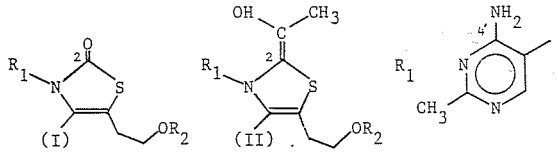
03. 5–1 CRYSTAL STRUCTURE OF THIAMIN THIAZOLONE – A TRANSITION-STATE ANALOG FOR THIAMIN PYROPHOSPHATE DEPENDENT ENZYMES. By Whanchul Shin and Young Chang Kim, Department of Chemistry, College of Natural Sciences, Seoul National University, Seoul 151, Korea

The structure of thiamin thiazolone(TT;I) resembles that of the metastable enamine(II), which is the immediate product of the decarboxylation of the pyruvate adduct, and its pyrophosphate binds to the thiamin-PP sites of the *E.coli* pyruvate dehydrogenase complex at least $2x10^4$ times more tightly than does thiamin-PP itself.



TT crystallizes in the monoclinic space group $P2_1/n$ with a=4.634(2), b=12.591(6), c=22.291(10)A, β=95.20(4)°, Z=4. The structure was solved by direct methods and refined to R=0.041 for 987 observed reflections measured with Cu Kα radiation on a diffractometer. Molecular conformation of TT containing a neutral 2-thiazolone instead of a thiazolium ring is quite different from either that(F) of thiamin or that(S) of C(2)-substituted thiamin. TT assumes a V conformation (ϕ_T=104°,ϕ_P=-74°), that has been observed in oxythiamin which is a strong antagonist of thiamin. The 4'-amino group of TT is hydrogen-bonded to C(2) oxygen. This is the first crystal structure that shows an *intramolecular* interaction of the 4'-amino group whose functional role in thiamin catalysis is not well established.

This research was supported by the grants from the Korea Science and Engineering Foundation(KOSEF).

03. 5–2 STRUCTURE OF DL-NORMETANEPHRINE HYDROCHLORIDE. By Rekha R. Pattanayek, J.K.Dattagupta, S.C.Bhattacharyya and N.N.Saha CεMB Division, Saha Institute of Nuclear Physics, Sector-I,Block'AF', Bidhan Nagar, Calcutta-700 064, India.

It is well known that the enzyme catechol O-methyl transferase when transfers the methyl group of S-adenosylmethionine to the 3-hydroxyl group of epinephrine and norepinephrine two metabolic products metanephrine and normetanephrine are formed in vivo, which constitute a major metabolic pathway of epinephrine and norepinephrine. The crystal structure of DL-metanephrine hydrochloride has already been reported by us (Acta Cryst(1983),C39, 91) and that of DL-normetarephrine hydrochloride forms the subject matter of the present paper. It crystallizes in the space group $P2_1/c$ with a=5·218(1), b=17·081(4), c=12·260(2)Å,β=91·50°(1), V=109·2 Å^3,Z=4, D_m=1·340, D_c=1·336 Mgm^{-3}, λ(Mo kα)=0·71073 Å, R=0·069 for 1651 reflections. The ethylamine side cnain is nearly planar, maximally extended and perpendicular to tne attacned pnenyl ring whicn is also planar. The distance of the amino nitrogen from the centre of the pnenyl ring is 5·11 Å, It has been observed that N-atom is gaucne to the hydroxyl group of the side cnain as in p-hydroxyepnedrine hydrochloride by us (Acta Cryst (1981),B37, 1439). The conformational features of the present molecule wnich incidentally has no direct drug action are similar to those of functionally active adrenergic drugs. It, therefore, appears tnat tne conformational features are not the only criteria for the biological activity.

03. 5–3 CRYSTAL STRUCTURE OF 3,4-DIMETHOXY-PHENETHYLAMINE (DMPEA) HYDROCHLORIDE DIHYDRATE ($C_{10}H_{16}O_2N^+Cl^-,2H_2O$). Rekha R. Pattanayek, J.K.Dattagupta ε N.N.Saha CεMB Division, Saha Institute of Nuclear Physics,Sector-I,Bl.'AF' Bidhan Nagar, Calcutta-700 064, India.

Crystal structures of some sympathomimetic drug molecules with bulky substituent groups at different sites are being studied by us in order to get a better idea as to which site or sites are more responsible for drug action. Here in DMPEA two methoxy groups have been substituted in the m- and p- positions of the benzene ring of the phenethylamine. DMPEA which is found in urine and in certain tissues, has no direct drug action but it forms physiologically active metabolite. N-acetyl DMPEA. Single crystal of DMPEA hydrochloride crystallises in monoclinic space group $P2_1/c$ with a=11·590(4), b=13·780(3), c=8·301(4) Å, β=94·89°(4) and Z=4. The crystal structure has been solved by heavy-atom method using diffractometric data. The structural parameters have been refined by full matrix least-squares method. In contrast to the conformation usually adopted by active sympathomimetic amines, the etnylamine sidechain in this compound is folded(τ_1=118·05, τ_2=-64·86 ε distance of N atom from centre of benzene ring, D_N=3·97 Å). It appears that the bulky methoxy groups have influenced the conformation of the molecule and its activity.

03. 5–4 STRUCTURES OF THREE ADENINE-CONTAINING COMPOUNDS. By V. Langer and K. Huml, Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, 162 06 Praha 6, Czechoslovakia.

The crystal and molecular structures of three adenine-containing compounds have been determined as part of a study of the transfer of energy and information in nucleic acids and their components. Single crystals serve as models for the interpretation of the results obtained by various methods of low-temperature reflection and emission spectroscopy for polynucleotides. The compounds under study were: 1:2 complex of adenine and N-methyl-2-pyrrolidone, $C_5H_5N_5.2C_5H_9NO$ (A1), adeninium bis-monochloracetate, $C_5H_5N_5.2CClH_2COOH$ (A2) and adeninium bis-trichloracetate, $C_5H_5N_5.2CCl_3COOH$ (A3). The summary of crystallographic data is given in the following table:

compound	A1	A2	A3
mol. weight	333.40	324.14	461.94
space group	$P2_1/c$	Pccn	$P2_1$
a (Å)	9.907(5)	24.154(2)	5.818(2)
b	21.962(7)	15.532(1)	26.173(9)
c	7.994(2)	7.136(1)	11.393(5)
β (°)	100.73(3)	90.00(0)	95.60(3)
Z	4	8	4
Dx (gcm^{-3})	1.296(1)	1.608(1)	1.776(1)
Dm	1.275(5)	1.591(4)	1.732(8)
obs. reflect.	1097	2904	2087
R	0.057	0.085	0.117

A comparison of molecular geometry and crystal packing as a function of degree and site of protonation of adenine molecule will be given.