Crystal Structure of a New Polymorph of Sulfabenzamide

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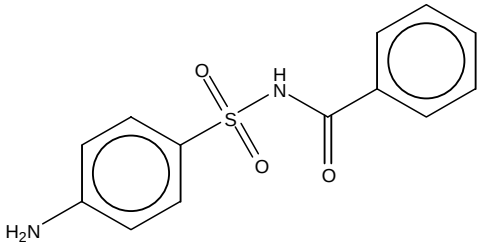
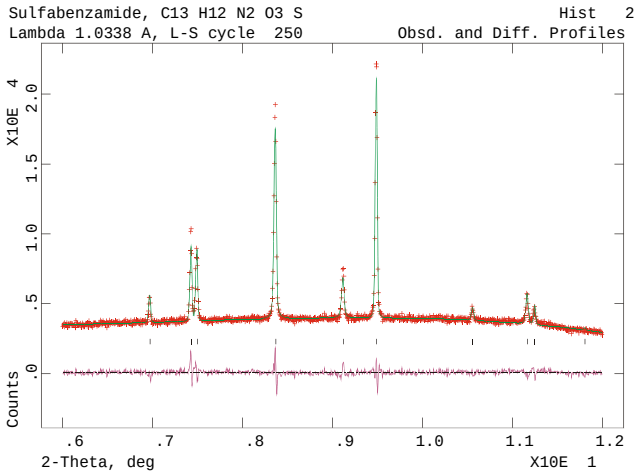
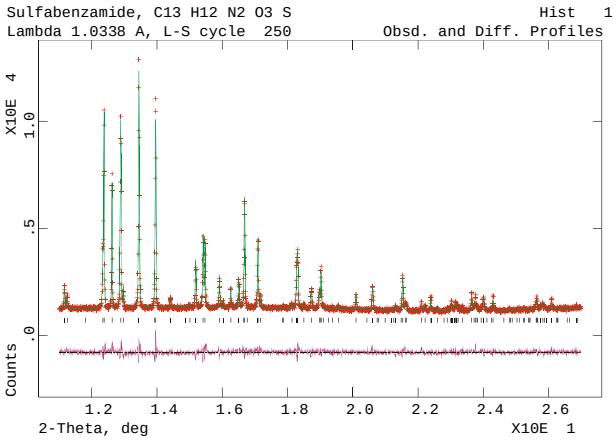
Introduction

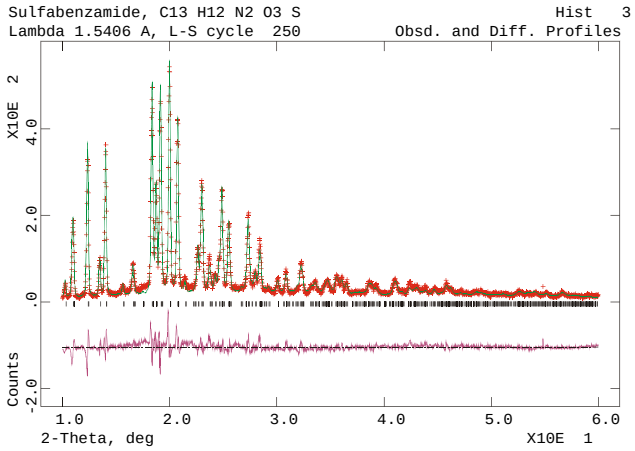
Although the crystal structure of the topical antibacterial sulfabenzamide, $C_{13}H_{12}N_2O_3S$ has been reported [1],



most commercial samples contain another polymorph as the major component. Knowledge of the structure of this "new" form would permit quantitative phase analysis of such mixtures using the Rietveld technique.





