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Crystal Structure of Arginine Diethyl Phosphate

S. FURBERG and J. SOLBAKK

Department of Chemistry, University of Oslo, Oslo 3, Norway

The crystal structure of the arginine salt of diethyl phosphoric acid has been derived by X-ray analysis based on 568 observed reflections measured on a photometer. The space group is $P3_121$, with $a = 9.25$ Å and $c = 34.25$ Å. The accuracy is low, with $R = 0.09$ and standard deviations of 0.02 – 0.09 Å in bonds between non-hydrogen atoms. The conformation of the diethyl phosphate ion is *gauche* about both P – O ester bonds. The protonated arginine zwitterions are linked together by two pairs of N–H···O hydrogen bonds to form ring-shaped dimers. The crystal consists of layers (001) of hydrogen-bonded ions.

In order to obtain information on the stereochemistry of the interaction between nucleic acids and basic proteins we have investigated the crystal structures of a number of salts between diethyl phosphoric acid and compounds related to arginine and lysine. The structures of the propylguanidinium [1] and putrescinium [2] salts have been reported and in this paper the arginine complex is described. The purpose of the work is to study the mode of bonding and the relative orientation of the two ions, not to derive accurate bond lengths and angles.

EXPERIMENTAL. CRYSTAL DATA

The compound was prepared by mixing equivalent amounts of arginine and diethyl phosphoric acid (prepared as previously described [1]) in aqueous solution. The solution was placed in a closed system containing some ethanol and crystals of the salt gradually precipitated. They had the shape of trigonal pyramids and cleaved very easily perpendicular to the three-fold axis.

Weissenberg photographs showed the crystals to be trigonal with Laue symmetry $\bar{3}m$. The $00l$ reflections are present only for $l = 3n$ and the space group was assumed to be $P3_121$. The cell dimensions were measured on a manual Picker diffractometer and found to be $a = 9.251(3)$ Å and $c = 34.25(2)$ Å. Flotation in mixtures of bromobenzene and *m*-xylene gave a density of 1.30 g/cm^3. There are six (calc. 6.05) formula units $(C_2H_5O)_2PO(OH).HOOC.CH(NH_2)(CH_2)_3.NH.C(NH)NH_2$ in the unit cell.

The reflections from a crystal of dimensions 0.2 – 0.4 mm were recorded on integrated Weissenberg photographs $h0l - h3l$ using Cu$K\alpha$ radiation. Their

Table 1. Positional and thermal parameters with estimated standard deviations. All values except those of B are multiplied by 10^4. The temperature factor is given by $\exp -(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)$.

Atom	x	y	z	B_{11}(B)	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
P	2811	0498	2479	111	128	10	138	15	11
	7	7	2	11	11	1	20	5	5
O1	2256	1743	2316	93	213	19	150	54	78
	16	18	4	29	36	2	60	13	14
O2	3469	−0035	2111	273	423	18	406	−8	−32
	24	25	5	42	52	2	81	16	19
O3	4340	1343	2718	136	177	17	121	−14	−17
	17	18	4	31	32	2	54	12	13
O4	1323	−0836	2656	220	251	19	342	12	32
	19	20	5	35	39	2	65	15	15
C1	3458	3152	2127	232	256	12	216	−9	39
	32	32	6	60	59	3	101	21	22
C2	2944	3757	1863	610	998	44	433	9	346
	58	76	14	149	229	8	310	62	79
C3	2276	−0957	1834	980	737	55	627	9	−238
	83	73	17	245	190	11	383	86	80
C4	2315	−2469	1741	218	1035	44	413	46	−75
	47	80	14	86	214	8	234	45	68
O5	5512	5371	2903	6.2					
	19	19	4	.4					
O6	3548	6092	2978	5.3					
	19	19	4	.4					
N1	7608	8893	4189	5.2					
	23	23	5	.4					
N2	9519	11581	4062	4.7					
	21	21	5	.4					
N3	6745	10670	4004	6.0					
	24	25	5	.5					
N4	4259	2906	3423	3.8					
	20	20	4	.4					
C5	7970	10370	4079	5.4					
	30	29	6	.6					
C6	5958	7421	4190	5.5					
	31	29	6	.5					

Table 1. Continued.

