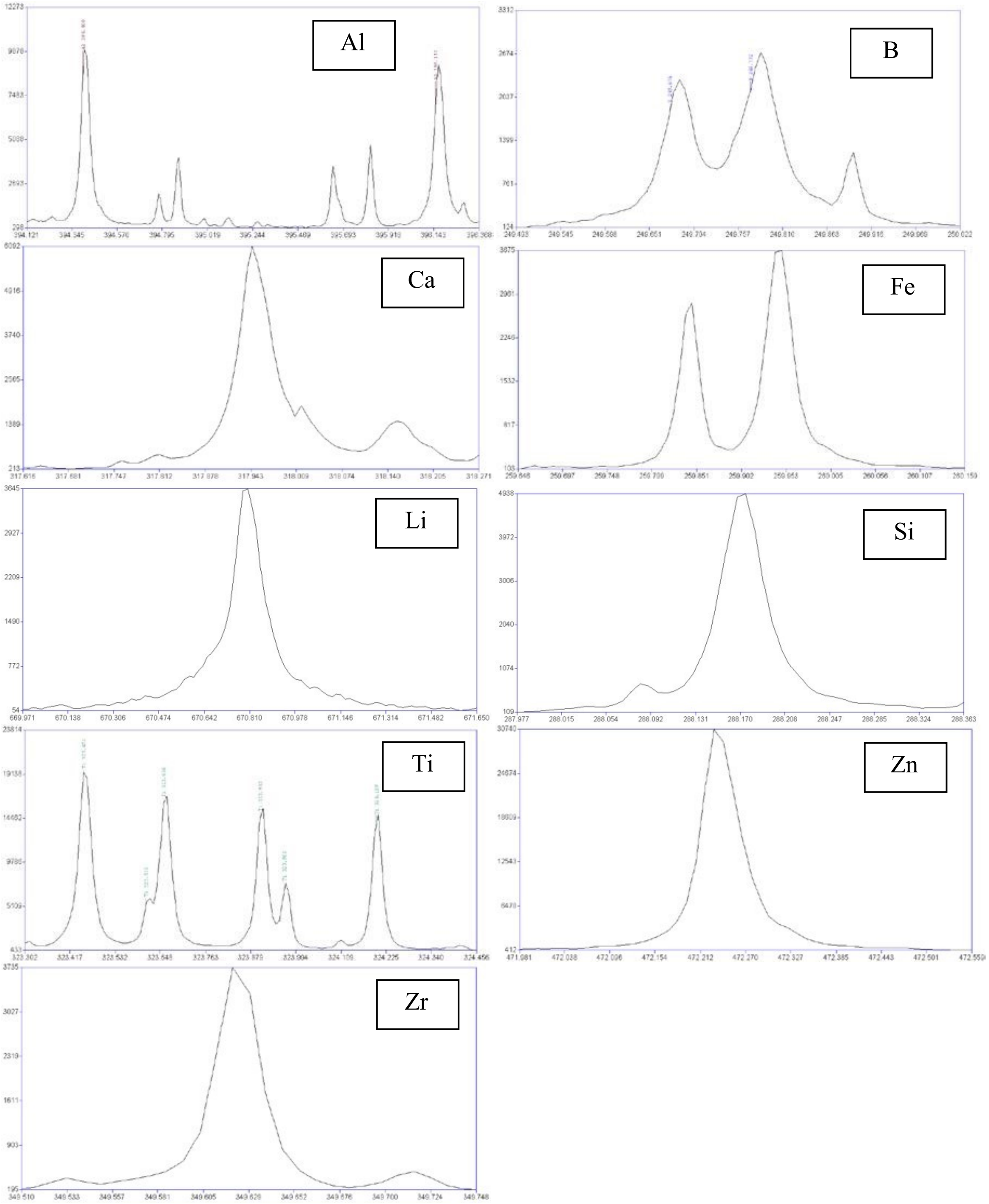

Figure 2: Plots of Peaks from GFC Powder X-axis is wavelength (nm) and Y-axis is intensity (counts).

