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APS Renewal Crystallography Instrumentation

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Mostly powder diffraction

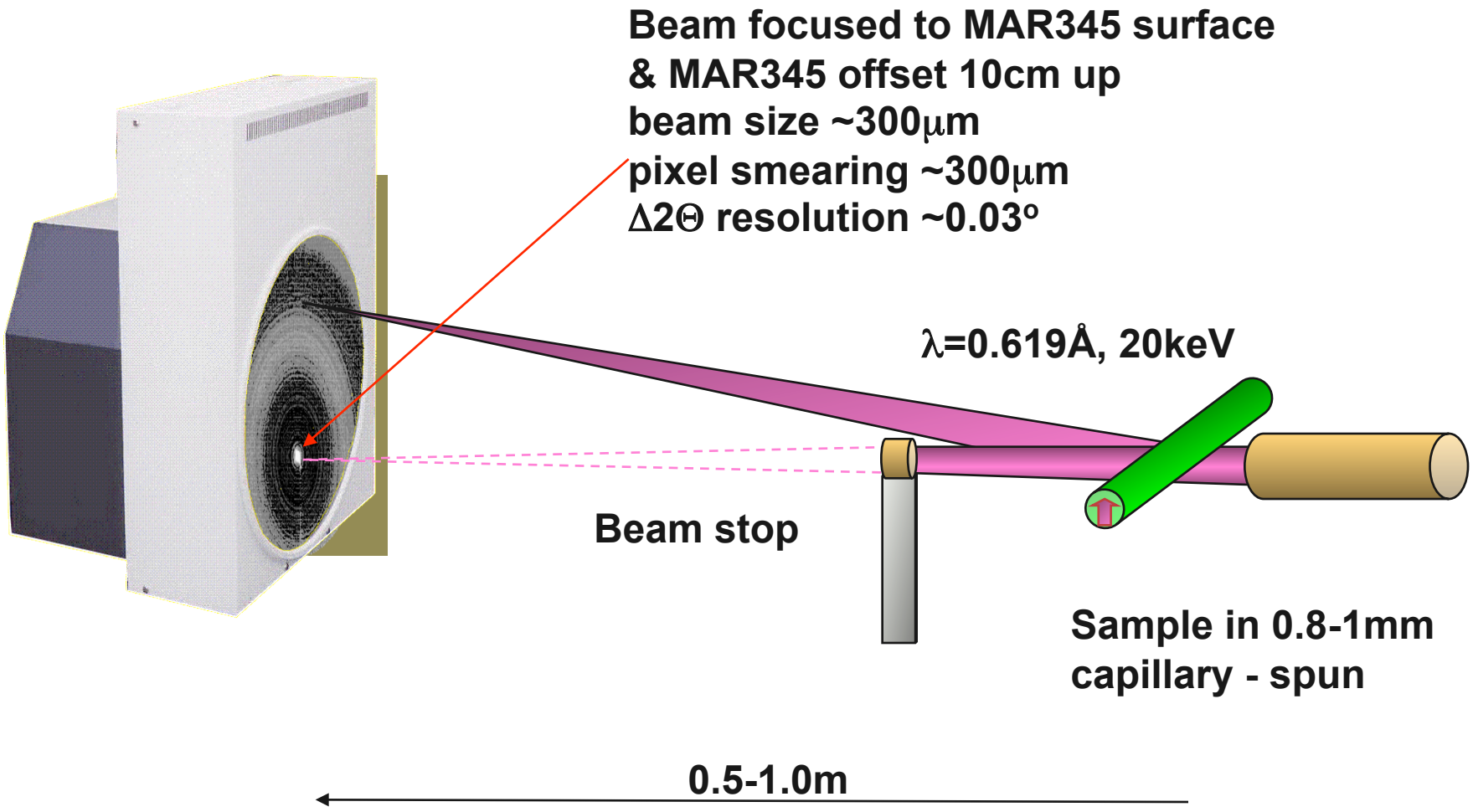
Why powder diffraction?
"Real samples/real time/real environments"
In situ structure analysis

"Powder diffraction is the structural tool for materials science."

"Definitive knowledge of the crystal structure of a material - inorganic, organic, or biological - is the gateway to understanding its physical properties, its chemical reactivity, and/or its biological functionality."

