EMSP

Environmental Management Science Program

Project # 81967

Radiochemical Analysis by High Sensitivity Dual-Optic Micro X-ray Fluorescence

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X-ray Optical Systems

NNSA

CHEMISTRY

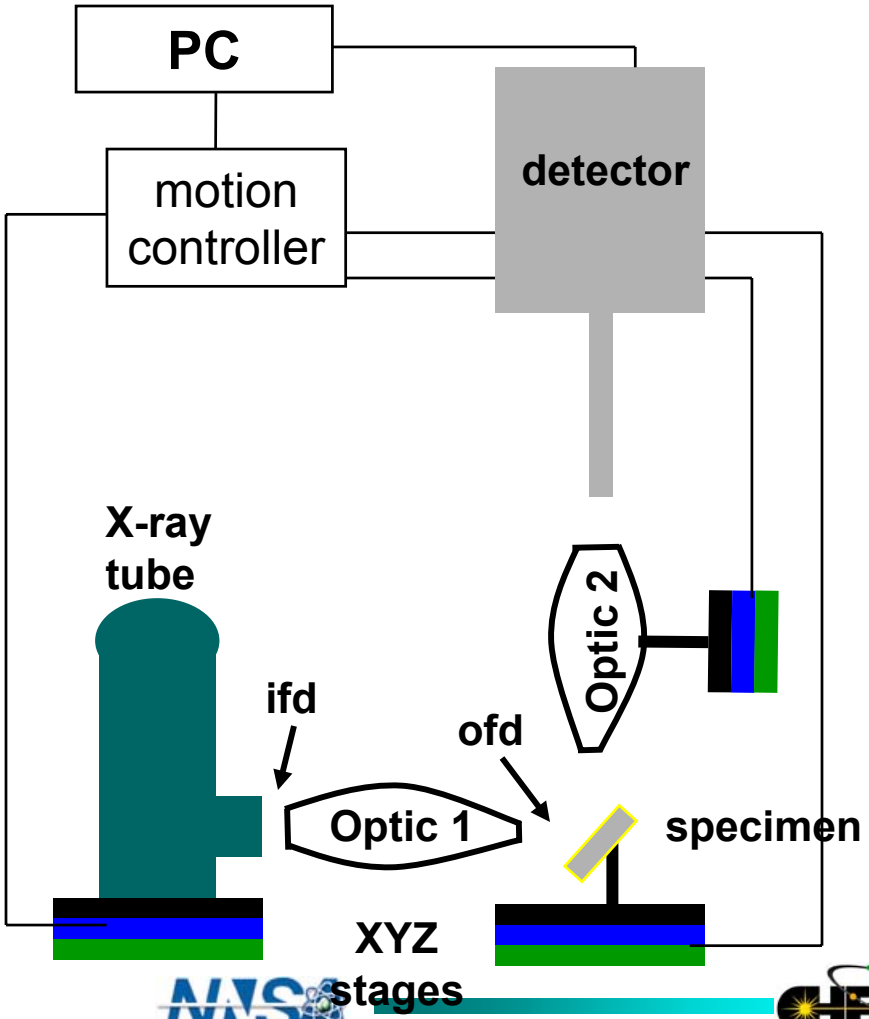
1943 - 2003 Los Alamos NATIONAL LABORATORY

Ideas That Change the World

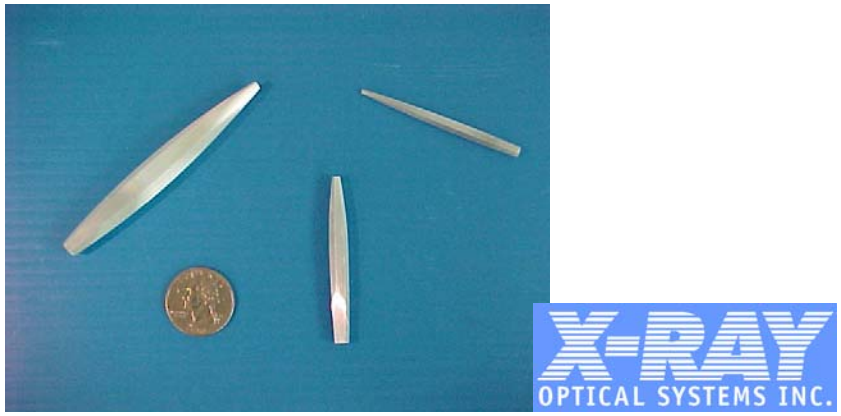
Objectives

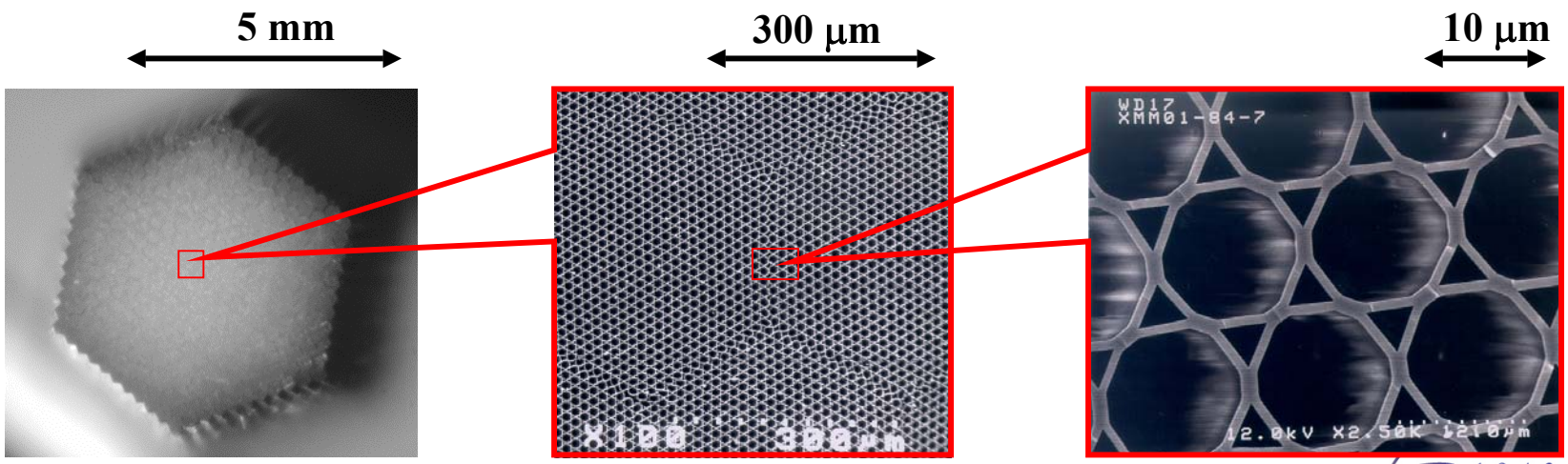
- Develop novel energy dispersive X-ray fluorescence spectrometer using dual X-ray optics to increase elemental analysis sensitivity
- Develop fundamental understanding of dual-optic coupling and optimize parameters for MXRF analysis
- Develop and test prototype MXRF instrument capable of analyzing radioactive samples for elements of interest

