Integration of 2D diffraction images

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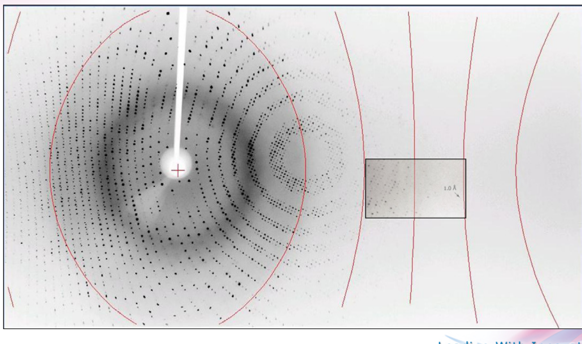
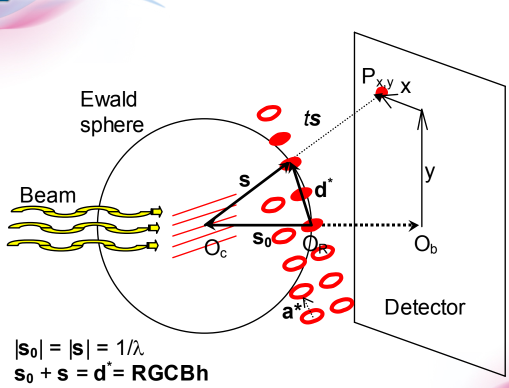
A diffraction image





$|\mathbf{s_0}| = |\mathbf{s}| = 1/\lambda$

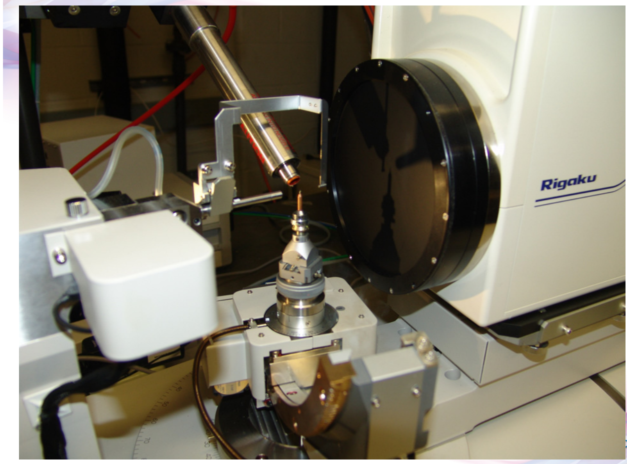
$\mathbf{s_0} + \mathbf{s} = \mathbf{d^*} = \mathbf{RGCBh}$

$\mathbf{s} = f(P_{x,y}, \phi, \text{Detector position})$

Diffraction math

$\mathbf{s_0} + \mathbf{s} = \mathbf{d^*} = \mathbf{RGCBh}$

h	Miller index (*h,k,l*)
B	Crystal orth. matrix (a,b,c,α,β,γ)
C	Crystal orientation matrix
G	Crystal goniometer matrix
R	Rotation axis matrix
d*	Reciprocal lattice vector
$\mathbf{s_0}$	Direct beam wavevector
s	Scattered beam wavevector



What you do

- Pick up crystal in loop, plunge into LN_2
- Put crystal on magnet on goniometer head and optical align
- Take a diffraction image or two
- Look at image(s) and decide whether to proceed
- Collect images, index, integrate, scale

Detector - Calibration

Stanton, et al. (1992) *J. Appl. Cryst*. **25**, 549-558.

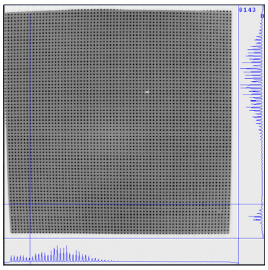
Dark current

Non-uniformity of response

Spatial distortion

Bad pixels

Zingers



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Reflections (from images)

- Find
  - X,Y, ϕ
- Index
  - Unit cell
  - Orientation
- Refine
  - Crystal
  - Detector
  - Source
- Predict / Strategy
  - Rot start, end
  - Completeness
- Integrate
  - *hkl*, Intensity, σ_I
  - Profile fitting
- Scale
  - Rmerge
  - $|\chi^2|$

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Integrate

- Predict reflection position
- Put box around reflection
- Assign pixels to Peak and Background
- Sum Peak, subtract Background
- Profile-fit
- Apply correction factors

