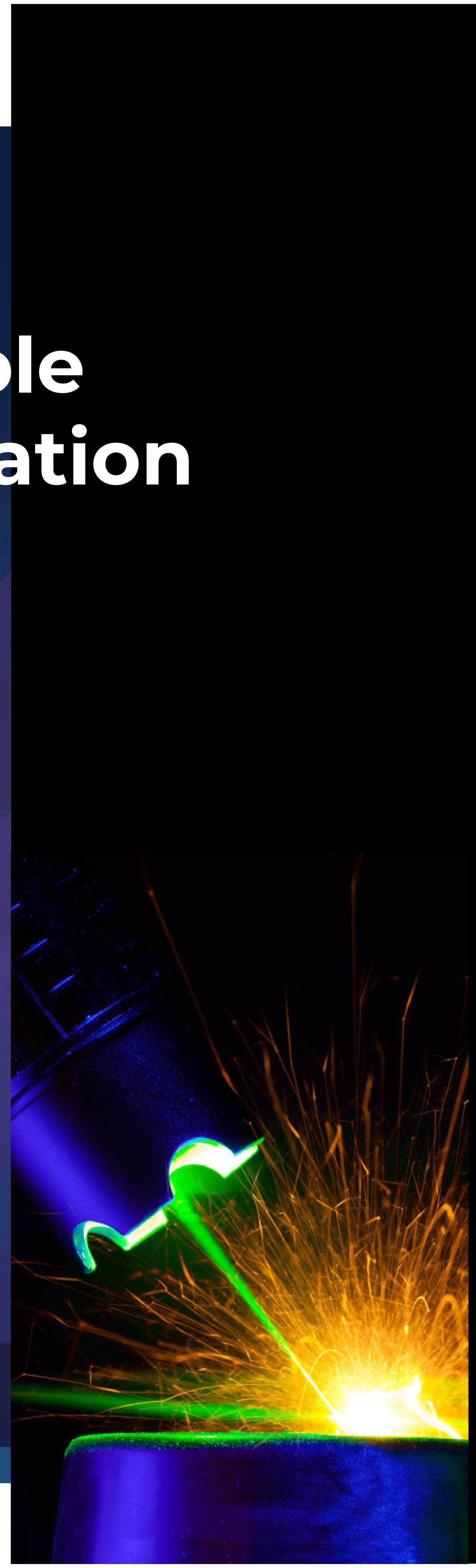
Exploring Portable LIBS Instrumentation Design



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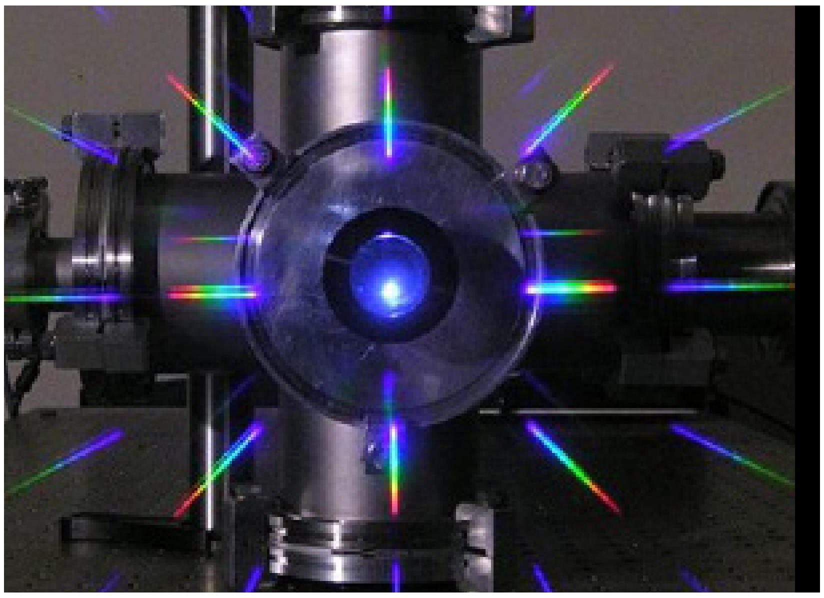

Introduction

Portable LIBS Instrument Design

Laser-Induced Breakdown Spectroscopy (LIBS) has long been used as an analytical technique for elemental analysis. LIBS is particularly attractive for detecting low atomic number elements since elements lighter than silicon cannot typically be seen using x-ray fluorescence (XRF) spectroscopy. As a result, LIBS is often utilized in applications such as lithium mining, analysis of carbon steel, and aluminum sorting. Additionally, LIBS is uniquely suited for soil and agricultural analysis due to the ability to detect carbon, nitrogen, and oxygen. LIBS has even been studied as a means of biomedical diagnostics. While this technology has been around since the 1960s, it has only been within the past decade that laser and spectrometer technology has evolved to the point where the form factor and power consumption are small enough to enable the production of handheld and portable LIBS systems.

In this webcast, Robert V. Chimenti (RVC Photonics / Rowan University) will begin by first introducing the fundamentals of portable LIBS spectrometers, focusing on understanding how the physics of plasma emission effect the overall system requirements. He will also provide a brief overview of some of the more common applications for portable LIBS technology. Afterward, Damon Lenski (Avantes) provides an overview of miniature spectrometer technology and the critical specifications when configuring a spectrometer for portable LIBS applications. Next, Jason Yager (Lumibird) will provide an overview of laser parameters essential for successful integration in portable LIBS instrumentation. Finally, all three presenters will wrap up the webinar by hosting a live round table question and answer session with the audience.

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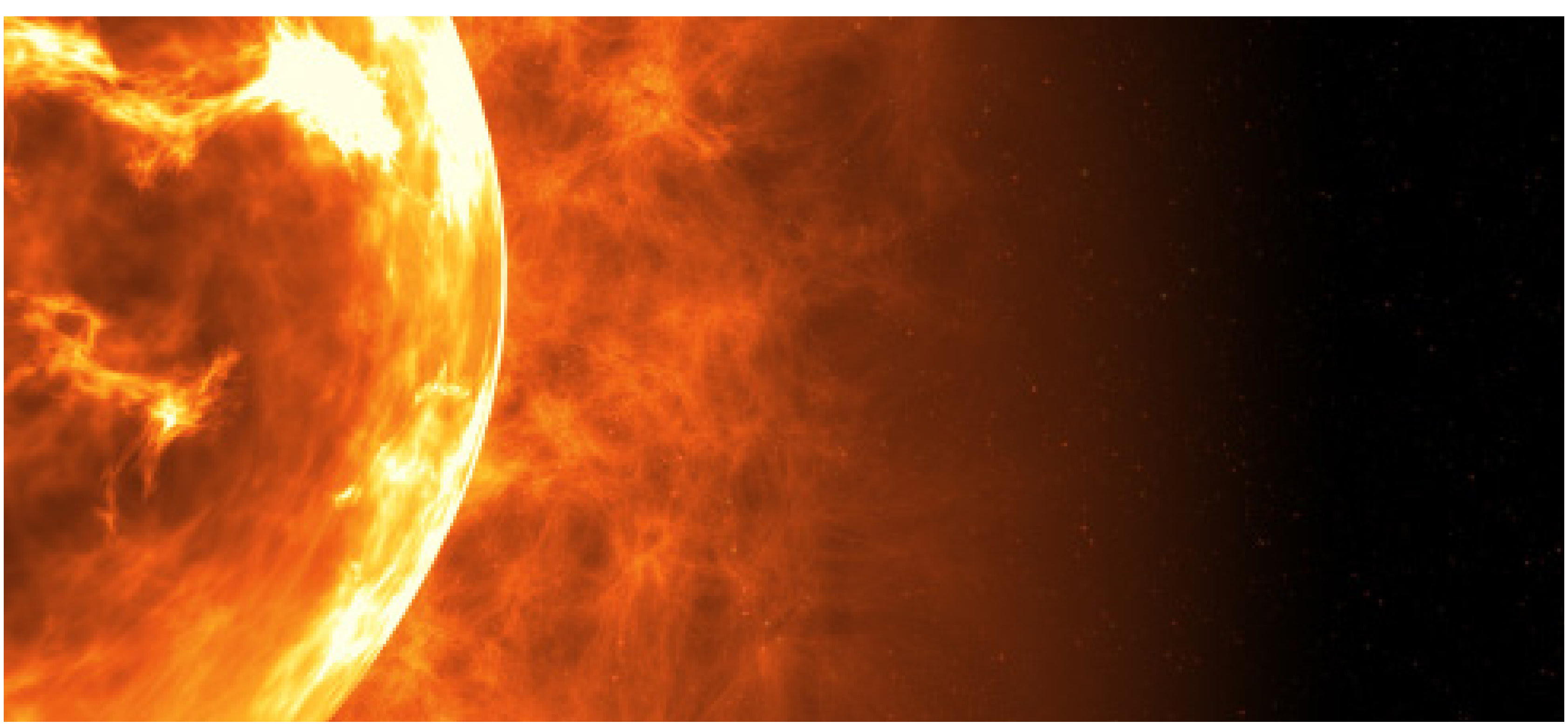

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Atomic Emission Spectroscopy

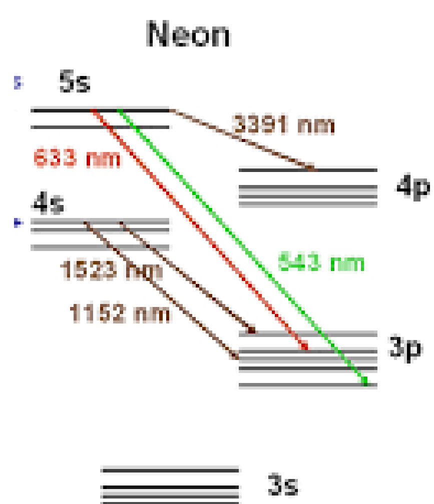
When the electrons in an atom are excited from the ground state to a higher energy level, they emit a unique spectral fingerprint associated with the allowed energy levels.

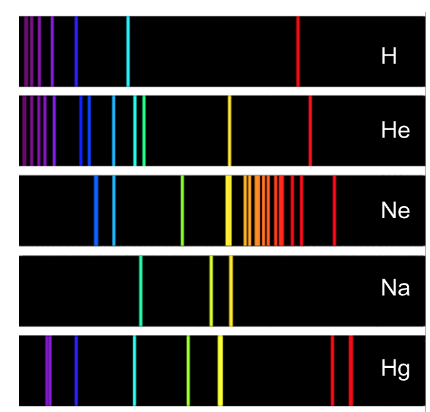
$$E_n = -\frac{M_r Z^2 q_e^4}{2\hbar^2}\frac{1}{n}, n = 1,2,3,..$$

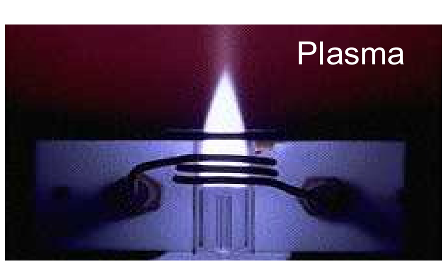
Single Electron Atom

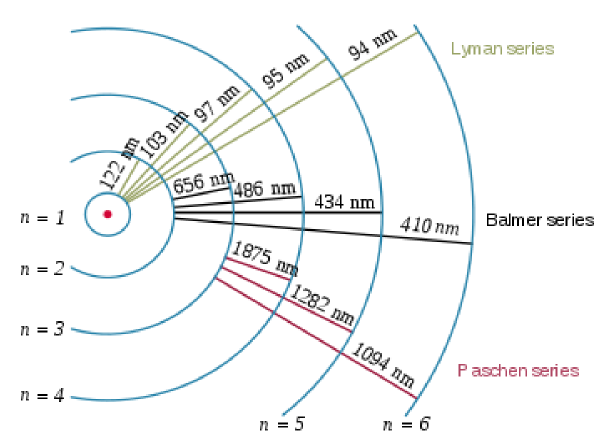
$$\text{H}: E_3 - E_2 = 1.9\text{eV} \Rightarrow 655.5\text{ nm}$$

$$\text{C}^{5+}: E_3 - E_2 = 68.1\text{eV} \Rightarrow 18.2\text{ nm}$$









Laser Induced Breakdown Spectroscopy

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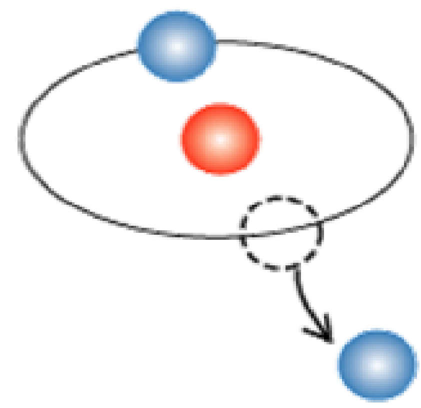
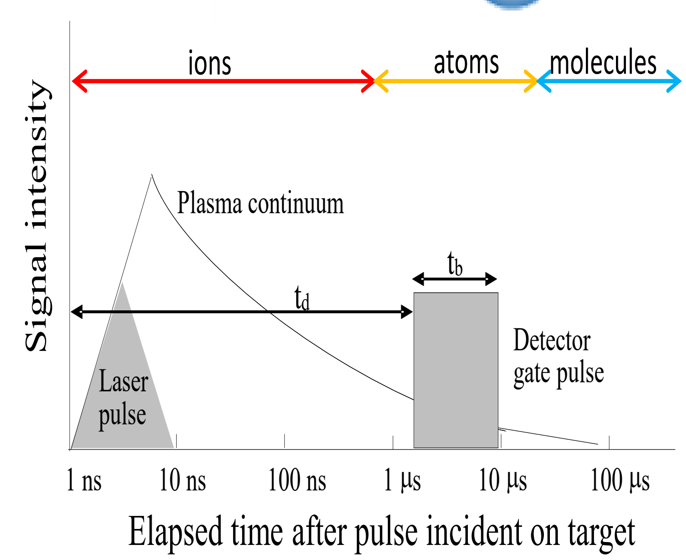
LIBS is an AES tedchnique that uses a focused high energy laser to ionize a sampe resulting in atomic emissions.



