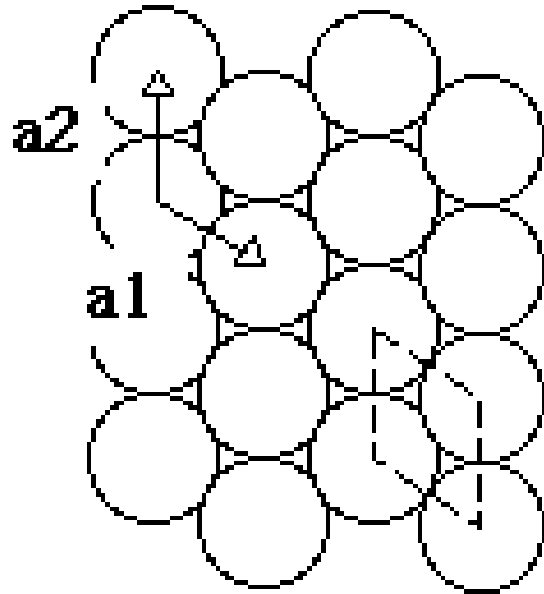
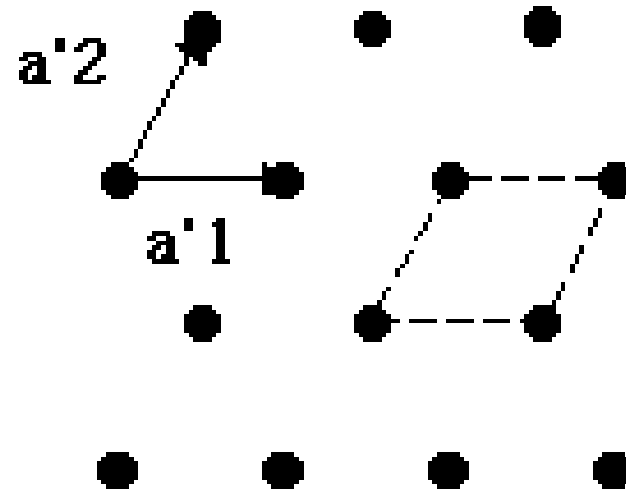