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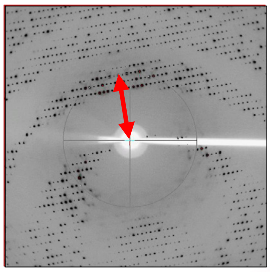
Direct beam position

- Direct beam shot
- Powder rings
- Ice rings
- Symmetry



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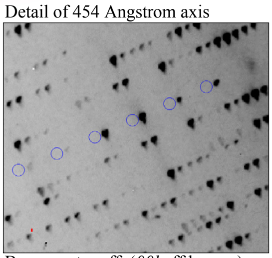
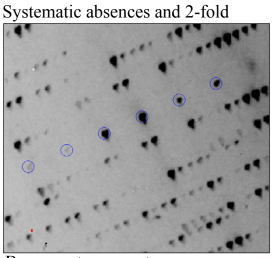
dtdisplay overlay

Detail of 454 Angstrom axis



Beam center off (*00l* off by one)

Systematic absences and 2-fold



Beam center correct

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Refine

- $\mathbf{s_o} + \mathbf{s_i} = \mathbf{RGCBh} = \mathbf{d^*_i}$
- Min $\chi^2 = \Sigma w_i \, (\mathbf{s_{i,obs}} - \mathbf{s_{i,calc}})^2$

  $= \Sigma w_i[\mathbf{s_{i,obs}} - (\mathbf{RGCBh} - \mathbf{s_o})]^2$
- Crystal (**B**, **C**): a, b, c, α, β, γ, Rot1, Rot2, Rot3
- Detector ($\mathbf{s_{i,obs}} = f(Det, X, Y, \phi, \boldsymbol{R})$)
  - Beam center, Distance, Rotations (2θ)
- Source ($\mathbf{s_o}$):Direction, wavelength

Leading With Innovation

Integrate

- Predict reflection position
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- Apply correction factors

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Integrate - Predict

$\mathbf{d^* = x_r = RGCBh}$ $\quad\quad$ $\mathbf{x_r - s_0 = s \rightarrow P(x,y)}$

Rocking curve

$R_i = 2L[\Delta d^* \cos\theta + (\delta\lambda/\lambda) d^* \sin\theta]$

Lorentz factor

$L = |\, 1.0 / (\, \mathbf{x_r} \cdot (\mathbf{r_1} \times \mathbf{s_0}))\, |$

Polarization factor

$p = 1 - [P_n * (\mathbf{s} \cdot \mathbf{s_n})^2 + (1 - P_n) * (\mathbf{s} \cdot \mathbf{n_p})^2]$

Oblique incidence correction factors

$O_1 = (1 - \exp(f_{obl})) / (1 - \exp(f_{obl} / \cos\alpha)$

$O_2 = \exp(f_{obl})) / \exp(f_{obl} / \cos\alpha)$

Kabsch (1988) *J. Appl. Cryst.* **21**, 916-924.
Zaleski, Wu & Coppens (1998) *J. Appl. Cryst.* **31**, 302-304.

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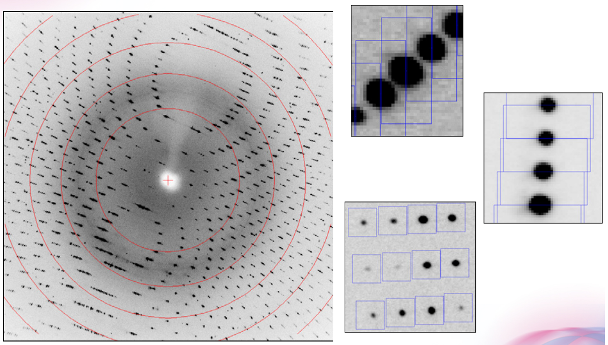
Integrate - Put box around



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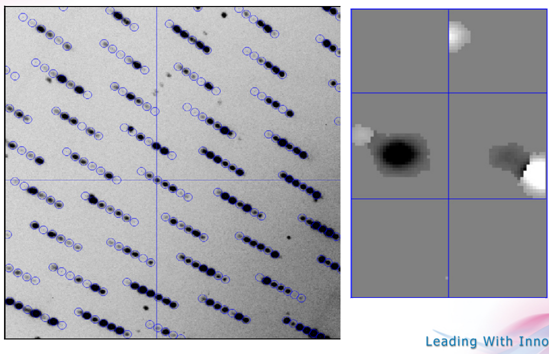
Integrate - Put box around



