

Data Reduction and Analysis

Part 1: Overview, Absorption

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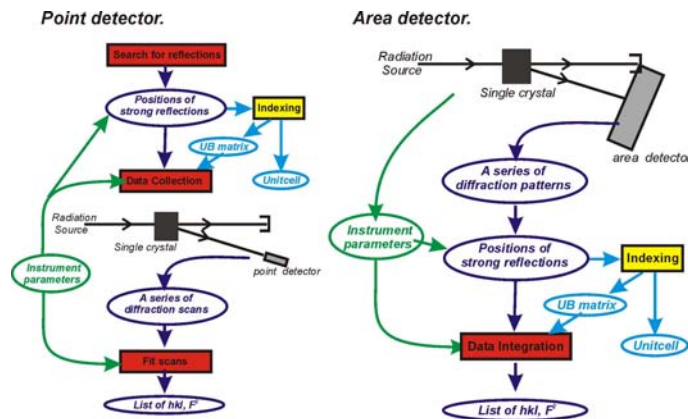
www.crystal.vt.edu

- Overview
- Absorption – the information needed

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Data collection....done and integrated!



- We now have a file:
- Integrated intensities
 - Indexing
 - Beam path information

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Intensity data quality:

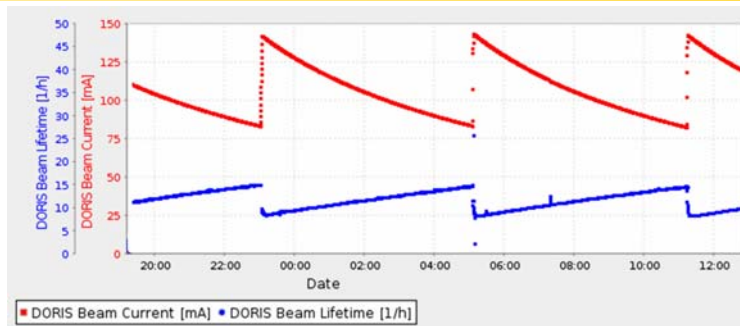
■ Intensity data is degraded by:

■ Limited access	Experiment design Integration
■ Low signal	
■ Scattering by cell components	Experiment design Integration
■ Shadowing by cell components	
■ Beam decay (synchrotron data)	Data Reduction
■ Absorption by cell components	
■ Diamond dips	

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Synchrotron: correction for primary beam



- independent monitoring of the primary beam intensity
- measurement of standard reflections (point detector)
- integration software takes into account the primary beam intensity
 - - information is fed into the program and used for scaling
 - - information is obtained from the average intensities of the frames
 - - information is obtained from the average intensities of the crystal

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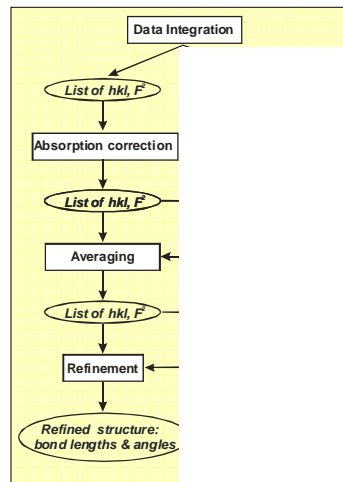
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Post-integration steps

- Data reduction
 - Critical for HP data
 - Absorption corrections
 - Averaging

- Refinement
 - Robust-resistant weighting
 - Restraints
 - Careful evaluation of outliers

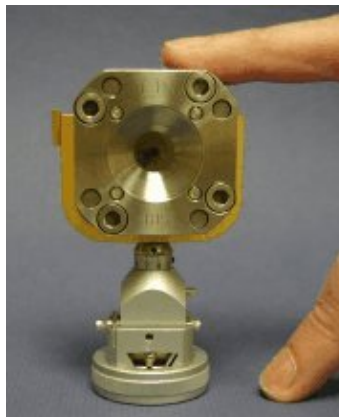
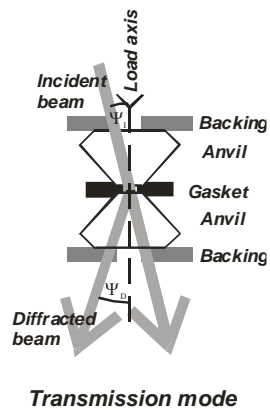
- Structure validation



