A Fundamental Study of Laser-Induced Breakdown Spectroscopy Using Fiber Optics for Remote Measurements of Trace Metals

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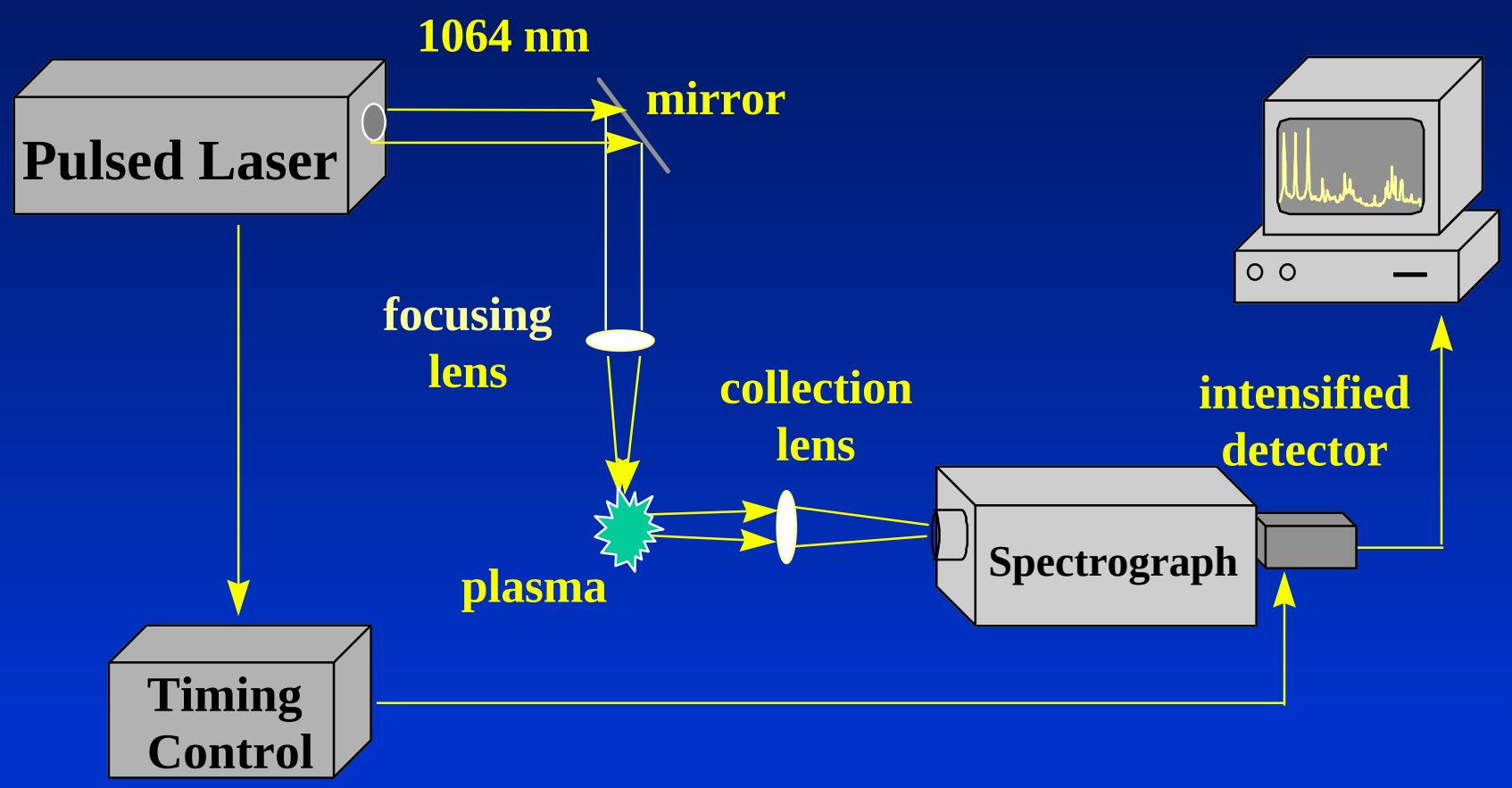
http://www.chem.sc.edu/goode/DOE_EMSP/IP98.htm

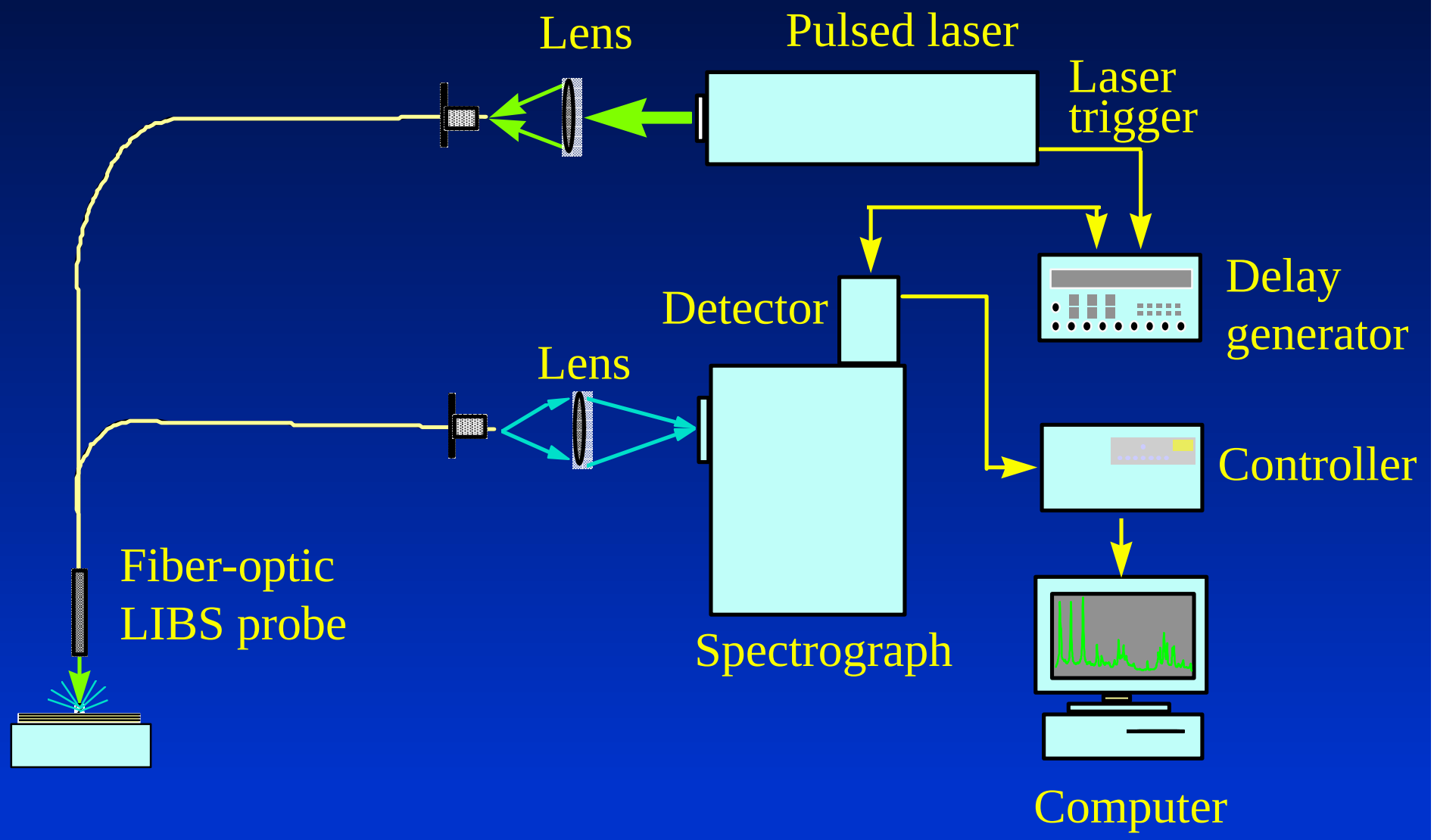
Laser-Induced Breakdown Spectroscopy

- Use laser to vaporize sample
- Laser electric field high enough to cause breakdown
- Monitor emission
- Fiber optics afford capability for remote analysis