Experimental

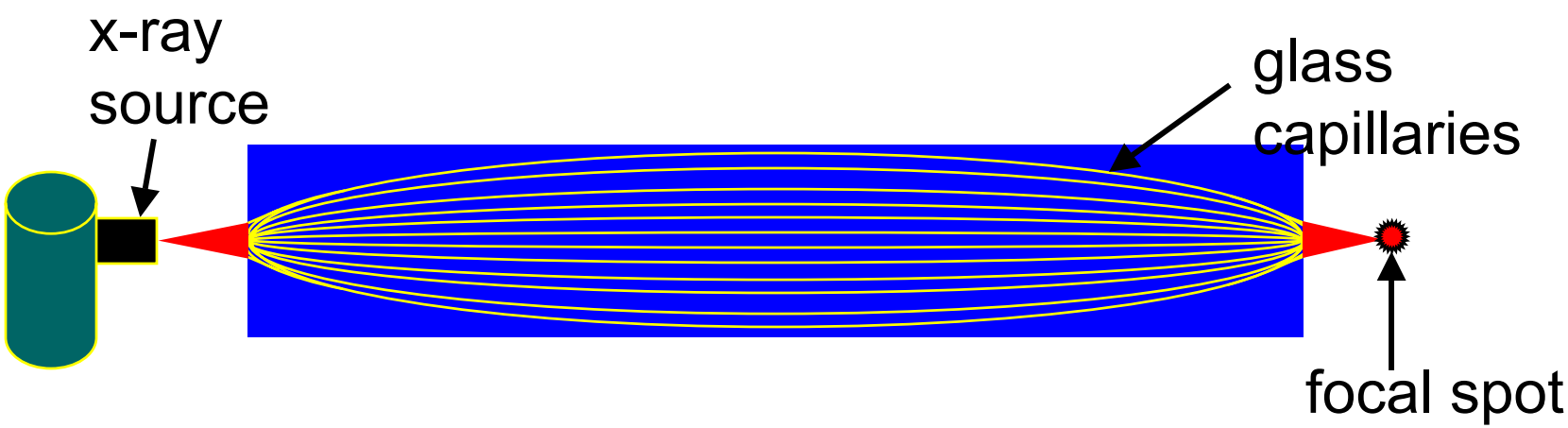


Polycapillary X-Ray Optics

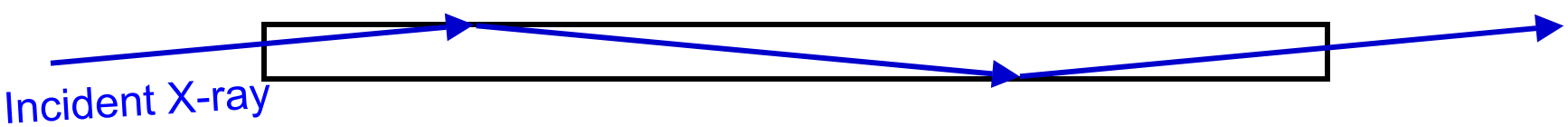




Polycapillary Optic



Individual capillary



X-ray Optics

- Increase resolution of polycapillary
- Lower background
- Reduce background radiation from radioactive samples

- Utilize dual polycapillary on both excitation and detection sides

Experimental

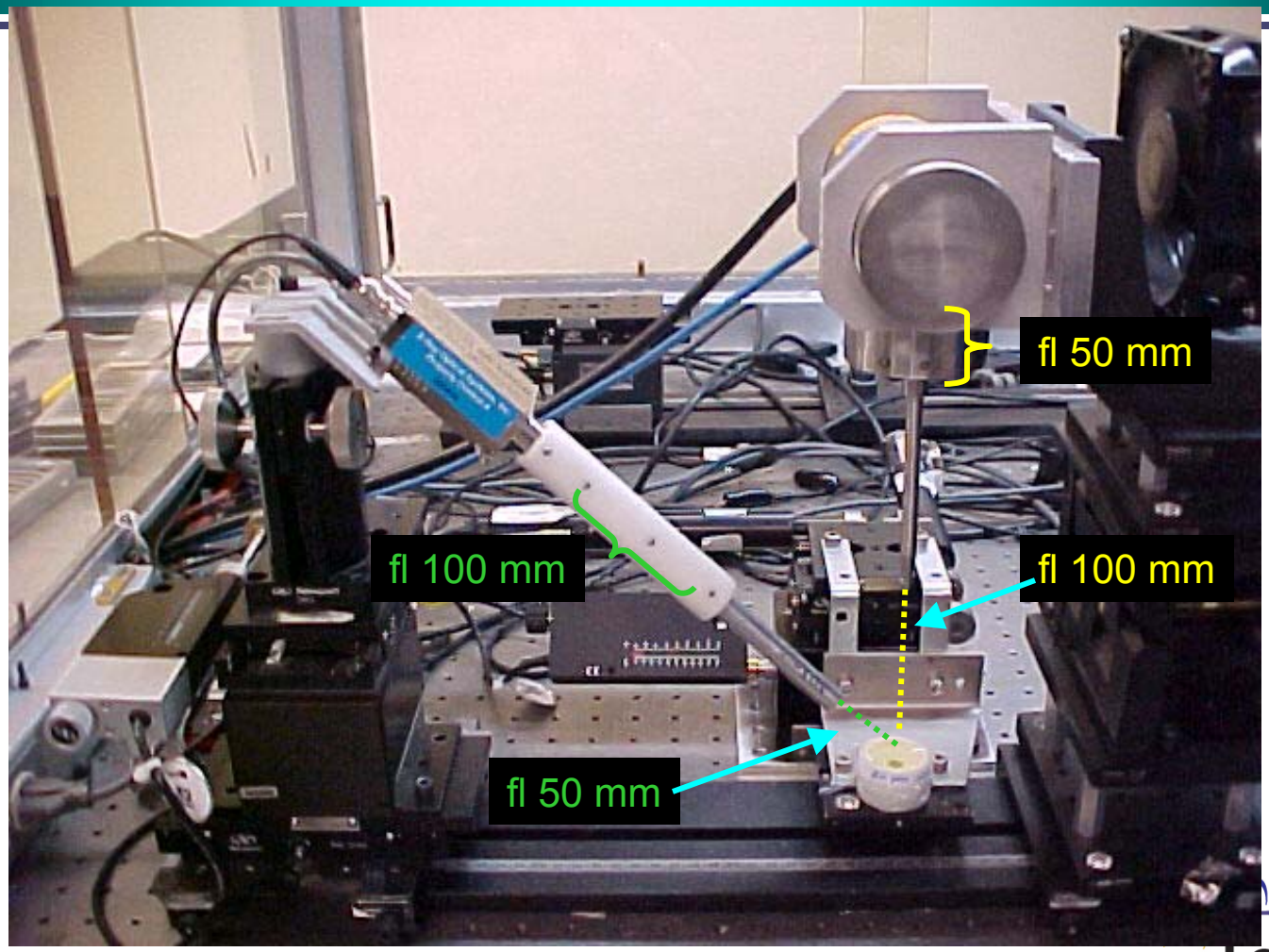
- Lab built system with data acquisition system
- Oxford 5011 Cu X-ray tube, 120 micrometer spot, 50kV, 1 mA (50 watts)
- AMPTEK 100 CR solid state detector, 200 eV at 5.9 keV, 5 mm^2 active area
- Capillary optics

