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DOE Bioenergy Technologies Office (BETO) 2023 Project Peer Review

Integrated LIBS-RAMAN-AI System for Real-Time, In-Situ Chemical Analysis of MSW

April 4, 2023

Technology Area Session: Feedstock

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Project Overview

ENERGY RESEARCH CENTER

- **Current State of Technology (SOT)**
  - Municipal Solid Waste (MSW) is a very heterogeneous material, which makes it difficult for sampling and analysis.
  - No ready-to-use rapid/real-time measurement techniques are available for MSW applications.
  - Critical review of real-time methods assessed for MSW quality monitoring:
    - Most techniques can only measure a limited number of parameters.
    - **Technologies need additional development to fully adapt them to the MSW environment.**
    - **New algorithms are needed for accurate measurements and automatic material identification.**
  - MSW processing presents similarities to other industries which could improve material management by using advanced analytical technology.

Project Overview

❑ Project Overall Goal

Improve throughput of characterization technology with a minimum target of 25% improvement over baseline characterization technology.

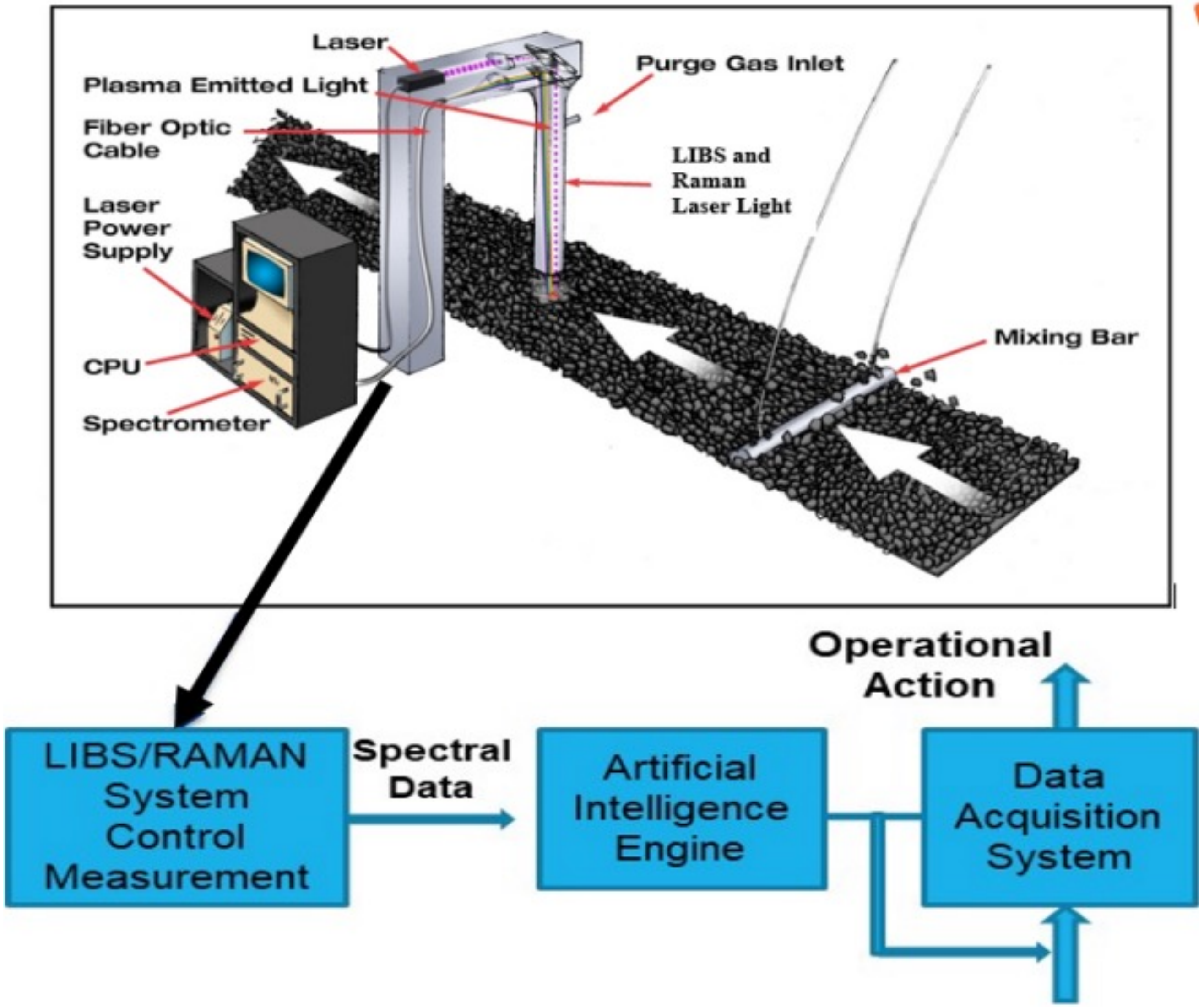
❑ Alignment with BETO FOA Goals

- ❑ Characterization of MSW to enable production of conversion-ready feedstocks
- ❑ Development of novel methods for rapid/real-time measurements
- ❑ Rapid analysis technology in conjunction with Artificial Intelligence (AI)
- ❑ Characteristics to be resolved must be critical to the specifications of feedstock entering reactors
- ❑ Sensors to be located on a conveying system and tested at multiple speeds
- ❑ Proposed method needs to be safe and reasonable for a typical industrial environment
- ❑ Provide a baseline for the conventional characterization method
- ❑ Perform a system performance and cost analysis to operate at a commercial scale
- ❑ Contribution to BETO's mission and priorities for production of sustainable aviation fuels

1 – Approach

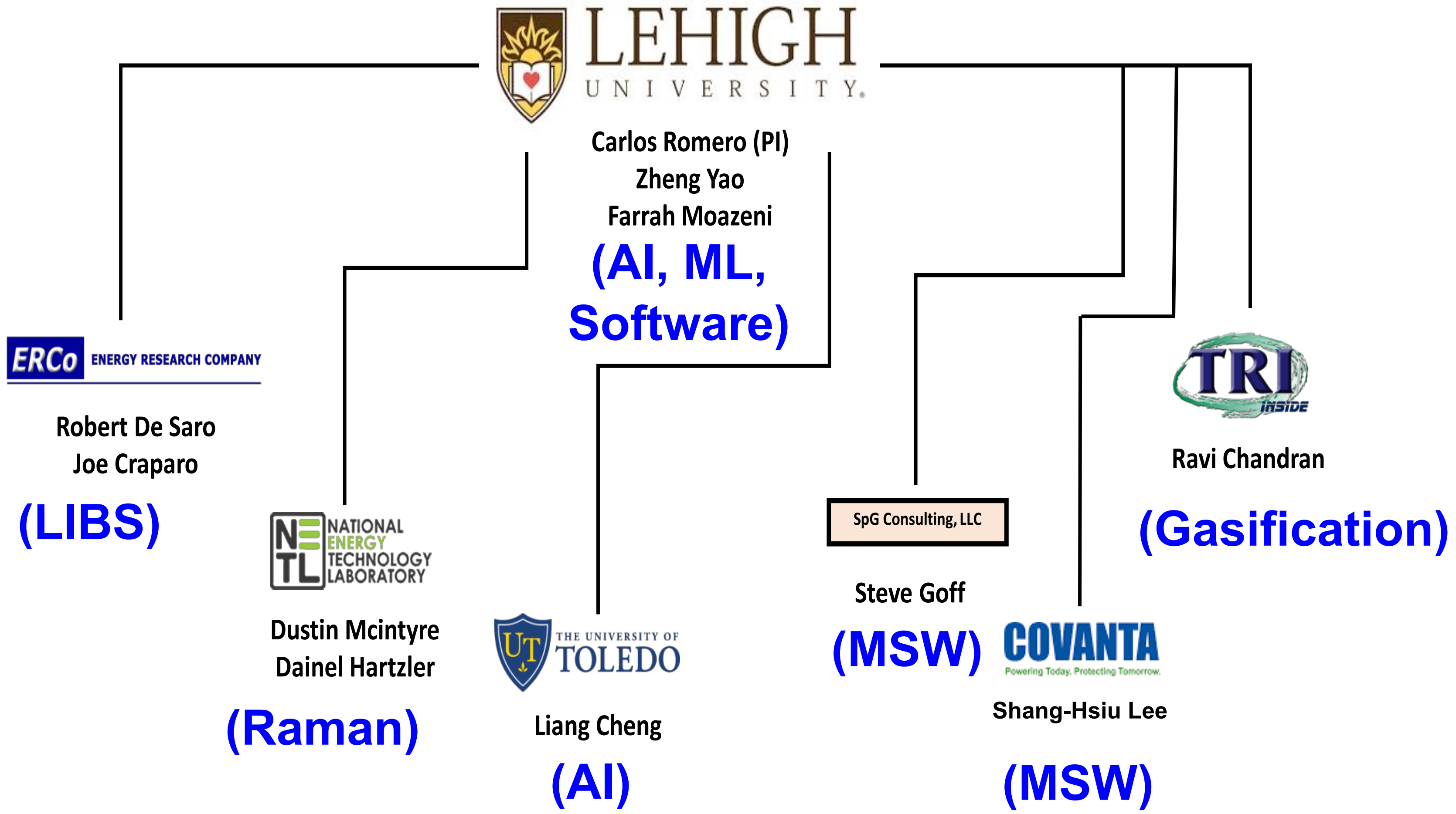
❑ Project Approach

- ❑ This project will develop and demonstrate a system that integrates Laser Induced Breakdown Spectroscopy (LIBS) and Raman Spectroscopy technology with advanced Machine Learning (ML) algorithms into a robust technology suited for rapid, in-situ and accurate analysis of MSW streams.
- ❑ The proposed technology will make it possible to resolve elemental, molecular and higher order characteristics of highly variable MSW.
- ❑ Technology will be demonstrated at TRI's Process Development Unit (PDU).