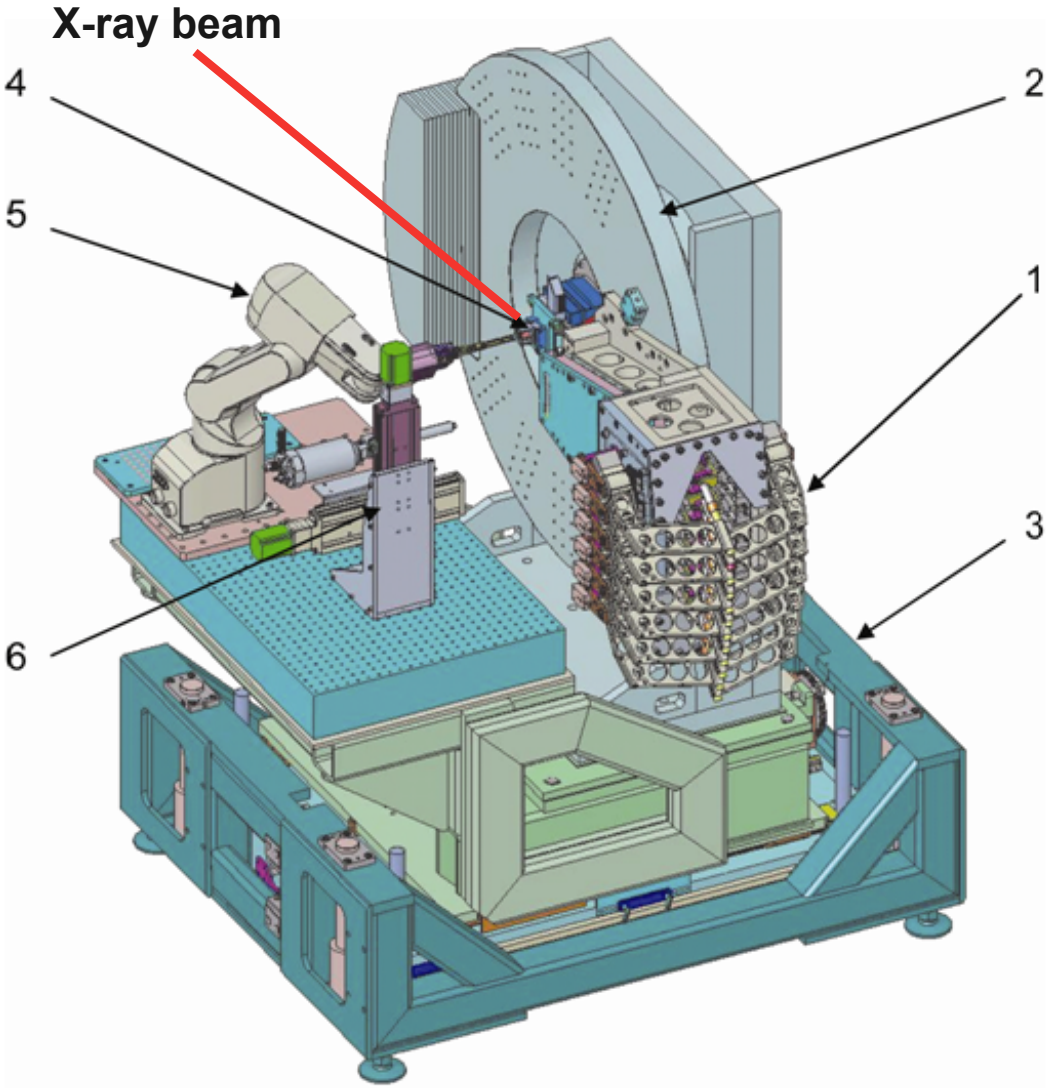
Buzz words: Oxides, intermetallics, high T_c super conductors, minerals, pharmaceuticals & proteins; resonant scattering, parametric studies, phase relationships, polymorphism, *ab initio* structure determination, PDF analysis, Rietveld refinement, complex formation, *in situ* reactions, etc.

Fundamentally a simple experiment – at "low resolution" NOT optimized!