Experimental

- Capillaries - XOS monolithic polycapillary

	Initial	#2
➔Excitation		
⇨ input focal length -	50 mm	55mm
⇨ output focal length -	100 mm	30mm
➔Detection		
⇨ input focal length -	50 mm	30mm
⇨ output focal length -	100 mm	200mm
➔Spot Size	120 μm	100 μm

Initial Design



Results of Initial Dual Capillary

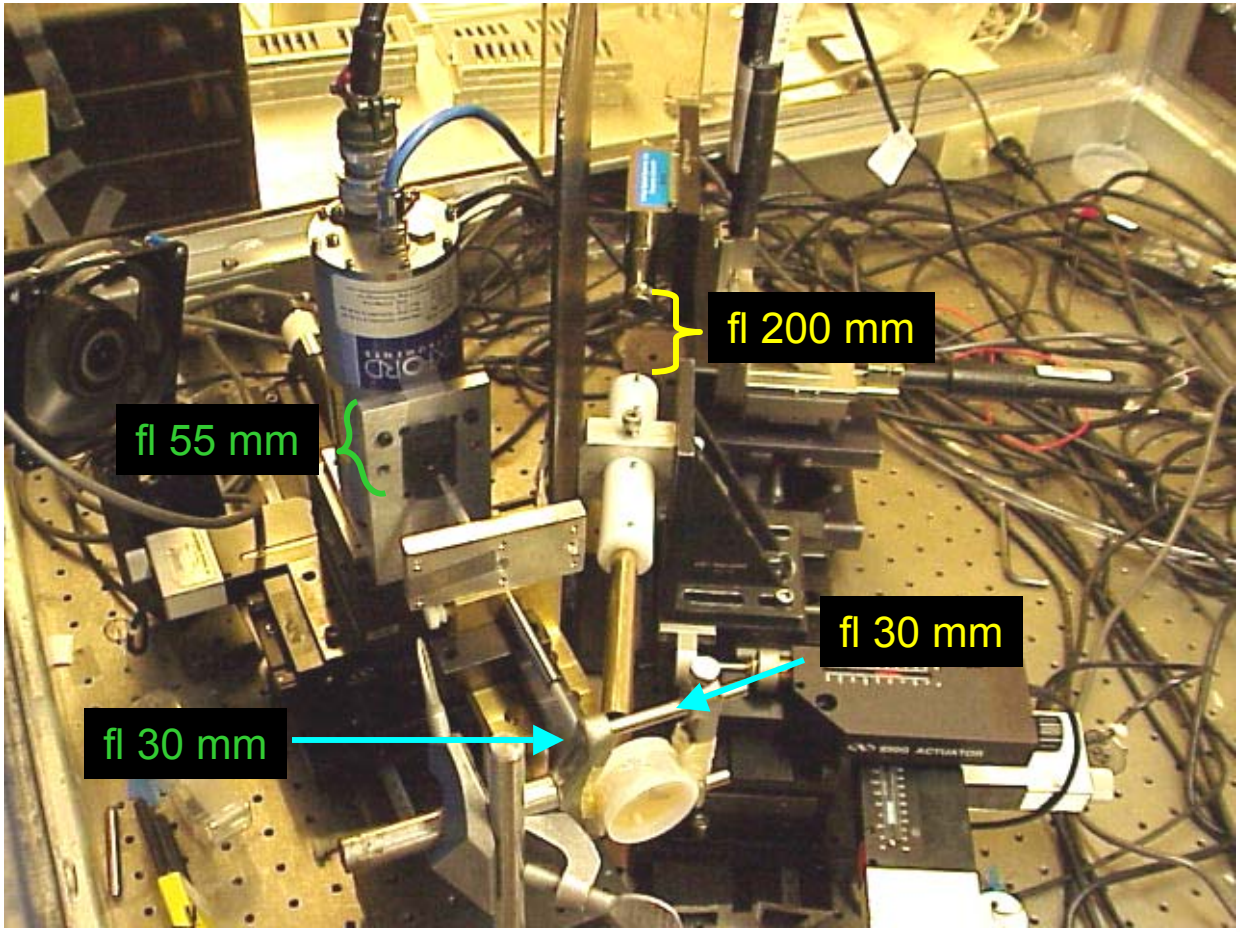
- Spatial Resolution increased by ~15%
- Intensity loss of a factor of 4
- Decreased background factor of 4
- Maintained S/N - increased overall sensitivity (not accounting for smaller area)
- Effective radiation filter

Design Modification

- Increase high energy throughput – decrease capillary diameter on both excitation and detection sides: loose some low energy throughput – longer optics165 mm excitation optic

- Decrease output fl of excitation to 30 mm; decrease input fl of detection to 30 mm

