Poster Presentations

[MS10-P21] Monomer Restraint Library for Supramolecular Crystallography Julian J. Holstein[a,b], Colm Browne[b], Oliver S. Smart[a], Gérard Bricogne[a]

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Supramolecular crystallography lies in between the typical domains of small- and macromolecular crystallography. Molecular weights and unit cells of supramolecular structures can be large. Low crystal stability can be caused by a large percentage (15%+) of disordered solvent regions in the asymmetric unit. In these cases diffraction up to atomic resolution cannot always be reached, even when 3rd generation synchrotron sources are used. These are typical characteristics of macromolecular structures. As supramolecular assemblies prefer to crystallise in centrosymmetric space groups (90%+) and do not contain biological building blocks (Proteins, DNA), they also share typical characteristics of small-molecule structures. Their structure refinement stability heavily relies on the introduction of additional conditions namely restraints or constraints that frame the structural model. Especially since 1991 [1] the extensive use of geometrical restraints became a standard technique in macromolecular structure refinement. In March 2012 the automatic restraint generator GRADE has been made publicly available as a web server, which is free to use. [2] GRADE queries the Cambridge Structural Database CSD using MOGUL. If experimental small-molecule information is lacking, GRADE performs semi empirical quantum mechanics (QM) calculations. Restraint dictionaries produced by GRADE can be directly used with BUSTER, COOT or REFMAC. A routine to translate GRADE restraint dictionaries into SHELX syntax (DFIX, DANG and FLAT commands) has been implemented recently and will be made available with the next update of the GRADE WEB SERVER.[2] SHELX fully supports the macromolecular residue construct, which is required to use GRADE restraints in structure refinements.
We have compiled a monomer restraint library for supramolecular ligand systems, which covers typical solvents and counter ions as well. These restraints have already proved helpful for the refinement of large and challenging supramolecular structures [3] and we will present the case of a supramolecular Co tetrahedron cage structure.

[1] R. A. Engh, R. Huber, *Acta Cryst.*, 1991, **A47**, 392 – 400
[2] GRADE WEB SERVER http://grade.globalphasing.org
[3] T. K. Ronson, C. Giri, N. K. Beyeh, A. Minkkinen, F. Topić, J. J. Holstein, K. Rissanen, J. R. Nitschke, *Chem. Eur. J.* 2013, **19**, 3374 – 3382

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