1 – Approach

- Project Team
- Relationship with Stakeholders
- Lehigh and DEI



1 – Approach

- Lehigh University is responsible for overall technical direction, timely execution, and management of the project.
- Tasks, Go/No-Go Decision Points, End of Project

BP1 10/1-21-4/30/22

BP2 5/1/22-4/30/23

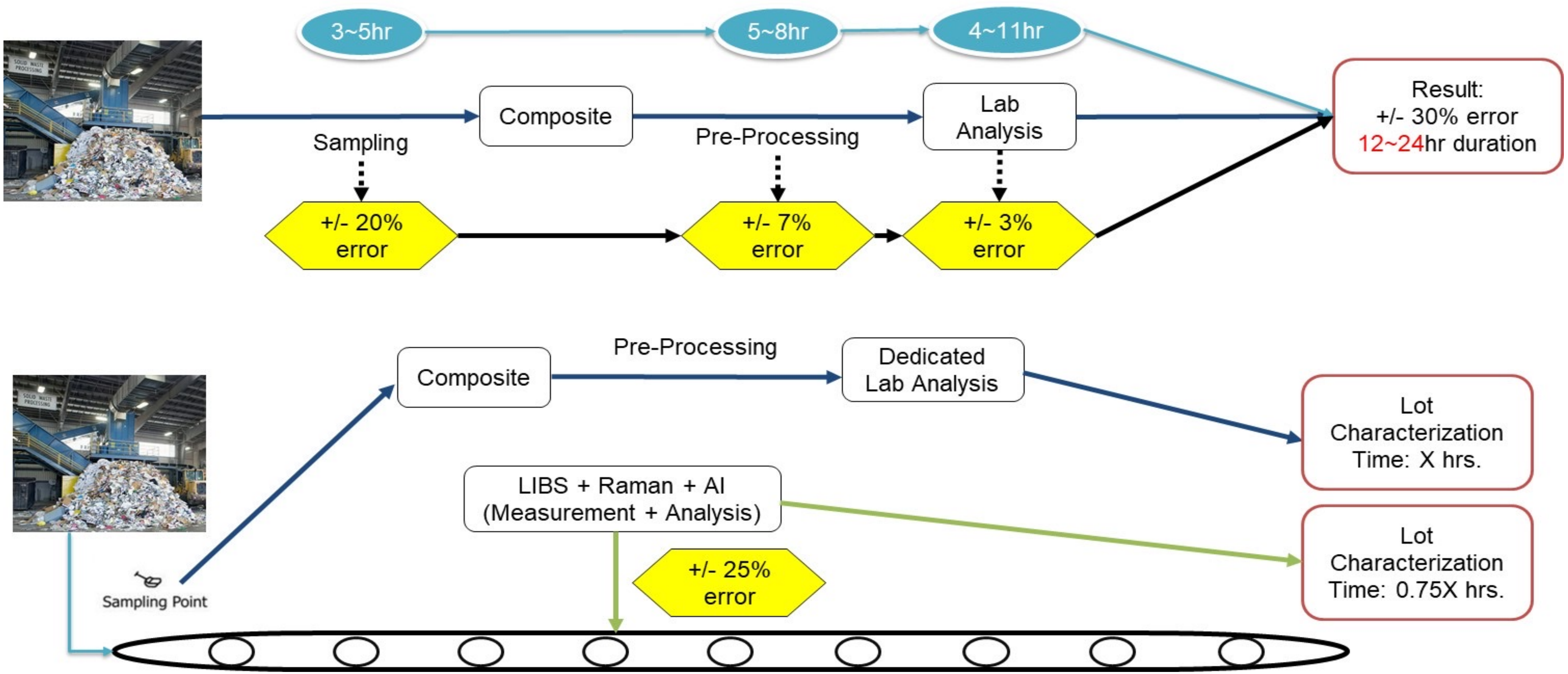
BP3 5/1/23-1/31/25

Budget Period	Schedule Months	Task Number	Task Description	Go/No-Go
1	1 – 3	1	Initial Verification	Month 3
2	4 – 12	2	Waste Sampling and Analysis	
	4 – 15	3, 4	Laboratory Test Program	Month 15
3	16 – 24	5	Prototype Development	
	25 – 33	6	PDU Test Program	
	16 – 36	7	Techno-Economic Analysis	Month 36

End of Project

1 – Approach

❑ Technical Metrics



1 – Approach

Technical Metrics

Validation Table	
Parameter/Performance	Final Target
Physical feature (partical size and/or shape?, density metric, rheology, contamination source)	1/2" minus for PDF Feedstock
Proximate Analysis	
Fixed Carbon (FC)	*</= +/- 25%*
Volatile Matter (VM)	*</= +/- 25%*
Moisture	*</= +/- 25%*
Total Ash	*</= +/- 25%*
Total Sulfur (S)	*</= +/- 25%*
Gross Heating Value	*</= +/- 25%*
Ash Analysis	
SiO2	Spectral Lines for Element, R2 > 0.7
Al2O3	Spectral Lines for Element, R2 > 0.7
Fe2O3	Spectral Lines for Element, R2 > 0.7
MgO	Spectral Lines for Element, R2 > 0.7
CaO	Spectral Lines for Element, R2 > 0.7
Na2O	Spectral Lines for Element, R2 > 0.7
K2O	Spectral Lines for Element, R2 > 0.7
TiO2	Spectral Lines for Element, R2 > 0.7
P2O5	Spectral Lines for Element, R2 > 0.7
Total alkali content [sum(Na2O+K2O) by wt.%/HV	*</= +/- 25%*
Ash Softening temperature	</= +/- 150 deg. F
Other Parameters	
Chlorine (Cl)	Demonstrated Ability to Identify
Cellulosic Content	Demonstrated Ability to Identify
Plastics Content	Demonstrated Ability to Identify
Lignin Content	Demonstrated Ability to Identify
LIBS/Raman Parameters	
Signal to Noise Ratio	3 to 1
Number of Laser Pulses Per Data Point	100 to 500
Number of prediction/time (final outcome)	10 minute frequency
Overall Improvement in Throughput of Characterization	> 25%

1 – Approach

❑ Critical Risks and Mitigation Strategies

Perceived Risk	Mitigation Strategy
Technical	
Issues with LIBS/Raman integration	Additional engineering will be carried out.
Instrument component failures	Components will be field repaired or replaced as needed.
Host site conditions	Field adjustments to equipment will be take. The team will factor in impacts on PDU vis prototype design.
Data accuracy and precision	Drift correction will be applied in the field, together with field calibration.
Financial	
PDU modification	Budget includes $12k for PDU sampling related costs. Team will adjust budget items to meet required modifications.
Availability of TRI PDU	The team will work with TRI to understand their PDU schedule and the work at Fulcrum Bioenergy plant.
Need for more data due to waste variability	Team will seek for no-cost extension to the project.

2 – Progress and Outcomes

ENERGY RESEARCH CENTER

- **Task 1: Initial Verification**
  - Completed sample management (chain of custody) plan verification
  - Completed laboratory test plan verification for LIBS and Raman analysis
  - Carried out visit to TRI’s Process Development Unit (PDU) in NC
  - Completed host site PDU preliminary prototype installation and test plan verification
  - Carried out Verification Team meeting
  - Prepared report on updated project plans and management, including SOPO update.

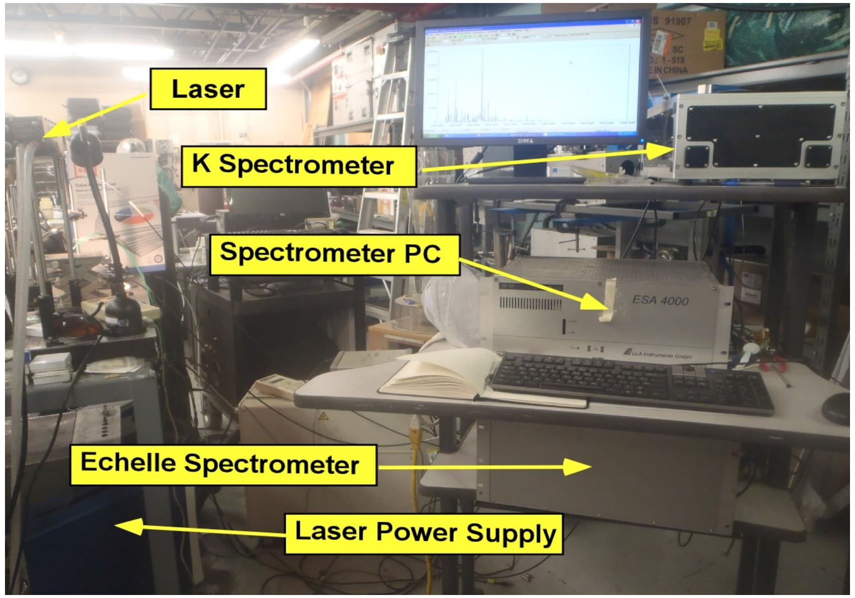
2 – Progress and Outcomes

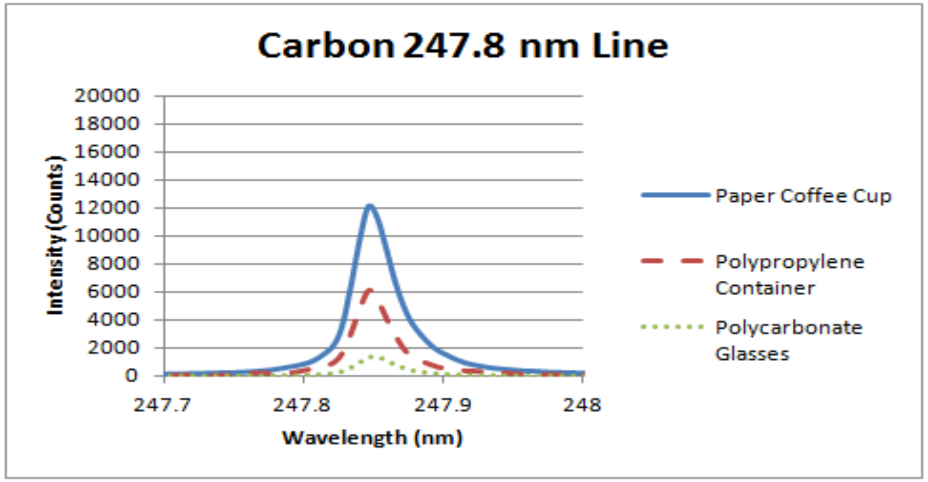
❑ Task 2: Waste Sample Procurement and Analysis

