

## STRUCTURE DETERMINATION OF CAFFEINE ANHYDROUS $C_8H_{10}N_4O_2$ FROM POWDER DIFFRACTION DATA WITH TOPAS

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TOPAS is a program package for profile and structure analysis, which has been developed with particular emphasis placed on improving stability and convergence. It is built around a general non-linear least squares system written specifically to integrate various types of powder diffraction and single crystal analyses. This includes the ability to refine on laboratory/synchrotron/CW and TOF neutron powder diffraction as well as single crystal data simultaneously. All currently employed profile fitting techniques such as single line fitting, whole power pattern fitting, whole powder pattern decomposition (according to Pawley and Le Bail) as well as Rietveld refinement are supported and can be combined for analysis of multi phase patterns.

A remarkable feature of TOPAS is the ability to determine structures by simulated annealing together with user defineable penalty functions, rigid bodies and lattice energy minimization techniques including user defineable force fields [1]. Structure solution can be performed using step intensity data as well as integrated intensities obtained from powder and single crystal data.

The ability to solve structures from step intensity data, i.e. without the need to extract integrated intensities, allows successful structure determination of large structures from poor quality powder data.

Recently the structure of the anhydrous compound of Caffeine monohydrate  $C_8H_{10}N_4O_2$  \*  $H_2O$  has been proposed. The powder pattern could be indexed monoclinic by using ITO. Several cells and space groups were found but finally two cells and space groups were found to be useful for further work. The cell volume of  $4450 \text{ \AA}^3$  is approx. 4.5 times larger than the cell of the monohydrate compound with one very long axis. The final result consists of 5 individual molecules in the asymmetric cell resulting giving a total amount of 20 molecules in the unit cell. The cell content has been approved by additional pycnometric density measurements. This structure determination represents one of the biggest ever solved structures using powder diffraction data and documents the effectiveness of real space structure determination methods.

### References:

- [1] Coelho, A.A. (2000): Whole-profile structure solution from powder diffraction data using simulated annealing. - J. Appl. Cryst., 33, 899 - 908.