03.2-11  CRYSTAL STRUCTURE OF GLYCINE ORTHOPHOSPHATE  
By J.K. Mohana Rao* and R. Thulasidass†, 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 (NH₄)(CH₂COOH·H₂PO₄) crystallizes in a tetramolecular unit cell. Its dimensions are a = 9.63, b = 7.89, c = 9.24 Å, β = 114° and the space group is P2₁/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 0.084 for 1000 observed reflections. All hydrogen atoms except one were located. The amino acid exists as a positive ion (NH₄⁺,CH₂COOH) in this structure and there is a strong C-O···O hydrogen bond between the carboxyl and the phosphate oxygen. 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.P. Padmanabhan, Neutron Physics Division, Bhabha Atomic Research Centre, Trombay, Bombay 400 085, India.

The prolyl residue can have two conformations ε and c with Cα and Cγ atoms on the same side of NCαCγCβ plane and the other with Cα on the opposite side of Cγ (Ramachandran et al. (1970), Biochem. et Biophys. Acta, 221, 165-181).

L-Prolyl-L-methionine crystallizes in the monoclinic space group P2₁ with a = 19.385(4) Å, b = 5.482(1) Å, 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 Cα atom of the ring statistically situated on both sides of NCαCβCγ plane. The bond lengths and bond angles for the peptide have values close to those expected for those for the pyrrolidine ring. The molecule is in the extended conformation (φ = 166°, θ = 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-AMINOBENZYL]-L-GLUTAMIC ACID HYDROCHLORIDE.  
By Uranada Chatterjee, J.K. Pattnaik and N.W. Saha, Saha Institute of Nuclear Physics, 92 A.P.C. Road, Calcutta-700 009, India.

N-[p-Aminobenzyl]l-glutamic acid (C₁₄H₃₄N₂O₇), a major portion of folic acid, is a sulfanilamide antagonist. The title compound crystallizes in the monoclinic space group P2₁ 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 0.15 for 839 diffractometer data. Para-aminobenzoic acid part of the molecule is linked to glutamic acid via a peptide-like linkage with O·N distance of 1.35 Å. The side chain in glutamic acid is buckled with Cα gauche to Cγ with respect to Cβ-Cγ (X=77.2°).

The α-carboxyl group is trans to Cβ with a torsion angle of C-C-O·O=−178.6°. The α-carboxyl group and the α-amino nitrogen are not co-planar, the angle of rotation (Y) of the C-N bond from the plane of the α-carboxyl group being 26°. 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···O, O-H···O and N-H···C. No intramolecular hydrogen bonds have been observed.

03.2-14  THE X RAY ANALYSIS OF HUMAN A.C.T.H.FRAGMENTS.  
By C.Précigou, B.Busetta, S.Geoffre 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) Å, b = 12.858(2) Å, c = 18.146(4) Å, space group P 2₁2₁2₁, with four molecules per unit cell. The final R value is 0.033. The molecules exist in the crystal as a swirritation. 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-glutamy1-L-histidyl-L-phenylalanine, the 4-7 fragment of A.C.T.H., crystallizes in the orthorhombic space group system with a = 4.3, b = 20.5, c = 27.7 Å.