Crystal lattice

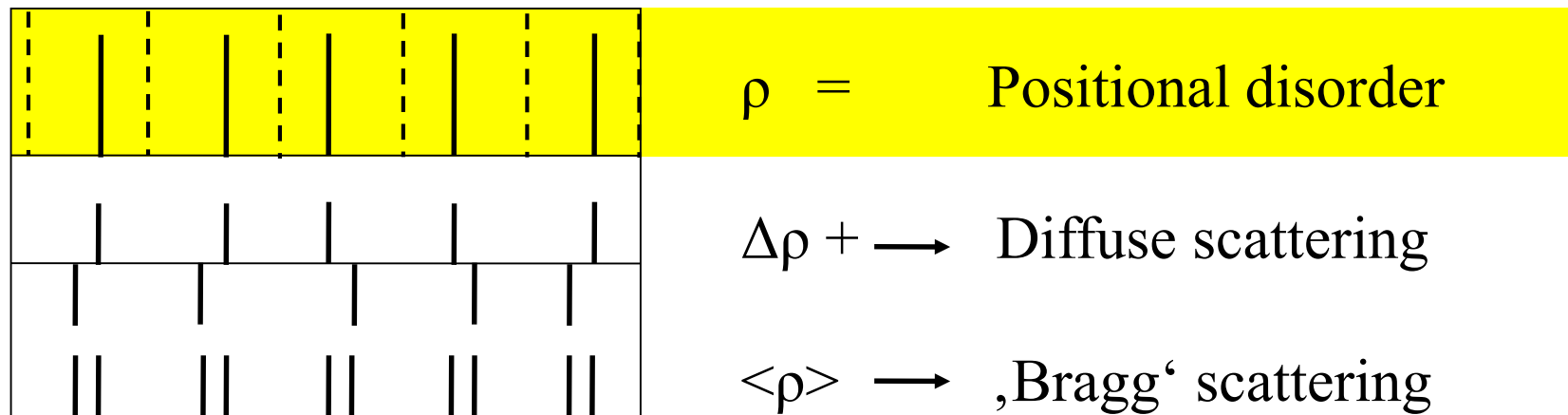
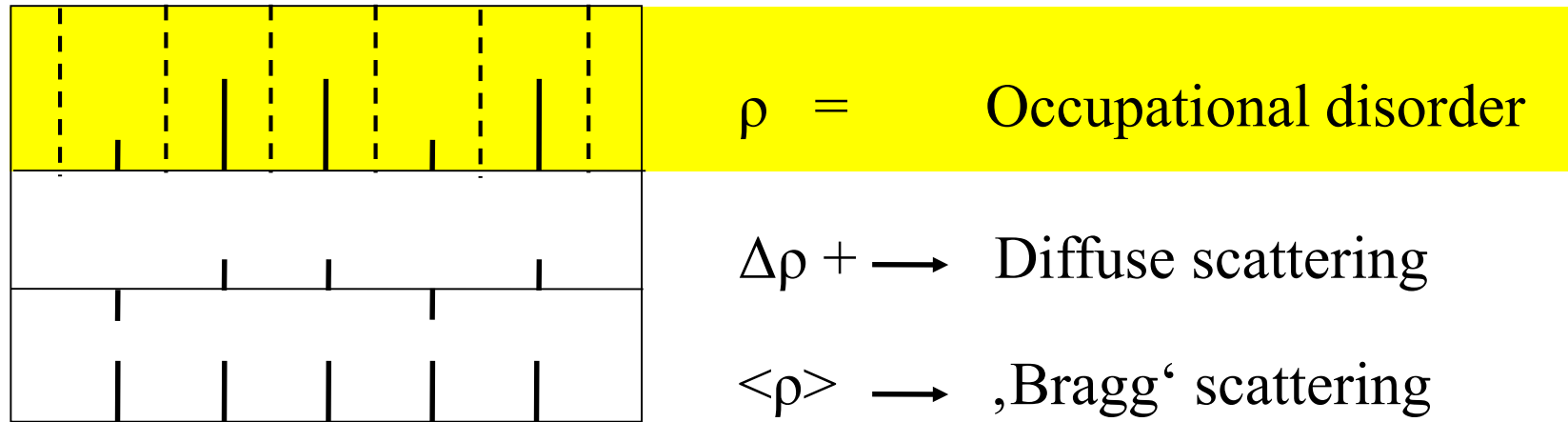