C7	5663	6550	3826	5.0
	27	27	6	.5
C8	3846	5056	3786	4.3
	27	27	5	.5
C9	3510	4043	3419	3.3
	24	24	5	.4
C10	4194	5155	3076	5.2
	29	28	6	.5

intensities were measured on a photometer. The layers were scaled by equivalent reflections. The fall-off in intensity was rapid and only 568 independent reflections had measurable intensities. Corrections for absorption and secondary extinction were not applied. All programs used are described in Ref. 3.

Table 2. Distances (Å) and angles (°). E.s.d. in parenthesis.

P–O1	1.58(1)	O1–P–O2	105(1)
P–O2	1.58(2)	O1–P–O3	113(1)
P–O3	1.48(2)	O1–P–O4	105(1)
P–O4	1.44(2)	O2–P–O3	101(1)
O1–C1	1.38(3)	O2–P–O4	115(1)
C1–C2	1.27(4)	O3–P–O4	118(1)
O2–C3	1.38(6)	P–O1–C1	117(1)
C3–C4	1.45(9)	O1–C1–C2	116(3)
O5–C10	1.28(3)	P–O2–C3	115(3)
O6–C10	1.32(3)	O2–C3–C4	110(5)
C9–C10	1.48(3)	O5–C10–O6	119(2)
C9–N4	1.52(3)	O5–C10–C9	121(2)
C9–C8	1.51(3)	O6–C10–C9	119(2)
C8–C7	1.56(3)	N4–C9–C10	108(2)
C7–C6	1.43(3)	C8–C9–C10	110(2)
C6–N1	1.45(3)	N4–C9–C8	113(2)
N1–C5	1.29(3)	C9–C8–C6	114(2)
C5–N2	1.31(3)	C8–C7–C7	113(2)
C5–N3	1.32(3)	C7–C6–N1	110(2)
		C6–N1–C5	126(2)
Hydrogen bonds		N1–C5–N3	119(2)
N1···O1′	3.08	N1–C5–N2	121(2)
N2···O4′	2.86	N2–C5–N3	120(2)
N2···O5′	2.78		
N3···O3′	3.01		
N3···O6′	2.93		
N4···O4′	2.79		
N4···O6′	2.77		
N4···O3	2.84		

Prime denotes an atom in any neighbouring ion.

Table 3. Observed and calculated structure factors.

h k	l	Fo	Fc
0 0	3	100	94
0 0	6	154	185
0 0	9	106	113
0 0	12	68	72
0 0	15	30	28
0 0	18	27	28
0 0	21	17	10
0 0	24	45	53
1 0	29	14	22
1 0	28	21	22
1 0	27	12	10
1 0	26	13	15
1 0	25	17	21
1 0	24	13	17
1 0	22	13	14
1 0	21	14	15
1 0	18	14	13
1 0	17	28	26
1 0	16	27	26
1 0	15	33	38
1 0	14	42	50
1 0	13	24	22
1 0	12	91	100
1 0	11	12	11
1 0	10	43	35
1 0	9	84	79
1 0	8	72	63
1 0	7	35	37
1 0	6	107	110
1 0	5	45	43
1 0	4	116	120
1 0	3	68	72
1 0	2	66	67
1 0	1	73	72
1 0	0	33	28
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1 0	-3	46	38
1 0	-5	122	120
1 0	-6	114	117
1 0	-7	159	177
1 0	-8	16	16
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1 0	-12	70	71
1 0	-13	42	48
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2 0	-2	64	66
2 0	-3	110	115
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2 0	-8	45	45
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2 0	-16	14	14
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5 0	-6	15	14
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2 3	-20	13	9
2 3	-22	16	9
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5 3	6	16	12
5 3	7	22	24
5 3	8	18	14
5 3	9	17	15
5 3	10	14	11
5 3	12	15	12
5 3	14	13	9

STRUCTURE ANALYSIS

The position of the phosphorus atom was derived from a sharpened Patterson map and repeated weighted Fourier syntheses eventually led to the correct structure. It was refined by block diagonal least squares calculations. In view of the small number of reflections, anisotropic temperature factors were applied only to the atoms of the diethyl phosphate ion. The atoms of the arginine ion were given individual isotropic temperature factors. A common isotropic temperature factor was assigned to the 25 hydrogen atoms, which were included in fixed calculated positions. It refined to $B = 6.6$ Å^2. The final value of R is 0.09, of R_w 0.12. The structure factors were given weights $(a + F + cF^2)^{-1/2}$, with $a = 2F_{min}$ and $c = 2/F_{max}$.

