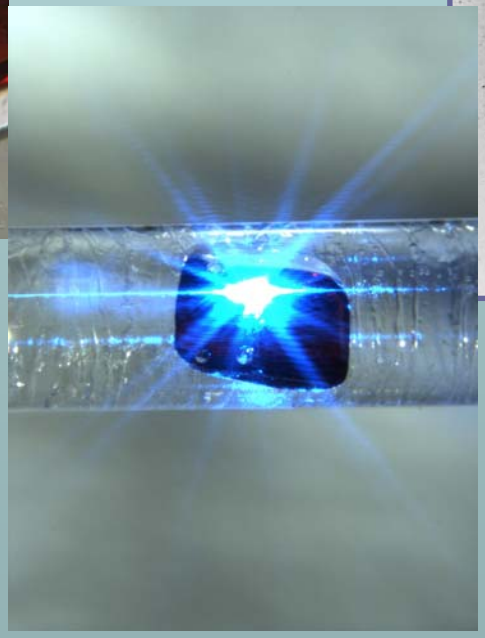
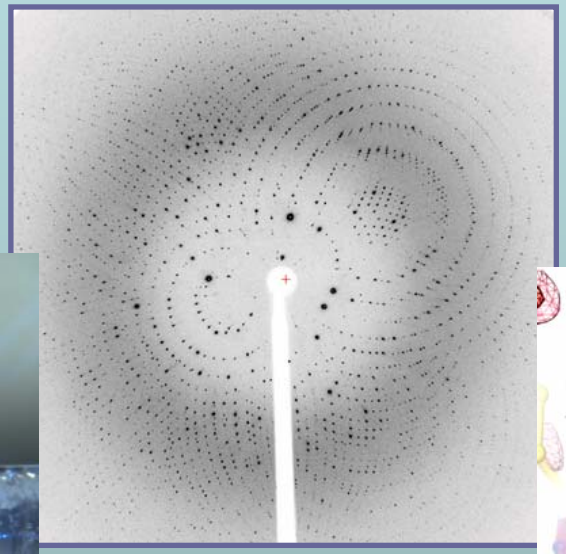
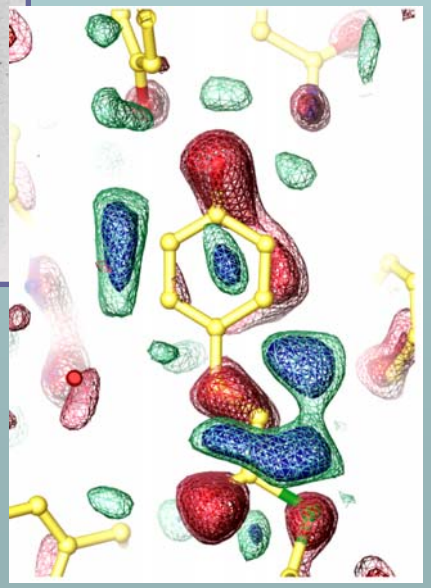
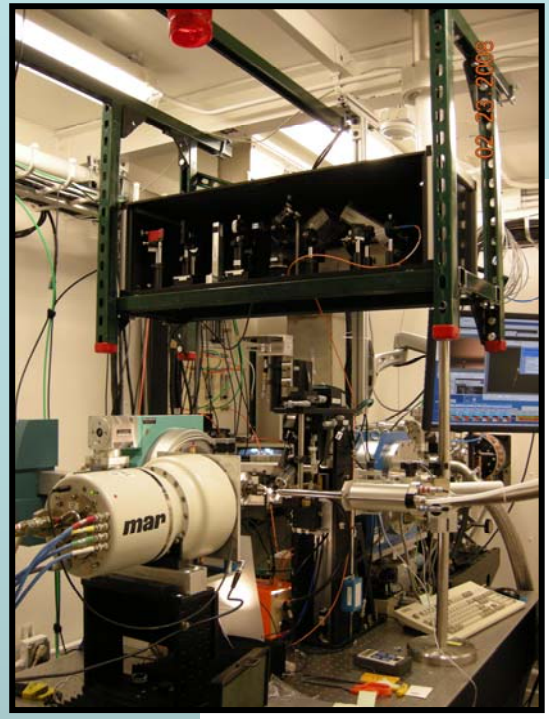
Time-Resolved Crystallography Experimental Set-up at 14-ID