Design #2



Dual Capillary Setup

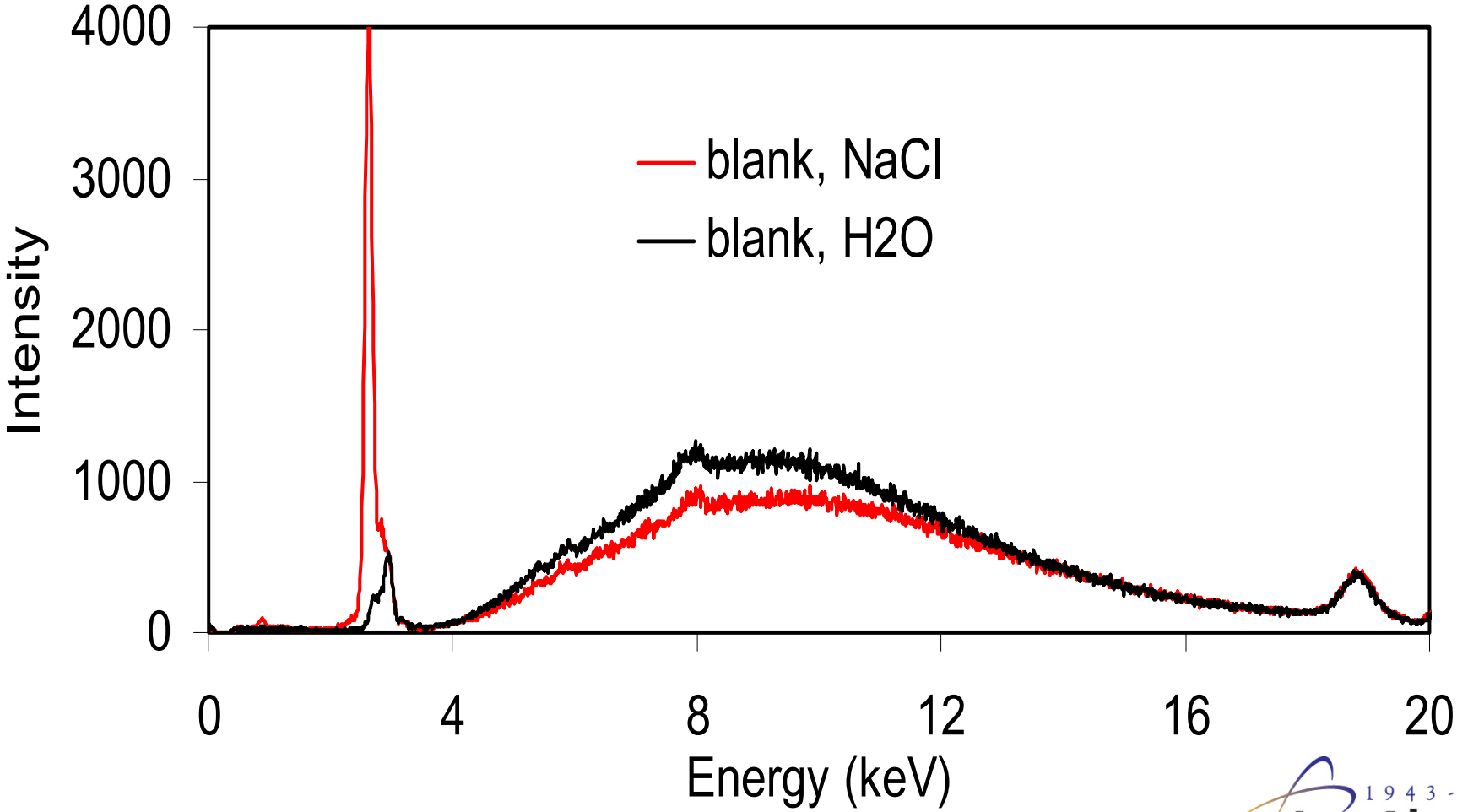
#2 Design Dual Capillary

- Primary – shielding of detector from radiation: need to measure elements in high radiation field
- Secondary – maintain sensitivity for elemental detection: high salt matrix 1M NaOH
  - Sr, Pb, Pt, Pd, Tc, Cu

