# Confocal X-ray Fluorescence Microscope Status and Future

George J. Havrilla
Los Alamos National Lab
Ning Gao, XOS

HLW Workshop - SRNL
January 19-21, 2005

# Overview

- Research Objectives
- Completed Research
- Additional Activities

# Research Objectives

- Name Change
  - Radiochemical Analysis by High-Sensitivity Dual-Optic Micro X-ray Fluorescence
  - "Radiochemical Analysis by High-Sensitivity Confocal Micro X-ray Fluorescence"

- Develop novel micro X-ray fluorescence instrument for radiochemical analysis

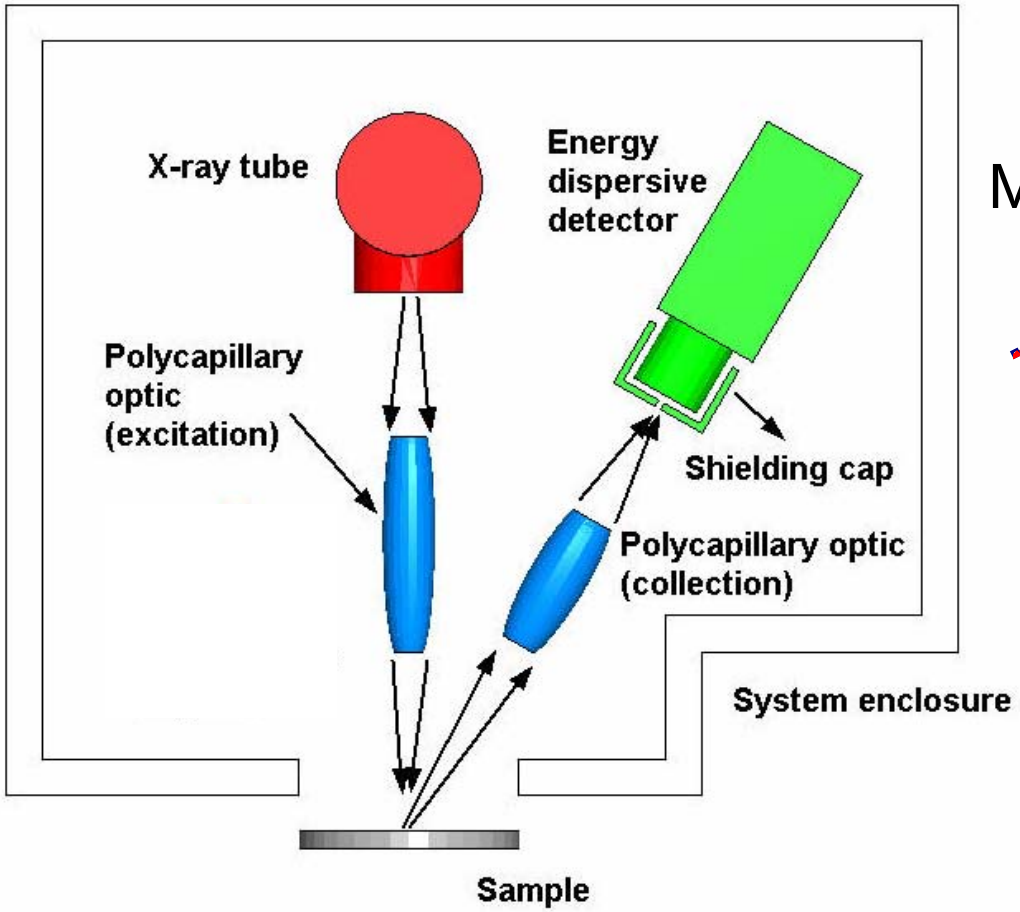
# Objectives

- Develop novel energy dispersive X-ray fluorescence spectrometer using dual X-ray optics to increase elemental analysis sensitivity

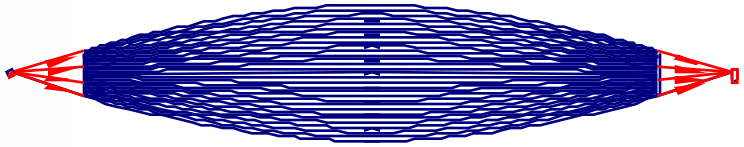
- Develop fundamental understanding of dual-optic coupling and optimize parameters for MXRF analysis

- Develop and test prototype MXRF instrument capable of analyzing radioactive samples for elements of interest