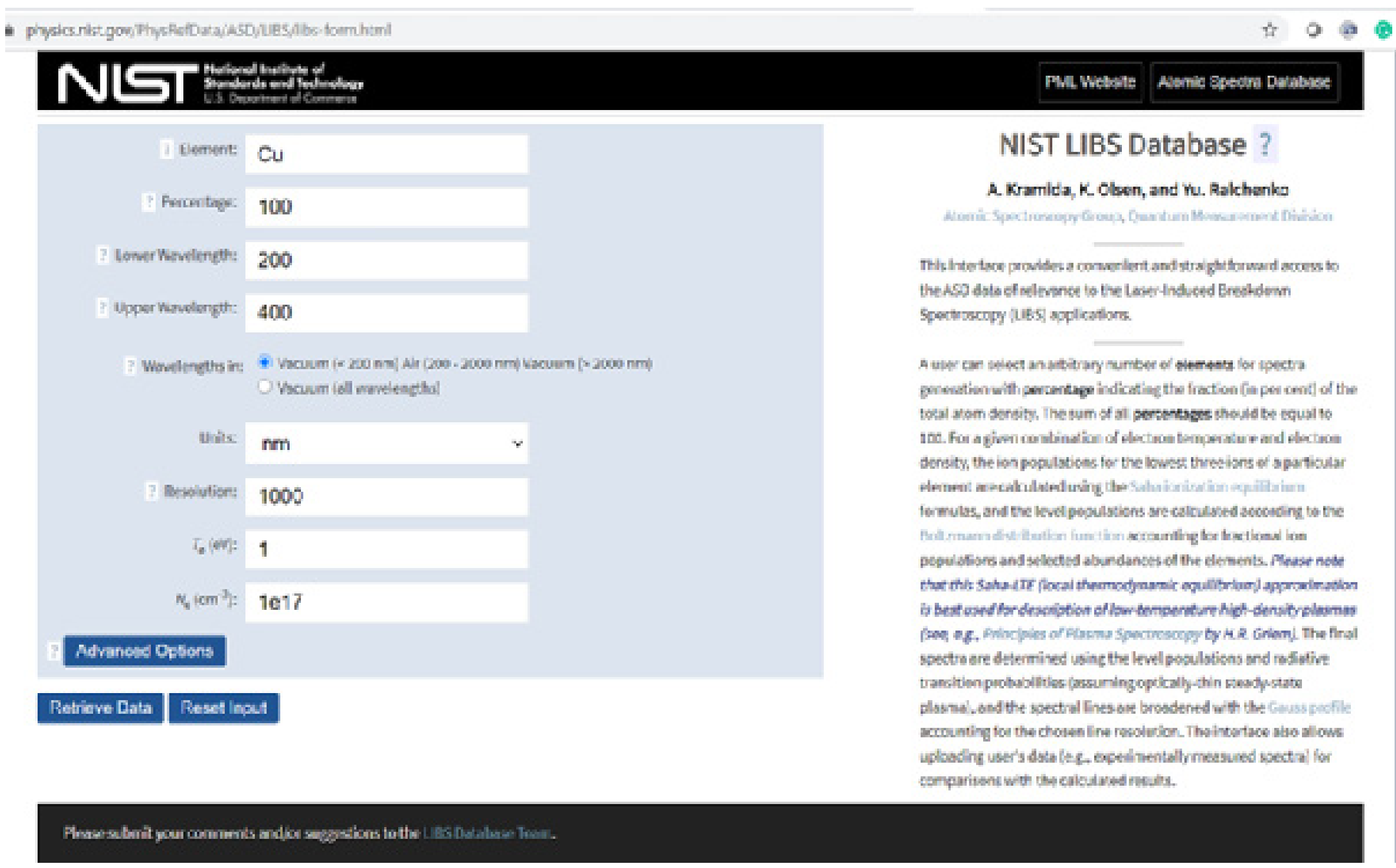
Advantages of LIBS

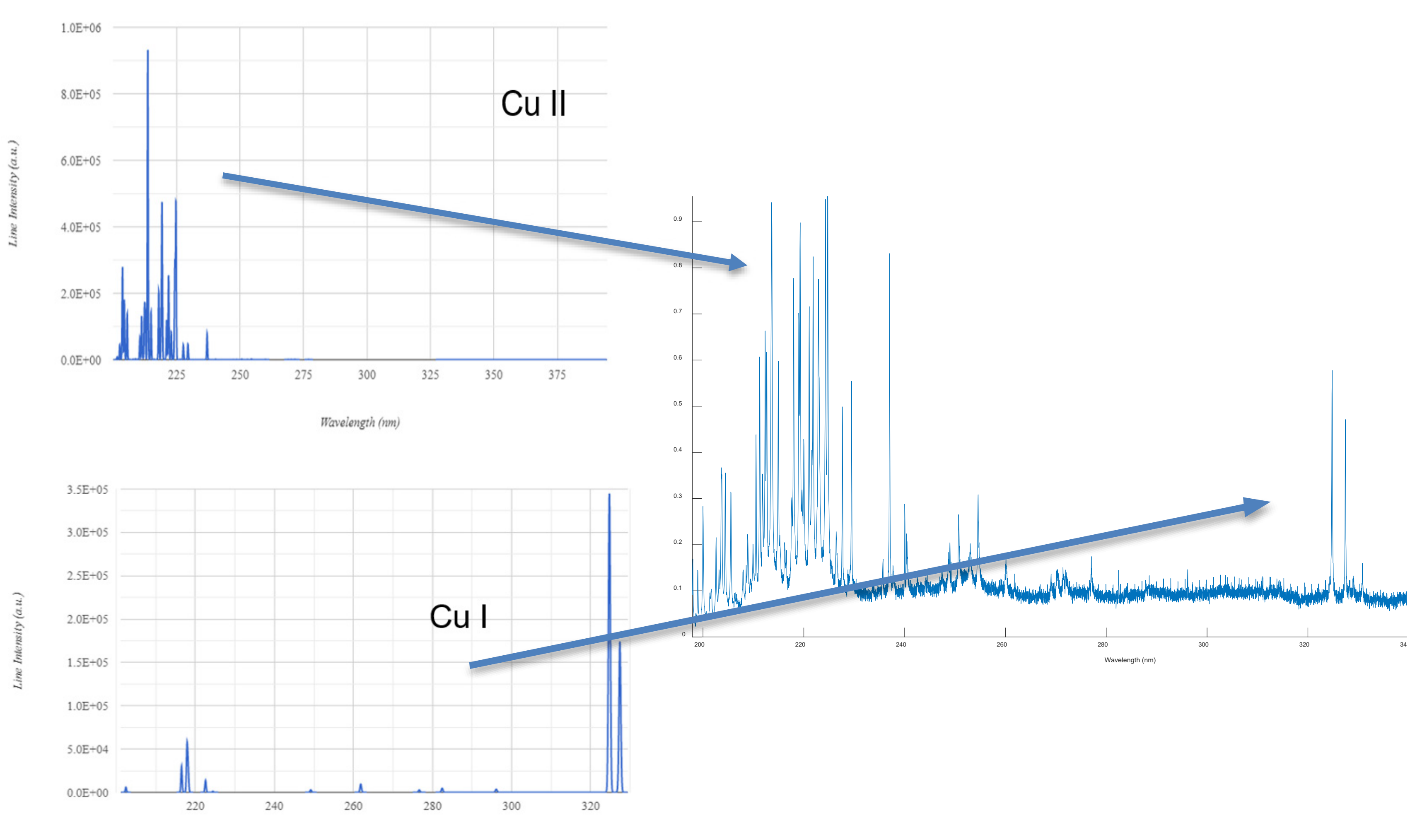
- No need for sample preparation
- Only AES technique that doesn't require prep
- Minimal Sample Consumption – "micro-destructive"
- µg level sample ablation per shot
- ~100 µm laser spot (typical)
- Rapid measurements (real-time)
- ~1 ms per spectrum
- Non-contact technique
- Analysis of samples behind a window
- Detection of smaller atoms including C
- XRF is typically limited to Si and up.



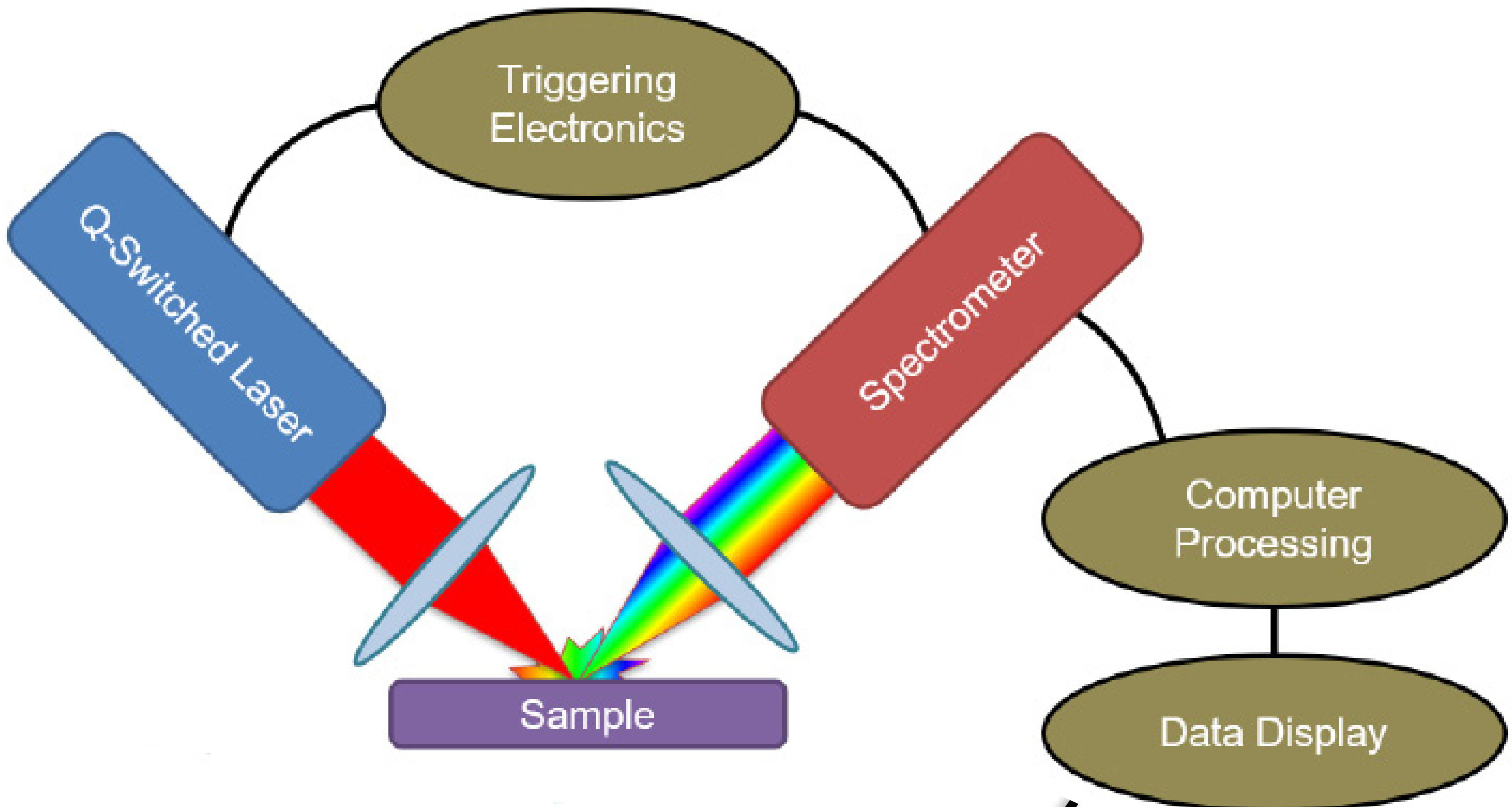
Typical threshold ~1011W/cm2 for gasses and ~1010W/cm2 for liquids, solids, and aerosols.