- ❑ Sources of MSW
  - ❑ Commercial Refuse Derived Fuel (RDF) from City of Ames
  - ❑ RDF from Fulcrum Bioenergy's Facility
  - ❑ Individual material samples (8 plastics, 3 papers, 3 others) by Lehigh Univ.
  - ❑ Additional MRF (materials recovery facility) stream samples from East Lansing Michigan (single-stream), and waste plastics from Peloskey Michigan(dual-stream MRF).
- ❑ Sample Distribution → ERCo, NETL and G&C Analysis Lab.
- ❑ Analysis → Proximate, ultimate, heating value, ash oxides, trace inorganic, fusion temperatures and target molecular compounds.
- ❑ Data Management
  - ❑ Lehigh University
  - ❑ Idaho National Lab's (INL's) Bioenergy Feedstock Library

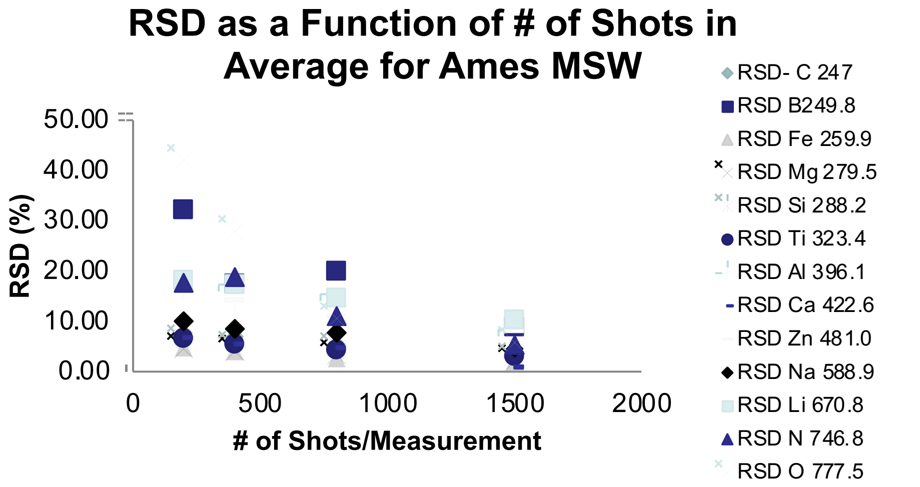
2 – Progress and Outcomes

- Task 3: LIBS Laboratory Development, Testing and AI Algorithm Development
  - Completed LIBS laboratory setup
  - Completed testing of individual material samples and in the process of testing MSW samples (processed and unprocessed)
  - Updated spectral data preprocessing
  - Excellent signal-to-noise on all elements of interest and relative standard deviation (RSD)





Sensitivity of LIBS Carbon Line for Different Materials

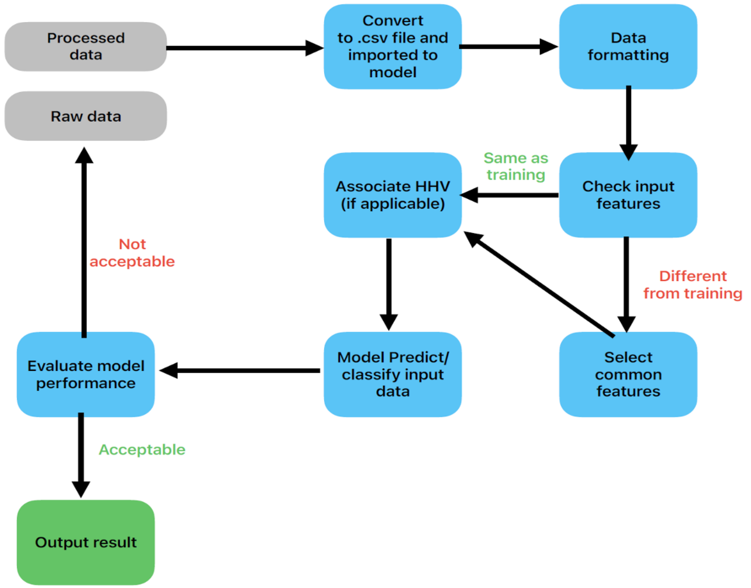


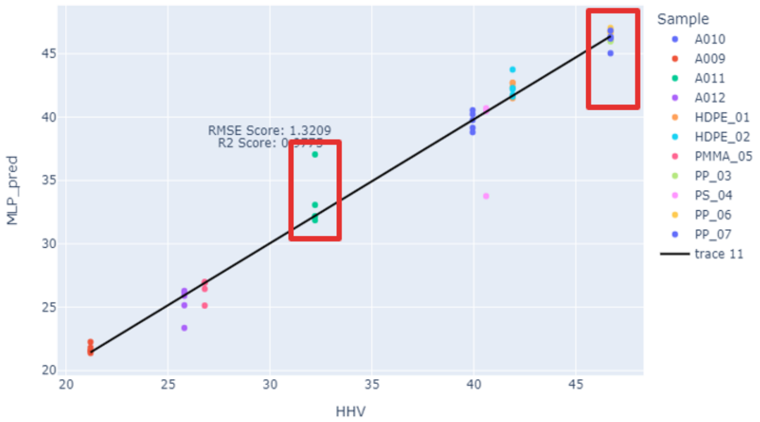
RSD as a Function of Number of LIBS Shots per Measurement

2 – Progress and Outcomes

❑ Task 3: LIBS Laboratory Development, Testing and AI Algorithm Development

- ❑ AI modeling underway (decision tree, support vector machine (SVM), random forest and multi-layer perceptron (MLP) neural networks).
- ❑ New approaches added to enhance repeatability of LIBS spectral data (intensity correction approach) and accuracy of prediction





Plastic	HHV
PMMA	26.8
HDPE	41.9
PP	46.7
PS	40.6
PVC	21.2
ABS	39.94
Polycarbonate	32.21
Acrylic	25.8

MLP Modeling Results Compared to Analytical Data

2 – Progress and Outcomes

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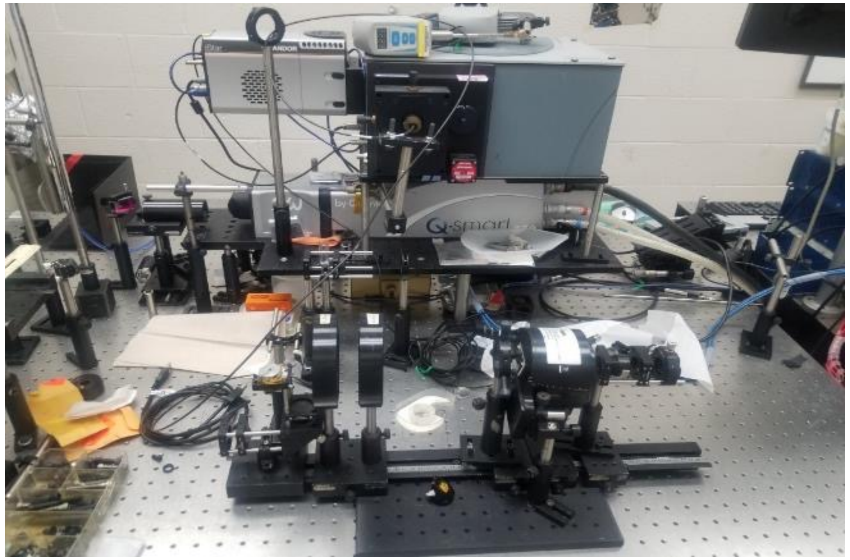
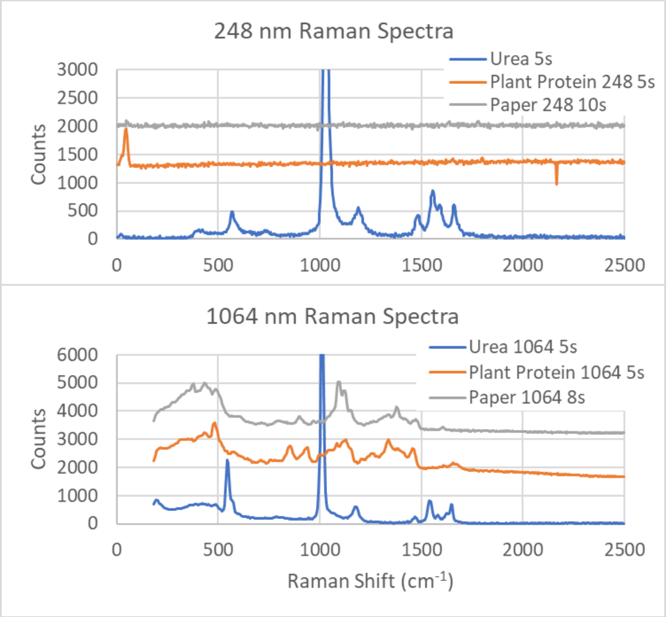
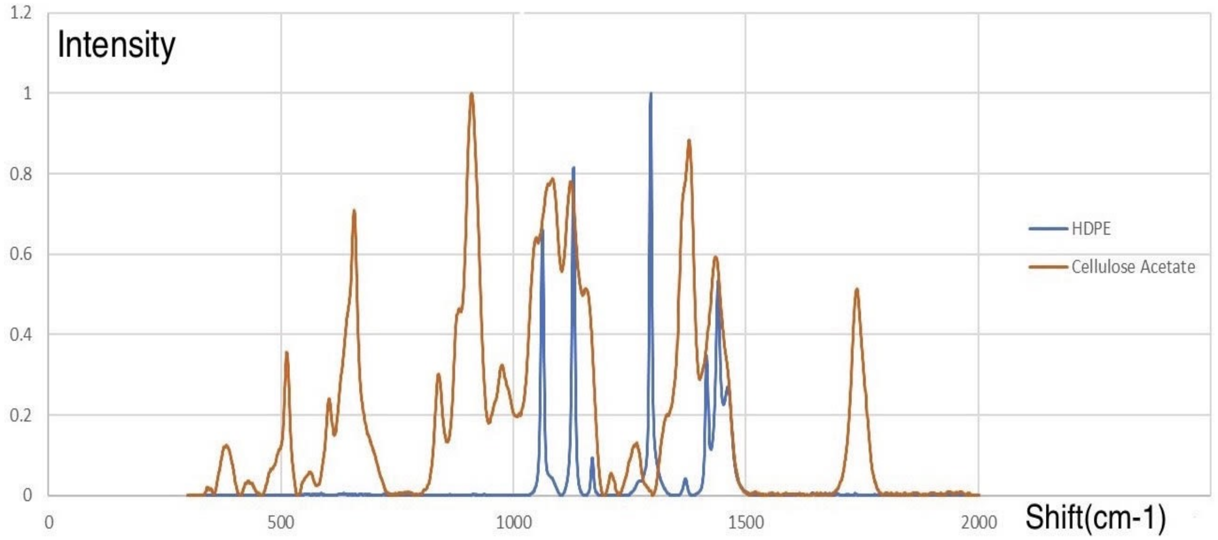
- **Task 4: Raman Laboratory Development, Testing and AI Algorithm Development**
  - Completed Raman laboratory setup. Switched from DUV-based to NIR-based system. Possibility of using an additional diode excitation laser at 850 nm.
  - Completed testing of individual material samples and in the process of testing MSW samples