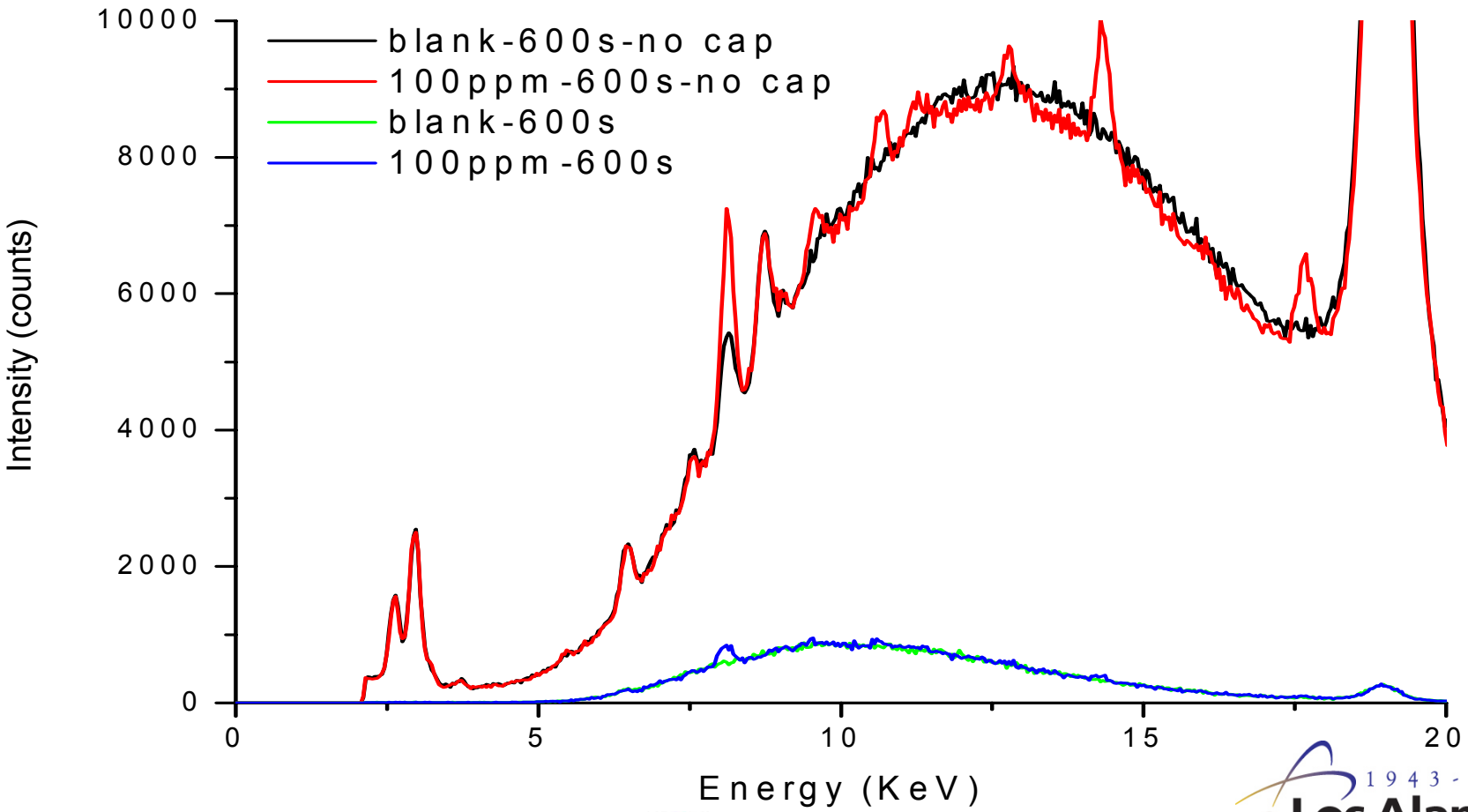
Analytes

- Sr, Pt, Pb, Pd, Mo, Cu
- Matrices
  - → H_2O
  - → 1 M NaCl – (1M NaOH)
- Sensitivity
- Background discrimination

