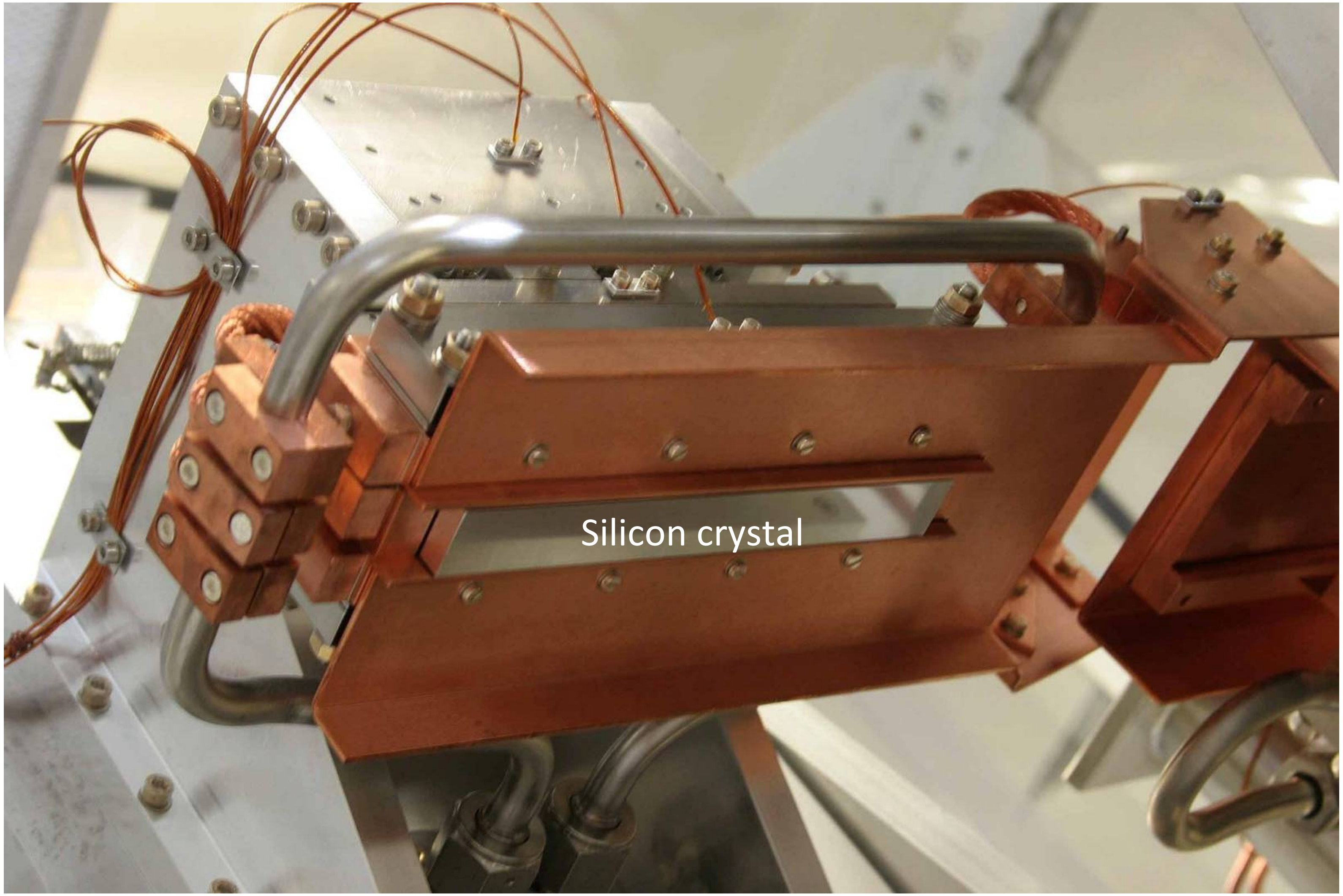
Cryogenic cooling of monochromator crystal

LN_2 cooling (Diamond)



Omitted topics

Electron analyzer

Crystal/multilayer analyzer

Ultra-fast detectors

Timing instruments

Detectors

Ionization chamber, solid state detectors (Silicon, Germanium…)

Detectors used in XAS experiments

Beam monitor

A. Ionization chamber (standard beam monitor in hard x-ray region)

a. Ambient gas pressure type

Control of absorbance: Mixing gas (N_2-He, N_2-Ar)

b. Pressurized gas type

Control of absorbance: Low vacuum, high vacuum

F detector

c. Lytle detector

Fluorescence detection where energy resolution is not required

B. Solid state detectors (SSD) Energy resolution: 135-220 eV @5.9 keV

Silicon (Li) Pure Ge Upto 100 pixels; Oyanagi, NIM A513, 340 (2003).

C. Silicon drift diode (SDD) Energy resolution: 130 eV @5.9 keV

Upto five elements (?)

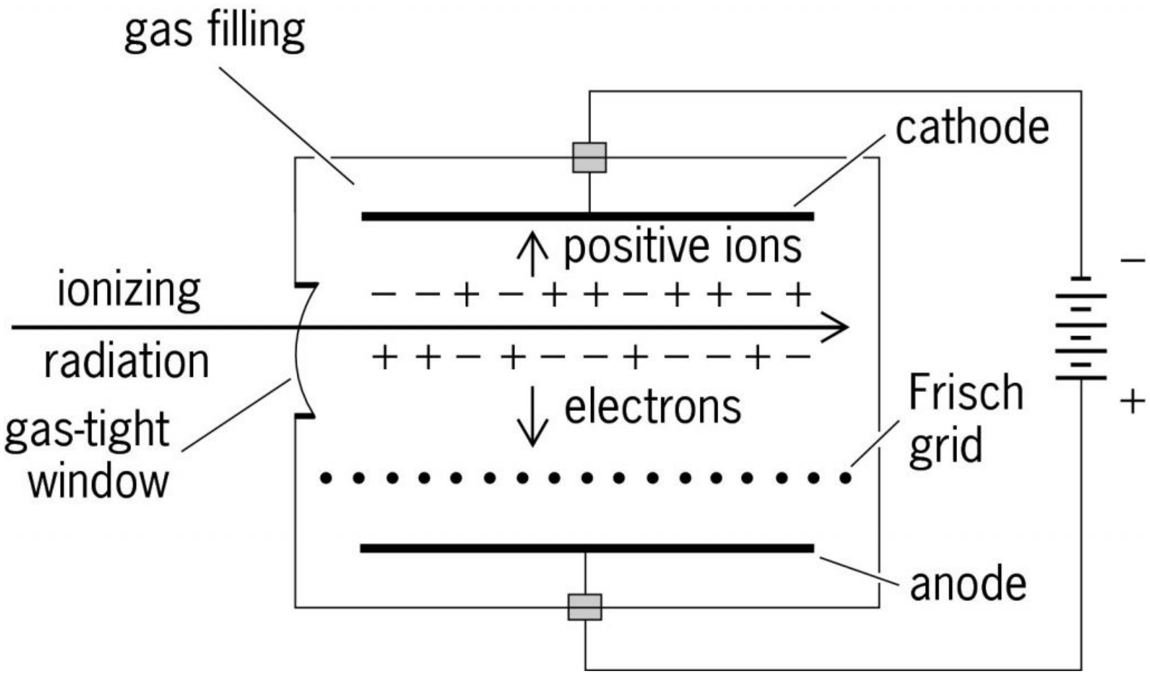
D. Si APD Fast response detector Energy resolution ≈26%

Kishimoto, RSI 63, 824 (1992)

E. Scintillation detector, NaI, plastic Energy resolution (NaI) ≈46%

F. Multilayer monochromator

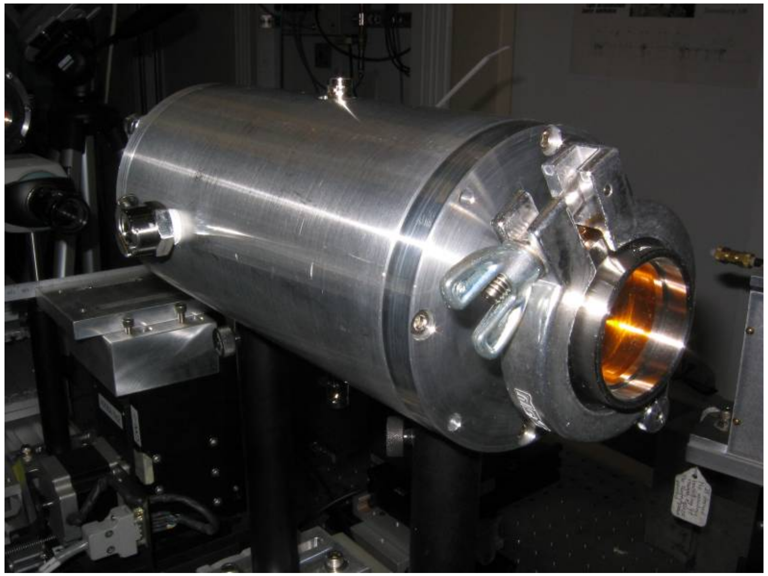
Ionization chamber -Principle



Note: frequent spike noise comes from a discharge
Low voltage is recommended

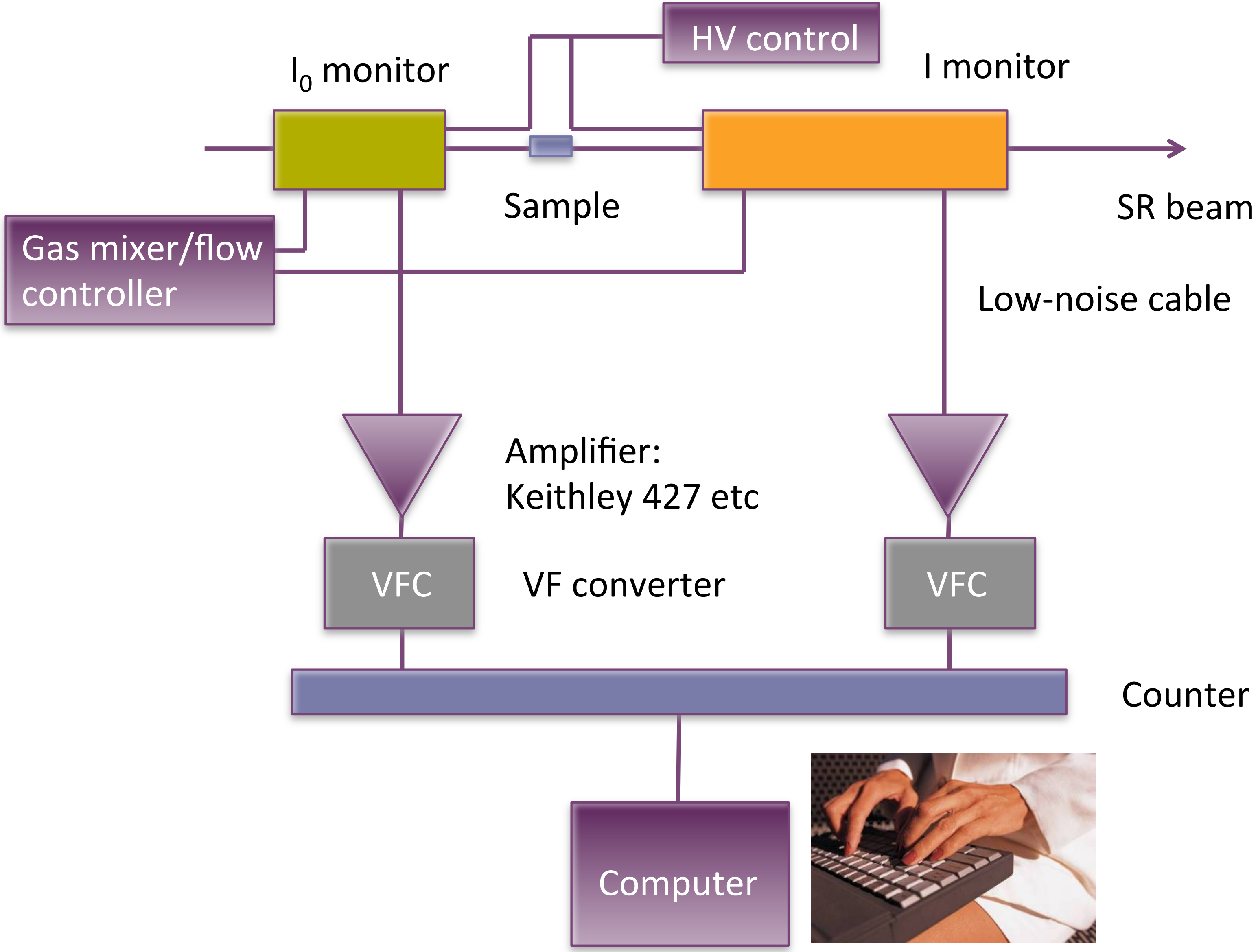
Two types of ionization chamber

@PF



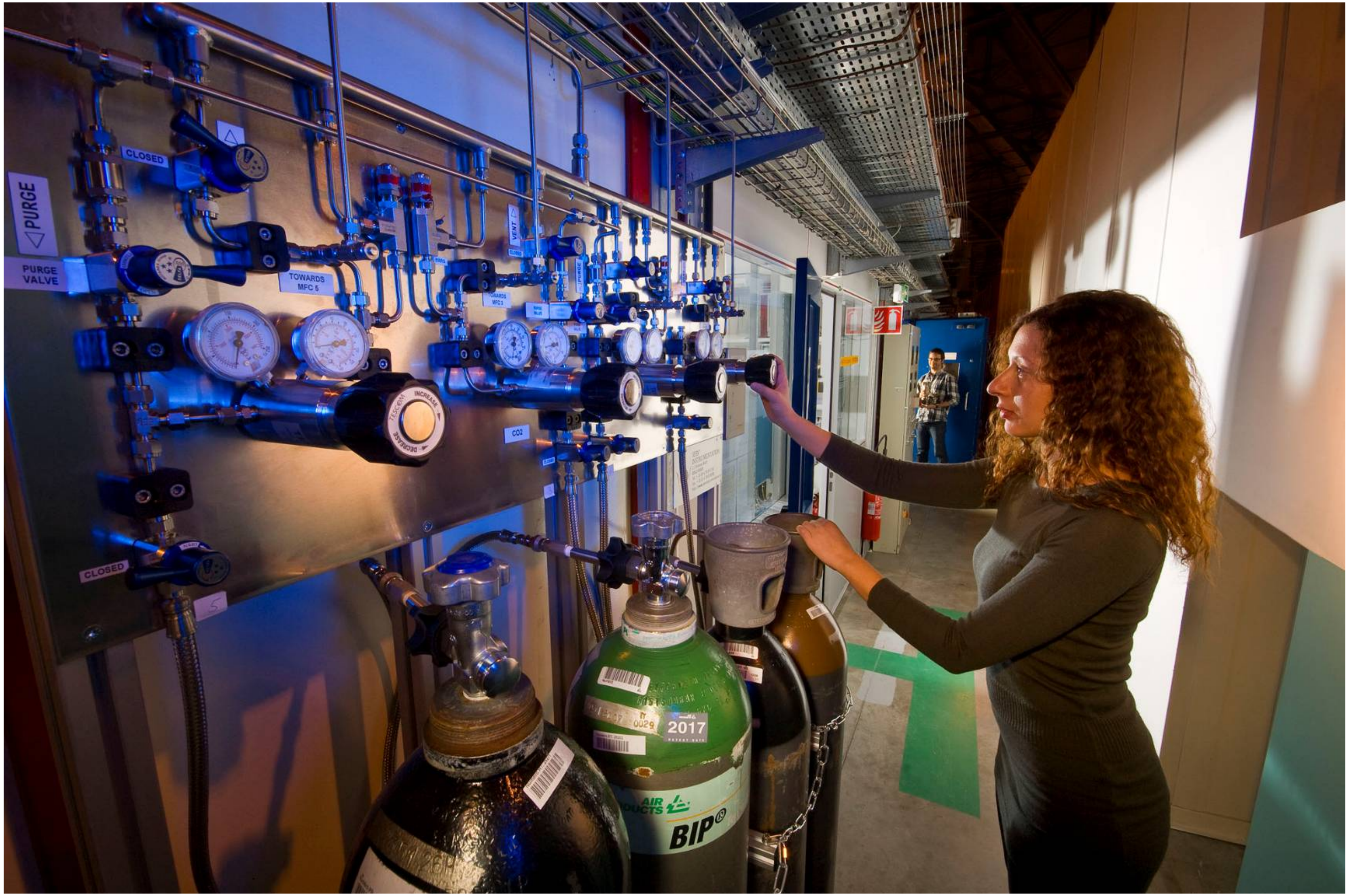
@CLS

Conventional setup (Analog amplifier)