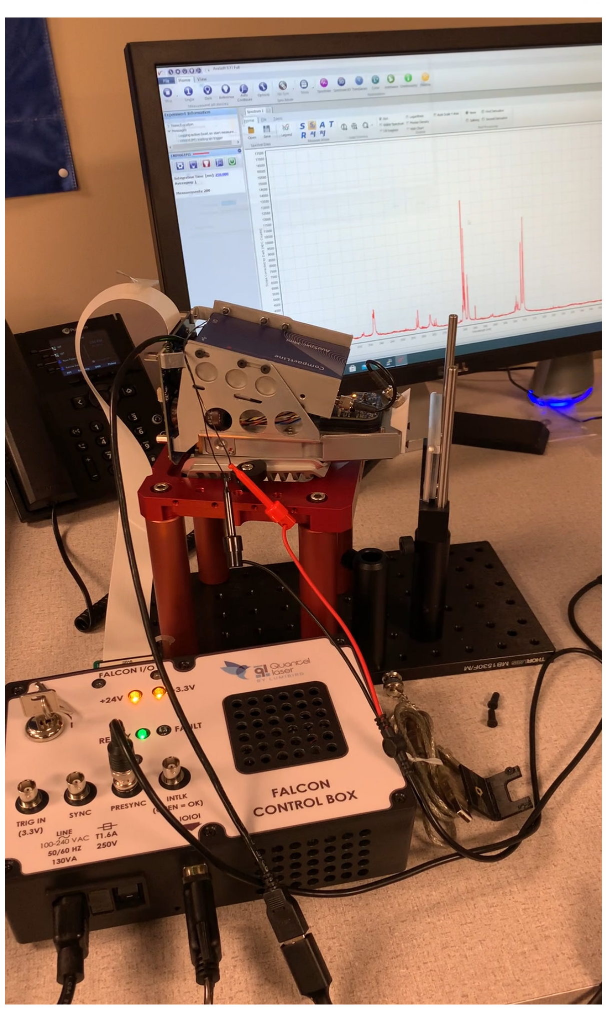
Interpreting LIBS Spectra

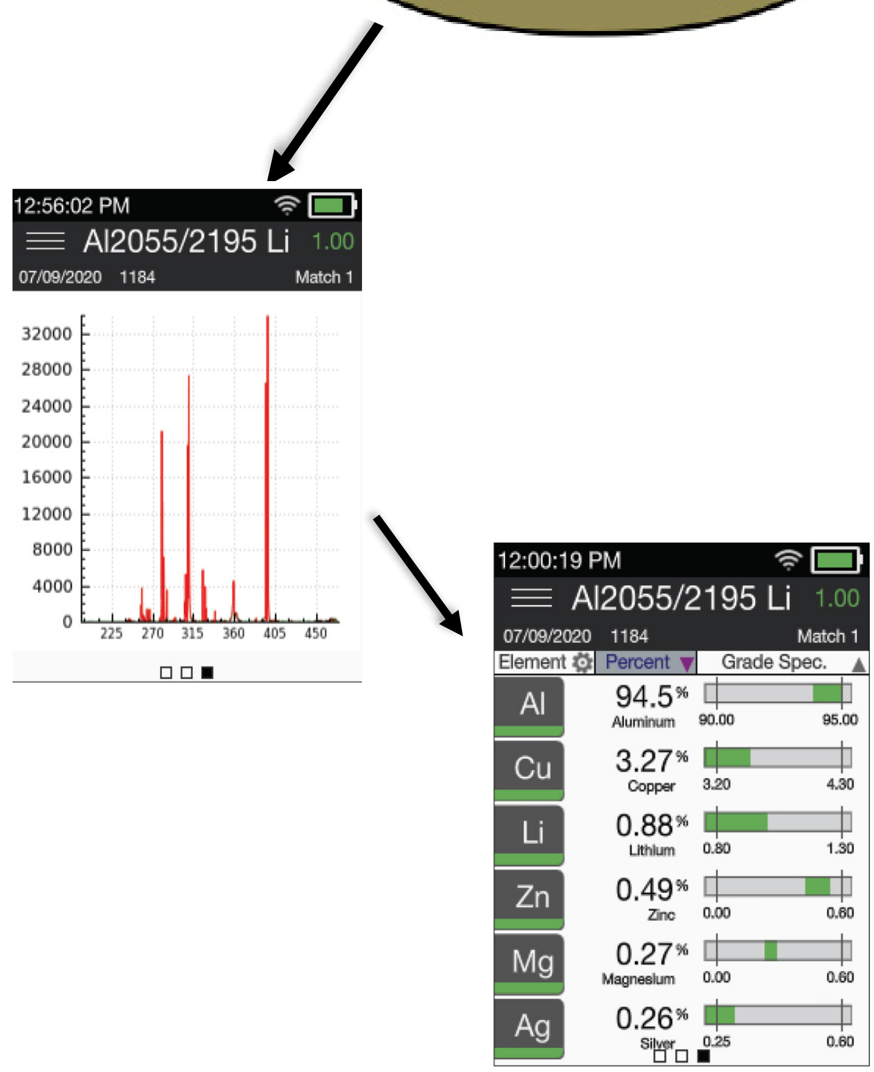




Components of a LIBS System







Portable LIBS Technology

The first handheld LIBS was released in 2013

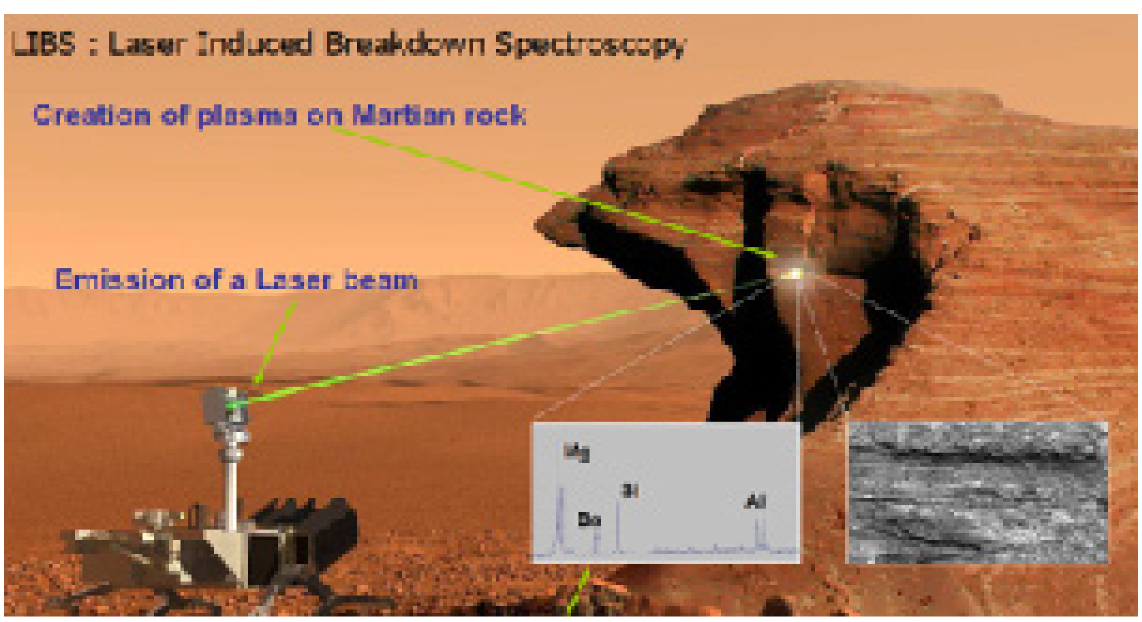
- *Prior to that portable "suit-case" size LIBS were available*
- *The first LIBS system landed on MARS in 2012*

Allows you to bring the lab to the sample

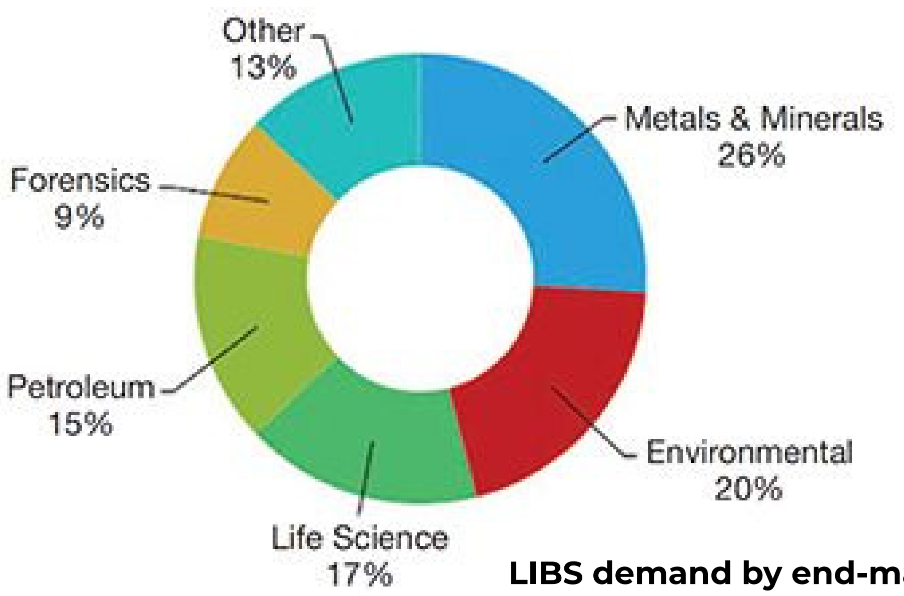
- *Ideal for in situ analysis*
- *Ergonomic Design*
- *On-Board Data Processing*

Currently Eroding the Portable XRF Market

- No need for controlled X-ray emitters and detectors
- No need for X-ray certified operators



