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Three Crystalline Forms of 9-(2-Hydroxyethyl)adenine Resulting from the Different Stacking of Hydrogen-Bonded Layers

BY AKIO TAKENAKA, MASAYUKI SHIBATA AND YOSHIO SASADA

Faculty of Science, Tokyo Institute of Technology, Nagatsuta, Midori-ku, Yokohama 227, Japan

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Abstract. Three crystalline forms of the title compound [$C_7H_9N_5O$, $M_r = 179.2$, $\lambda(\text{Cu } K\alpha) = 1.54184 \text{ \AA}$, room temperature] were examined by X-ray diffraction. Form (I): orthorhombic, $P2_12_12_1$, $a = 13.566$ (2), $b = 8.271$ (1), $c = 7.340$ (1) \AA , $V = 823.6$ (2) \AA^3 , $Z = 4$, $D_x = 1.445 \text{ g cm}^{-3}$, $F(000) = 376$, $\mu = 8.88 \text{ cm}^{-1}$, $R = 0.070$, 701 observed reflexions. Form (II): orthorhombic, $P2_12_12_1$, $a = 13.572$ (3), $b = 8.248$ (2), $c = 14.508$ (3) \AA , $V = 1624.1$ (6) \AA^3 , $Z = 8$, $D_m = 1.466$, $D_x = 1.466 \text{ g cm}^{-3}$, $F(000) = 752$, $\mu = 9.01 \text{ cm}^{-1}$, $R = 0.047$, 1478 observed reflexions. Form (III): monoclinic, $P2_1/c$, $a = 13.543$ (1), $b = 8.290$ (1), $c = 22.591$ (2) \AA , $\beta = 104.97$ (1)°, $V = 2450.2$ (4) \AA^3 , $Z = 12$, $D_x = 1.457 \text{ g cm}^{-3}$, $F(000) = 1128$, $\mu = 8.96 \text{ cm}^{-1}$, $R = 0.105$, 2339 observed reflexions. In the three forms, the values of a and b remain nearly the same, but those of c are in the ratio of 1:2:3. In all three structures, adenine moieties are connected by N(6)H...N(1) and N(6)H...N(7) hydrogen bonds to form infinite ribbons along b , and the ribbons are linked side by side *via* O(12)H...N(3) to construct hydrogen-bonded pleated sheets. The sheet in form (I) consists of one crystallographic kind of molecule, A , while that in form (II) contains two kinds of molecules, B and C . Form (III) has two kinds of sheet: one consists of D and the other of E and F molecules. There are no significant differences in molecular dimensions among the molecules A – F , and the four kinds of sheets are almost the same. However, three different stacking modes exist between successive layers. Different combinations of the stacking modes result in poly-

morphism. Disorder along c^* observed in forms (I) and (III) and twinning in form (III) are interpreted in terms of the stacking modes.

Introduction. Polymorphism, twinning and disorder owing to layer stacking can give much information for intermolecular interactions of complicated molecules. However, detailed reports on these crystallographic aspects for organic substances are not common, one example being that of *o*-chlorobenzamide (Kato, Takaki & Sakurai, 1974).

The present compound, which was synthesized as a model of protein–nucleic acid interactions, gives rise to three crystalline forms; the values of one of the lattice parameters are in the approximate ratio 1:2:3, while the other two do not change significantly. Forms (I) and (III) reveal features of disorder about this multiplicable lattice vector, dependent on the crystallization conditions, and form (III) is also constituted of finely polysynthetic twins. The present paper describes the varieties of stacking of the typical hydrogen-bonded molecular layers, found in the structures of the three forms.

Experimental. The title compound was kindly supplied by Dr Minoru Ohki, Research Institute, Wakamoto Pharmaceutical Co. Ltd. By cooling a saturated aqueous solution of the compound, plate crystals were deposited. Observation under a microscope indicated that the crystals were composites of fine lamellae, whose end-on photograph (Fig. 1) suggests poly-

synthetic twins. Weissenberg photographs about the a and b axes showed appreciable streaks along c^* and twinning, the twinning plane being the ab plane. These crystals are called form (III), since the value of c is about three times that of form (I) described next.