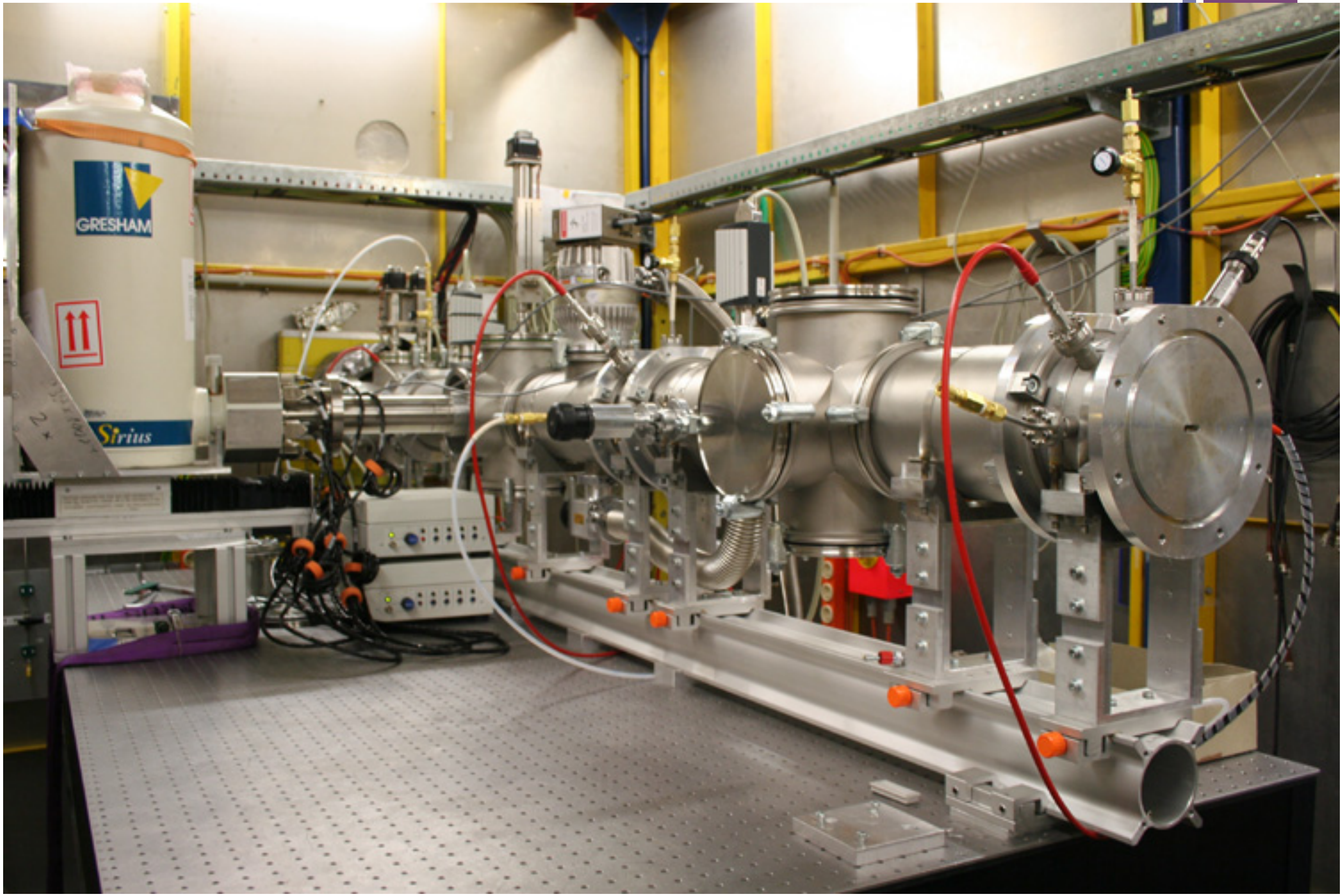


+ Ionnization gas control

Controlled gas flow makes a stable signal output



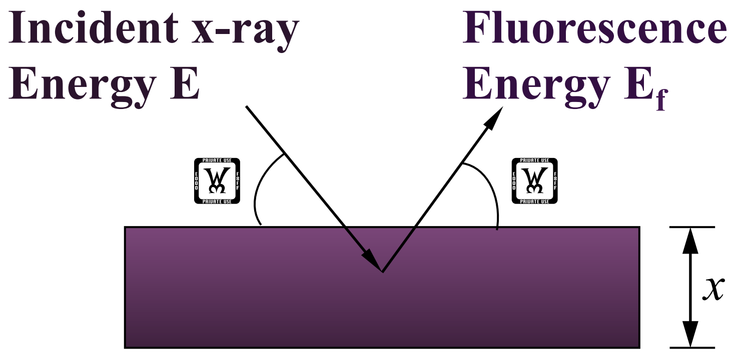
@ESRF



Pressurized ionization chamber setup at APS

Fluorescence intensity estimation

For x-rays incident on a slab of sample with thickness of x:



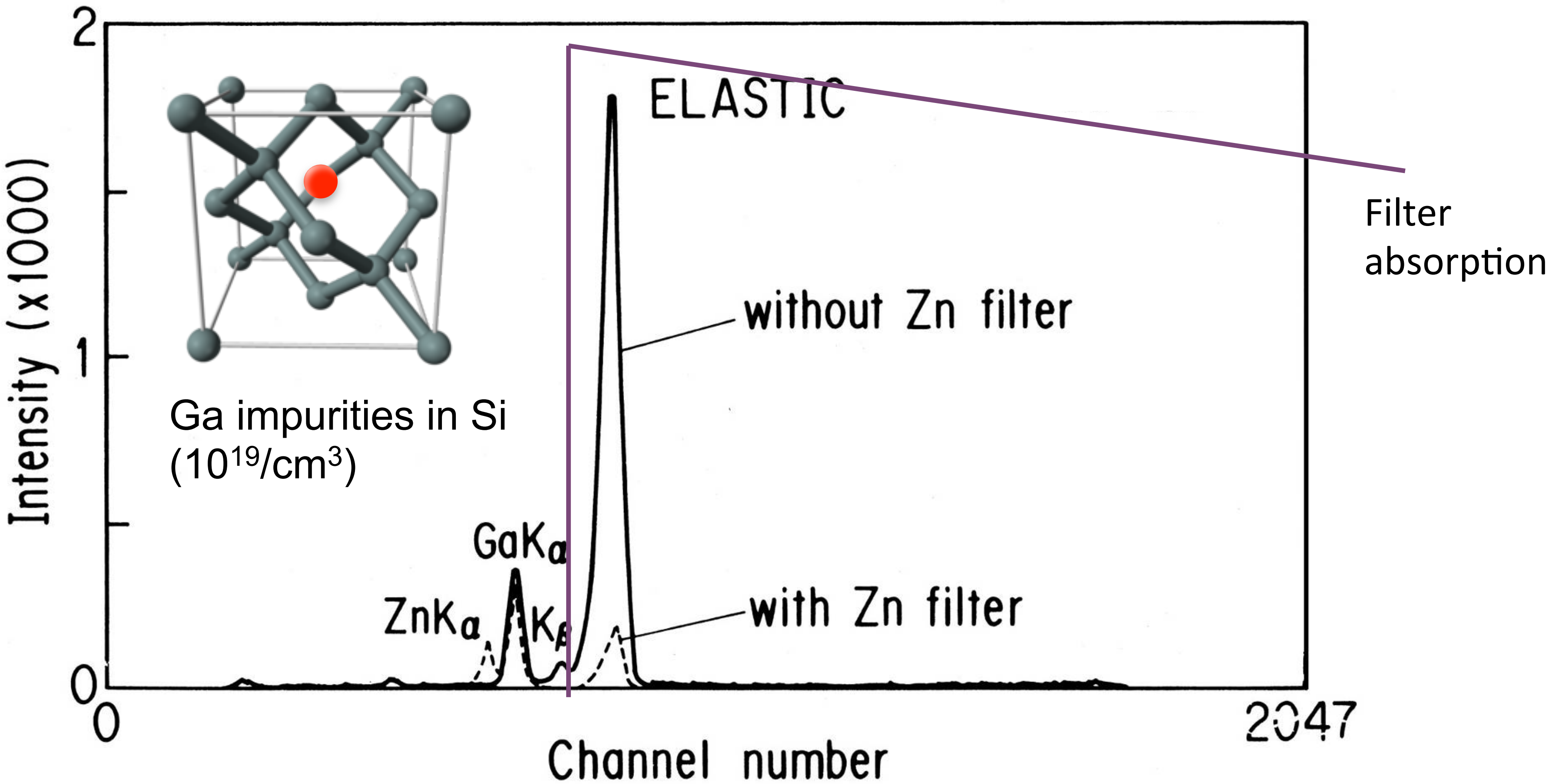
The fluorescence intensity $I_f(E)$ accepted by a detector with a solid angle of $\Omega/4\pi$ is:

$$I_f(E) = I_0(\Omega/4\pi)\varepsilon\mu_{Se}(E)/\sin\Theta \frac{1-\exp[-(\mu(E)/\sin\Theta+\mu(E_f)/\sin\Phi)x]}{\mu(E)/\sin\Theta+\mu(E_f)/\sin\Phi} \quad (1)$$

$I_f(E) \approx I_0 \times 8.8 \times 10^{-4} \times 0.012 \approx 1 \times 10^{-5} I_0$, lower than I_0 by 5 orders.

Fluorescence detection technique (Z-1 filter)

With a sacrifice of ½ intensity reduction, one order of magnitude
Of elastic x-ray background is removed by a fluorescence filter