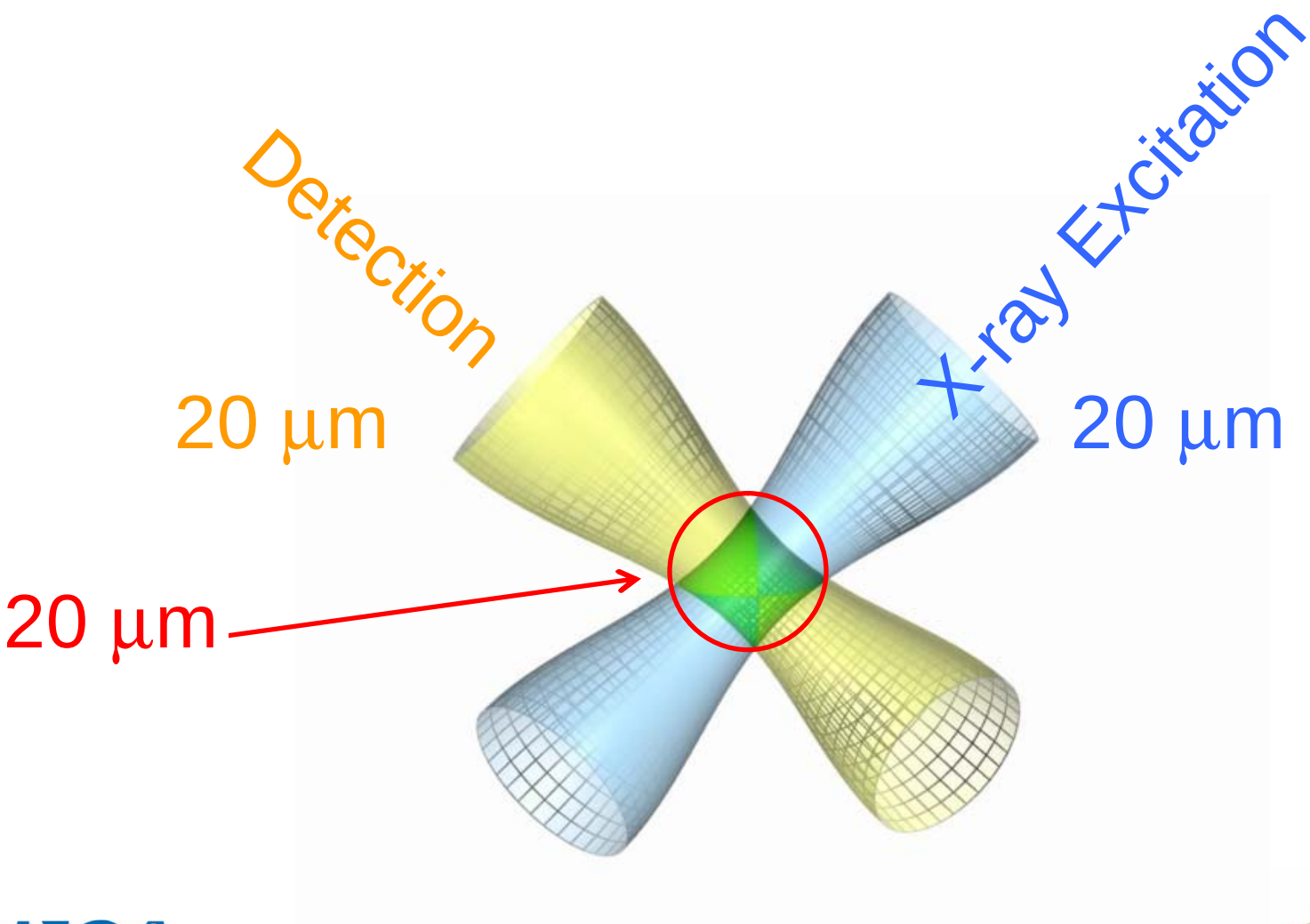
# Conceptual Design



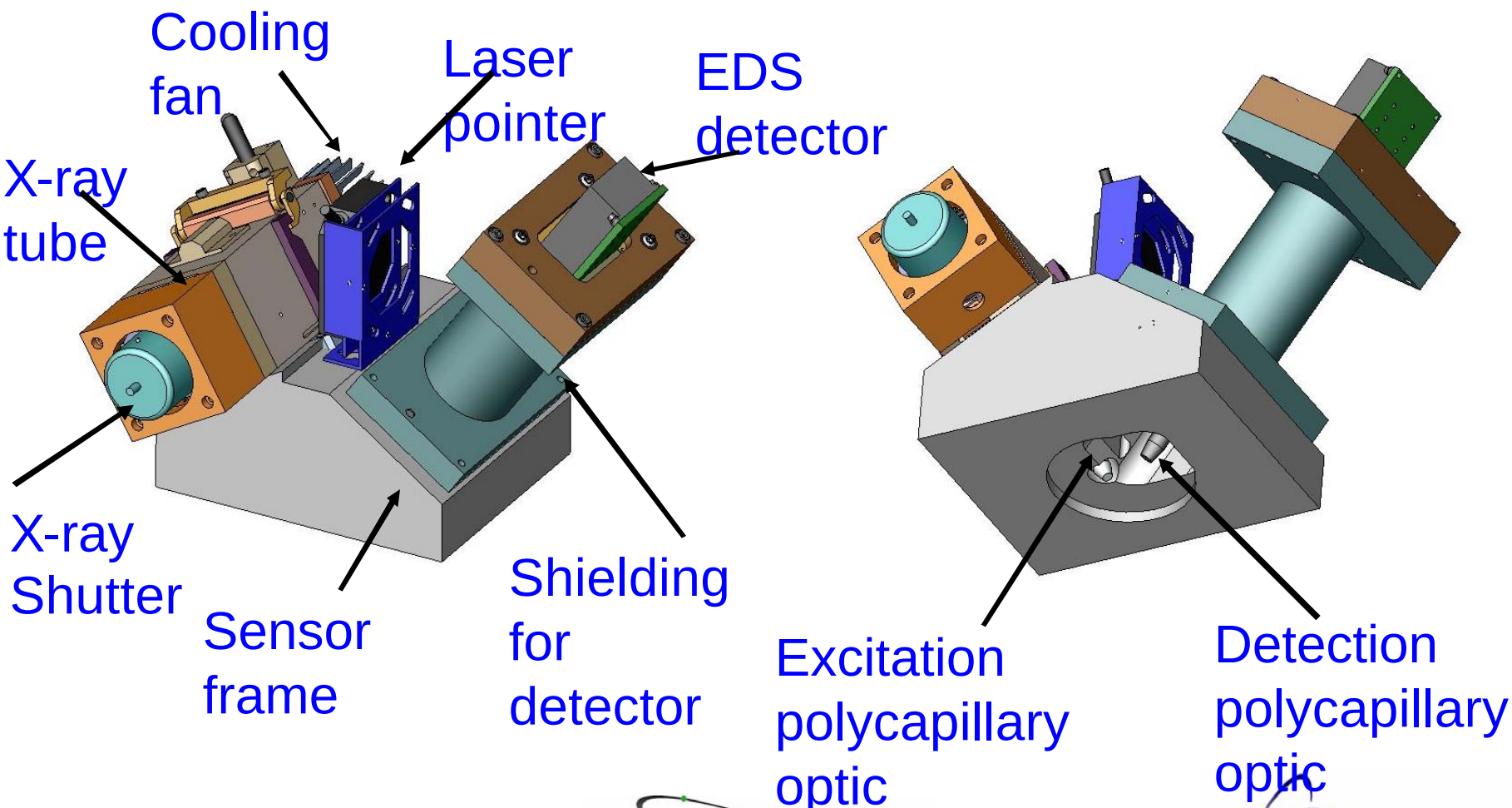

Monolithic Polycapillary Optic



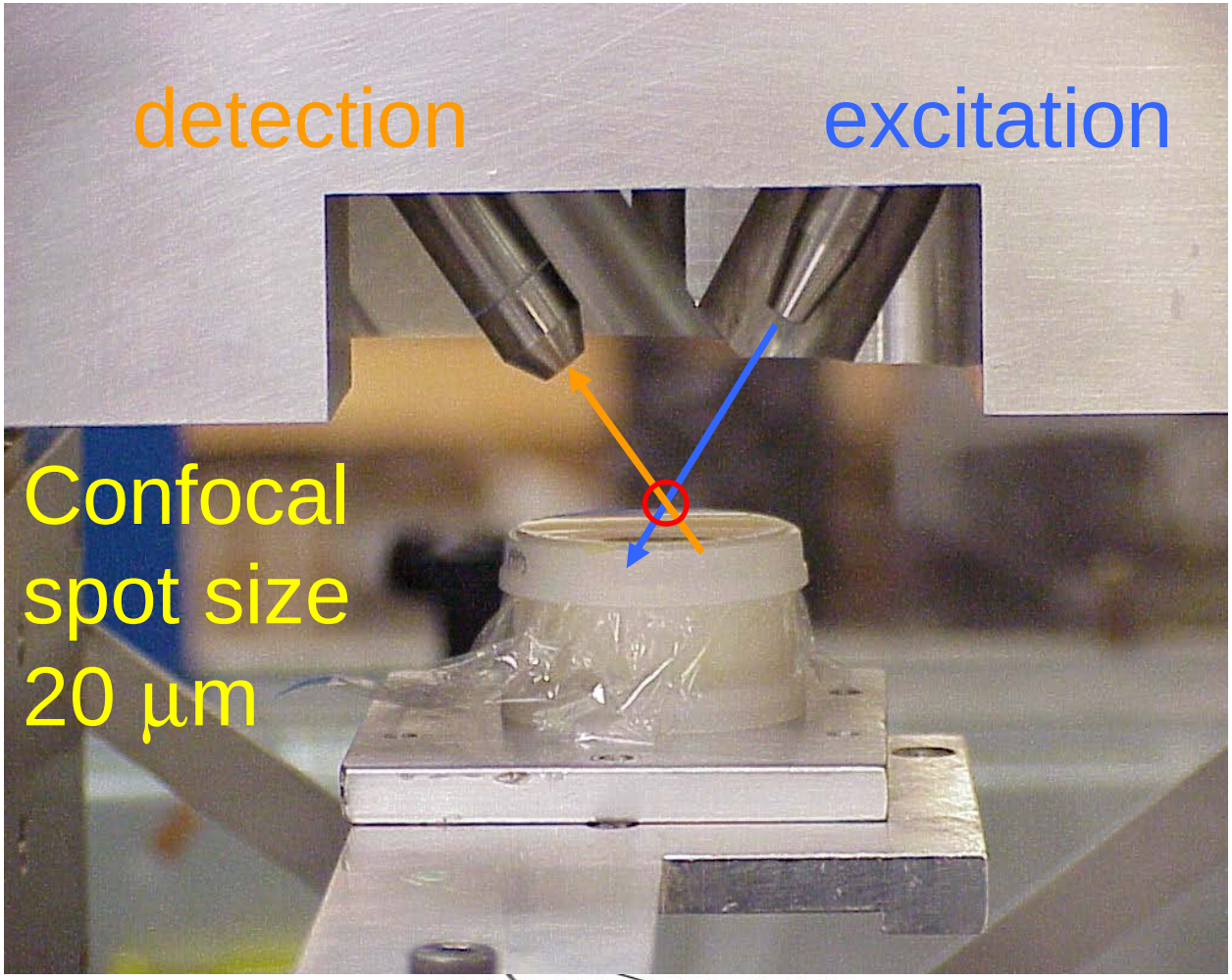
# Confocal Volume



# Instrument Design

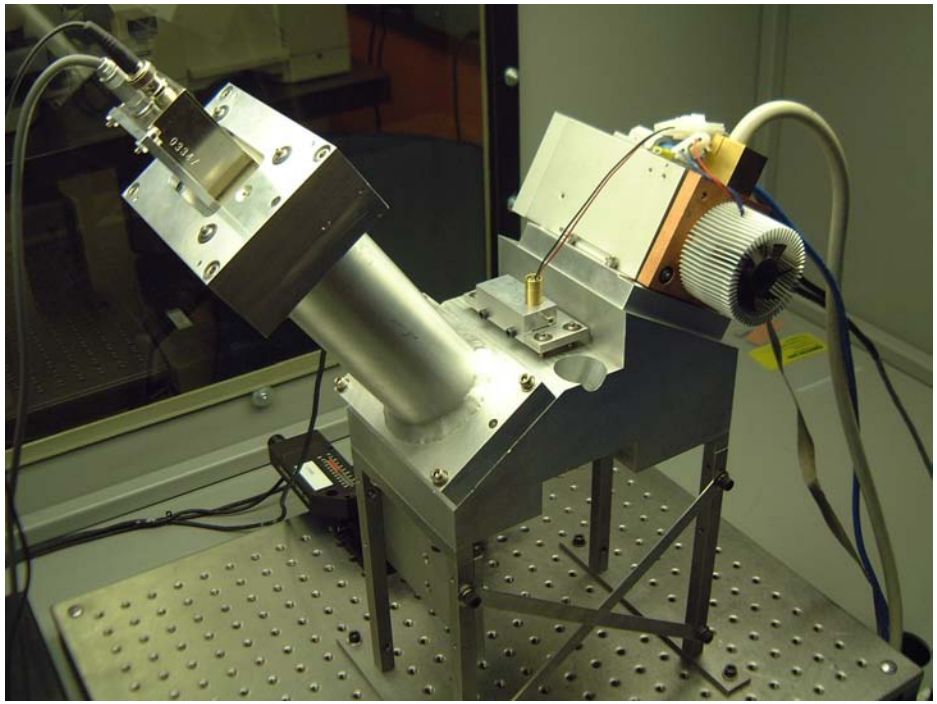