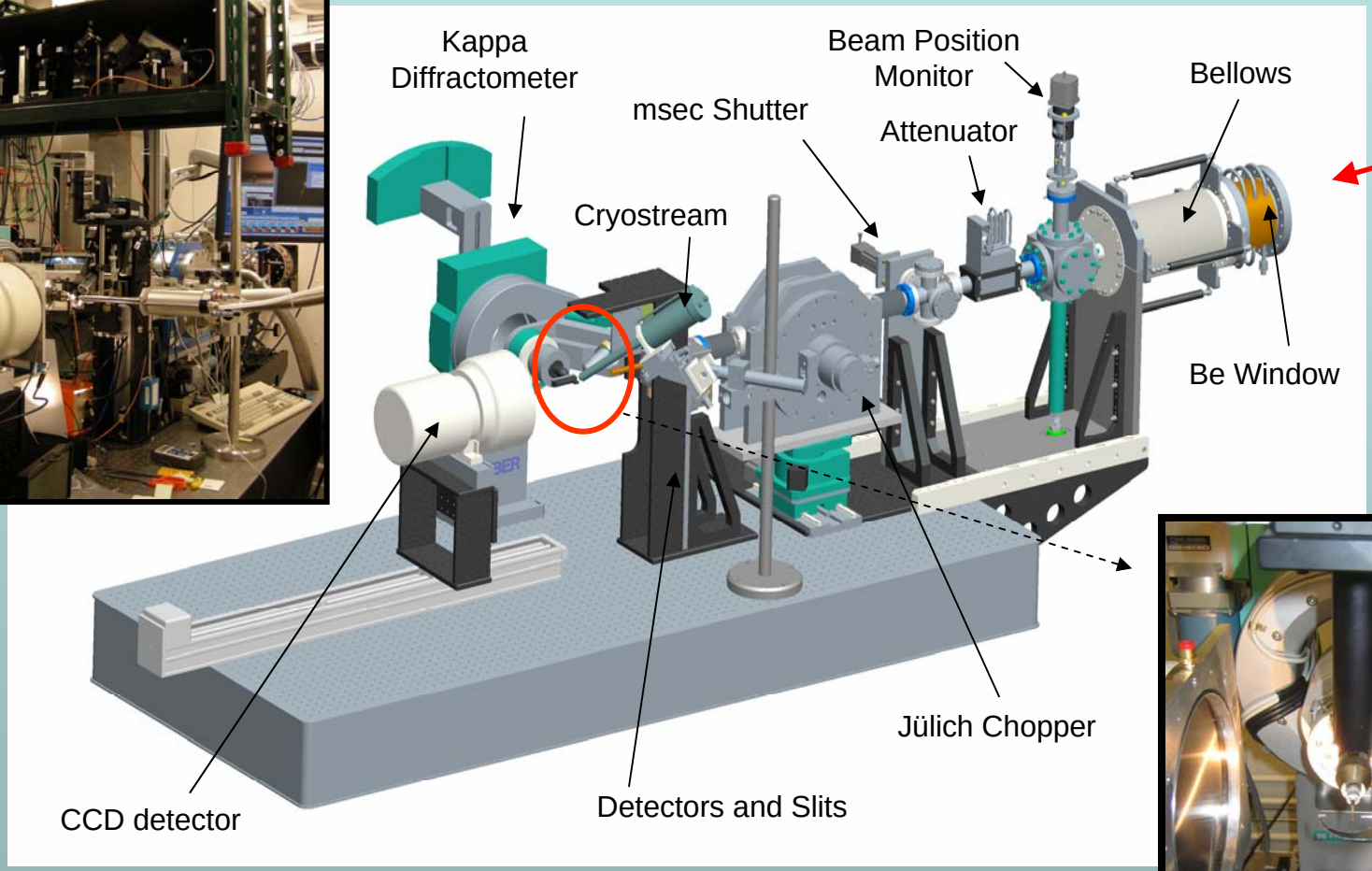






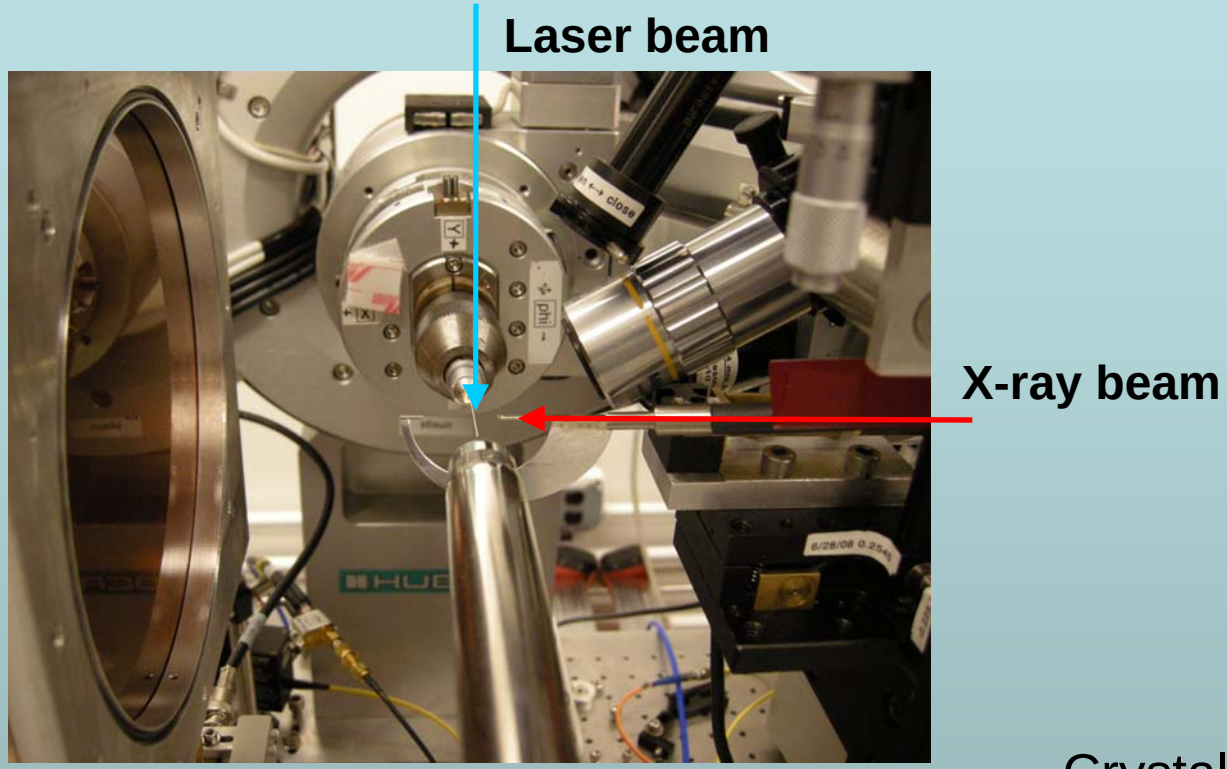
14-ID-B Station

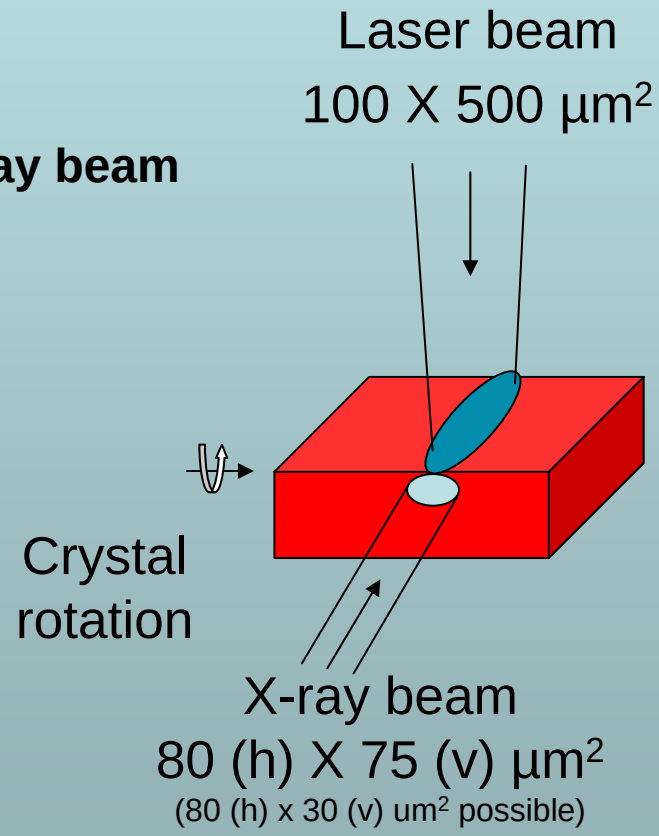




Sample environment

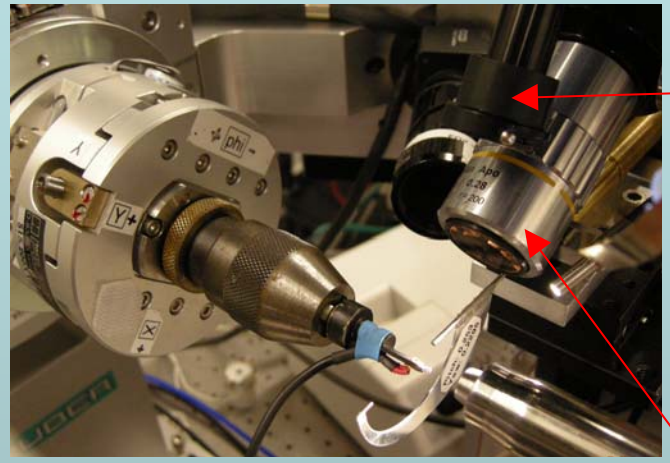
Sample Environment





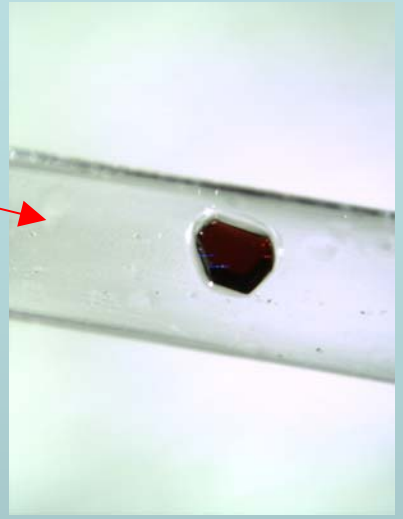
- **Overlap of x-ray and laser beams is critical**

Sample Viewing



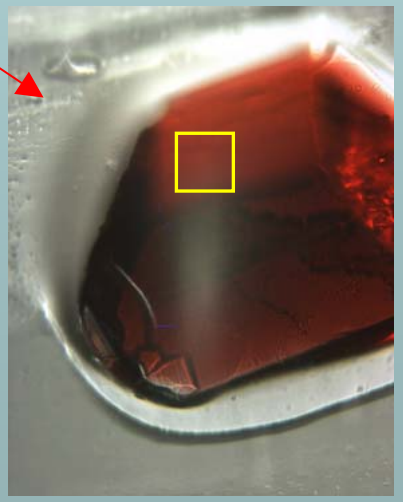
