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Application of Laser Induced Breakdown spectroscopy (LIBS) to International Safeguards

~~LIBS Instrumentation Development Applications to International Safeguards and NNV Activities~~

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Abstract

Laser Induced Breakdown Spectroscopy (LIBS) has been under development here at Los Alamos National Laboratory and laboratories in the nation and worldwide for over 40 years to address a number of analysis problems involving gases, liquids, and solids. The focus of our development effort has been the design, construction, and performance testing of LIBS instrumentation to address the needs of the international safeguards community for rapid fixed lab or in field measurements on samples of interest.

In this paper we will highlight our development efforts on a backpack LIBS system that can be used to rapidly analyze surfaces contaminated with depleted uranium, metals, alloys, and samples in general that are important in the processing and reprocessing of special nuclear materials. We will also discuss an automatic sample identification feature that we have integrated into this system and are currently testing. Finally, we will discuss our recent results using a medium resolution LIBS system (res. approximately 20,000) to analyze mixed oxide samples of uranium and thorium in a stearic acid binder and some preliminary results on analyzing a similar uranium / thorium sample in the presence of a stainless steel matrix.

Applications of Laser Induced Breakdown Spectroscopy (LIBS) to International Safeguards

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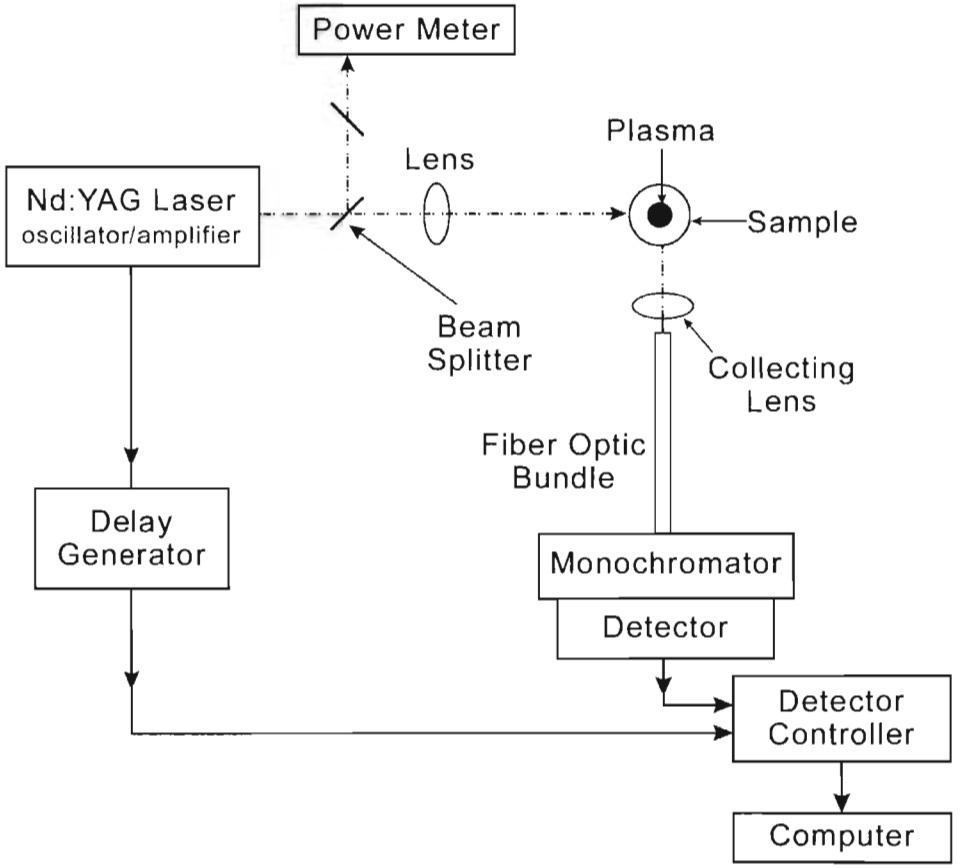
What is LIBS?

- LIBS (Laser-Induced Breakdown Spectroscopy) is a laser based technique that involves the use of a focused pulse laser beam to generate a microplasma

- The emission from the plasma is wavelength resolved using a spectrograph / detector / computer system to determine the elemental composition of the samples of interest