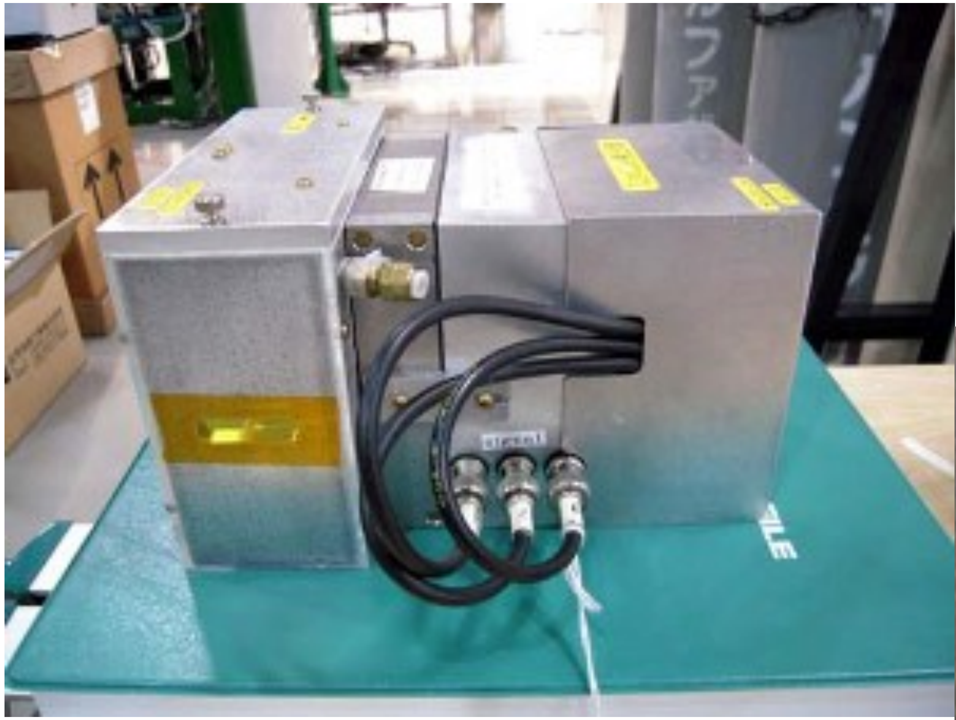
Lytle detector

Basically ionization chamber

Soller slit assembly to remove scattering and fluorescence background

www.exafsco.com/

@SagaLS



@APS

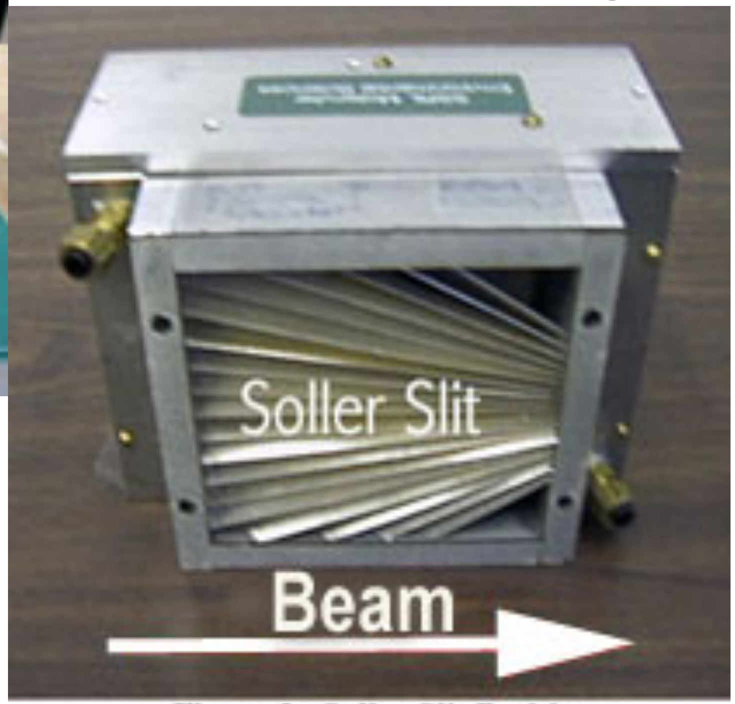


Figure 2. Soller Slit Position

Note: easy-to-use and low-cost fluorescence detector

Note: never use in multi fluorescence signal sample

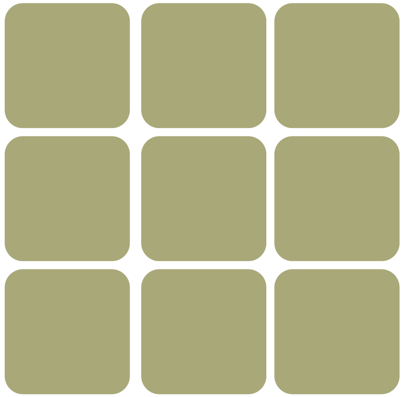
NaI array

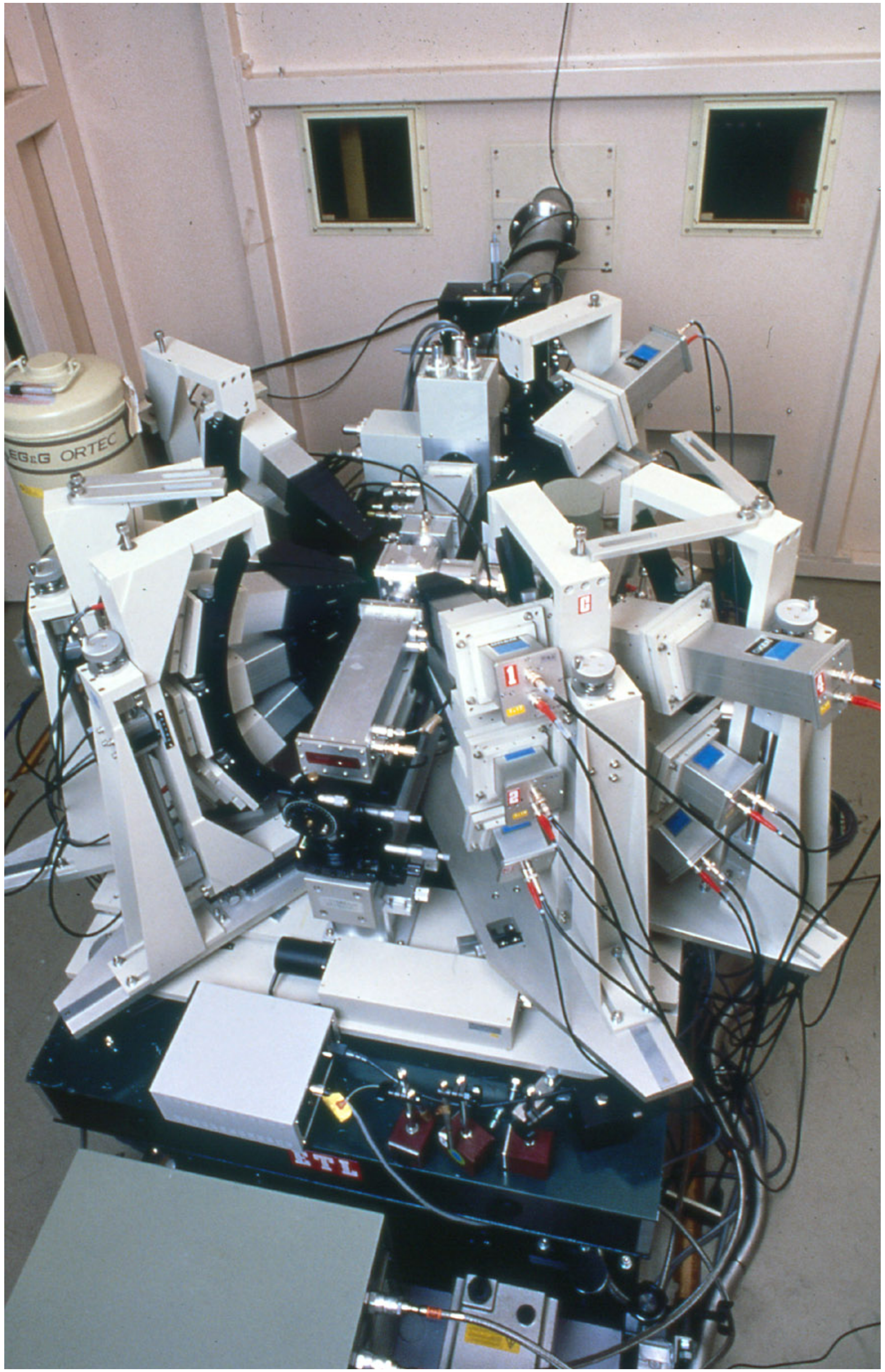
For simple case, i.e., a single fluorescence line, energy resolving power becomes unimportant

Lytle detector
Si APD (DE≈26%)
NaI array (40-50%)

High coverage of solid angle but …
Low energy resolution

18% of 4p
DE=41%E





Focused beamline with a NaI array
BL4C, 10C@PF

SSD (Solid State Detector) -a CMOS device for x-rays

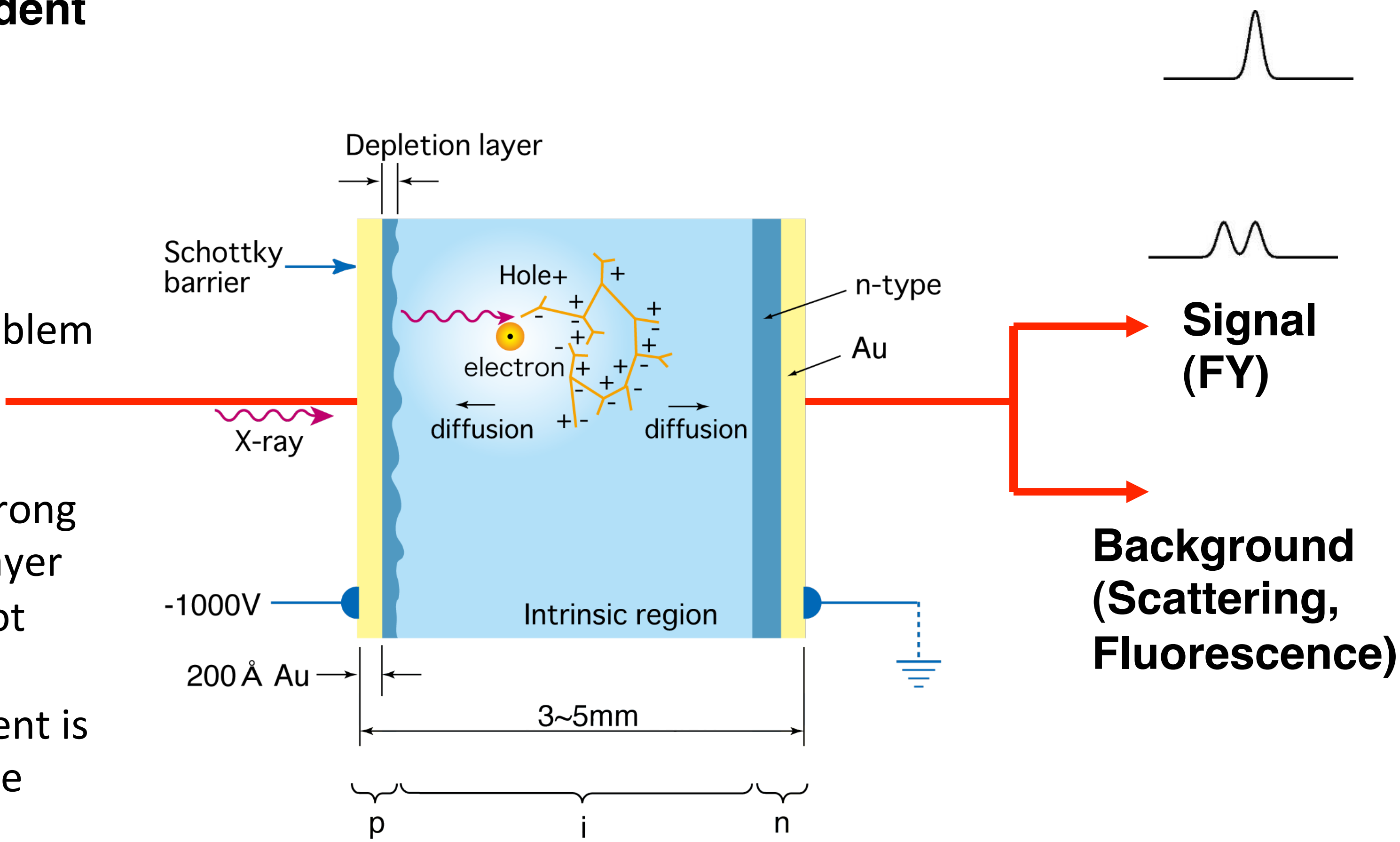
Each pixel acts as an independent SSD device

Signal (fluorescence) must be separated from elastic scattering And other backgrounds