Wide field camera

3.2 x 2.4 mm FOV



High resolution camera

0.632 x 0.476 mm FOV

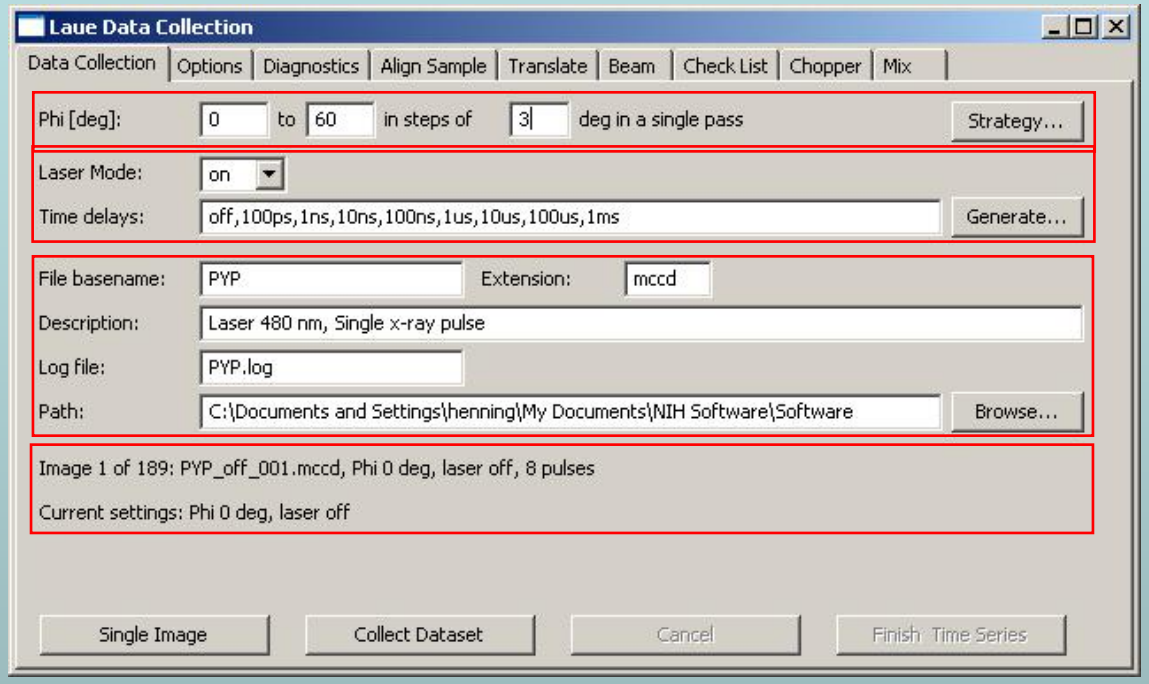


Yellow box indicates approximate beam size on the sample 100 x 100 microns (H x V)

- High resolution cameras allow for precise sample alignment
- Small focal spot allows for working with small crystals or for collecting multiple data sets from one crystal