The atomic parameters are given in Table 1 and observed and calculated structure factors in Table 3. The atomic scattering factors used were those of Hanson *et al.*[4] for non-hydrogen atoms and of Stewart *et al.*[5] for hydrogen atoms.

RESULTS AND DISCUSSION

The bond lengths and angles are given in Table 2 and Fig. 1. They are all normal within the limits of error. The accuracy is low because of the small number of reflections and the large thermal vibrations of the ethyl groups, and only the general features of the molecular and crystal structure will be discussed.

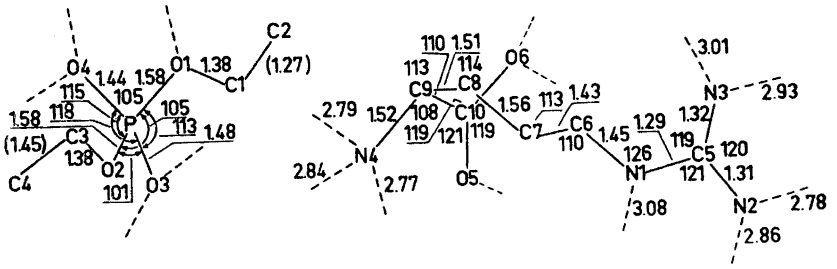

Fig. 1. Bond lengths (Å) and angles (°).

The diethyl phosphate ion. The structure of this ion is essentially the same as found in the propylguanidinium salt,[1] the conformation being *gauche* about both P – O ester bonds (dihedral angles O2 – P – O1 – C1 and O1 – P – O2 – C3 are 58.4° (1.8°) and 66.0° (3.5°), respectively). However, the positions of the terminal methyl groups deviate much from the expected *anti* conformation, the dihedral angles about bonds C3 – O2 and C1 – O1 being 151° and 128°, respectively. These deviations are probably due to crystal forces. They bring the methyl hydrogen atoms closer to layers (001) of the ethyl hydrogen atoms in the structure. The ethyl groups vibrate strongly the mean B-values for C1, C2, C3, and C4 being 6.5, 18.8, 17.3, and 21.3 Å^2, respectively, whereas they lie in the range 3.3 – 7.3 Å^2 for the other atoms.

The arginine ion. The arginine molecule occurs in the crystal as a protonated zwitterion (-OOC).CH(NH_3^+)(CH_2)$_3$(NH)C(NH_2)$_2^+$, as found in other salts of arginine.[6,7] This is indicated mainly by the scheme of hydrogen bonding, the α-amino group taking part in three such bonds, the guanidyl group in five.

The conformation of the arginine molecule in four different crystals has been discussed by Ramachandran *et al.*[6] In all structures nearly planar guanidyl and carboxylate groups are linked together by a chain of coplanar carbon atoms, but the dihedral angles about $C_\alpha - C_\beta$ and C_δ – N vary and the molecules have different conformations in the crystals. In the present structure the atoms of the carboxylate group (C9, C10, O6, and O5) are coplanar to within 0.04 Å, those of the guanidyl group (N1, C5, N2, and N3) to within 0.02 Å, whereas the maximum deviation of atoms C9, C8, C7, C6, and N1 from their best plane is 0.05 Å. The angles between the latter plane and those of the carboxylate and guanidyl groups are 89° and 82°, respectively. Atom N4 of the NH_3^+ group is 0.31 Å away from the carboxylate plane, and C6 0.13 Å from the guanidyl plane. The dihedral angle C7 – C8 – C9 – N4 is 75° (C9 – H *anti* to C7 – C8) and the one about C6 – N1 (C_δ – N) is 91°. Each of these values is

similar to what is found in structures previously investigated.[6] However, in none of these this particular combination of the two dihedral angles occurs and the present structure appears to represent an arginine conformation previously not observed. Its main characteristics is that the carboxylate and guanidyl groups point to the same side of the C9 – C8 – C7 – C6 – N1 chain, with C – O and C – N bonds very roughly parallel. This conformation would appear stereochemically favourable for the formation of cyclic dimers through pairs of N – H···O hydrogen bonds, as found in the present structure.