Technical Problem

SiO_2 forms strong passivation layer but GeO_2 is not strong

Leakage current is A noise source

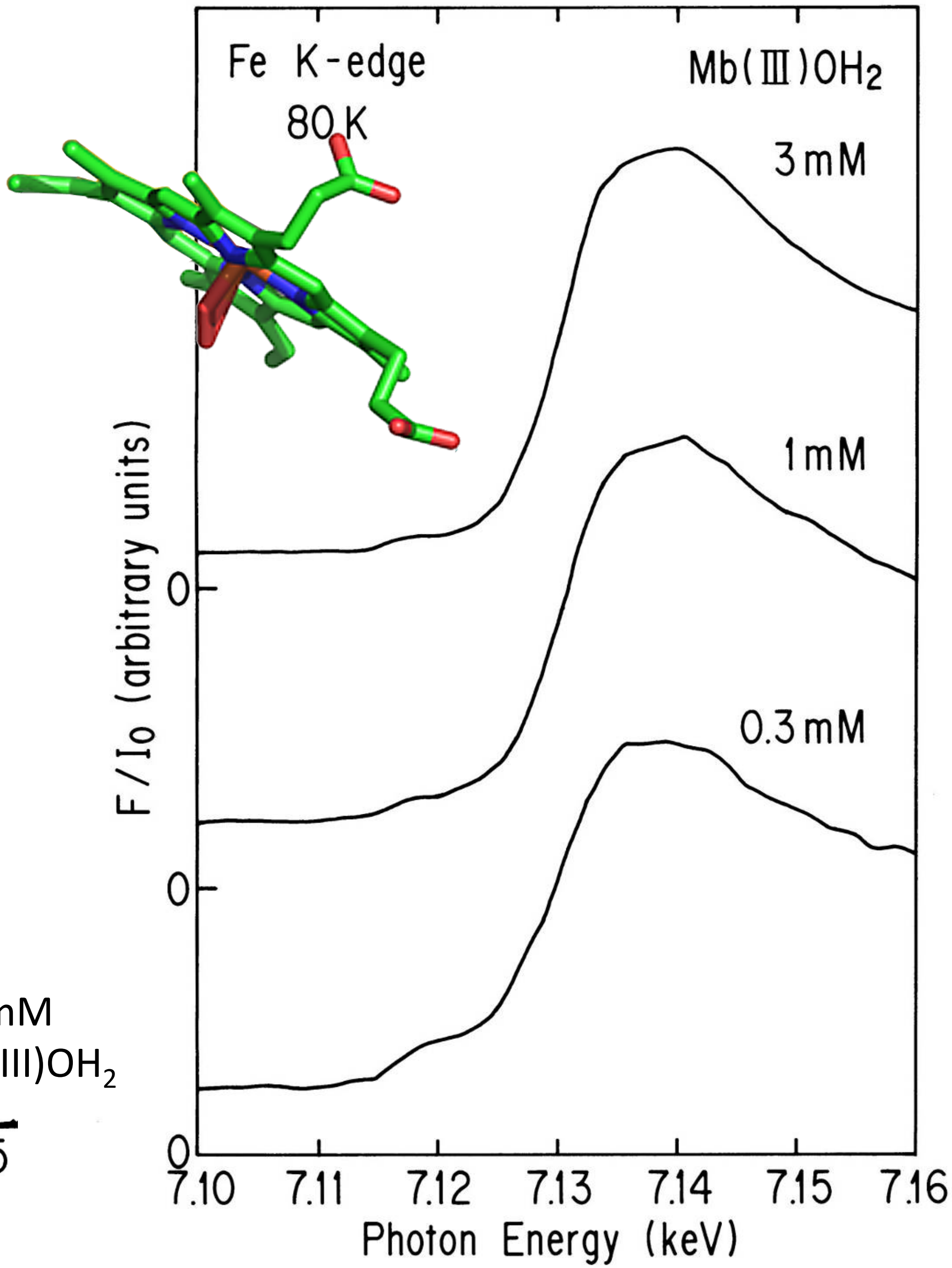


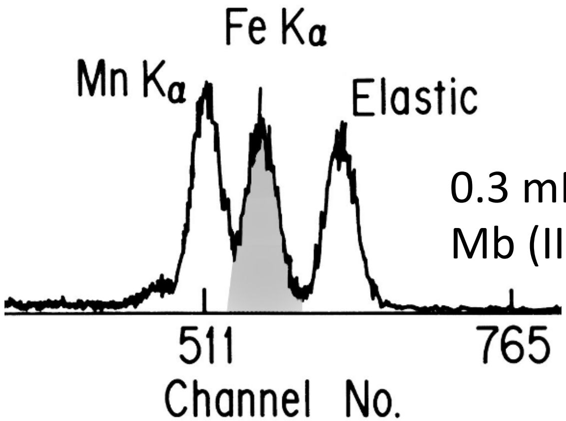
+ Fe K-XANES

Sample: Mb(III)OH_2

Signal: Fe fluorescence
Background: Filter fluorescence plus elastic scattering

Statistics: Proportional to the square root of the accumulated number of photons



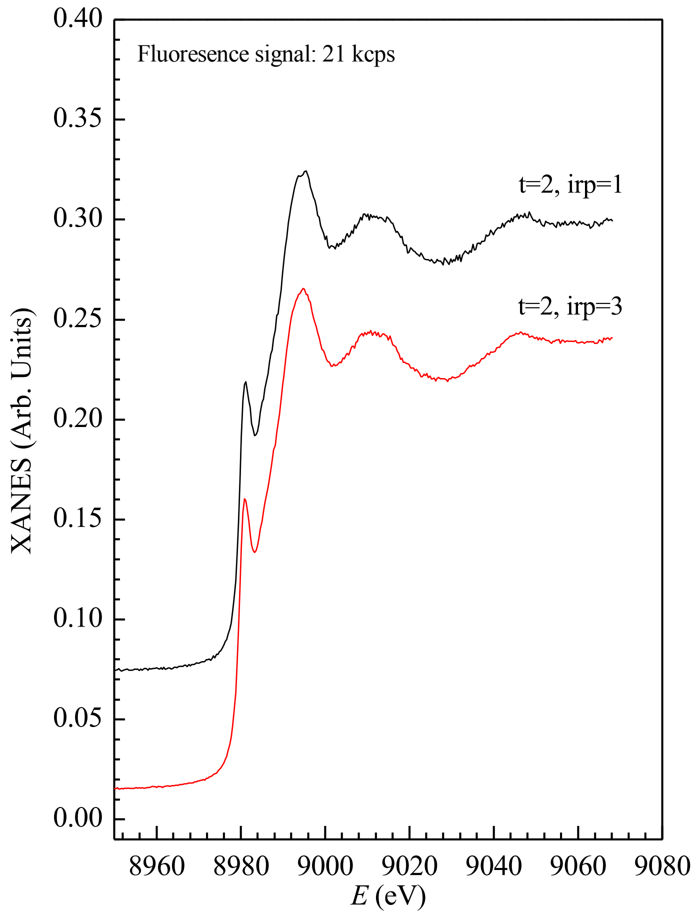


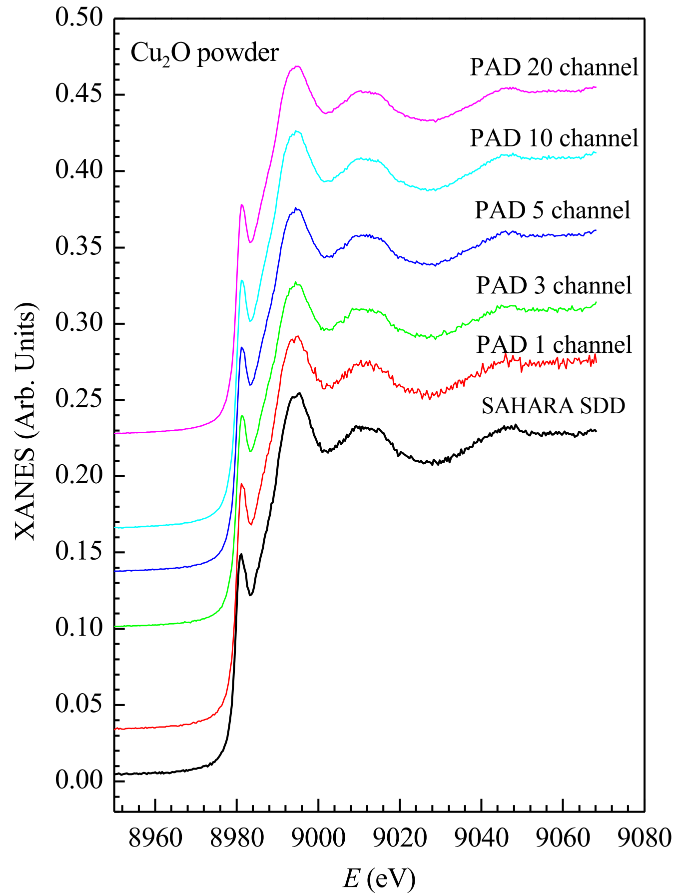
0.3 mM
Mb (III)OH_2

Statistics in fluorescence XAFS

a. Repeating scans

b. Segmented detection





+ Evolution of pixel array detector

1st Generation

5 mm x 5 mm
100 pixels
215 eV@5.9keV

No. 1 Spring-8 (Oyanagi)

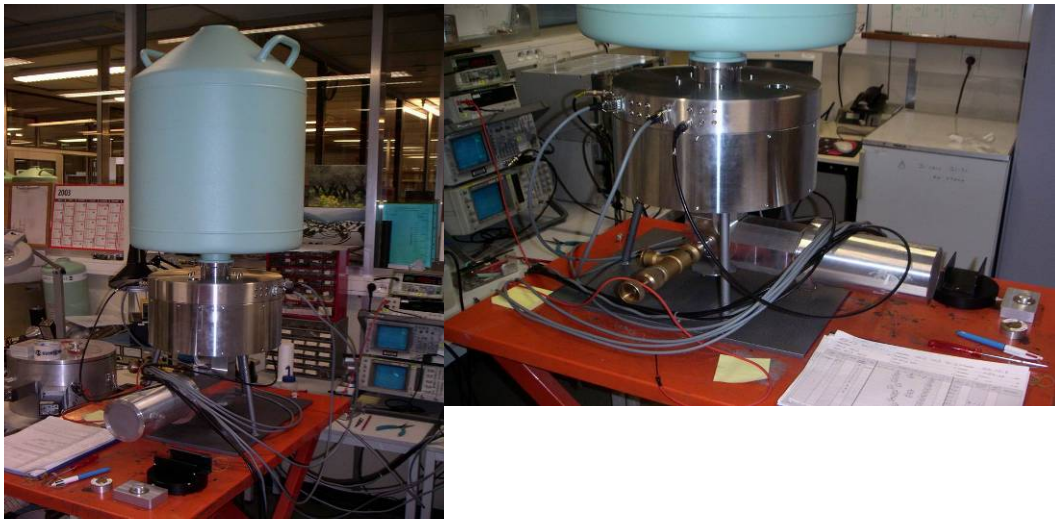
No. 2 Photon Factory (Oyanagi)

2nd Generation

5 mm x 5 mm
36 pixels

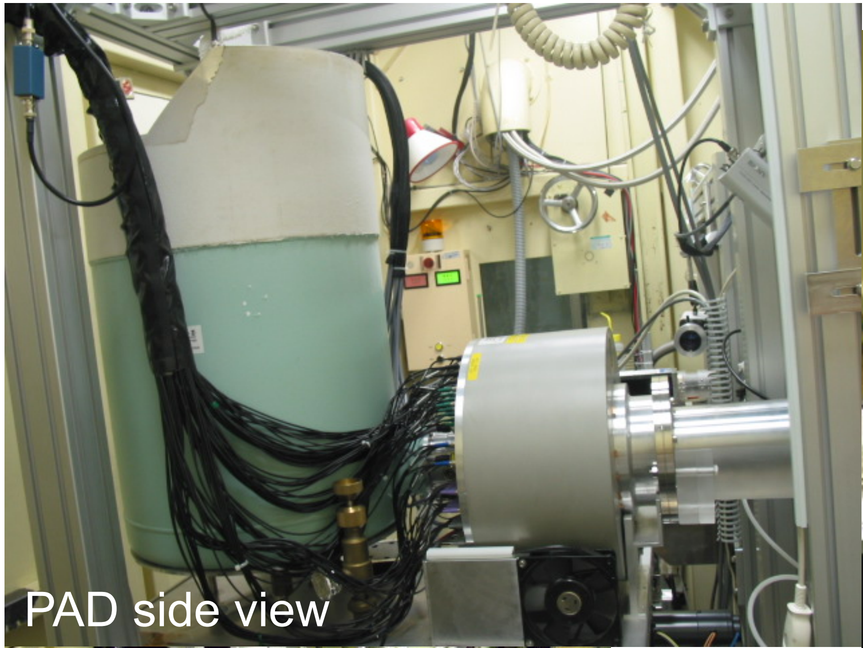
165 eV@5.9keV

No. 3 Australian Beamline at PF (Foran et al.)