Laue Collect

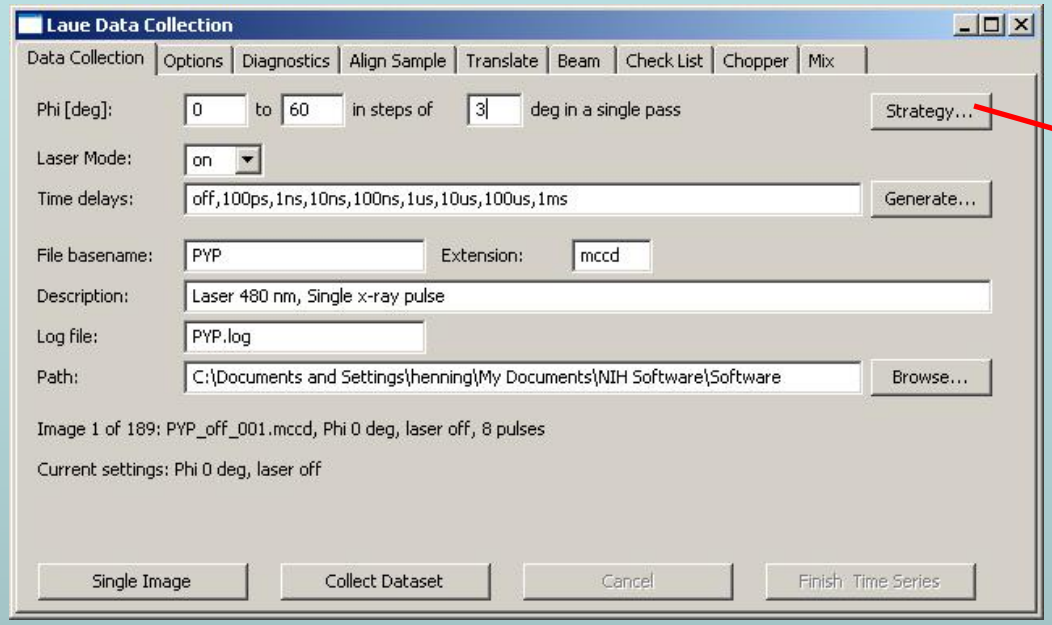
Written by Friedrich Schotte

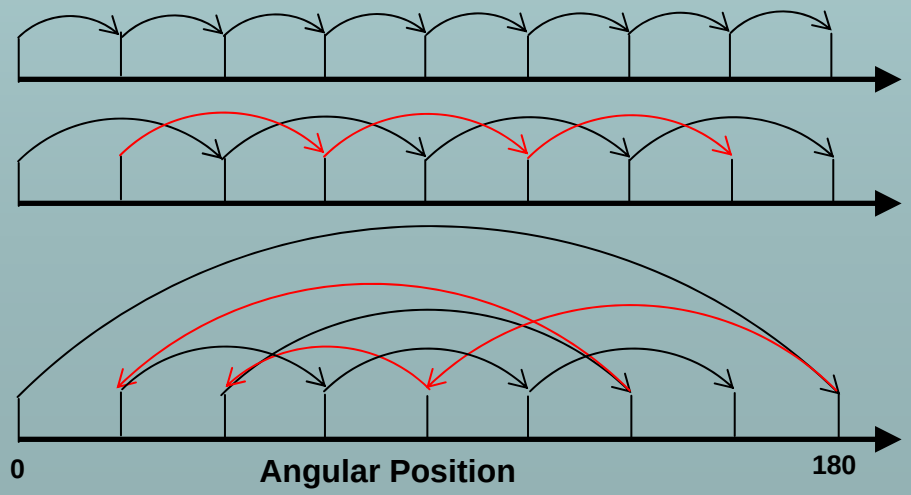


- Data collection interface written in Python
- Tabbed layout
- Used for both single crystal and solution studies

- Data Collection
  - Determine data collection strategy
  - Laser operation and time delays
  - Input basic information: file name, directory and description
  - Provides status information