Evaporation of an unsaturated aqueous solution gave bicuneiform crystals, and the crystal face exhibited fine stripes. Streaks along c^* were observed in Weissenberg photographs. These crystals are referred to as form (I).

If a dilute aqueous solution was kept for a long time, slow evaporation produced columnar crystals. Weissenberg photographs indicated neither apparent disorder nor twinning. From the value of c , these crystals are called form (II).

The crystal of form (I) giving minimum streaks was carefully chosen, and that of form (III) was cut out through the cleavage plane ab to exclude the other twinning component, although the streaks remained.

Rigaku four-circle diffractometer, Cu $K\alpha$ radiation, ω -scan speed 8° min^{-1} . Five reference reflexions monitored every 50 reflexions, no significant changes. Corrections for Lorentz and polarization effects, but not for absorption. Standard deviations assigned by $\sigma^2(|F_o|) = \sigma_p^2(|F_o|) + q|F_o|^2$, where σ_p was from counting statistics and q was estimated from the intensity variation of the reference reflexions (McCandlish & Stout, 1975). Experimental details for the individual crystals are given in Table 1.

Structures were solved by the direct method and refined by block-diagonal least squares minimizing $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2(F_o)$. All H atoms in form (II) and some of those in forms (I) and (III) were found on difference maps. Details of the structure determination are listed in Table 2. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *LSAP80* (Takenaka & Sasada, 1980), *DCMS82* (Takenaka & Sasada, 1982) and *LISTUP* (Takenaka & Sasada, 1983).

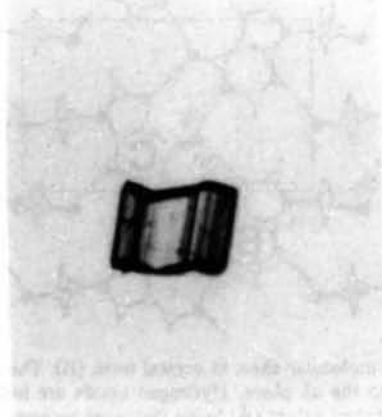


Fig. 1. End-on photograph of the crystal of form (III).

Table 1. Details of the experiments

	Form (I)	Form (II)	Form (III)
Crystal size (mm)	0.4 × 0.3 × 0.05	0.8 × 0.5 × 0.5	0.25 × 0.2 × 0.1
For cell parameters			
No. of reflexions	30	30	43
range of 2θ ($^\circ$)	16.1 ~ 38.4	33.6 ~ 50.8	19.2 ~ 44.3
For intensity measurement			
scan width ($^\circ$ in ω)	1.5	2.2	2.0
range of 2θ ($^\circ$)	3 ~ 125	3 ~ 125	3 ~ 125
range of h	0 ~ 15	0 ~ 15	-15 ~ 15
range of k	0 ~ 9	0 ~ 9	0 ~ 9
range of l	0 ~ 8	0 ~ 16	0 ~ 24
No. of reflexions	793	1508	3522
No. of zero-reflexions	90	26	1099
q	4.13×10^{-5}	1.35×10^{-5}	3.45×10^{-5}
F_{int}	1.22	0.75	3.63

Table 2. Details of structure analysis

	Form (I)	Form (II)	Form (III)
No. of reflexions ($F_o > 3\sigma$)	701	1478	2339
R	0.070*	0.047	0.105*
wR	0.074	0.045	0.071
S	3.859	5.152	2.722
$\Delta\rho_{\text{max}}$ ($e \text{ \AA}^{-3}$)	0.36	0.32	0.77
Maximum shift (σ)	x, y, z B	x, y, z B	x, y, z B
C	0.05 0.04	0.04 0.02	0.23 0.34
N	0.02 0.03	0.02 0.03	0.32 0.25
O	0.06 0.03	0.03 0.03	0.34 0.20
H	0.42 2.51	0.43 0.27	2.25 1.21

* Somewhat high owing to disorder.