Raman Spectral for DUV- and NIR-Based Systems

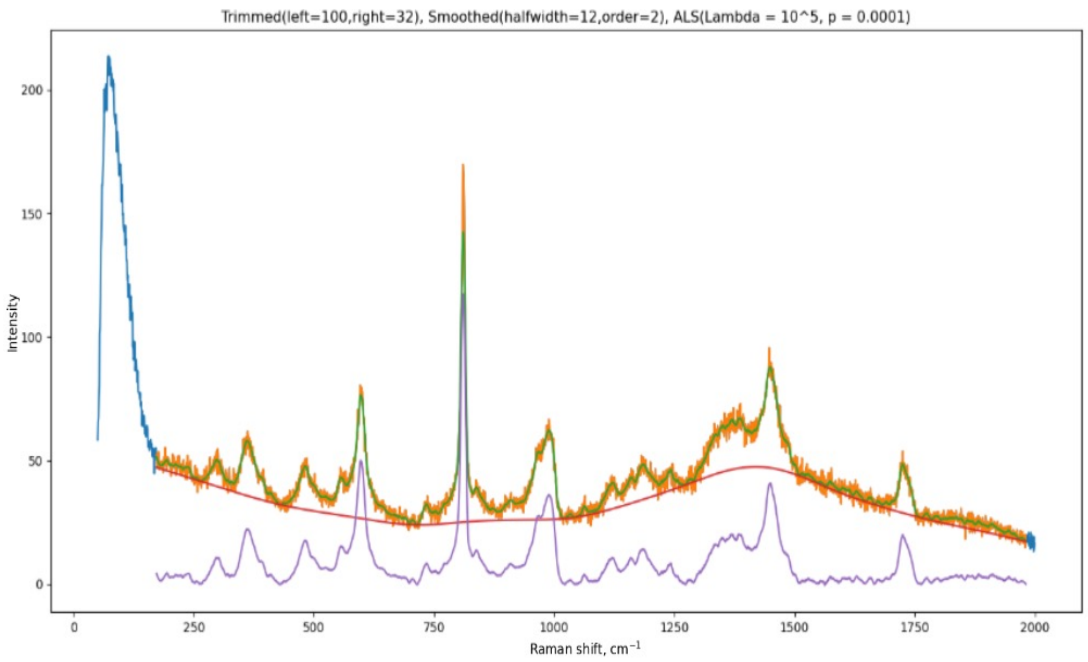


Raman Spectrum for High Density Polyethylene and Cellulose

2 – Progress and Outcomes

❑ Task 4: Raman Laboratory Development, Testing and AI Algorithm Development

- ❑ AI modeling underway (wavelength range selection, outlier/cosmic ray removal, spectral smoothing, baseline correction, normalization).
- ❑ In the process of assessing spectral band assignment to classification groups



Unprocessed and Processed Raman Spectrum

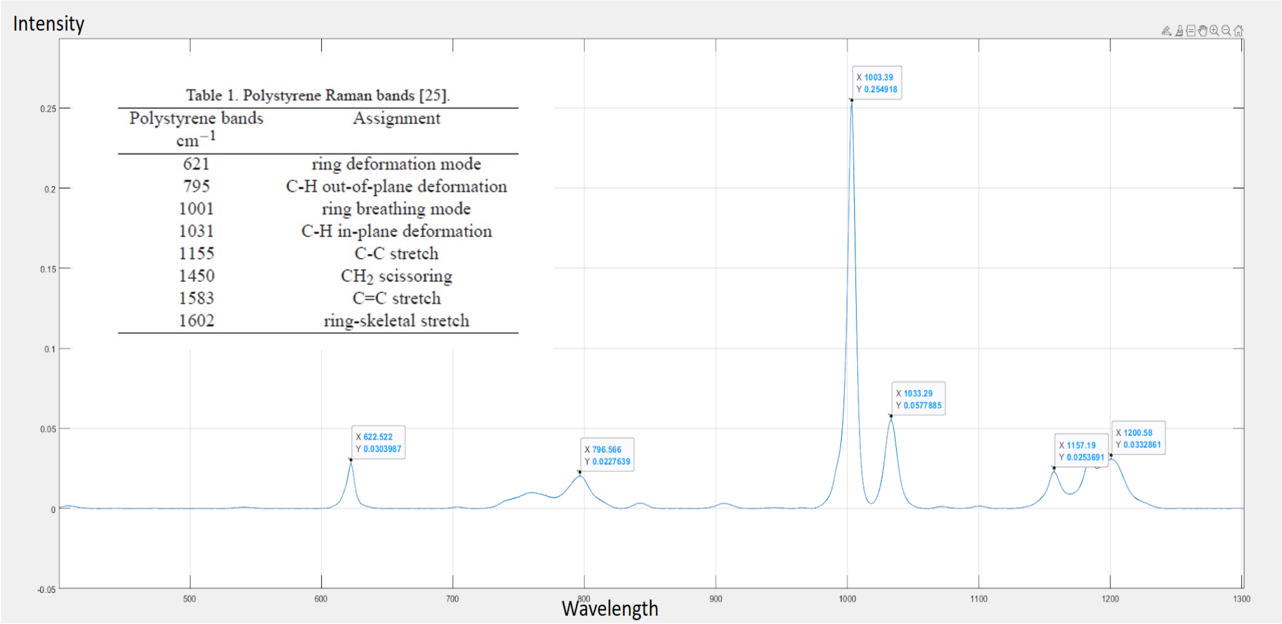


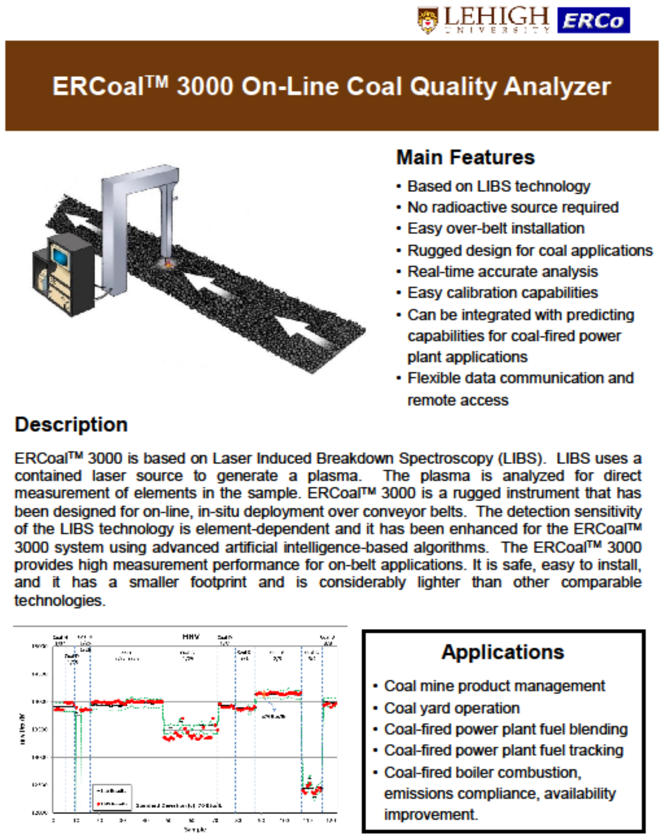
Table 1. Polystyrene Raman bands [25].

Polystyrene bands cm^{-1}	Assignment
621	ring deformation mode
795	C-H out-of-plane deformation
1001	ring breathing mode
1031	C-H in-plane deformation
1155	C-C stretch
1450	CH_2 scissoring
1583	C=C stretch
1602	ring-skeletal stretch

Identification of Raman Bands for Polystyrene

3 – Impact

- **Technological Advancements to be Derived from this Project**
- Advance the capabilities of LIBS and Raman (individually) to identify and quantification MSW material.
- Advanced AI algorithms to improve optical techniques measurement accuracy and precision.
- A first generation of integrated LIBS/Raman/AI measurement.
- **<u>Value Proposition</u>**
  - Enable continuous measurement of processed MSW properties to confirm product specifications
  - Substantially eliminate cost of sampling, compositing and laboratory analysis
  - Improved MSW processing system performance and RDF product QA/QC.
  - Reduce operator interaction with equipment and waste – improved plant safety
  - Feed-forward process control of downstream RDF gasification conversion system
  - Increased revenue and reduced Operating and Maintenance (O&M) costs should yield less than 1 year payback for projected instrument cost in the range of $300 - $500K.
- **Commercialization Potential**



Example of Commercialization Partenership between Lehigh University and ERCo for the Coal-Fired Power Industry

Summary

- Project goal is an improvement in throughput of MSW measurement characterization technology by the development of advanced spectroscopic techniques linked to artificial intelligence.
- Project has assembled a unique team of qualified groups that includes an instrumentation company, a national lab, academic institutions and industrial companies aligned with MSW management and processing.
- Protocols, laboratory plans, full-scale prototype host site, metrics and project initial verification has been arranged/completed.
- Material samples have been procured and analyzed for parameters of interest.
- LIBS and Raman laboratories are up and running. AI model development is progressing. Results up to date are encouraging.

Quad Chart Overview

Timeline

- *Project start date: 10/01/2021*
- *Project end date: 01/31/2025*

	FY22 Costed	Total Award
DOE Funding	*$499,258*	*$2,810,431*
Project Cost Share *	*$101,092*	*$702,673*

TRL at Project Start: 2
TRL at Project End: 6

Project Goal

Improve throughput of MSW measurement characterization technology with a minimum target of 25% improvement over baseline characterization technology.

End of Project Milestone

Improvement in throughput of waste characterization over current approaches of manual waste sampling and laboratory analysis. The end of project verification should confirm program requirement of improvement in waste characterization frequency of at least 25% improvement over baseline. The measurement accuracy of the new on-line system will be determined based on ASTM standard D6543 for coal, adapted to MSW.