Data Collection Strategy



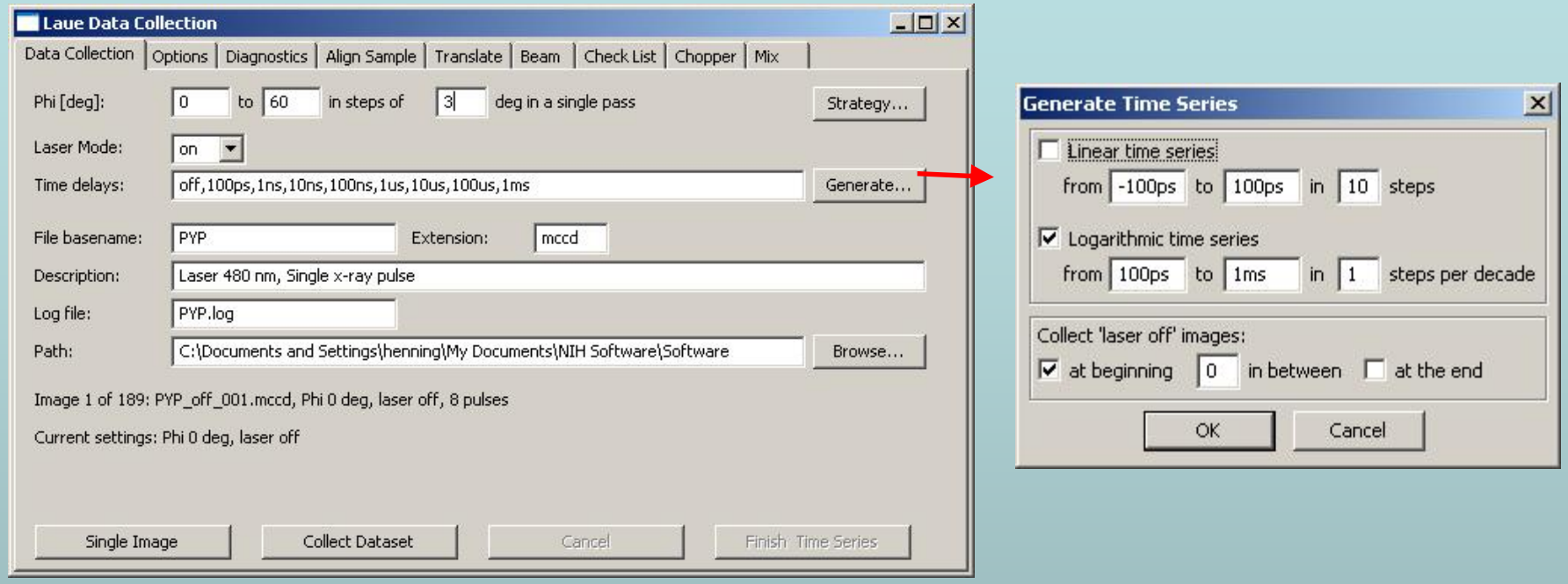


Single Pass

Two Interlaced Passes

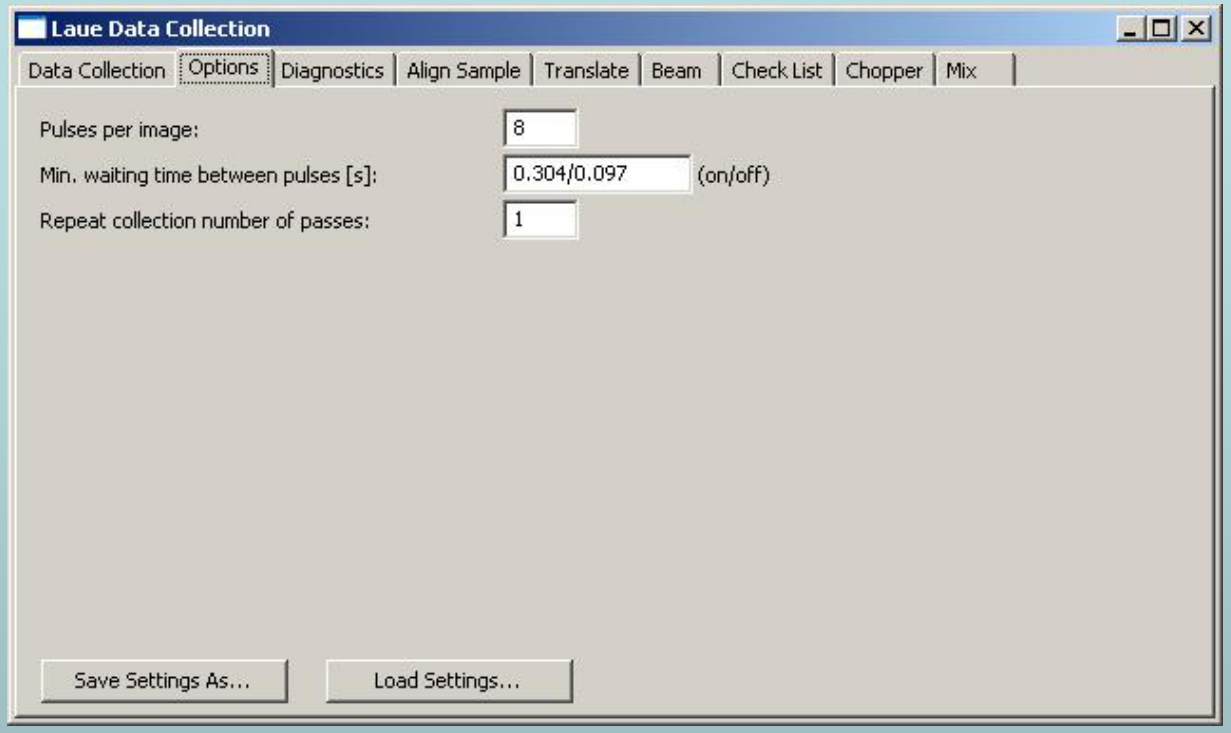
Filling gaps

Pump Probe Delay



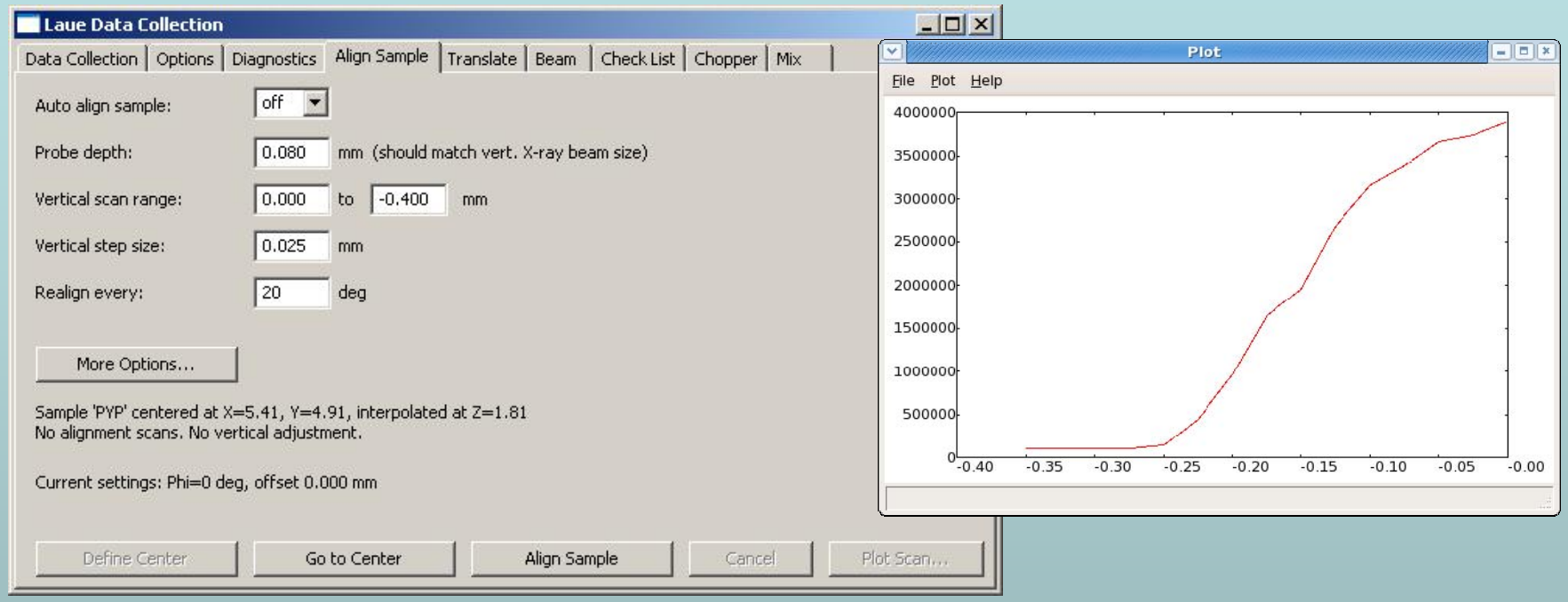
- Linear times series: 0, 500ps, 1ns, 1.5ns
- Logarithmic time series: 0, 100ps, 1ns, 10ns, 100ns, 1us
- Can also add time sequence manually
- Off images can be collected periodically

Data Collection Options



- Multiple pump-probe pulses may be required for a single image
  - 4-20 pulses are typical for macromolecular materials
  - A single pulse could be used if the sample diffracts well or the chemistry requires only one pulse (irreversible reactions)
- Waiting time between pulses
  - Lifetime of the excited state may be long
  - Large temperature jump due to the laser

