

03.5-19 CRYSTAL AND MOLECULAR STRUCTURE OF 2-IMINO-1,3-DIHYDRO-4,6-DIMETHYL PYRIMIDINIUM CHLORIDE By Amit Halder, A.K. Basak, S.K. Mazumdar of Crystallography Division, Saha Institute of Nuclear Physics. and S. Chaudhuri, Bose Institute, Calcutta, India.

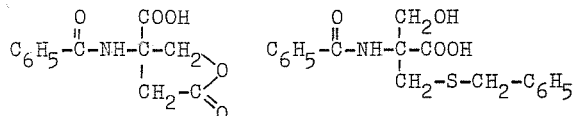
The title compound crystallizes in the orthorhombic space group, $Pna2_1$ with $a=14.269(3)$, $b=8.022(2)$, $c=6.675(2)$ Å. Solved by heavy atom method and refined to a final R of 0.048. The cation exist as imino tautomer. The exocyclic C-N bond length clearly establishes its double bond character and endocyclic bond angles at N(1) and N(3) increases because of protonation. There is an indication of strong conjugation between the imino nitrogen and the pyrimidine ring. The molecules form chloride bridged hydrogen bonded chains extended infinitely along the glide direction. The chloride ions are stacked between the pyrimidine bases.

Mol. Formula : $C_6H_{10}N_3Cl$.

This work forms a part of the doctoral dissertation of Amit Halder.

03.5-21 ABSOLUTE CONFIGURATION AND CRYSTAL STRUCTURE OF THE NEW α -SUBSTITUTED AMINOACID DERIVATIVES. By M. Bukowska-Strzyżewska and W. Wieczorek, Institute of General Chemistry, Technical University, 36 Żwirki, 90-924 Łódź, Poland.

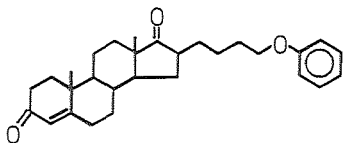
Dextro-rotatory α -hydroxymethyl-N-benzoil-aspartic acid lactone (I) crystallizes in the orthorhombic system: $P2_12_12_1$, $a=10.337(1)$, $b=18.115(1)$, $c=6.063(1)$, $Z=4$. Laevorotatory S-benzyl- α -hydroxymethyl-N-benzoilocysteine (II) crystallizes in the monoclinic system: $P2_1$, $a=8.972(1)$, $b=10.004(1)$, $c=10.302(1)$, $\beta=106.37(11)$, $Z=2$.



The structures has been solved by direct methods and refined by full-matrix least-squares to a final R=0.04 (1519 reflections) for I and R=0.06 (1331 reflections) for II. The absolute configuration was determined by Hamilton method and confirmed by comparing the Bijvoet's pairs. The molecule I adopts the R absolute configuration. The trans configuration of α -amino and β -carboxylate groups was observed for the first time in the derivative of the aspartic acid. The molecule II adopts the R absolute configuration. The elongation of the $C_{sp^3}-C_{sp^2}$ bond between C^α and $C(COOH)$ atoms was observed. The hydrogen bonds system was analyzed.

03.5-20 THE STRUCTURE OF 16 β -PHENOXYBUTYL-4-ANDROSTENE-3,17-DIONE. By W.L. Duax[†], Z. Gałdecki^{*}, P. Grochulski^{*}, A.J. Solo^{**} and Z. Wawrzak^{*}, [†]Medical Fundation of Buffalo, New York, 14203, U.S.A., ^{*}Technical University of Łódź, 90-924 Łódź, Poland, ^{**}State University, New York, Buffalo, N.Y. 14260 U.S.A.

The purpose of the structure determination is to verify positions of the substituents at C(16) and C(17). The compound crystallized in the monoclinic space group $P2_1$ with unit cell parameters: $a=19.803(4)$, $b=7.1389(7)$, $c=8.849(1)$ Å, $\beta=102.61(1)^\circ$, $V=1220.9$ Å³, $Z=2$, $D_x=1.181$ Mg m⁻³. The intensities of 2703 independent reflections were collected on a CAD4 diffractometer using $CuK\alpha$ radiation. The structure was solved by direct methods (MULTAN) using the weighted technique. The positions of all H atoms were found from difference Fouries syntheses. The structure refinement is in progress. Current R = 0.096. Dr. Solo, who synthesized the compound has assumed that the compound is a 4-cholesten derivative, but from X-ray analysis we have found, that it is 4-androsten one.