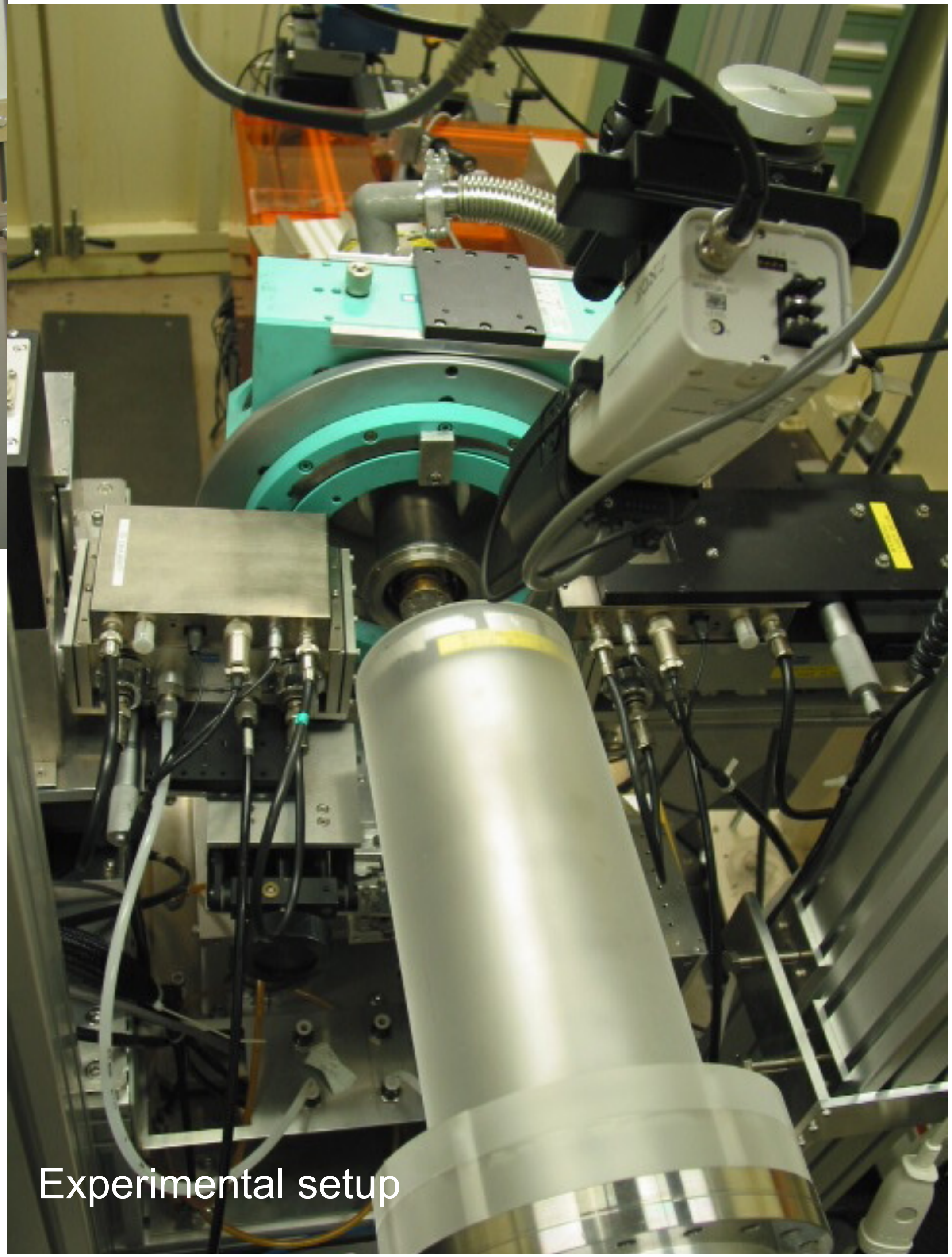
3rd Generation

Now in operation in Australian light source



PAD side view

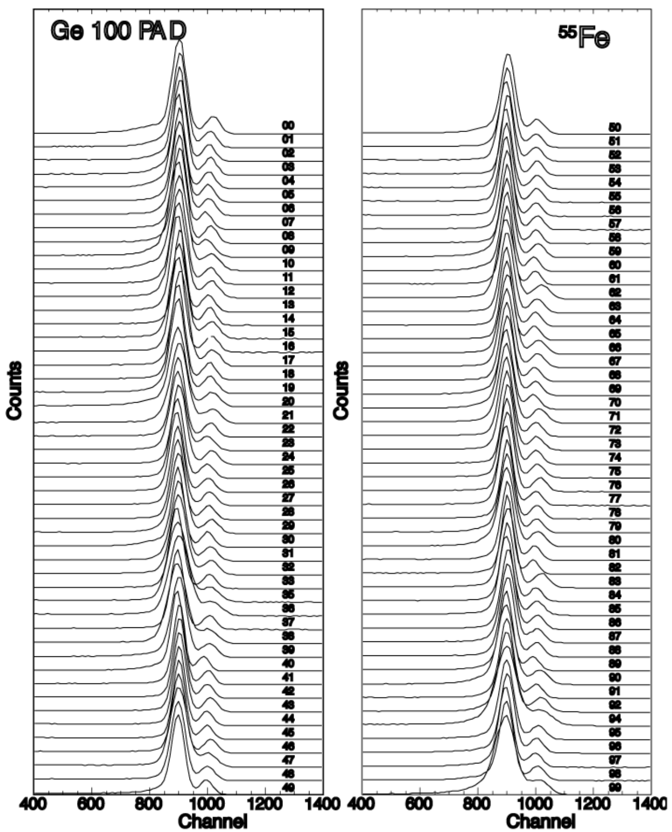
PAD front view



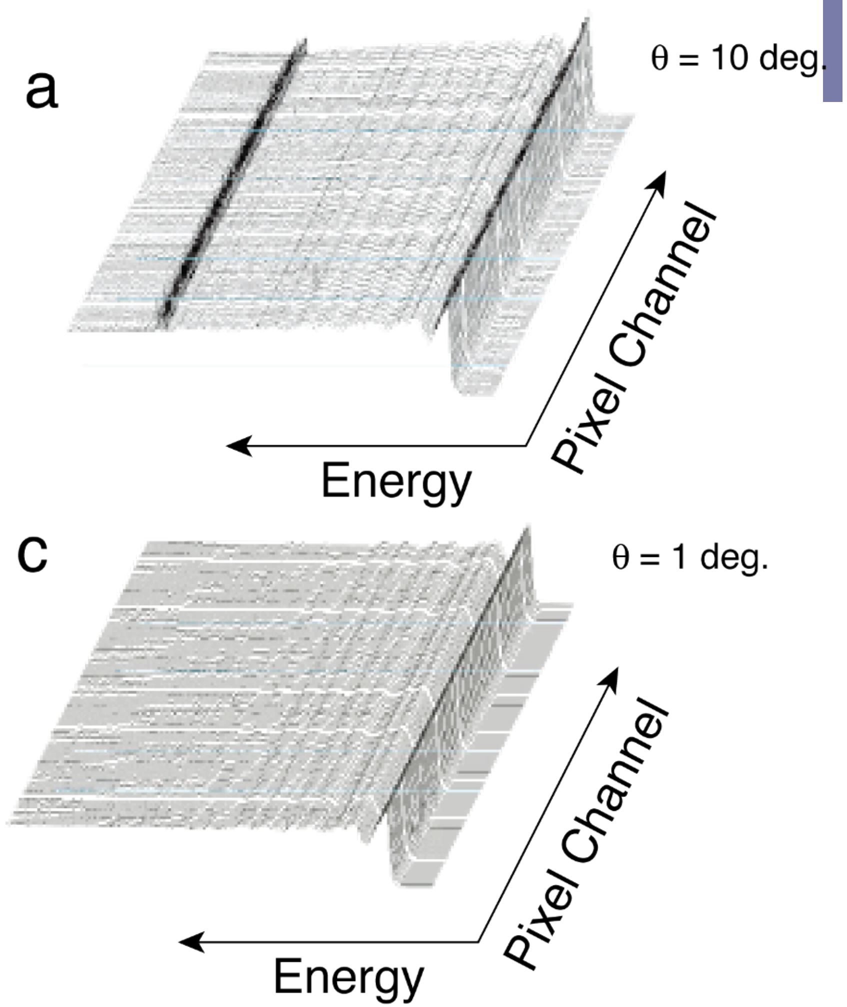
Experimental setup

Advantage of PAD

Real time fluorescence monitoring over a large segmented solid angle

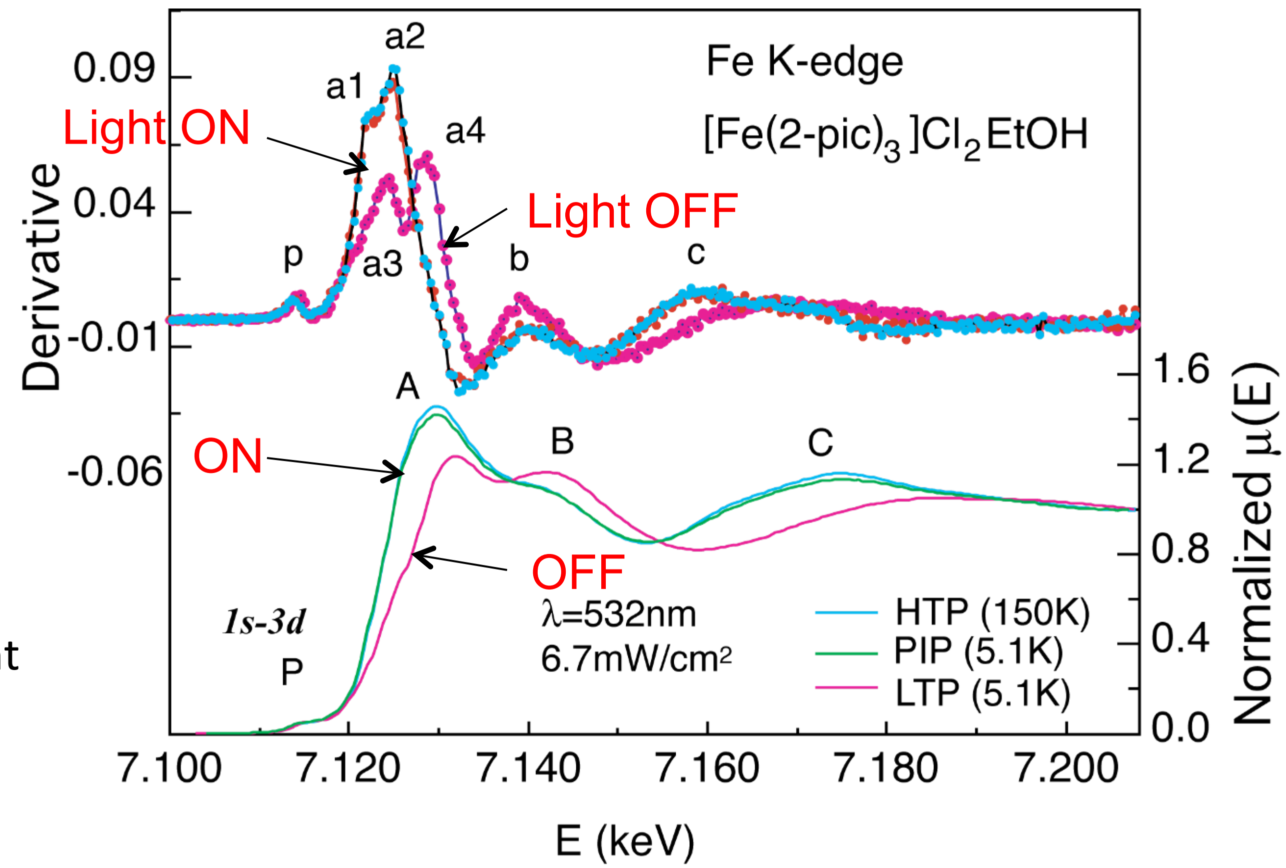


MCA output for 100 pixels



Glitches (scattering, standing wave, other elements) are completely removed.

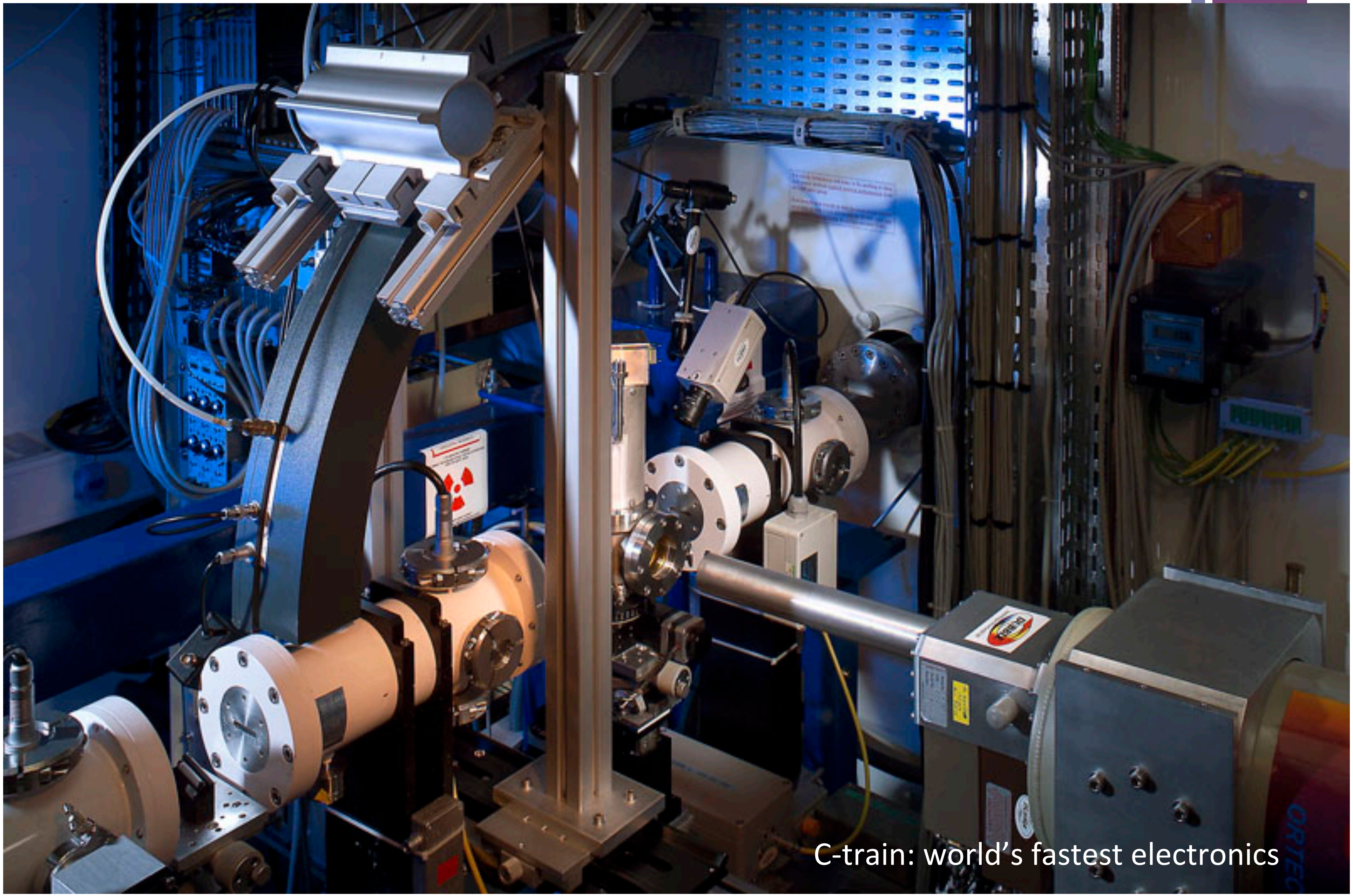
Photo-induced spin crossover –fluorescence detection by Ge PAD



Sample weight <1 mg

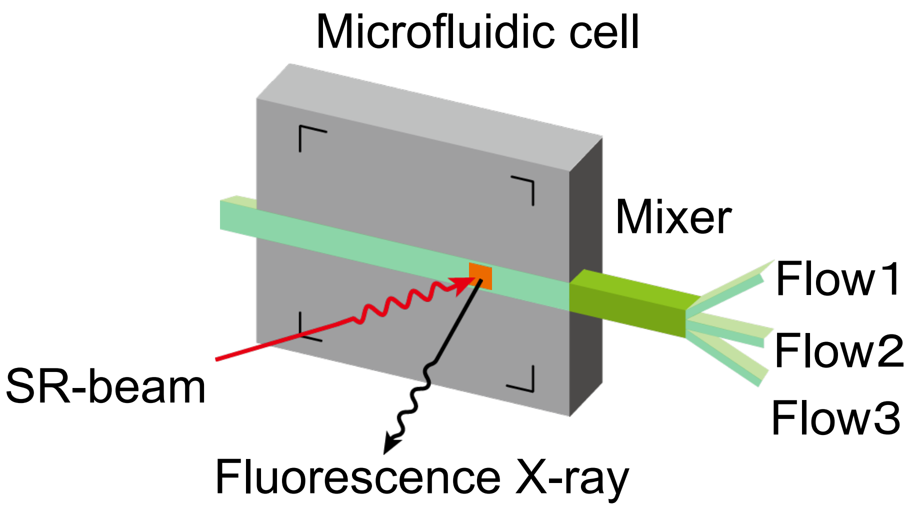
Laser illumination changes Fe spin state from S=0 to S=2
No symmetry breaking!

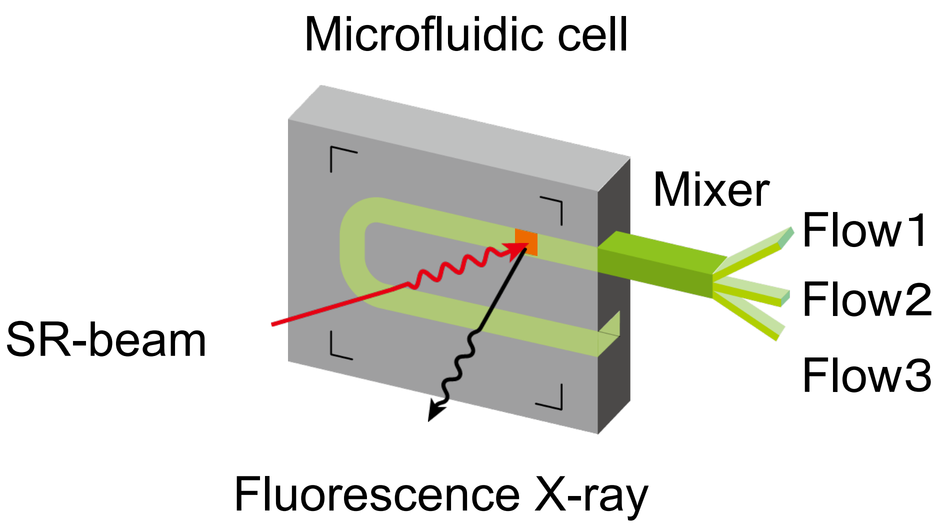
Oyanagi et al., J. of Luminescence 119, 361 (2006).



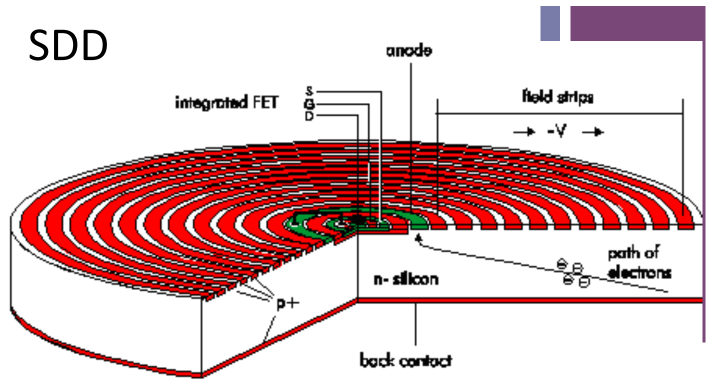
Vacuum-tight ion chambers and a fast SSD @ESRF