Crystal Edge Scanning

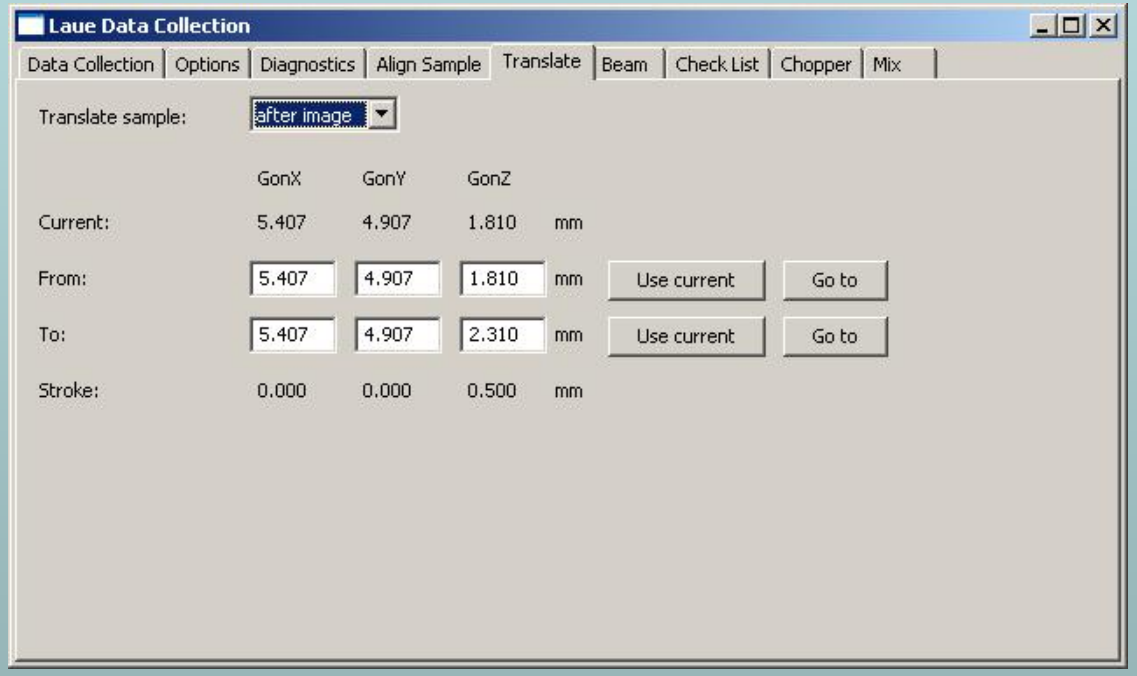


Scan edge of crystal with x-ray beam in order to determine the optimum position during data collection

- Dependent on laser penetration depth

Crystal Translation

- Radiation damage
  - Large crystals can be translated to a fresh surface during the data collection
  - May have to switch samples if crystals are too small