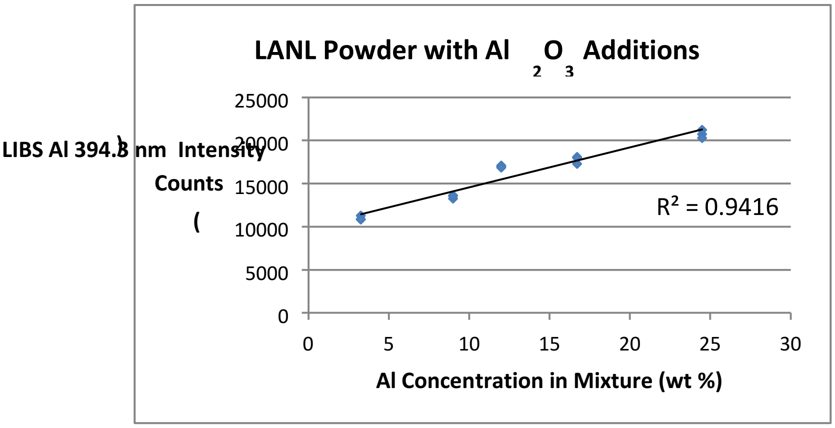

Figure 3: Calibration Curve for Aluminum with Al_2O_3 Additions Each concentration was tested 3 times.

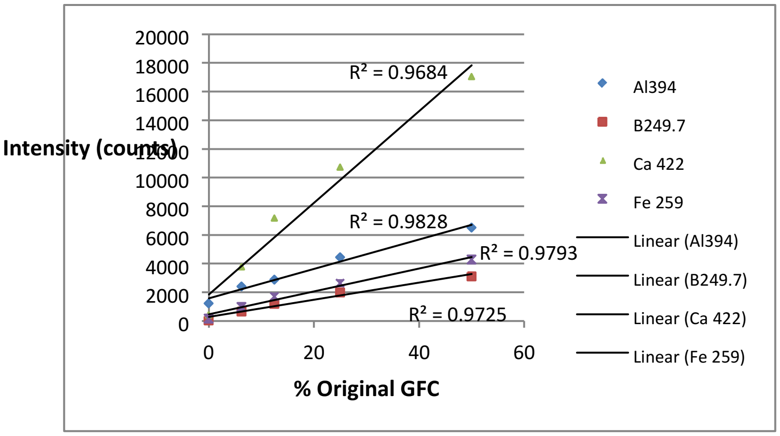

Figure 4: Calibration Curves for Al, B, Ca, and Fe with SiO_2 Dilutions

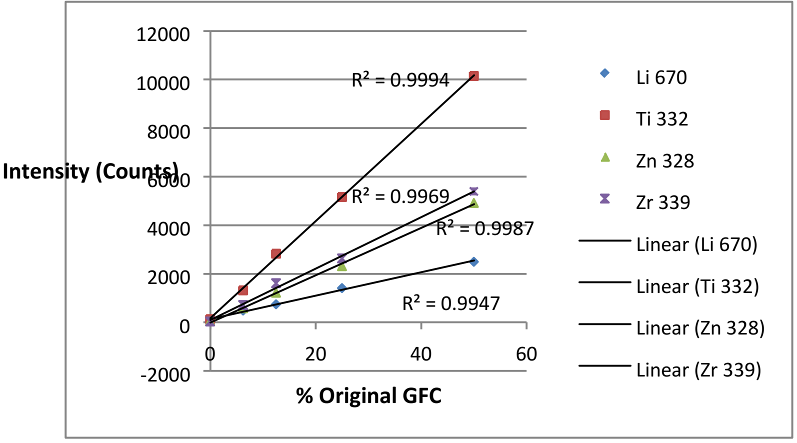

Figure 5: Calibration Curves for Li, Ti, Zn, and Zr with SiO_2 Dilutions

3 Next Steps

The slurry sample will be analyzed next with the OnSpec system. In order to do this analysis, we will constantly stir the slurry with a magnetic stirrer to ensure it is homogenized during the LIBS measurements. Because the sample is small, we will only be able to analyze the surface of the slurry, which is another reason to ensure that the slurry is very well stirred. We will document the peak heights of the elements of interest, as was done above with the GFC powder.

4 Conclusion

The ability of the OnSpec system to quantitatively measure elements of interest in the GFC powder was shown in a series of experiments. With further refinement of the technique, such as optimizing the laser energy, the spectrometer parameters, and the choice of spectral peaks used for analyses, we expect to achieve higher sensitivity and accuracy in the future.

We will next demonstrate the ability of the OnSpec system to measure peaks for these elements in the slurry.

A final report will then be submitted together with a proposal containing a work plan for further development. The report will include LIBS measurements of the slurry and a plan for utilizing Raman spectroscopy for measuring the supernatant components.