

FA4-MS12-P06**Improving the S-FFT Algorithm for Positive and Negative Density-function Peaks.**

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Some years ago the direct-methods origin-free modulus sum function (S) [1] was adapted to the processing of intensity data from density functions with positive and negative peaks [2]. That implementation used phase relationships explicitly. Although successfully applied to different situations where the number of reflections was small, its generalization to larger problems required avoiding the time-consuming manipulation of quartet terms. To circumvent this limitation, a modification of the more recent S-FFT algorithm [3] (that maximizes S with only Fourier transforms) was investigated. The resulting S₂-FFT algorithm proved highly effective for crystal structures with positive and negative scatterers in the presence of at least one moderate scatterer in the unit cell [4]. To increase its versatility, the S-FFT algorithm has been adapted now to density functions formed exclusively by positive and negative peaks of similar strength. The results are very promising and some examples describing different situations are analysed in detail.

[1] Rius, J., *Acta Cryst. A*, **1993**, 49, 406. [2] Rius, J.; Miravittles; C.; Allmann, R., *Acta Cryst. A*, **1996**, 52, 634. [3] Rius, J.; Crespi, A.; Torrelles, X., *Acta Cryst. A*, **2007**, 63, 131. [4] Rius, J.; Frontera, C., *Acta Cryst. A*, **2008**, 64, 670.

Keywords: direct methods; phase refinement method; ab-initio structure determination

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New Modelling for Disordered Atoms in Free-form Based Hybrid Refinement and Visual Representation. Mustapha Sadki. *Inorganic Chemistry Department, University of Oxford, OX1 3QR, UK.*

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The number of structures flagged in the CSD as disordered has doubled over the last 25 years, so that the treatment of disorder has become a significant hindrance to fully automatic structure determination.

In a disordered structure, the classical partially occupied anisotropic displacement parameter model is not always appropriate [1]. A new treatment has been devised which integrates a free-form refinement of the disorder together with traditional refinement of other parameters. The method optimises and illustrates the free-form, as well as quantifies and refines related parameters.

We investigate a free form disorder refinement method to optimally combine classic refinement with an iterative process between object and diffraction pattern, overwriting known information in each space.

To optimise the region of interest (ROI) boundaries (in our case, a disordered region), the ROI is modelled after each

iteration as a free form with *deformable models*, where a surface is defined within the density domain, able to move under the influence of internal forces coming from within the model itself and external forces computed from the density values. We use the *gradient vector flow* (GVF) field [2] for the external force, computed as a diffusion of the gradient vectors of a density-value. The GVF was preferred to other forces because of its large capture range and its ability to move a deformable model into boundary concavities.

Given the results obtained from a classic refinement which indicated a potential disorder, the initial free-form configuration is defined within the ROI, in the real space where the optimisation process of a deformable model will operate. Thus the main loop of the refinement process consists of two parts of residuals: one computed as known with all non-disordered sites; the other using the structure factors, obtained by inverse- transform of the optimised ROI. Other parts can be included as usual, such as restraints. The resulting refined parameters and phases produce a new density which when added to the current ROI define a domain where a new ROI optimisation operates. At the final iteration, the refined form represented as *2-simplex meshes* is then visualised, jointly with the refined non-disordered sites, using a volumetric visualisation method, instead of a surfacic one based on iso-surface.

We will discuss how this method leads to improved parameter estimates and a more favourable and comprehensive 3D representation. Notwithstanding, a new specification for a CIF file is required to support the free-form representation.

[1] P. v. d. Sluis and A. L. Spek. BYPASS, **1990**, *Acta Cryst. A* 46, 194-201. [2] C. Xu and J. L. Prince. Snakes, shapes, and gradient vector flow. *IEEE Trans. on Image, Processing*, 7(3):359-369, March **1998**.

Keywords: refinement disorder; computational modelling methods; computational crystallography

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Olex2: Workflow and User Experience. Horst Puschmann^a, Luc Bourhis^a, Richard Gildea^a, Oleg Dolomanov^a. ^a*Department of Chemistry, Durham University, Durham, UK.*

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Olex2 [1] is an analysis and visualisation program for small-molecule crystal structures. It integrates seamlessly with the SHELX [2] package as well as our own cctbx/smtbx-based structure solution and refinement utilities [3,4]. All aspects of the structure determination and publication process are presented in a single, workflow-driven package – from structure solution, refinement and CIF preparation through to the final step of generating publication reports and tables.