Full pattern in one exposure – time 1-30s

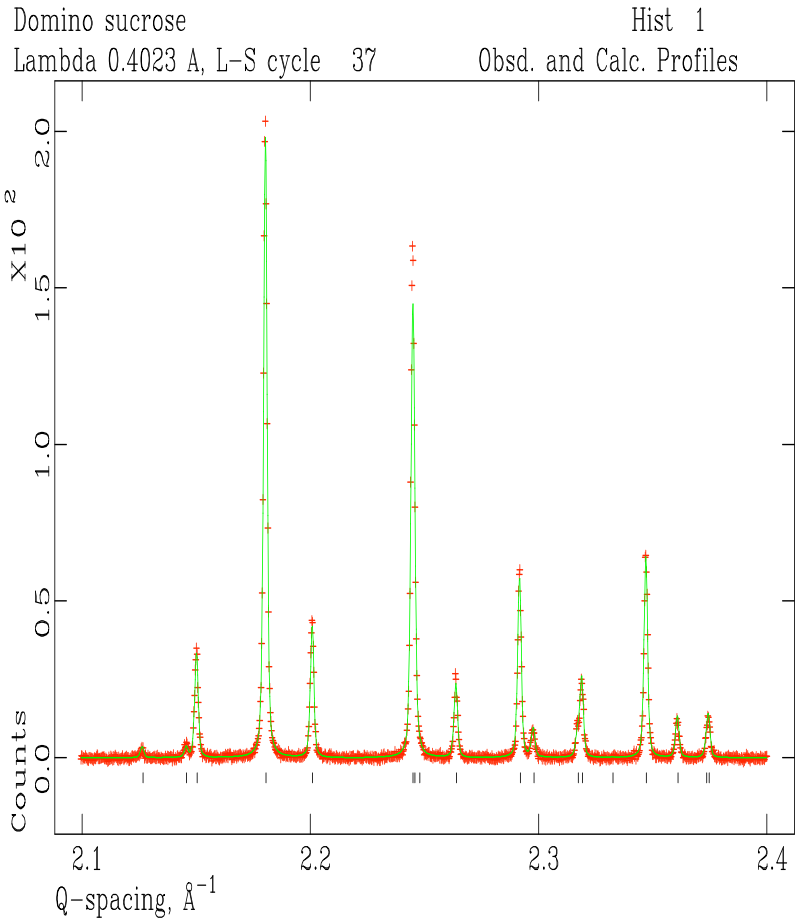
Complex experiment: Crystal/analyzer system – high resolution (11BM-B)