Applications of Portable LIBS

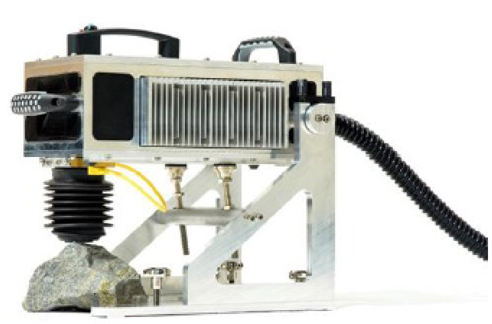


LIBS demand by end-market for 2018

TDA's Industry Data

Metal Alloy Sorting

- Recycling, Scrap &QA
- Carbon Steal
- Aluminum Alloys



On-Site Mining Testing

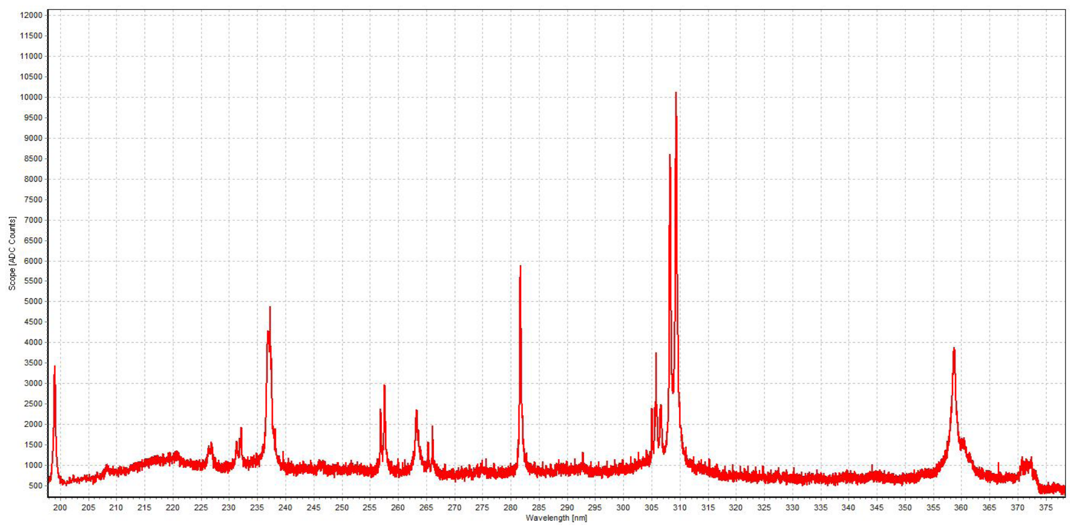
- Lithium in Brine
- Gold in Rock Samples
- Bitumen Content in Oil Sands

Agriculture

- Contaminants in Soil and Water
- Soil Nutriesnts Detection

Aluminum Sorting Example

Al-1100 vs Al-6063

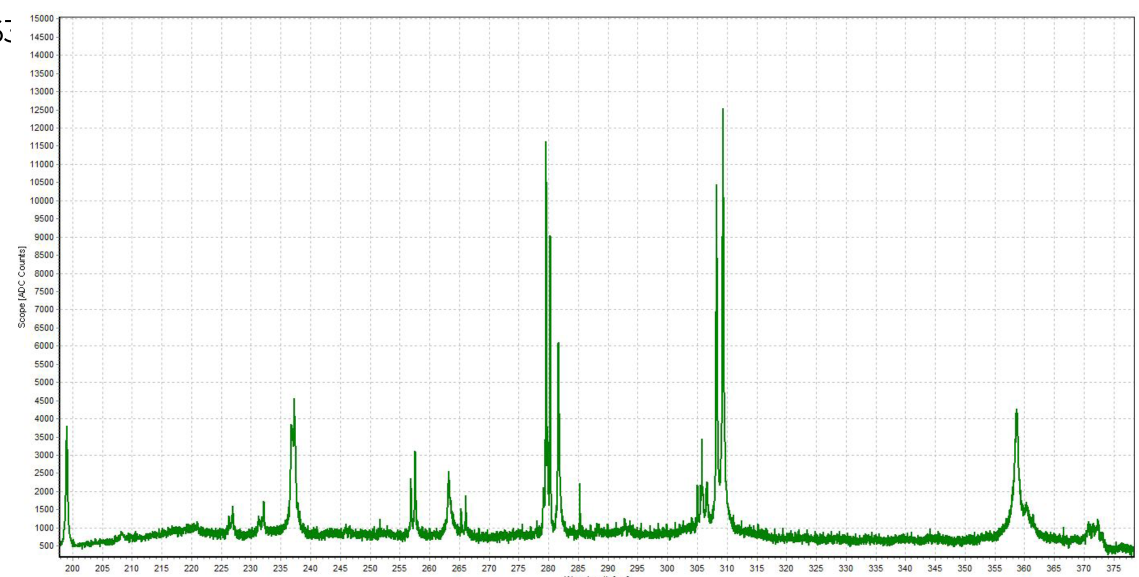


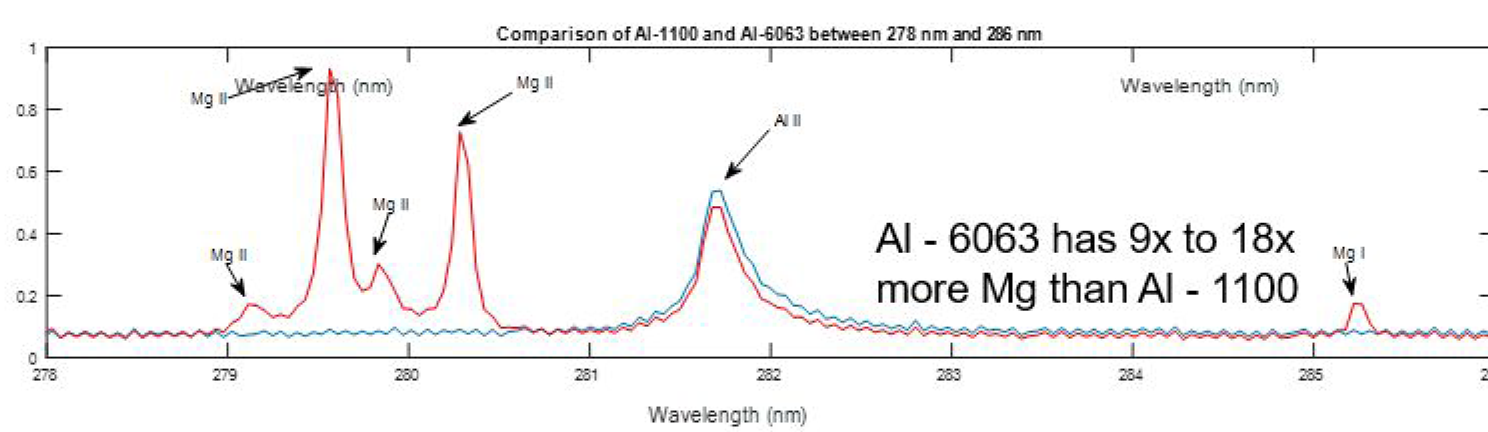
Characteristics of Aluminum-1100

- Al: 99.0–99.95%
- Cu: 0.05–0.20%
- Fe: 0.95% max
- Mg: 0.05% max
- Si: 0.95% max
- Zn: 0.1% max
- Other: 0.15% max

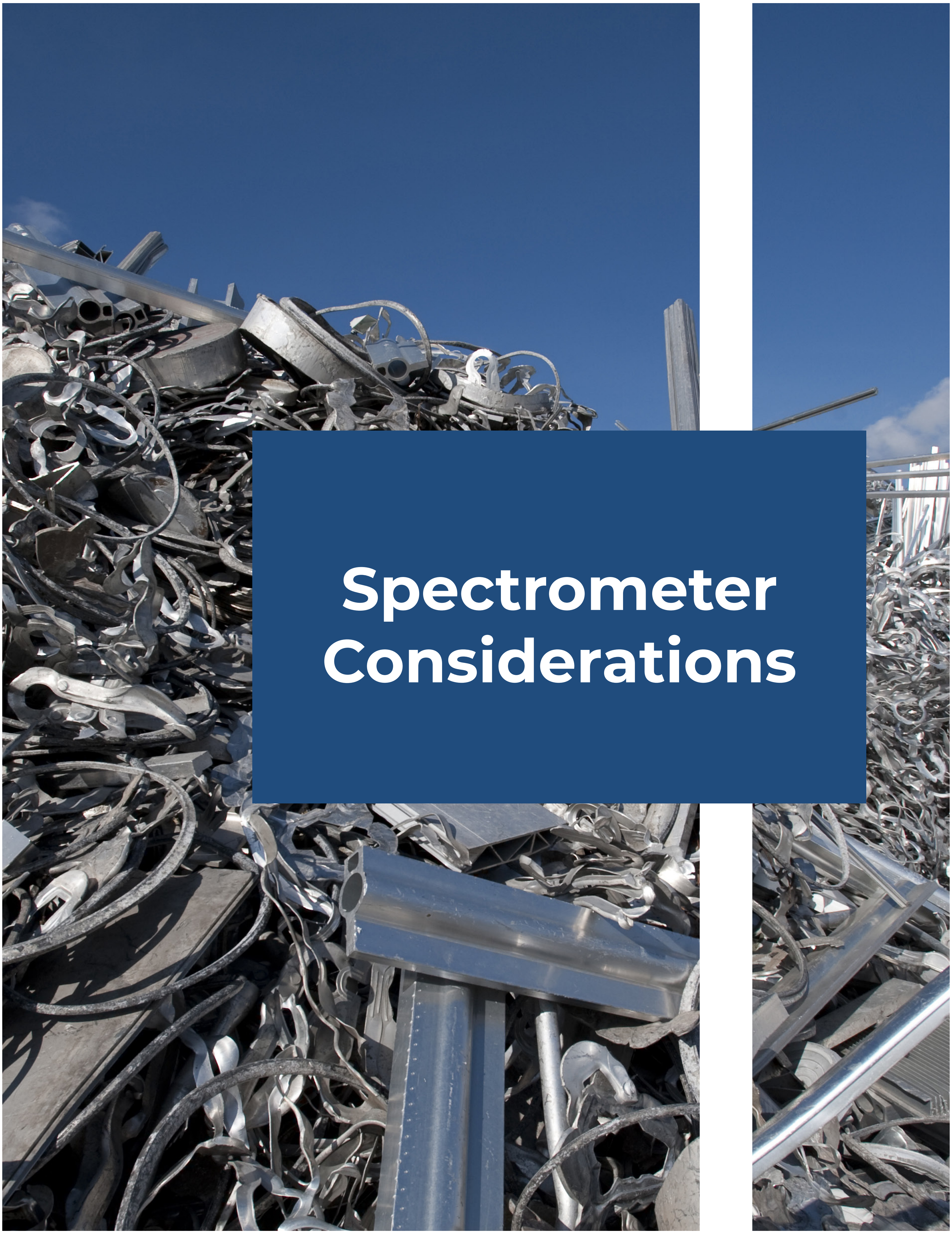
Characteristics of Aluminum-6063

- Al: 97.5–99.30%
- Cr: 0.10% max
- Cu: 0.10% max
- Fe: 0.35% max
- Mg: 0.45 -0.90%
- Si: 0.20 -0.60%
- Ti: 0.10% max
- Zn: 0.1% max
- Other: 0.15% max





Click to See and Download the Experiment Data



Spectrometer Considerations

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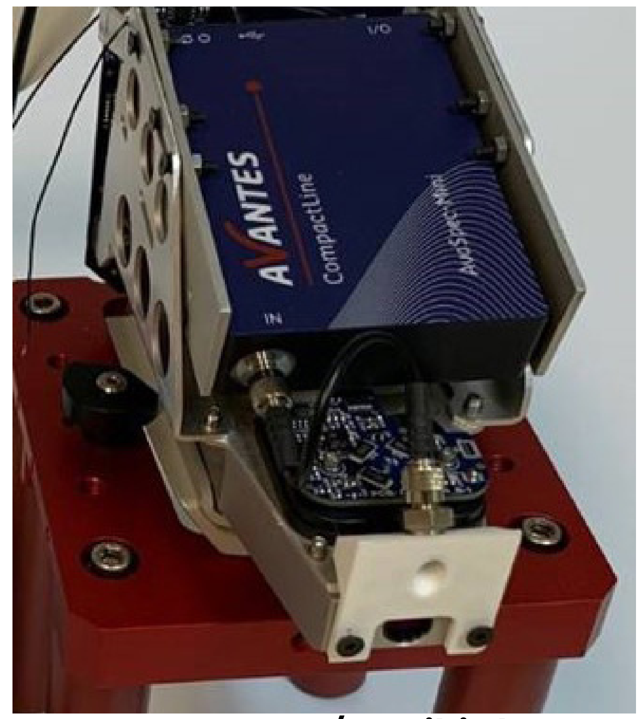
Spectrometer Considerations for Portable LIBS



Avantes/Lumibird Demo Kit

Avantes Spectrometers for Portable LIBS contain the following features:

- Configurability
- Robustness
- Size
- Resolution
- Sensitivity
- Speed/Timing
- Communication