03.5-22 CRYSTAL AND MOLECULAR STRUCTURES OF SOME ACYLANILIDE FUNGICIDES. By J. C. J. Bart, R. Scordamaglia and M. Calcaterra, Istituto G. Donegani S.p.A. (Montedison), Novara, Italy.

Some microbicidal anilide derivatives which control *Phytophthora infestans* on potato, *Plasmopara viticola* on grape, *Pseudoperonospora cubensis* on squash, *Peronospora tabacina* and *Phytophthora nicotianae* on tobacco, *Pseudoperonospora humili* on hops, and *Sclerospora* on corn were analyzed by x-ray diffraction in order to gain information about the molecular conformation. The following active compounds were considered:

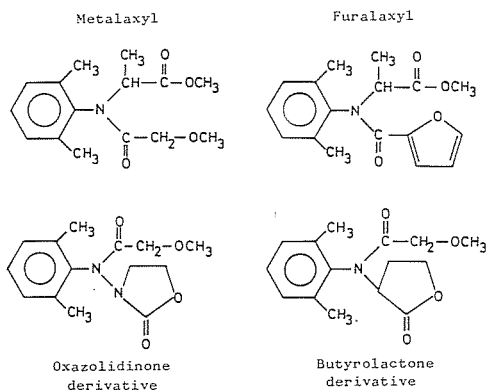
DL-methyl-N-(2,6-dimethylphenyl)-N-(2-methoxyacetyl)alaninate or metalaxyl ($P2_1/c$, $a=7.851(3)$ Å, $b=13.119(10)$ Å, $c=15.107(7)$ Å, $\beta=101.71(6)^\circ$, $Z=4$, $R=0.079$ for 1203 F(hkl));

DL-methyl-N-(2,6-dimethylphenyl)-N-(2-furoyl)alaninate or furaxyl (Cc , $a=15.997(12)$ Å, $b=7.929(7)$ Å, $c=14.064(11)$ Å, $\beta=118.70(5)^\circ$, $Z=4$, $R=0.068$ for 852 F(hkl));

N-(2-methoxyacetyl)-N-(2,6-xylyl)-3-amino-1,3-oxazolidin-2-one ($P2_1/n$, $a=9.363(1)$ Å, $b=12.715(1)$ Å, $c=12.360(1)$ Å, $\beta=102.64(10)^\circ$, $Z=4$, $R=0.062$ for 1175 F(hkl));

N-(2-methoxyacetyl)-N-(2,6-xylyl)- α -amino- γ -butyrolactone ($P2_1/n$, $a=9.600(3)$ Å, $b=11.964(8)$ Å, $c=12.802(4)$ Å, $\beta=101.25(3)^\circ$, $Z=4$, $R=0.055$ for 1686 F(hkl)).

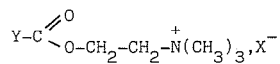
The molecular conformations of metalaxyl and furalaxyl are similar with the dimethylphenyl ring being almost perpendicular to the amidic plane of the molecule, as is also the case for the two isostructural compounds with the oxazolidinone and butyrolactone rings. The amidic nitrogen atom is sp^2 hybridized. As in most N-substituted anilides, metalaxyl and furalaxyl exist preferentially in the *exo* isomeric form with the phenyl ring *trans* with respect to the carbonyl group. The oxazolidinone ring is in a half chair form and the butyrolactone ring in a typical envelope conformation.



03.5-23 LOW TEMPERATURE STUDIES OF CHOLINE ESTERS

By Birthe Jensen, Department of Chemistry BC, Royal Danish School of Pharmacy, Universitetsparken 2, DK-2100 Copenhagen, Denmark.

The crystal structures of many salts of choline esters are known.



Strong thermal motion or disorders are often observed in these salts, and the accuracy of the structures is generally rather low. A survey of the known structures has disclosed a strong correlation between the magnitude of some bond angles and the actual conformation. Many types of theoretical studies of molecular flexibility still make use of crystallographically observed geometry, and it was found worth while to collect low temperature data for a selection of choline ester salts, in order to get more precise knowledge about the correct values of bond lengths and angles. Results will be presented for methoxycarbonylcholine iodide, carbamoylcholine chloride and bromide, acetylcholine chloride, and succinylcholine iodide, representing the three known conformers *trans-trans*, *trans-gauche* and *gauche-gauche*.