Understanding Poor Data

A non-Uk, non-Kcod user, non-CRYSTALS user e-mailed to say that he had was having problems refining a structure, and could CRYSTALS do a better job?

Crystal Data\textsuperscript{2}

C\textsubscript{6}H\textsubscript{10}F\textsubscript{35}O\textsubscript{17}P\textsubscript{4}Ru\textsubscript{6}
P2\textsubscript{1}/a, 21.54 12.54 28.76 93.52
17845 reflections, 155 non-H atoms
Two different molecules per asymmetric unit
R\textsubscript{1} = 12.4\% (3258 with F>4\sigma(F))

Simple plots may indicate the over-all quality of the data. Characteristic shapes are easily recognised.

- Completeness vs resolution: Low completeness in high resolution shells often points to failures in the reflection searching process, since zero or negative intensities should count towards the completeness.
- Wilson Plot: The gradient of the Wilson plot is a measure of the overall temperature factor, and should normally correspond to a value of about 0.02 to 0.05. A very low or negative value may be symptomatic of neglect of a theta dependent absorption correction, or a serious failure in the data processing.
- Intensity distribution vs resolution: Most of the reflection in the "Poor Data" set have |F|<3\sigma(F). Though weak data can be important, this is only significant if it overpowers the contributions from overlapping reflections.

The scatter plot of the systematic absences (above) is reasonably symmetric about zero. There are no strong reflections, whose presence might have indicated either an incorrect space group or twinning.

A plot of |<Fo-Fc>| against azimuth and declination (below, left) shows a systematic variation over reciprocal space. This is consistent with a failure in the multi-scan inter-frame scaling, perhaps indicating inadequate redundancy, or a slow movement of the crystal with time.


Refinement

- Low completeness alone does not indicate a bad data set. In the absence of space group ambiguities, disorder, twinning etc, completeness as low as 70% can still give a reliable structure. When it is due to weak data, there will be problems with the refinement.
- The Wilson Plot and the text diagnostic (right), suggest that the high angle data are pretty worthless. These suggest trying a refinement using only the data with Theta < 18.5º.

Disorder

If disorder seems likely, the electron density map may indicate whether a discrete-atom model is appropriate (above). However, there is no a priori reason why a resolved atomic model should be suitable.

In the metal organic framework material (below), a packing diagram shows that the 5 O-peaks form part of a chain running right through the crystal. The difference density in this region is a "soup", best represented with the discrete Fourier transform of the electron density in the cavity between the well-resolved atoms.

If you would like to try the beta version, contact us by e-mail.

\textsuperscript{†} Richard Cooper has recently re-worked the graphical interface to give dramatically improved performance with most graphics cards. This will be available together with lots of other goodies shortly.

12402 Reflections processed

Minimum ratio = 0.753 at Phi=0.0, Major=25.6 Minor=17.6
Maximum ratio = 1.153 at Phi=0.0, Major=25.6 Minor=0.0
Average ratio = 0.8965

A new version of CRYSTALS will be released late Autumn 2009†