Configurability

Numbers of Grating Options

- Gratings have variable sensitivity over their range

Slit Size Options

- Balance Throughput with resolution

Order supressing filter options

- control second order effects

Detector Options

- 2048 or 4096 - more pixels =higher resolution

Purge capabilities

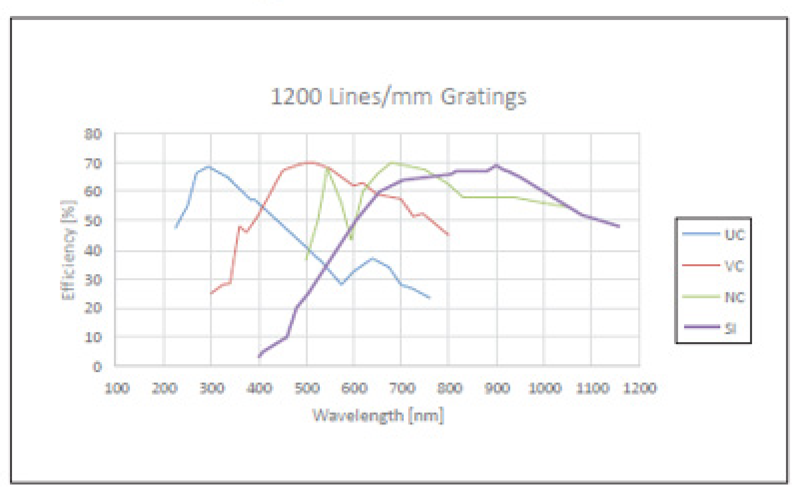
- For vacuum UV <185 nm

Multi-channel capabilities

- *Broader range @ higher resolution*
- *Greater range of detected elements/more specificity*

Grating Efficiency Curves

1200 lines/mm Gratings



AvaSpec-ULS Optical Bench

AvaSpec-Mini Spectrometer

Robustness

Thermal Mechanical

- Wavelength stability in different environments
- On board thermistor in spectrometer for wavelength correctionwith temperature drift
- Device MUST be tested – Avantes 100% thermal testing

Shock and Vibration

- Ability to withstand impact and vibration
- AvaSpec-Mini designed for integration in portable instruments – fixed slit, optics

Minimal Power Requirements

- Reliant on battery power with laser consuming most of budget
- AvaSpec-Mini 500 mA

Size

Size matters with portable instrumentation

Overall instrument footprint

- Trade off between size and resolution - function of optical focal length

Weight

- Minimal contribution to total device weight <200 grams

Correlation to wavelength range

- High resolution optical design constrains wavelength coverage
- Range is inversely proportional to resolution
- More than one instrument may be required to cover

Resolution

Resolution is KING in LIBS

- Target resolution of 0.1-0.2 nm (FWHM)

Key Determinants

- Slit size (width)
- slit width controls light throughput and resolution

Grating

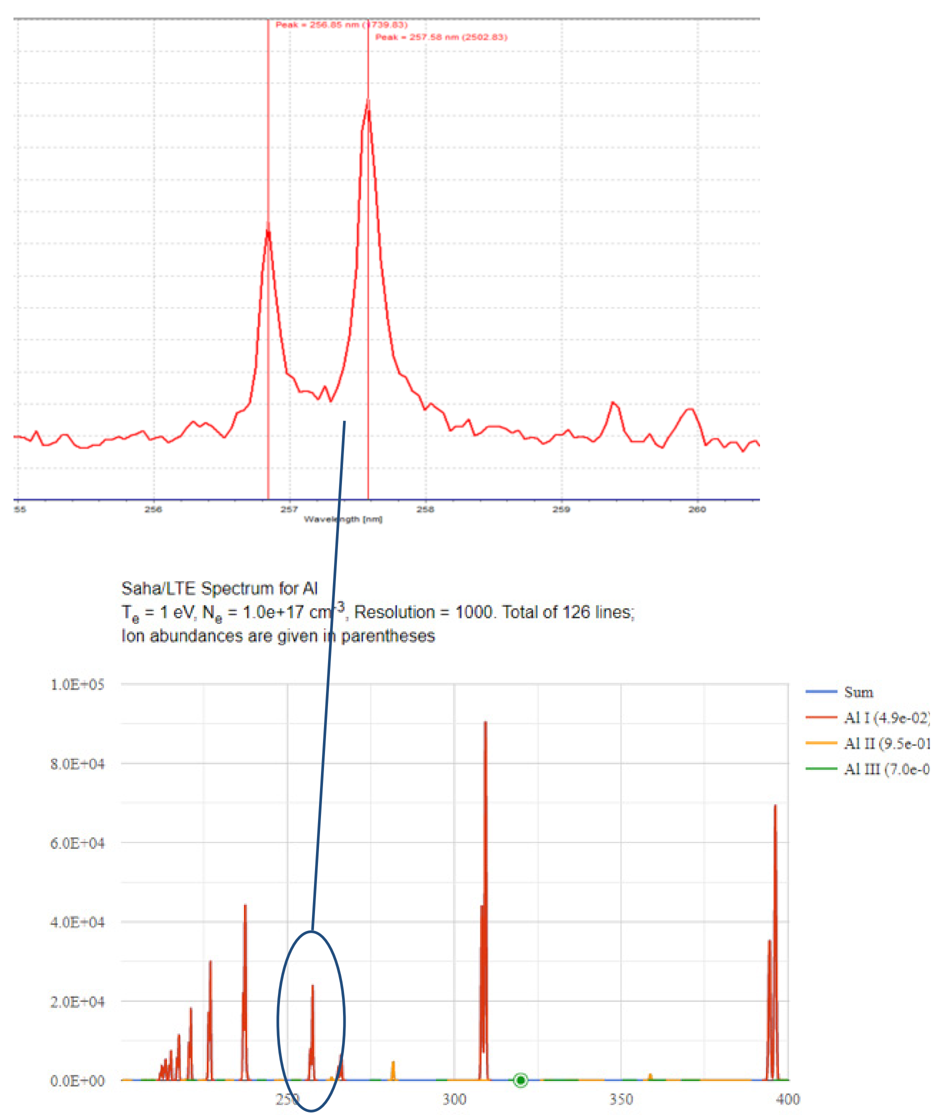
- Slit size (width)
- Grating dispersion should be adequate to allow for 8-15 pixels per nm for adequate spectral resolution – typical 1200 -2400 grooves/mm for portable = 0.1-0.15 nm FWHM

Pixel Count

- Detector must have adequate pixel count to provide resolution/datapoints over desired range
- 2048, 4096

Optical Design

- *Should favor resolution*
- *Numerical aperture correlated to res*



Sensitivity

Every photon counts!

Rep rate vs laser power

Slit size (width)

Cylindrical detector lens

- Utilizes slit height with larger core fibers to gain sensitivity – more photons on detector

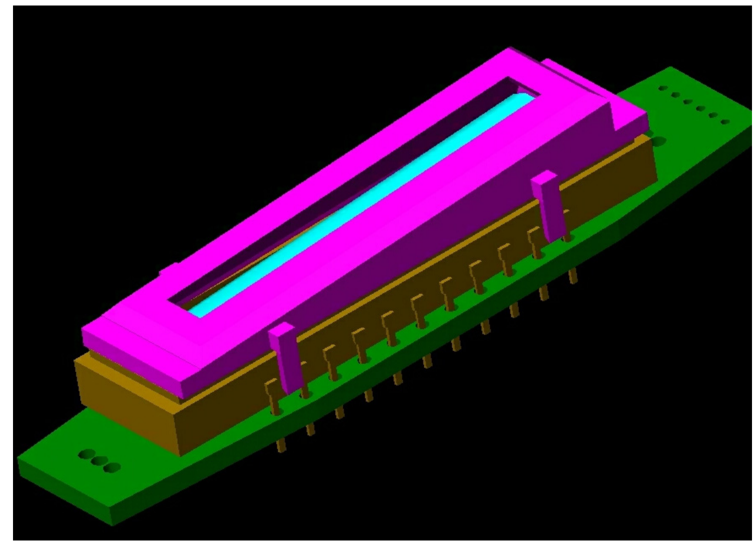
Detector sensitivity/Grating efficiency

Grating dispersion

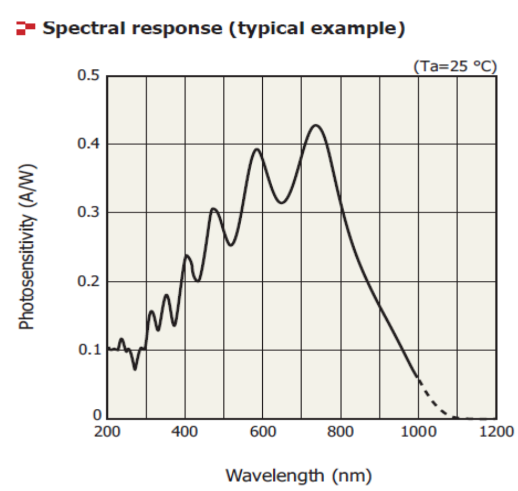
- Higher groove density = Higher dispersion & Lower photon density

Signal to noise

- May be improved with on board averaging
- Partly determined by laser fluence



Detector Collection Lens



Detector QE Response Curve

Speed/Timing

Must be able to measure repeatably at the same moment in time…every time!

Integration time minimums

- Detector dependent
- 1 ms or lower

Laser Delay Programmability

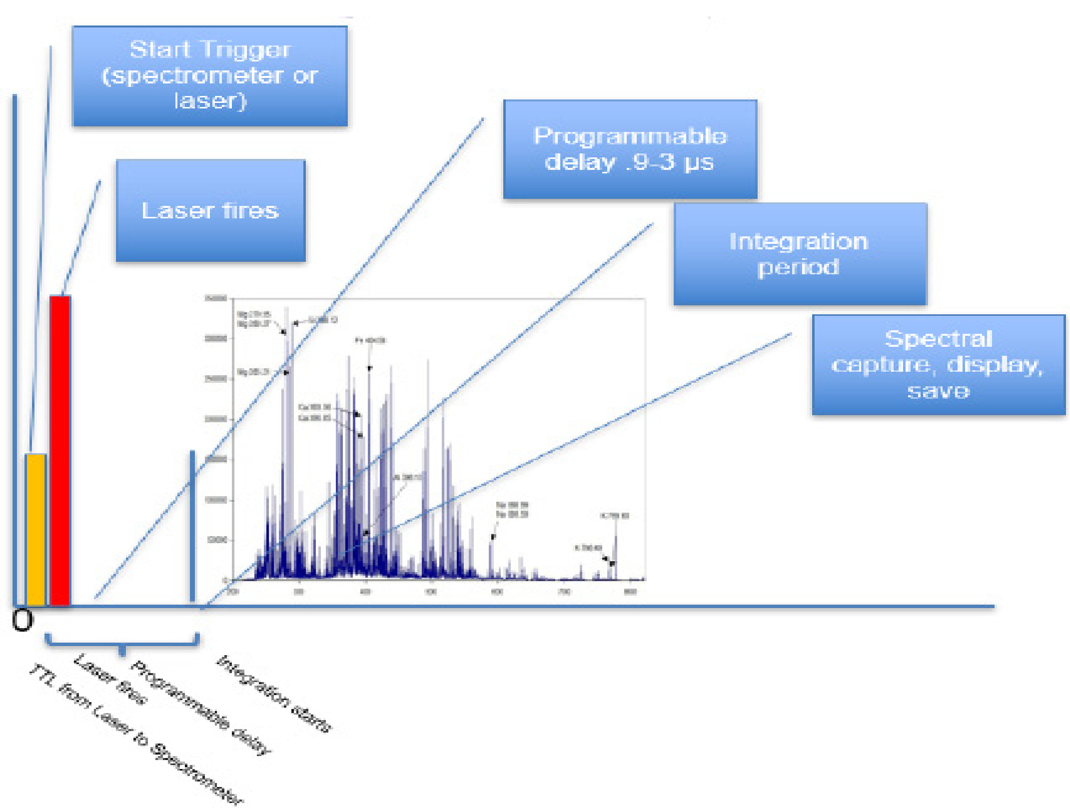
- Subject to spectrometer electronics
- 890 ns to 1 microsecond