The final atomic coordinates and equivalent isotropic temperature factors are given in Table 3.* The atomic numbering system is illustrated in Fig. 2. Crystals of forms (I), (II) and (III) contain one, two and three independent molecules, respectively. These are referred to as molecules A – F .

Discussion. Molecular structures. Table 4 shows the bond distances in the six independent molecules observed in the three crystalline forms. The molecular dimensions of the adenine moieties in the two independent molecules in form (II) agree well with those in the related compounds, for example, adenosine (Lai & Marsh, 1972). In view of the large standard deviations for forms (I) and (III), it is meaningless to discuss subtle variations in the individual bond distances, especially in molecules A , D , E and F . Comparison of the molecular dimensions as a whole, however, suggests that A is different from B or C . On the other hand, the differences between A and F and those among B , C , D and E are not significant. As will be shown below, molecules A and F have a very similar environment and so do molecules B , C , D and E . It is interesting that the environmental change may affect the molecular dimensions. The conformational difference of the hydroxyethyl group will be discussed below.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond angles, deviations of atoms from the least-squares planes, detailed geometries of hydrogen bonds and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42765 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Fractional coordinates and equivalent isotropic temperature factors

B values are calculated from the anisotropic thermal parameters: $B = 8\pi^2(U_1 + U_2 + U_3)/3$, where U_1 , U_2 and U_3 are the principal components of the mean-square displacement matrix U . Values in $\langle \rangle$ are the anisotropy defined by $[\sum(B - 8\pi^2 U_i)^2/3]^{1/2}$ and those in $()$ are e.s.d.'s referring to the last decimal place.

	x	y	z	$B(\text{\AA}^2)$
(a) Form (I)				
N(1)A	0.3907 (4)	-0.3913 (7)	-0.203 (1)	3.2 (21)
C(2)A	0.2983 (6)	-0.432 (1)	-0.179 (2)	4.3 (26)
N(3)A	0.2156 (4)	-0.3433 (7)	-0.173 (1)	3.5 (23)
C(4)A	0.2406 (5)	-0.1856 (8)	-0.196 (1)	2.8 (9)
C(5)A	0.3351 (5)	-0.1232 (8)	-0.224 (1)	2.4 (16)
C(6)A	0.4130 (5)	-0.2308 (8)	-0.225 (1)	2.6 (9)
N(6)A	0.5074 (5)	-0.1929 (8)	-0.248 (1)	2.9 (17)
N(7)A	0.3301 (4)	0.0470 (7)	-0.235 (1)	2.8 (11)
C(8)A	0.2345 (5)	0.0798 (8)	-0.220 (1)	3.3 (16)
N(9)A	0.1768 (4)	-0.0549 (7)	-0.1923 (9)	2.9 (13)
C(10)A	0.0705 (5)	-0.060 (1)	-0.164 (1)	3.2 (17)
C(11)A	0.0174 (6)	-0.087 (1)	-0.340 (1)	3.9 (20)
O(12)A	-0.0845 (3)	-0.1065 (7)	-0.304 (1)	3.9 (20)

