Table 2. *Comparison of bond lengths in amide groups*

Substance	Distance (Å) C–N	Distance (Å) C=O
Sulfazecin[a]	1·374 (7)	1·210 (7)
Unstrained β-lactam[b]	1·367 (4)	1·210 (4)
Penicillins[c]	1·392 (4)	1·194 (4)
Cephalosporins[d]	1·38 (1)	1·21 (1)
Δ^2-Cephalosporins[e]	1·339 (7)	1·223 (7)
	1·36	1·20
	1·334	1·228
Free amide[f]	$1{\cdot}32_5$	1·24

References: (*a*) Present work; (*b*) Parthasarathy (1970); (*c*) Domiano, Nardelli, Balsamo, Macchia & Macchia (1979); (*d*) Sweet (1972); (*e*) Sweet & Dahl (1970); Kobelt & Paulus (1974*a*); Kobelt & Paulus (1974*b*); (*f*) Marsh & Donohue (1967).

demonstrates that Sweet & Dahl's concept is also applicable to the present antibiotic.

The crystal packing involves seven independent intermolecular hydrogen bonds, five of which are between sulfazecin molecules with the remaining two between sulfazecin and the methanol of crystallization. The amino N(23) donates three hydrogens to the carbonyl O(14), and the sulfo O(6) and O(7) of the symmetry-related molecules. Two amide nitrogens N(12) and N(17) donate hydrogens to the carbonyl O(19) and O(9) atoms, respectively. As for the oxygens of the carbonyl group, O(25) accepts a hydrogen from the hydroxyl group of methanol, and O(26) donates its hydrogen to the hydroxyl oxygen of the methanol. The sulfo O(8) does not participate in hydrogen bonding. These hydrogen bonds connect molecules of sulfazecin and methanol of crystallization with each other and form a three-dimensional network in the crystal.

The authors are grateful to Dr E. Ohmura for his encouragement throughout this work. We also thank Drs M. Nishikawa and T. Kishi for useful suggestions.

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Acta Cryst. (1981). **B37**, 1628–1631

The Structure of 6-(6-Aminohexanamido)hexanoic Acid Hydrate*

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(*Received* 8 *April* 1980; *accepted* 28 *May* 1980)

Abstract. $C_{12}H_{24}N_2O_3.H_2O$, $M_r = 262{\cdot}3$, monoclinic, $P2_1/c$, $a = 17{\cdot}649\,(2)$, $b = 4{\cdot}915\,(1)$, $c = 17{\cdot}516\,(2)$ Å, $\beta = 107{\cdot}26\,(1)^\circ$, $V = 1451{\cdot}1\,(3)$ Å^3, $D_c = 1{\cdot}200$ Mg m^{-3}, for $Z = 4$, $\mu(\text{Cu}\,K\alpha) = 0{\cdot}741$ mm^{-1}. The structure was solved by the direct method and refined anisotropically by the block-diagonal least-squares procedure; $R = 0{\cdot}051$ for 1710 non-zero reflections. The molecules are present in the crystal as zwitterions. Each zwitterion forms four N–H···O bonds with adjacent molecules to form a three-dimensional network. The water of crystallization also engages in the N–H···O and O–H···O hydrogen

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* Work supported in part by a Grant-in-Aid (No. 434024), Ministry of Education, Science and Culture, Japan, and the earlier work supported in part by the US Atomic Energy Commission Grant AT(30-1)-1715.

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bonds, which reinforce the hydrogen-bond network in the crystal. The hydrogen-bond lengths are: NH···O 2·694 (2), 2·775 (2), and 2·902 (3) and OH···O 2·634 (2) and 2·923 (3) Å.

