Semi-automated Rietveld Refinement of Molecular Crystal Structures with *DASH* & *TOPAS*.

Jacco van de Streek, Institute for Inorganic and Analytical Chemistry, Frankfurt University, Frankfurt am Main, Germany. jacco@chemie.uni-frankfurt.de.

With the improvement of powder diffractometers—both at synchrotron facilities and in the laboratory—and increases in computer power, the number of molecular crystal structures that can be routinely determined from powder diffraction data, as well as their complexity, is on the increase (see *e.g*. (Fernandes *et al.*, 2007) and references therein).

As a result of this trend, the number and complexity of Rietveld refinements is increasing accordingly. Due to the presence of severe peak overlap in the powder patterns of molecular crystal structures, Rietveld refinements of such patterns need to be able to cope with parameters that are correlated or ill-determined; improved computer power does not change this. The two strategies that are usually adopted to keep Rietveld refinements stable are well known:

1. Rather than refining all parameters at once, subsets of parameters are refined in a well-controlled order.
2. Chemical restraints or constraints are introduced, *e.g*. by restraining all bond lengths to chemically sensible values.

Although several good programs for Rietveld refinement are available, the implementation of these two strategies is at the moment still up to the user. This leads to tedious, manual setting up of restraints and constraints and an equally tedious, manual switching on and off of refinable parameters.

These two strategies, which require an intimate knowledge of chemistry, crystallography and powder diffraction, have now been implemented in the program *DASH* (David *et al.*, 2006), in such a way that *DASH* can be used to direct a Rietveld refinement in *TOPAS* (Coelho, 2006) by generating input files and modifying the resulting output files so that they can be used as new input files again. *DASH* automatically perceives bonds and bond types, aromatic rings (for planarity restraints) and atoms on special positions, and *DASH* automatically switches refinable parameters on and off. Even for molecular compounds with many restraints and constraints, high-quality Rietveld refinements can now be carried out routinely in a matter of minutes (Figure 1).

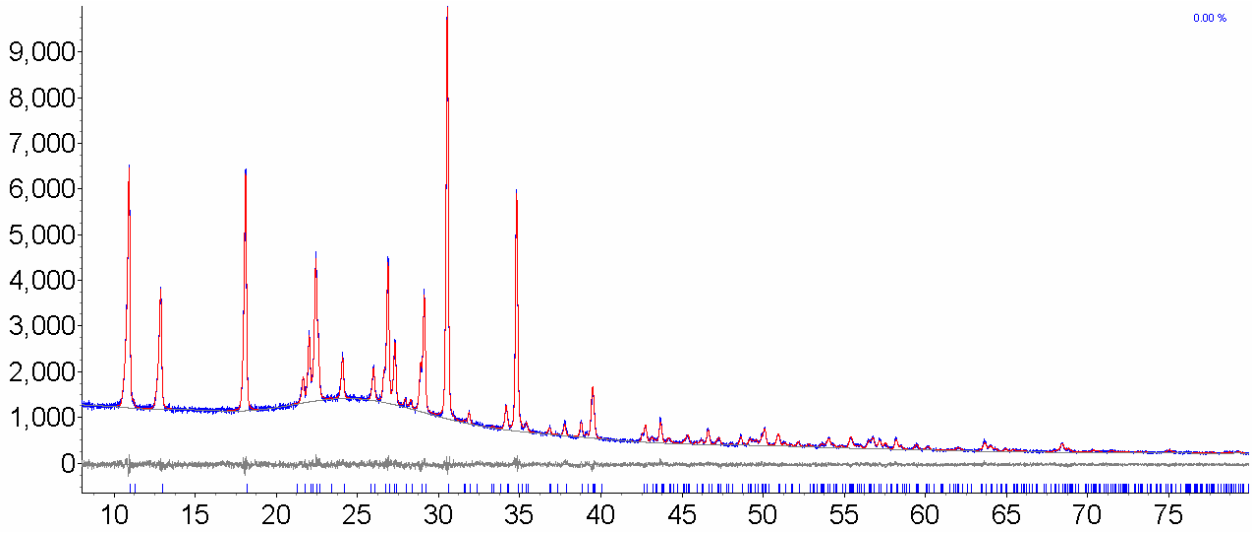

Figure 1. Result of the Rietveld refinement of pamoic acid with *DASH* and *TOPAS*, including anisotropic peak broadening, peak asymmetry, bond-length restraints, valence-angle restraints and planarity restraints for aromatic ring systems. The observed (blue), calculated (red) and difference patterns (black) are shown. R_{wp} = 3.65%, R_p = 2.75%, χ^2 = 1.05.

References

Coelho, A. A. (2006) TOPAS-Academic Version 4.0

David, W. I. F., Shankland, K., van de Streek, J., Pidcock, E., Motherwell, S. & Cole, J. C. (2006). *J. Appl. Cryst.* **39**, 910-915.

Fernandes, P., Shankland, K., Florence, A. J., Shankland, N. & Johnston, A. (2007). *J. Pharm. Sci.* **96**, 1192-1202.