Typical LIBS Apparatus

Schematic of a typical LIBS apparatus



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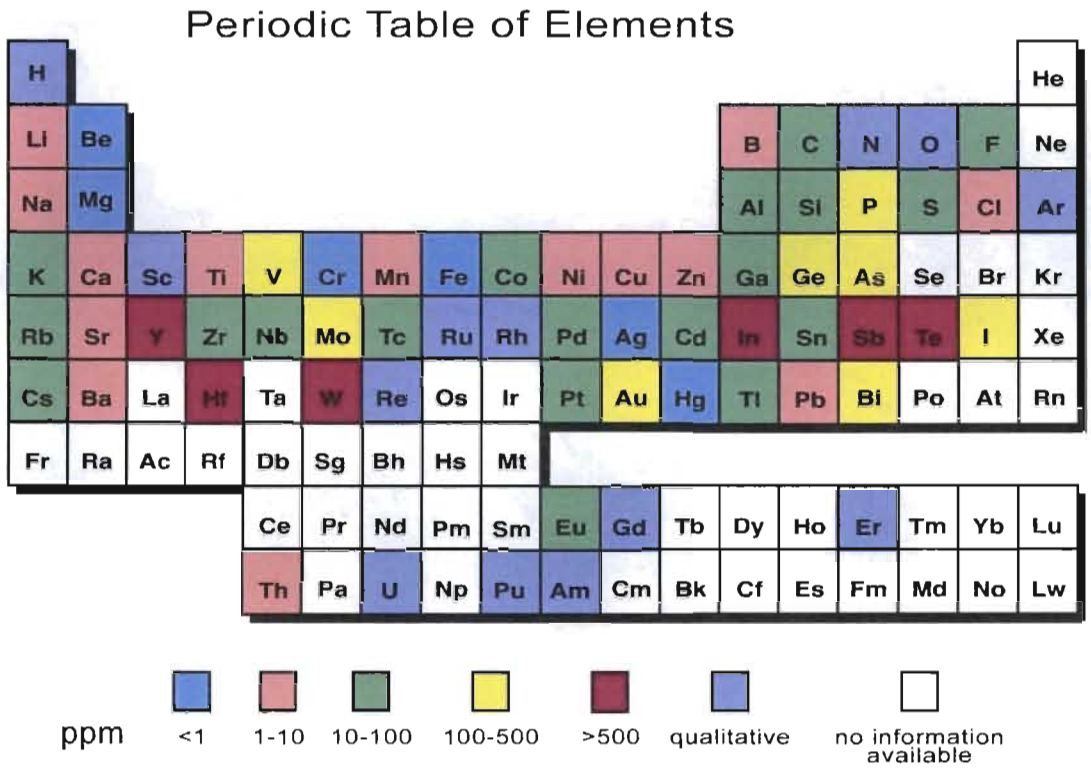
LIBS Advantages

- Little to no sample preparation required
- Analysis in real time / minutes
- Highly configurable
  - Portable, fixed lab, and transportable field deployable systems possible
  - Remote analysis in harsh environments using conventional or fiber optics in hot cells and gloveboxes
- Sensitivity (ppm – subppm)
- Ratios of elements (Pu/Cm, U/Cm, Pu/U, U/Pu…etc)
- Isotopic measurements of actinides (Pu, U…etc)
- Analysis of all elements in the Periodic Table

Elemental Coverage



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CHEMISTRY

NNSA

LIBS Instrumentation Development

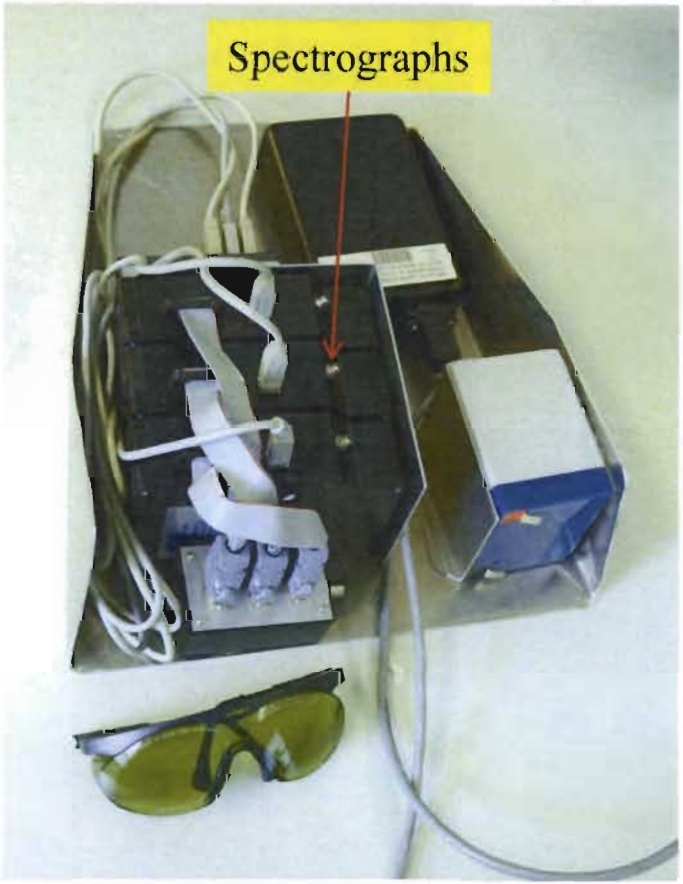
- Portable / backpack mounted LIBS system
  - In-field sample identification, screening, and analysis

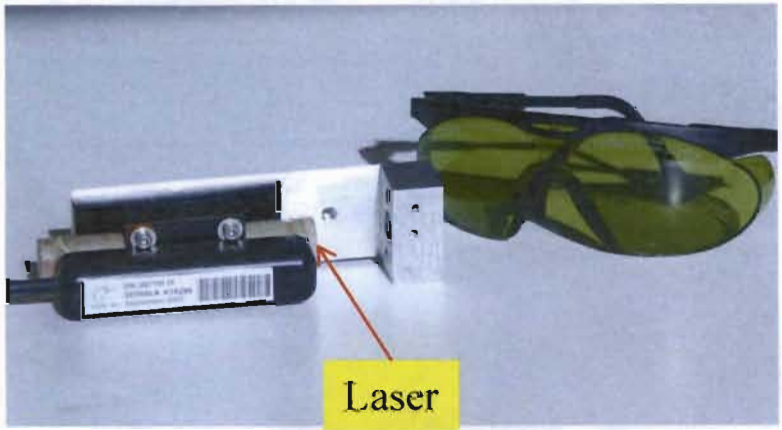
- Rack / cart mounted high resolution LIBS system
  - Transportable, in-field isotopic analysis or DA lab pre-screening tool