Introduction. Bodor, Bednowitz & Post (1967) and Takenaka, Yamasaki, Yamamoto, Furusaki & Nitta (1966) have determined the crystal structure of ε-aminocaproic acid, $NH_2(CH_2)_5COOH$, which undergoes polycondensation in the solid state to form Nylon 6. Bodor *et al.* related some structural features of Nylon 6 to the crystal structure of ε-aminocaproic acid. 6-(6-Aminohexanamido)hexanoic acid (condensed linear dimer of ε-aminocaproic acid), $NH_2(CH_2)_5$-$CONH(CH_2)_5COOH$, which has the same functional groups as ε-aminocaproic acid, has a long induction period in polycondensation in the solid state (Macchi, Morosoff & Morawetz, 1968). To elucidate the structural difference between these two related compounds the crystal structure analysis of the present compound has been carried out.

The crystals were supplied by Mr E. Macchi at the Polytechnic Institute of Brooklyn in 1964. Using intensity data collected by multiple-film Weissenberg photographs or by the stationary-crystal stationary-counter method, efforts were made in 1965 and 1966 to solve the structure by the Patterson function and related techniques and also by symbolic addition (Karle & Karle, 1963), without success.

A crystal, which had been kept for 15 years, with approximate dimensions 0·38 × 0·40 × 0·08 mm, was used in the present study. The unit-cell dimensions were determined by a least-squares fit using 2θ values of 25 strong reflections measured on a Rigaku automated four-circle diffractometer. Nickel-filtered Cu $K\alpha$ radiation was used.

Integrated intensities were measured by the θ–2θ scan technique on the same diffractometer. The scan rate and scan width were 4° min^{-1} and $\Delta(2\theta) = (2{\cdot}0 + 0{\cdot}3 \tan \theta)°$, respectively. Backgrounds were counted for 7·5 s at both ends of a scan. A total of 1833 (1712 non-zero) independent reflections were measured up to $2\theta = 110°$. The usual Lorentz and polarization corrections were applied to the intensity data, but absorption corrections were ignored.

The structure was solved by direct methods (*MULTAN* 74, Main, Woolfson, Lessinger, Germain & Declercq, 1974). The non-hydrogen atoms were located on an E map. The structure was refined anisotropically by the block-diagonal least-squares procedure (*HBLS* V, Ashida, 1973) minimizing $\sum w(\Delta F)^2$. The H atoms were located by a difference synthesis. The water of crystallization was found and included in the subsequent refinement. At the final stage the 004 and 104 reflections, which were considered to be affected by extinction, were excluded from the refinement. The final R value is 0·051 for 1710 non-zero (0·055 for all) reflections ($R_w = 0{\cdot}064$). The weighting scheme used was $w = [\sigma_{cs}(F) + a|F_o| + b|F_o|^2]^{-1}$ for $|F_o| > 0$ and $w = c$ for $|F_o| = 0$, where σ_{cs} is the standard deviation obtained from counting statistics. Values of a, b and c used in the final refinement are −0·1060, 0·0071 and 1·5386, respectively. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are listed in Table 1.*