FT
→

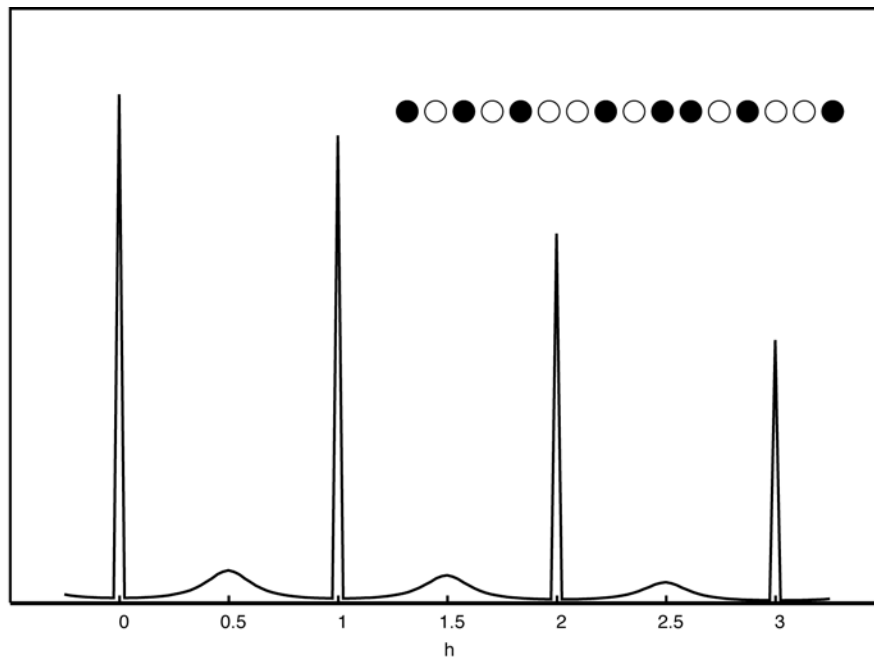
Reciprocal lattice



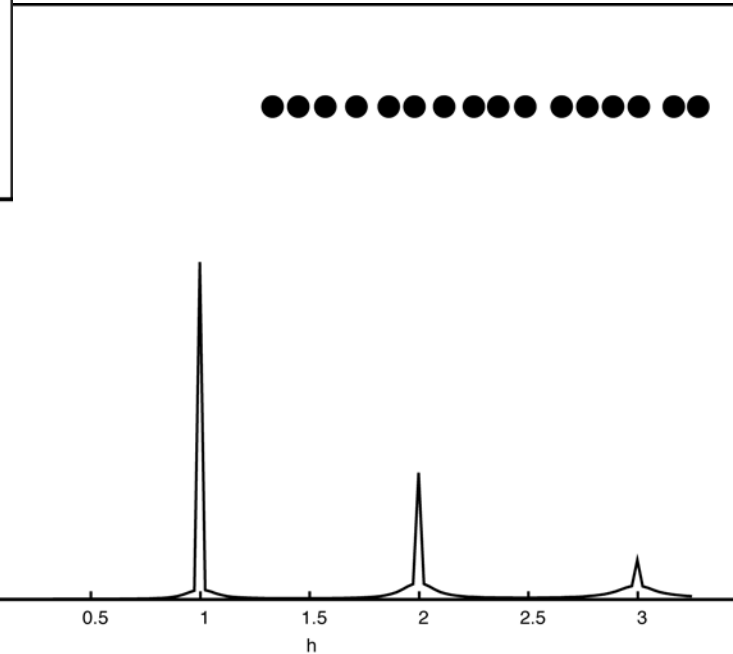
Schematic representation of relation between disorder and scattering



Bragg diffraction and diffuse scattering



Occupational
disorder



Positional
disorder

**A caveat concerning
routine crystal structure analysis from Bragg data**

**A crystal structure derived from Bragg data only
shows the scattering density
of a single unit cell which represents an average
over the time of the experiment and all unit cells of the
crystal.**

Which information is needed?

- Connectivity only?
- Geometry to within 0.01 Å or better?
- Optimal description of the average structure of disordered parts?
- Qualitative information from diffuse scattering?
- Quantitative information from diffuse scattering?

Plan experiment accordingly!

Required size and quality of crystals? Careful mounting of a single single-crystal on a loop!

Wave length (minimize absorption and consequent, unwanted fluorescence background)?

Attainable resolution of data?

Precision of data, signal/noise, exposure time?

Preliminary experiment to check the chosen conditions!

If you do not know the best conditions for your particular sample, explain to the group crystallographers or the beam line scientists what it is you want to do and ask for help with your specific problem.

