ESRF	**Experiment title:** Structure determination of $C_{18}H_{10}N_2O_3$ by energy minimization and high resolution powder diffraction	**Experiment number:** CH-189
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Report:

The crystal structure of 2,5-Dihydroxy-benzo[de]benzo[4,5]-imidazo[2,l-a]isochinolin-7-one, $C_{18}H_{10}N_2O_3$ has been solved by a combination of energy minimization calculations [l] and Rigid Body Rietveld Refinement. Single crystals could not be grown and therefore we used Synchrotron powder data to confirm the anticipated structure and to allow for the refinement of structural details.

Data collection was carried out at room temperature using 0.84979 Å from double monochromator and 9 parallel Ge (111) analyzers. The compound crystallizes in $Pna2_1$ with Z=4 and lattice parameters of: a= 13.2748(3) Å, *b= 20.9543(6)* Å, *c= 4.7796(* 1) Å.

The arrangement of the molecules inside the unit cell was calculated by minimizing the intermolecular energy using self-developed methods [2]. The obtained packing showed a calculated powder diagram very similar to the experimental one.

This crystal structure was subsequently refined by Rigid-Body Rietveld methods (R-p = 4,3 %, R-wp= 5,9 %, R-F = 6,7 %, R-F^2 = 10,7 %) [Fig. 1]. The following parameters have been refined: profile (3 parameters), zero shift, lattice parameters, scale factor, overall temperature factor, background (4 parameters), position and orientation of the molecules, 4 intramolecular parameters (length, width, planarity, 1 angle) [Fig. 2]. This is one of the largest molecules, whose crystal structure was determined from powder data.

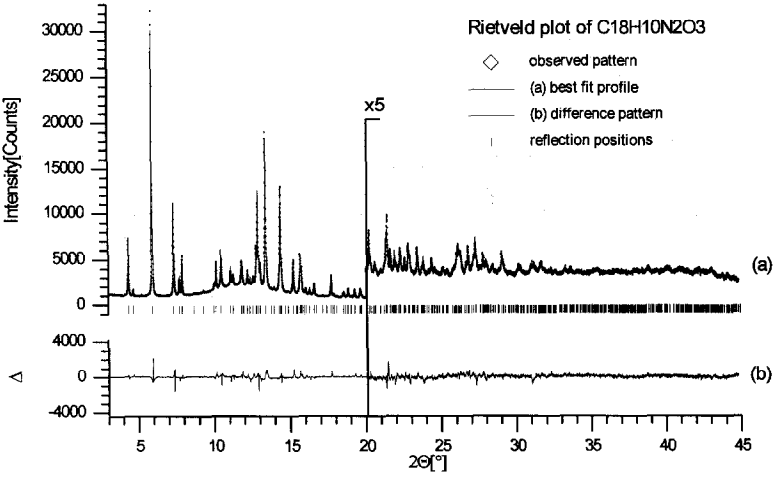

Fig 1:
Rietveld plot of $C_{18}H_{10}N_2O_3$ using *Pna2₁* symmetry. The pattern at higher angle (>20° 2Θ) is enlarged by a factor of 5.

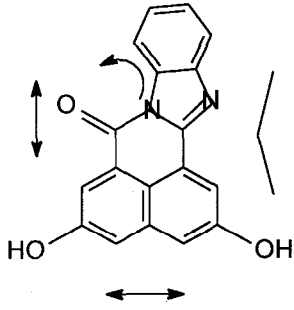

Fig. 2:
Model of the molecule and some intramolecular degrees of freedom: length, width, planarity and rotation angle around N (left).

References:

[l] M. U. Schmidt (1997), DGK meeting 1997 in Hamburg, collected abstracts, 179.
[2] M. U. Schmidt & U. Englert (1996), J. Chem. Soc. Dalton Trans., 2077-2082.