Table 1. *Fractional atomic coordinates with e.s.d.'s in parentheses*

	x	y	z	B_{eq}/B_{iso} (Å^2)
O(1)	0·0764 (1)	0·8322 (4)	0·14176 (9)	5·7
O(2)	0·1047 (1)	0·5313 (4)	0·06217 (9)	5·1
O(3)	0·5310 (1)	1·1698 (3)	0·1161 (2)	5·4
O(*W*)	0·0449 (1)	0·9386 (4)	0·27658 (9)	4·7
N(1)	0·4945 (1)	0·7407 (4)	0·1288 (2)	3·8
N(2)	0·94880 (9)	1·1709 (4)	0·0722 (1)	3·3
C(1)	0·1134 (2)	0·6273 (5)	0·1295 (2)	3·1
C(2)	0·1715 (2)	0·4931 (5)	0·2006 (2)	3·7
C(3)	0·2491 (2)	0·4107 (5)	0·1854 (2)	3·6
C(4)	0·2998 (2)	0·6539 (5)	0·1782 (2)	3·4
C(5)	0·3748 (2)	0·5748 (5)	0·1573 (2)	3·7
C(6)	0·4260 (2)	0·8173 (5)	0·1545 (2)	3·9
C(7)	0·5413 (2)	0·9231 (5)	0·1105 (2)	3·6
C(8)	0·6084 (2)	0·8102 (5)	0·0825 (2)	4·0
C(9)	0·6695 (2)	1·0231 (5)	0·0784 (2)	3·6
C(10)	0·7432 (2)	0·8972 (5)	0·0644 (2)	3·8
C(11)	0·8067 (2)	1·1070 (5)	0·0638 (2)	3·4
C(12)	0·8839 (2)	0·9706 (5)	0·0640 (2)	3·7
H(1)	0·151 (2)	0·355 (5)	0·219 (2)	3·9 (6)
H(2)	0·185 (2)	0·631 (5)	0·248 (2)	2·8 (6)
H(3)	0·233 (2)	0·294 (5)	0·131 (2)	2·3 (5)
H(4)	0·278 (2)	0·286 (5)	0·234 (2)	3·8 (6)
H(5)	0·266 (2)	0·783 (4)	0·132 (2)	1·9 (5)
H(6)	0·317 (2)	0·763 (5)	0·233 (2)	3·4 (6)
H(7)	0·359 (2)	0·488 (4)	0·111 (2)	2·0 (5)
H(8)	0·408 (2)	0·436 (5)	0·201 (2)	3·2 (6)
H(9)	0·393 (2)	0·948 (5)	0·118 (2)	3·8 (6)
H(10)	0·442 (2)	0·902 (5)	0·215 (2)	3·9 (6)
H(11)	0·506 (2)	0·569 (5)	0·132 (2)	3·0 (6)
H(12)	0·583 (2)	0·738 (5)	0·023 (2)	3·4 (6)
H(13)	0·636 (2)	0·657 (5)	0·121 (2)	2·7 (5)
H(14)	0·643 (2)	1·161 (5)	0·036 (2)	3·8 (6)
H(15)	0·687 (2)	1·123 (4)	0·135 (2)	1·6 (5)
H(16)	0·733 (2)	0·805 (5)	0·011 (2)	2·8 (6)
H(17)	0·771 (2)	0·755 (5)	0·115 (2)	3·9 (6)
H(18)	0·786 (2)	1·218 (5)	0·015 (2)	2·5 (6)
H(19)	0·816 (2)	1·234 (4)	0·113 (2)	1·7 (5)
H(20)	0·879 (2)	0·888 (5)	0·012 (2)	2·7 (5)
H(21)	0·904 (2)	0·851 (5)	0·117 (2)	4·3 (7)
H(22)	0·939 (2)	1·252 (4)	0·032 (2)	1·4 (5)
H(23)	0·996 (2)	1·089 (5)	0·084 (2)	3·7 (6)
H(24)	0·953 (2)	1·289 (5)	0·122 (2)	2·7 (6)
H(*W*1)	0·065 (2)	0·910 (5)	0·232 (2)	3·9 (6)
H(*W*2)	0·023 (2)	1·077 (5)	0·265 (2)	3·5 (6)

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* Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35393 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. Fig. 1 is a schematic drawing of the molecule (Johnson, 1976) with the atom numbering. Similarly to ε-aminocaproic acid (Bodor *et al.*, 1967), the molecules are present in the crystal as zwitterions.

Bond lengths and angles are listed in Table 2. The $C(sp^3)$–$C(sp^3)$ bond lengths range from 1·505 (3) to 1·525 (3) Å, and the average value (1·520 Å) is the same as that in ε-aminocaproic acid. The two $C(sp^3)$–$C(sp^2)$ bonds are 1·511 (3) and 1·516 (3) (av. 1·514 Å). The average C–H, N–H, and O–H lengths are 1·03, 0·90, and 0·87 Å, respectively. Bond angles in the molecule are normal; the O–C–O angle is 123·2 (2)°, and the average C–C–C, C–C–H, C–N–H, N–C–H, H–C–H, and H–N–H angles are 112·7, 109, 109, 108, 110, and 110°, respectively.

