Identifying problems in your single crystal diffraction data

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Structure determination from single crystal diffraction data occasionally runs into problems during the refinement, analysis and publication stages: either the headline quality measures (R-values, residual density) are worse than anticipated or online validation tools [1] throw up warnings which require investigation, correction, or justification.

Crystal structure data – typically in .ins and .hkl format – can be easily imported into the crystal structure analysis package CRYSTALS [2] where a number of useful plots and analyses of the data are available. We find that these tools enable a rapid overview and identification of problems, whether we are looking at our own data, refereeing data for journals, or assisting colleagues with problematic datasets.

Problems can arise due to shortcomings in the model or errors and omissions in the data itself. Examples presented here include: (i) identification of problems from the surprisingly underused plot of calculated against observed structure factor magnitude, which straightforwardly finds outliers, twinning, and extinction as shown in Fig. 1; (ii) use of Wilson plot and systematic absence data to find space group errors and the resolution limits above which data is poorly integrated as shown in Fig. 2; (iii) use of contoured generalised Fourier sections to suggest improvements in parameterisation of problems; (iv) identifying sources of disagreement between different methods for determining absolute structure [3].

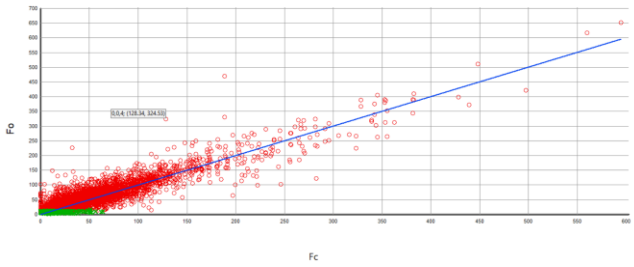

Figure 1. Observed against calculated structure factor magnitudes. This old dataset had several clearly contaminated observations consistent with undetected twinning. The result was an incorrect and complicated disordered structural model.

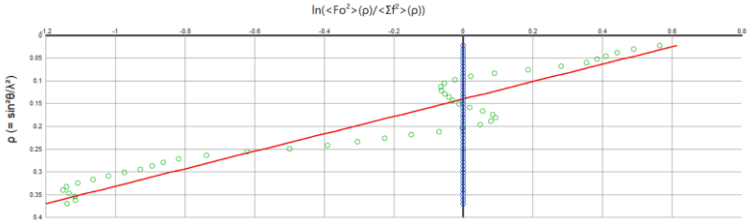

Figure 2. The Wilson plot identifies a resolution limit above which structure factor intensities are being systematically overestimated, which can lead to underestimation of displacement parameters. In the figure above, the limit is approximately $\sin^2\theta / \lambda^2 > 0.34$ so the impact on the refinement will be minimal.

Examples and discussion of the causes of these problems will be presented. Interested crystallographers or reviewers are encouraged to install the CRYSTALS package which is available from http://www.xtl.ox.ac.uk/tag/crystals-release.1.html or https://github.com/ChemCryst/crystals/releases

[1] Spek A. L. (2020) *Acta Cryst.* E**76**, 1-11.

[2] Betteridge P. W., Carruthers J. R., Cooper R. I., Prout K. & Watkin D. J. (2003) *J. Appl. Cryst* **36**, 1487.

[3] Watkin D. J. & Cooper R. I. (2016) *Acta. Cryst.* B**72**, 661-683.

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