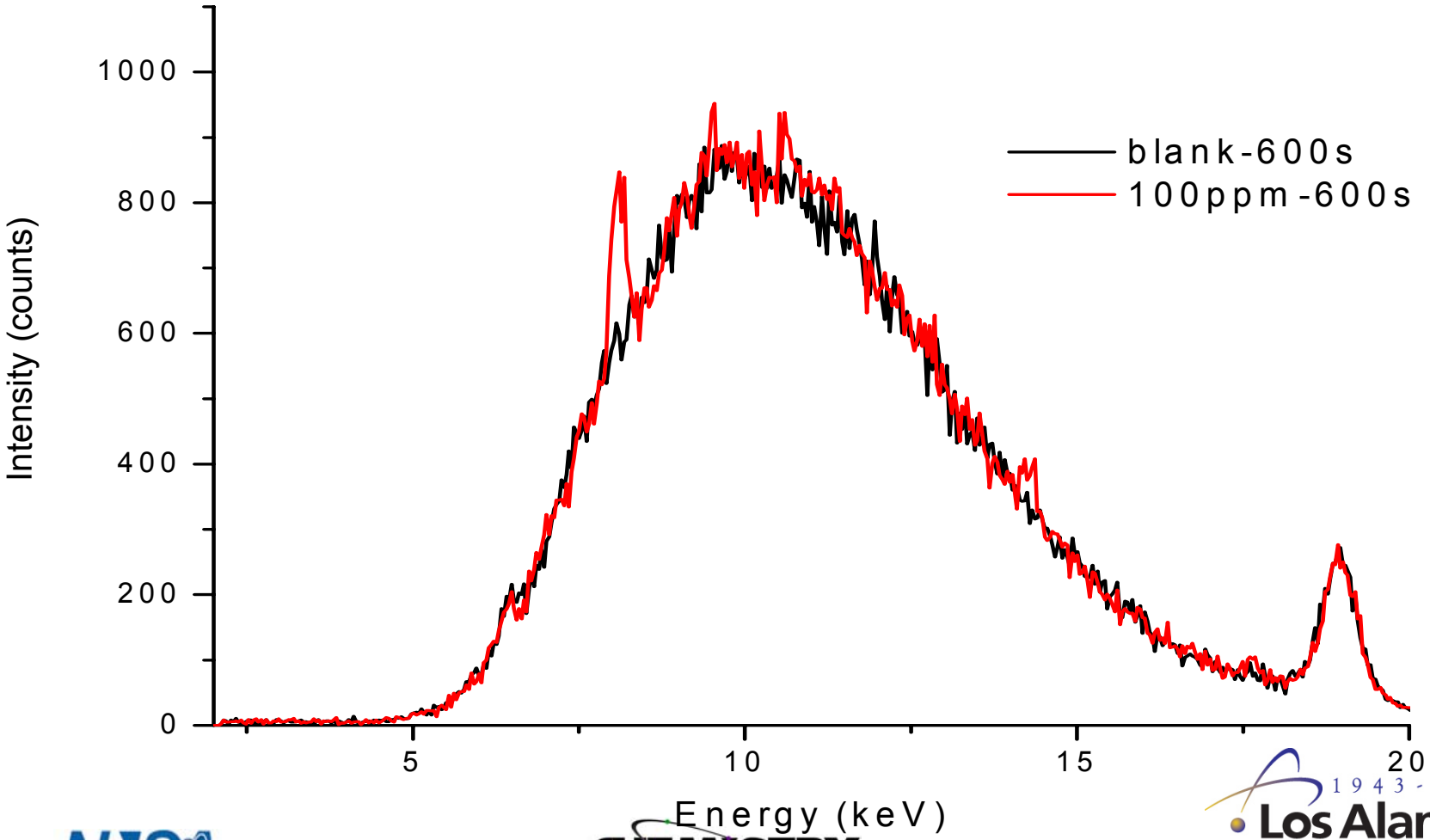
Matrix Comparisons



Spectra of 100 ppm 1 M Salt Matrix



100 ppm in 1M Salt Matrix



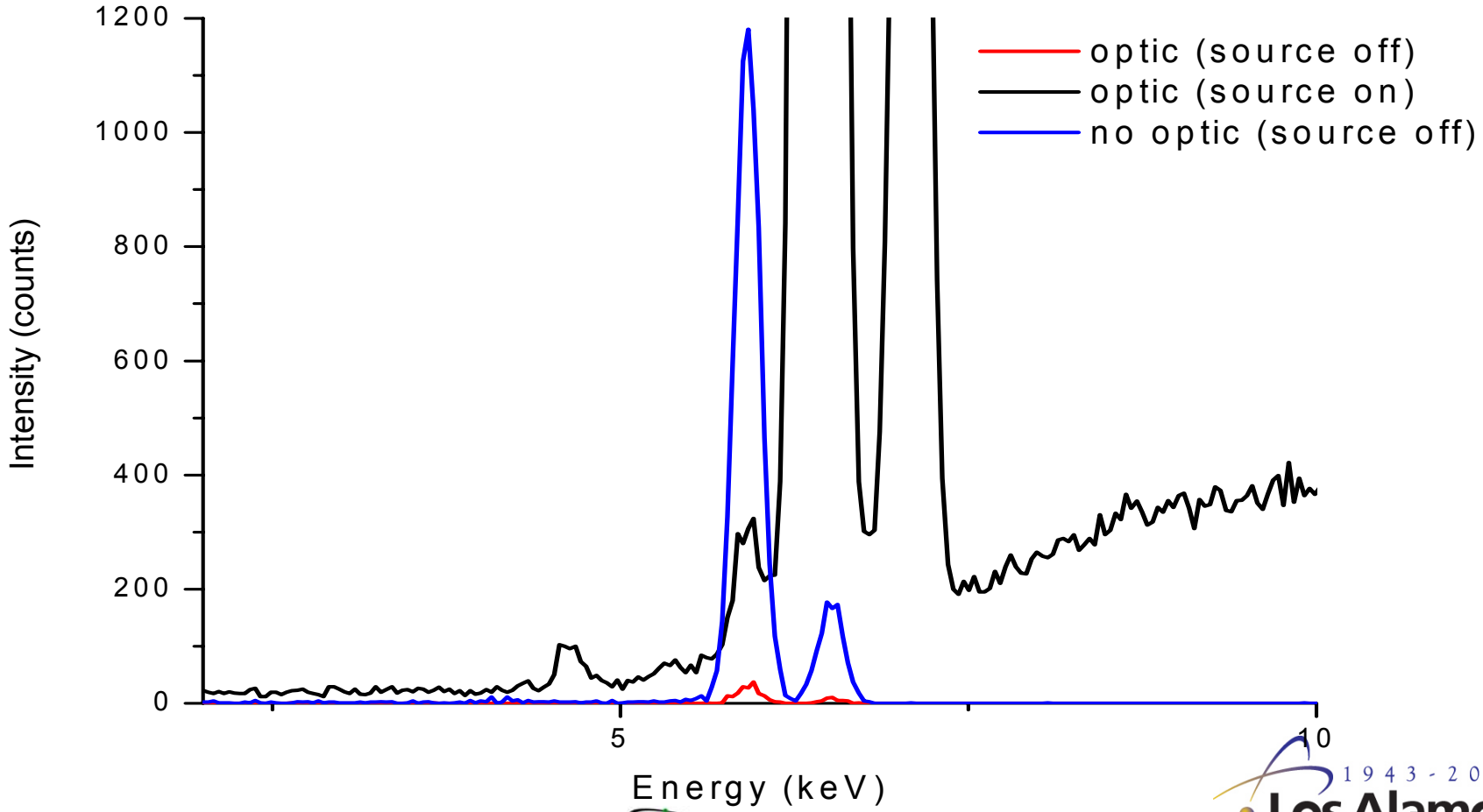
Comparison of Detection Limits

	Blank Optic	Blank No Optic	Sample Optic	Sample No Optic	Sample blank subtracted	Sample no optic blank subtracted	Optic LOD (ppm)	No Optic LOD (ppm)
Cu	599	5363	847	7247	248	1884	1	1
Pt	882	6527	952	7251	70	724	5	1
Pb	843	7949	938	8685	95	736	4	2
Sr	316	8266	408	10027	92	1761	2	1
Mo	80	5639	105	6587	25	948	4	1
Pd	18	4130	20	4012	2	-	26	-

Iron^{55} Radiation Background Reduction

- Objective is to reduce unwanted radiation on the detector
- Improve sensitivity
- Improve dynamic range of detector with radioactive materials

Fe 55 Source Excitation



Radiation Rejection for Fe^{55}

	Optic Source Off	Optic Source On	No Optic Source Off	No Optic Source On
Mn	31	303	1115	1575
Fe	5	18489	108	20757

Rad rejection factor of 20 with optic

Results

- Long focal length dual capillary can be an effective radiation shield – factor of 20 reduction
- Some loss of sensitivity due to lower solid collection angle
- Some increased sensitivity increase through background rejection and Compton and Rayleigh rejection
- Improved high energy throughput