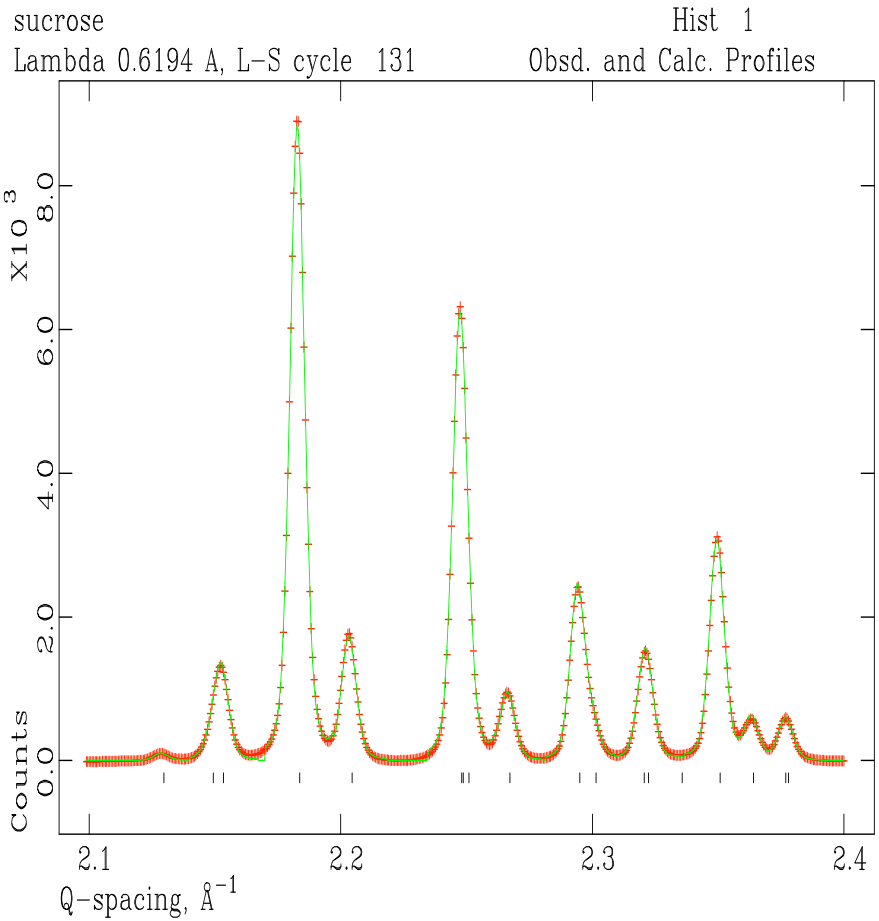


$\Delta 2\Theta$ resolution ~0.005°
Essentially the "ultimate"
(at sample limits)

Sequential data collection
– a "scan"
Time 2-60min
Suffer time dependent effects:
Radiation damage
Kinetics studies not possible
limited in situ studies