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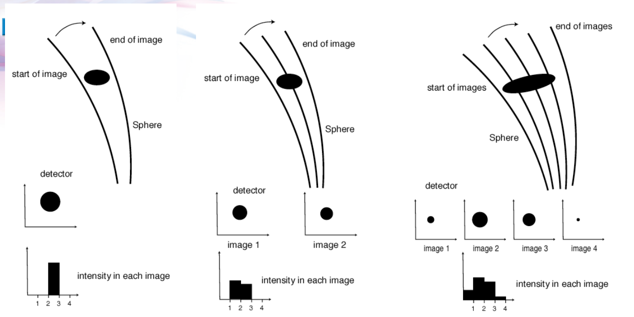

A fully-recorded spot is entirely recorded on one image

Partials are recorded on two or more images

“Fine-sliced” data has spots sampled in 3-dimensions

Perhaps best processed with a 3D program (eg d*TREK, XDS)

Elspeth Garman, Oxford

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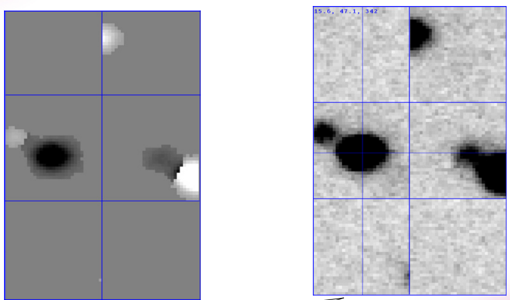
Integrate - Background



Background: Least-squares fit to a plane

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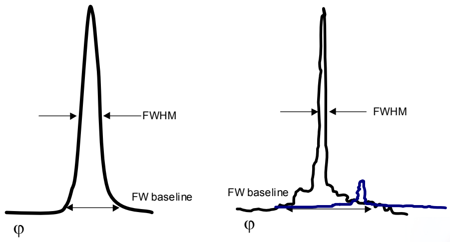
Rigaku

Refine (crystal mosaicity)

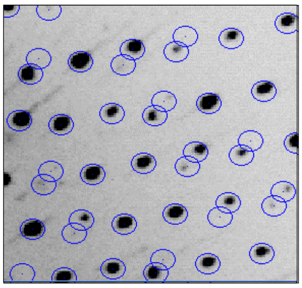
Rocking curve

$$R_i = 2L[\Delta d^* \cos\theta + (\delta\lambda/\lambda) d^* \sin\theta]$$



Rigaku

Refine (crystal mosaicity)



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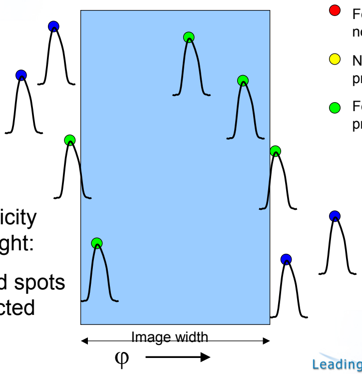
Rigaku

- Not in image, not predicted
- Found in image, not predicted
- Not in image, predicted
- Found in image, predicted



Mosaicity just right:

Found spots predicted

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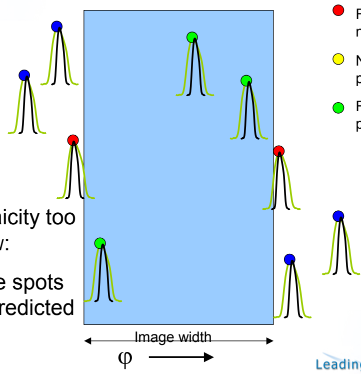
Rigaku

- Not in image, not predicted
- Found in image, not predicted
- Not in image, predicted
- Found in image, predicted



Mosaicity too narrow:

Some spots not predicted

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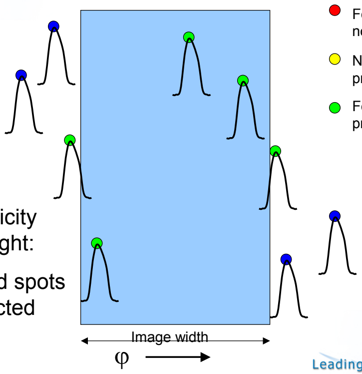
Rigaku

- Not in image, not predicted
- Found in image, not predicted
- Not in image, predicted
- Found in image, predicted



