What data need to be measured?

Scattering from:

specimen in its container

empty specimen container

standard or calibration specimen

What data need to be measured?

Scattering from:

- specimen in its container
- empty specimen container
- standard or calibration specimen
- dark count (noise)
- detector dead time
- background

What data need to be measured?

Scattering from:

specimen in its container

empty specimen container

standard or calibration specimen

dark count (noise)

detector efficiency

background

transmissions of each specimen

incident beam intensity

What data need to be measured?

Scattering from:

specimen in its container

empty specimen container

standard or calibration specimen

to get absolute intensity measurements, standard must have known scattering intensity

What data need to be measured?

Scattering from:

specimen in its container

empty specimen container

standard or calibration specimen

dark count (noise)

includes errant x-rays - measure w/ strongly absorbing material in place of specimen

may vary with time

What data need to be measured?

Scattering from:

specimen in its container

empty specimen container

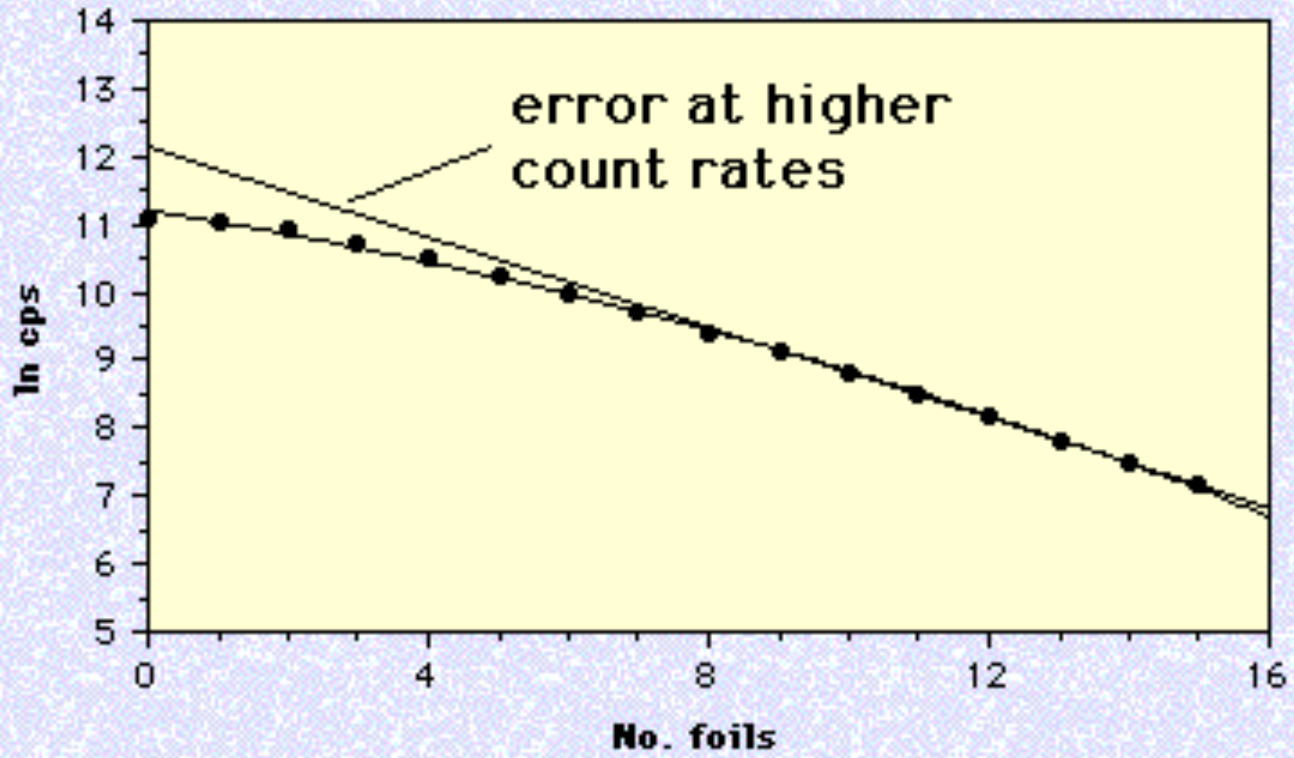
standard or calibration specimen

dark count (noise)

detector efficiency

scale each datum to that of a detector w/ constant efficiency

What data need to be measured?



$$I/I_o = \exp(-\mu * x)$$

$$\ln I = \ln I_o - \mu * x$$

straight line

$$\text{true cps} = \frac{\text{observed cps}}{1 - \tau \cdot \text{observed cps}}$$

What data need to be measured?