LIBS Backpack Mounted System

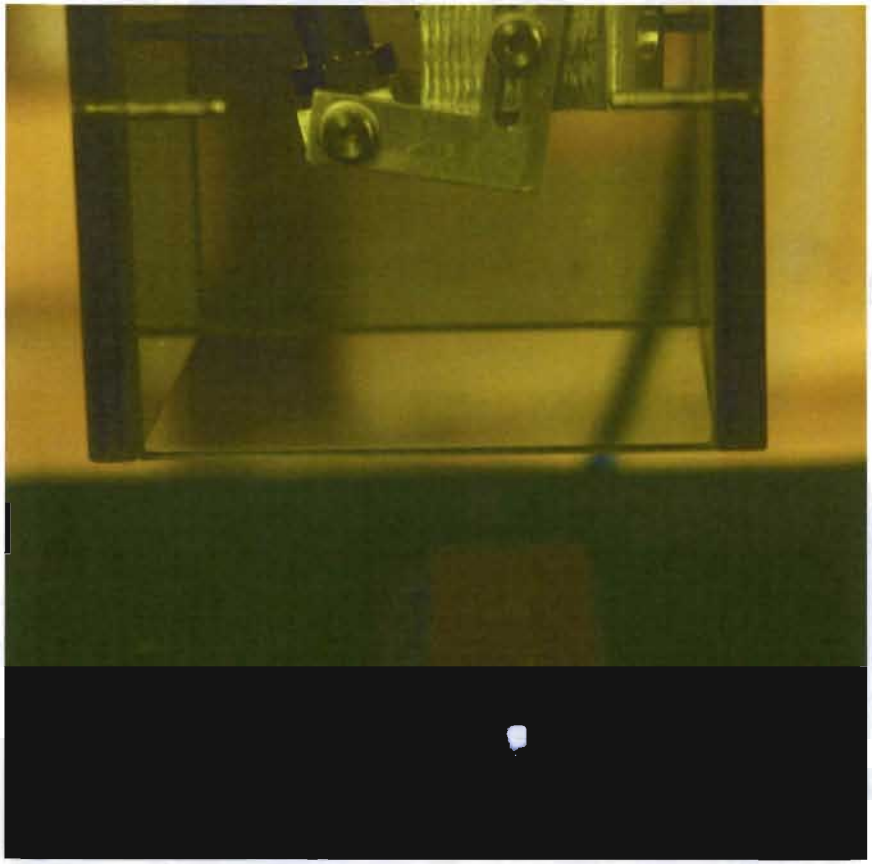
Field Test Geological Site in Arizona

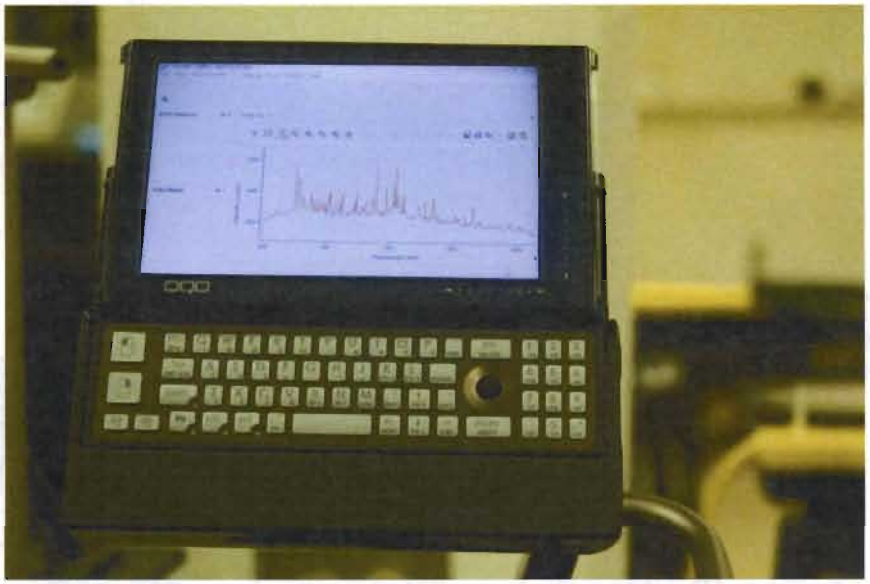
System Components (spectrometers, electronics, laser, sampling head)