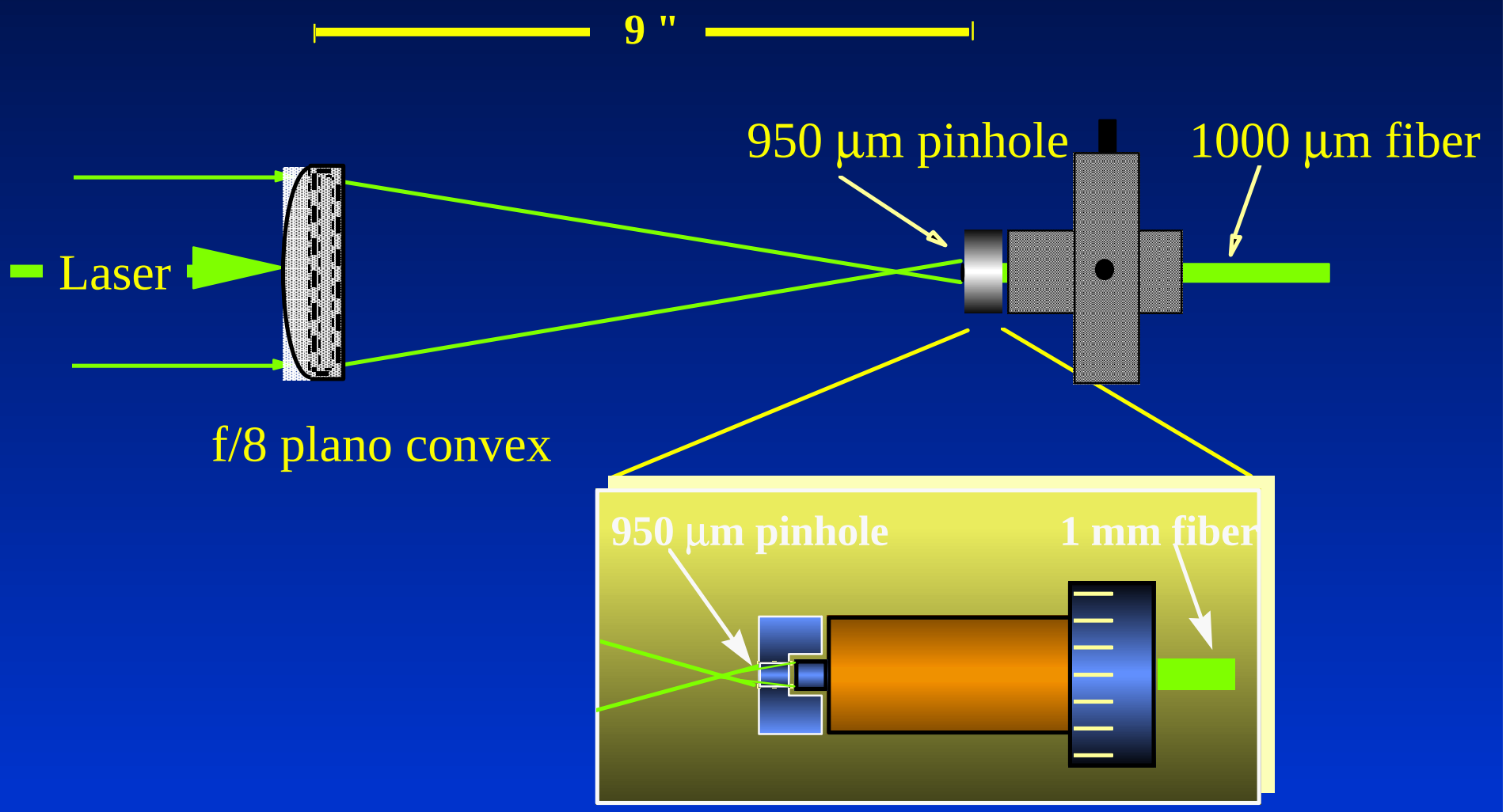
Time-Resolved LIBS Apparatus



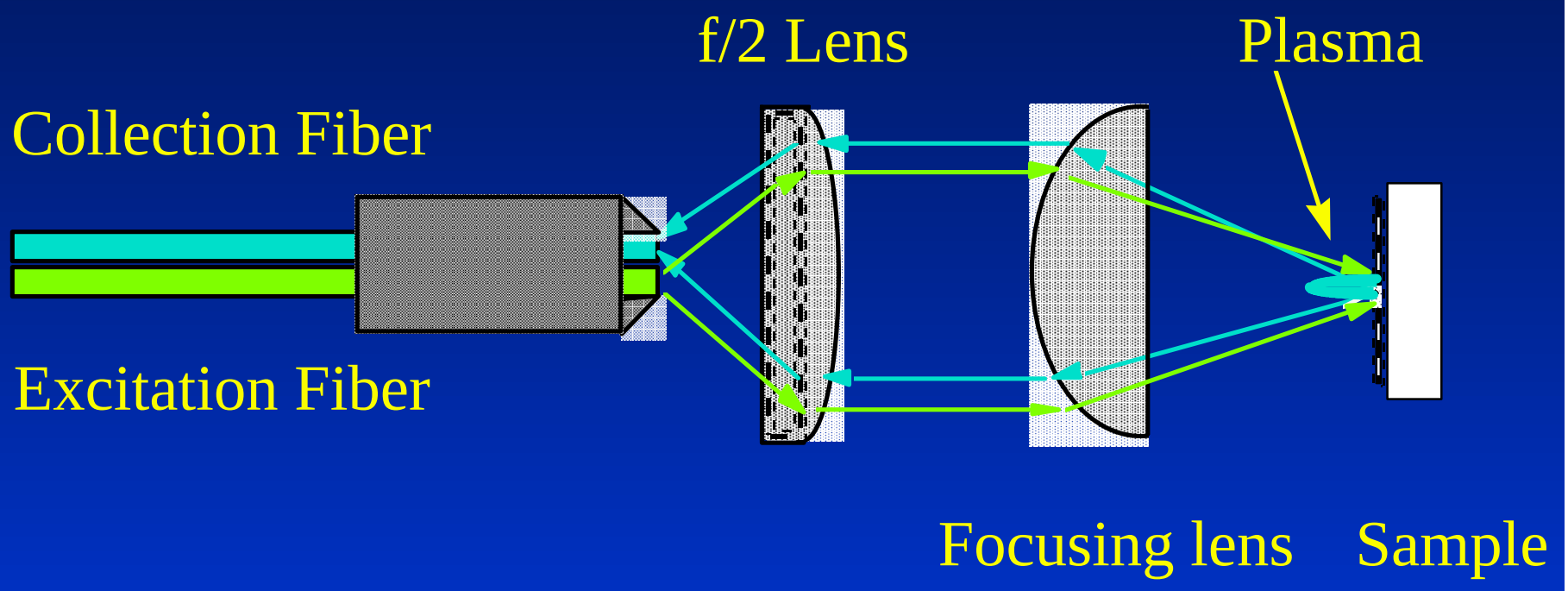
Fiber-Optic LIBS System Configuration



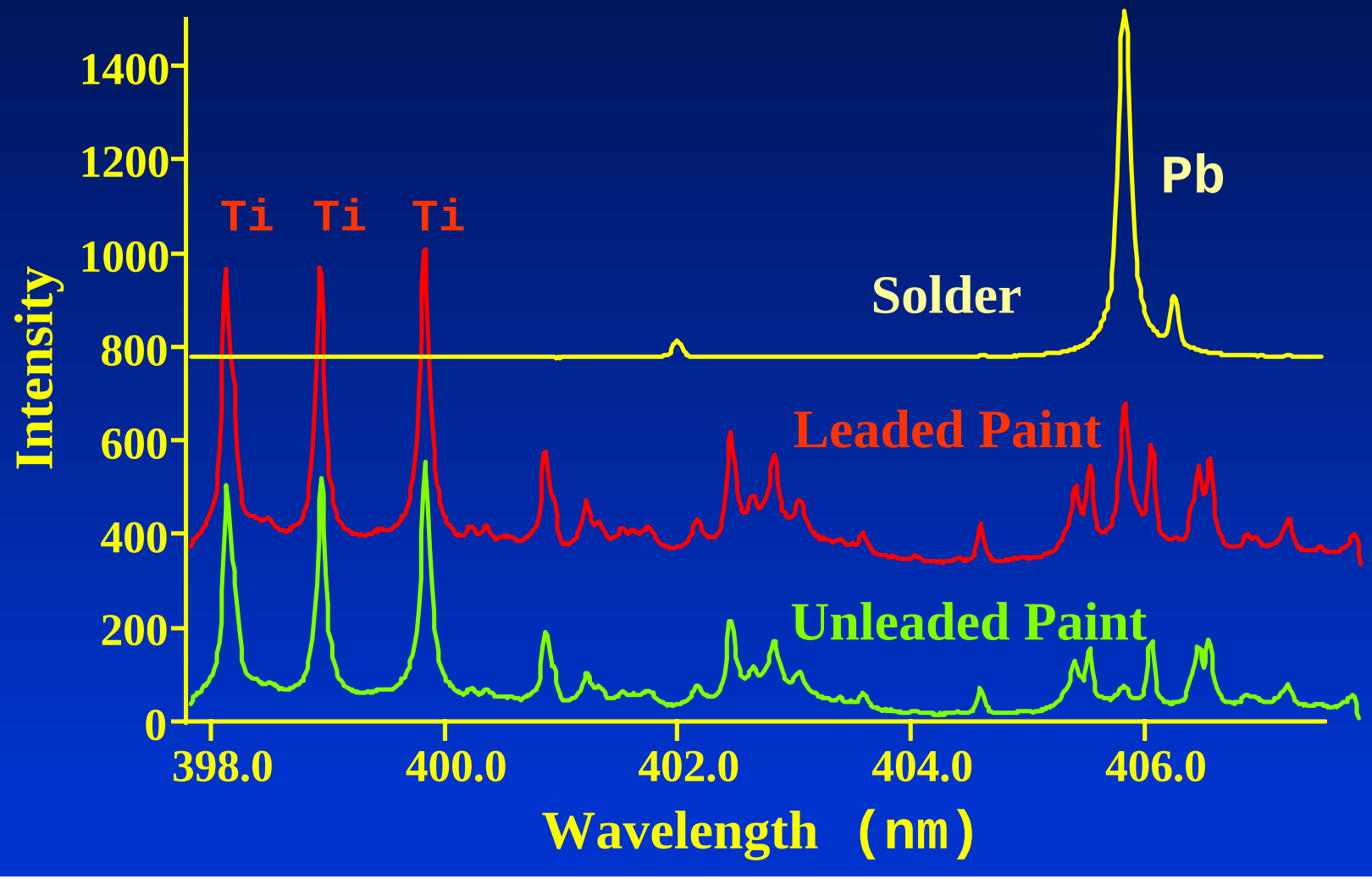
Laser to Fiber Launch Design



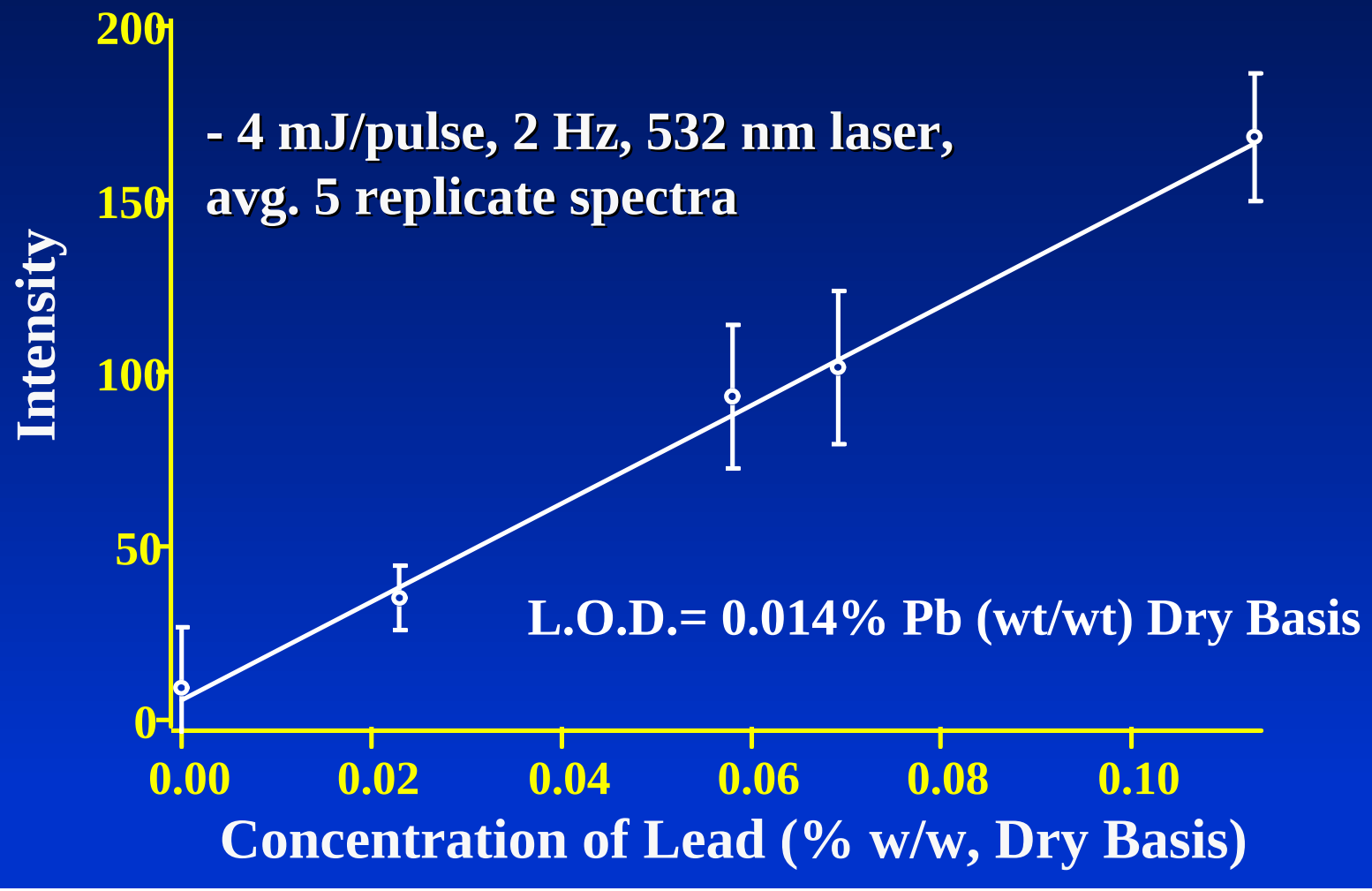
Fiber-Optic LIBS Probe Design



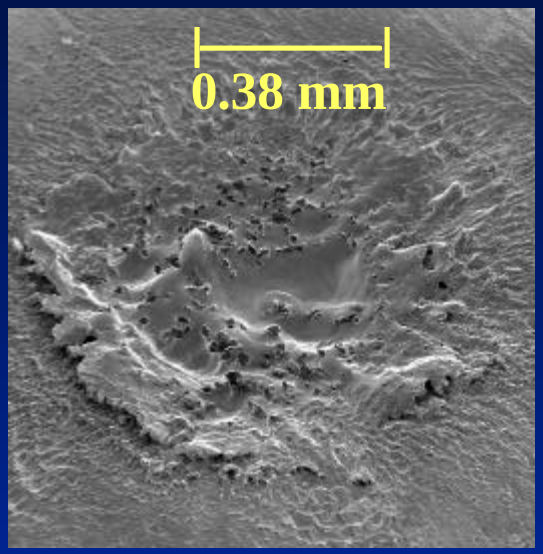
Lead in Paint Using Fiber-Optic LIBS Probe