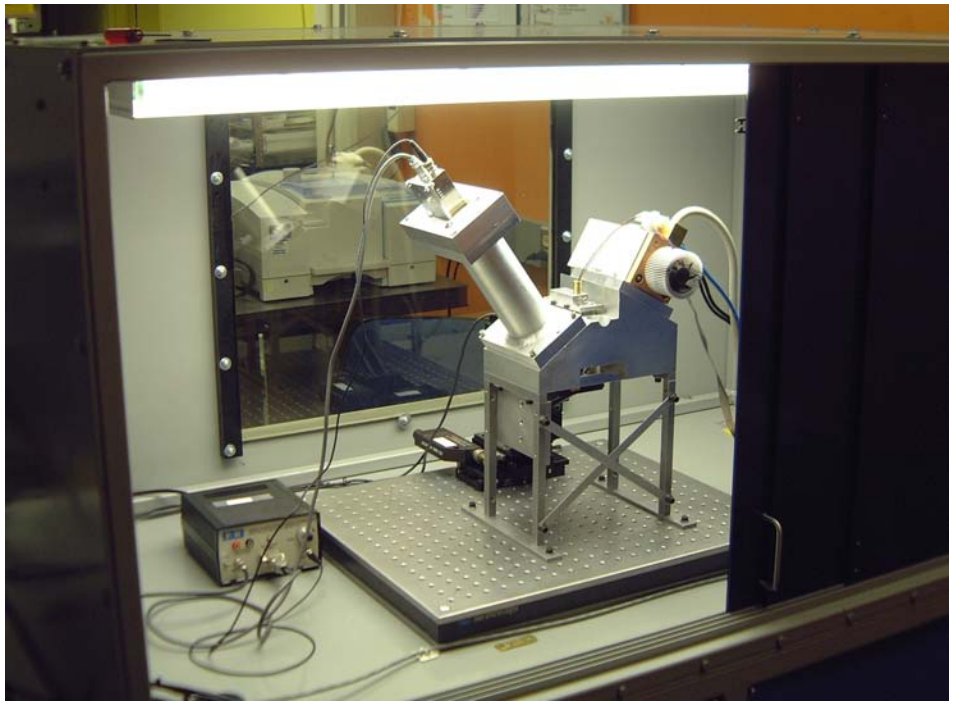
# Optical Arrangement



# Confocal Instrument

# Confocal Instrument





# Instrument Specifications

- Ag X-ray tube, 50 kV, 0.5 mA, X-Beam

- Excitation and detection optics are ~30 micrometer focal spots with ~10 mm working distance