Mosaicity just right:

Found spots predicted

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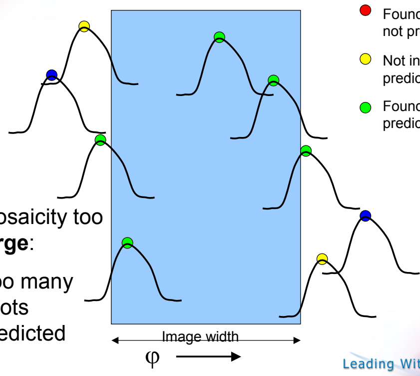
Rigaku

- Not in image, not predicted
- Found in image, not predicted
- Not in image, predicted
- Found in image, predicted



Mosaicity too **large**:

Too many spots predicted

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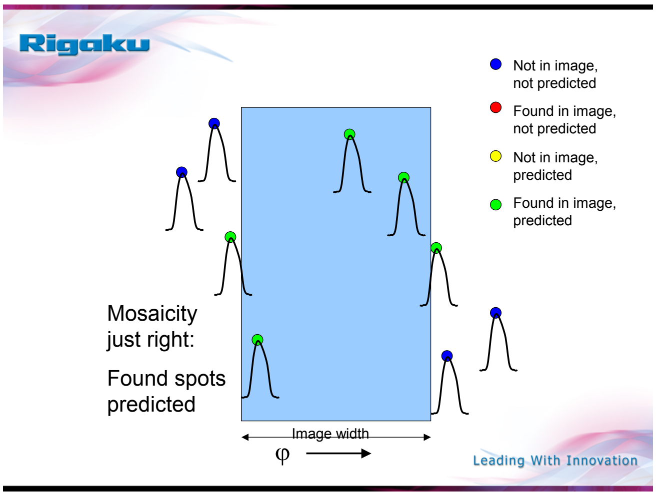
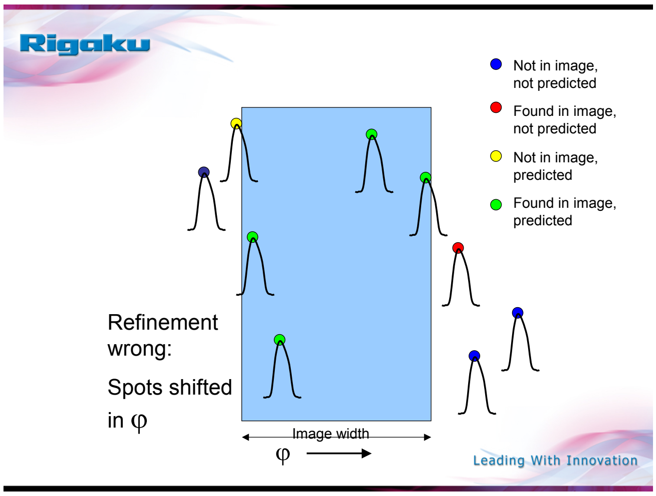
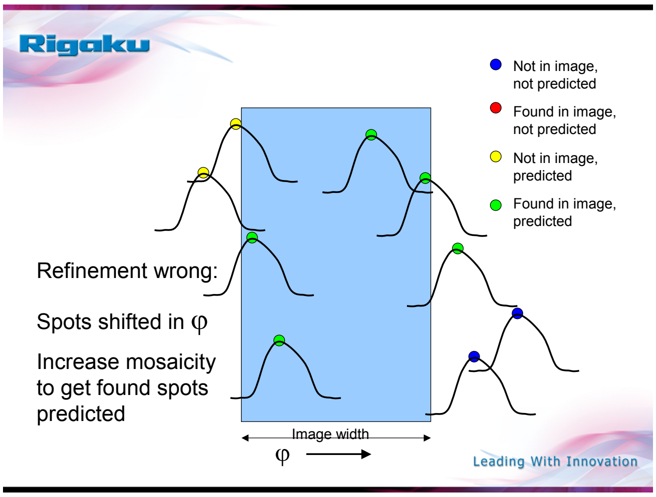
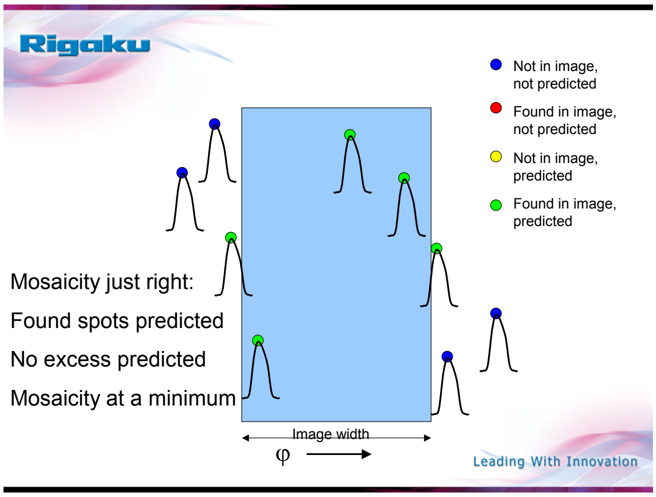