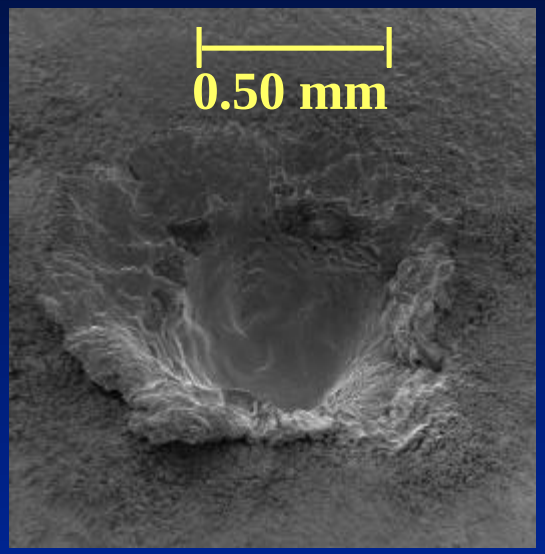


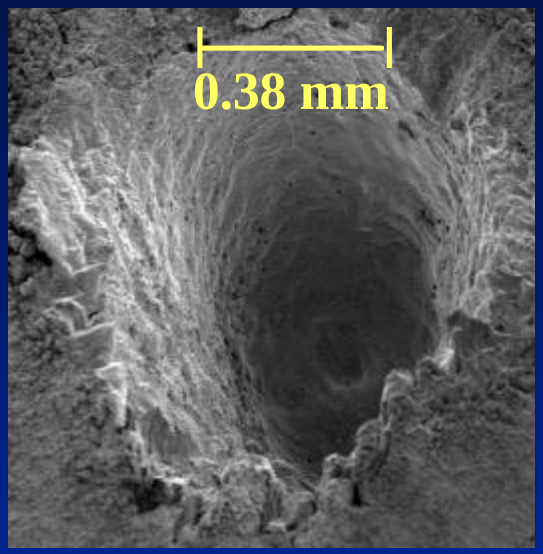
Leaded Paint Calibration Using Fiber-Optic Probe