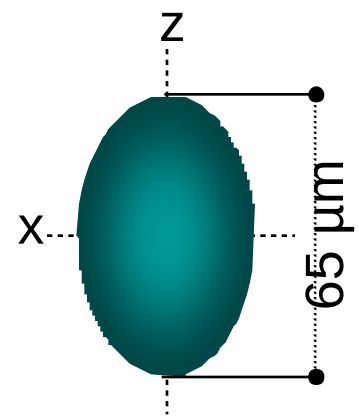
# Beam Characterization

Beam dimensions at 30 kV, 0.3 mA

z: 65 µm

y: 30 µm

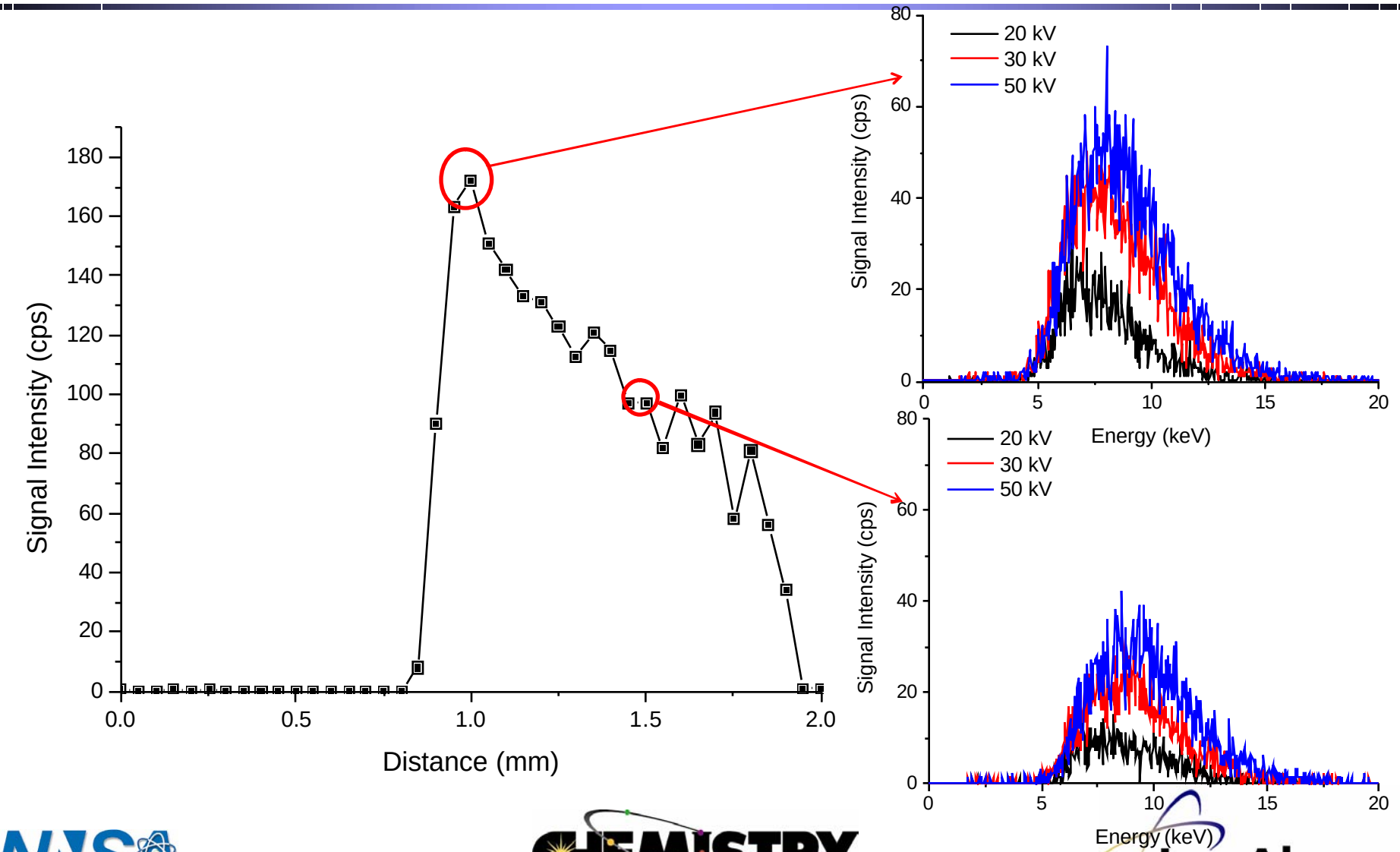
x: 30 µm



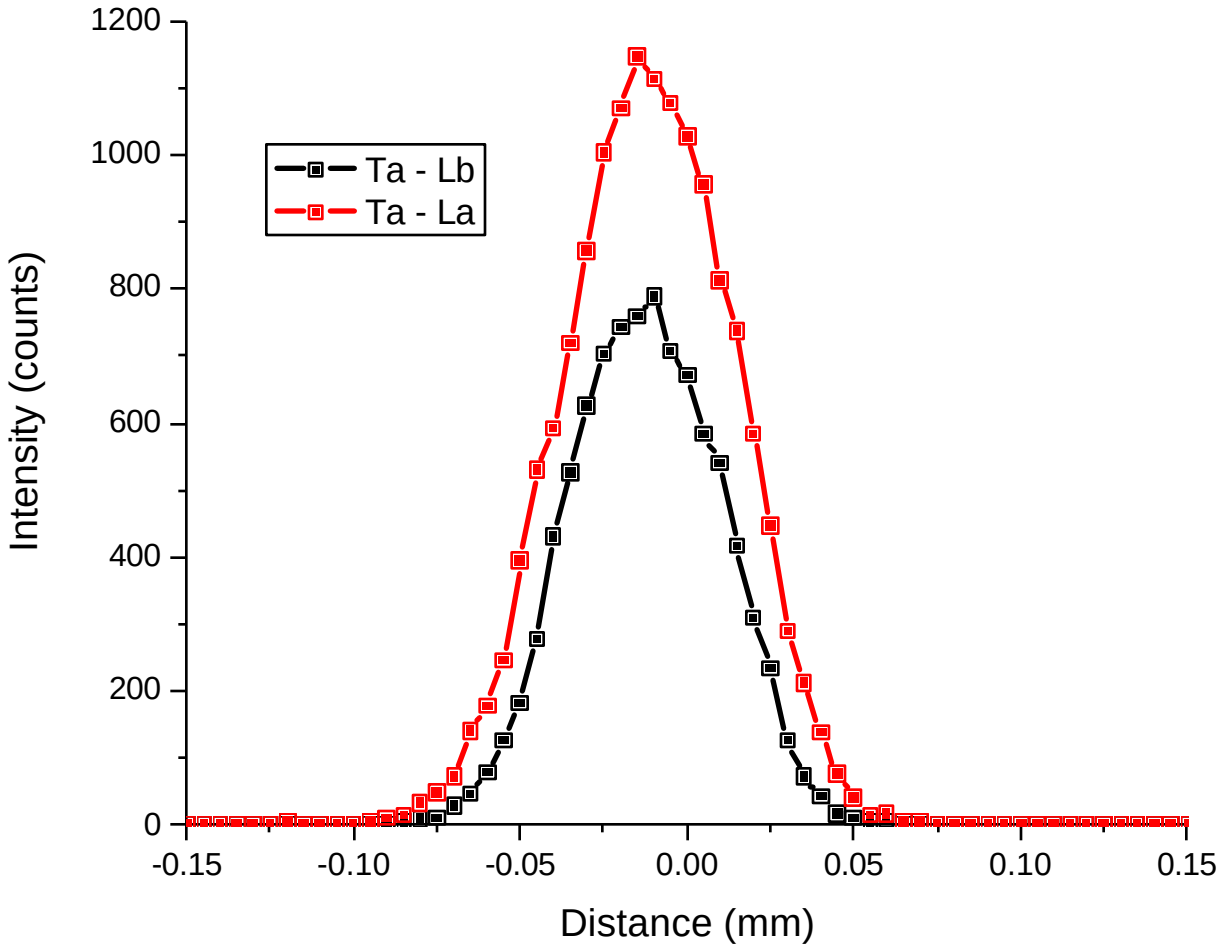

**Z:** as the voltage or current increased the beam size remained at ~60µm

**Y and X:** no trend, based on voltage or current, was apparent for the beam size. Beam size ranged from 25-30 µm

# Emission Profile



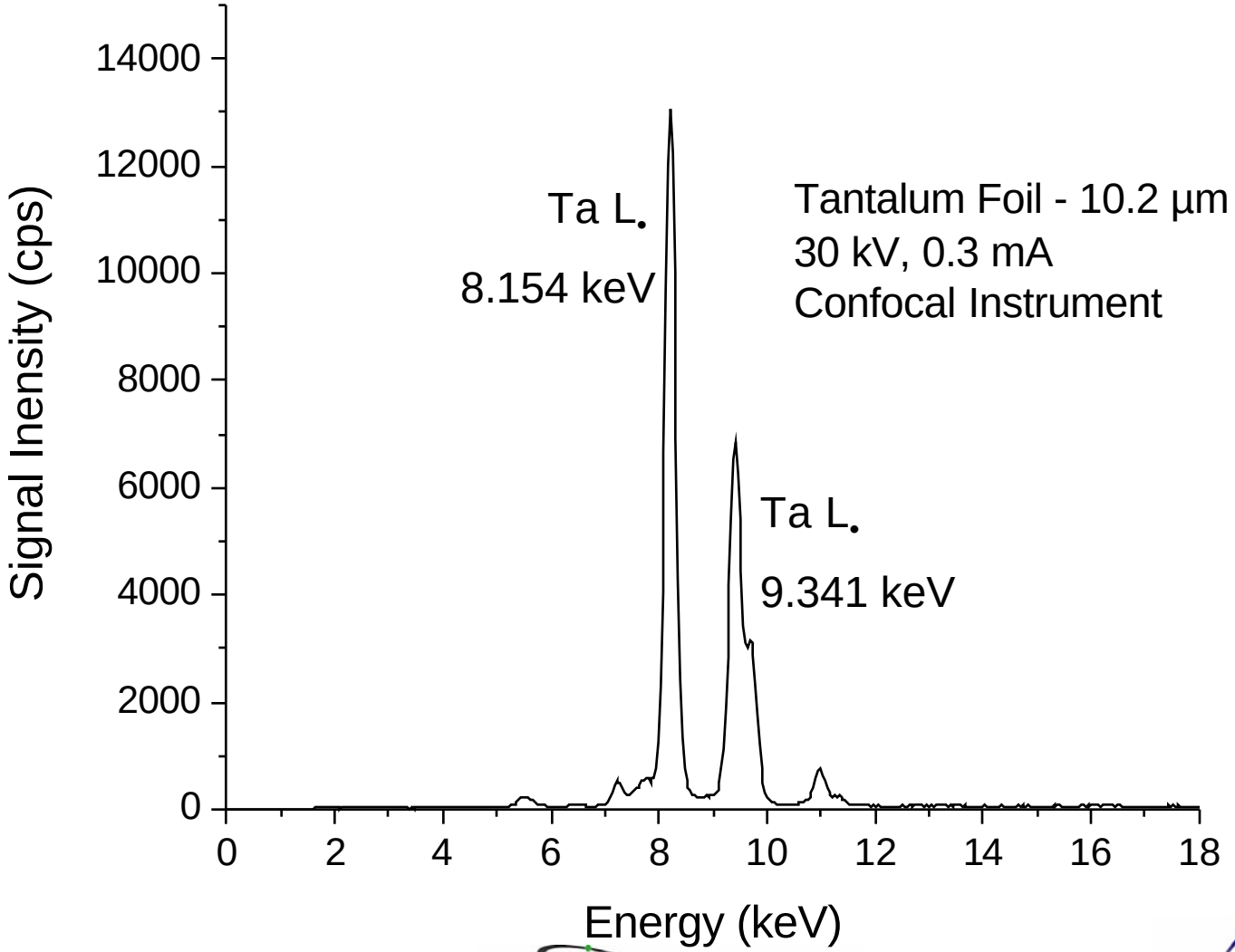