Cutaway View of Prototype

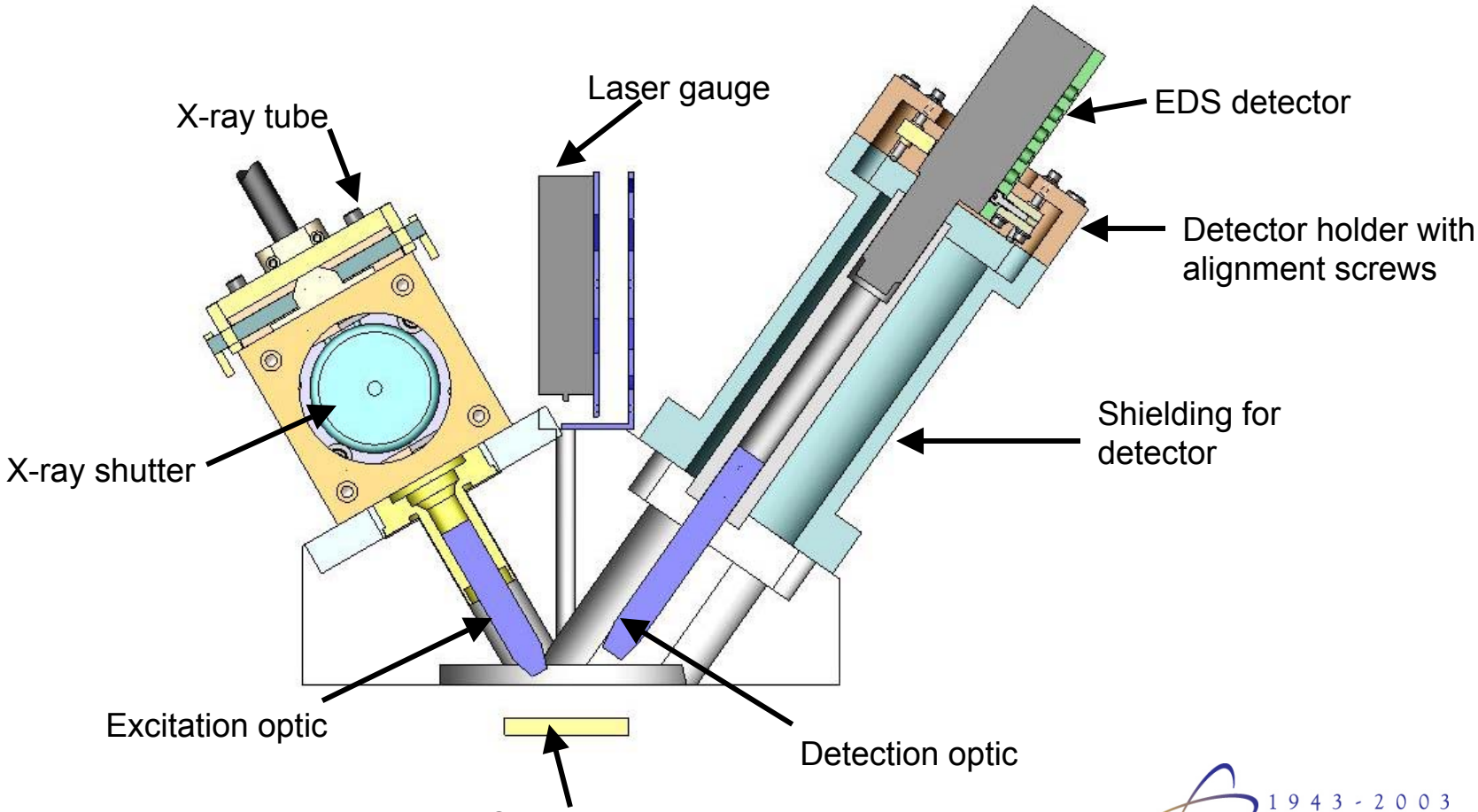
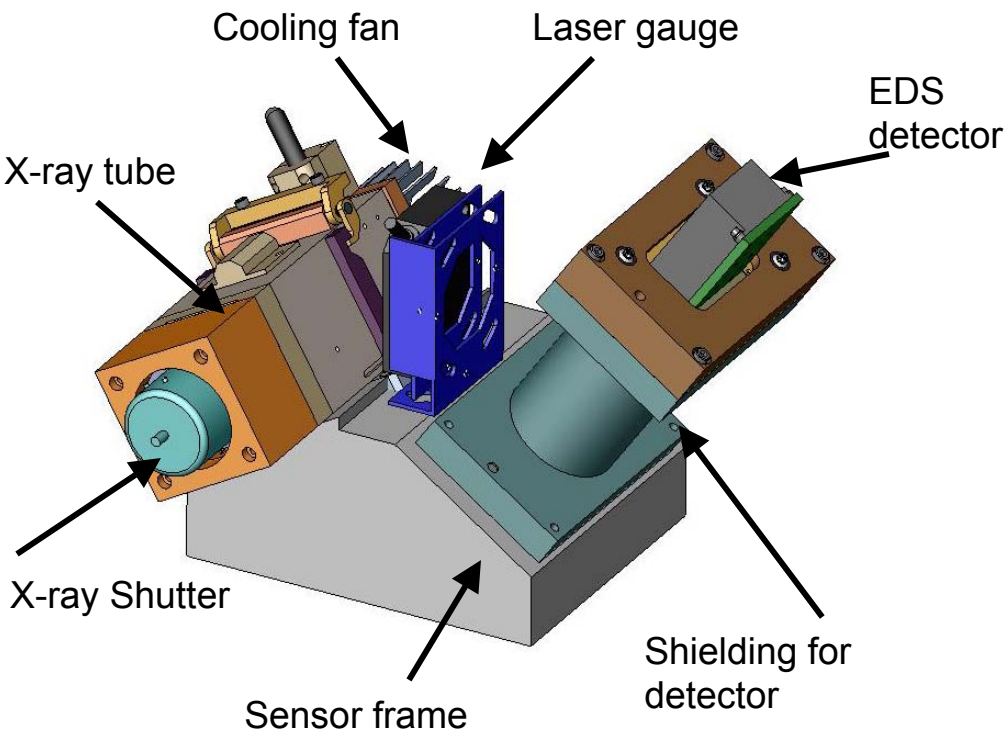
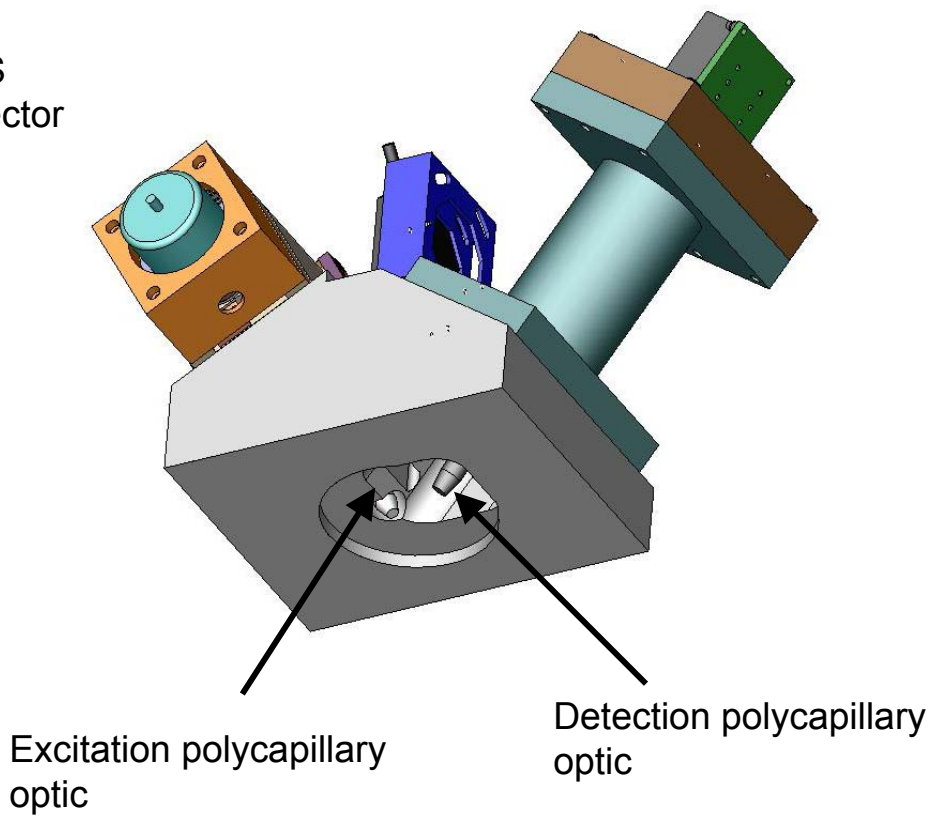
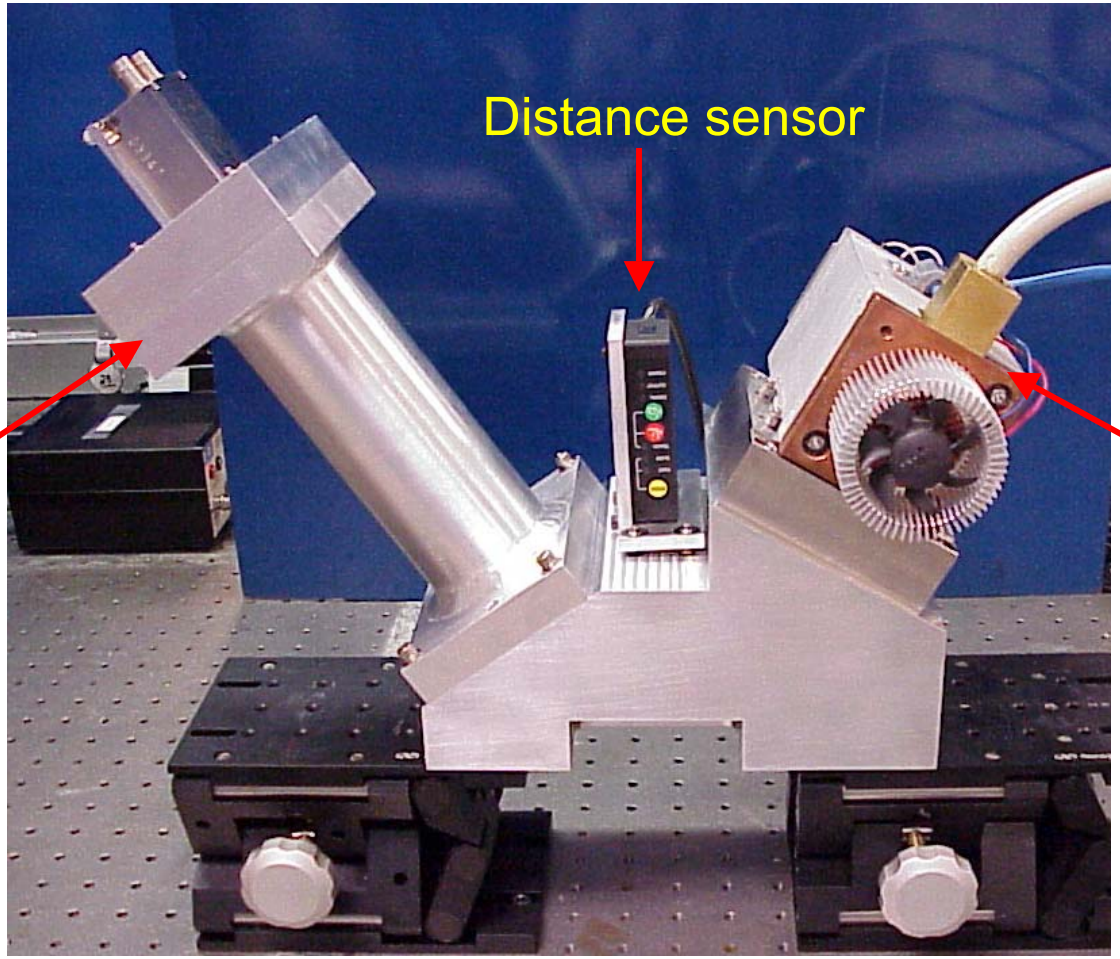