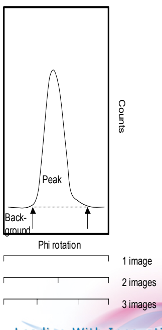






Images - Expectations

- Poisson counting
  Quantity Q, var(Q) = Q, $\sigma(Q) = Q^{1/2}$
- Simple integration
  Intensity = Σ(Peak - Background)
  var(Int) = var(Peak)
  + var(Background)



Integrate - Profile fitting

Two major assumptions

1. Reflections have the same profile:
   Same shape
   Same distribution
2. Reflections are predicted accurately

Diamond (1969) *Acta Cryst* **A25**, 43-55.
Ford (1974) *J Appl Cryst* **7**, 555-564.
Rossmann (1979) *J Appl Cryst* **12**, 225-238.
Kabsch (1988) *J Appl Cryst* **21**, 916-924.

Integrate - Profile fitting

Integrate - Profile fitting

Integrate - Profile fitting

Integrate - Profile fitting

Reference profile = Superposition of pixel values

Poor predictions

= Poor superposition

Good predictions

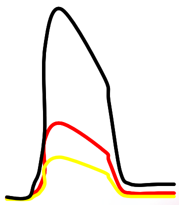
= Good superposition

Integrate - Profile fitting

Integrate - Profile fitting

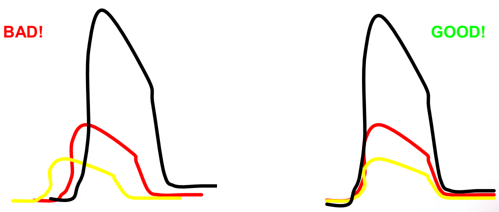
Two major assumptions

1. Reflections have the same profile:
   - Same shape
   - Same distribution
2. Reflections are predicted accurately

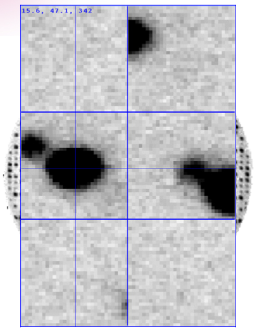


Integrate - Profile fitting II

Bad predictions = Bad reference profile



Integrate - Profile fitting III



$$f = \sum_i \frac{(p_i - Ic_i)^2}{v_i}$$

$$I_{prof} = \frac{\sum_i \frac{p_i c_i}{v_i}}{\sum_i \frac{p_i^2}{v_i}}$$

Variations

Integrate in 2D, later post-refine and sum partials
MOSFLM, denzo, HKL2000

Integrate in 3D, refine as you go along
XDS, MADNES, d*TREK

Box & spot size – user input or automatic; fixed or plastic

More details

How much are Bragg peaks rasterized?
What about powder rings such as from ice?
Wide slice 5 degree images?
Or fine slice 0.3 degree images?
Or 0.5 degree images?
What about systematic and erratic errors?
Bad pixels, shadows, moving shadows – mask them out
Zingers
$K\alpha_1/K\alpha_2$ at high 2θ - shift vectors are calculated and applied
Scale and get statistics as you go along
Update refinement continually
Detector gain
Spot overlap

Which bottle has naturale water and which has sparkling?

Scaling

- Correction of systematic errors
- Outlier rejection
- Validation of sigmas

$$\sigma_{adj}^2 = (\sigma_{in} E_{mul})^2 + (I_{in} E_{add})^2$$

Scaling

Correction of systematic errors

- different crystal volumes
- different exposure times
- different detectors
- radiation damage
- wavelength dependent factors
- different or fluctuating source intensities
- different absorption due to different paths through the crystal and other matter