Funding Mechanism

DE-FOA-0002423 - FY21 Bioenergy Technologies Office (BETO) Feedstock Technologies and Algae FOA.

Topic Area 1: Characterization of Municipal Solid Waste to Enable Production of Conversion-Ready Feedstocks.

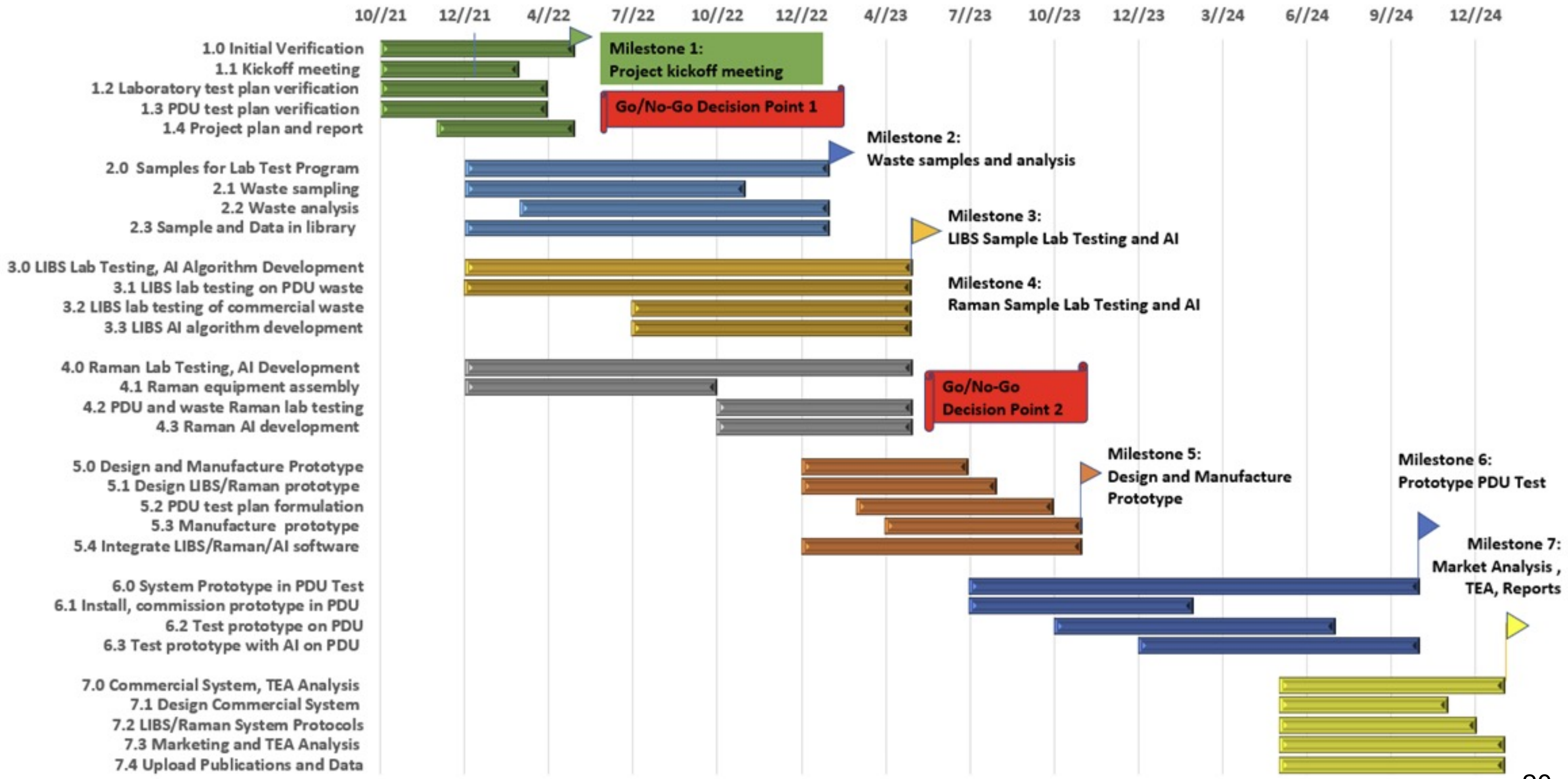
Subtopic 1b: Development of Novel Methods for Rapid/Real-Time Measurements.

Project Partners

- *DOE National Energy Technology Lab, Energy Research Co., ThermoChem Recovery International, Inc., SpG Consulting, Covanta, The University of Toledo.*

Additional Slides

Project Management Plan



Overview of Recipient Resources and Capabilities

❑ Lehigh University - Energy Research Center (ERC)

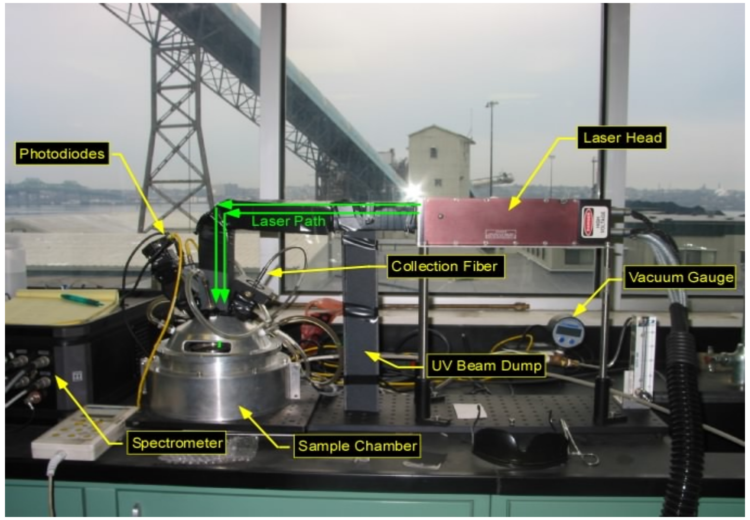
- The ERC has considerable experience managing large federally funded projects in the field of energy and power generation. The ERC is currently leading large DOE funded projects, for example, Flexible Power Plant Operation with Thermal Energy Storage Utilizing Thermosiphons and Cementitious Materials, Improvement of Power Plant Dry Cooling Technology through Application of Cold Thermal Energy Storage.
- The ERC has carried out sponsored projects that involve instrumentation and control (i.e., soft sensors, distributed control upgrades), smart software (i.e., on-line optimizers, AI models and expert systems), and technology field deployment (i.e., installation of Boiler OP technology at more than 30 power plants).
- In collaboration with ERCo, the ERC has participated in projects that integrate the LIBS technology with AI:

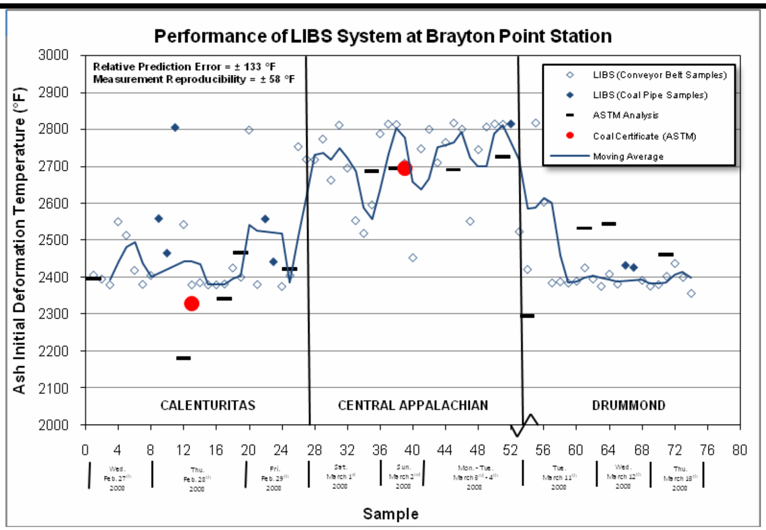
  (1) Coal Lab-STAC, (2) Biomass Lab-NYSERDA, (3) Coal Field Demo-PPL,

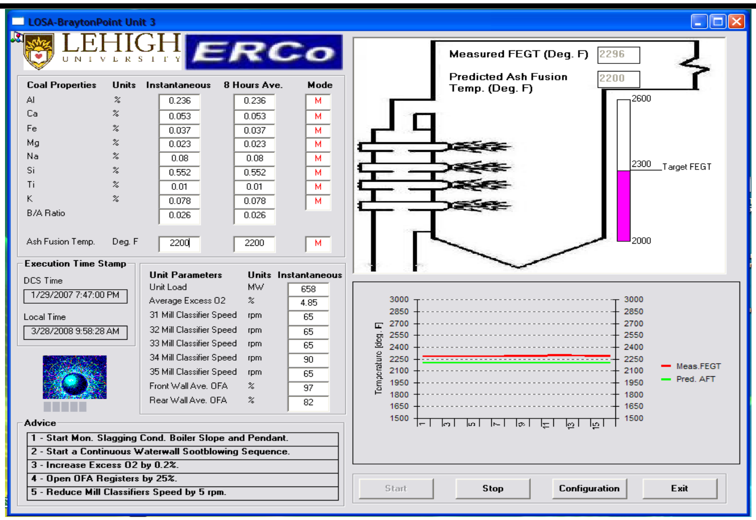
  (4) Coal Commercial System-EPRI & FirstEnergy, (5) MSW Lab-APCI,

  (6) Gasification Feedstock-EPRI, (7) Coal Mining-China.