	x	y	z	$B(\text{\AA}^2)$
(b) Form (II)				
N(1)B	0.2964 (2)	-0.4004 (4)	-0.1016 (3)	3.4 (17)
C(2)B	0.2025 (3)	-0.4448 (5)	-0.0870 (4)	4.2 (23)
N(3)B	0.1223 (2)	-0.3522 (4)	-0.0837 (3)	3.6 (18)
C(4)B	0.1463 (3)	-0.1949 (5)	-0.0948 (3)	2.6 (7)
C(5)B	0.2393 (2)	-0.1318 (5)	-0.1091 (3)	2.6 (6)
C(6)B	0.3177 (2)	-0.2432 (4)	-0.1132 (3)	2.6 (8)
N(6)B	0.4119 (2)	-0.2028 (4)	-0.1278 (3)	3.1 (14)
N(7)B	0.2354 (2)	0.0364 (4)	-0.1172 (2)	3.0 (11)
C(8)B	0.1405 (3)	0.0690 (5)	-0.1069 (3)	3.1 (11)
N(9)B	0.0835 (2)	-0.0641 (4)	-0.0940 (2)	2.9 (10)
C(10)B	-0.0246 (3)	-0.0684 (6)	-0.0840 (3)	3.6 (16)
C(11)B	-0.0722 (3)	-0.0817 (7)	-0.1777 (3)	4.2 (19)
O(12)B	-0.1752 (2)	-0.1087 (4)	-0.1685 (3)	5.5 (35)
N(1)C	-0.4835 (2)	0.1006 (4)	-0.1478 (3)	3.2 (14)
C(2)C	-0.3890 (3)	0.0584 (5)	-0.1535 (4)	3.9 (20)
N(3)C	-0.3090 (2)	0.1525 (4)	-0.1538 (3)	3.4 (16)
C(4)C	-0.3344 (3)	0.3086 (4)	-0.1469 (3)	2.6 (5)
C(5)C	-0.4290 (2)	0.3710 (4)	-0.1389 (3)	2.4 (5)
C(6)C	-0.5069 (2)	0.2583 (4)	-0.1412 (3)	2.5 (6)
N(6)C	-0.6020 (2)	0.2992 (4)	-0.1386 (3)	3.4 (18)
N(7)C	-0.4263 (2)	0.5398 (4)	-0.1344 (3)	3.1 (11)
C(8)C	-0.3324 (3)	0.5734 (5)	-0.1413 (3)	3.4 (12)
N(9)C	-0.2729 (2)	0.4405 (4)	-0.1485 (2)	2.9 (11)
C(10)C	-0.1657 (2)	0.4387 (6)	-0.1620 (3)	3.3 (14)
C(11)C	-0.1124 (3)	0.4013 (6)	-0.0733 (3)	3.7 (16)
O(12)C	-0.0097 (2)	0.3828 (4)	-0.0927 (2)	3.9 (18)