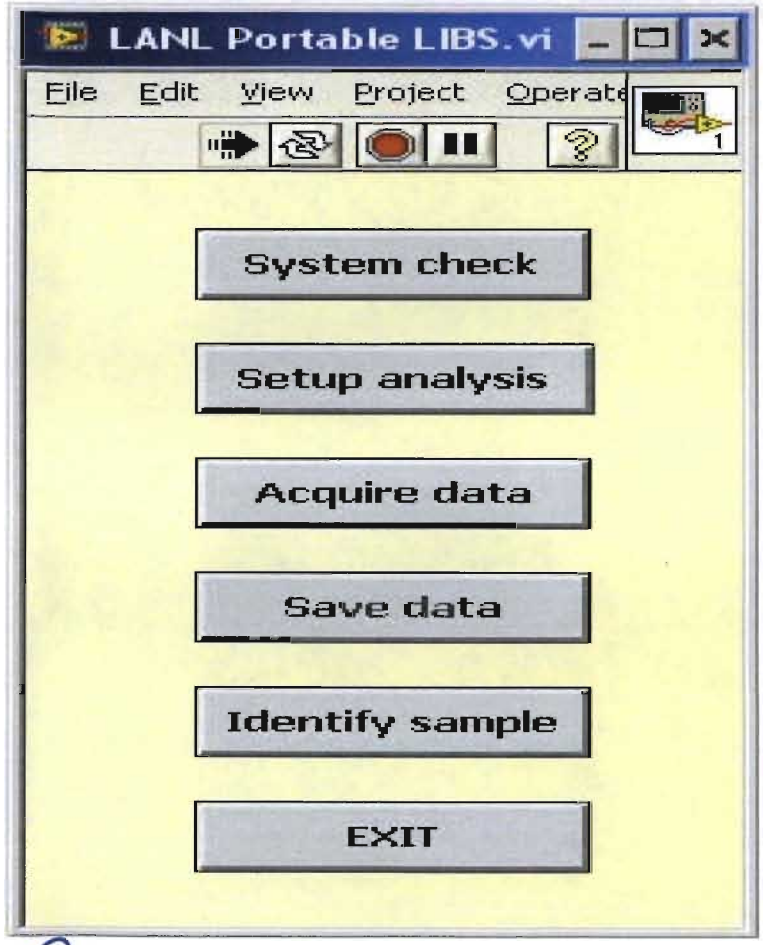


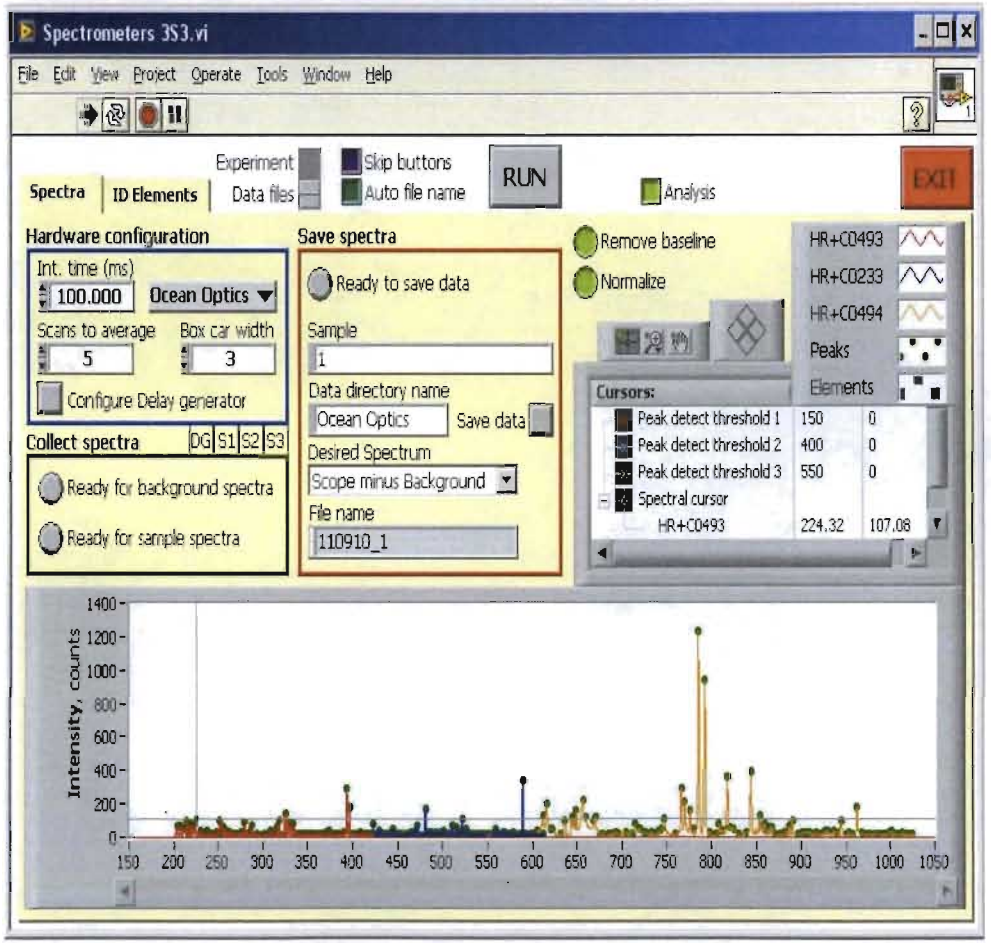
System Components (LIBS Spark on graphite, Pocket PC Data collection and Analysis)