The dihedral angle between O(1),O(2),C(1) and C(1),C(2),C(3) is 43·4 (2)°. As shown in Table 3, only the conformation around the C(2)–C(3) bond, which is next to the carboxyl end, is *gauche* (τ = 69·7°). The conformations around the other C–C and C–N bonds are all *trans*, as are those in the stable form of Nylon 6.

The crystal structure is shown in Fig. 2. Four slightly bent molecules are packed in an antiparallel way in a shallow unit cell. Each molecule lies approximately parallel to the *a* axis and is bonded to two adjacent

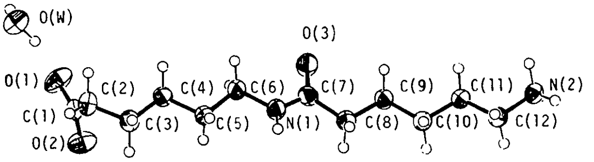

Fig. 1. Molecular structure and numbering scheme. Non-hydrogen atoms are shown as thermal ellipsoids at the 50% probability level.

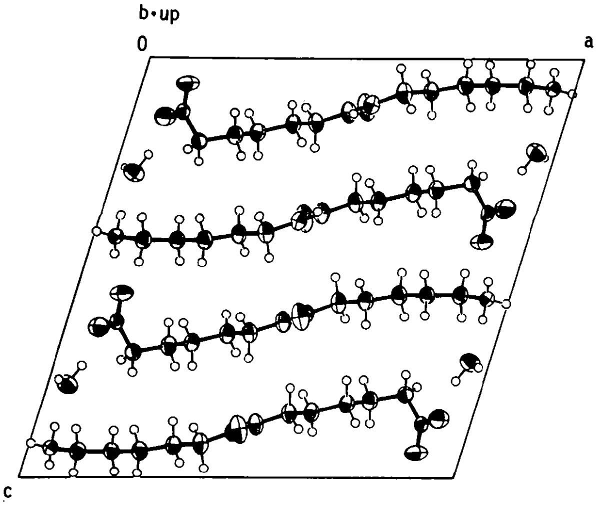

Fig. 2. Crystal structure projected along the *b* axis.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(2)–C(3)	1·524 (3)	C(6)–N(1)	1·459 (3)
C(3)–C(4)	1·521 (3)	C(13)–N(2)	1·485 (3)
C(4)–C(5)	1·523 (3)	(Average	1·472)
C(5)–C(6)	1·505 (3)		
C(8)–C(9)	1·519 (3)	C(7)–N(1)	1·321 (3)
C(9)–C(10)	1·524 (3)		
C(10)–C(11)	1·525 (3)	C(1)–O(1)	1·253 (3)
C(11)–C(12)	1·519 (3)	C(1)–O(2)	1·236 (3)
(Average	1·520)	(Average	1·245)
C(1)–C(2)	1·511 (3)	C(7)–O(3)	1·234 (3)
C(17)–C(8)	1·516 (3)		
(Average	1·514)		
C(2)–H(1)	0·88 (3)	N(1)–H(1)	0·87 (3)
C(2)–H(2)	1·04 (2)	N(2)–H(22)	0·79 (2)
C(3)–H(3)	1·07 (2)	N(2)–H(23)	0·90 (3)
C(3)–H(4)	1·06 (3)	N(2)–H(24)	1·03 (3)
C(4)–H(5)	1·06 (2)	(Average	0·90)
C(4)–H(6)	1·07 (3)		
C(5)–H(7)	0·88 (2)	O(*W*)–H(*W*1)	0·96 (3)
C(5)–H(8)	1·06 (3)	O(*W*)–H(*W*2)	0·78 (3)
C(6)–H(9)	0·98 (3)	(Average	0·87)
C(6)–H(10)	1·09 (3)		
C(8)–H(12)	1·07 (3)		
C(8)–H(13)	1·03 (3)		
C(9)–H(14)	1·01 (3)		
C(9)–H(15)	1·06 (2)		
C(10)–H(16)	1·01 (3)		
C(10)–H(17)	1·12 (3)		
C(11)–H(18)	0·99 (3)		
C(11)–H(19)	1·03 (2)		
C(12)–H(20)	0·97 (3)		
C(12)–H(21)	1·07 (3)		
(Average	1·03)		
O(1)–C(1)–O(2)	123·2 (2)	C(1)–C(2)–C(3)	113·4 (2)