# Step Size Resolution – 5 micrometers



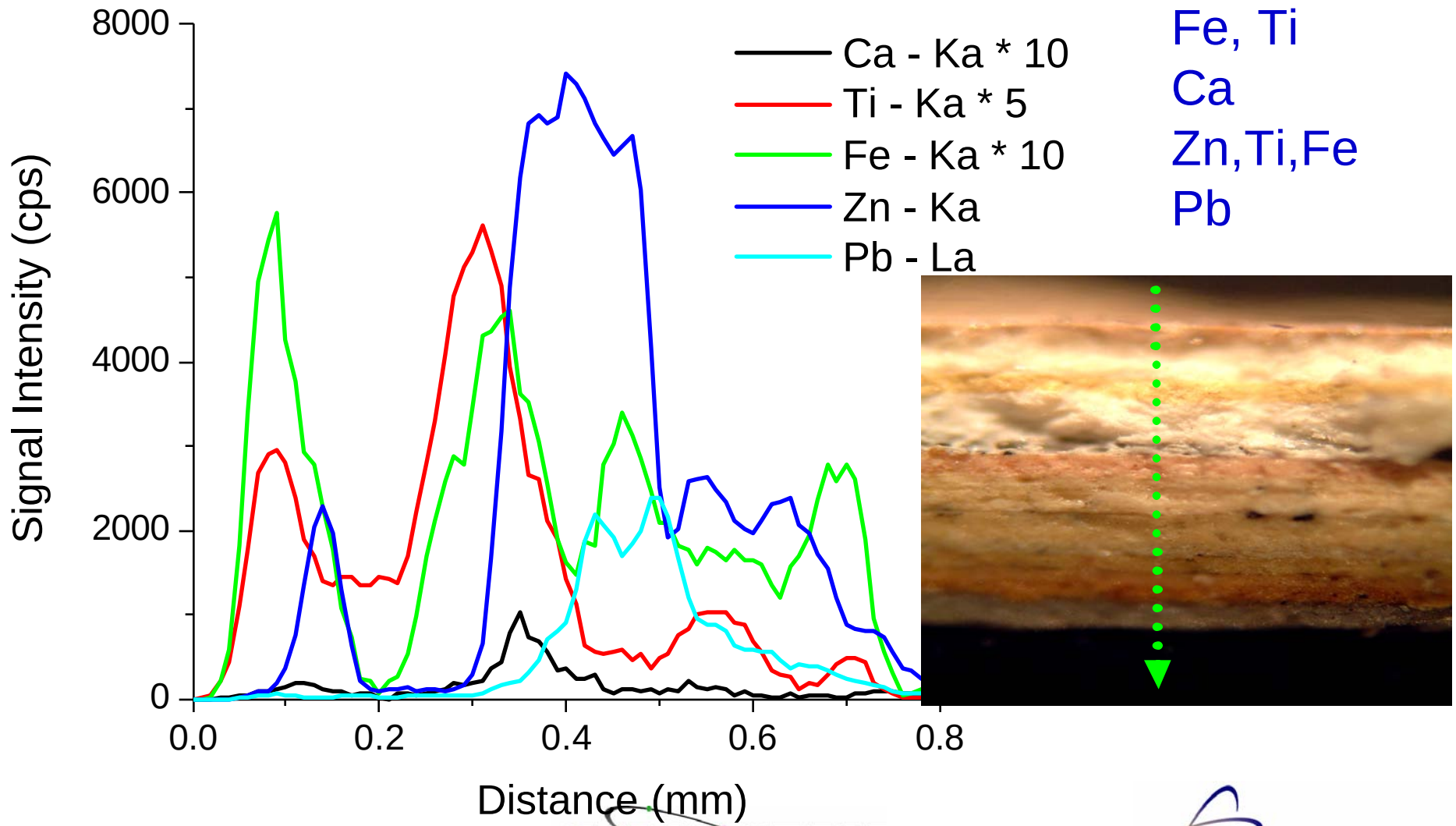
# Data Acquisition

- Point – spectrum of selected spots
- Line – either elemental line scans or full spectra across the line
- Depth – line scan in the z-direction: into the sample - NONDESTRUCTIVE
- 2D – x, y array scan
- 3D – x, y array scans at various depths