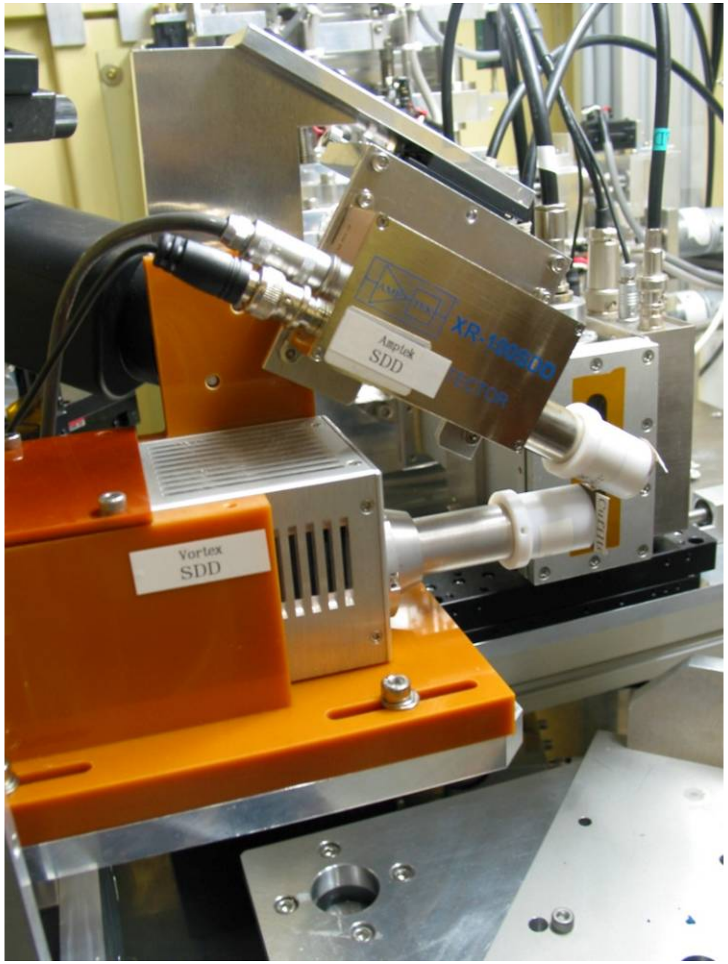
XAFS experiment using aicrofluidic cell

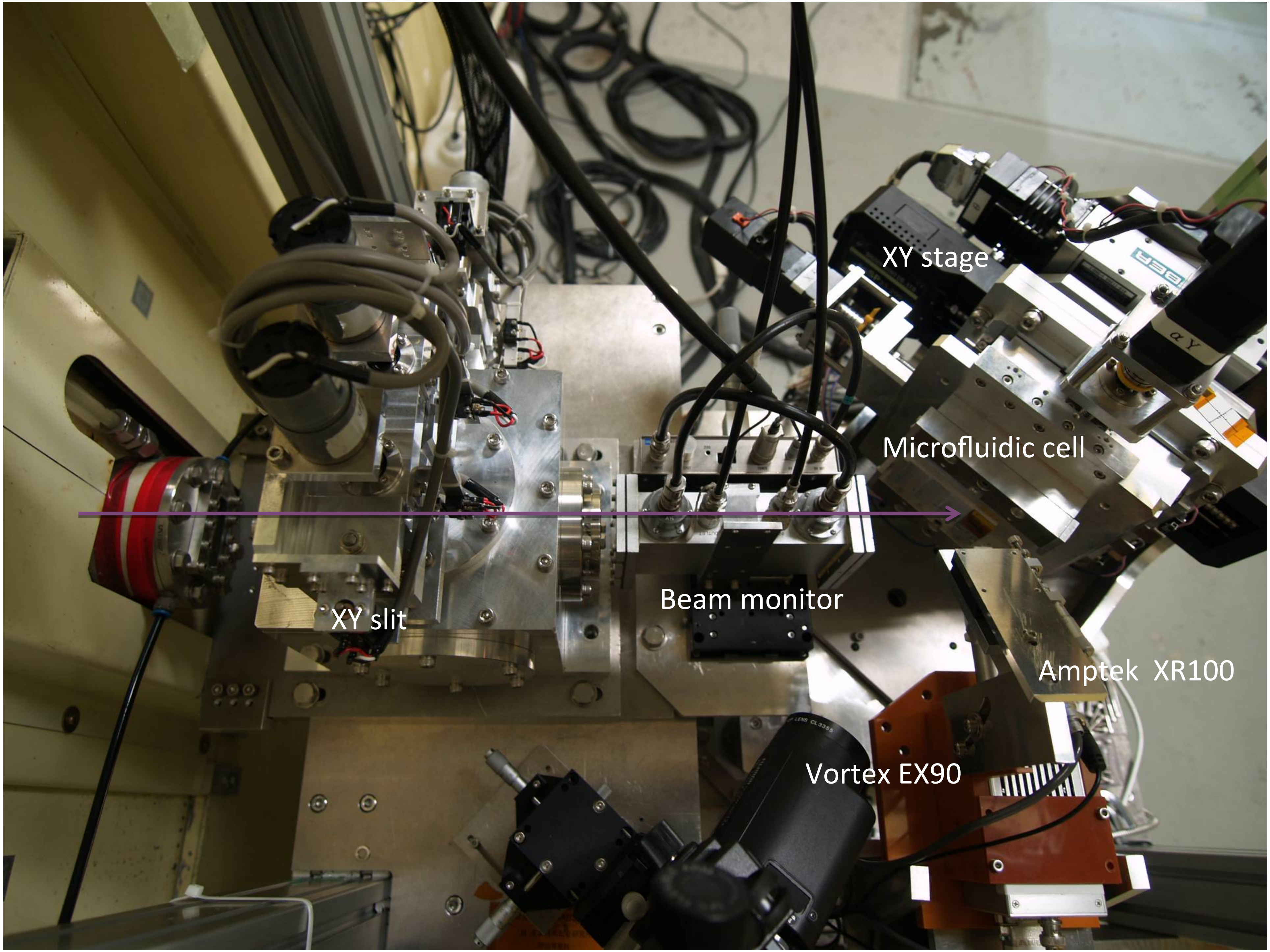




SDD






XY stage
Microfluidic cell
Beam monitor
XY slit
Amptek XR100
Vortex EX90

+

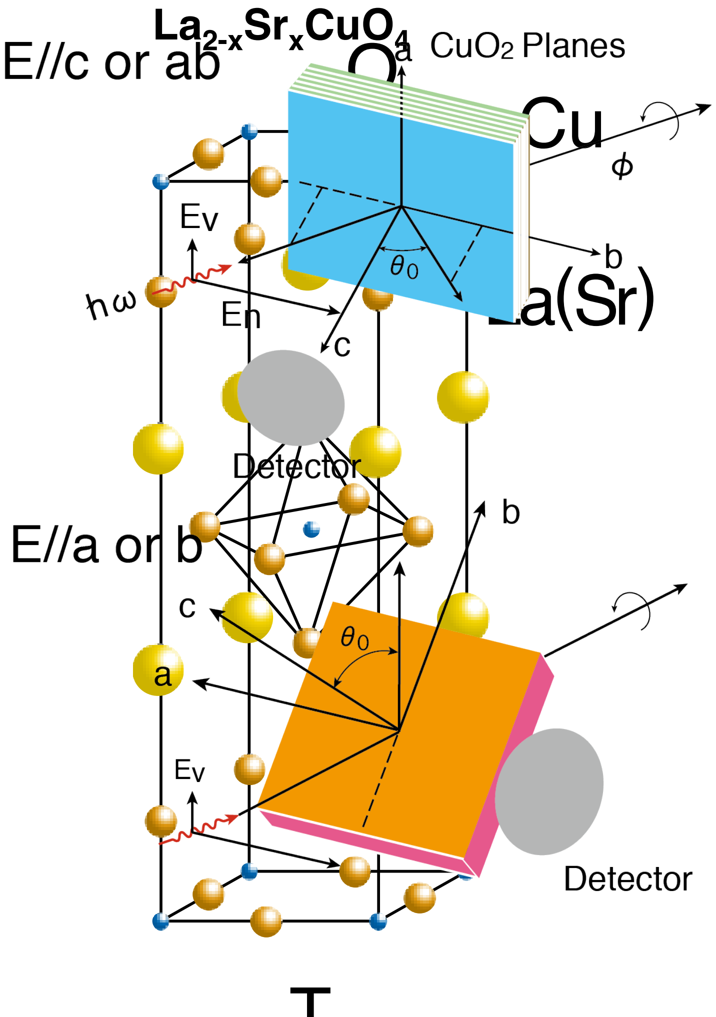
Omitted topics

- Energy dispersive EXAFS
- Quick scan
- Pump & probe

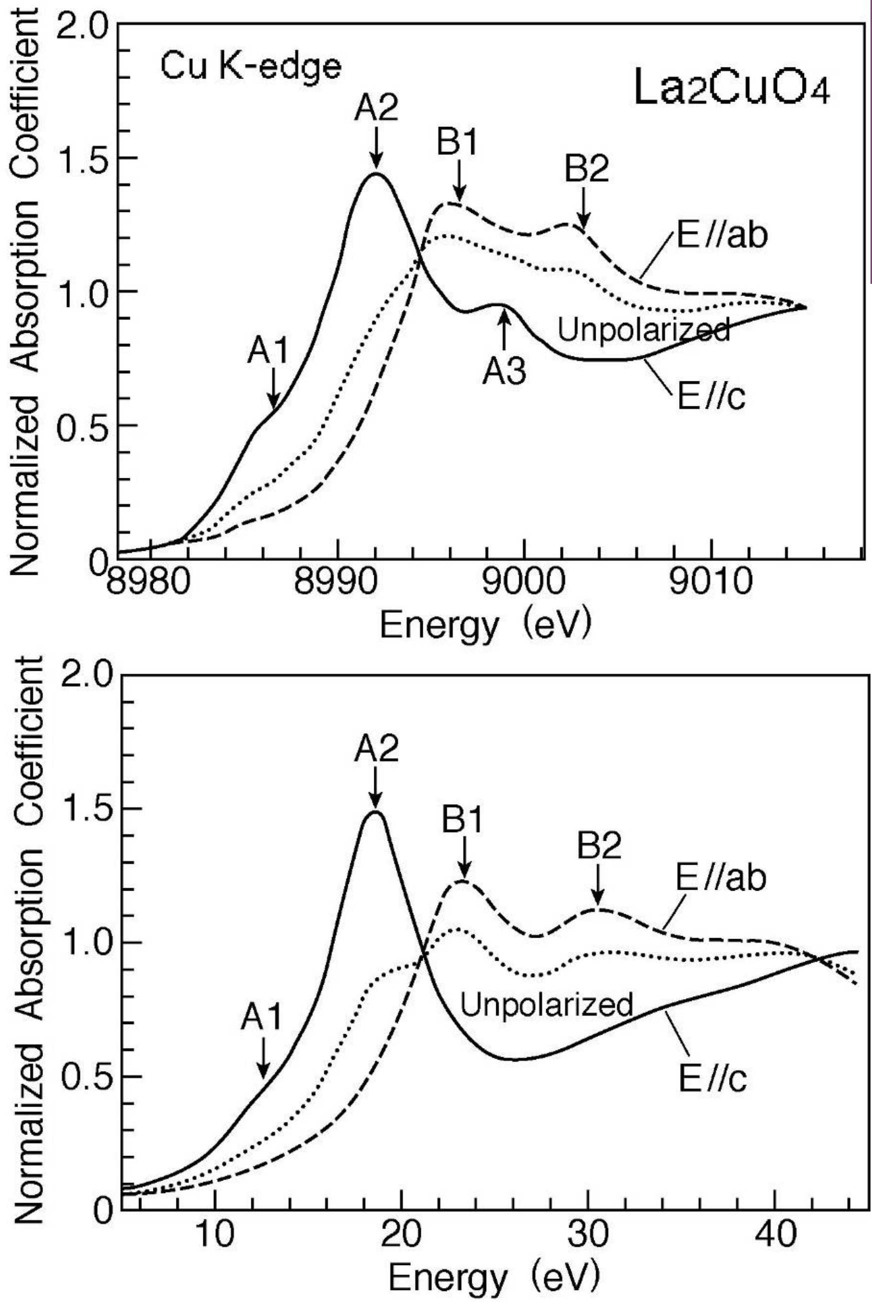
Measurement modes geometry

Transmission and fluorescence

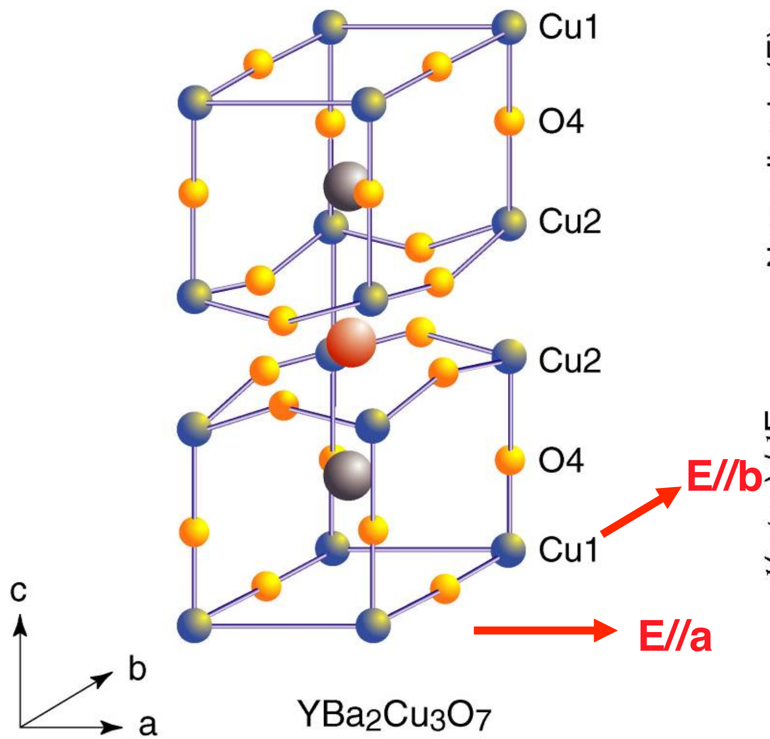
+ Geometries

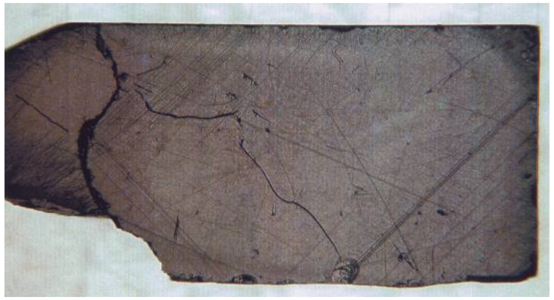


Generalized single crystal orientation

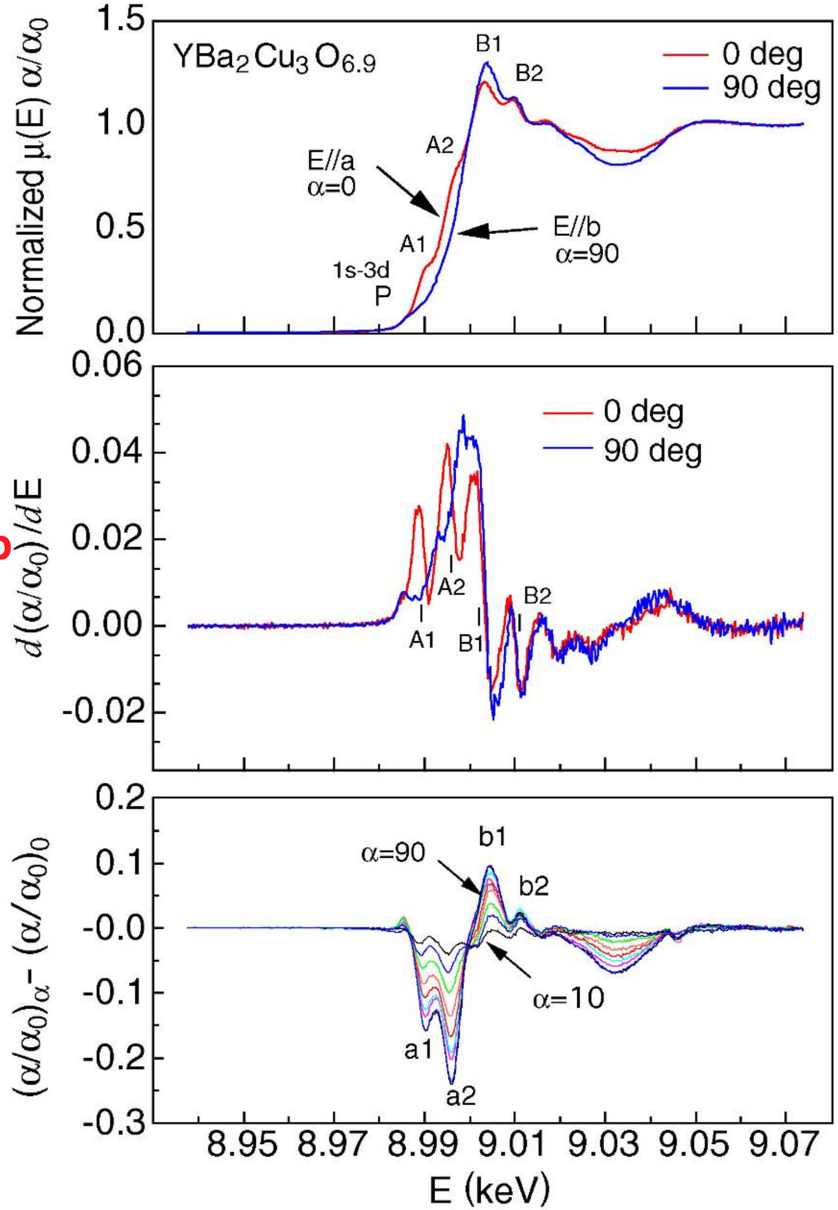


+ In-plane anisotropy





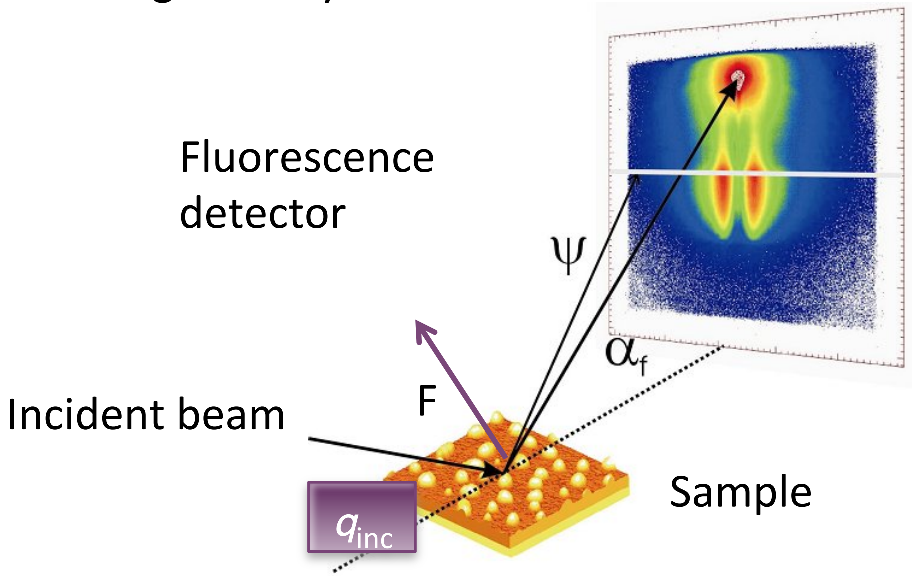