LIBS Backpack System Control Interface





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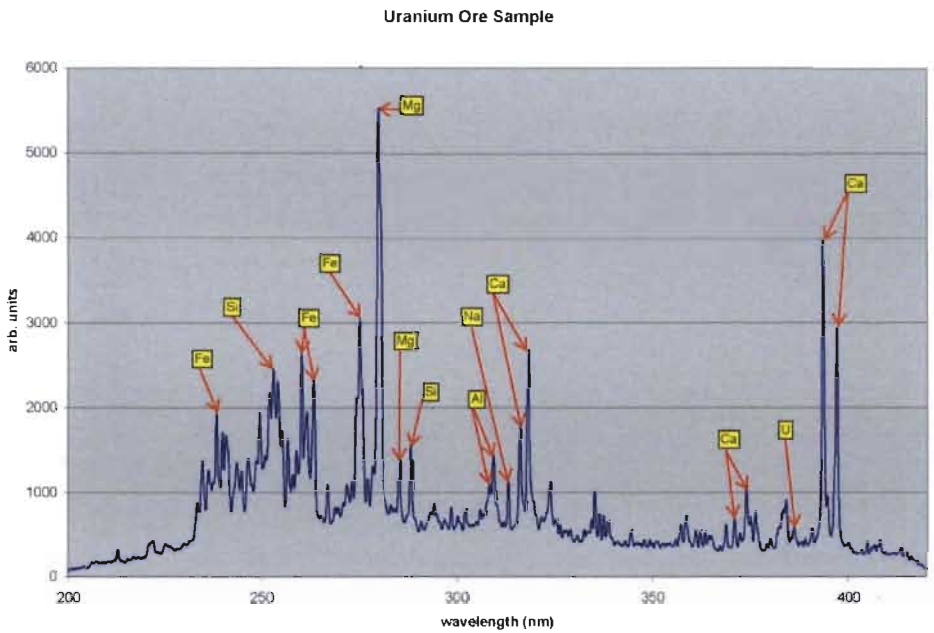
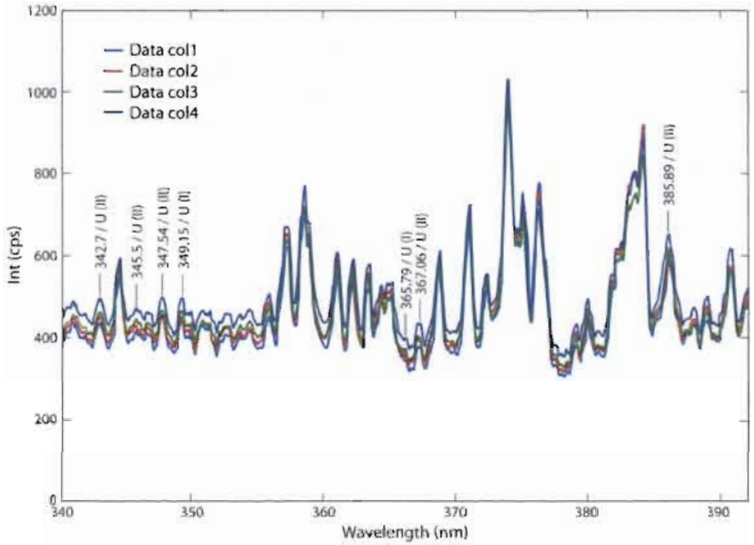
Uranium Ore sample



UORE Spectra 340 – 392 nm Region

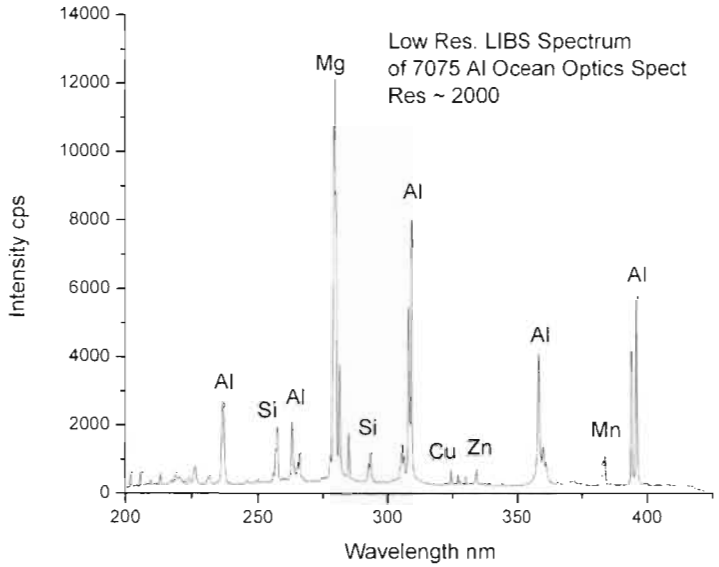
UORE Spectra Region 340 - 392 nm

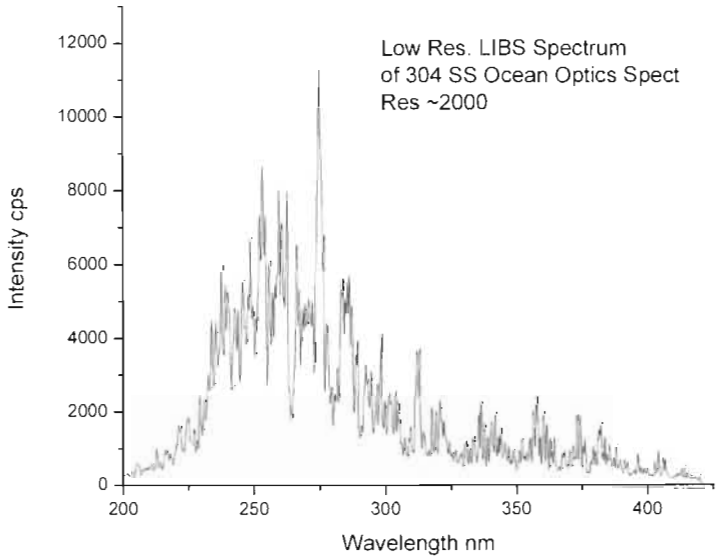


Uranium Peak Assignments

Ionization State	Wavelength nm	Wavelength nm	Ionization State
U II	268.37	389.4	U II
U II	270.63	399.82	U II
U II	277.00	401.78	U II
U II	278.44	409.19	U II
U II	295.63	411.61	U II
U II	302.22	415.4	U II
U II	310.24	424.3	U II
U II	311.16	436.1	U I
U II	339.47	462.7	U II
U I	350.76	547.5	U II
U II	353.4	548.01	U II
U II	367.01	556.4	U II
U II	385.9	597.6	U I
U II	387.4	682.8	U I