		C(2)–C(3)–C(4)	112·8 (2)
C(1)–C(2)–H(1)	112·6 (17)	C(3)–C(4)–C(5)	113·2 (2)
C(1)–C(2)–H(2)	107·9 (12)	C(4)–C(5)–C(6)	112·3 (2)
C(2)–C(3)–H(3)	106·0 (11)	C(7)–C(8)–C(9)	113·4 (2)
C(2)–C(3)–H(4)	104·9 (13)	C(8)–C(9)–C(10)	112·3 (2)
C(3)–C(4)–H(5)	109·0 (11)	C(9)–C(10)–C(11)	113·0 (2)
C(3)–C(4)–H(6)	109·6 (13)	C(10)–C(11)–C(12)	111·3 (2)
C(4)–C(5)–H(7)	106·3 (13)	(Average	112·7)
C(4)–C(5)–H(8)	108·6 (12)		
C(5)–C(6)–H(9)	107·0 (15)	N(1)–C(6)–H(9)	111·0 (15)
C(5)–C(6)–H(10)	104·7 (13)	N(1)–C(6)–H(10)	113·7 (13)
C(7)–C(8)–H(12)	107·1 (13)	N(2)–C(12)–H(20)	103·1 (14)
C(7)–C(8)–H(13)	108·5 (12)	N(2)–C(12)–H(21)	103·9 (13)
C(8)–C(9)–H(14)	108·8 (13)	(Average	108)
C(8)–C(9)–H(15)	107·1 (11)		
C(9)–C(10)–H(16)	114·4 (13)	H(1)–C(2)–H(2)	103·5 (20)
C(9)–C(10)–H(17)	108·3 (12)	H(3)–C(3)–H(4)	110·5 (17)
C(10)–C(11)–H(18)	107·0 (12)	H(5)–C(4)–H(6)	109·4 (17)
C(10)–C(11)–H(19)	110·1 (11)	H(7)–C(5)–H(8)	108·0 (18)
C(11)–C(12)–H(20)	110·3 (14)	H(9)–C(6)–H(10)	108·6 (19)
C(11)–C(12)–H(21)	107·7 (13)	H(12)–C(8)–H(13)	112·0 (17)
C(3)–C(2)–H(1)	110·6 (17)	H(14)–C(9)–H(15)	108·1 (17)
C(3)–C(2)–H(2)	108·1 (12)	H(16)–C(10)–H(17)	111·7 (17)
C(4)–C(3)–H(3)	110·9 (11)	H(18)–C(11)–H(19)	108·2 (17)
C(4)–C(3)–H(4)	*111·5 (13)*	*H(20)–C(12)–H(21)*	*119·8 (19)*
C(5)–C(4)–H(5)	107·4 (11)	(Average	110)
C(5)–C(4)–H(6)	108·2 (13)		
C(6)–C(5)–H(7)	112·6 (13)	H(22)–N(2)–H(23)	111·2 (20)
C(6)–C(5)–H(8)	109·1 (12)	H(23)–N(2)–H(24)	103·3 (19)
C(9)–C(8)–H(12)	106·3 (13)	H(22)–N(2)–H(24)	114·9 (19)
C(9)–C(8)–H(13)	109·6 (12)	(Average	110)
C(10)–C(9)–H(14)	112·6 (13)		
C(10)–C(9)–H(15)	107·7 (10)	C(6)–N(1)–H(11)	115·6 (14)
C(11)–C(10)–H(16)	102·9 (13)	C(8)–N(1)–H(11)	121·6 (14)
C(11)–C(10)–H(17)	106·4 (12)	(Average	119)
C(12)–C(11)–H(18)	110·3 (12)		
C(12) C(11) H(19)	109·0 (11)	C(12)–N(2)–H(22)	107·2 (14)
(Average	108·6)	C(12)–N(2)–H(23)	111·6 (15)
		C(12)–N(2)–H(24)	108·7 (13)
		(Average	109)