Data comparison



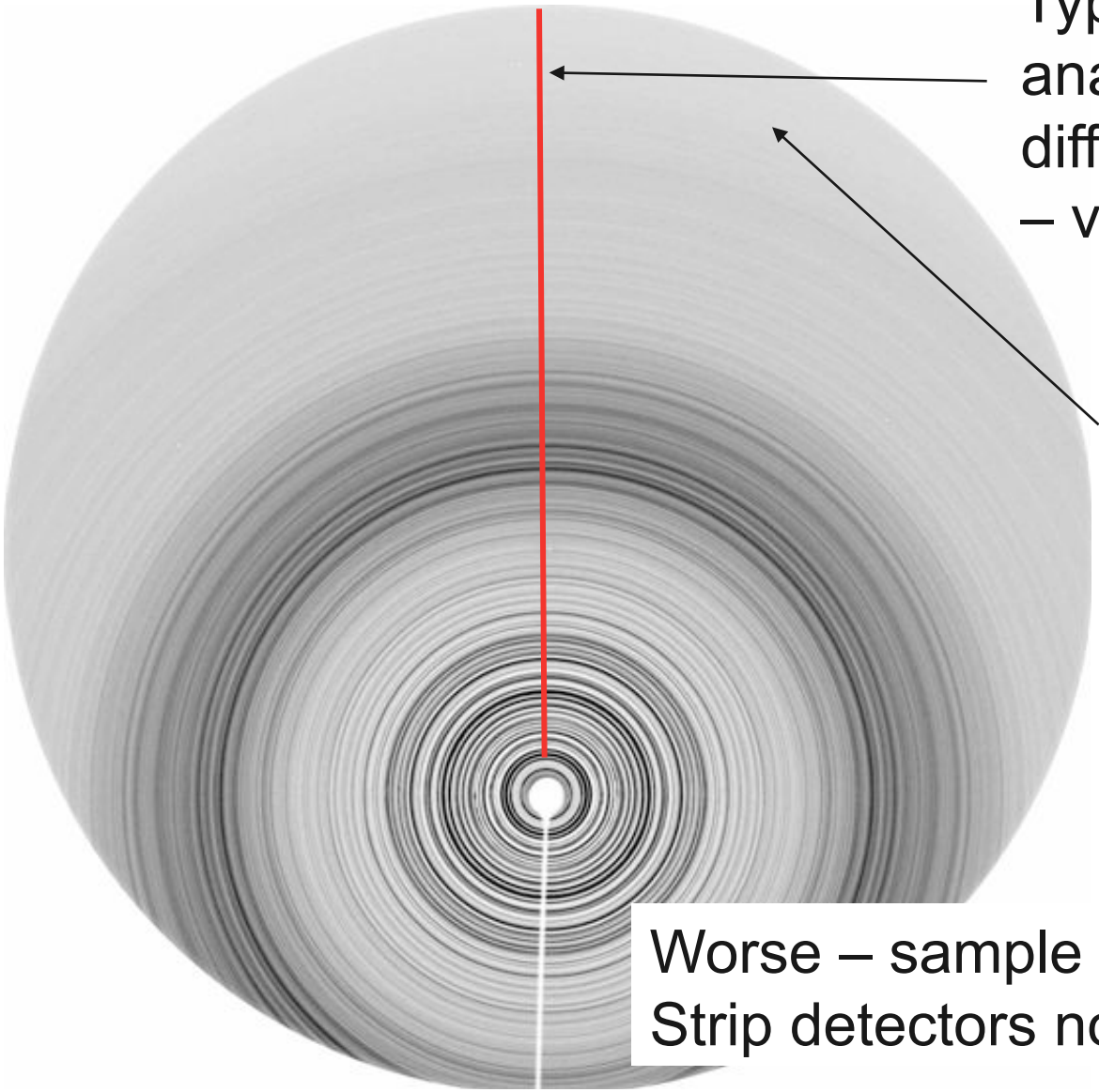


11BMB – part of 10min scan

1BM/MAR345 – 1sec exposure

3-4X broader peaks!

Powder pattern - rings



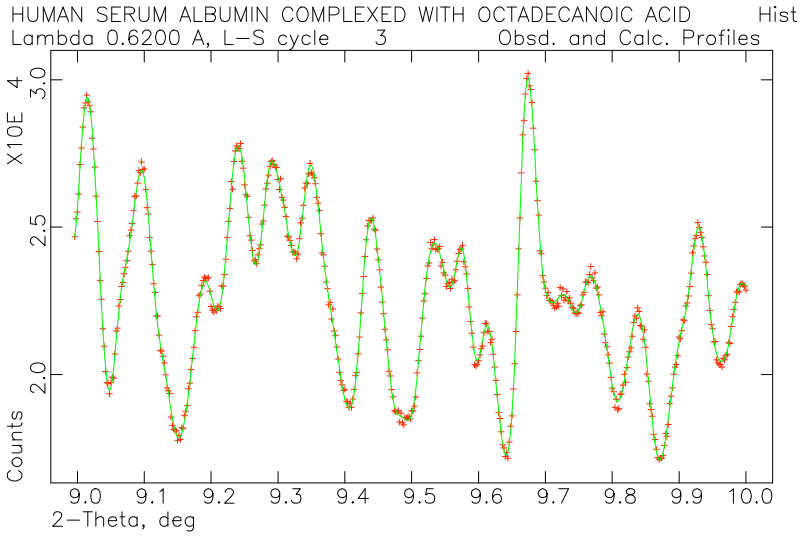
Typical 2Θ scan – analyzer/detector diffractometer – very small solid angle

Rest is wasted!

(especially at high angles)

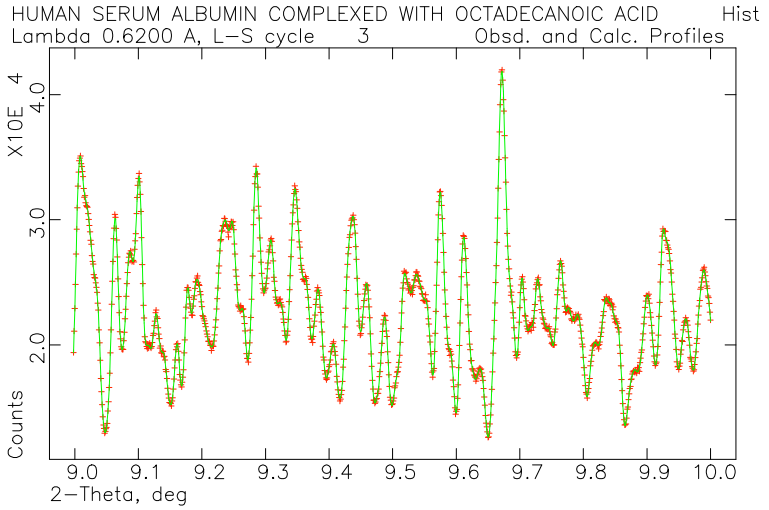
Worse – sample damage during scan
Strip detectors no real solution

Instrumentation challenge – high res. area detector



Current: MAR345 IP
300μm spatial resolution

Wish ☺ :
30μm resolution
match analyzer/detector resolution & sample contribution



Powder Instrument design

Need: Design “state of the art” high resolution imaging powder diffractometer

- Optimize for high resolution area detector
- Guinier geometry – vertical & horizontal beam focusing
- Optimal detector surface – curved over 3D?
- Optimal detector coverage – mostly vertical?
- Other considerations – special environment issues – different instrument?

