

03.2-11 CRYSTAL STRUCTURE OF GLYCINE ORTHOPHOSPHATE
By J. K. Mohana Rao* and R. Thulasidhass†, Department of Physics, Madurai-Kamaraj University, Madurai-625021, India. *Present address: Department of Biological Sciences, Purdue University, West Lafayette, Indiana 47907. †Present address: Department of Physics, G.V.N. College, Kovilpatti, Tamil Nadu, India.

Studies on amino acid phosphate compounds are expected to be an important source of information for understanding the protein-nucleic acid interactions and with that end in view, the crystal structure of the title compound was studied. Glycinium orthophosphate ($\text{NH}_3^+\text{CH}_2\text{COOH H}_2\text{PO}_4^-$) crystallizes in a tetramolecular unit cell of dimensions $a = 9.63$, $b = 7.89$, $c = 9.24$ Å, $\beta = 114^\circ$ and the space group is $P2_1/c$. Good single crystals were grown from a saturated aqueous solution containing glycine and orthophosphoric acid in stoichiometric proportions. Three-dimensional intensity data were collected by the multiple film equi-inclination Weissenberg technique using $\text{CuK}\alpha$ radiation. The crystal structure, solved by the Patterson and the Fourier methods, was refined to an R value of 0.08 for 1000 observed reflections. All hydrogen atoms except one was located. The amino acid exists as a positive ion ($\text{NH}_3^+\text{CH}_2\text{COOH}$) in this structure and there is a strong $\text{O}^-\cdots\text{H}^+\cdots\text{O}$ hydrogen bond between the carboxyl and the phosphate oxygens. The phosphate groups themselves are linked by hydrogen bonds and form extended chains along the b and c axes.

03.2-12 STUDIES ON CONFORMATION OF PROLYL RESIDUE IN PEPTIDES: THE CRYSTAL AND MOLECULAR STRUCTURE OF L-PROLYL-L-METHIONINE MONOHYDRATE. By V.S. Yadava and V. M. Padmanabhan, Neutron Physics Division, Bhabha Atomic Research Centre, Trombay, Bombay 400 085, India.

The prolyl residue can have two conformations - one with C^γ and C^δ atoms on the same side of $\text{NC}^\alpha\text{C}^\beta\text{C}^\delta$ plane and the other with C^δ on the opposite side of C^γ (Ramachandran et al. (1970), Biochem. et Biophys. Acta, 221, 165-181).

L-Prolyl-L-methionine crystallizes in the monoclinic space group $P2_1$ with $a = 19.385(4)$, $b = 5.482(1)$, $c = 6.414$ Å, $\beta = 93.21(8)^\circ$ and $Z = 2$. From the Trombay computer-controlled diffractometer data (1072 observed reflections), the crystal structure was solved by direct methods and refined by the least-squares procedure to an R index of 0.084.

The crystal structure is a disordered one. The pyrrolidine ring exists in two conformations in the ratio of 3:2, with C^γ atom of the ring statistically situated on both sides of $\text{NC}^\alpha\text{C}^\beta\text{C}^\delta$ plane. The bond lengths and bond angles for the peptide have values close to those expected except those for the pyrrolidine ring. The molecule is in the extended conformation ($\psi = 166^\circ$, $\phi = 70^\circ$) and in *trans* configuration ($\omega = 168^\circ$). The sulphur and the terminal methyl group have large thermal parameters. The hydrogen bonds through the water molecule stabilize the structure.

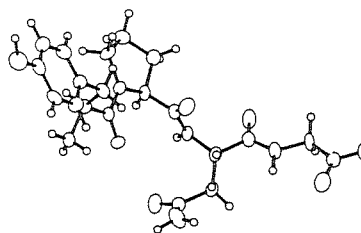
03.2-13 CRYSTAL STRUCTURE OF N-(p-AMINO BENZOYL)-L-GLUTAMIC ACID HYDROCHLORIDE. By Chandana Chatterjee, J.K. Dattagupta and N.N. Saha, Saha Institute of Nuclear Physics, 92 A.P.C. Road, Calcutta-700 009, India.

N-(p-Aminobenzoyl)-L-glutamic acid ($\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_5$), a major portion of folic acid, is a sulfanilamide antagonist. The title compound crystallises in the monoclinic space group $P2_1$ with $a=11.819(3)$, $b=4.924(1)$, $c=12.085(1)$ Å, $\beta=102.4(1)^\circ$, $Z=2$. The structure was solved by direct methods and refined by block-diagonal least-squares technique, with anisotropic temperature parameters for nonhydrogen atoms and isotropic ones for hydrogens, to an R value of 0.12 for 819 diffractometer data. Para-aminobenzoic acid part of the molecule is linked to glutamic acid via a peptide-like linkage with C-N distance of 1.33 Å. The side chain in glutamic acid is buckled with C^δ gauche to C^α with respect to $\text{C}^\beta-\text{C}^\gamma$ ($\chi^2=77.2^\circ$). The α -carboxyl C is *trans* to C^γ with a torsion angle of $\text{C}-\text{C}^\alpha-\text{C}^\beta-\text{C}^\gamma = -178.6^\circ$. The α -carboxylic group and the α -amino nitrogen are not coplanar, the angle of rotation (ψ') of the C-N bond from the plane of the α -carboxylic group being 26.7° . All the available hydrogen atoms take part in hydrogen bonding and the structure is stabilised by a three-dimensional network of hydrogen bonds of types $\text{N}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$. No intramolecular hydrogen bonds have been observed.

03.2-14 THE X RAY ANALYSIS OF HUMAN A.C.T.H. FRAGMENTS by G. Précigoux, B. Busetta, S. Georffre and M. Hospital, Laboratoire de Cristallographie, Université de Bordeaux I - 33405 - Talence-Cedex - France.

Among several crystallization trials with numerous fragments (or analogs) of human A.C.T.H., only two gave large enough crystals for X-ray study.

The tetrapeptide L-tyrosyl-L-prolyl-L-asparaginyl-L-glycine, the 23-26 fragment, crystallizes by free diffusion between a concentrated peptide solution in methanol-water and chloroform. The crystal is orthorhombic, $a = 8.896(2)$, $b = 12.858(3)$, $c = 18.146(4)$ Å, space group $P 2_1 2_1 2_1$ with four molecules per unit cell. The final R value is 0.033. The molecule exists in the crystal as a zwitterion. The peptide main chain is in extended conformation. The rather high density ($1.44 \text{ Mg}\cdot\text{m}^{-3}$) is explained by a strong intermolecular hydrogen bond network. There is no intramolecular hydrogen bond.



The tetrapeptide L-methionyl-L-glutamyl-L-histidyl-L-phenylalanine, the 4-7 fragment of A.C.T.H., crystallizes in the orthorhombic system with $Z = 4$, $a = 20.5$, $b = 27.7$, $c = 4.8$ Å.