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Absorption in the DAC: geometry

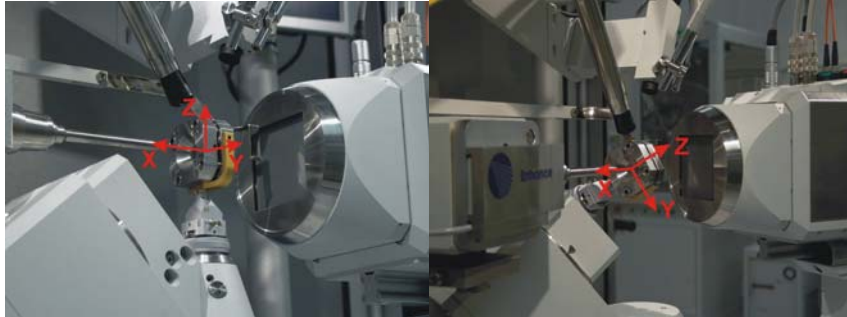


- We need beam path directions relative to DAC
 - (and thus relative to crystal as well)

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Absorption in the DAC: defining beam paths



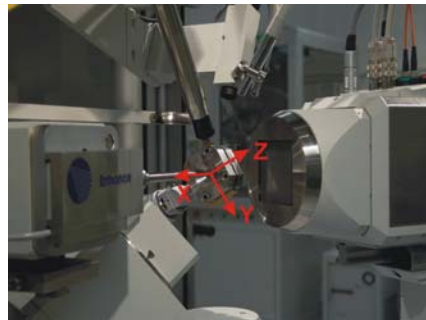
- The DAC is fixed to the goniometer head
- A natural Cartesian reference basis is therefore a basis fixed to the goniometer head (phi-axis system)
- Define beam paths on this axial system

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Defining beam paths

- Incident beam path
 - Only goniometer angles
- Diffracted beam path
 - Goniometer angles
 - Detector position
 - Spot position on the detector (area detectors only)
- Information can be provided in two ways:
 - PD: goniometer angles
 - PD & AD: Direction cosines of I-beam and D-beam
 - In SHELX hkl files, direction cosines relative to crystal axes!



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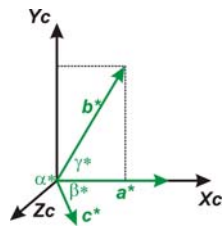
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The UB matrix

- Defines the orientation of the reciprocal lattice vectors of the crystal with respect to the goniometer:

$$\underline{h\phi} = \underline{UB} \cdot \underline{h}$$

- B** is a matrix that transforms reciprocal space vectors (hkl) from reciprocal lattice basis to an orthonormal basis:



$$\mathbf{B} = \begin{pmatrix} a^* & b^* \cos \gamma^* & c^* \cos \beta^* \\ 0 & b^* \sin \gamma^* & -c^* \sin \beta^* \cos \alpha^* \\ 0 & 0 & 1/c \end{pmatrix}$$

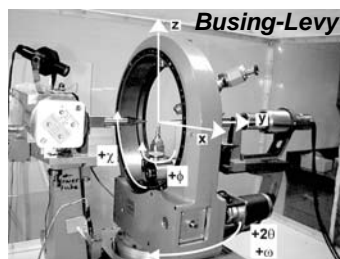
- The **B** matrix (or **UB**) is needed to convert the SHELX direction cosines from crystal system to phi-axis system (Allen et al, 2000)

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Defining beam paths with the UB matrix

- The definition of the phi-axis system, and thus **U** and **UB**, is different in different software!
 - Axis directions when diffractometer circles at zero