High-quality $YBa_2Cu_3O_y$ crystal



+ Grazing incidence geometry

Surface-sensitive geometry

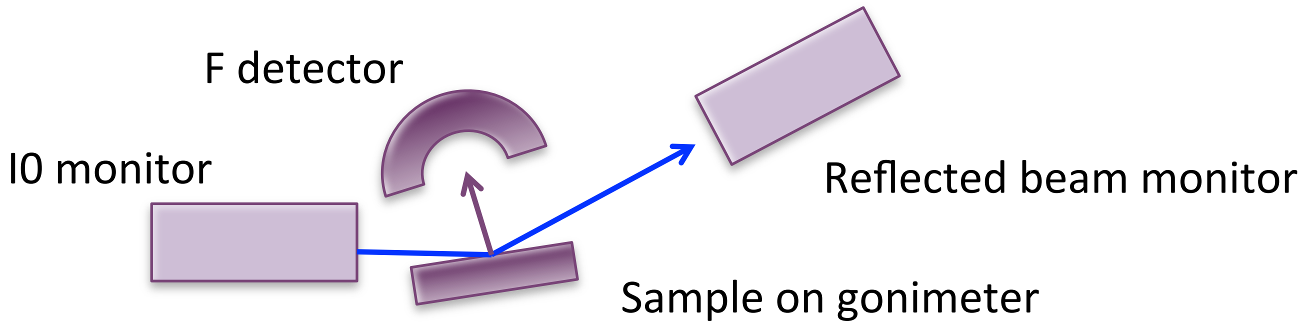
Surface scattered x-rays



Sensitivity ≈0.1 ML

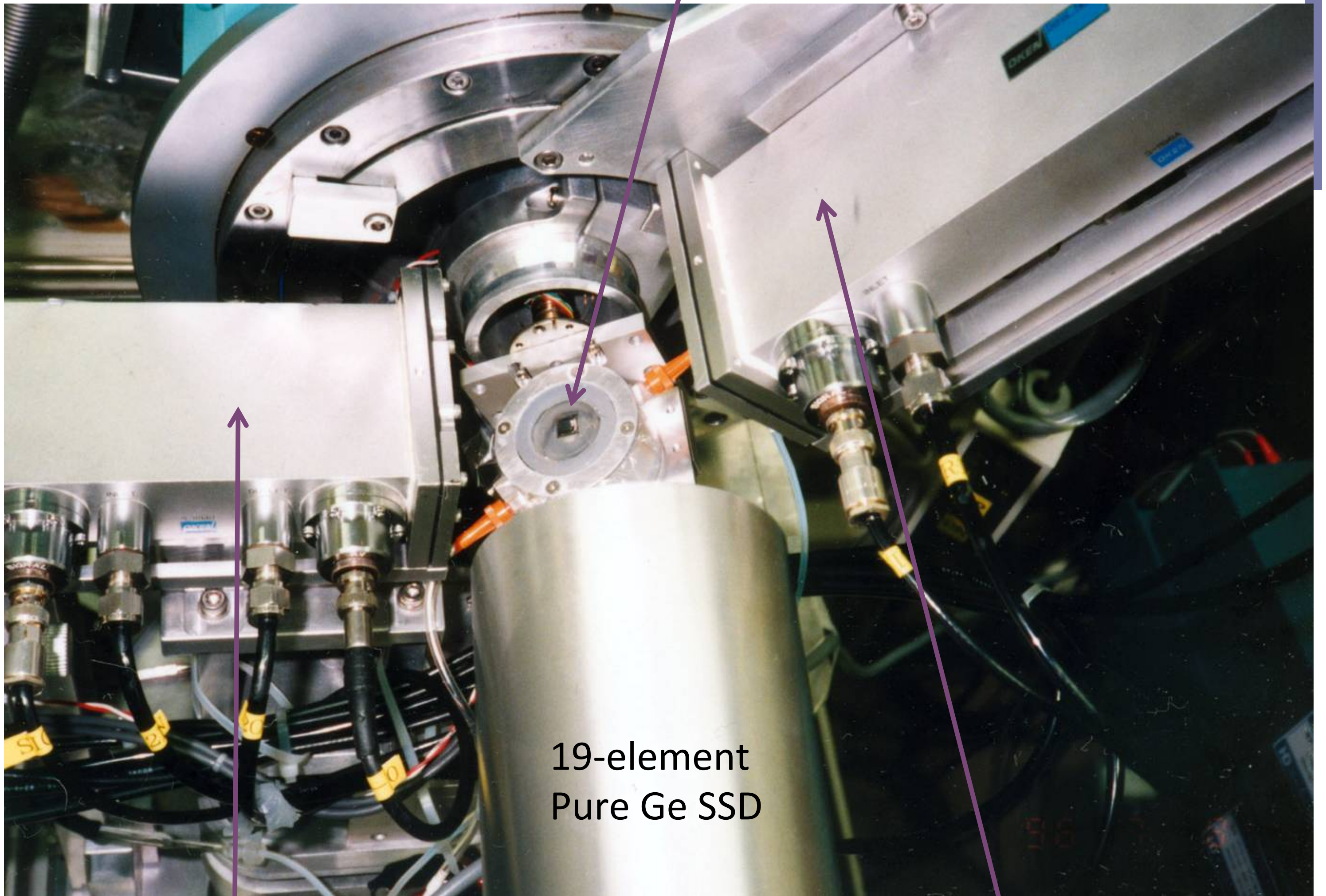
In a total reflection regime, $q_{inc} < q_c$

Selective excitation of surface with background reduction is possble
Increasing surface sensitivity to 0.1 monolayer (ML) level



XSW (X-ray Standing Wave) set up
Zegenhagen & Oyanagi @BL13/PF

Electrochemistry cell (GaAs substrate)

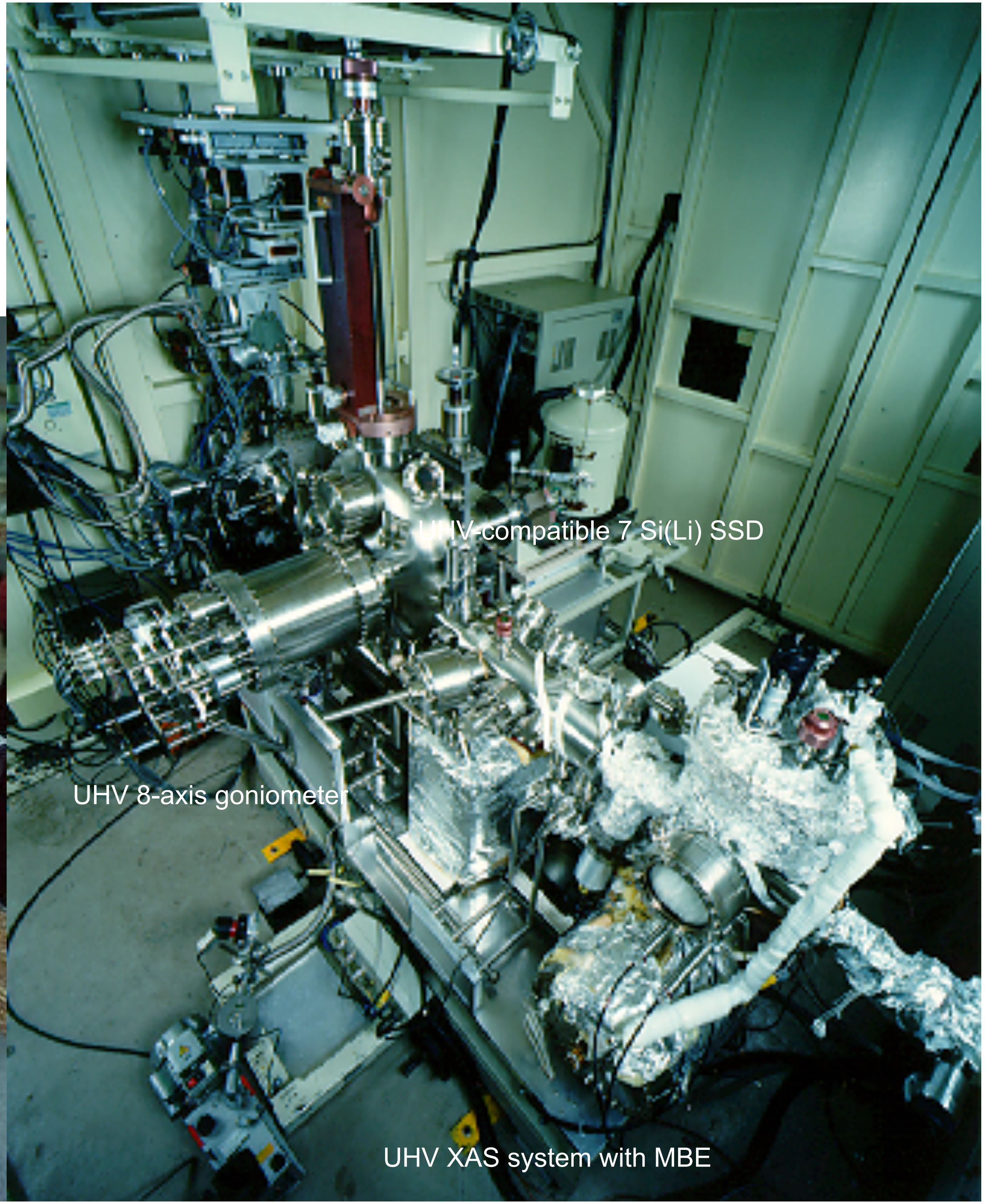


ii (reflected beam)monitor
(l=280 mm, N_2 or N_2+Ar gas)

i_0 monitor (l=140 mm, N_2 gas)

UHV fluorescence experiment

Rev. Sci. Instrum. 66, 5477 (1995)