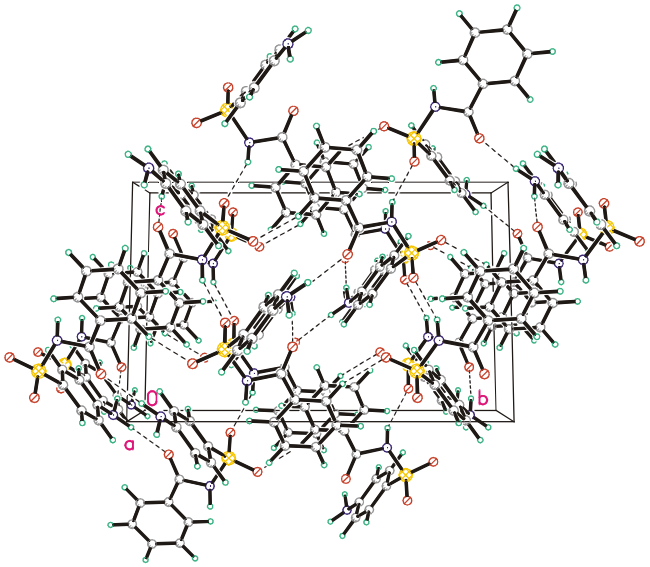


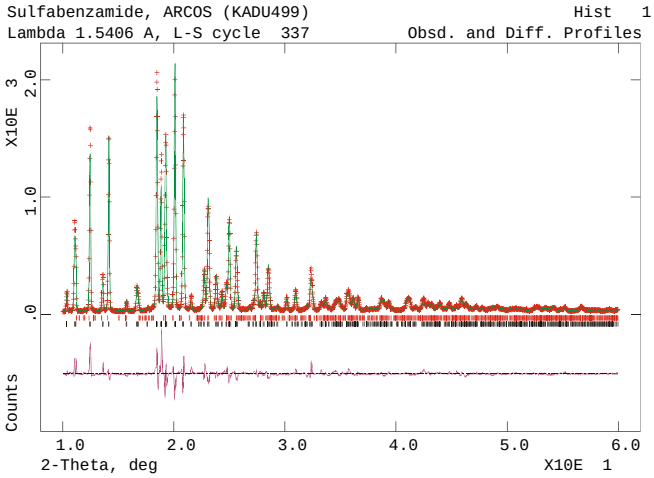
Methods and Materials

Both synchrotron (12.7 keV, λ = 1.03377 Å) and laboratory powder patterns could be indexed on a primitive monoclinic cell having a = 8.17063(9), b = 15.98003(18), c = 10.38238(10) Å, β = 104.1811(9)°, and V = 1314.29(3) Å^3. The space group from the laboratory pattern was ambiguous, but the higher resolution of the synchrotron pattern permitted unambiguous determination of the space group as $P2_1/c$. A trial structure was derived using the simulated annealing program DASH, and refined using GSAS. The final refinement of 69 variables using 6920 observations yielded the combined residuals wRp = 0.0533, Rp = 0.0341, and χ^2 = 4.173. Extensive use was made of rigid bodies and soft constraints.

Discussion

The single molecule in the asymmetric unit is linked to its neighbors by a network of hydrogen bonds. Each amino group bridges two carbonyl groups, and the amido nitrogen donates a proton to one of the sulfone oxygens. These N-H···O=S linkages result in chains parallel to the c-axis. Some of the torsion angles differ significantly from those in the two independent molecules in the DAWRAU structure. A search of the Cambridge Structural Database indicate that phenyl-sulfone torsion angles cluster strongly around ±90°, as is observed here; the phenyl-sulfone linkage appears relatively rigid. Likewise, the S-N-C=O torsion angles in the CSD cluster tightly around 0°; the values observed here and in DAWRAU are typical. Although four torsion angles were allowed to vary in the simulated annealing process, the





number could have been reduced to two to make the problem smaller. The sulfonamide and benzamide torsion angles exhibit greater variability in the CSD, and the largest conformational differences among the sulfabenzamide structures lie in these angles. Quantum chemical calculations on the crystal structure are in progress.

Trosion	This Work	DAWRAU Mol. 1	DAWRAU Mol. 2
C5-C4-S14-N17	-84.8	-83.3	-99.6
C3-C4-S14-N17	95.9	101.1	83.1
C4-S14-N17-C19	-57.3	-65.5	67.6
S14-N17-C19-O20	-5.7	13.5	-7.4
N17-C19-C21-C26	9.0	-17.6	-31.2
N17-C19-C21-C22	-169.4	161.6	149.4

The structure of this "new" polymorph demonstrates its utility by permitting us to conclude that commercial reagent sulfabenzamide, purchased from Fisher ACROS, consists of 97% of this new polymorph and 3% of the DAWRAU polymorph.

Acknowledgments

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References

[1]. J. Rambaud, R. Roques, S. Alberola, and F. Sabon, *Bull. Soc. Chim. Fr.*, 51 (1980); CSD Refcode DAWRAU.