The crystal structure. The crystal consists of layers (001) of thickness $c/3$ (11.4 Å) related by the 3_1 axis. One layer is shown in Fig. 2. The layers have

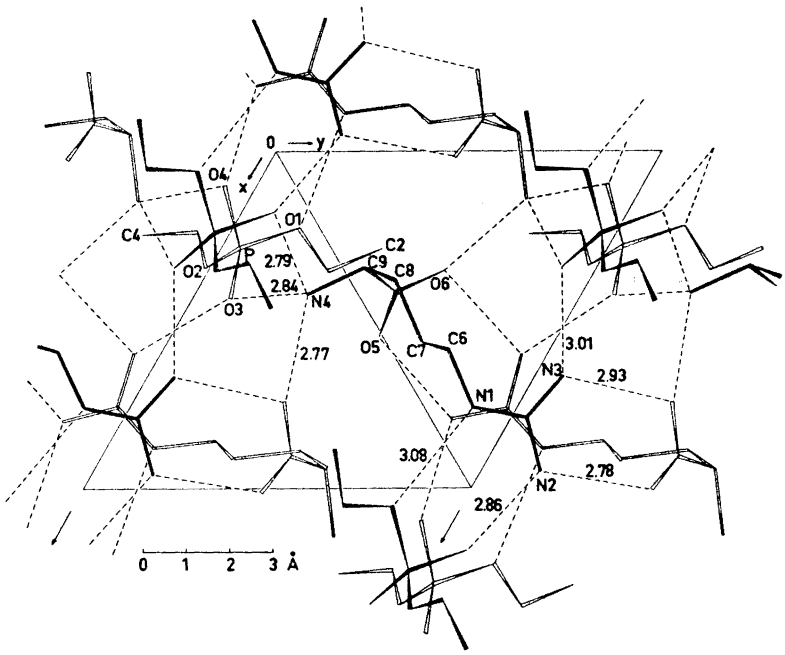

Fig. 2. One layer of the structure viewed along the 3_1 axis.

twofold axes of symmetry and the ethyl groups of adjacent layers interlock so as to give corrugated sheets of ethyl groups at roughly van der Waals distances from each other. Between these sheets are the phosphate and arginine ions which are held together by an extensive system of N – H···O hydrogen bonds, one for each hydrogen atom bonded to nitrogen. The main structural unit in a layer is a dimer of arginine ions related by a twofold axis of symmetry and held together by two pairs of hydrogen bonds (lengths 2.78 Å and 2.93 Å) between the guanidyl group in one molecule and the carboxylate group in the other, forming an 18-membered ring system. Two of the hydrogen bonds between a dimer and neighbouring diethyl phosphate ions, N1 – H···O1 (length 3.08 Å) and N2 – H···O4 (length 2.86 Å), are nearly parallel and link

the guanidyl group to two oxygen atoms in the same diethyl phosphate ion. Ester oxygen atoms like O1 are in general not involved in hydrogen bond formation, but in the present structure the distance H···O1 of 2.04 Å is considerably smaller than van der Waals contact and it seems reasonable to consider it a weak hydrogen bond. A similar bond (3.09 Å) occurs also in the propylguanidinium salt.[1]

The guanidyl group takes part in five hydrogen bonds, all of which are approximately linear and roughly lying in the guanidyl plane. The NH_3^+ group forms three hydrogen bonds (lengths 2.78 Å, 2.84 Å, and 2.81 Å) in nearly tetrahedral arrangement, two to different diethyl phosphate groups and the third to O6 in a neighbouring dimer.

There are no hydrogen bonds between the layers. This explains the good (001) cleavage of the crystals.

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