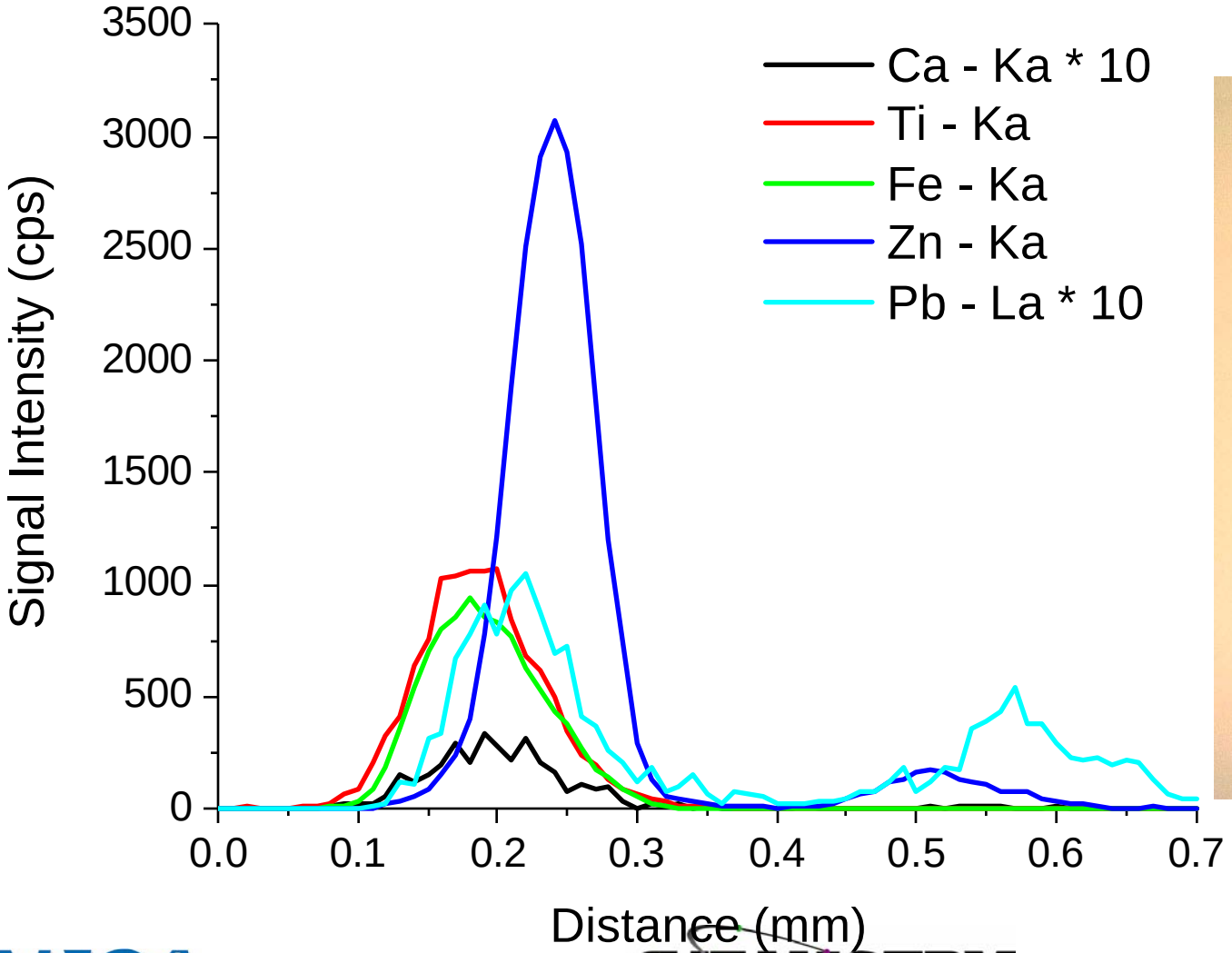
# Point Spectrum

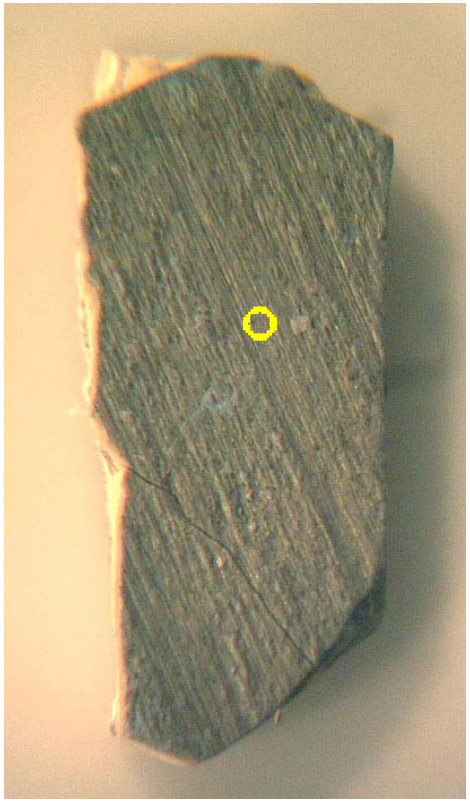


# Line Scans

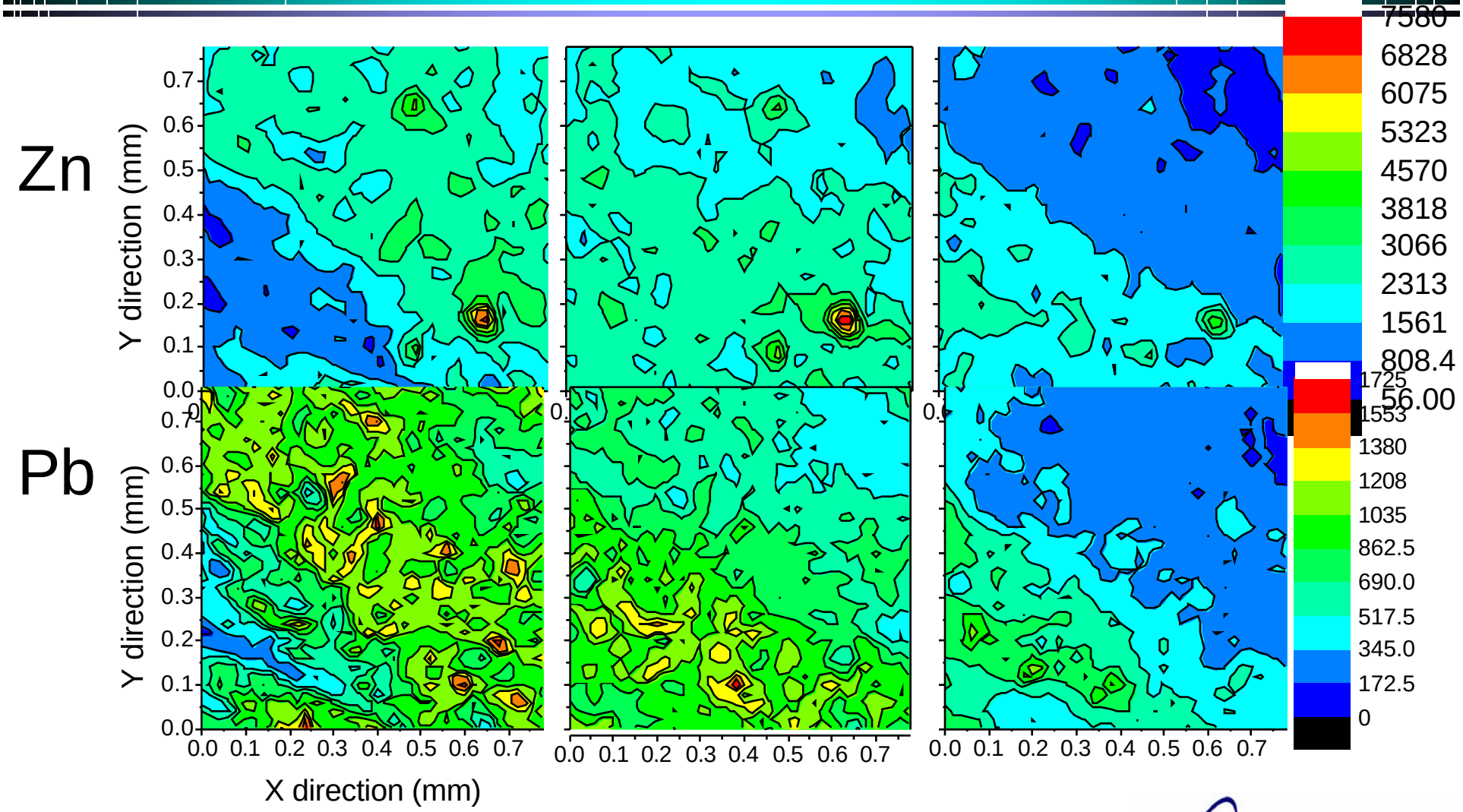

8000
6000
4000
2000
0
Signal Intensity (cps)
0.0
0.2
0.4
0.6
0.8
Distance (mm)
Ca - Ka * 10
Ti - Ka * 5