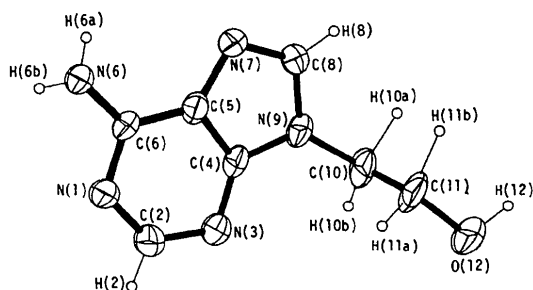
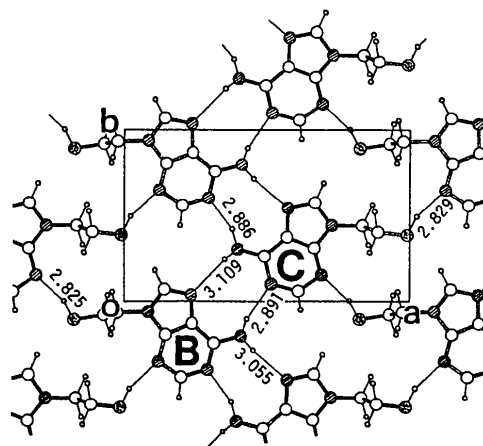
	x	y	z	$B(\text{\AA}^2)$
(c) Form (III)				
N(1)D	0.3210 (4)	-0.5140 (6)	0.0912 (3)	3.0 (22)
C(2)D	0.2319 (5)	-0.5619 (9)	0.0982 (4)	3.9 (25)
N(3)D	0.1526 (4)	-0.4703 (7)	0.1042 (3)	3.6 (24)
C(4)D	0.1756 (5)	-0.3100 (9)	0.1027 (3)	2.6 (7)
C(5)D	0.2664 (5)	-0.2474 (8)	0.0968 (3)	2.5 (8)
C(6)D	0.3424 (5)	-0.3566 (8)	0.0903 (3)	2.3 (4)
N(6)D	0.4345 (4)	-0.3189 (7)	0.0830 (3)	2.8 (16)
N(7)D	0.2628 (4)	-0.0790 (7)	0.0953 (3)	2.8 (17)
C(8)D	0.1689 (5)	-0.0495 (8)	0.1000 (3)	2.9 (13)
N(9)D	0.1138 (4)	-0.1825 (7)	0.1054 (3)	2.6 (9)
C(10)D	0.0076 (5)	-0.1904 (9)	0.1100 (3)	3.2 (17)
C(11)D	-0.0637 (5)	-0.199 (1)	0.0467 (4)	4.2 (23)
O(12)D	-0.1643 (4)	-0.2245 (6)	0.0508 (3)	4.7 (27)
N(1)E	0.5375 (4)	-0.0147 (7)	0.0777 (3)	2.9 (22)
C(2)E	0.6298 (5)	-0.0571 (9)	0.0730 (4)	3.5 (16)
N(3)E	0.7081 (4)	0.0361 (7)	0.0676 (3)	3.2 (20)
C(4)E	0.6824 (5)	0.1914 (9)	0.0685 (3)	2.7 (7)
C(5)E	0.5896 (5)	0.2533 (8)	0.0737 (3)	2.4 (11)
C(6)E	0.5128 (5)	0.1433 (8)	0.0769 (3)	2.5 (9)
N(6)E	0.4182 (4)	0.1840 (7)	0.0793 (3)	3.2 (21)
N(7)E	0.5912 (4)	0.4220 (7)	0.0711 (3)	3.0 (11)
C(8)E	0.6852 (5)	0.4588 (9)	0.0655 (3)	3.2 (12)
N(9)E	0.7417 (4)	0.3217 (7)	0.0628 (3)	2.8 (14)
C(10)E	0.8456 (5)	0.3168 (9)	0.0547 (3)	2.9 (16)
C(11)E	0.9222 (5)	0.289 (1)	0.1145 (3)	3.5 (16)
O(12)E	1.0206 (3)	0.2678 (6)	0.1028 (2)	3.8 (25)
N(1)F	0.1142 (4)	0.2677 (7)	0.2659 (3)	3.8 (24)
C(2)F	0.2131 (6)	0.309 (1)	0.2749 (4)	4.7 (29)
N(3)F	0.2971 (4)	0.2210 (7)	0.2769 (3)	3.9 (32)
C(4)F	0.2664 (5)	0.0647 (9)	0.2700 (3)	3.1 (10)
C(5)F	0.1680 (5)	0.0022 (8)	0.2602 (3)	2.3 (13)
C(6)F	0.0876 (5)	0.1083 (8)	0.2589 (3)	2.7 (9)

Table 3 (cont.)

	x	y	z	$B(\text{\AA}^2)$
N(6)F	-0.0101 (4)	0.0722 (7)	0.2504 (3)	3.1 (16)
N(7)F	0.1699 (4)	-0.1707 (7)	0.2552 (3)	3.0 (12)
C(8)F	0.2693 (5)	-0.2020 (9)	0.2616 (3)	3.2 (13)
N(9)F	0.3321 (4)	-0.0662 (7)	0.2705 (3)	3.1 (13)
C(10)F	0.4451 (5)	-0.0622 (9)	0.2814 (3)	3.1 (21)
C(11)F	0.4711 (5)	-0.028 (1)	0.2230 (4)	4.8 (30)
O(12)F	0.5800 (4)	-0.0121 (6)	0.2377 (3)	4.5 (22)