Overview of Recipient Resources and Capabilities







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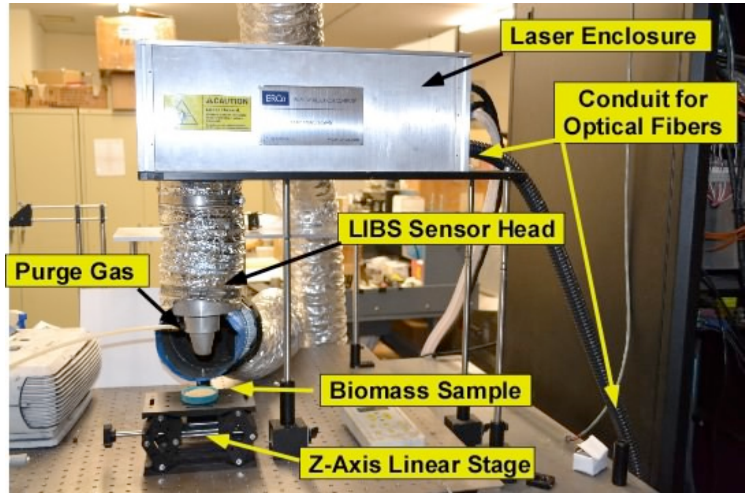

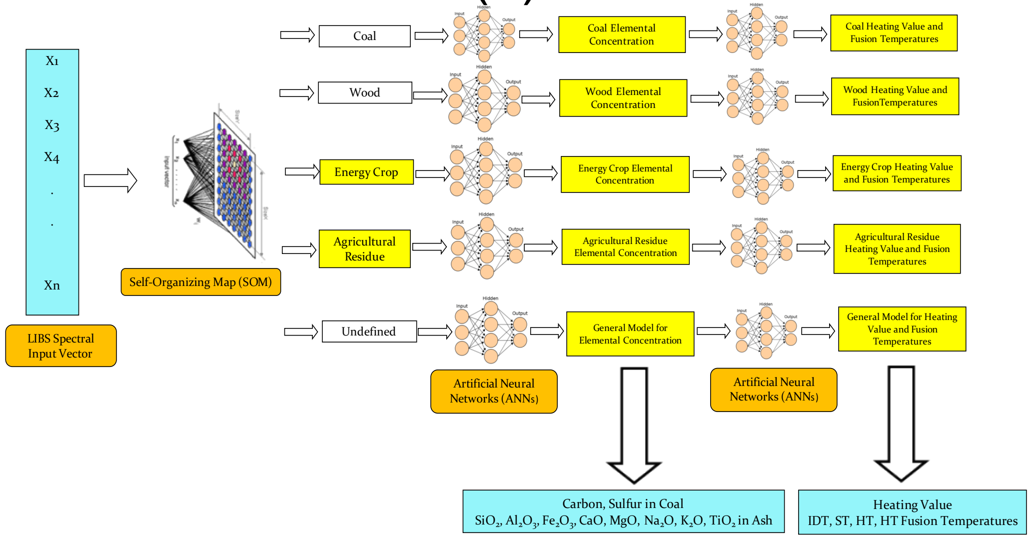

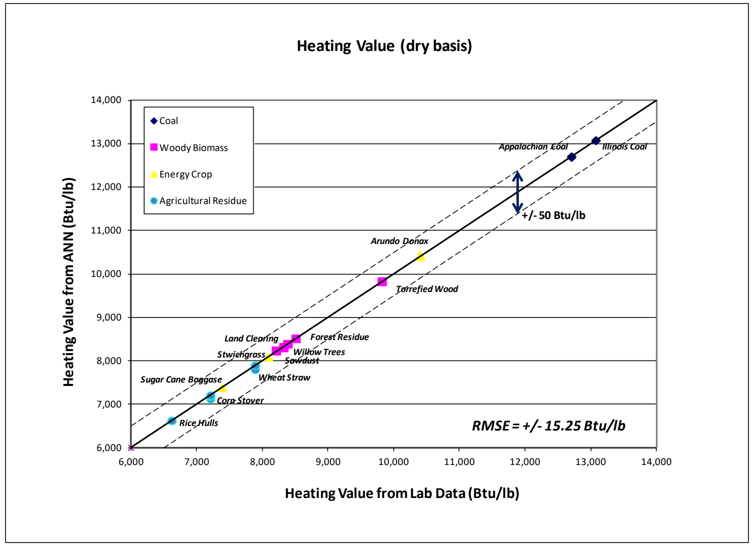

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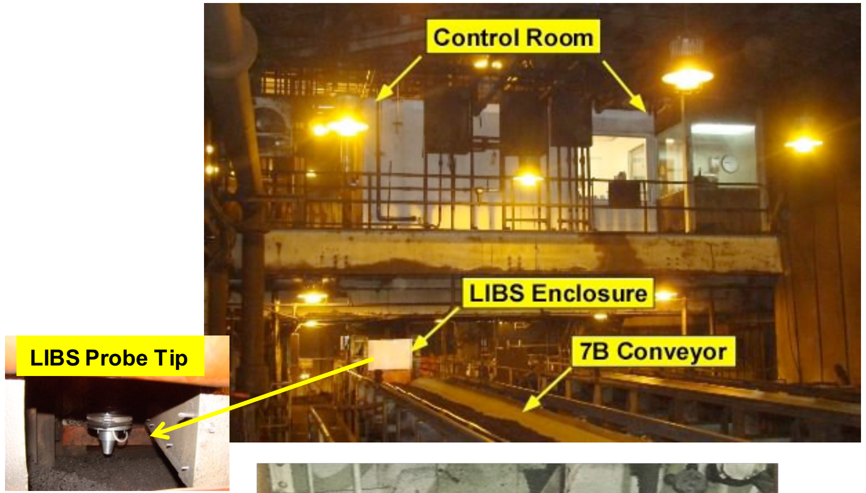
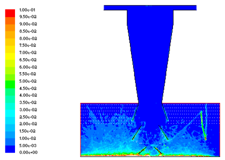
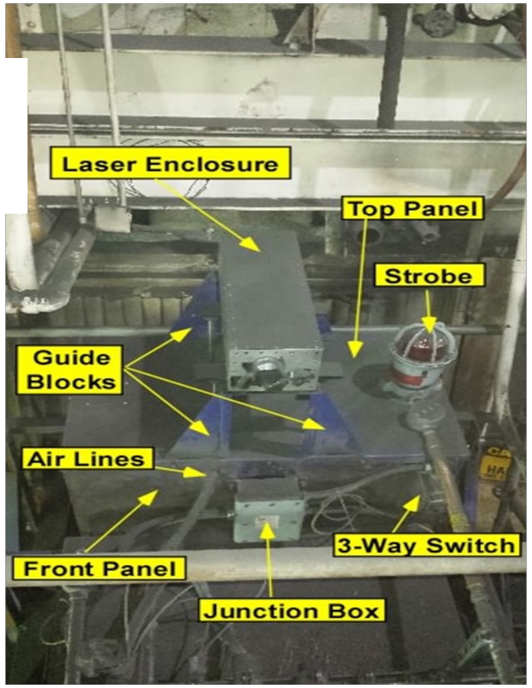
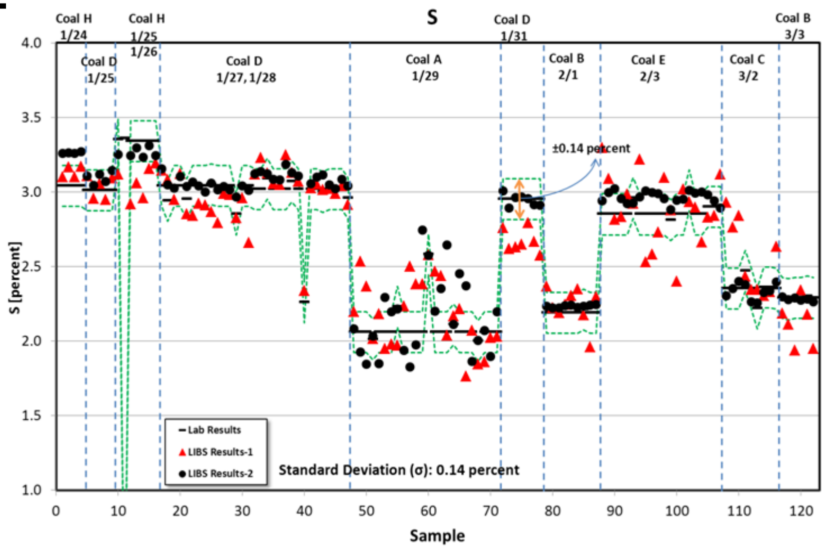
Overview of Recipient Resources and Capabilities









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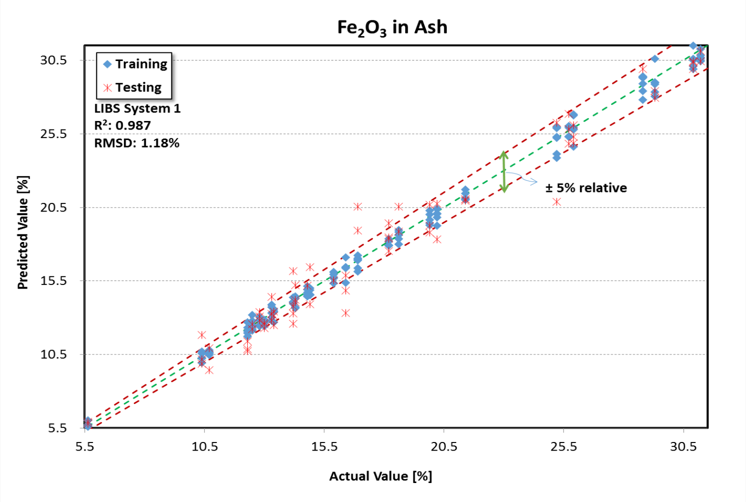