Diagram of Prototype





Picture of Prototype



EDS detector with polycapillary optic

X-beam with polycapillary optic

Timeline

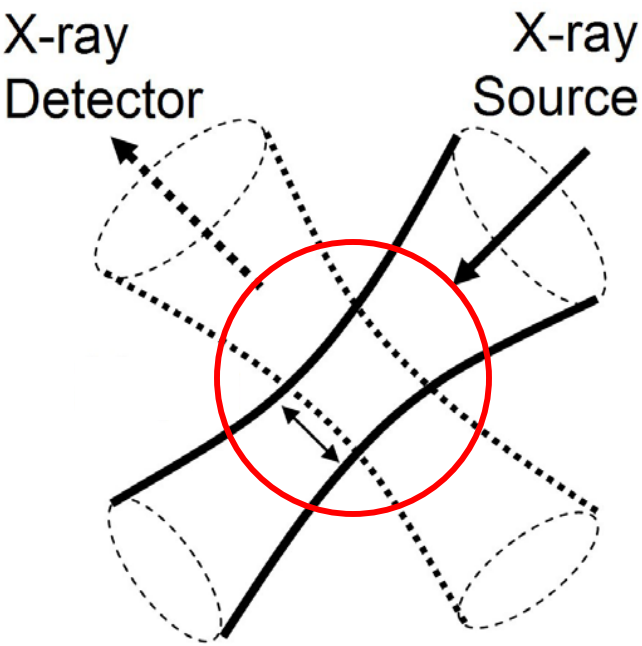
- On target – building prototype, expected delivery August 03
- Testing and evaluation of prototype at LANL 1st & 2nd quarter FY'04
- Potential site testing at SRS or INEEL – funding issues for site support and logistics to keep instrument cold

Relevance

- Increased speed and sensitivity of elemental characterization - what? and how good?
  - ➔input waste characterization for clean-up process
  - ➔output waste for process monitoring and effectiveness
  - ➔reduced cost for sampling, rad protection, sample prep, sample treatment
  - ➔increased safety
  - ➔improved operations overall

Unexpected Benefit – Confocal MXRF

Confocal MXRF



- 3D elemental mapping
- Layered material probe
- Nondestructive
- Potential for speciation via **lab based** EXAFS and XANES

EMSP
Environmental Management Science Program

X-Ray Optical Systems, Albany, NY

- Dr. Ning Gao - Research Scientist
- Mr. David Gibson - President
- Design, fabrication, testing and application investigation of X-ray optics
- Forefront of X-ray optic technology
  - ➔novel air particulate sampling and analysis
  - ➔DCC development for elemental speciation **without** SR radiation