Table 4. Bond distances (\AA)

	A	B	C	D	E	F
N(1)—C(2)	1.31 (1)	1.342 (6)	1.332 (6)	1.32 (1)	1.328 (9)	1.35 (1)
N(1)—C(6)	1.37 (1)	1.339 (5)	1.343 (5)	1.338 (8)	1.351 (8)	1.368 (9)
C(2)—N(3)	1.34 (1)	1.330 (6)	1.335 (6)	1.35 (1)	1.34 (1)	1.34 (1)
N(3)—C(4)	1.36 (1)	1.348 (5)	1.337 (5)	1.367 (9)	1.336 (9)	1.36 (1)
C(4)—C(5)	1.40 (1)	1.381 (5)	1.388 (5)	1.37 (1)	1.39 (1)	1.39 (1)
C(4)—N(9)	1.39 (1)	1.375 (5)	1.372 (5)	1.359 (8)	1.371 (9)	1.401 (9)
C(5)—C(6)	1.38 (1)	1.407 (5)	1.408 (5)	1.41 (1)	1.40 (1)	1.39 (1)
C(5)—N(7)	1.41 (1)	1.393 (5)	1.394 (5)	1.398 (8)	1.400 (8)	1.439 (8)
C(6)—N(6)	1.33 (1)	1.338 (5)	1.335 (5)	1.336 (8)	1.339 (9)	1.321 (9)
N(7)—C(8)	1.33 (1)	1.324 (5)	1.309 (5)	1.326 (9)	1.346 (9)	1.341 (9)
C(8)—N(9)	1.38 (1)	1.356 (5)	1.366 (5)	1.355 (9)	1.380 (9)	1.393 (9)
N(9)—C(10)	1.46 (1)	1.474 (5)	1.468 (5)	1.470 (9)	1.466 (9)	1.487 (9)
C(10)—C(11)	1.49 (1)	1.509 (6)	1.509 (6)	1.51 (1)	1.49 (1)	1.48 (1)
C(11)—O(12)	1.42 (1)	1.421 (6)	1.431 (5)	1.41 (1)	1.436 (9)	1.43 (1)

Fig. 2. A view of molecule B , with the atom-numbering system. Thermal ellipsoids are drawn at the 50% probability level.Fig. 3. The molecular sheet in crystal form (II). The sheet is nearly parallel to the ab plane. Hydrogen bonds are indicated by thin lines, attached values (\AA) being distances between the donor and acceptor atoms.

Structural relationship among the three crystalline forms. The unit-cell dimensions of the three forms are characteristically correlated. The values of a and b are nearly the same in all three forms; the maximum variations are only 0.2 and 0.5% for a and b , respectively. On the other hand, the values of c are in the approximate ratio 1:2:3, the deviation from this integral ratio being about 1.2%.

The crystal structure analysis explains these correlations well. The crystals are composed of hydrogen-bonded molecular sheets which are stacked mainly by van der Waals forces. The structure of the sheet in form (II) is illustrated in Fig. 3. Adenine moieties are linked through N(6)H...N(1) and N(6)H...N(7) hydrogen bonds, to form ribbons along the b axis. The ribbons are connected side-by-side through O(12)H...N(3) hydrogen bonds. Thus, molecular layers nearly parallel to the ab plane are constructed. The same structural motif is observed in forms (I) and (III), which is indistinguishable from Fig. 3 except for the respective origin. In form (II), molecules B and C are related by a pseudo twofold screw axis (0.4058, y , -0.1246); the maximum atomic deviation from a true 2_1 axis is 0.03 Å along a , 0.04 Å along b and 0.12 Å along c in the adenine moiety. The layer in form (I) is composed of molecules A related to each other by the crystallographic 2_1 axis along b . Form (III) contains two independent layers: one consists of molecules D and E related by a pseudo 2_1 axis and the other of molecules F related by a true 2_1 axis.