Data Transfer Speeds

- 5-10 ms/spectra or faster

Jitter

- Uncertainty after trigger as to the start of integration



Communication/ Control

Protocols for interfacing with CPU

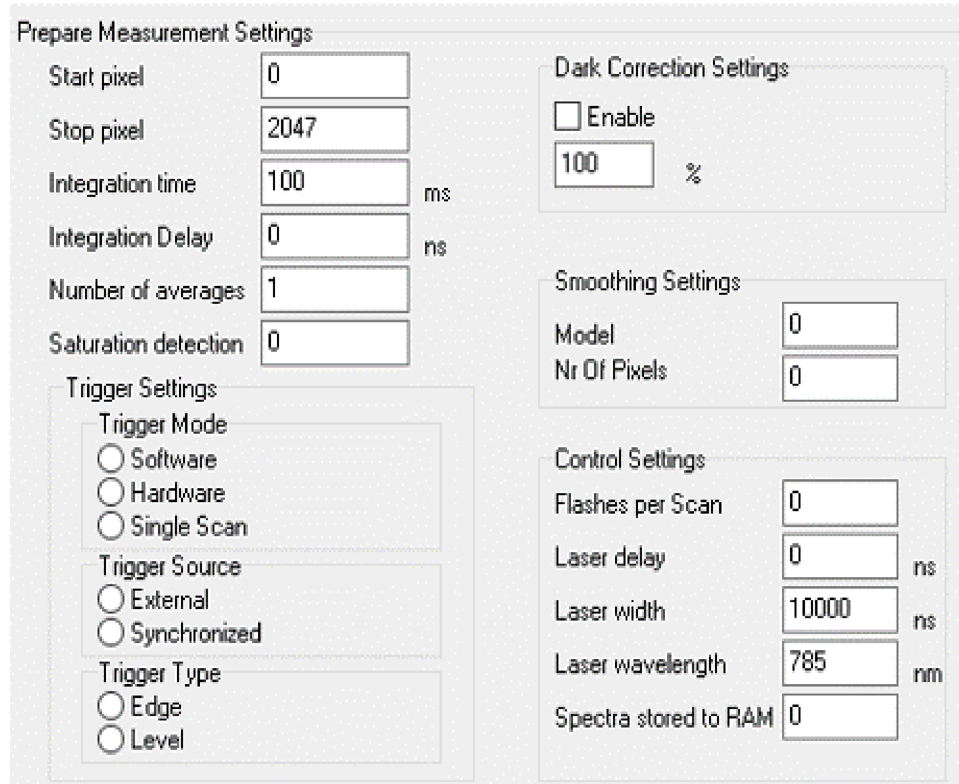
- Linux
- Windows
- Mac

Software Options

- Labview, C#, C++, Visual Studio, Ubuntu, etc

USB2/USB3/Ethernet

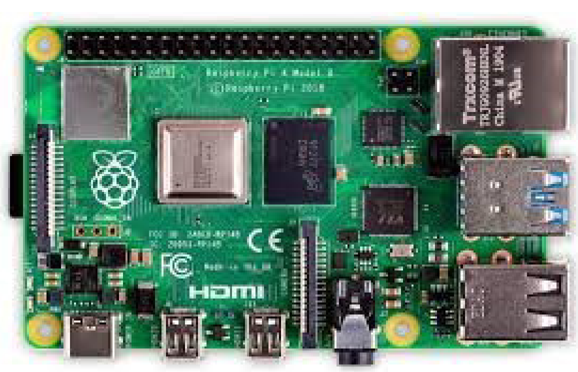
Laser control parameters integrated in software



Laser/Interface

Spectrometer triggering laser

Laser triggering spectrometer



Raspberry Pi

Beaglebone

Optical Coupling Considerations

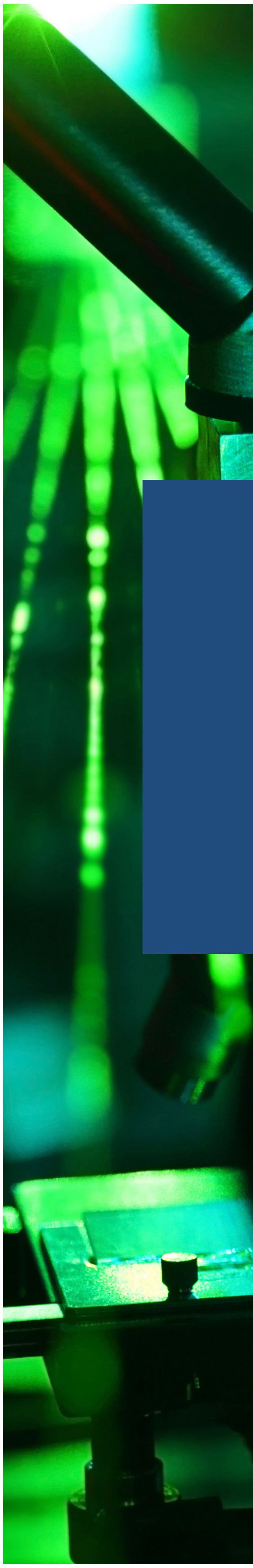
Spectrometer illumination options

Free space

- Superior throughput with suitable optics
- Collimating/focusing lenses
- Necessary for VUV applications

Fiber optic

- Greater flexibility
- High temperature/harsh environments
- Configuration options
    - Bundles
    - Round to linear fibers



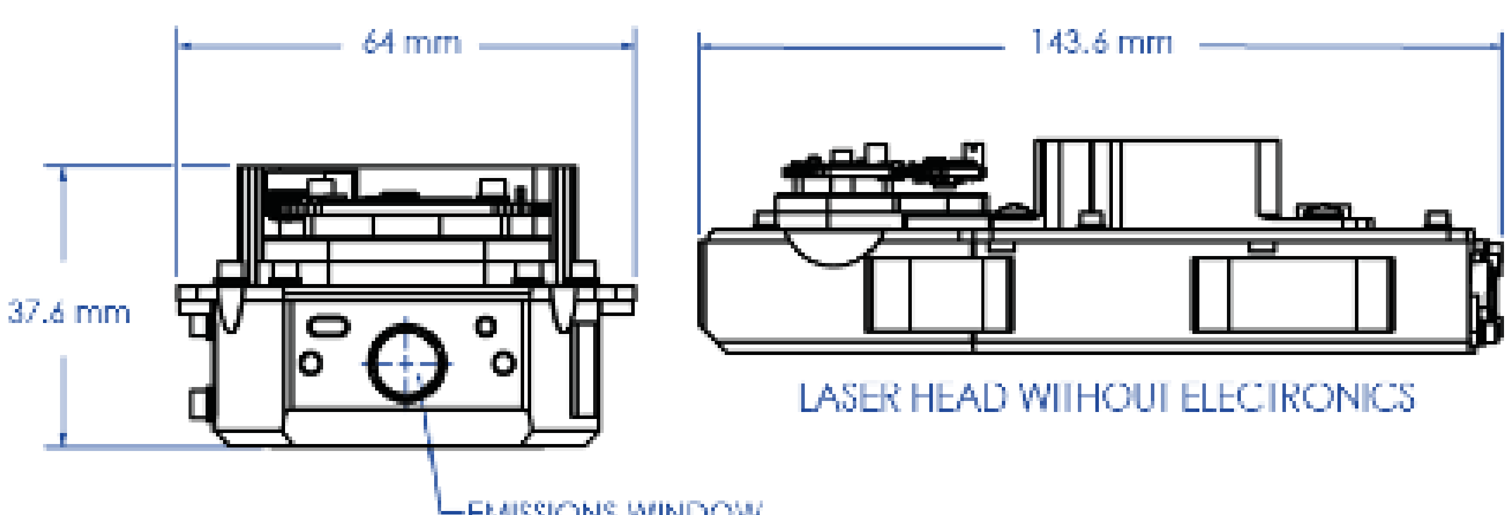
Laser Sources

17 Design Considerations for Industrialized Laser Sources for Handheld Devices

- Miniaturization for handheld applications
- Improved efficiency for battery operation
- Passive vs Active Q-switching Timing
- Environmental Reliability
- Energy and beam quality
- High rep rate vs low rep rate
- Wavelengths

Compact Design for Handheld LIBS

- Battery Powered
- Diode-Pumped Solid-State
- Sealed IP66 housings
- Folded Resonators
- Intracavity Wavelength Conversion



Lumibird Falcon LIBS Laser

Passive vs Active Q-switching

Passive Q Switching

Technology

- Cr4+:YAG solid-state passive Q-switch material

Repetition Rate

- Higher - kHz

Pros

- *Eliminates the need for a high voltage power supply*
- *Lower Cost*
- *Reduce packaging size and weight*
- *Raster Scanning*

Cons

- Timing for Plasma Signal
- Can drill a hole quickly shadowing the plasma and limiting the LIBS signal

Active Q Switching

Technology

- Acousto-optics Modules (AOM)
- Electro-Optical Devices (EOM)

Repetition Rate

- Lower – 10's of Hz

Pros

- *Precise Timing*
- *Simplified Electronics*
- *Higher Energies for better for surface preparation – removing oxidation like rust, oils & debris*

Cons

- *Higher Cost*
- *Allows for Carbon detection*
- *Increase size and complexity*
- *Can take longer*

What is M2 and why does it matter

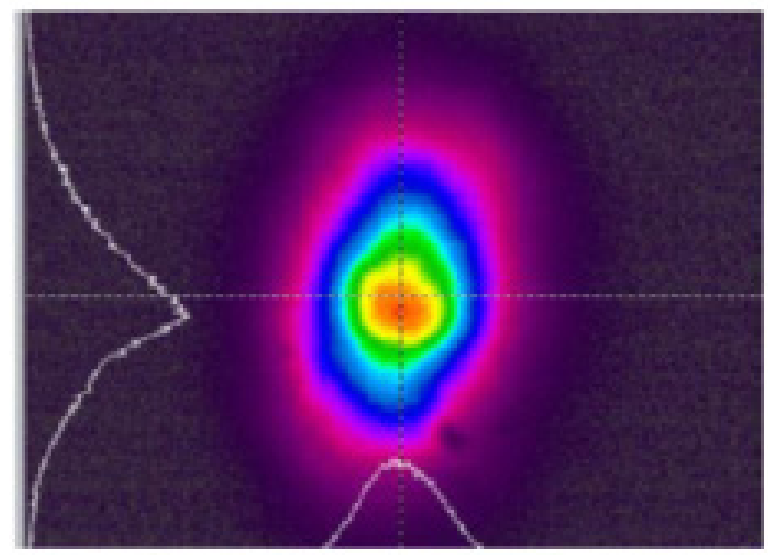
M2 is a measure of the focusability of the beam. It is the ratio of the beam quality compared to a diffraction limited beam

$$M^2 = \frac{\pi \cdot BPP}{4 \cdot \lambda}$$

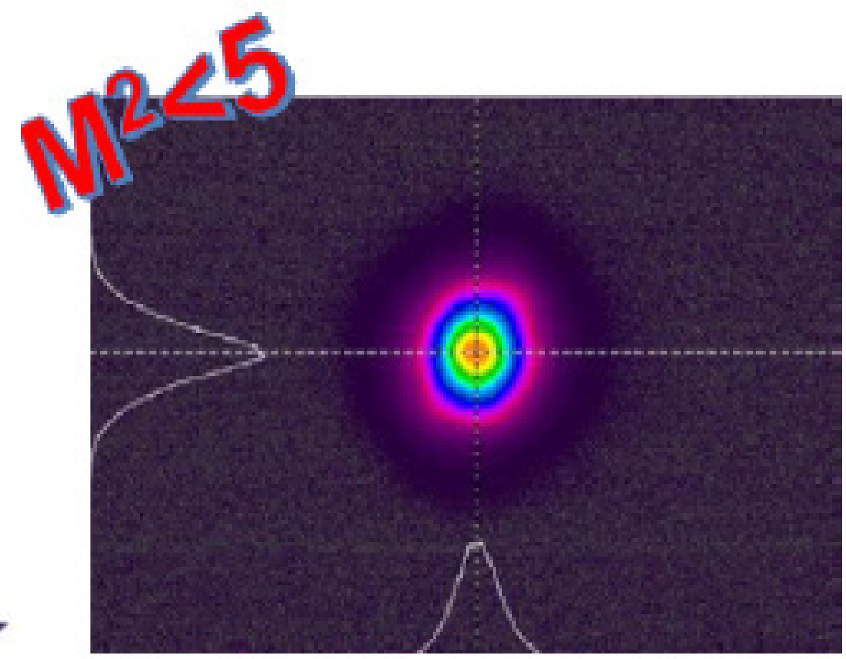
$BPP = \varphi \cdot \theta$
Beam Parameter Product