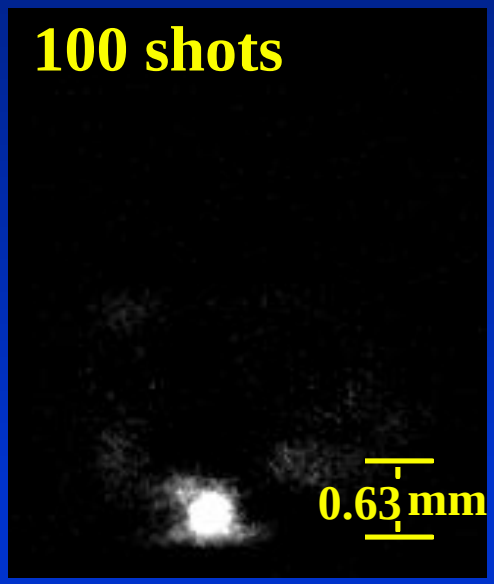


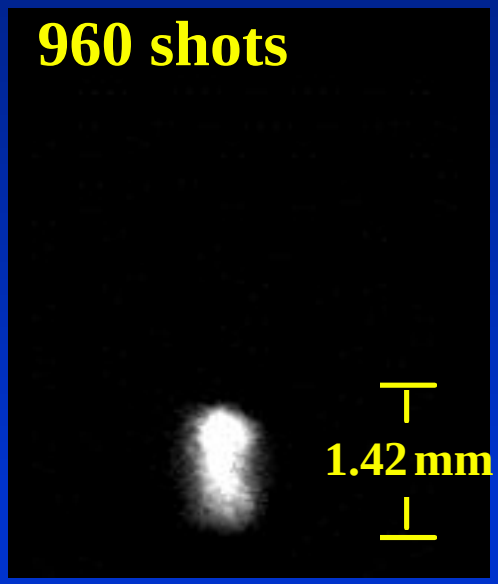
Lead Crater Depth and Plasma Height

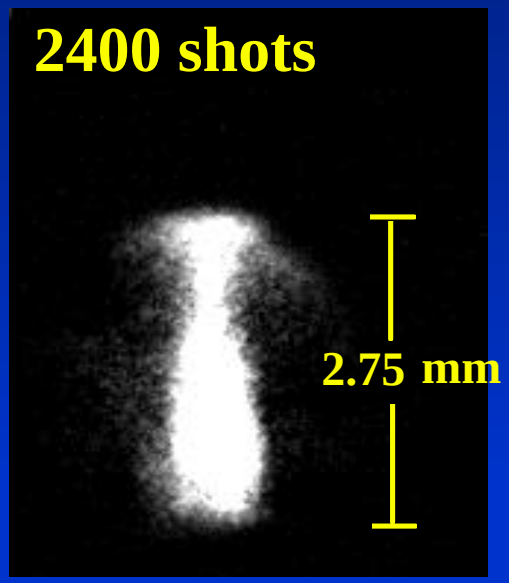


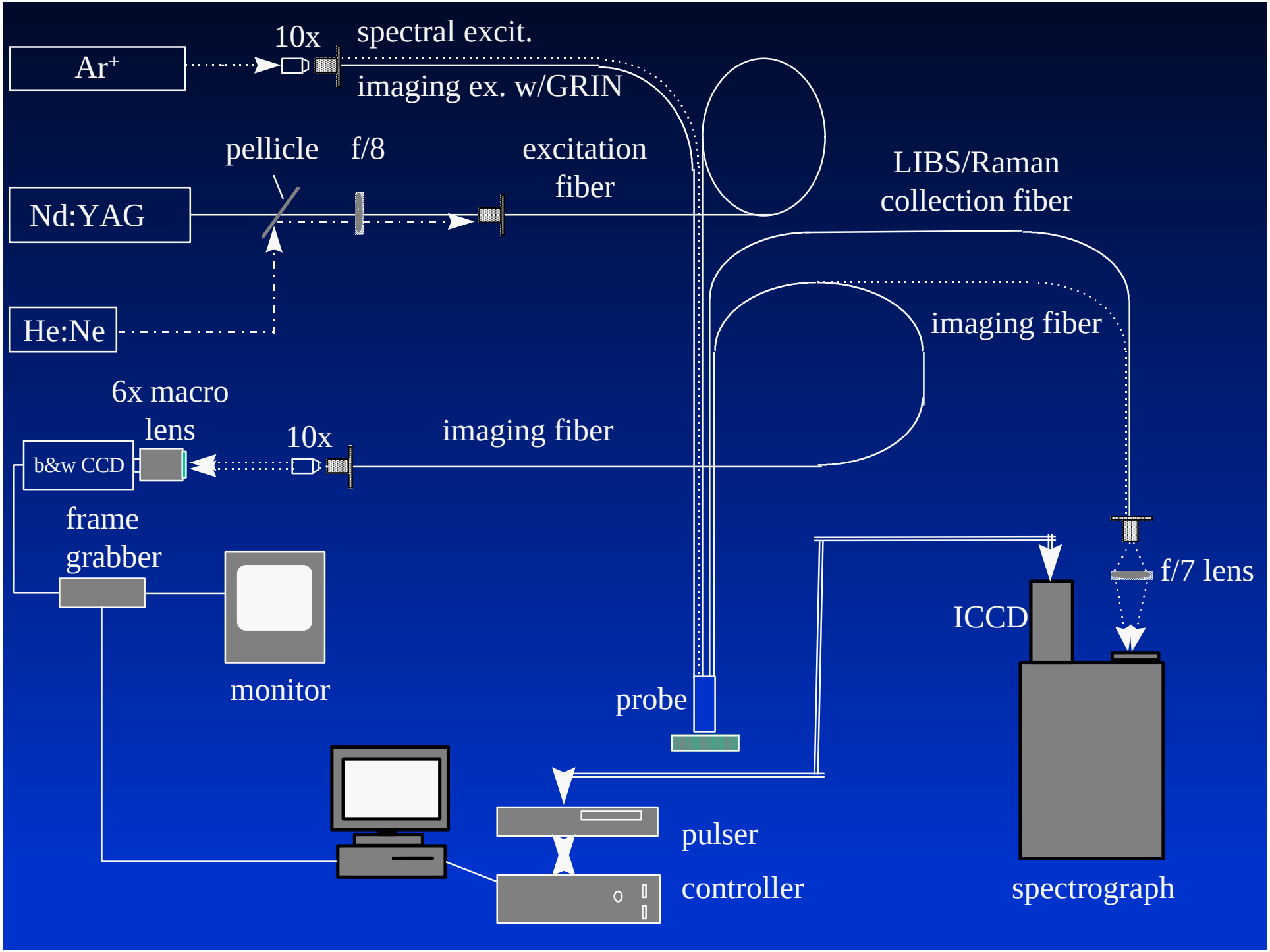


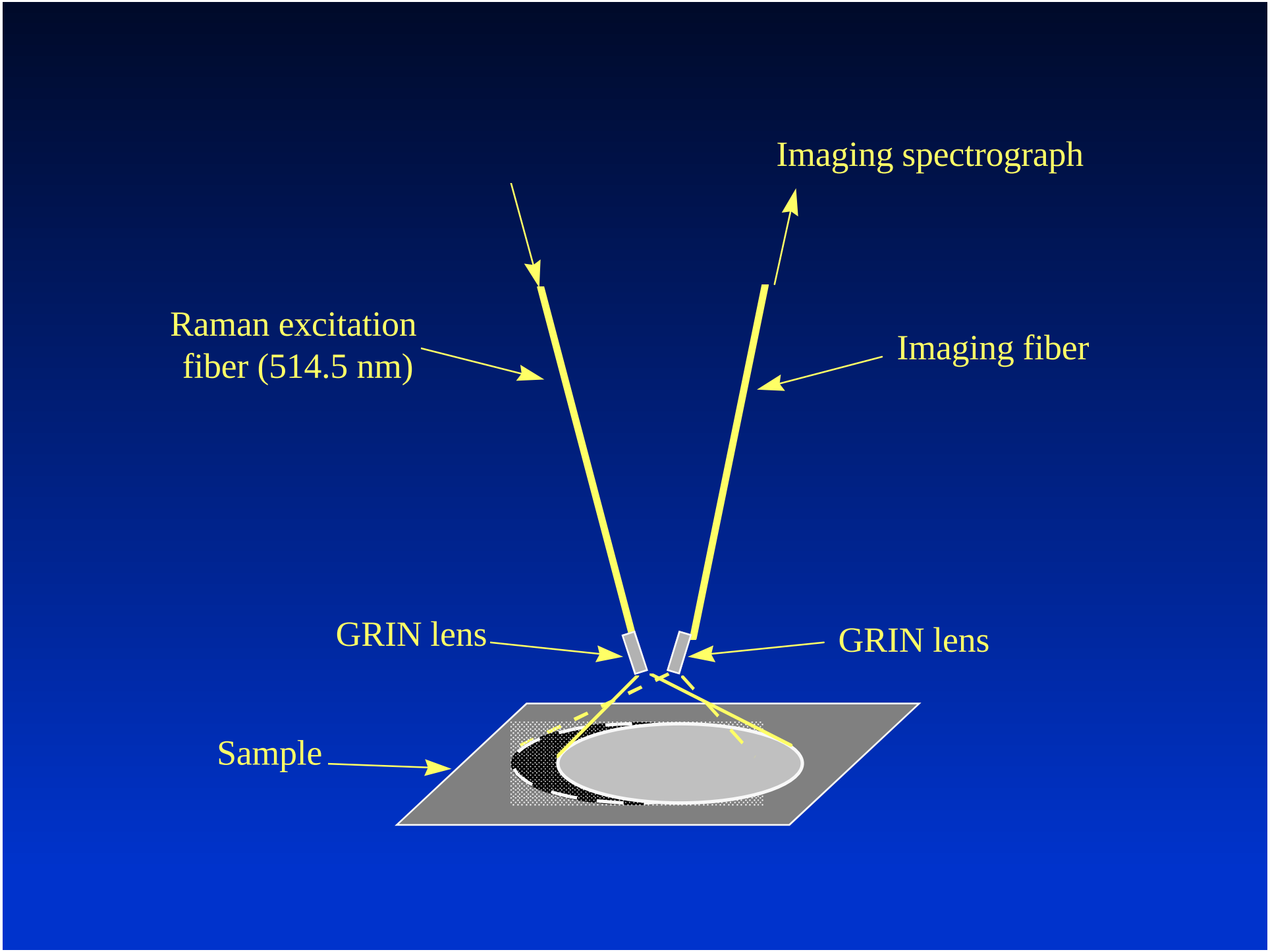


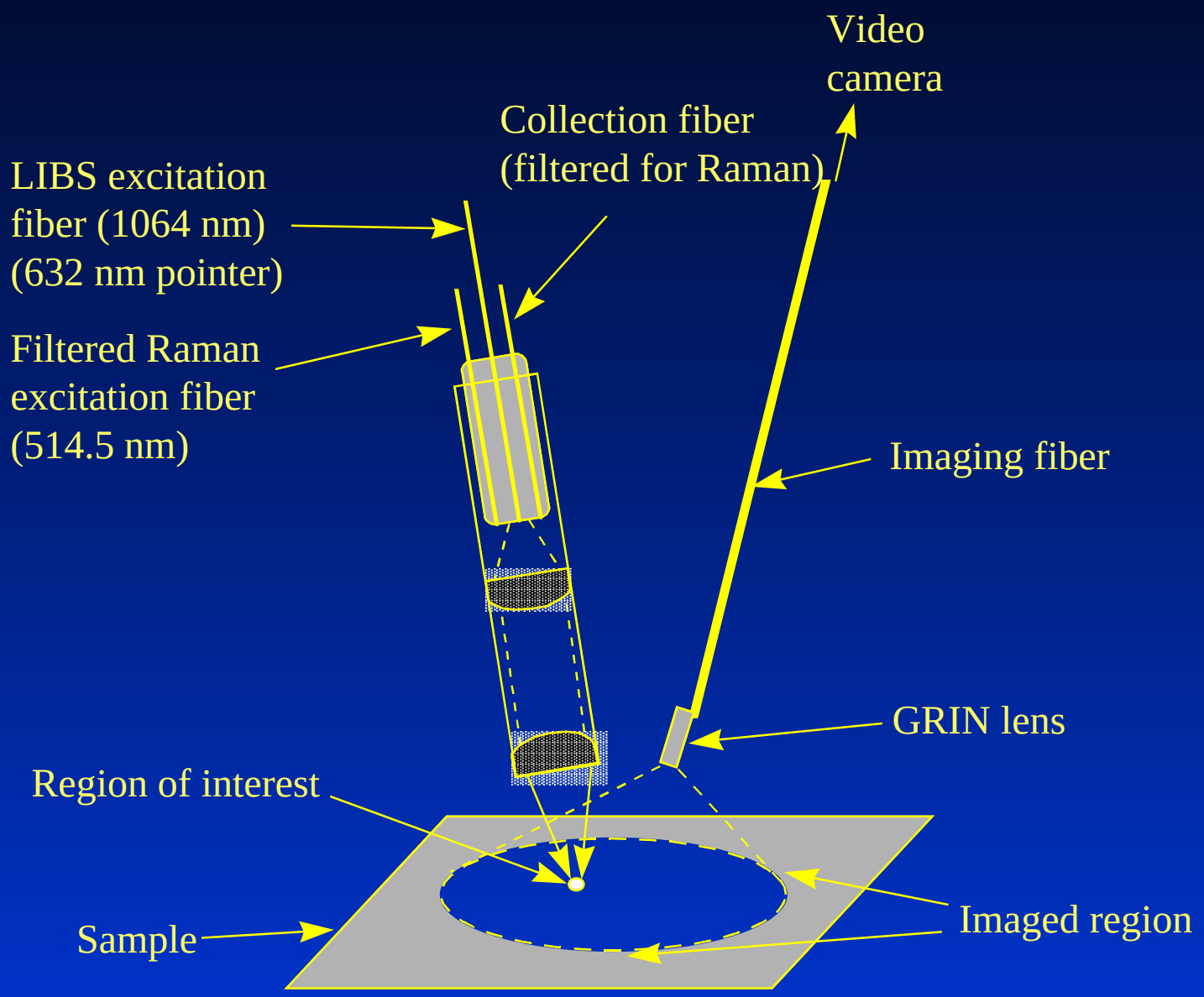