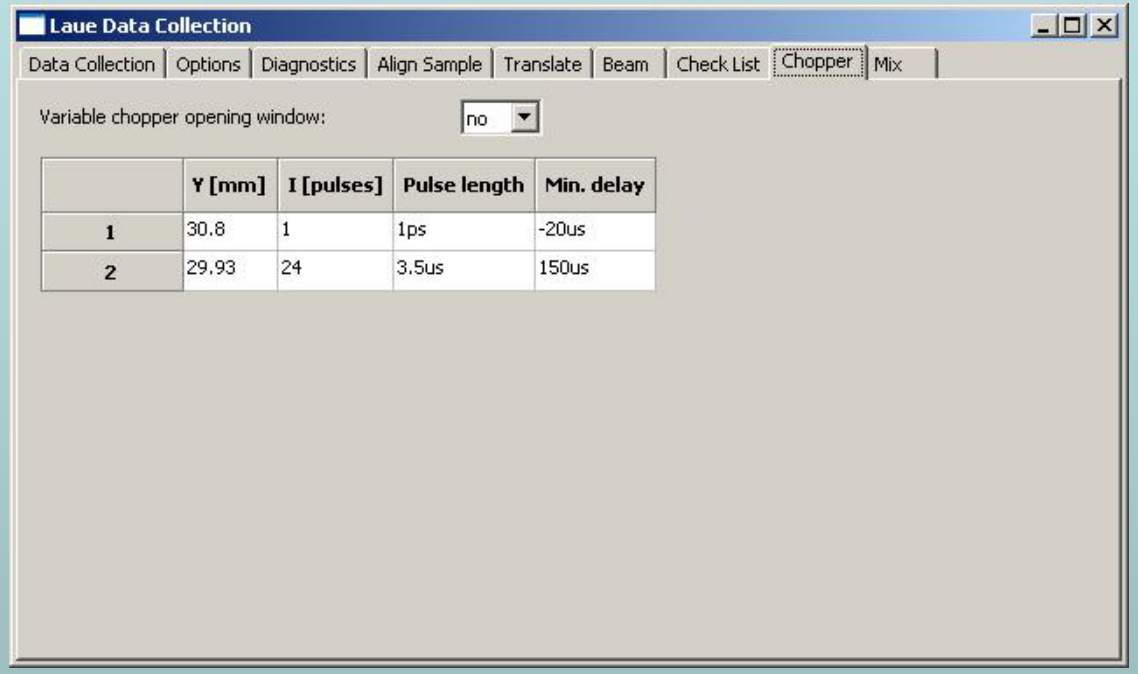


Translate options
- After image
- Continuous (TWAX)
- Linear stage (TWAX)
- Off

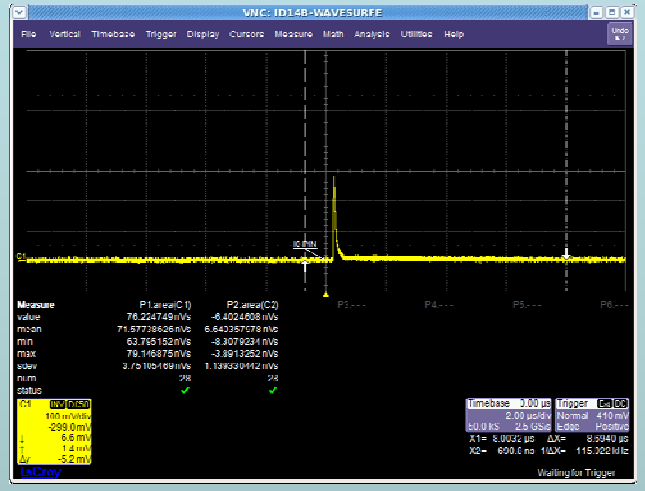
Crystal translation

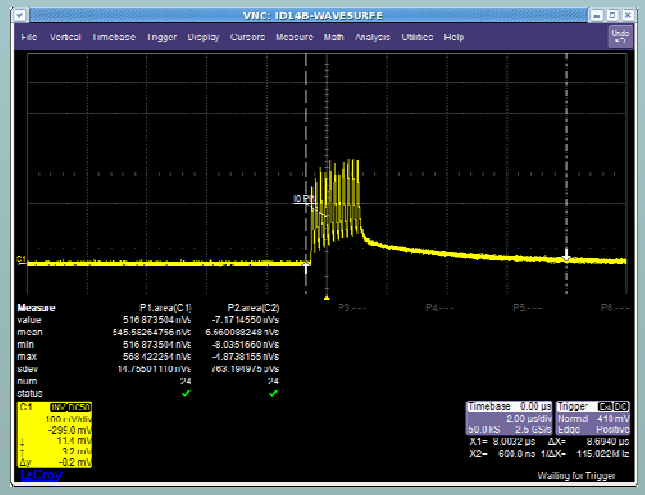
Yellow box is the x-ray/laser beam

Variable Pulse Isolation



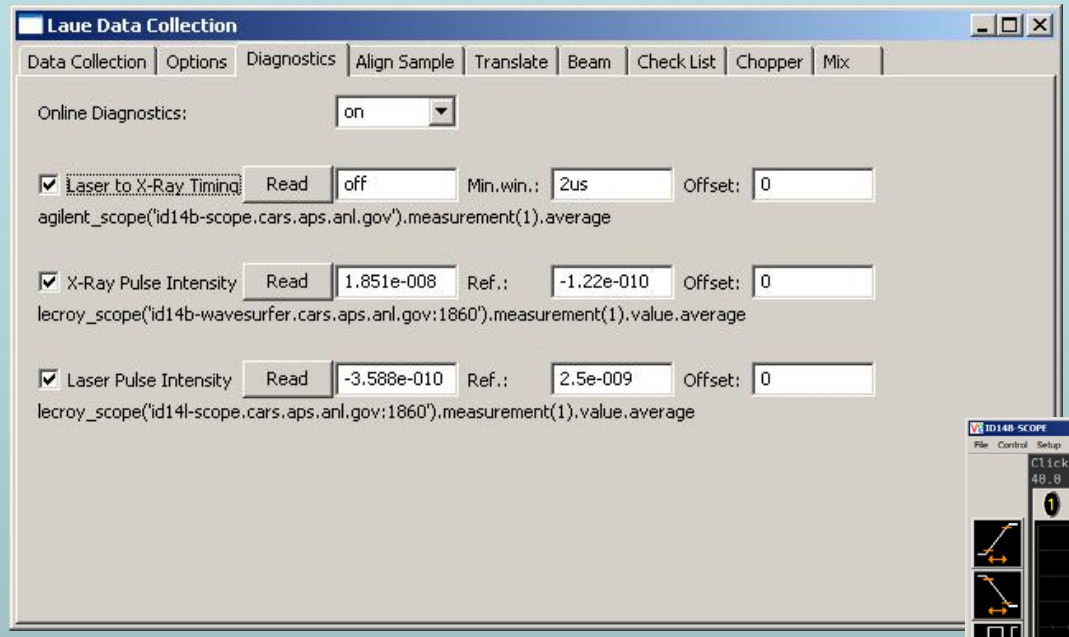
	Y [mm]	I [pulses]	Pulse length	Min. delay
1	30.8	1	1ps	-20us
2	29.93	24	3.5us	150us





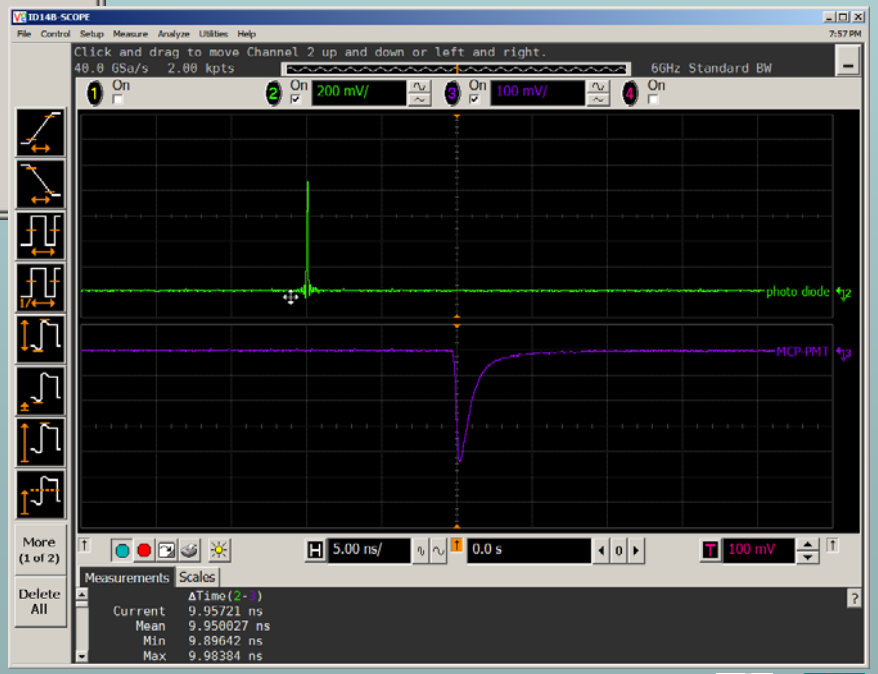
- Vertical position of Julich chopper is adjusted to change number of pulses
- Can be used to increase flux at longer time delays
- Used in 24 bunch mode

Online Diagnostics



Information logged for every image

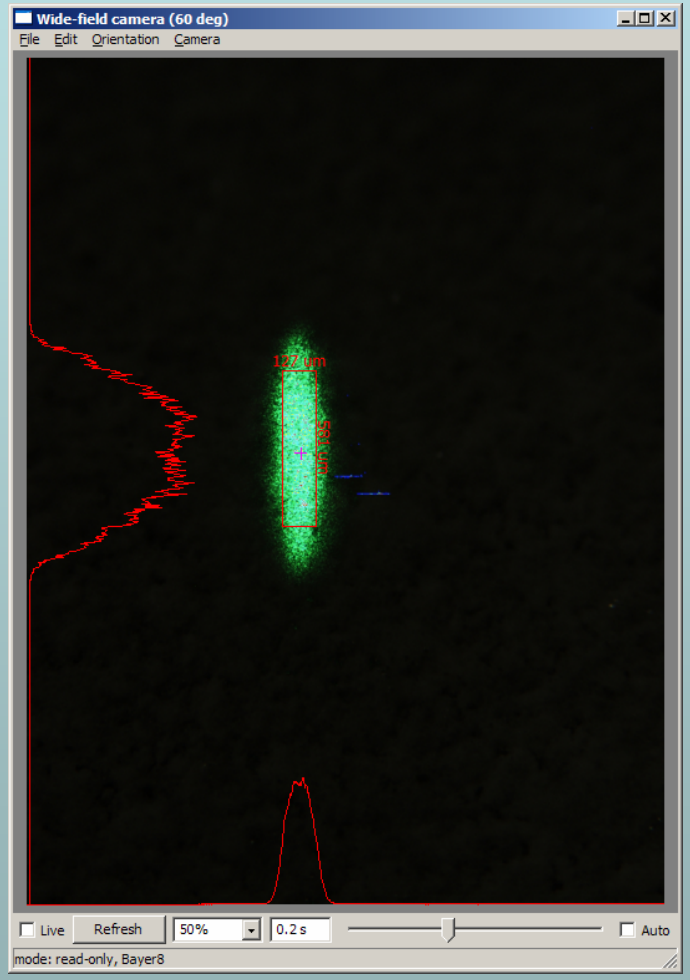
- Laser to X-ray time delay
- X-ray intensity
- Laser intensity