φ:= Beam Diameter

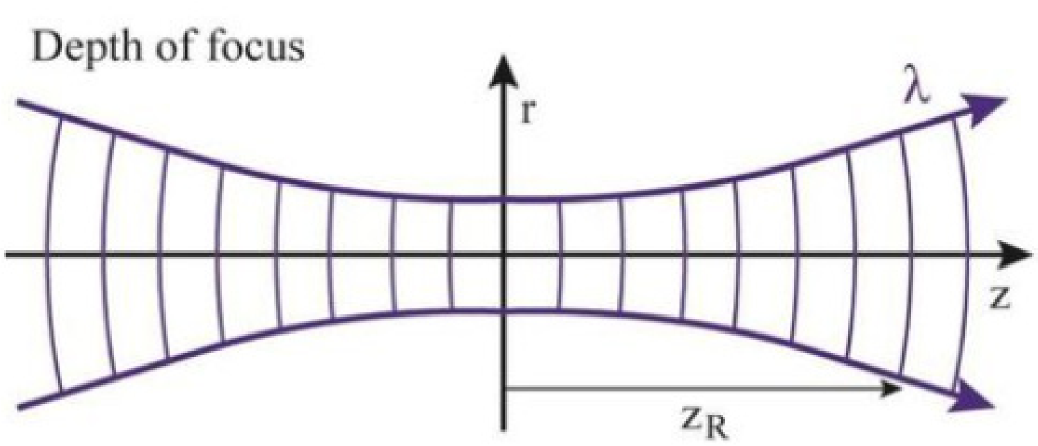
θ:= Divergence (Full Angle)



Falcon Near Field



Falcon Far Field



Laser Considerations

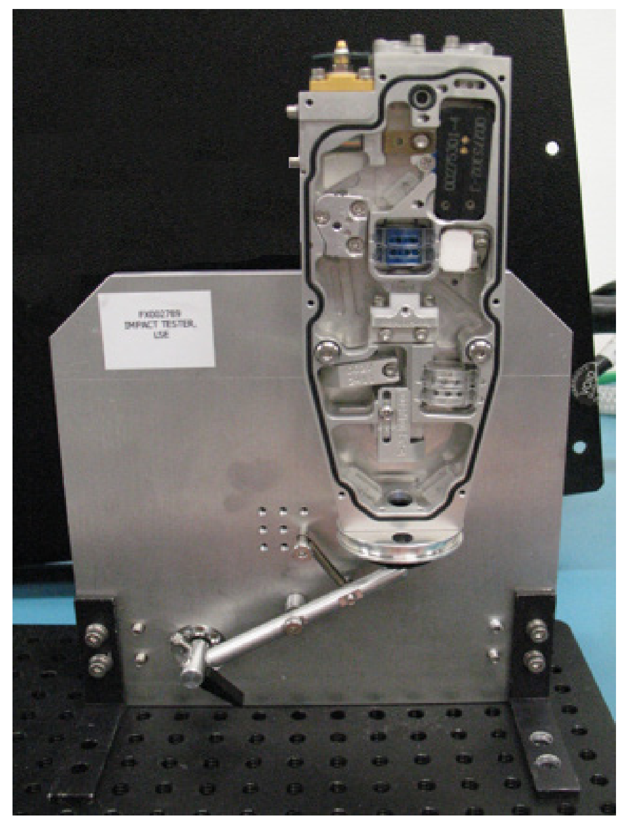
Wavelengths and Pulse Widths

1064nm Near-infrared

- Readily Available
- Higher energy photons for Surface Prep and Carbon LIBS
- Nano-second pulses produces good LIBS signal for most metals

1.5μm Short-infrared

- Wavelength is "eyesafe"
- Lower energy photons
- Nano-second pulses produces good LIBS signal for most metals

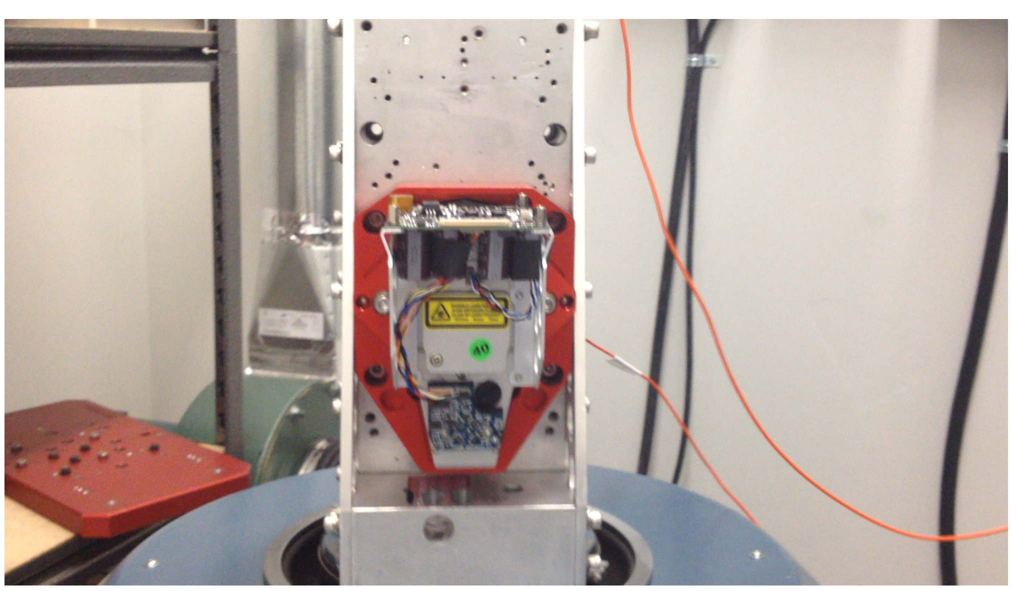


Falcon Optical Bed Assembly on Impact Test Fixture: Drop resistant 1 meter

Vibration Testing

Testing per MIL-STD-810H
Electro-dynamic Shaker
Drop Tests
Lumibird provides In-house Shock and Vibration Testing

- Random Vibe 60 minutes (7 grms)
- Shock Test (15 g peak)

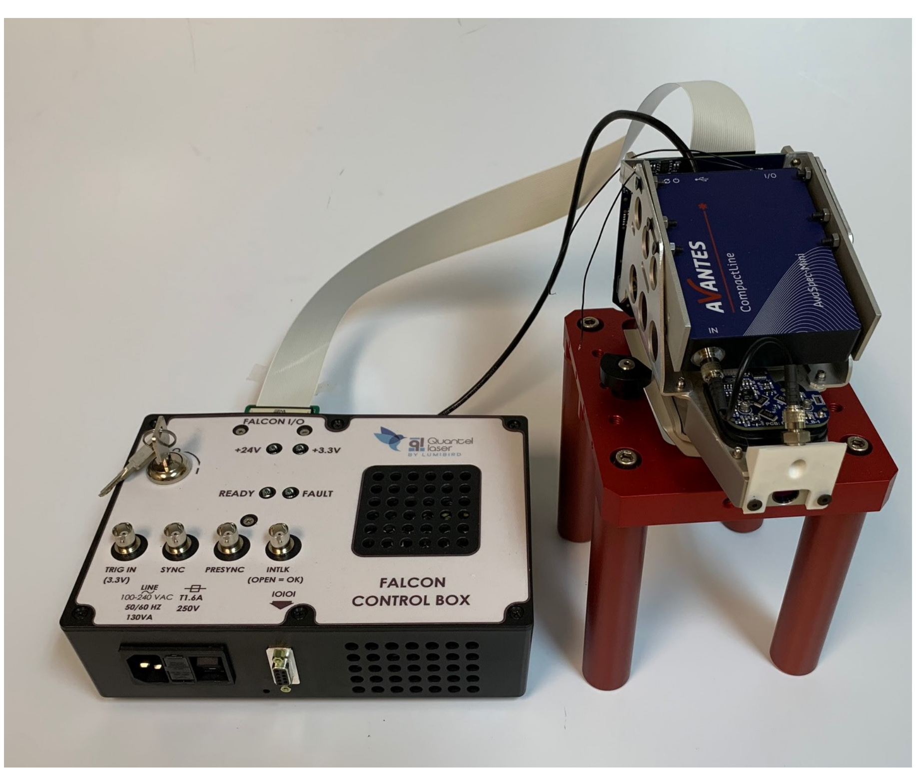


Temperature Stabilization

- Optical Cavity design needs to include temperature stabilization from the beginning
- Needs to address warmup times
- Are there thermal shuts offs at high temp?
- Typical specs include <5% variation over 5 to 35°C
- Storage Temps -40 to 80°C
- Feedback loops for temperature stability of the output energy

Lumibird uses a proprietary temperature feedback loop to ensure a stable output energy over the entire operating temperature

Demonstration Kit

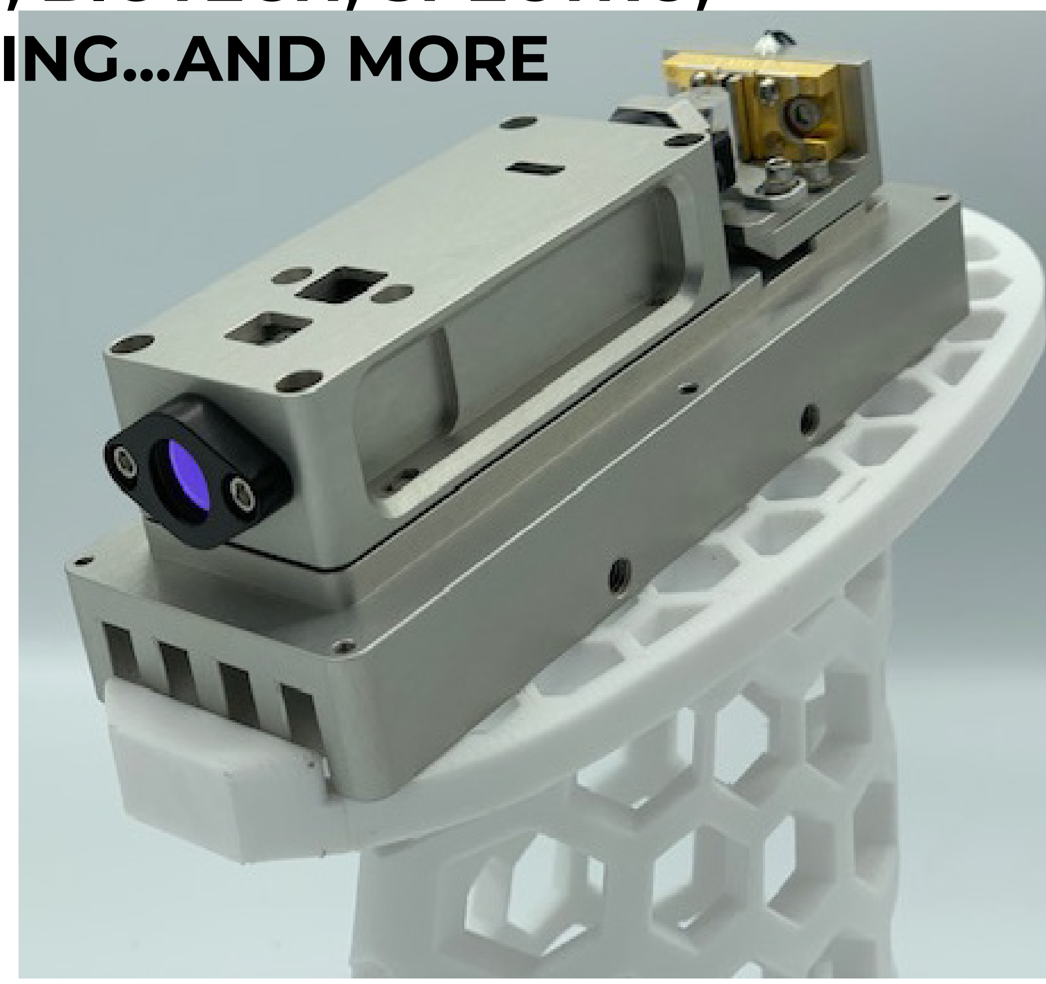


Features

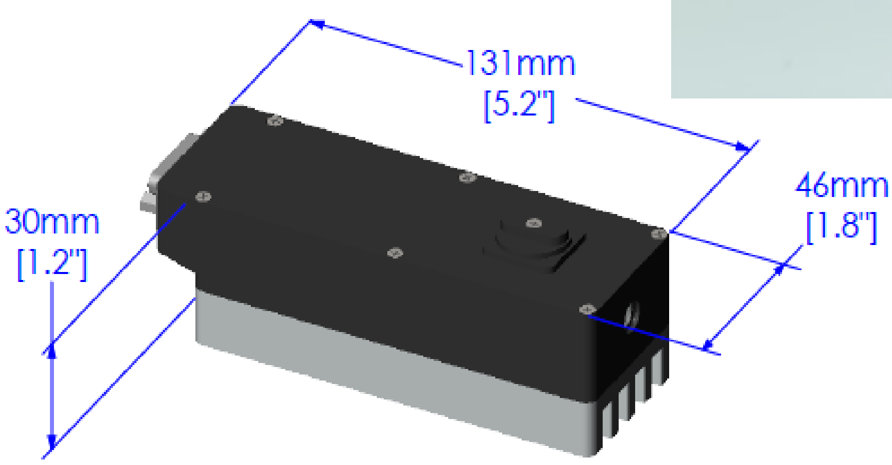
- Mounting Bracket
- Control Boards
- Heat Sync
- Control Box
- Optical Table Mounting
- ESD Protection Pack