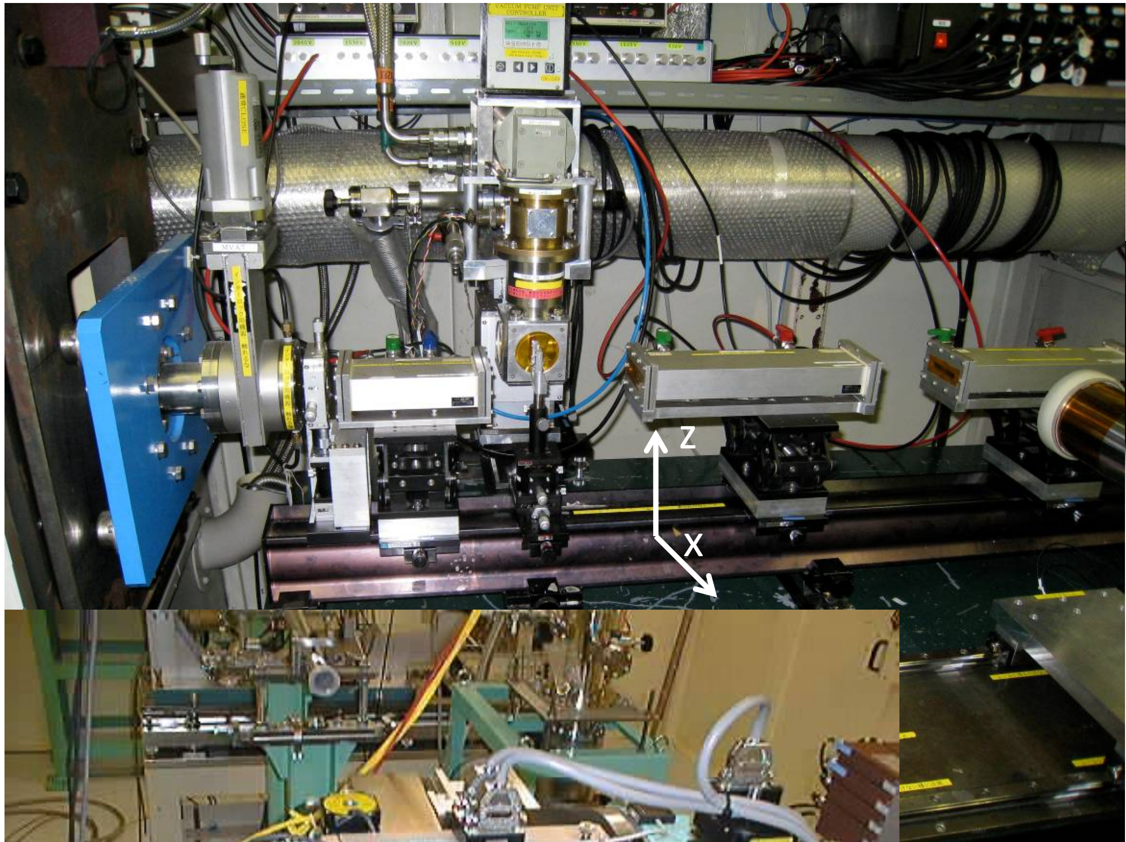


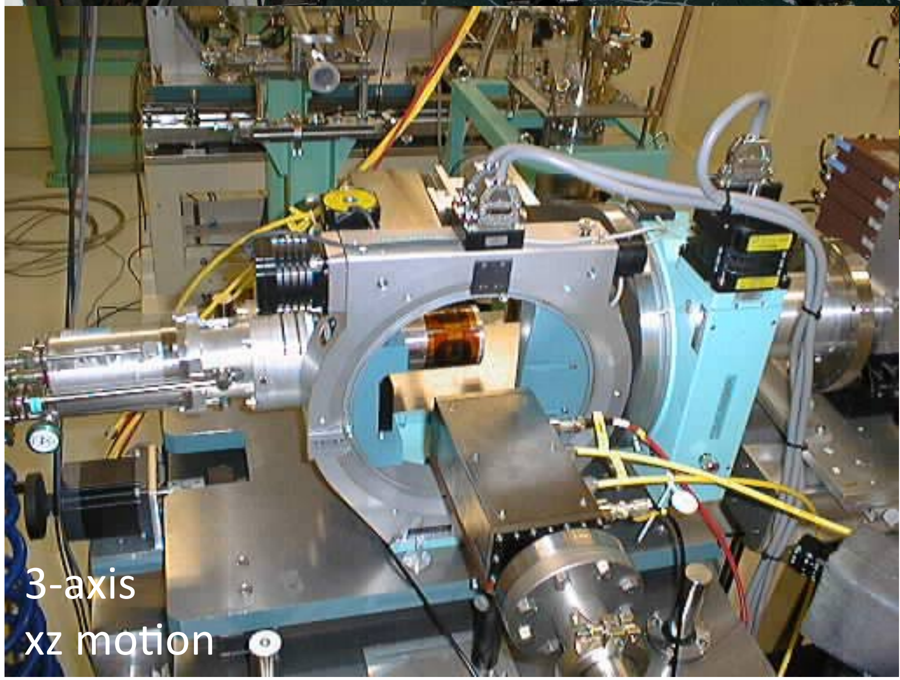
Sample preparation methods

Some hints for better experiments

+ Cryostat

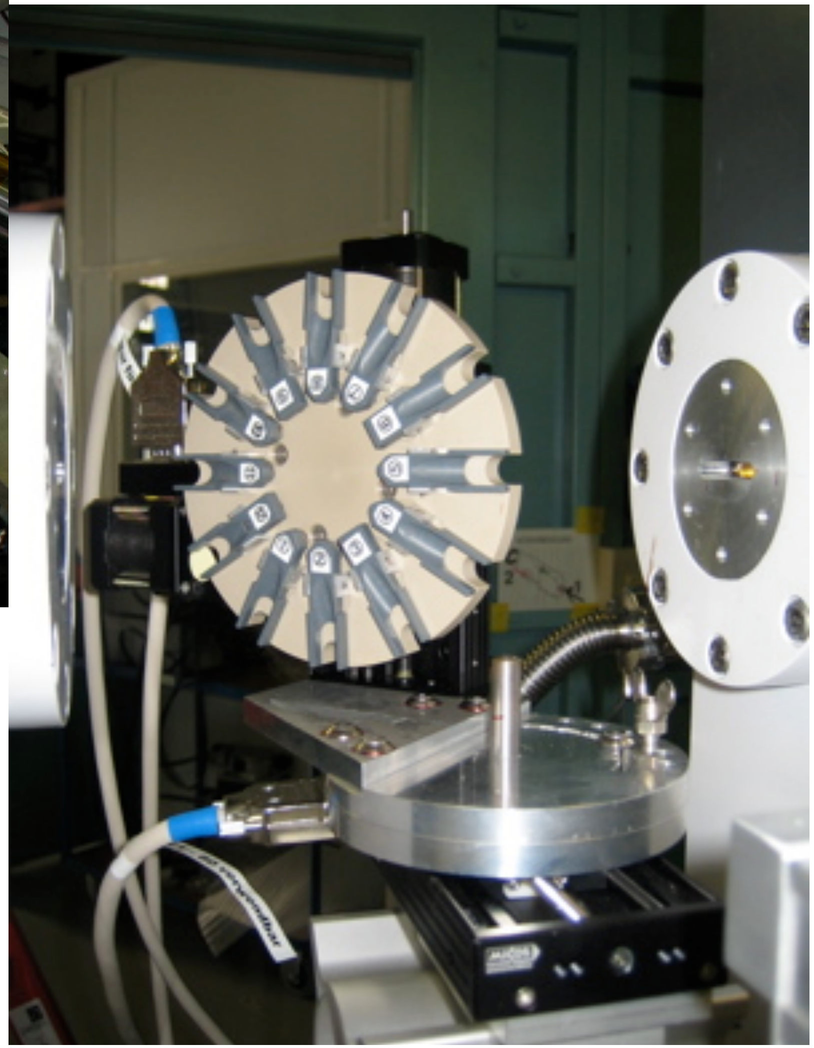
Closed cycle He cryostat @KEK





Closed cycle He cryostat with flexible tube @SPring8

Multi sample holder @ANKA



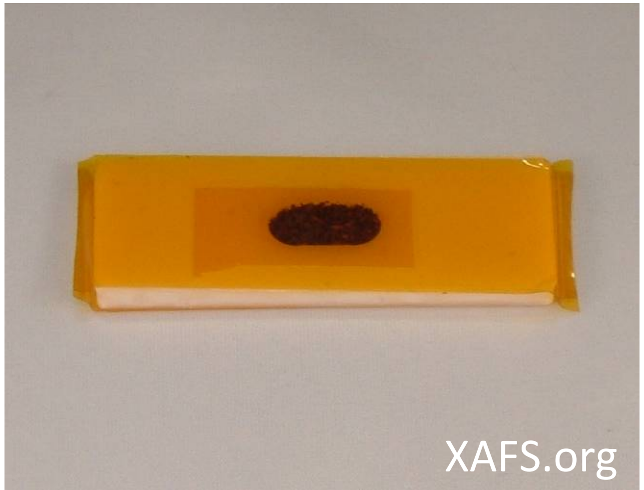
Sample preparation

General

Detailed description of sample preparation is available at xafs.org by the following researchers

<u>Grant Bunker</u>
Matt Newville
Rob Scarrow
Scott Calvin

URLs for each description available at xafs.org



Transmission experiment (Powder specimen)

http://www.xafs.org/Experiment/DoublyContainedSamples

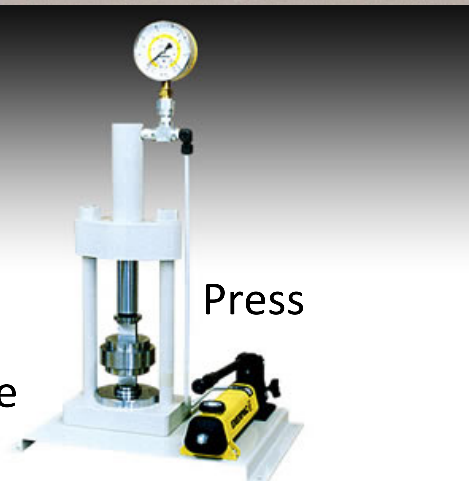
Homogeneity and right thickness/concentration

Fluorescence experiment (single crystal)

Orientation and surface roughness

Temperature dependence

Stress-free good thermal contact

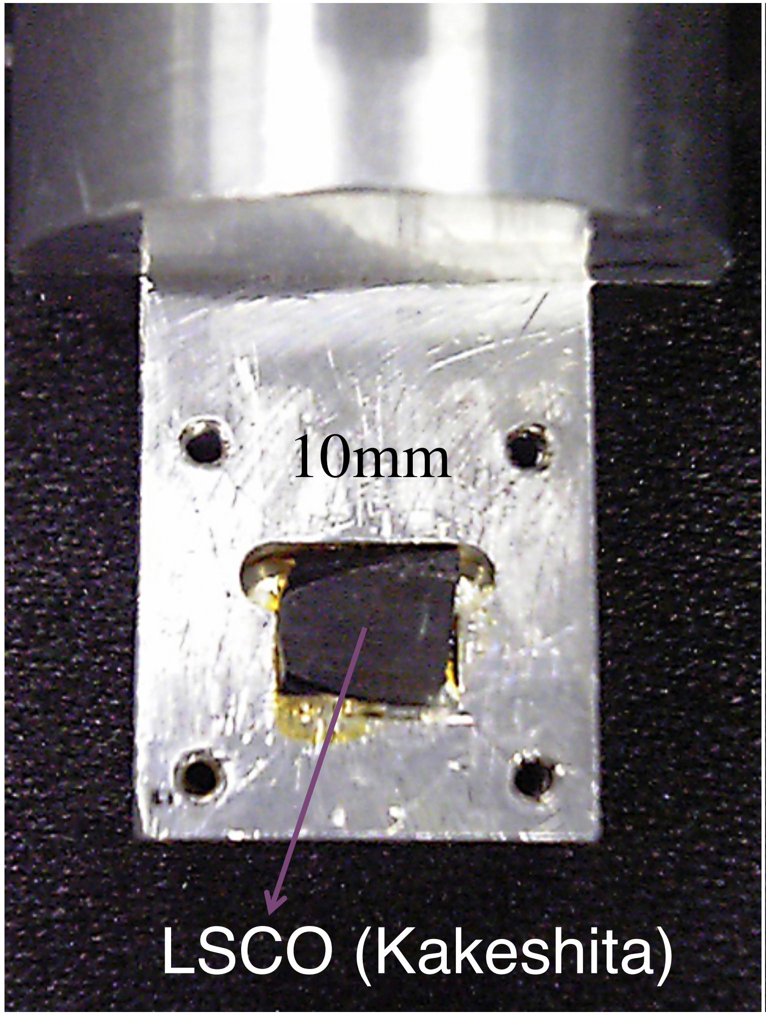


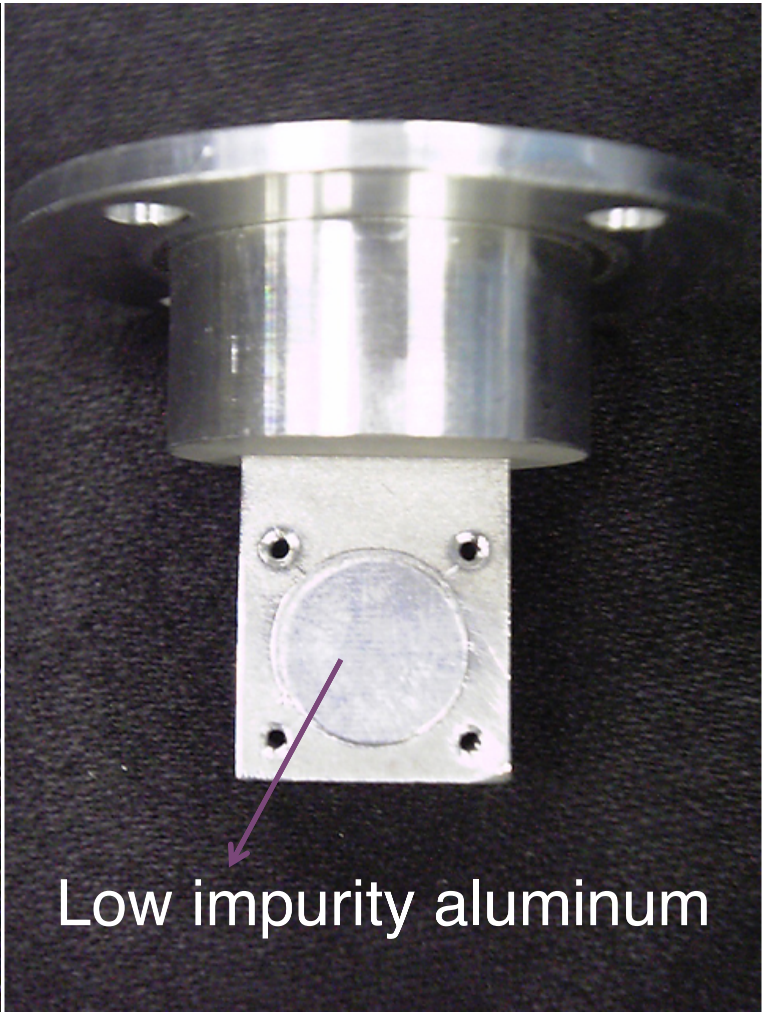
Press

Pellet sample

Sample mounting (bulk single crystals)

Large single crystal mounted on an aluminum base (left), smaller crystal mounted on impurity-free base (right)





LSCO on a standard type

2-4mm

Low-impurity type

1mm or less

+ Enjoy your experiment –Survaival tips

Anatoly Frenkel at NSLS beam line X16C.

- **S** - Size Up the Situation
- **U** - Use All Your Senses, Undue Haste Makes Waste
- **R** - Remember Where You Are
- **V** - Vanquish Fear and Panic
- **I** - Improvise
- **V** - Value Living
- **A** - Act Like the Natives
- **L** - Live by Your Wits, *But for Now*, Learn Basic Skills

taken from the U.S. Army Survival Manual