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Crystals: Refinement and Validation Tools. David J. Watkin[a], Richard I. Cooper[b], Amber L. Thompson[a]. [a]*Chemical Crystallography, Department of Inorganic Chemistry, Oxford.* [b]*oXray Ltd., Oxford.*
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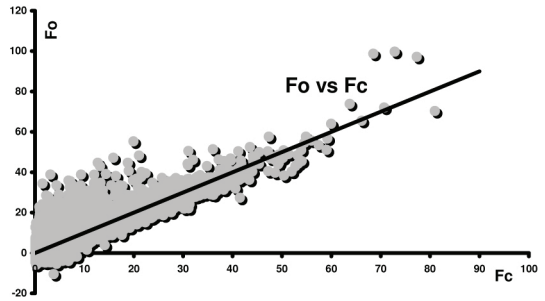
The basic technology for the refinement of small molecule structures has remained largely unchanged for 30 years. A standard refinement program will use a least-squares method to optimise parameters and have a range of constraints and restraints available to deal with troublesome problems. The programmers' task includes providing tools to help the users identify and resolve problems.
One kind of troublesome problem is that in which the R-factor is inexplicably high for what appear to be fair diffraction images. The crystallographer is faced with the task of deciding whether the data is in fact faulty, or whether the structural model is inadequate. The web-service checkCIF/PLATON [1] will pinpoint some sources of difficulty. However, there are cases where crystallographic experience is needed. In these cases, graphical displays can be exploited to make use of human pattern recognition abilities. Histograms, scatter-plots and iso-surfaces can draw the eye to trends or outliers which might not be evident in a simple table of values.



The powerful refinement tools available in CRYSTALS [2] are now being supplemented by a wide variety of graphical and statistical diagnostic and validation tools. The summer 2009 release of CRYSTALS will include these tools and examples of their use.

There will be a live demonstration of Crystals at the ECM25 Software Fair.

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Handling of Cell Errors and Their Effect on Derived Parameters. James Haestier[a], N. Dave Brown[a], Mustapha Sadki[a], Amber L. Thompson[a], David J. Watkin[a]. [a]*Chemical Crystallography, Inorganic Chemistry Department, University of Oxford.*
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Area diffractometers output standard uncertainties (s.u.s) on cell parameters that are an order of magnitude smaller than those on atomic coordinates. Herbstein[1], referring to area diffractometers, reported "little (or no) reliance can be placed on currently reported s.u.s from such diffractometers" yet area diffractometers continue to output cell parameter s.u.s with much the same magnitude now as then. The real uncertainties could be up to an order of magnitude greater, but as a consequence of the current output, it is generally felt that their effect on dependent parameters can be neglected. This means that the standard uncertainties of most derived parameters are underestimated. In addition, Kennard and Taylor[2] suggest that the s.u.s on atomic parameters derived by Normal Matrix Least Squares refinement are also underestimated. For calculations of s.u.s of the dihedral angle, Shmueli[3] revealed that the omission of covariances can cause discrepancies as large as 20-25%. Currently there is no cif definition for the variance-covariance matrix (VcV) for the atomic parameters, so programs attempting to compute uncertainties for derived parameters from a published cif have only the isolated parameter s.u.s to work with.

Part of the brief of the Age Concern Software Project is to re-visit common calculations and either reformulate them in a clear and consistent way or, if the published algorithms were incomplete, to put them on a sound mathematical basis. Since rigorous derivation of the standard uncertainties of derived parameters has been consistently neglected, it was felt useful to provide a general analysis that includes all sources of error in the anticipation that some day proper treatment of the data would yield valid VcV matrices.

Our experience from deriving the effect of cell parameter s.u.s on the TLS calculation[4] leads us to believe that if the atomic-parameter VcV in crystal space and the unit cell VcV matrix could be combined to give an augmented parameter VcV matrix in orthogonal space, the computation of valid s.u.s on any derived parameter would be greatly simplified. This manipulation has currently been achieved for all systems except triclinic. The work is on-going.

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Keywords: software; uncertainties; analysis