Low Res. LIBS Spectra of Al7075 and 304SS



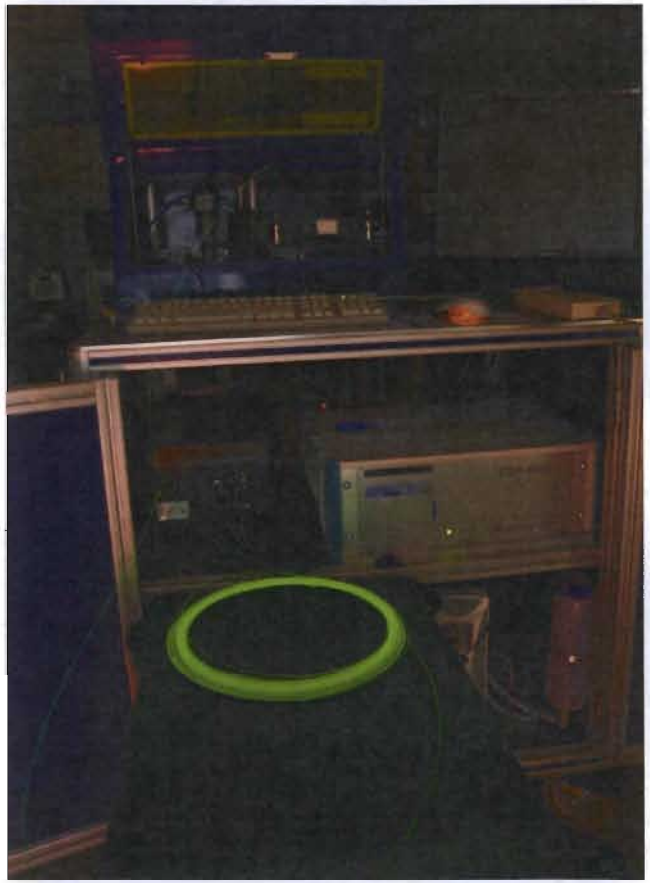


Samples Analyzed with the LIBS Backpack System

- Magnets, AlNiCo, SmCo, NdFeB
- Steels, 350 and 250 Marging, 304 and 316 SS, A36 HRS
- Carbon Steel Series 451 – 460
- Al Alloys, 6061, 7075, and 2024
- Carbon fiber and Graphite
- Natural Abundant Uranium (SRM from NIST, USA) and Ore
- Precision of ~92 % Achieved for Sample Identification
- All spectra bet. 200 and 1000nm was used

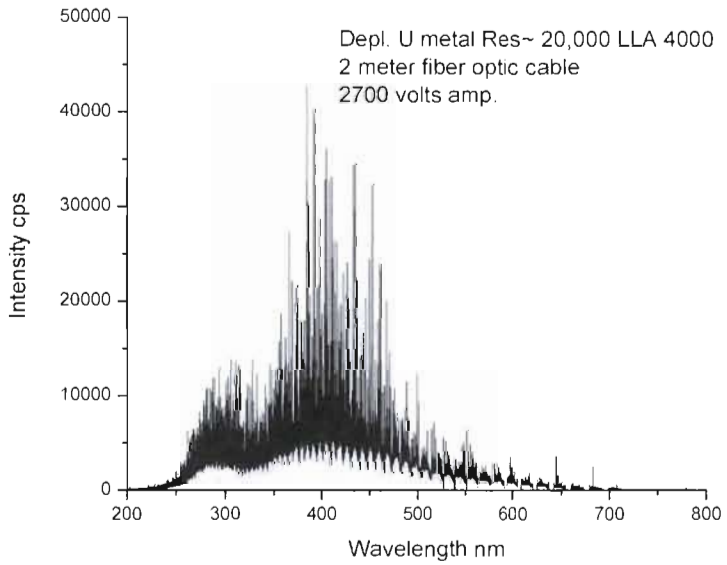
Rack / Cart Mounted LIBS System Res. (20,000)

