s1.m3.p2 **New structures solved using ab initio direct methods.** Gábor Bunkóczi, Judit É. Debreczeni and George M. Sheldrick, *Lehrstuhl für Strukturchemie, Georg-August Universität, Tammannstr. 4, 37077 Göttingen, Germany. E-mail: gsheldr@shelx.uni-ac.gwdg.de*

Keywords: Direct methods; Dual-space recycling; SHELX

The structure determination of biological peptides like antibiotics and microproteins is complicated by unique factors. There are only a few crystallisation screens available that are directed to this substance class, and the asymmetric unit is usually comparable to that in average protein structures, which constitutes a significant barrier when trying to overcome the phase problem. In addition, refinement of these structures is complicated by unusual residues and requires high-resolution data.

Three structures belonging to this class have recently been determined using *ab initio* direct methods. Viscotoxin B2 is a thionin with 46 amino acid residues, but with two independent molecules that correspond to about 800 atoms. Asymmetric unit of the antibiotic feglymycin was thought to consist of about 500 independent atoms with oxygen as the heaviest element present. The amphomycin-analogue tsushimycin is an 11-residue Ca^{2+}-binding cyclopeptide with a long fatty acid sidechain, and contains about 1300 atoms in 12 independent molecules.

In all cases, data were available to atomic resolution, but resolution limit varied from higher than 1.0 Å to barely atomic with 3σ intensity between 1.1-1.2 Å. SHELXD [1] was employed by first locating heavier elements (viscotoxin: 6 S, tsushimycin: 24 Ca^{2+}) and then expanding the structure with peaklist optimisation. For feglymycin, where there is no element heavier than oxygen, a portion of the structure was searched that was thought to be large enough to give an indication for success. Solution was achieved in all cases with varying success rates, which were considerably dependent on the number of heavier atoms and was the highest for tsushimycin and the lowest for the equal-atom structure feglymycin. In the refinement, feglymycin proved to be merohedrally twinned, and taking the twin operation into account, the size of the asymmetric unit corresponds to more than 1000 atoms with that feglymycin is by far the largest equal-atom structure solved so far.

[1] Sheldrick, G. M., Hauptman, H. A., Weeks, C. M., Miller, M. & Usón, I. (2001). *International Tables for Crystallography*, Vol. F, edited by E. Arnold & M. G. Rossmann, pp. 333-351. Kluwer Academic Publishers, Dordrecht.

s1.m3.p3 **A Direct Approach towards High Resolution Structure Determination without Fourier Inversion.** Karl. F. Fischer[a], Armin Kirfel[b] and Helmuth W. Zimmermann[c], [a]*Technische Physik, D-66041 Saarbrücken, Germany,* [b]*Mineralogisch-Petrologisches Institut, Poppelsdorfer Schloss, D-53115 Bonn, Germany, and* [c]*Institut für Angewandte Physik, Staudtstr. 3 D-91058, Germany. E-mail: karl.fischer@mx.uni-saarland.de*

Keywords: Structure determination; High resolution; Quasi-homometry

For determining one-dimensional centrosymmetric structures or inspecting structural details in projections of known structures we have developed an approach which is based on a point scatterer model and its "geometrical structure amplitudes". [1] The method provides higher resolution than a Fourier sum, needs less observations and detects either a unique unambiguous solution or all possible scatterer arrangements that are compatible within the experimental uncertainty.

The significant gain in direct space resolution was successfully tested in a feasibility study concerning a possible pseudosymmetric split (< 0.1 Å) of the (La, Sr) position in $(La_{0.5}Sr_{1.5})MnO_4$ [2].

Theoretical progress has been made towards relieving some of the initial restrictions imposed on the structure model, i.e. we can show that:

– special positions can be included,
– point scatterers with unequal scattering powers both positive and negative can be handled,
– two-dimensional point structures can be solved, as proven by test calculations.

Additional details of the method as well as test examples, including a case of quasi-homometry, will be presented.

[1] Fischer, K.F., Kirfel, A., Zimmermann, H. (2004). *Annual DGK-meeting*, Jena (2004).
[2] Kirfel, A., Fischer, K.F. (2004). *Annual DGK-meeting,* Jena (2004)..