Detectors

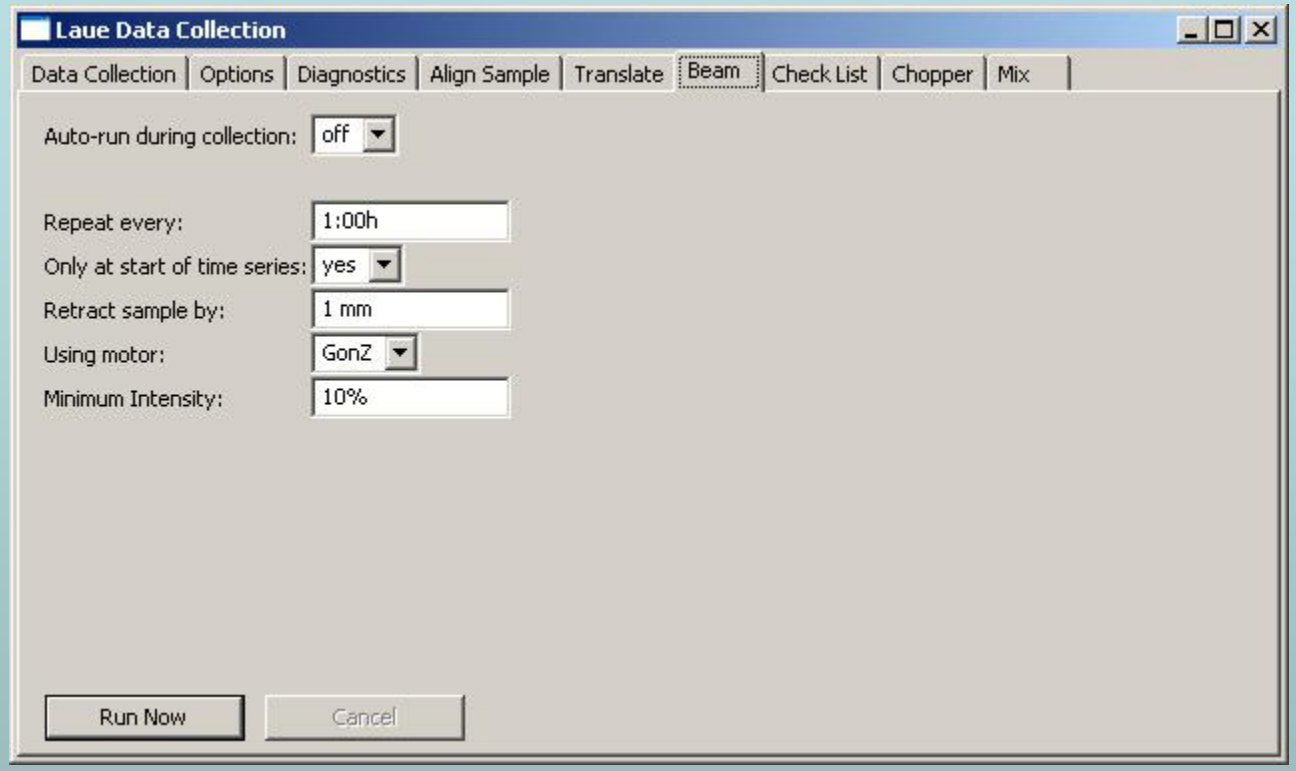
- Microchannel Plate (MCP)
- Silicon diodes
- Micro Ion Chamber
- Laser diodes

Real Time Beam Profiles



- Use sample viewing cameras to view the laser beam profile and size in real time
- Developing similar techniques for use with the x-ray beam

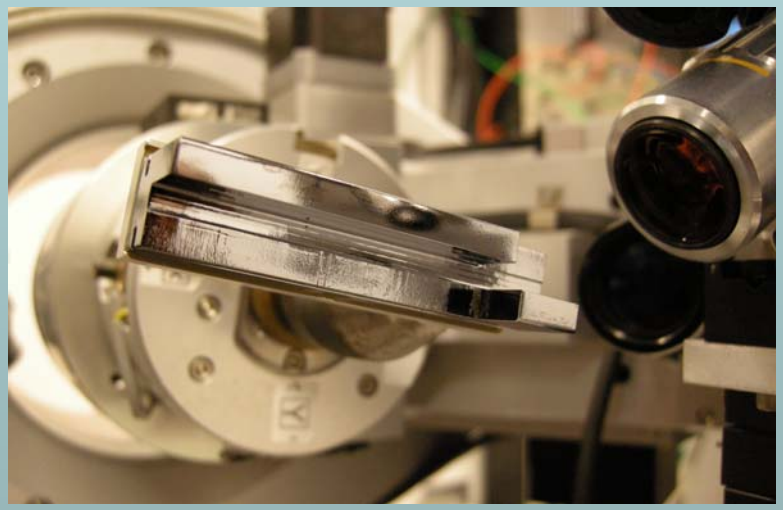
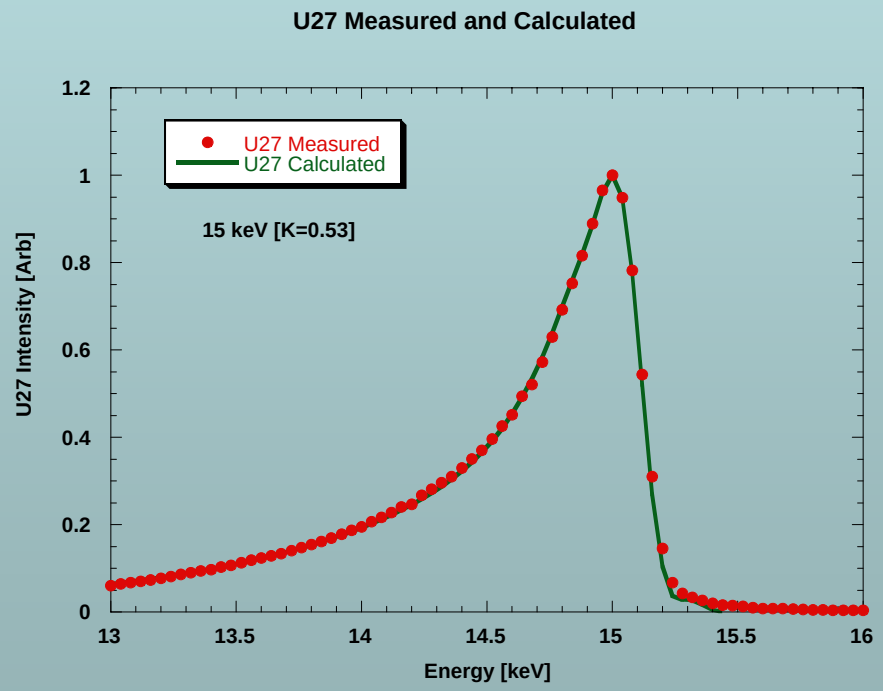
Automated X-ray Alignment



- X-ray beam can automatically be optimized at periodic intervals during the data collection

Incident Spectrum

- Needed for the data analysis
- Can be extracted directly from the diffraction data by Precognition software
- Can be measured using a channel cut monochromator mounted at the sample position



Channel Cut
Monochromator