While Olex2 combines a wide variety of sophisticated crystallographic tools (space-group determination, symmetry tools, charge-flipping structure solution, smtbx-based refinement routines, tools to deal with disorder efficiently, void calculation, electron density viewer, polyhedral volume calculation and many more), this presentation will focus on workflow aspect of this software:

It is not about what the program *does* but about what *results* are expected.

Olex2 enables any scientist with an interest in single-crystal diffraction to perform all aspects of structure solution, refinement and publication tasks correctly and, in routine cases, without the need to refer to a trained crystallographer. This is achieved through a strong emphasis on workflow, combined with clear and indicative visual feedback of what is happening in any given task, on the way towards a publishable small-molecule structure. This makes Olex2 a very useful aid in teaching of small-molecule crystallography.

Olex2 is open source software and is available free of charge to academic users from <http://www.olex2.org>

[1] O. Dolomanov, L. Bourhis, R.J. Gildea, J.A.K. Howard and H. Puschmann *J. Appl. Cryst.*, **2009**, 42, 339-341. [2] G.M. Sheldrick, **2008**, *Acta Cryst.* A64, 112-122. [3] L.J. Bourhis, O.V. Dolomanov, R.J. Gildea, J.A.K. Howard and H. Puschmann, **2009**. *In preparation*. [4] L.J. Bourhis, Grosse-Kunstleve, R. W. & Adams, P. D., **2007**. *IUCr Commission on Crystallogr: Computing Newsletter*, No. 8, pp. 74-80. <http://www.iucr.org/resources/commissions/crystallographic-computing/newsletters/8>

Keywords: software for crystallography; small molecule crystallography; computer-aided crystallographic teaching

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The Ambiguous Solutions from Different Automatic Structure Solution Programs. F. Betül Kaynak^a, Lars Eriksson^b. ^a*Hacettepe University, Department of Physics Engineering, 06800, Ankara, Turkey.* ^b*Department of Structural Chemistry, Arrhenius Laboratory, Stockholm University, 106 91, Stockholm, Sweden.*

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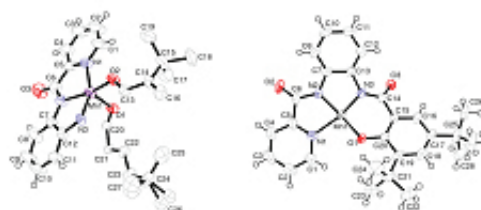
The manganese (III) complex with a diamide ligand has been synthesized by Lien-Hoa Tran. This complex was found to catalyze both epoxidation of (Z)- and (E)-stilbene with high conversion and the oxidation of benzyl alcohol to benzaldehyde. In this study we present the two different results of the different automatic structure solution programs.

First the structure was solved by direct methods using the SHELXS-97 [1] program. But the structure predicted from chemical and spectral analysis was not confirmed by X-ray analysis of the single crystal. Even though it had a satisfactory R(F) value of 0.0588 ($wR(F) = 0.1153$) and the minimum and maximum values for the residual density were -0.340 and $0.460 \text{ e}\text{\AA}^{-3}$ respectively (Figure 1a).

Then SIR92 [2] was used in order to find the correct structure. The structure supported by chemical and spectral analysis was found (Figure 1b). At the final convergence limit the reliability indices are as follows: $R(F) = 0.0425$, $wR(F) = 0.0772$ and $g.o.f. = 0.839$. And the minimum and maximum values for the residual charge density were -0.222 and $0.227 \text{ e}\text{\AA}^{-3}$ respectively.

In this study we see that the structure validation values such as R(F), $wR(F)$, ρ_{\max} , ρ_{\min} , $g.o.f.$, etc. are not satisfactory in

order to define the correct crystal structure. It is interesting that both structures are “well behaved” but one of them is better.



(a)

(b)

Figure 1. (a) Structure found by SHELXS-97 and (b) SIR92

[1] Sheldrick, G. M., *Acta Cryst.*, **1997**, A64, 112-122. [2] Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M., *J. Appl. Cryst.*, **1994**, 27, 435.

Keywords: structure validation; SHELX; SIR