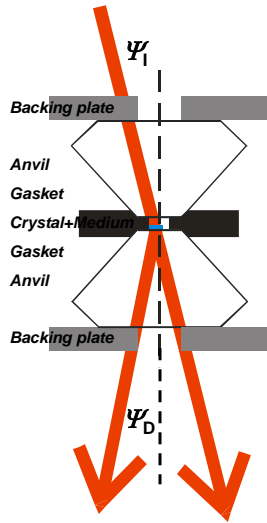


- And you need to know:
 - Type of goniometer (kappa or Eulerian)
 - Circle parities
 - Conventions used by your absorption program

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Absorption corrections



- **Absorption by**
 - Anvils + backing plates
 - Pressure medium
 - Crystal
 - Gasket
- **Psi scans or SADABS**
 - Not recommended
- **Integration over the crystal**
 - Non-analytic
 - Replace by summation over Gaussian grid of points on the crystal:

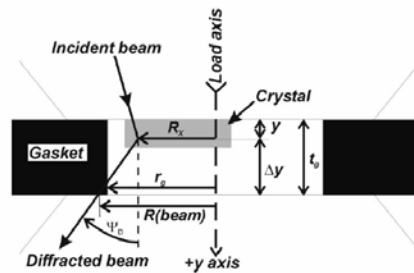
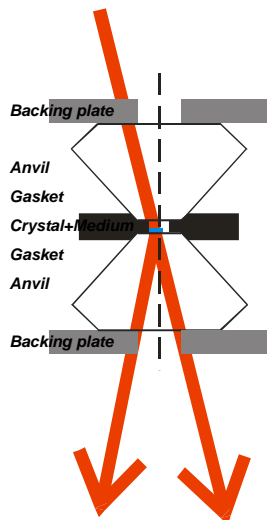
$$T = V^{-1} \int_V \exp\left[-\sum_i \mu_i(t_{iI} + t_{iD})\right] dV.$$

$$T = V^{-1} T(\Psi_I) T(\Psi_D) \int_V \exp\left[-\sum_i \mu_i(t_{iI} + t_{iD})\right] dV,$$

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Gasket shadowing



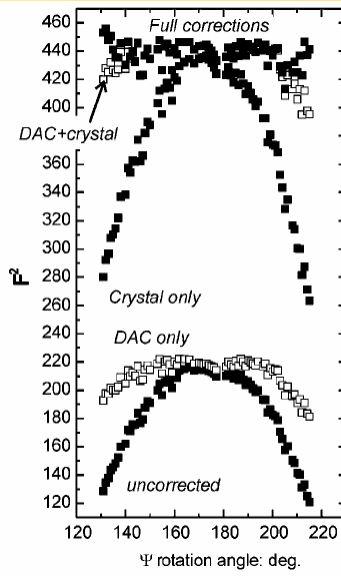
$$T = V^{-1} T(\Psi_I) T(\Psi_D) \int_V \exp[-\mu_x(t_{I,x} + t_{D,x})] \\ \times \exp[-\mu_g(t_{I,g} + t_{D,g})] \exp[-\mu_m(t_{I,m} + t_{D,m})] dV.$$

- **Need description of:**
 - Anvils + backing plates
 - Pressure medium
 - Crystal
 - Gasket

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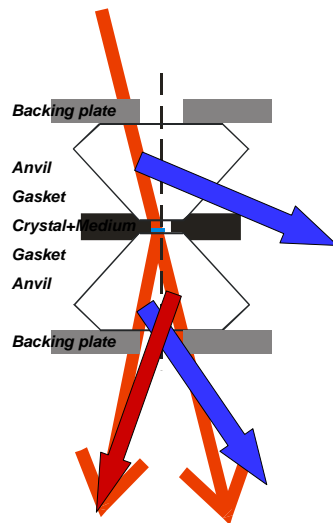
Test models with psi scans



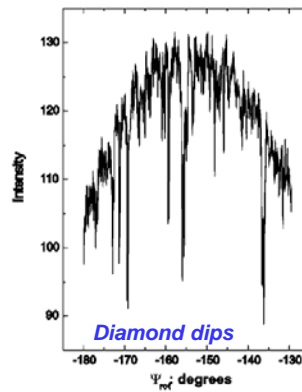
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Diamond reflections



- Diamond reflections in to detector
- Diamond dips



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