Scattering from:

- specimen in its container
- empty specimen container
- standard or calibration specimen
- dark count (noise)
- detector efficiency
- background
  - e.g., pure solvent, or pure matrix material

What data need to be measured?

Scattering from:

- specimen in its container
- empty specimen container
- standard or calibration specimen
- dark count (noise)
- detector efficiency
- background
- transmissions of each specimen
  - cut beam intensity, measure direct beam I w/ & w/o specimen

What data need to be measured?

General procedure:

1. Scale all data to incident beam monitor (synchrotron beam decays w/ time)

What data need to be measured?

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2. Correct for detector efficiency

What data need to be measured?

General procedure:

1. Scale all data to incident beam monitor (synchrotron beam decays w/ time)

2. Correct all data for detector efficiency

3. Correct all data for empty cell and dark count

Now have:

$$I^{corr}_{specimen} = (I_{specimen} - I_{dark}) - (T_{cell\ full}/T_{cell\ empty})(I_{cell\ empty} - I_{dark})$$

$$I^{corr}_{bkgrd} = (I_{bkgrd} - I_{dark}) - (T_{bkgrd}/T_{cell\ empty})(I_{cell\ empty} - I_{dark})$$

$$I^{corr}_{std} = (I_{bkgrd} - I_{dark}) - (T_{std}/T_{cell\ empty})(I_{cell\ empty} - I_{dark})$$

What data need to be measured?

General procedure:

$$I^{corr}_{specimen} = (I_{specimen} - I_{dark}) - (T_{cell\ full}/T_{cell\ empty})(I_{cell\ empty} - I_{dark})$$

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$$I^{corr}_{std} = (I_{bkgrd} - I_{dark}) - (T_{std}/T_{cell\ empty})(I_{cell\ empty} - I_{dark})$$

4. Get absolute *I*s

$$I_{abs} = (I_{specimen}\, f(\mathbf{q}) / I_{std})\ (t_{std}\, T_{std} / t_{specimen}\, T_{specimen})$$

$$I_{bkgrd} = (I_{bkgrd}\, f(\mathbf{q}) / I_{std})\ (t_{std}\, T_{std} / t_{bkgrd}\, T_{bkgrd})$$

What data need to be measured?

General procedure:

$$I^{corr}_{specimen} = (I_{specimen} - I_{dark}) - (T_{cell\ full}/T_{cell\ empty})(I_{cell\ empty} - I_{dark})$$

$$I^{corr}_{bkgrd} = (I_{bkgrd} - I_{dark}) - (T_{bkgrd}/T_{cell\ empty})(I_{cell\ empty} - I_{dark})$$

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$$I_{abs} = (I_{specimen}\, f(\mathbf{q}) / I_{std})\ (t_{std}\, T_{std} / t_{specimen}\, T_{specimen})$$

$$I_{bkgrd} = (I_{bkgrd}\, f(\mathbf{q}) / I_{std})\ (t_{std}\, T_{std} / t_{bkgrd}\, T_{bkgrd})$$

5. Subtract bkgrd

What data need to be measured?

Background treatment - complex

need to consider what bkgrd should be -

w/ solns or dispersions, bkgrd usually includes scattering

from solvent or dispersion medium & sample cell

What data need to be measured?

