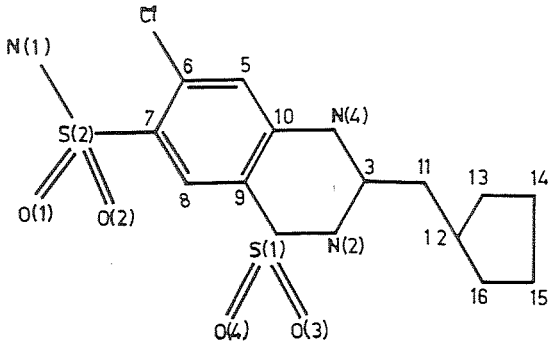
03.3–10 CRYSTAL AND MOLECULAR STRUCTURE OF 6-CHLORO-3-CYCLOPENTYLMETHYL-3,4-DIHYDRO-7-SULFAMOYL-2H-1,2,4-BENZOTHIADIAZINE-1,1-DIOXIDE. By A. K. Basak, S. Chaudhuri and S. K. Mazumdar, Crystallography and Molecular Biology Division, Saha Institute of Nuclear Physics, Sector I, Block 'AF', Bidhannagar, Calcutta-700 064, India.

2H-1,2,4-Benzothiadiazine-1,1-dioxides and its derivatives constitute a group of drugs used as diuretic and antihypertensive agents. The present crystallographic investigation of the title compound, bearing the commercial name NAVIDREX, was undertaken in order to study the geometry and conformation of the molecule vis-à-vis those of other molecules of this group.



Crystal data :
crystals from acetone, molecular formula $C_{13}H_{18}N_3O_4S_2Cl$, M_r = 379.89, space group=Pbca, with a=9.441(4), b=29.633(11), c=12.167(8) Å, V=3404(5)Å^3, Z=8, D_m=1.485 Mgm^{-3}, D_x=1.482 Mgm^{-3} μ=4.85 cm^{-1}, F(000)=1584.

The structure was determined by Patterson and heavy-atom methods and refined by full-matrix least-squares to a final R=0.056 for 2075 'observed' [$I \geq 3\sigma(I)$] reflections. The endocyclic torsion angles in the thiadiazine ring indicate that it has a half-chair conformation. The phenyl ring shows a slight puckering with the atoms, C(9) and C(10), at its junction with the thiadiazine ring having maximum deviations from planarity. The cyclopentane ring has also a half-chair conformation. The unusually large thermal parameters observed for the atoms in this ring may be attributed to pseudorotation. Of the four hydrogen atoms available for hydrogen bonding it has been possible to locate two, both being attached to the thiadiazine ring nitrogens, N(2) and N(4). These hydrogens form intermolecular N-H...O hydrogen bonds to stabilize the structure.

03.3–11 THE CRYSTAL AND MOLECULAR STRUCTURE OF SODIUM CEFTIZOXIME. By A. Miyamae, S. Koda and Y. Morimoto, Fujisawa Pharmaceutical Co., Ltd., Analytical Research Laboratories, 2-1-6, Kashima, Yodogawa-ku, Osaka 532, Japan.

Sodium ceftizoxime is one of the new cephalosporine antibiotics which is characterized by an aminothiazolyl-methoxyimino group in the C_7-side chain and no substituent at the C_3-position.
Crystals are colourless sticks and belong to the orthorombic system, space group $P2_12_12_1$, with a=7.84, b=23.19, c=51.29Å and Z=20 (five independent molecules per asymmetric unit). The intensities of 8357 independent reflections were measured using a Rigaku automatic four-circle diffractometer with graphite monochromated CuKα radiation.
The structure has been solved by a combination of the direct method using MULTAN and Fourier technique. The refinement of positional and isotropic temperature parameters has been carried out by a block-matrix least-squares method using 5760 reflections with $F \geq 3\sigma(F)$. R-value is now 0.30, and further refinement is in progress. Five independent molecules take similar stereochemistry in the crystal and packed along the b axis.
As to the exocyclic amido group at the C_7-position, it appears that intermolecular hydrogen bonds between N(13) and O(15) atoms with distances of 2.6 - 2.8A link the molecules along the b axis and this type of hydrogen bonds is just like the β-sheet structure found in protein.
In the C_7-side chain, the methoxymino group adopts a syn configulation and the amino-thiazole ring is mainly amino-type structure. Furthermore these two groups are quasi coplanar.