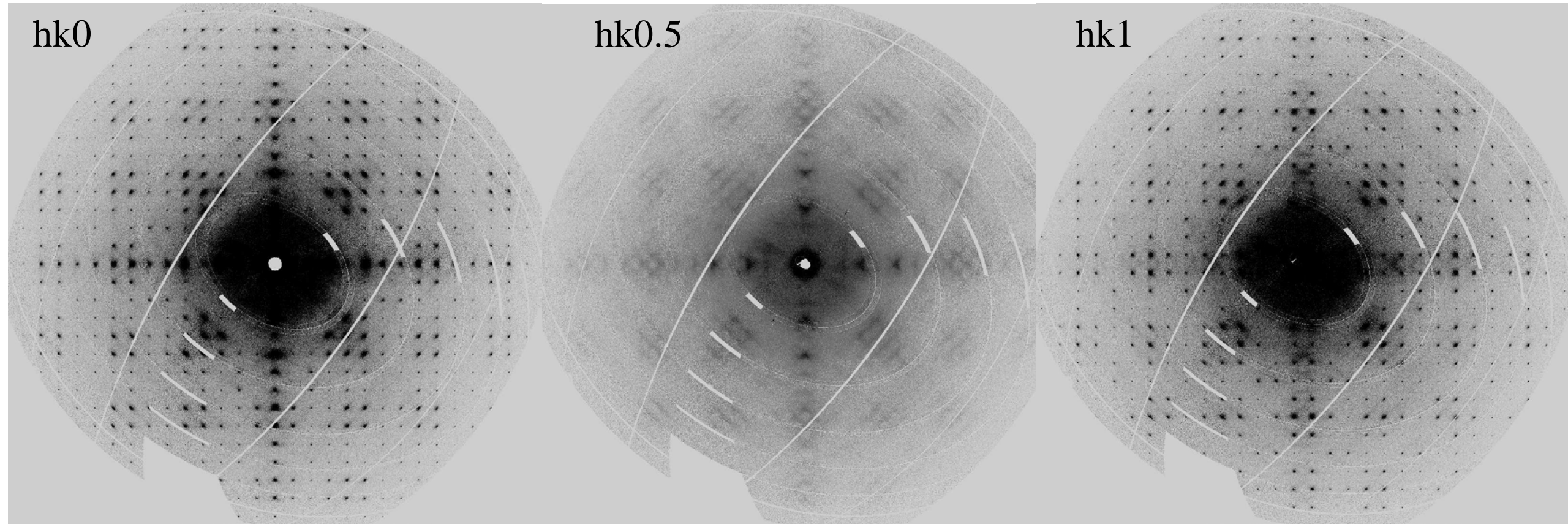
Extract the best possible data from the experiment especially if your sample is of poor quality!

Careful determination of orientation of crystal on diffractometer (orientation matrix) and optimal cell constants. Optimal integration of the Bragg intensities.

Inspect your data visually. Reconstruction of precession pictures, i.e. sections through reciprocal space including and excluding Bragg layers

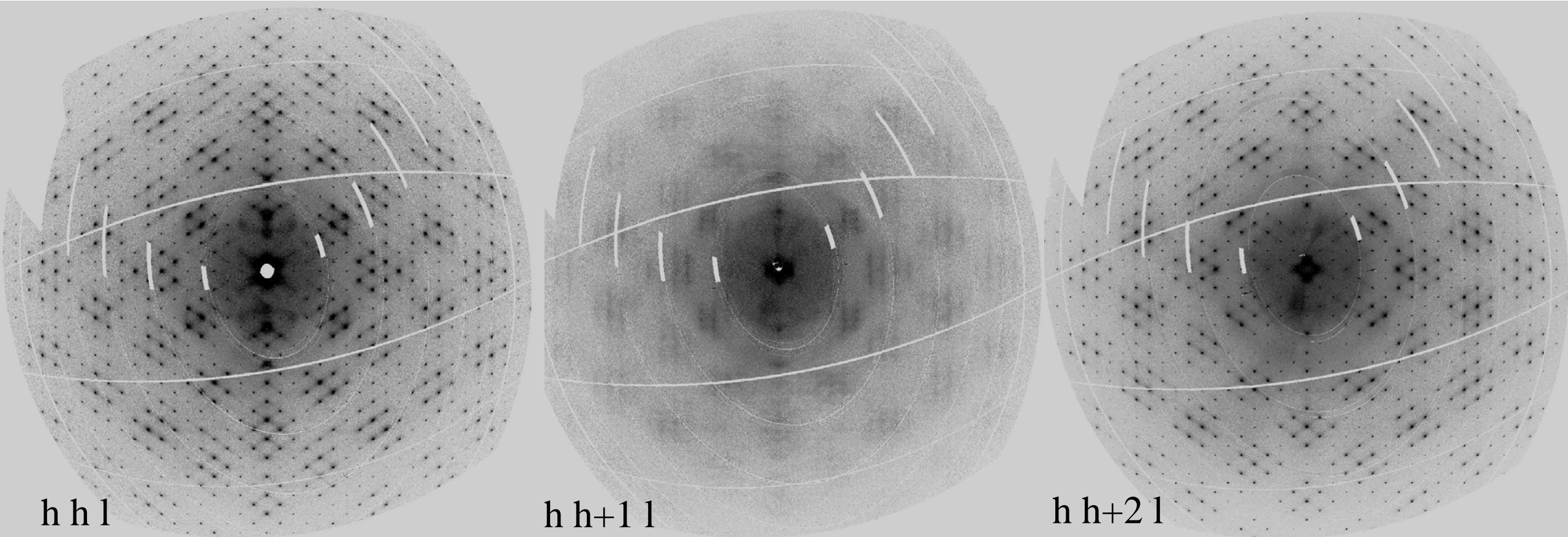
Knowing about the quality of the data may save a lot of time when interpreting them!

