03.2-11 CRYSTAL STRUCTURE OF GLYCINE ORTHOPHOSPHATE

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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 ($NH_3^+CH_2COOH\ H_2PO_4^-$) crystallizes in a tetramolecular unit cell of dimensions a = 9.63, b = 7.89, c = 9.24 Å, β = 114° 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 CuKα 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 ($NH_3^+CH_2COOH$) in this structure and there is a strong $O\cdots H\cdots O$ 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.

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The prolyl residue can have two conformations – one with C^γ and C' atoms on the same side of $NC^\alpha C^\beta C^\delta$ plane and the other with C^γ on the opposite side of C' (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, β = 93.21(8)° 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 $NC^\alpha C^\beta C^\delta$ 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 (ψ = 166°, ϕ = 70°) and in trans configuration (ω = 168°). 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-AMINOBENZOYL)-L-GLUTAMIC ACID HYDROCHLORIDE.

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N-(p-Aminobenzoyl)-L-glutamic acid ($C_{12}H_{14}N_2O_5$), 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, β=102·4(1)°, 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 $C^\beta—C^\gamma(\chi^2=77·2°)$. The α-carboxyl C is trans to C^γ with a torsion angle of $C-C^\alpha-C^\beta-C^\gamma = -178·6°$. The α-carboxylic group and the α-amino nitrogen are not co-planar, 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 N-H····Cℓ, O-H····O and N-H····O. No intramolecular hydrogen bonds have been observed.

03.2-14 THE X RAY ANALYSIS OF HUMAN A.C.T.H.FRAGMENTS

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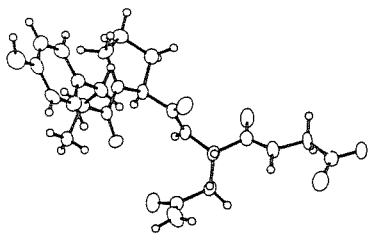
Among several crystallization trials with numerous fragments (or analogs) of human A.C.T.H., only two gave large enough crystals for X-ray study.

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 $P\ 2_12_12_1$ 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 $Mg.m^{-3}$) 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.