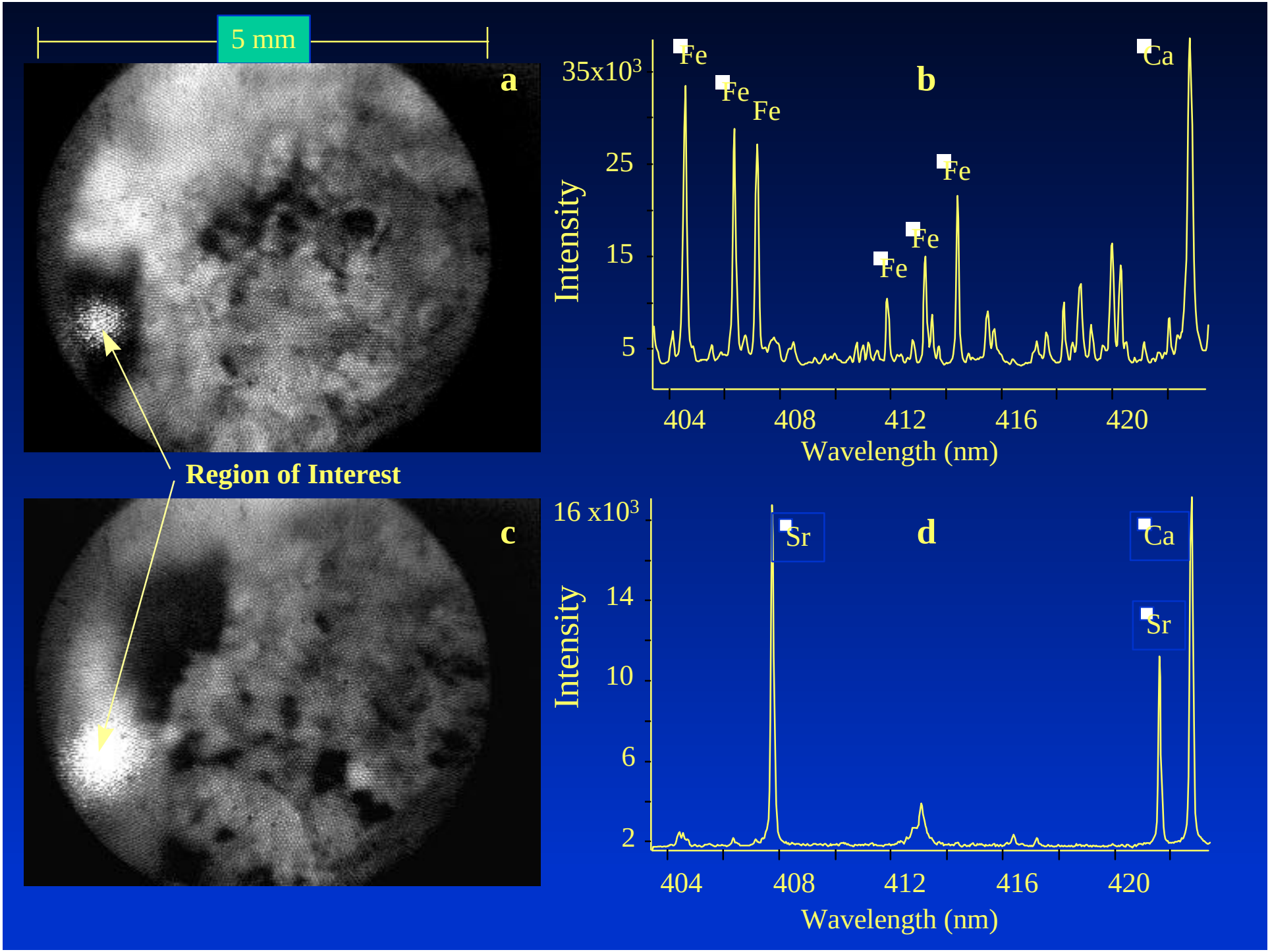




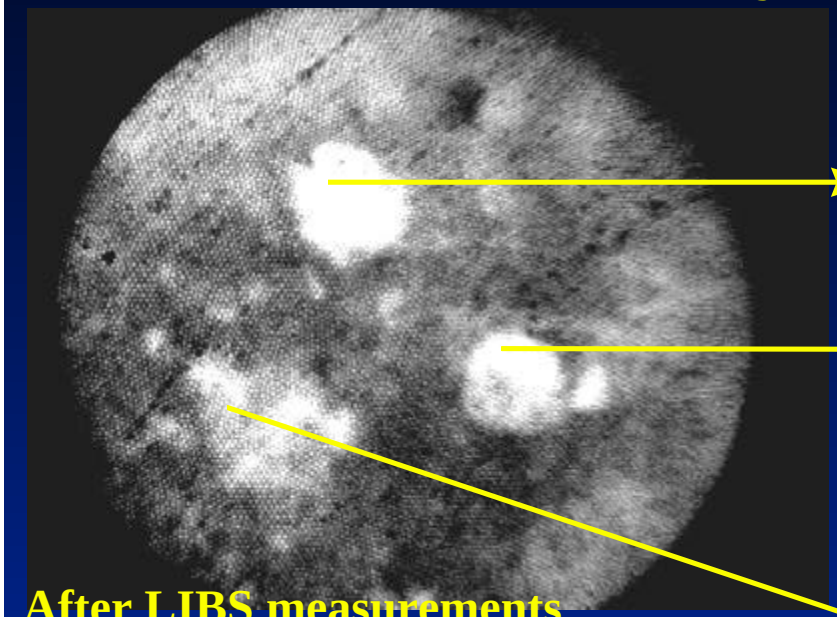

Ar+
10x
spectral excit.
imaging ex. w/GRIN
pellicle
f/8
excitation
fiber
Nd:YAG
LIBS/Raman
collection fiber
He:Ne
imaging fiber
6x macro
lens
10x
imaging fiber
b&w CCD
frame
grabber
f/7 lens
ICCD
monitor
probe
pulser
controller
spectrograph

