CRYSTALLOGRAPHY IN BIOCHEMISTRY AND PHARMACOLOGY

03.3.10 CRYSTAL AND MOLECULAR STRUCTURE OF 6-CHLORO-3-CYCLOPENTYL-3,4-DIHYDRO-7-
SUBFAMOTHIAZOLE-2(1,2,4)-BENZOTHIAZOLIDINE-1,1-DIOXIDE. By S. K. Banak, S. Chaudhuri
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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-a-vis those of other molecules
of this group.

Crystal data:
Crystals from acetone, molecular formula
C\textsubscript{13}H\textsubscript{18}N\textsubscript{3}O\textsubscript{4}S\textsubscript{2}Cl, \textit{I} \textit{m}=379.89, space group \textit{Pbca},
with \textit{a}=9.441(4), \textit{b}=29.633(11), \textit{c}=12.167(8) \AA, \textit{V}=3404(5) \AA\textsuperscript{3}, \textit{Z}=8,
\textit{D}_m=1.485 g cm\textsuperscript{-3}, \textit{D}_x=1.482 g cm\textsuperscript{-3}, \textit{\mu}=4.85 cm\textsuperscript{-1}, \textit{F}(000)=1584.

The structure was determined by Patterson and heavy-atom methods and refined by full-matrix
least-squares to a final \textit{R}=0.056 for 2075 'observed' [I \textgreater; 3\textit{\sigma}(I)] reflections. The endo-
cyclic 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 hydrogen
groups form intramolecular N-H \cdots O hydrogen bonds to stabilize the structure.

03.3.11 THE CRYSTAL AND MOLECULAR STRUCTURE OF SODIUM CEFTIZOXIME. By A. Miyamae, S. Koda
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Sodium ceftizoxime is one of the new cephalosporine antibiotics which is characterized by
an aminothiazolyl-methoxyimino group in the C\textsubscript{7}-side chain and no substituent at the
C\textsubscript{3}-position.

Crystals are colourless sticks and belong to the orthorhombic system, space group \textit{P}\textsubscript{2}1\textsubscript{2}1\textsubscript{2}1,
with \textit{a}=7.84, \textit{b}=23.19, \textit{c}=51.29 \AA and \textit{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 CuKa 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 \textit{F} > \textit{\sigma}(\textit{F}).
\textit{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 \textit{b} axis.

As to the exocyclic amido group at the C\textsubscript{7}-position, it appears that intermolecular
hydrogen bonds between N(13) and O(15) atoms with distances of 2.6 - 2.8 \AA link the molecules
along the \textit{b} axis and this type of hydrogen bonds is just like the \beta-sheet structure found
in protein.

In the C\textsubscript{7}-side chain, the methoxyimino group adopts a syn configuration and the amino-
thesiazole ring is mainly amino-type structure.

Furthermore these two groups are quasi coplanar.