Inspecting diffraction/scattering data



If no diffuse can be seen, this does not mean that there is no disorder.
The diffuse may simply be too weak to be seen under the chosen conditions.

Inspecting diffraction/scattering data



If no diffuse can be seen, this does not mean that there is no disorder.
The diffuse may simply be too weak to be seen under the chosen conditions.

Organization of remaining lectures

EXPERIMENTAL ASPECTS: WHAT TO CONSIDER BEFORE YOU START MEASURING AND MODELING YOUR DIFFRACTION DATA

- lecture 2 by Simon Teat:
Factors affecting data quality and consequent structure modeling.
Recognizing and optimizing them.
- lecture 3 by Kevin Gagnon:
Overview of the nuances of getting an optimal data set, from crystal picking to data processing. Recognizing when to quit, and when you need help.

Organization of remaining lectures

LEAST-SQUARES REFINEMENT FOR COMPLEX DISORDERED STRUCTURES

- lecture 4: Modeling electron density: the model and its parameters, the essence of least-squares refinement, its potential and pitfalls. Illustrated with examples.
- lecture 5: Modeling disorder: use of analytical and geometric non-diffraction information. Illustrated with examples. Reciprocal space criteria for assessing the quality and reliability of an electron density model. R-factors, statistics for $|F_{\text{obs}}^2 - F_{\text{model}}^2|$, systematic trends in the list of 'most disagreeable reflections
- lecture 6: Direct space criteria for assessing the quality and reliability of an electron density model. Standard uncertainties, correlation coefficients, Q-peaks vs contoured difference Fourier synthesis. Modeling disorder vs the SQUEEZE procedure. Illustrated with examples.

Suggested Reading

- 'Comparison of silver and molybdenum microfocus X-ray sources for single-crystal structure determination' L. Krause, R. Herbst-Irmer, G.M. Sheldrick, D. Stalke, J. Appl. Cryst. (2015). 48, 3–10 (good info on data processing and data quality)
- 'Optimizing the model of a crystal structure' H.B. Bürgi (Lecture notes from the Zürich school of Crystallography 2013. See <http://yaghi.berkeley.edu/links.html#lectures>)
- 'Crystal Structure Refinement - A Crystallographer's Guide to SHELXL'. P. Muller, R. Herbst-Irmer, A. Spek, Th. Schneider, M. Sawaya, OUP 2006
- 'PLATON SQUEEZE: a tool for the calculation of the disordered solvent contribution to the calculated structure factors'. A. L. Spek, Acta Cryst. C 71 (2015) 9–18.

Organization of remaining lectures

BEYOND THE BRAGG DATA: STRATEGIES AND EXAMPLES FOR PROCESSING AND INTERPRETING DIFFUSE SCATTERING DATA

- lecture 7: Data processing between the Bragg reflections (spurious Bragg peaks, powder rings, other artefacts, symmetry averaging). Information from the 3D-PDF and 3D- Δ PDF functions.
- lecture 8: Monte Carlo (MC) modeling of disordered structures. Differential evolution for optimization of MC parameters. Statistical description of disordered structures.