Cart / Rack Mounted LIBS System Components

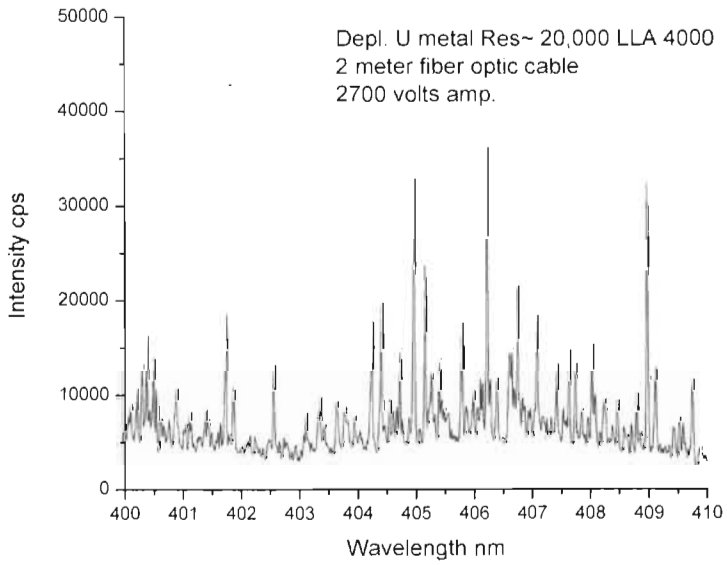


LIBS Spectrum of Depleted Uranium Sample

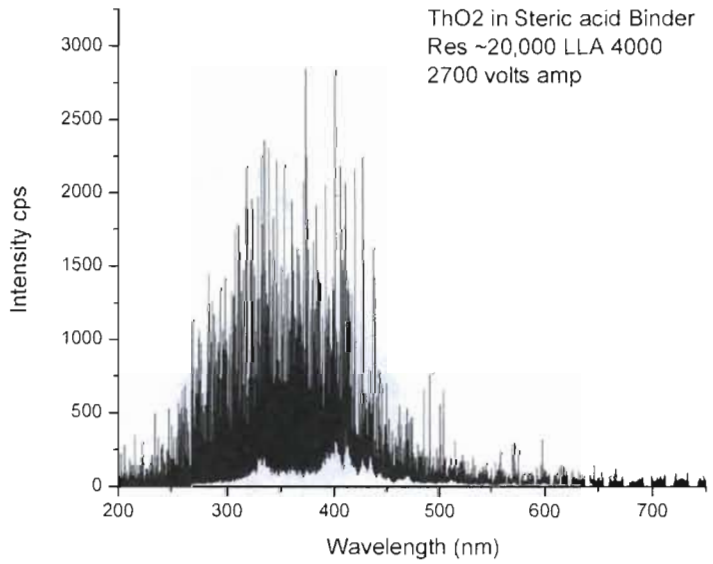
Full Spectrum

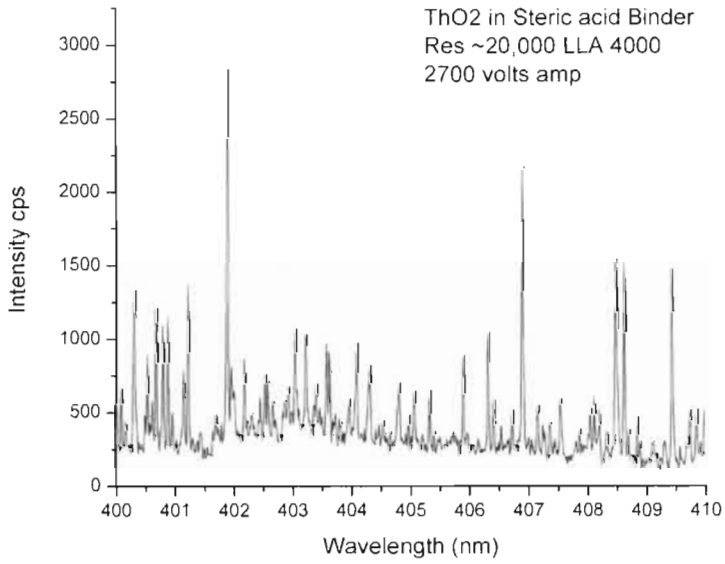


400 – 410 nm Region



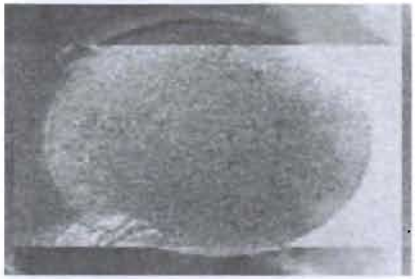
LIBS Spec. of ThO2 in a Stearic acid binder LLA 4000



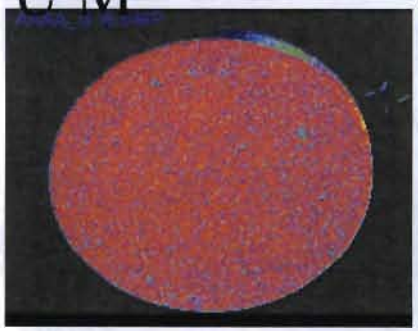


Elemental Images for ThO_2/dUO_2

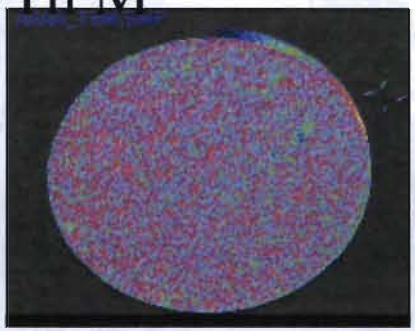
White light image (montage)