The hydrogen-bond geometries in the three crystalline forms are very close to each other: the distances for N(6)H...N(1) are 2.868 (8)–2.900 (8) Å, for N(6)H...N(7) 3.017 (8)–3.115 (8) Å and for O(12)H...N(3) 2.807 (7)–2.852 (7) Å. The implication of hydrogen bonds in the present crystals for protein–nucleic acid interactions has been mentioned in a previous paper (Takenaka, Shibata & Sasada, 1984).

Fig. 4 shows the crystal structures of the three forms projected along the b axes, and clearly indicates the

stacking features of the molecular sheets described above. In form (I), any successive layers are related by the crystallographic twofold screw axis along a or c . The molecular superposition of the two layers is illustrated in Fig. 5(a). Such a stacking mode is denoted as p . Since the sheet is polar along b , the mode p is antiparallel stacking with respect to the polarity. As seen from Fig. 4, this stacking mode p is found in forms (II) and (III), although the crystallographic symmetry elements are lacking in the latter form. Form (II) contains another stacking mode q , in which the successive layers are related by the crystallographic 2_1 axis along b , so that it is parallel stacking with respect to the sheet polarity. The molecular superposition diagram for the stacking q is given in Fig. 5(d). Form (III) has, in addition to mode p , a third mode r in which the successive layers are related by the centre of symmetry so that it is antiparallel stacking (Fig. 5e). Thus, the layer structures of these three forms are represented, in terms of stacking mode, as pp for form (I), $pqqq$ for (II) and $pprrppr$ for (III).

The complementarity in stacking of mode p seems to be less favourable than those in modes q and r , as seen from Fig. 4. This is compatible with the slightly smaller density of the crystals of form (I) compared with those of (II) and (III). A semi-quantitative analysis was carried out by calculating the energy function between successive layers with Giglio's (1969) parameters. Electrostatic terms were omitted because it is inadequate to include them in such local interactions. Dispersion and repulsion terms for all atom pairs with interatomic distances less than 60 Å were summed. The values for stacking p are -74.8, -76.9 and -76.5 kJ mol⁻¹ for forms (I), (II) and (III), respectively. The energy for q is -78.2 kJ mol⁻¹, while that for r -79.4 kJ mol⁻¹. This estimation supports the above-mentioned argument, but the energy difference among the three stacking modes is very small.

The conformation of the hydroxyethyl group reveals significant variations, as listed in Table 5. Molecules A

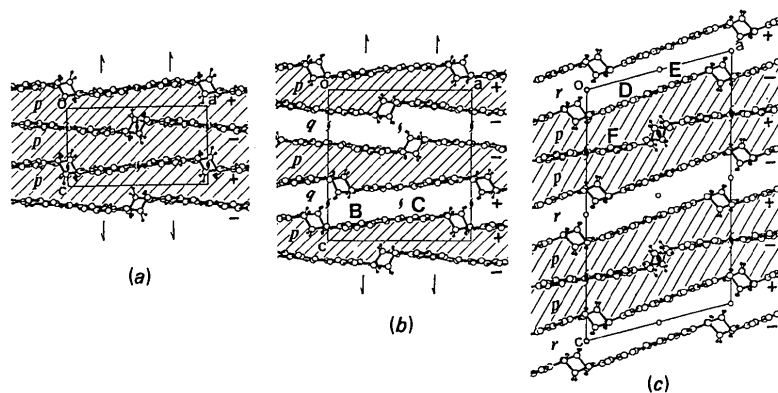


Fig. 4. Crystal structures viewed along the b axis. Stacking in mode p is hatched. + and - represent the sheet polarity. (a) Form (I). (b) Form (II). (c) Form (III).

