PS02.03.15 NEW DEVELOPMENTS OF THE TWIN VARIABLES APPROACH TO THE CRYSTAL STRUCTURE DETERMINATION. G. Tsoucaris, A. Mishnev*, A. Hountas#, Universite de Paris-Sud, Centre Pharmaceutique, Chatenay-Malabry, France, *Latvian Institute of Organic Synthesis, Riga, Latvia, #Agricultural University of Athens, Athens, Greece

In the development of a new approach to the crystal structure solution based on the concept of "twin" variables (Hountas & Tsoucaris (1995), Acta Cryst. A51, 754-763) we investigate: 1) dependence of the procedure on the parameters involved, 2) the role of random starts for Ψ-sets, 3) an attempt for transferring of phase information from low (LR) to high (HR) resolution. The basic procedure implies gradient search of the minimum of the global minimization function in the Ψ-variables space. Relation between E's and Ψ's is expressed by convolution eq. $E(H) = \Sigma\Psi(K)\,\Psi^*(K-H)$ (1) and regression eq. $\Psi(H) = \Sigma\, E(K)\,\Psi(H-K)$ (2).

1. Parameter optimization. To control the number of accepted E's in the extension process we calculate the acceptance criterion AMIN for the value of _E_ by empirical formula AMIN = 0.5 + N/1000, where N is the current number of E's. We also introduce a) Sim-type weights in eq.(2) and gradient expression for M_{mod}, b) recombination formula in the form $\Psi_{new} = (\Psi_{old} + \Psi_{new})/2$.

2. Multisolution algorithm. 100-150 random starts for Ψ-sets produced several good solutions with the mean phase errors (MPE) in the range of 25-35° for 150-200 E's. The best set contained 210 phases with MPE of 27° (test structure with 41 nonhydrogen atoms in P1).

3. From LR to HR. An important characteristic of the method is the decoupling of E-set, containing the moduli information, and primitive Ψ-variables. Thus part of the Ψ-set can be located outside the observed sphere. This amounts to modeling LR E-data with HR Ψ-set, and thus introducing a bias towards atomicity. After transferring of information from the LR E's to the Ψ-set containing HR components, one extends the phase and modulus information to the HR E's in the next step by means of eq. (1).

PS02.03.16 DEVELOPMENT OF *SnB* VERSION 2. Charles M. Weeks, Russ Miller, Hauptman-Woodward MRI, 73 High Street, Buffalo, New York 14203-1196, USA

The ***Shake-and-Bake*** algorithm is a powerful formulation of direct methods which alternates reciprocal-space phase refinement with filtering in real space to impose constraints. As implemented in the distributed version (1.1) of the ***SnB*** computer program, ***Shake-and-Bake*** combines peak picking in real space with optimization *via* either parameter-shift reduction of the minimal function value or tangent-formula refinement [Miller *et al.* (1994), *J. Appl. Cryst.*, **27**, 613-621]. It employs a multisolution approach in which the starting set of trial structures consist of randomly positioned atoms. The ***SnB*** program has provided *ab initio* solutions for structures containing as many as 600 independent nonH atoms, provided that diffraction data are available to 1.2Å.

An upgraded version of ***SnB***, which permits density modification followed by inverse Fourier transformation to be used in place of peak picking and subsequent structure-factor calculation, is under development. Using a simple modification scheme such that $\rho_{mod}=\rho$ if $\rho>C^*\sigma_\rho$ and $\rho_{mod} = 0$ if $\rho<C^*\sigma_\rho$, where C is a constant in the range 2.0-3.0, a 6-8 fold reduction in overall CPU time has been achieved for crambin, a 400-atom test structure. When SAS tangent-formula refinement was incorporated into this version of ***SnB*** and applied to invariant values estimated for 2Å error-free SAS data [Hauptman (1982). *Acta Cryst.* **A38**, 632-641] for cucumber basic protein, the success rate (*i.e.*, percentage of trials going to solution) was improved over that obtained for SAS tangent-formula refinement alone.

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PS02.03.17 DIRECT DETERMINATION OF PHASE FROM THREE BEAM CONVERGENT BEAM DIFFRACTION PATTERNS OF CENTROSYMMETRIC CRYSTALS. J. Etheridge, A. F. Moodie and C. J. Humphreys, Dept of Materials Science and Metallurgy, University of Cambridge, Pembroke Street, Cambridge, CB2 3QZ, U.K

The symmetries of three beam diffraction from a centrosymmetric crystal are such that the distribution of scattered intensity uniquely determines both the phase and the magnitude of the three structure amplitudes. In particular, the phase can be read directly from convergent beam diffraction patterns where the three beam approximation retains some validity, whilst the magnitudes can be determined from three distances measured on these patterns. Technically, this result derives from the fact that orientations can be found along which the general symmetry of the special unitary group, SU(3), factors into three subgroups in SU(2). This result is independent of thickness. The experimental conditions under which these results can be exploited are discussed quantitatively. Experimental convergent beam electron diffraction patterns are used to illustrate the method.

PS02.03.18 PHASE DETERMINATION BY DUAL SPACE METHODS OF ELECTRON CRYSTALLOGRAPHY: A COMPARISON OF I23 & $I2_13$ STRUCTURAL PHASES A. Schwartzman, P. Goodman,School of Physics, University of Melbourne, Parkville, Australia 3052, A.W.S.Johnson, E.M.Centre, University of Western Australia, Nedlands, W.A., Australia 6009.

No discussion on direct methods of structural phase determination can be considered complete without reference to the dual space methods of electron crystallography, whereby Fourier space and direct space data are collected by means of CBED and HREM respectively on the same sample. The method is based on the well-known relationship that phases in one space amount to displacements in the other. In this paper we illustrate this method by showing how the look-alike space group pairs $I23/I2_13$ and $I222/I2_12_12_1$, commonly held to be indistinguishable since the space-group-forbidden reflections characterising the 2-fold screw axis are already extinguished by lattice centering, may be resolved in this way.

We present data on the crystal systems $Bi_{12}GeO_{24}$ (I23) and $Ba_3Fe_2O_5Cl_2$ ($I2_13$). The former compound shows accurate 2-fold symmetry in the [001] CBED pattern, while the $I2_13$ compound shows a weak departure from symmetry in the FOLZ reflections, due to 3-dimensional dynamic interaction. However distinction is much more unequivocal using dual space methods. High-resolution [001]-projection micrographs for $Ba_3Fe_2O_5Cl_2$ show a molecular structure as required by the projected horizontal 2_1 operators. Direct observation of this operation is possible in real space since this results in a unit-cell displacement, spatially distinct from the centering displacement caused by body centering.