Background treatment - complex
- need to consider what bkgrd should be -
- w/ solns or dispersions, bkgrd usually includes scattering from solvent or dispersion medium & sample cell

Standard specimen
- reproducible
- stable
- known scattering data
- scattering must be high
- isotropic scattering

Instrument resolution

See *J. Appl. Cryst.* (1990). **23**, 321–333

Need to consider

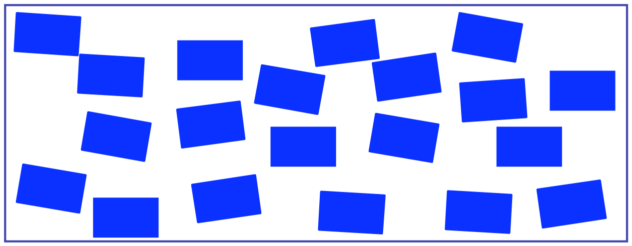
wavelength spread
collimation effects
detector resolution

Instrument resolution

Wavelength spread

Lab x-rays + monochromator

Monochromator crystal set to scatter characteristic line - wavelength spread is result of combined effects of
- natural line width
- mosaic spread of monochromator crystal
- collimation before & after crystal



Instrument resolution

Wavelength spread

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Synchrotron spectrum cont<u>s</u> - wavelength spread determined only by mosaic spread of monochromator & collimation

Instrument resolution

Wavelength spread

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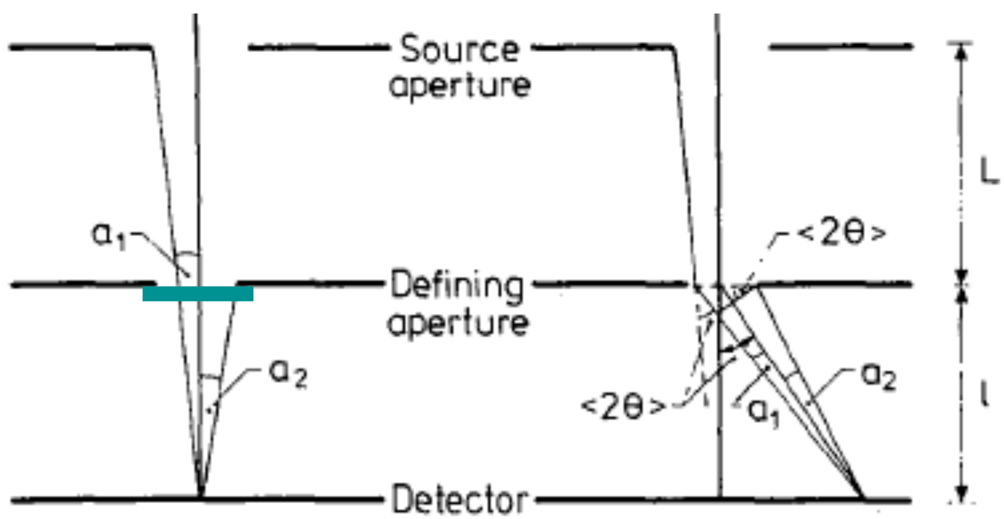
Synchrotron spectrum cont$^{\underline{s}}$ - wavelength spread determined only by mosaic spread of monochromator & collimation

Both conventional & synchrotron X-ray sources - wavelength spread ~$\Delta\lambda/\langle\lambda\rangle$ typically $<10^{-3}$ & neglected in most cases

Instrument resolution

Collimation



Instrument resolution

Detector

3 contributions to spatial resolution

division of detector into pixels

method of detection

method of position determination

latter 2 dominant

Instrument resolution

Combined resolution fcn

$$R(\mathbf{q}, \langle \mathbf{q} \rangle) = (2\pi\sigma\sigma_{C2})^{-1} \times \exp\left[-\frac{1}{2}\left(\frac{(q_1 - \langle q \rangle)^2}{\sigma^2} + \frac{q_2^2}{\sigma_{C2}^2} \right) \right]$$

$$\sigma_{C2} = \langle k \rangle \Delta\beta_2 / 2(2 \ln 2)^{1/2} \quad \Delta\beta_2 = 2r_1/L - \frac{1}{2}\frac{r_2^2}{r_1}\frac{\cos^2\langle 2\theta \rangle}{l^2 L} \times (L + l/\cos\langle 2\theta \rangle)^2 \qquad \text{for } a_1 \geq a_2$$

σ - parameter related to FWHM of function

ex: $\sigma_\lambda = \Delta\lambda / [2(2 \ln 2)^{1/2}]$

Instrument resolution

Combined resolution fcn

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