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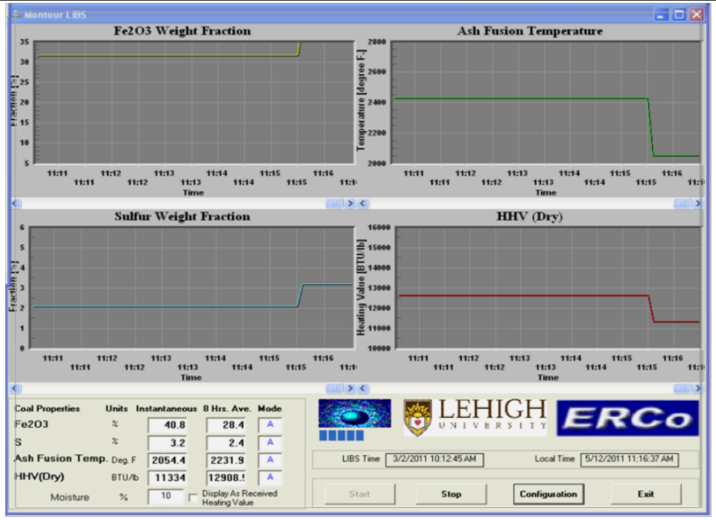
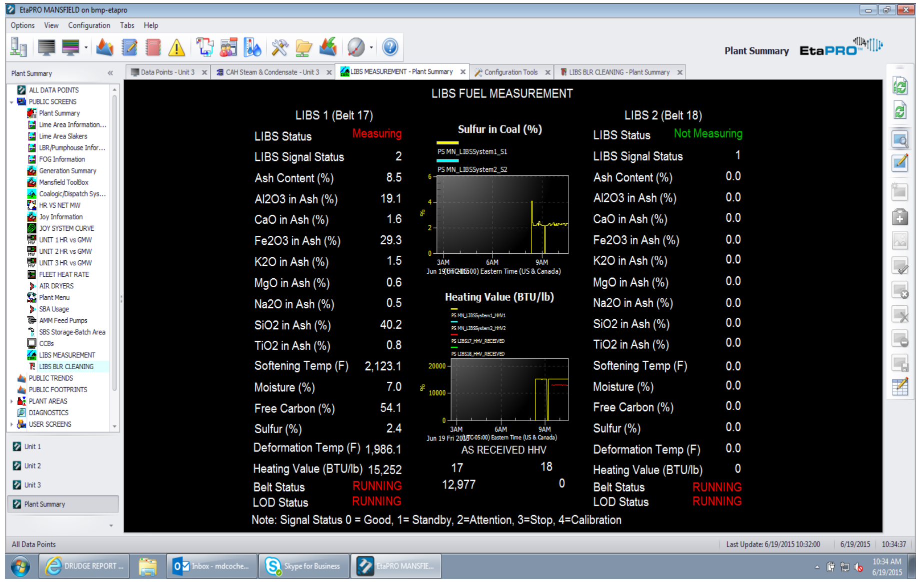
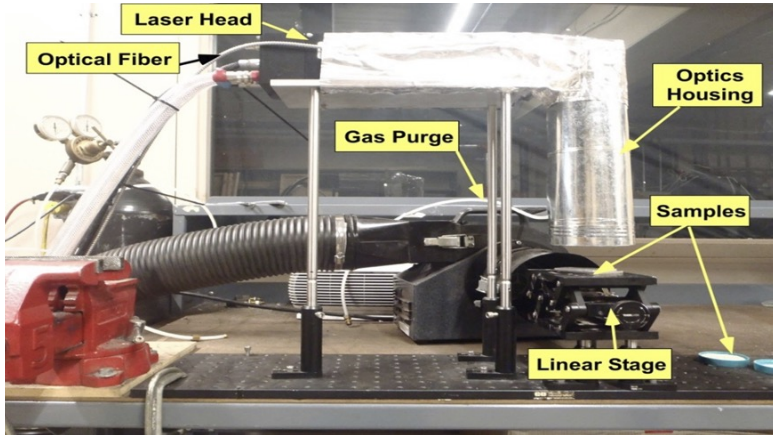




Overview of Recipient Resources and Capabilities



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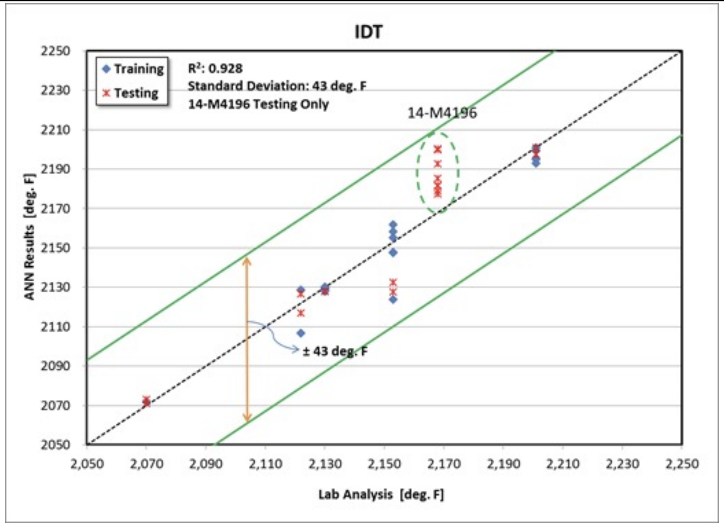

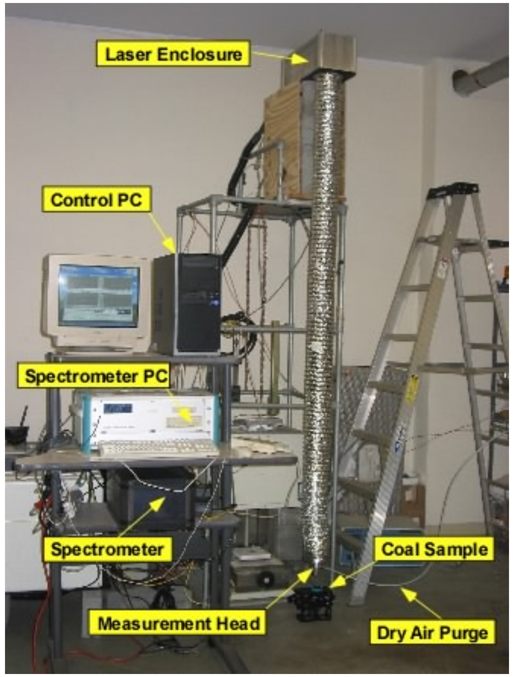

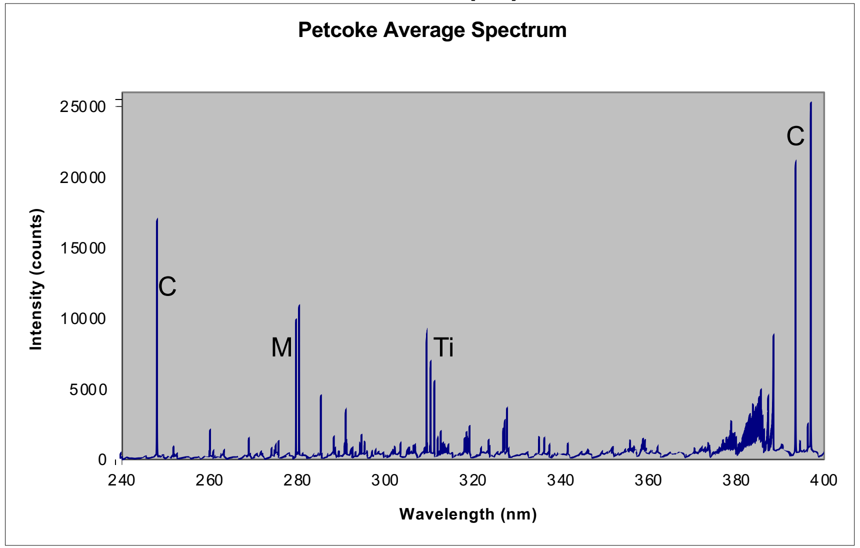

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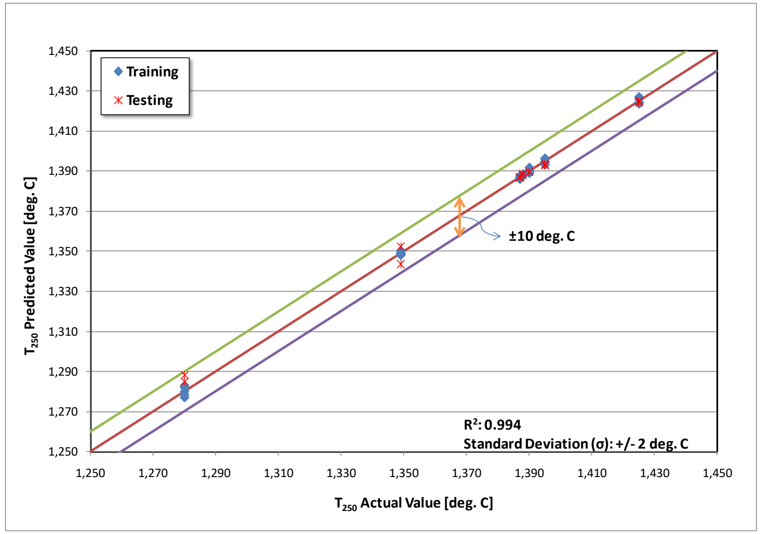
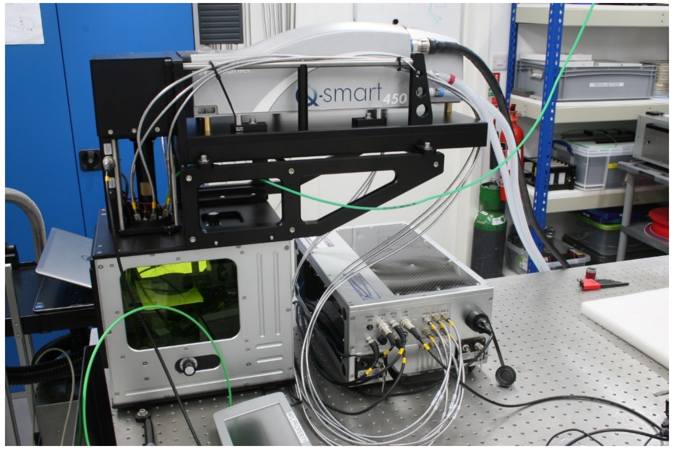
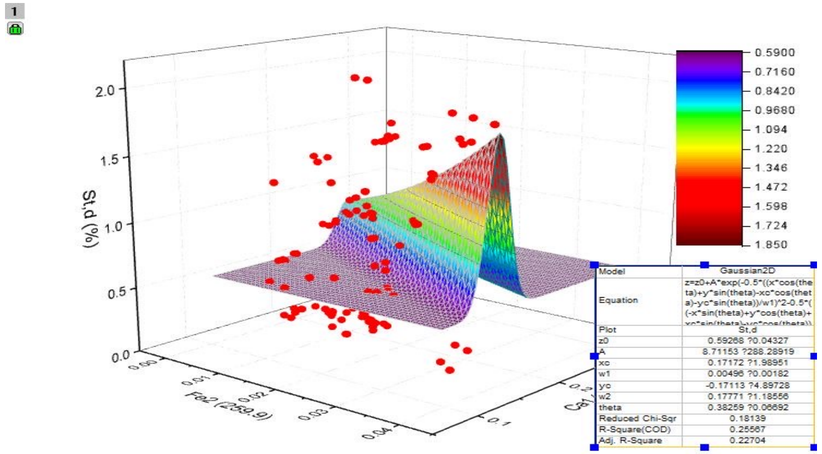
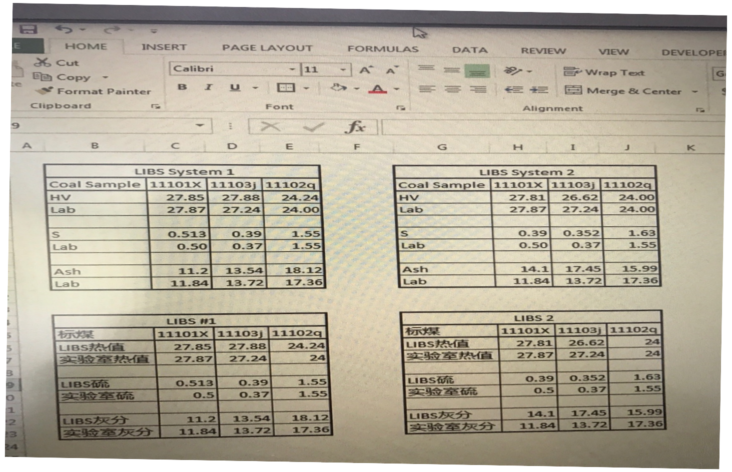