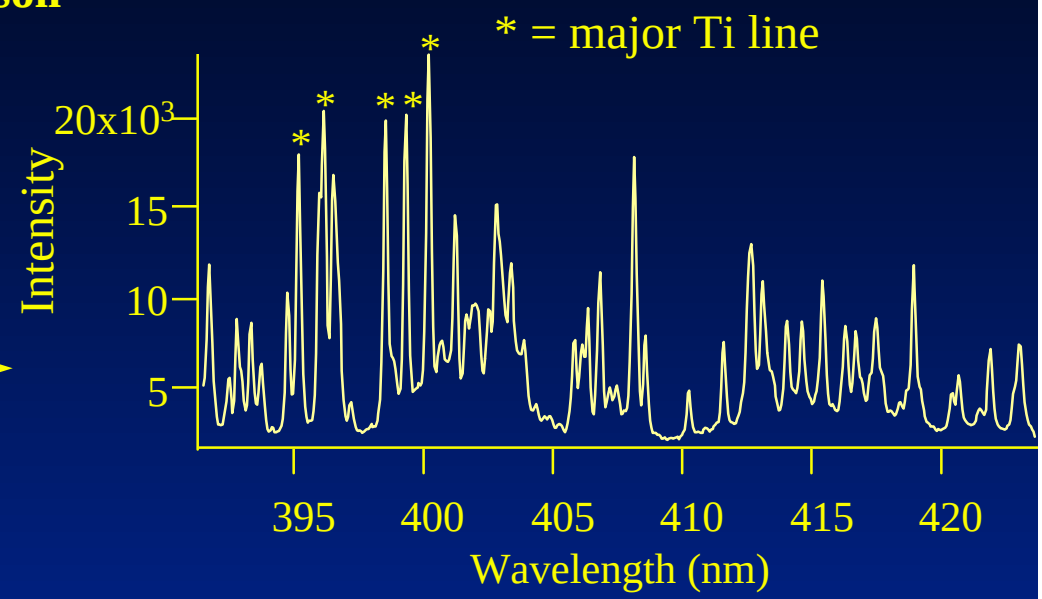

Imaging spectrograph
Raman excitation
fiber (514.5 nm)
Imaging fiber
GRIN lens
GRIN lens
Sample


Video
camera
Collection fiber
(filtered for Raman)
LIBS excitation
fiber (1064 nm)
(632 nm pointer)
Filtered Raman
excitation fiber
(514.5 nm)
Imaging fiber
GRIN lens
Region of interest
Sample
Imaged region

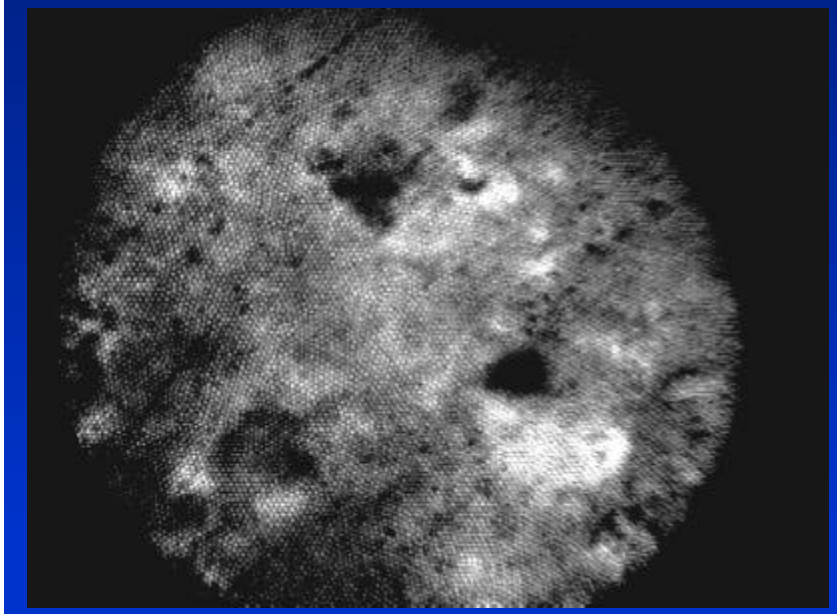

5 mm
a
b
c
d
Region of Interest
Fe
Fe
Fe
Fe
Fe
Fe
Ca
Sr
Sr
Ca
35x10³
25
15
5
16 x10³
14
10
6
2
Intensity
404
408
412
416
420
Wavelength (nm)

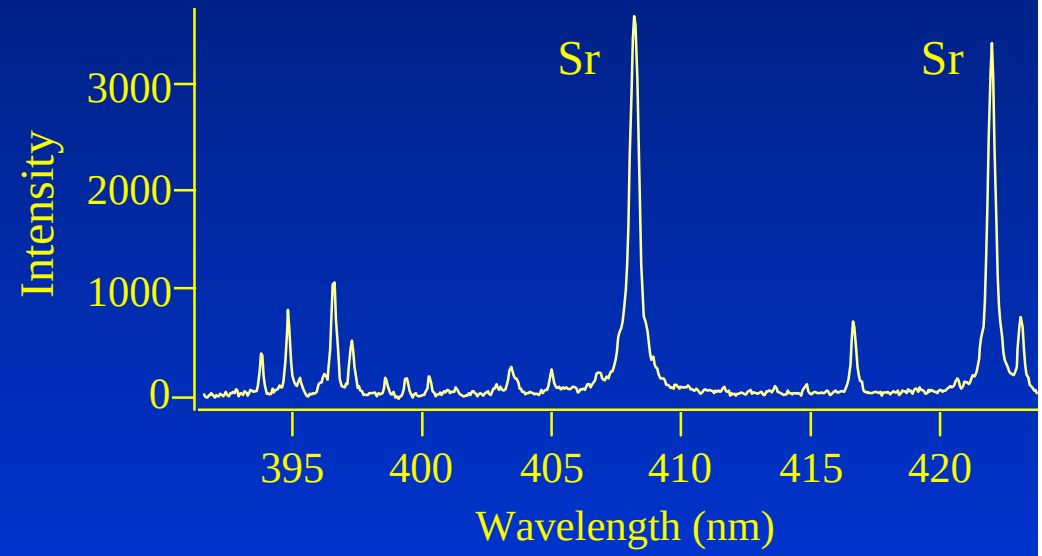
Darkfield image of TiO_2 and $Sr(NO_3)_2$ on soil





After LIBS measurements





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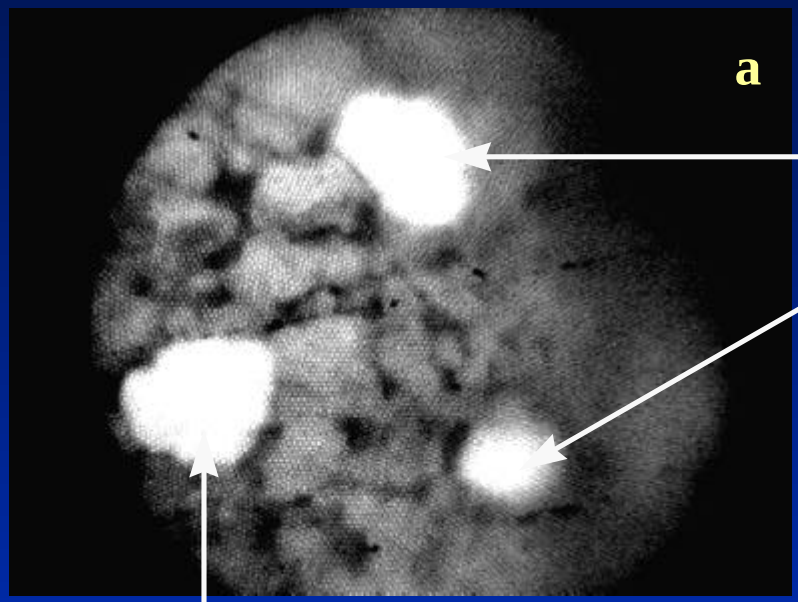
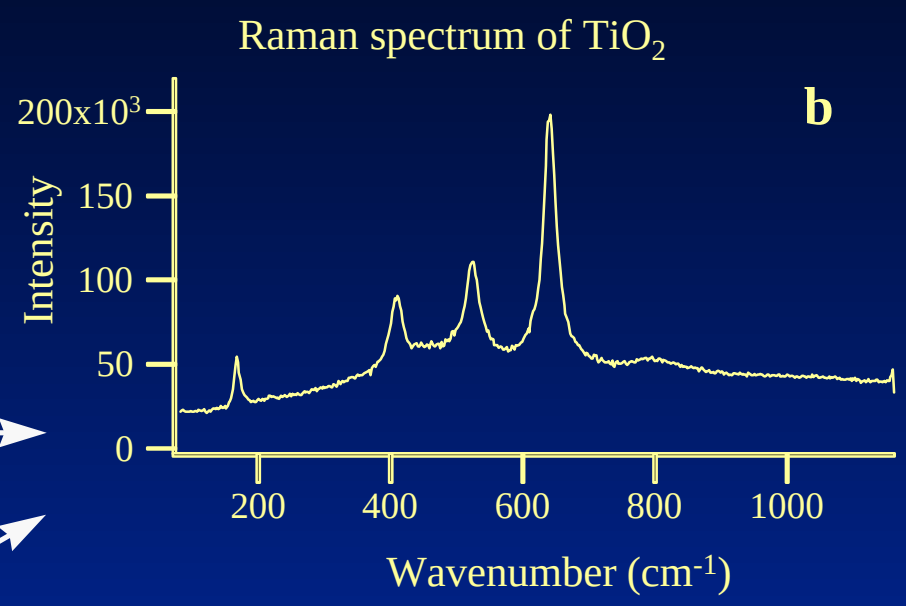
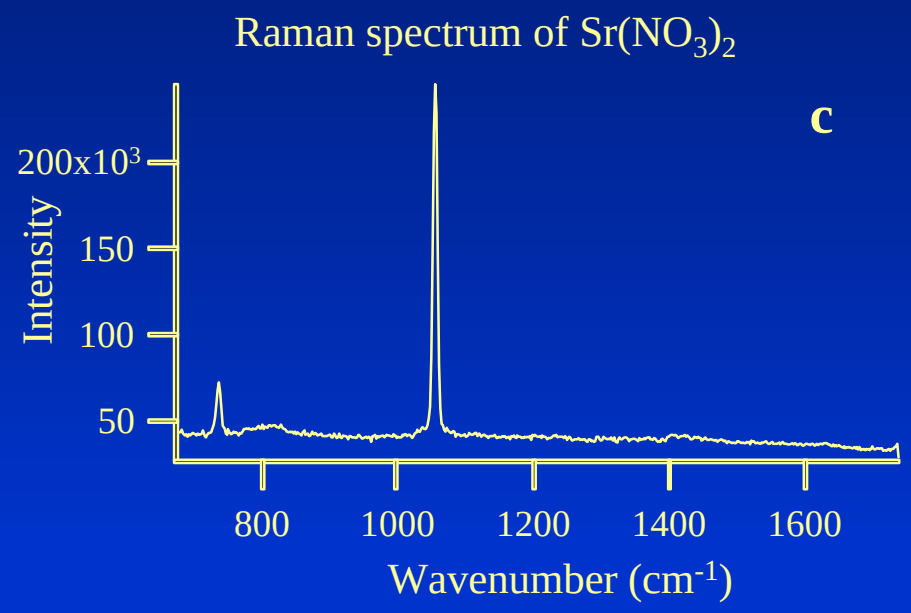
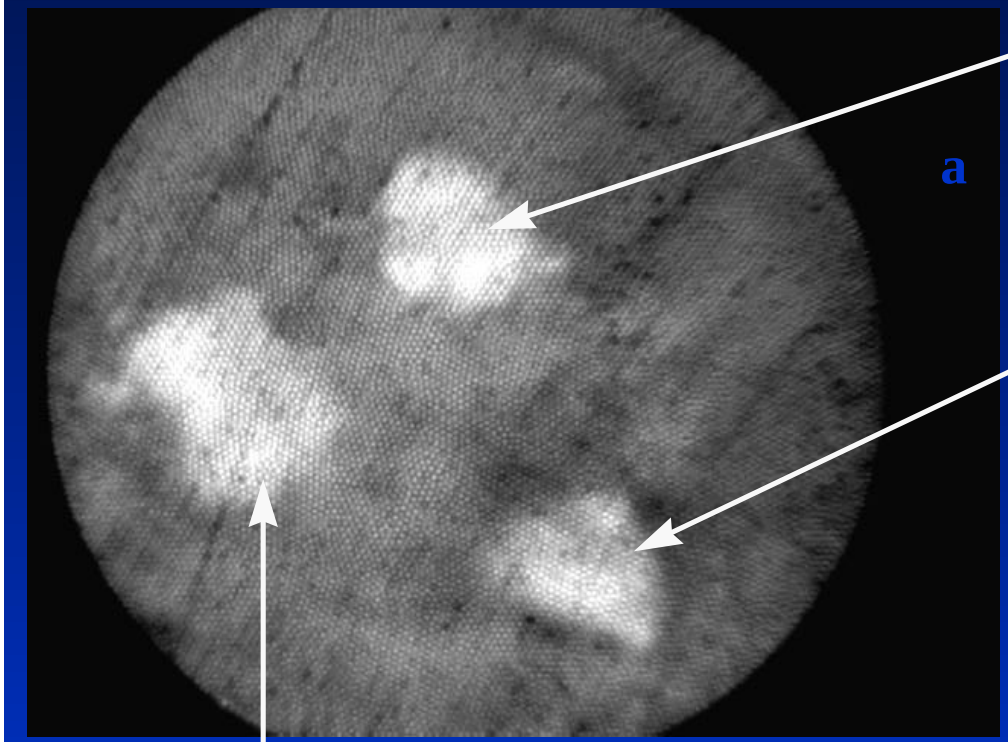