Fe - Ka * 10
Zn - Ka
Pb - La
Fe, Ti
Ca
Zn,Ti,Fe
Pb

# Depth Profiles – 20 micrometer steps

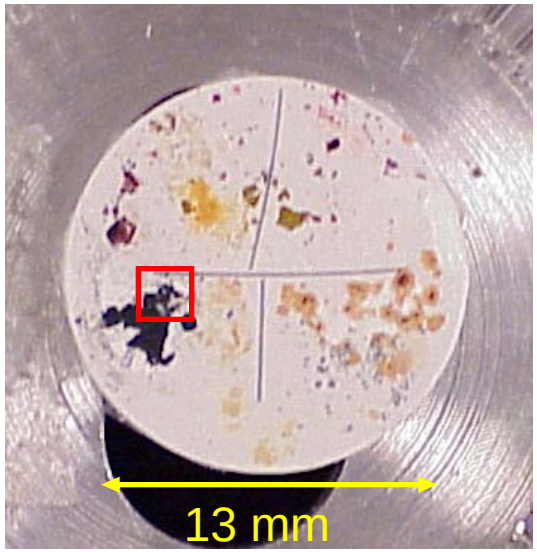


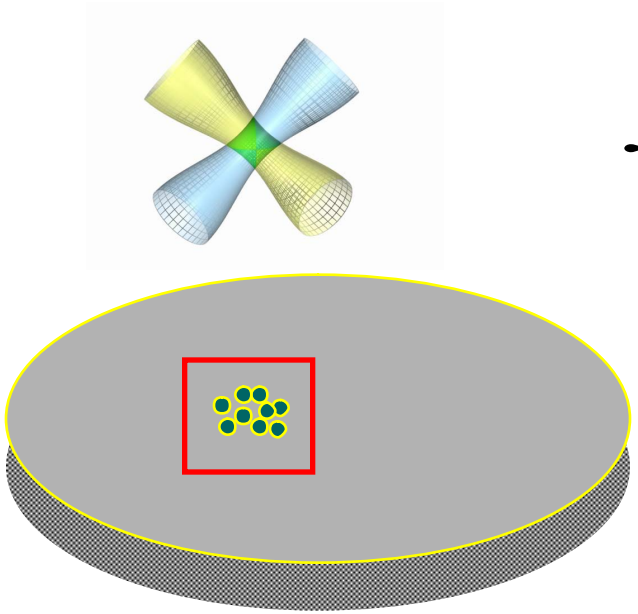



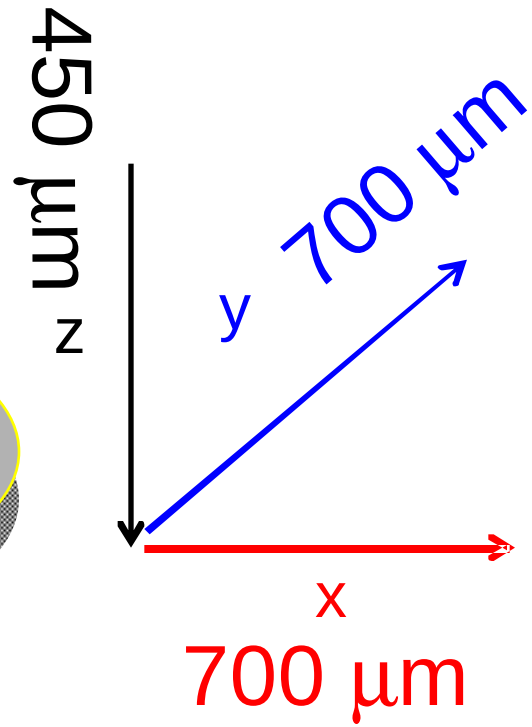
# 3D Array Depth Profiling – 20 µm steps