Impact: “Real samples/real time/real environments” (even for proteins)

In situ reactions/kinetics; phase changes, small samples, controlled sampling volumes (develop 2D collimation)

Instrument Operations

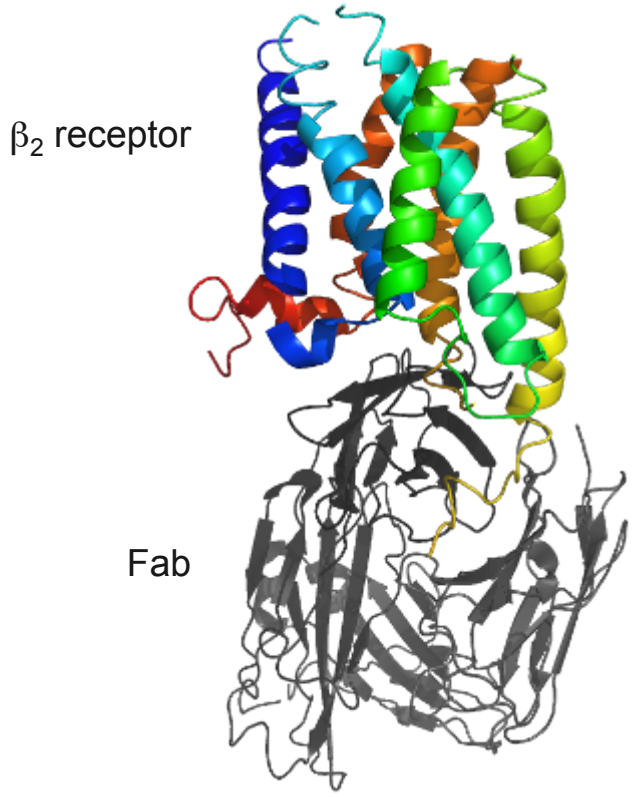
Need: Facility wide automation & design support

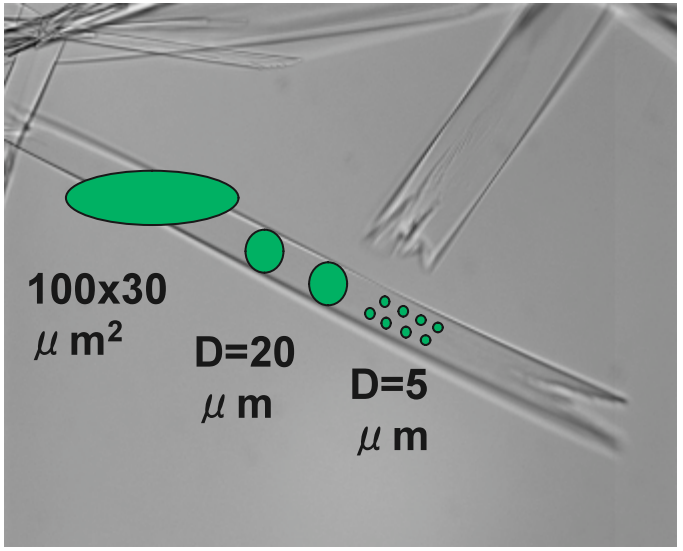
- Current status – wide range of capabilities at APS
  - High level of automation ← → almost no automation
  - Macromolecular beam lines – high automation
    - *Simple energy change – enter value/select element edge*
    - *Fast! & User selected*
    - *Auto optimization during operation*
    - *Heavy investment in mechanicals & engineering*
  - Materials beam lines - little automation
    - *Manual energy changes – not user controlled*
    - *Excruciatingly slow & error prone*
    - *No optimization*
    - *Done cheap as possible & little cross-sector common design effort*

MM payoff of attention to design:

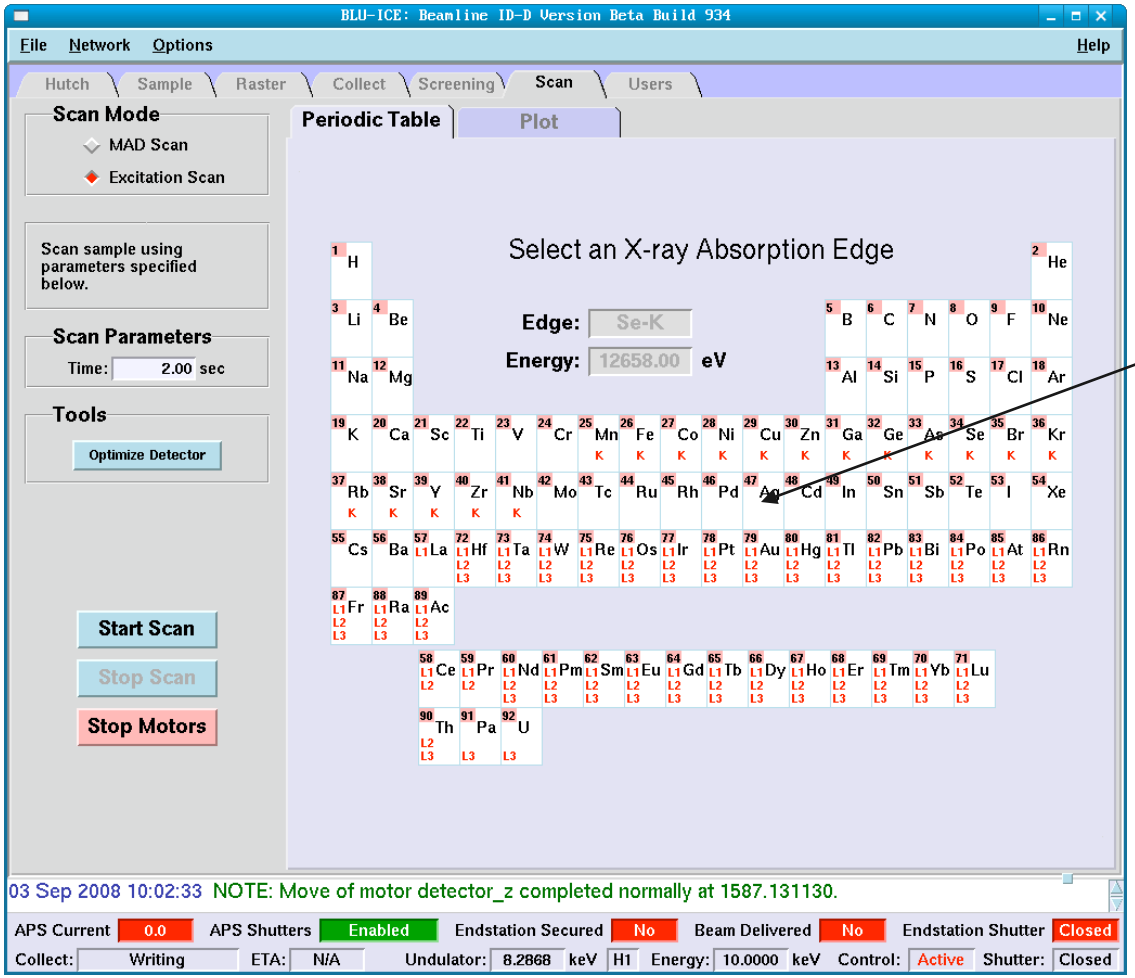
User Needs Drove Development of the Mini-beam - Radiation Sensitivity

S. G. F. Rasmussen, H.-J. Choi, D. M. Rosenbaum, T. S. Kobilka, F. S. Thian, P. C. Edwards, M. Burghammer, V. R. P. Ratnala, R. Sanishvili, R. F. Fischetti, G. F. X. Schertler, W. I. Weis, B. K. Kobilka, "Crystal structure of the human β_2 adrenergic G-protein-coupled receptor", *Nature* **450**, 383-387 (2007).