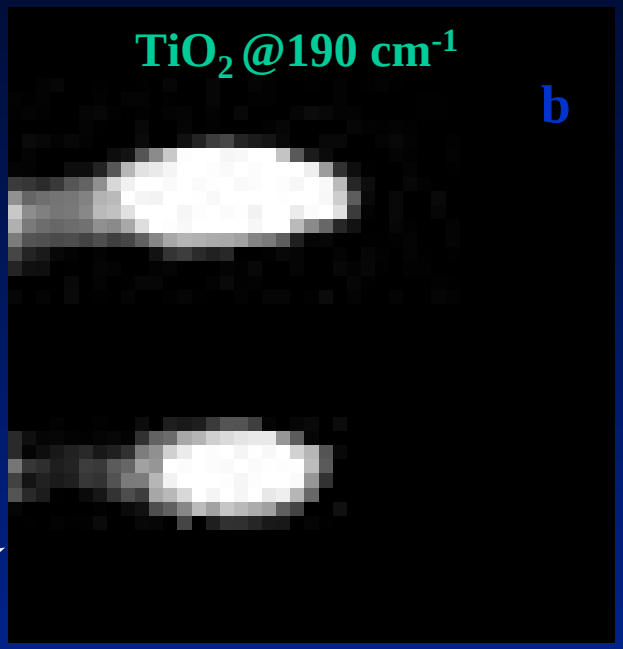


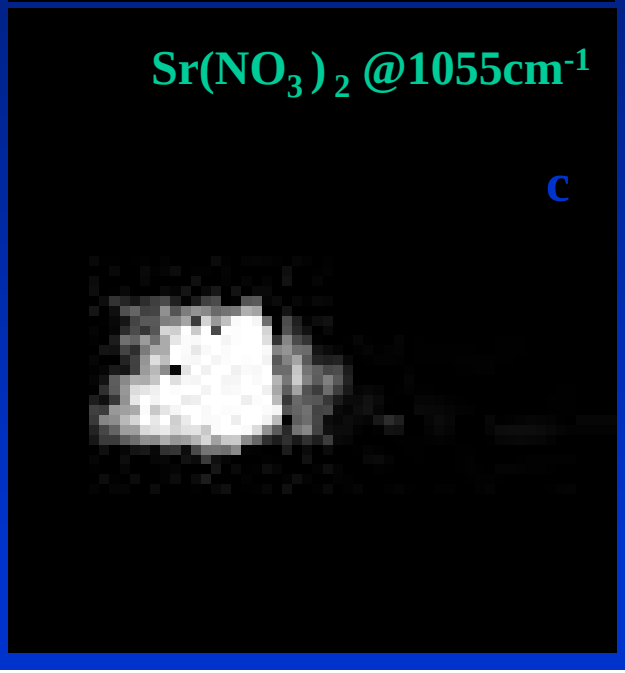


Raman Images

Darkfield image of TiO_2 and $Sr(NO_3)_2$ on soil





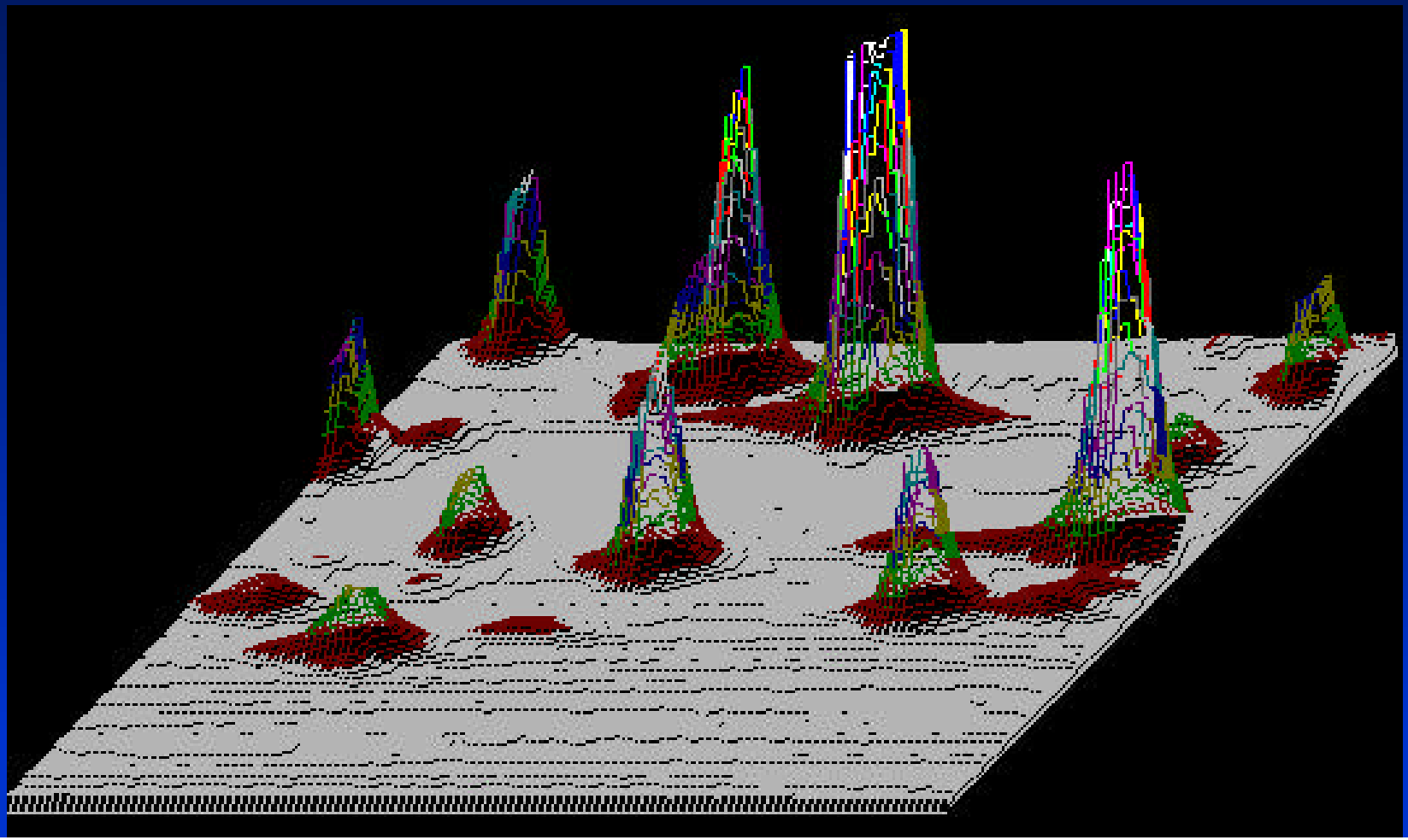


Using High Wavelength Resolution

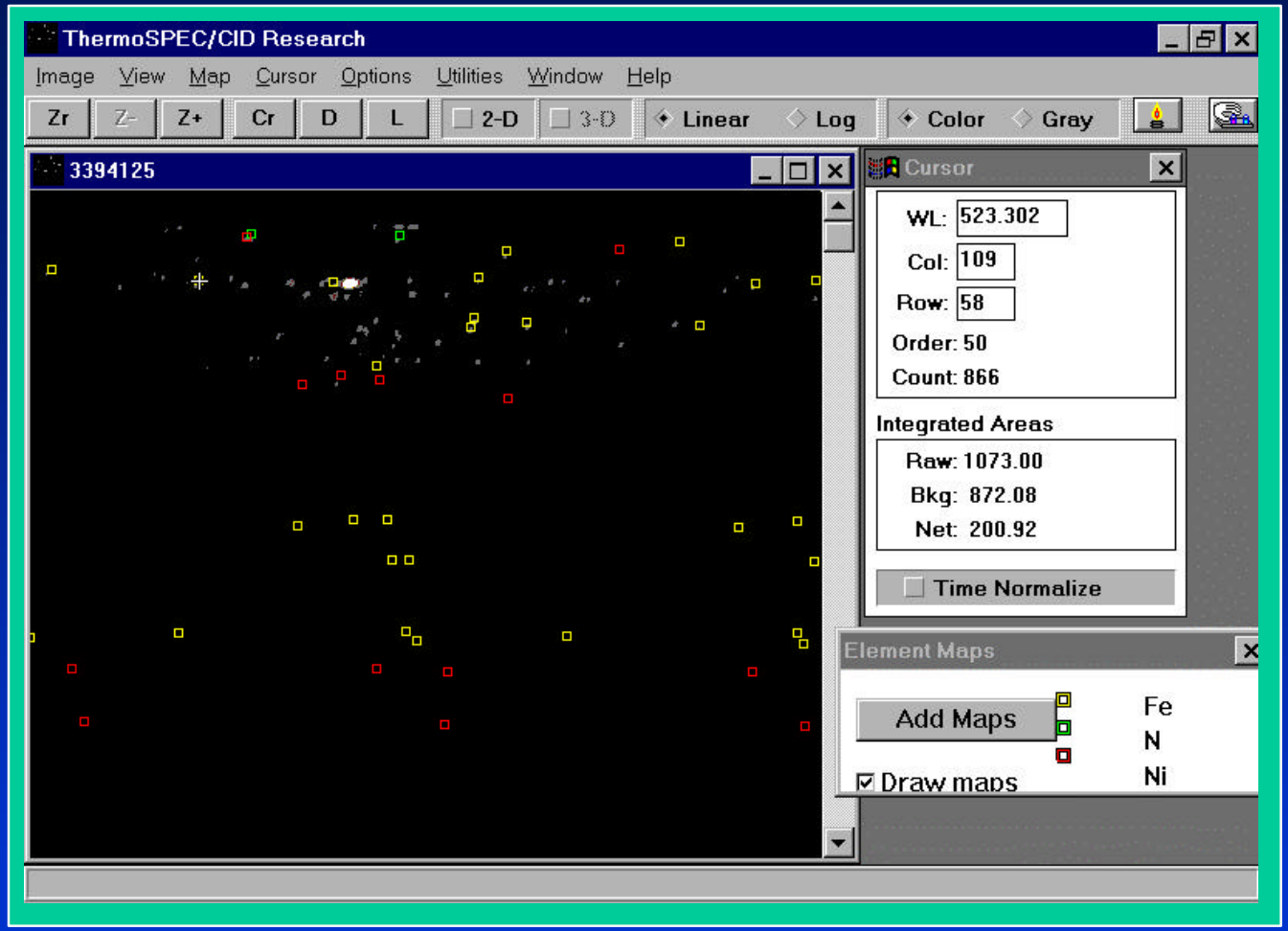
If the major source of noise is the continuum background

- Eliminate the background by time resolution
- Use wavelength resolution to distinguish the atomic lines from the continuum background