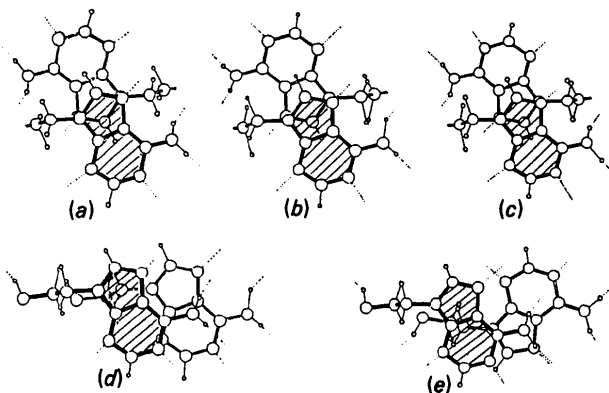


Fig. 5. Superposition of the molecules between successive layers. (a), (b) and (c) show stacking mode *p* in forms (I), (II) and (III), respectively, (d) shows *q* in form (II), and (e) shows *r* in form (III).

in form (I) and *F* in form (III) take the same stacking mode *p* at both sides of the molecular plane. On the other hand, the layer consisting of molecules *B* and *C* in form (II) and that containing *D* and *E* in form (III) have different modes at both sides of the layer. Component molecules in the former case show the single torsion angle τ_1 of about 96° , while in the independent molecules in the latter case there are observed two torsion angles of about 88 and 102° . Perhaps such subtle adjustment in the torsion angle improves the packing efficiency between the layers.

Disorder and twinning. Since the energy difference among the stacking modes is quite small, as indicated above, disordering could also occur by random change of the modes. In fact, appreciable streaks along c^* in Weissenberg photographs were found for crystals of forms (I) and (III).

A similar interpretation is applicable to the polysynthetic twin of form (III). Examination of Weissenberg photographs of the twinned crystals indicates that $a' = -a$, $b' = -b$ and that the c^* axes are common for the twin components. From the crystal structures shown in Figs. 4 and 5, it is most plausible that the

Table 5. Torsion angles ($^\circ$) for the hydroxyethyl group

Form	Stacking	Molecule	τ_1	τ_2
Form (I)	<i>p</i>	<i>A</i>	94.1 (1)	174.6 (6)
Form (III)	<i>p</i>	<i>F</i>	98.1 (8)	175.3 (5)
Form (II)	<i>q</i> and <i>p</i>	<i>B</i>	88.1 (4)	171.9 (3)
		<i>C</i>	102.7 (4)	173.5 (3)
Form (III)	<i>r</i> and <i>p</i>	<i>D</i>	88.7 (8)	173.6 (6)
		<i>E</i>	100.8 (8)	173.8 (5)

τ_1 : C(8)—N(9)—C(10)—C(11), τ_2 : N(9)—C(10)—C(11)—O(12).

stacking mode *r* or *p* in the twinning plane is replaced by mode *q*.

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Structure of a 9-(2-Carboxyethyl)guanine 1,6-Hexanediamine (2:1) Complex

BY SHOJI OUCHI, AKIO TAKENAKA AND YOSHIO SASADA

Faculty of Science, Tokyo Institute of Technology, Nagatsuta, Midori-ku, Yokohama 227, Japan

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Abstract. 1,6-Hexanediammonium bis[3-(2-amino-1,6-dihydro-6-oxo-9-purinyloxy)propionate] tetrahydrate, $2C_8H_8N_5O_3 \cdot C_6H_{18}N_2^+ \cdot 4H_2O$, $M_r = 634.65$, triclinic,

0108-2701/86/101340-04\$01.50

$P\bar{1}$, $a = 11.169$ (2), $b = 13.885$ (2), $c = 11.004$ (2) Å, $\alpha = 108.44$ (1), $\beta = 102.77$ (1), $\gamma = 97.67$ (1)°, $V = 1539.6$ (6) Å³, $Z = 2$, $D_m = 1.377$, $D_x = 1.369$ g cm⁻³,

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