"I'd like to see" - Bluice (MM data acquisition) equivalent



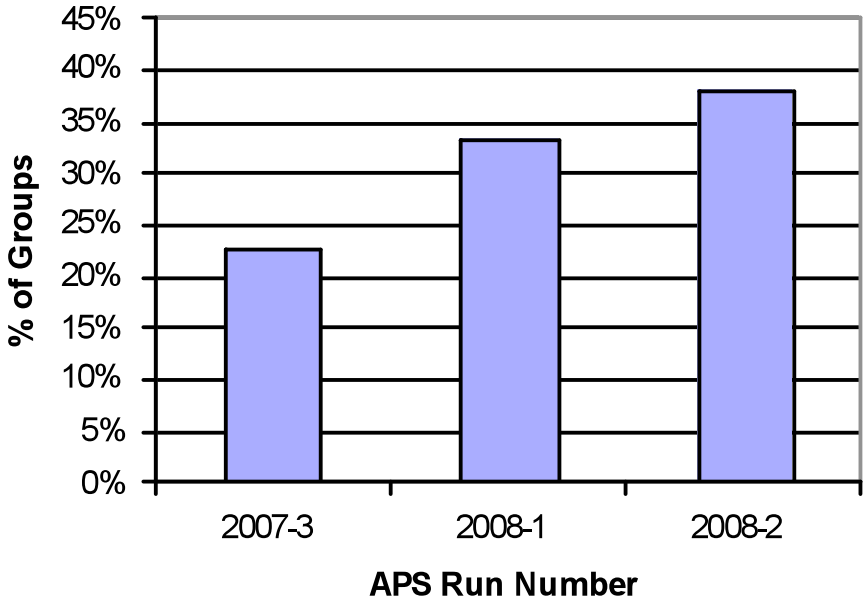
Choose edge for data collection

11BMB – only manual energy change → Std. Ops. Fixed E

Requires – careful mechanical & controls design

Future at APS? – from current trends

Increased Use of Automounter – MM experience; 11BM similar

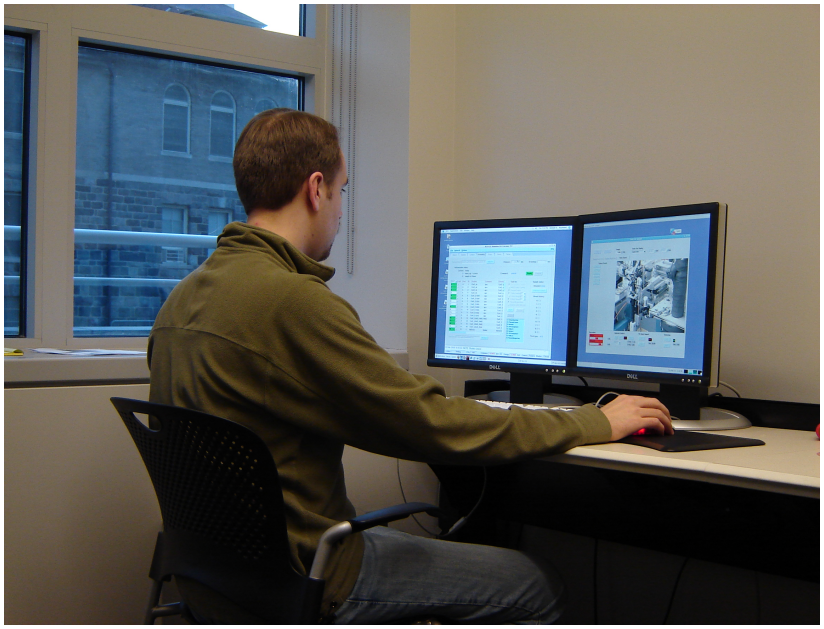


23-ID-D - ALS-style pucks only: ~50% automounter use in 2008-1.

23-ID-B - Rigaku-style pucks only: ~19% automounter use in 2008-1.

Less need for users to shop up at APS!

Remote beamline control with NX implemented



Todd Geders using remote controls at U. Michigan

- Same technology as at SSRL & others
- Two computers open per beamline (one for data collection & one for processing)
- Extra controls for remote operation in BluIce

Compliance with Argonne's enhanced security requirements: access is restricted not only to the time of an experiment and specified user name, but also to a list of IP domains from which users work.

Future of APS science – remote experiments!