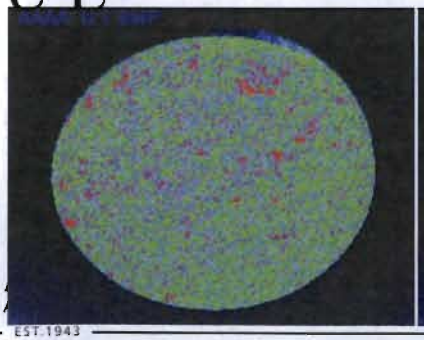
U M



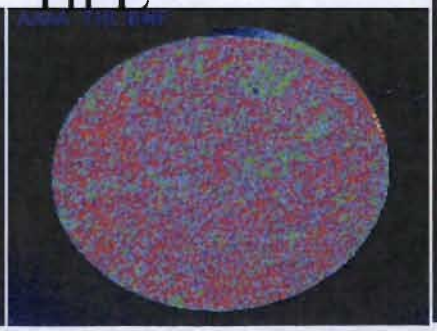
Th M



U L

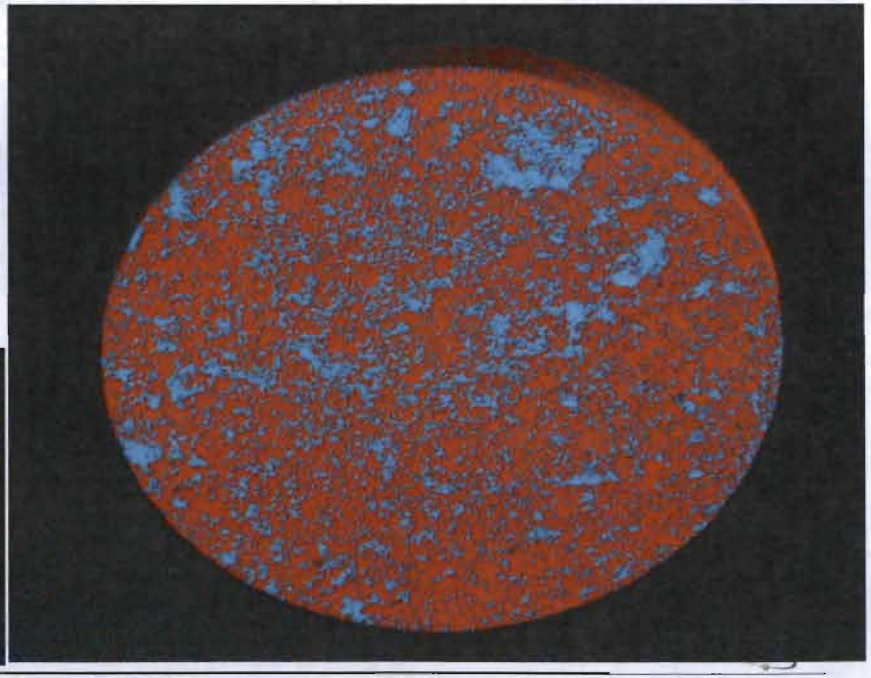


Th L

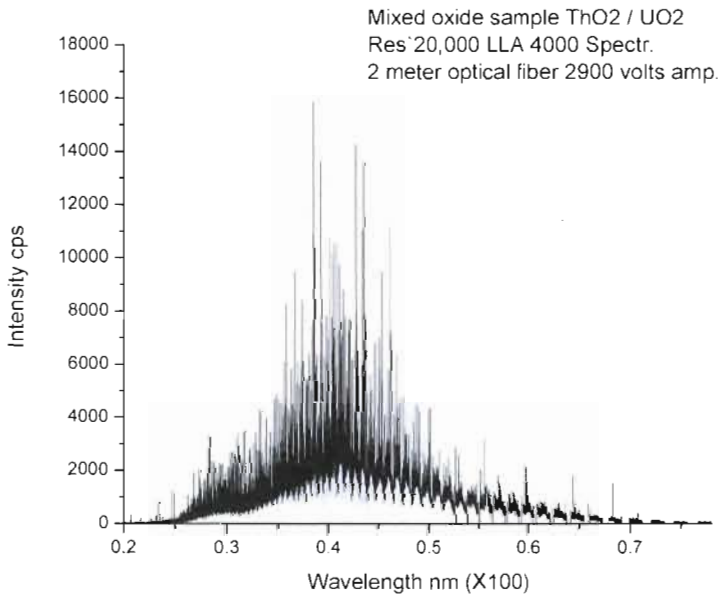


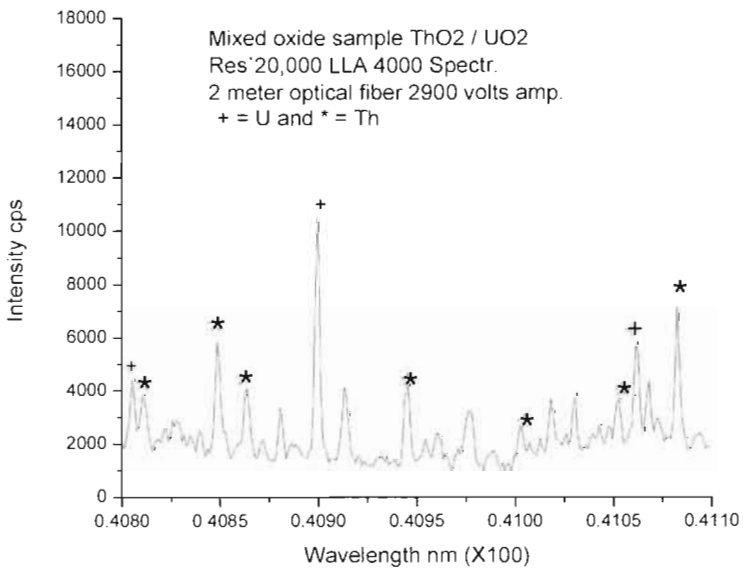
Overlay

Red = ThO_2 Blue = dUO_2



LIBS Spectrum of ThO2 / UO2 in Stearic Acid Binder





Conclusions

- LIBS instrumentation developed to support IAEA safeguards applications as part of the Next Generation Safeguards Initiative (NGSI)
- Technology is mature but the application is novel and highly flexible
  - Portable instrument (identify presence of actinides, analyze alloys, plastics, metals, and other samples)
  - High resolution fixed lab system (isotopic, elemental, and ratio analysis (Cm/Pu, Cm/U…etc))
  - Rack / cart mounted field deployable high resolution LIBS system (measurements through glovebox, hot cell windows or with fiber optic probes of lengths up to 75 meters)
  - Fiber Optic Probes Harsh Environments Hotcells Gloveboxes