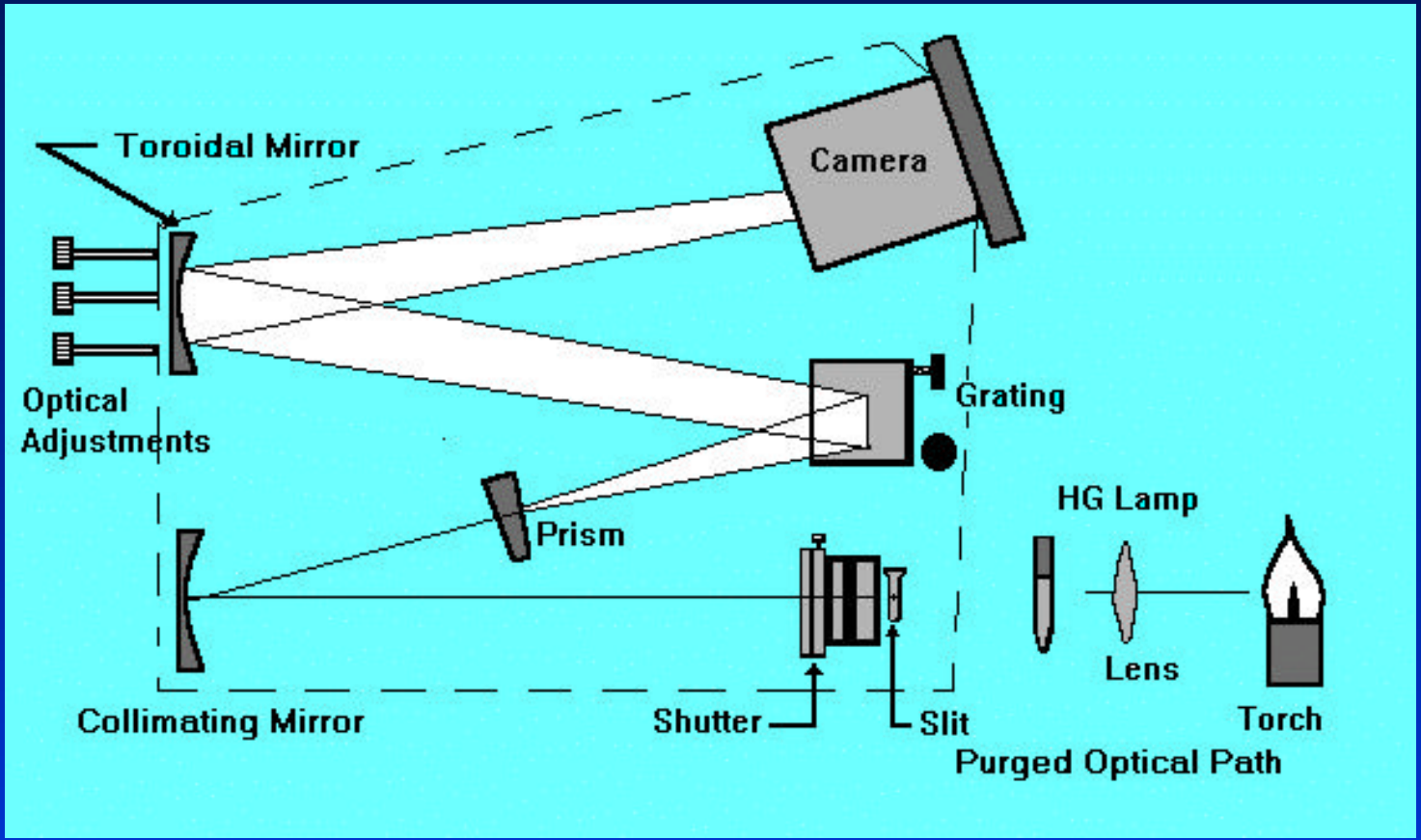
Information dispersed in two dimensions



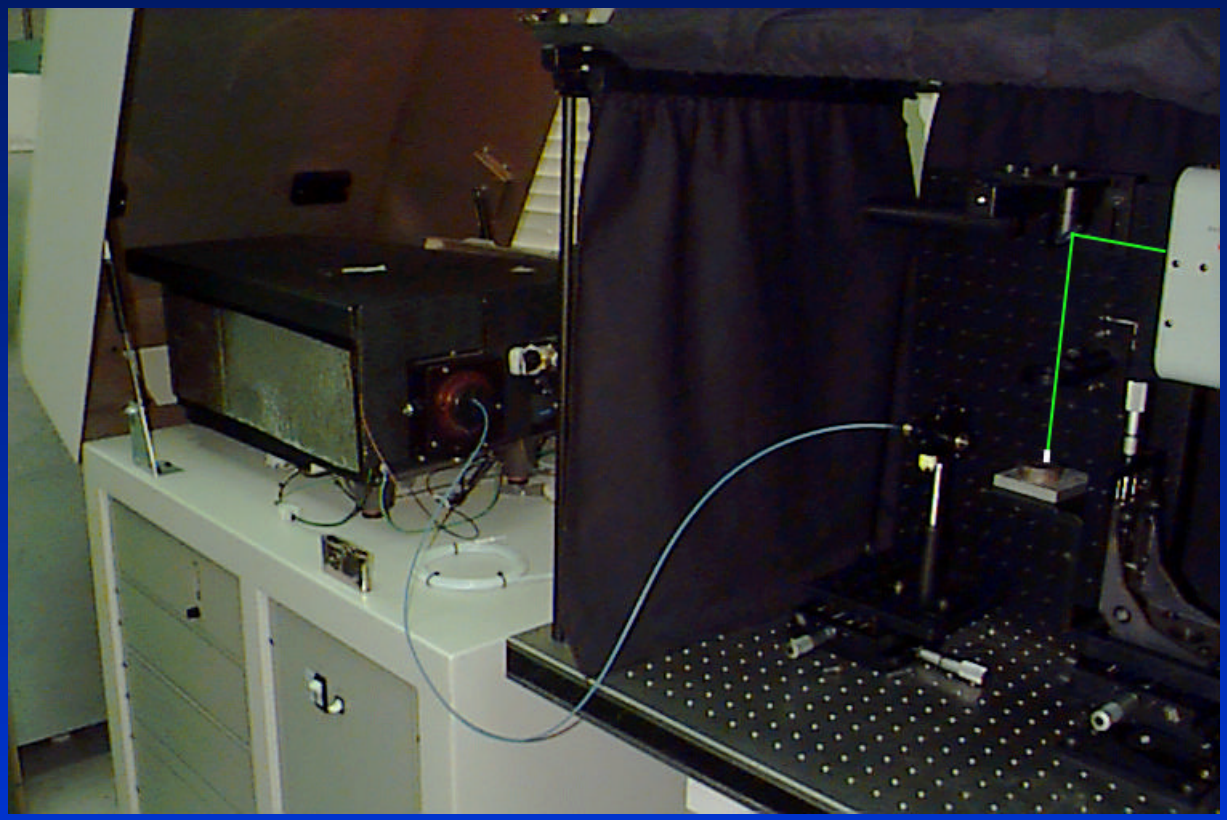
Information dispersed in two dimensions



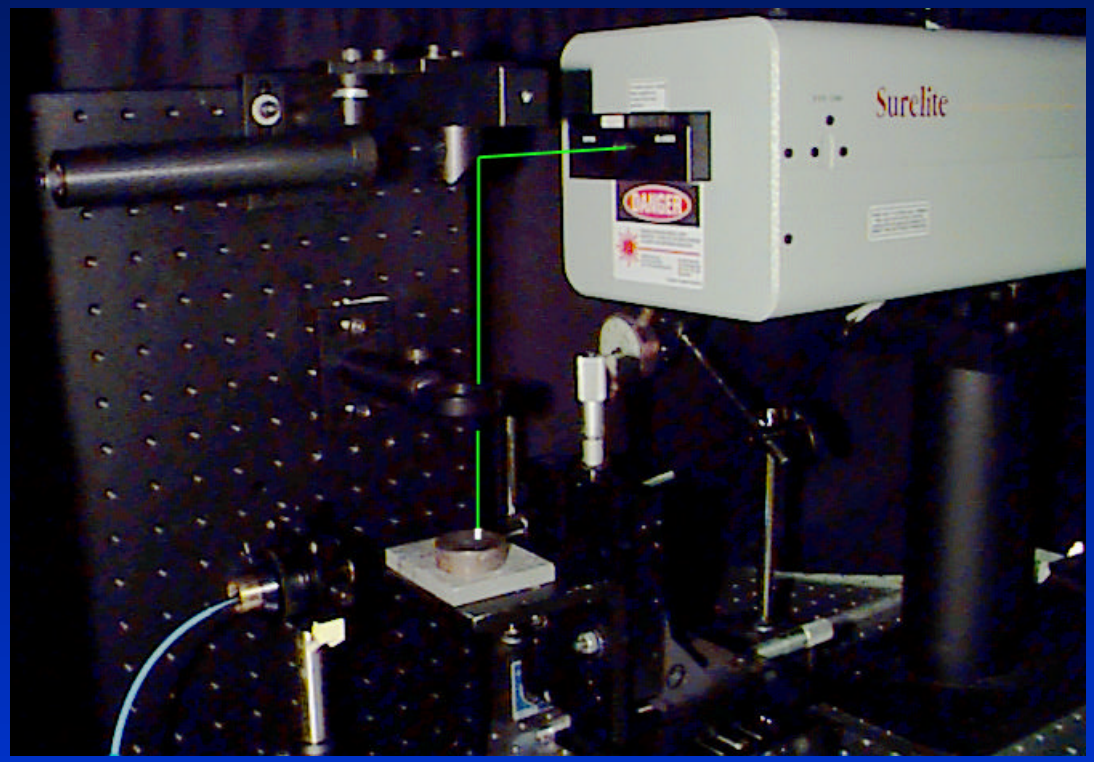
Echelle Spectrometer



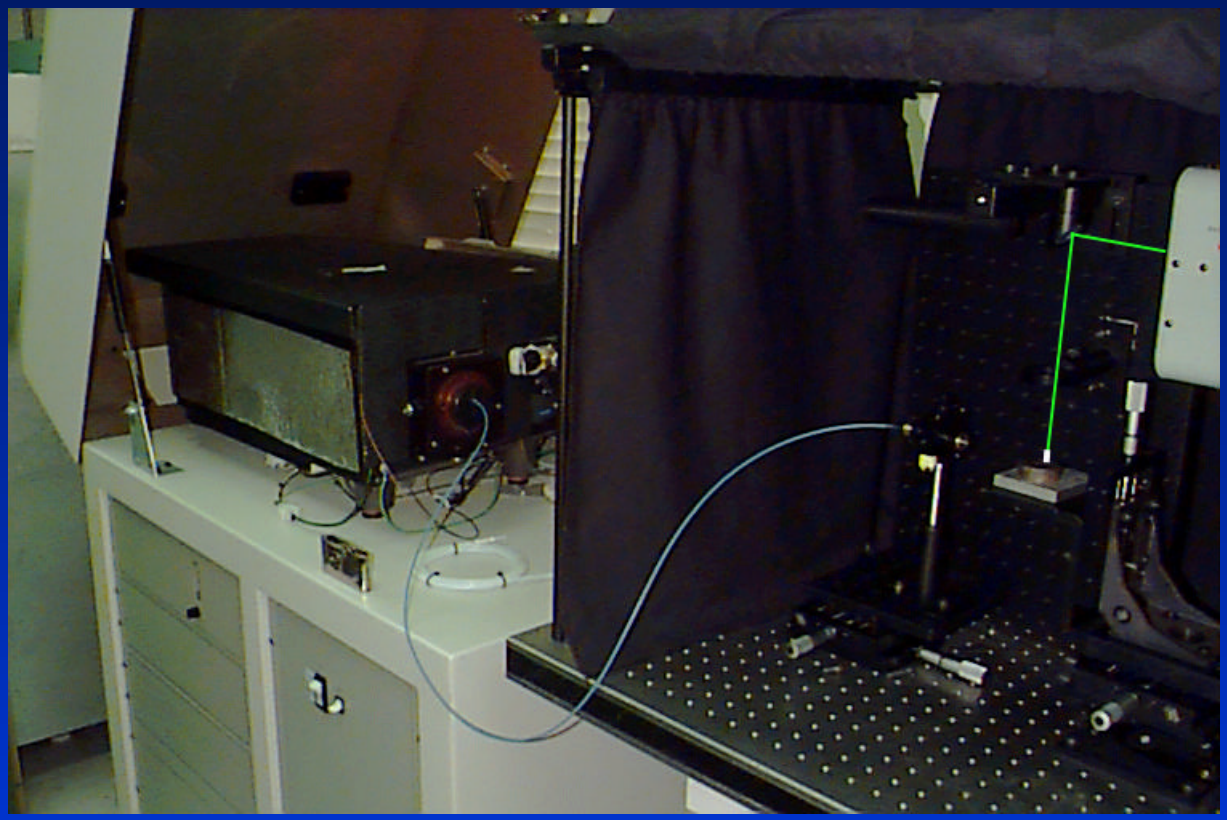
Collection of emission *via* fiber optic



Experimental apparatus used to generate plasma



Collection of emission *via* fiber optic



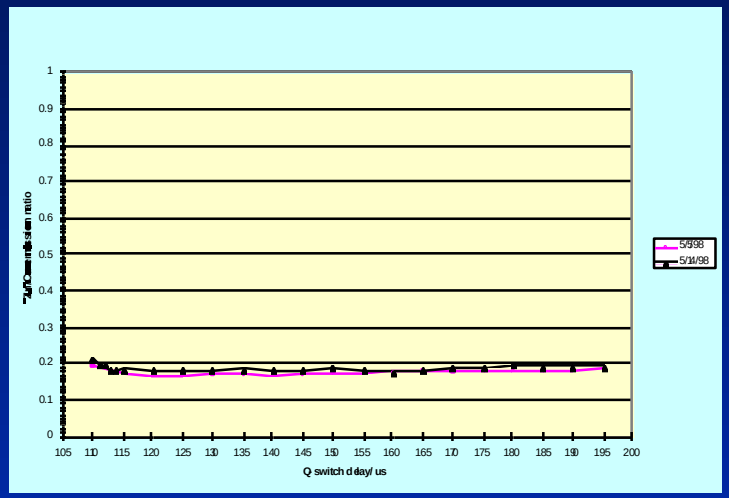
Matrix effects

- Use binary alloy (brass samples)
- Examine signals from zinc (volatile) and copper (nonvolatile)
- Vary laser power
- Vary focal depth

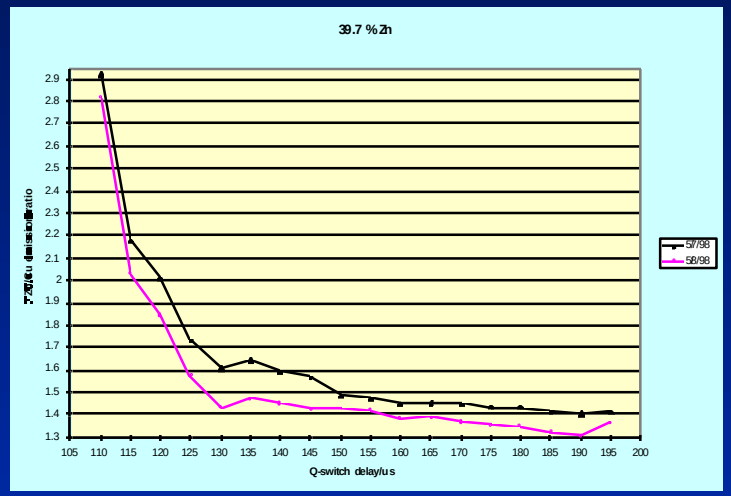
Studying selective volatilization

- Measure zinc and copper emission from brass standards
- Perform measurements while varying laser power (Q-switch delay)
- See if ratio is independent of power and proportional to concentration

Effect of Laser Power

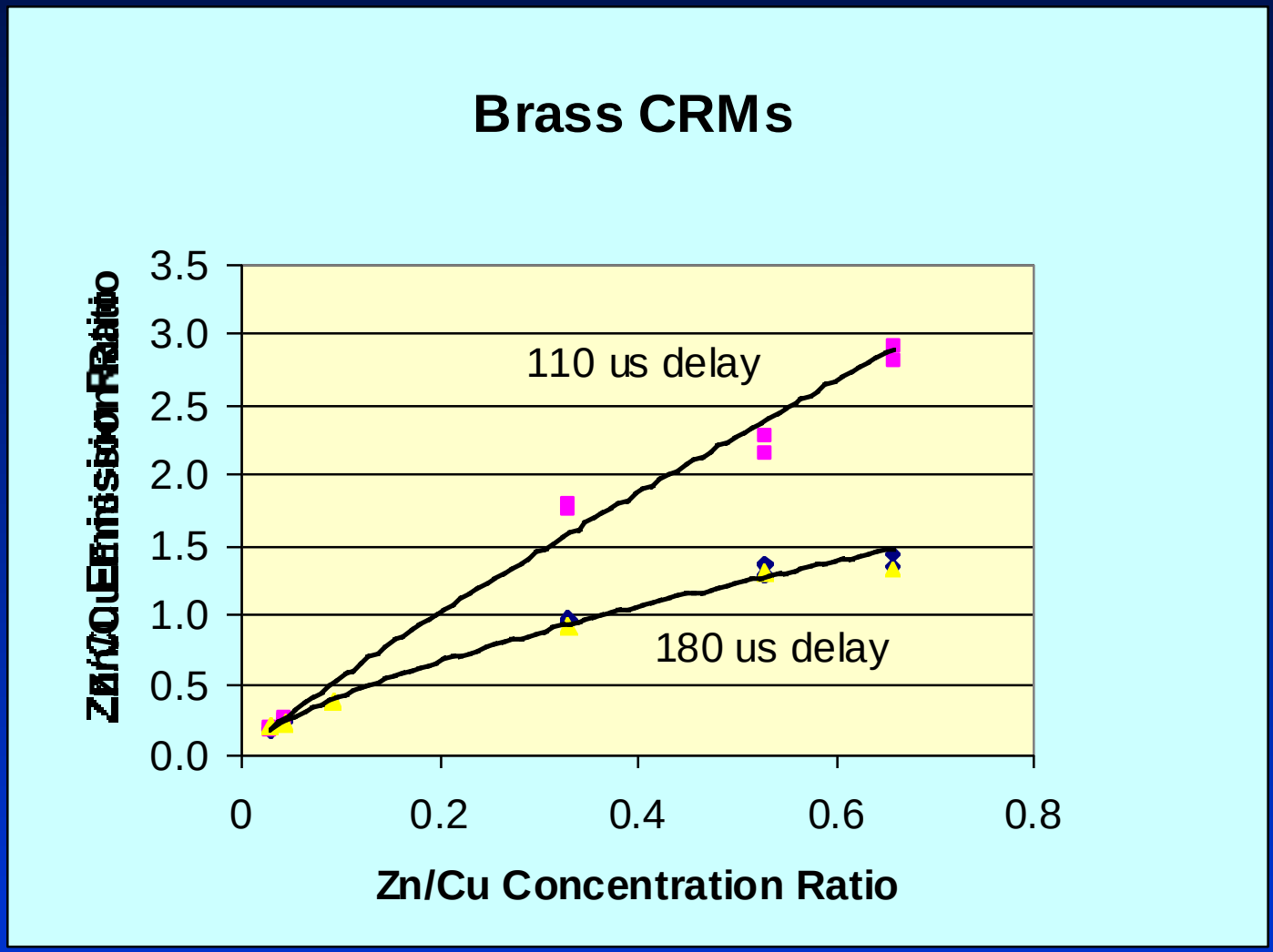


2.86% Zn



39.7% Zn

Calibration Plot



Segregate excitation effects from vaporization effects

- Brass samples, known composition
- Laser ablation into solution
- Dissolution
- Chemical analysis by ICP-MS
- Determine if materials vaporized in proportion to concentration
- Determine factors that affect selective and nonselective vaporization