NEW PRODUCT COMING SOON

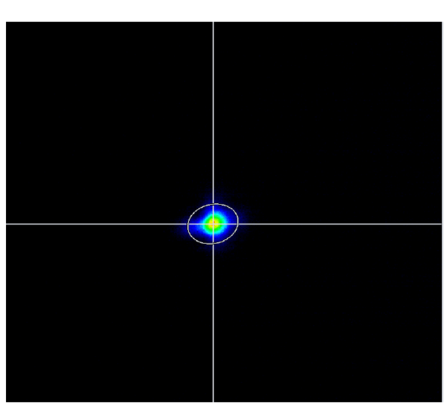
Designed for LIBS, BIOTECH, SPECTRO, RANGING, & SENSING...AND MORE



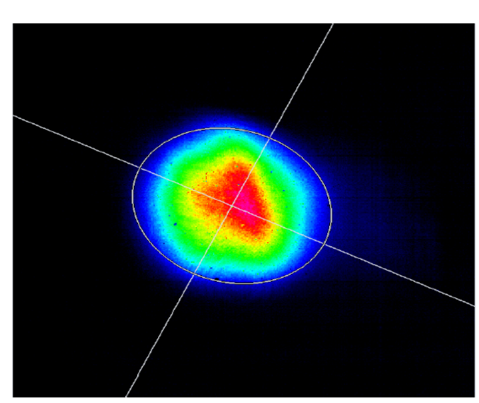
- DPSS Nd:YAG
- 1064nm
- Up to 50Hz
- 10mJ
- 6-13ns
- M2<2
- <1.5mrad Low Divergence Linear Resonator
- Passive Q-Switch
- 2.5mm Beam Diameter



Far Field



Near Field



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Summary of Critical Factors for Portable LIBS design.

Miniaturization

- Both the laser and spectrometer must be compact and light weight

Power Consumption

- High efficiency lasers and on-board electronics reduce battery requirements

Environmental Ruggedization

- Each component must be tested for sock, vibration, and temperature

Spectral Range and Resolution

- Portable LIBS often requires both large spectral range necessitating multichannel spectrometers

Precise Triggering Electronics

- Time gating is essential to measure atomic emissions, requiring fast low jitter trigger electronics

Vacuum UV – Ar Purge

- To measure carbon and other light elements with spectral bands below 190 nm

Presenters

AVANTES

MEMBER OF THE NYNOMIC GROUP

Avantes, part of the Nynomic Photonics group, is the leading innovator in the development of fiber-optic spectroscopy instruments and systems. Headquartered in the Netherlands, Avantes celebrated 26 years in business in 2020. Globally we have more than 36,000 spectrometers installed and growing. Avantes North America is based in Louisville, CO, with Asian headquarters in Beijing, China. We also work with over 40 distributors worldwide. Additionally, we are an ISO 9001: 2015 certified manufacturer of spectrometers.

Our mission is to provide state of the art, innovative measuring equipment that helps mankind live longer, healthier, and saving the planet for generations to come.

NYNOMIC
THE PHOTONICS GROUP

Lemnatec
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LAYTEC
Knowledge is key

tec5 USA
Technology for Spectroscopy

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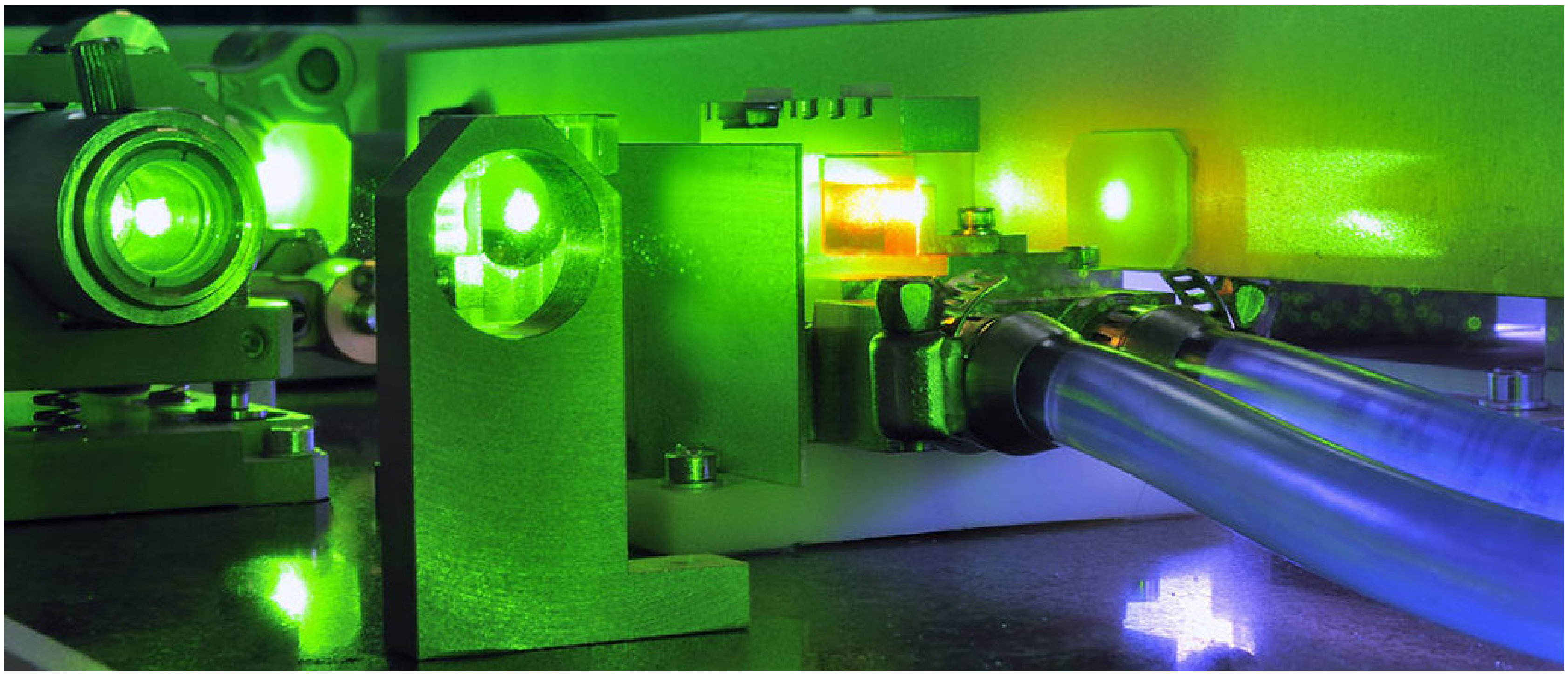
SPECTRAL ENGINES

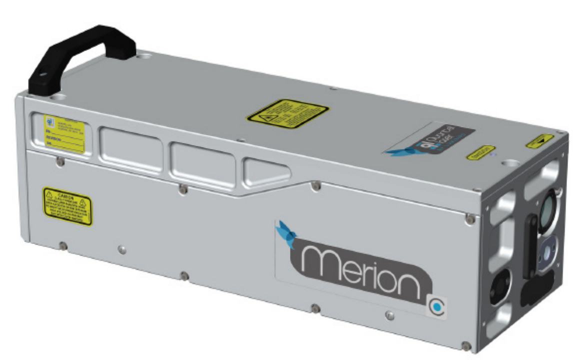
m·u·t
efficient photonic solutions

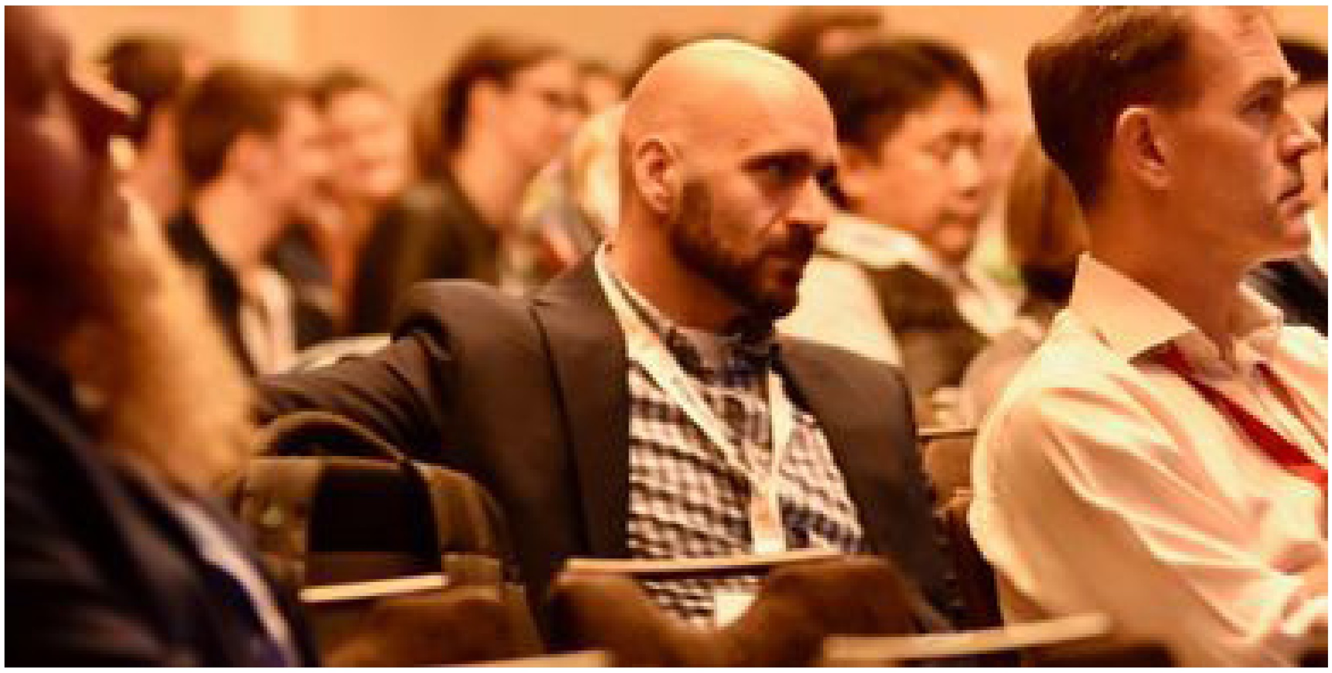


LUMIBIRD
MORE THAN LASERS

Lumibird Laser division, incorporating Keopsys, Quantel and SensUp, offers state-of-the-art lasers for industrial, defense, scientific and medical applications. With 50 years of experience in 3 key technologies: fiber lasers, solid-state lasers and laser diodes, Lumibird boasts 800 employees worldwide, with production in France, Slovenia, and the United States. Lumibird is your international partner and foremost source for LIBS laser systems from mJ to Joules

Our mission is popularizing lasers. We aim to open up widespread access to lasers through innovation and designing increasingly high-performance lasers that are aligned with end users' constraints, particularly in terms of miniaturization.





RVC Photonics

Robert V. Chimenti

Director & Principal Consultant, RVC Photonics

July 2018 - Present

Freelance Technical Writing, Professional/Technical Training, Applications Development, Instrument Evaluation, Optical DesignServices, and Technical Marketing Consulting

Instructor/Adjunct Professor, Rowan University College of Science and Mathematics

Spring 2013 - Present

M.S. in Electro-Optics

Working in Optics Since 2002

Lasers since 2005

Laser Spectroscopy since 2009

Thank you

If you would like to learn more about developing a handheld LIBS device, Avantes has the expertise to guide you to solutions for your measuring challenges.
Inquiries are welcome.

Follow up Questions can be directed to the following:

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