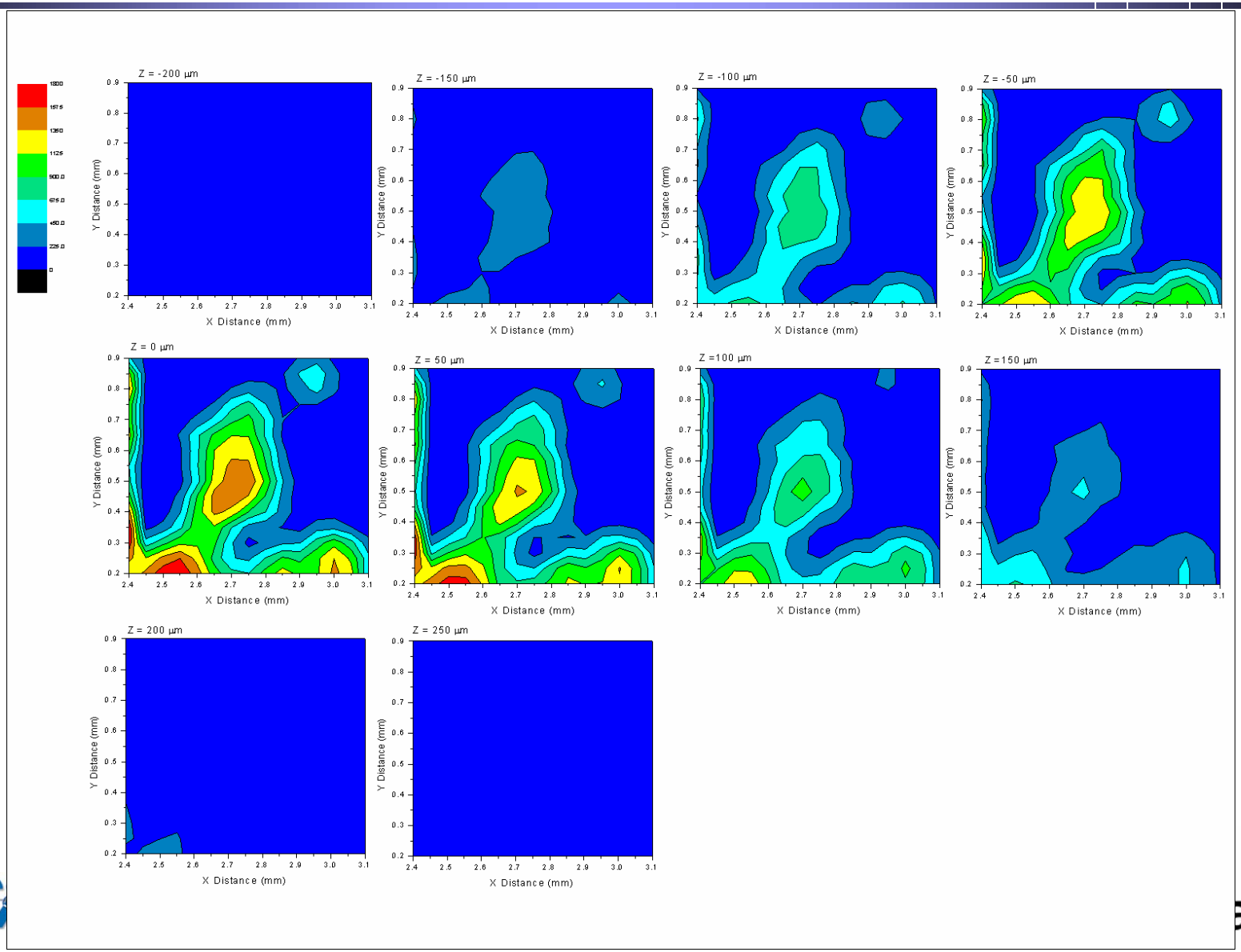
# 3D Elemental Mapping



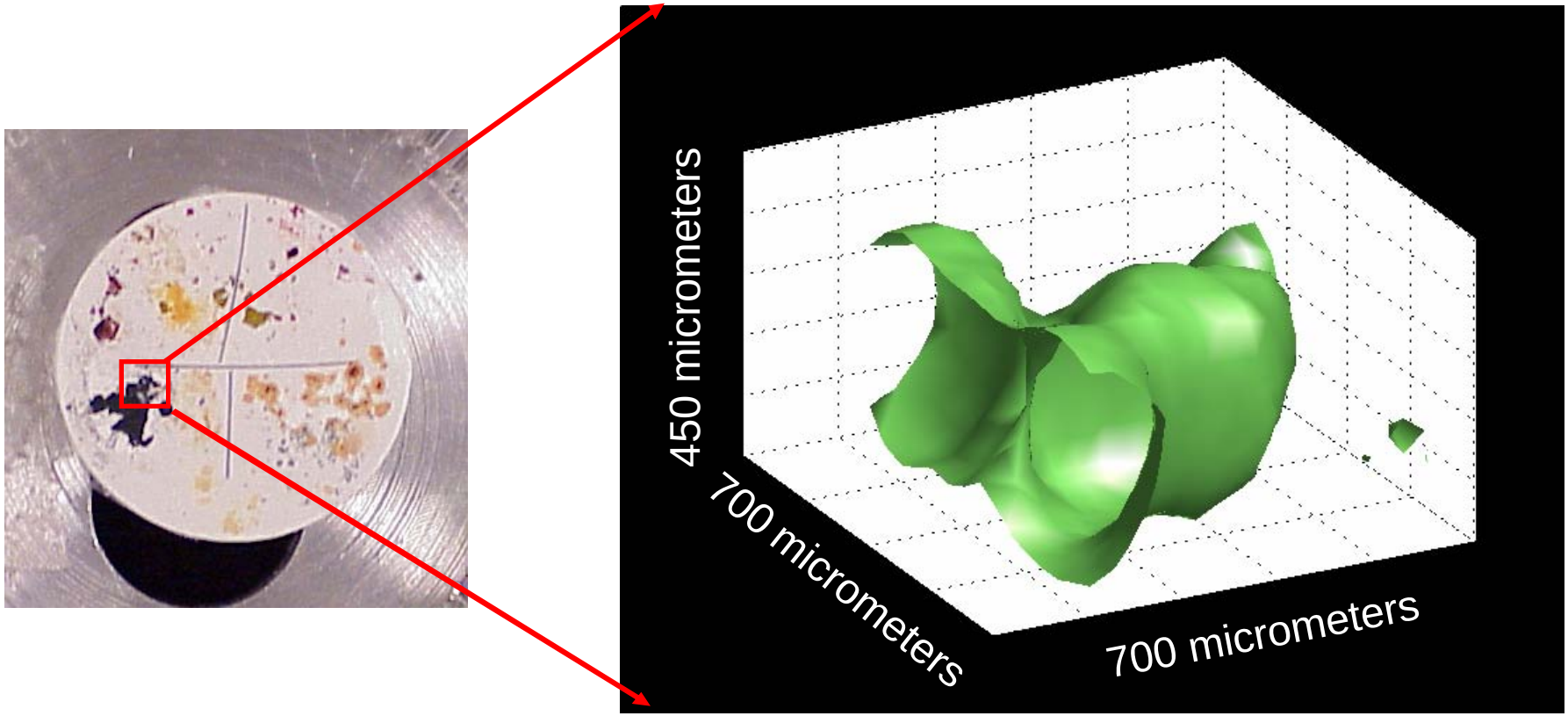





# 2D Elemental Maps of Iron



# 3D Elemental Map of Iron



# Results – alpha prototype

- Model analyte system 100-ppm each (Sr, Pt, Pb, Pd, Mo, Cu) with 1 M sodium chloride test matrix for the sodium hydroxide tank matrix. All target elements were detected below 10-ppm with a 600-second counting time. Pd had a detection limit of 26 ppm.

- Iron 55 source for radiation background reduction with (confocal) optic design. Radiation rejection measurements showed a factor of 20 rejection ratio using an iron 55 source.

# Results

- Confocal MXRF provides unique analytical capabilities for materials characterization
  - Nondestructive elemental analyses
  - Depth profiles
  - 3D elemental mapping
  - Has potential for increased sensitivity due to reduced background
  - Radiation rejection for hot sample characterization

# Plans

- Prototype Testing
  - Demonstrate radiation rejection on hot samples
  - Determine sensitivity, LOD for target analytes

- Applications are limitless

# Relevance

- Needs
  - rapid, sensitive, radiochemical analyses
- Potential
  - at/in-tank analyses
  - on-line/at-line analyses
- Provides new approach to elemental analysis that will aid in streamlining tank waste characterization and clean-up processes

# Implementation

- Follow-on funding
  - development of hardened system
    - shielding
  - lab testing of hot samples
    - determine radiation limits
  - evaluation for in-line process monitoring
    - model cold flowing system

# Ultimate Goal

- Develop an on-line instrument capable of measuring the desired analytes in SRS and RL HLW process lines.
- Areas of interest to be addressed
  - High ionic strength liquid analyses
  - $Sr^{90}$ detection with high sensitivity
  - Improve TRU detection
  - Improved radiochemical analysis sensitivity, 0.5% in glass and 0.05% for nuclides