Overview of Recipient Resources and Capabilities







LIBS System 1			
Coal Sample	11101X	11103j	11102q
HV	27.85	27.88	24.24
Lab	27.87	27.24	24.00
S	0.513	0.39	1.55
Lab	0.50	0.37	1.55
Ash	11.2	13.54	18.12
Lab	11.84	13.72	17.36

LIBS System 2			
Coal Sample	11101X	11103j	11102q
HV	27.81	26.62	24.00
Lab	27.87	27.24	24.00
S	0.39	0.352	1.63
Lab	0.50	0.37	1.55
Ash	14.1	17.45	15.99
Lab	11.84	13.72	17.36

LIBS #1			
标煤	11101X	11103j	11102q
LIBS热值	27.85	27.88	24.24
实验室热值	27.87	27.24	24
LIBS硫	0.513	0.39	1.55
实验室硫	0.5	0.37	1.55
LIBS灰分	11.2	13.54	18.12
实验室灰分	11.84	13.72	17.36

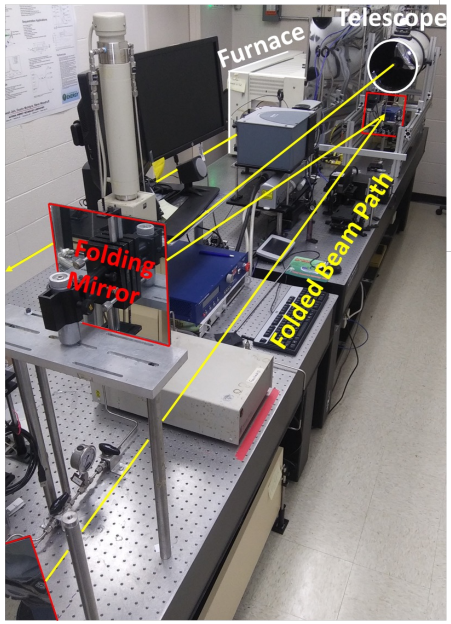
LIBS 2			
标煤	11101X	11103j	11102q
LIBS热值	27.81	26.62	24
实验室热值	27.87	27.24	24
LIBS硫	0.39	0.352	1.63
实验室硫	0.5	0.37	1.55
LIBS灰分	14.1	17.45	15.99
实验室灰分	11.84	13.72	17.36

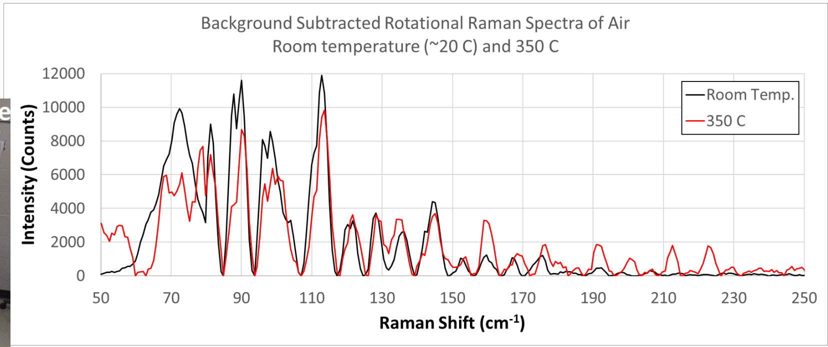
(7)

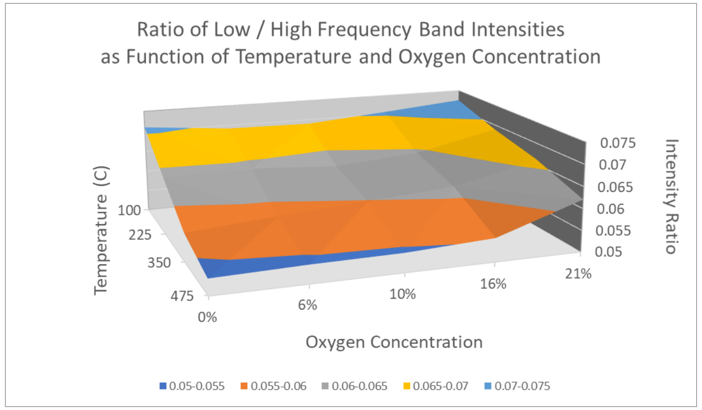
Overview of Recipient Resources and Capabilities

❑ National Energy Technology Laboratory (NETL)

- ❑ Recent work includes:
  - Upgrades and a redesign of the subsurface LIBS sensor system
    - To address issues identified during testing and add capabilities
    - Field testing is planned for the later half of 2022
  - Raman LIDAR chemical and physical analysis of gasses (i.e., composition, concentration, and temperature) within a fossil fueled boiler. See Figures.
    - The project demonstrates the simultaneous determination of spatially resolved gas temperature and composition up to 475 C and at 15 m distance. See 2D calibration curve (lower right).







Overview of Recipient Resources and Capabilities

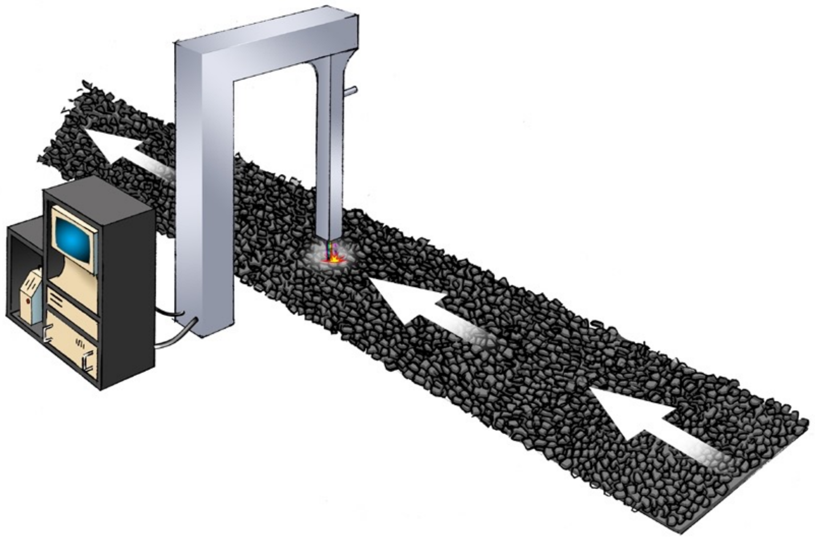
❑ Energy Research Company

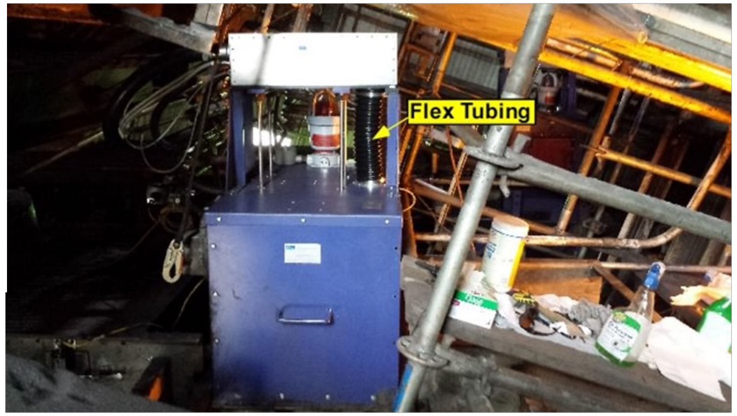
- R&D Company founded 1991
- Laser Diagnostics
- Industrial Applications
- OEM for Laser System for Aluminum Industry
- Indirect Rates Approved by DOE
- DCAA Audited
- Formed Joint Venture, Melt Cognition, for Commercial Sales of LIBS for Metals Industry
- License to Altek for Aluminum Industry

❑ What We Do

- Application Oriented Laser-based Sensing
- Industrial
- Military and Security
- Other

Overview of Recipient Resources and Capabilities





Coal at Commercial Operating Power Plants

Overview of Recipient Resources and Capabilities

❑ ThermoChem Recovery International, Inc. (TRI)

- TRI's feedstock flexible, 2-stage gasification system forms a vital part of the Integrated BioRefinery (IBR) that converts biomass, agri-waste, MSW, etc. into any combination of fuels, power, heat, and chemicals. TRI is currently involved in commissioning the 500 dtpd MSW to SAF IBR at Fulcrum BioEnergy's Project Sierra site in Reno, NV.
- TRI has built a fully integrated IBR Process Demonstration Unit (PDU) comprising the various independent unit operations, such as feeding, gasification, syngas clean-up and synthesis of syngas to fuels into a combined, energy efficient system to demonstrate the feedstock-to-liquid production. TRI has run long duration test campaigns (1,000's of h) on this platform with MSW and woody biomass. This facility is at the TRI Advanced Development Center (ADC) in Durham, NC and will host project prototype testing in YR3 of the project.
- Dr. Ravi Chandran is TRI's Chief Technology Officer with over 30 years of technology development experience in the areas of gasification, steam reforming, pulse combustion, power generation and waste treatment.

Overview of Recipient Resources and Capabilities

- **ThermoChem Recovery International, Inc. (TRI)**
  - The most prominent of ADC facilities is the largest fully integrated, feedstock-to-fuel Process Demonstration Unit (PDU) biorefinery on the East coast (possibly worldwide). This facility can transform a variety of raw waste materials, including processed MSW, into drop-in transportation fuels and valuable consumer chemical products entirely on the premises. Every process required in a commercial biorefinery is replicated here.