Table 3. *Internal rotation angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)–C(3)–C(4)	69·7 (3)	O(1)–C(1)–C(2)–C(3)	−137·5 (3)	C(6)–N(1)–C(7)–C(8)	−178·3 (2)
C(2)–C(3)–C(4)–C(5)	−176·1 (2)	O(2)–C(1)–C(3)–C(3)	42·2 (3)	N(1)–C(7)–C(8)–C(9)	−168·4 (2)
C(3)–C(4)–C(5)–C(6)	−177·2 (2)			C(7)–C(8)–C(9)–C(10)	169·9 (2)
C(4)–C(5)–C(6)–C(7)	−175·8 (2)	C(6)–N(1)–C(7)–O(3)	1·9 (4)	C(8)–C(9)–C(10)–C(11)	−175·5 (2)
C(5)–C(6)–N(1)–C(7)	169·8 (2)	O(3)–C(7)–C(8)–C(9)	11·4 (4)	C(9)–C(10)–C(11)–C(12)	170·4 (2)
				C(10)–C(11)–C(12)–N(2)	−173·8 (2)

molecules at both ends by N(2)H(23)···O(1) [2·775 (2) Å] hydrogen bonds to form an infinite chain of molecules along the *a* axis. Each molecule is also connected, by hydrogen bonds between amido groups at the middle part of the molecule {N(1)H(11)···O(3) [2·902 (3) Å]}, to the molecules above and below along the *b* axis. A wavy sheet of parallel molecules is thus formed. These sheets stack parallel to one another along the *c* axis, and are on one side bonded by an N(2)H(22)···O(2) [2·694 (2) Å] hydrogen bond and on the other by two hydrogen bonds N(2)-H(24)···O(*W*) [2·930 (2) Å] and O(*W*)H(*W*1)···O(1) [2·634 (2) Å] *via* a water molecule. The water molecules of crystallization themselves are connected by a weak O(*W*)H(*W*2)···O(*W*) [2·923 (3) Å] hydrogen bond along the *b* axis.

Under the combined influences of heat and high vacuum, the existence of crystalline water probably increases the vapor pressure in the reaction vessel appreciably, which controls the vaporization of water molecules released by the polycondensation and affects the solid-state conversion of the present dimer molecule to Nylon 6. This is consistent with the fact that the solid-state polycondensation of the ε-aminocaproic acid hardly takes place if the crystal is sealed in a small ampoule.

Computations throughout the study were carried out on an ACOS series 77 NEAC System 700 computer at the Crystallographic Research Center, Institute for Protein Research, Osaka University.

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Acta Cryst. (1981). B**37**, 1631–1634

Tetra-*tert*-butylcyclotetraphosphane, $[PC(CH_3)_3]_4$

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(*Received* 23 *December* 1980; *accepted* 4 *March* 1981)

Abstract. $C_{16}H_{36}P_4$, monoclinic, $P2_1/c$, $a = 9{\cdot}391$ (1), $b = 14{\cdot}029$ (2), $c = 16{\cdot}854$ (2) Å, $\beta = 95{\cdot}80$ (1)°, $V = 2196$ Å^3, $Z = 4$, D_o (by flotation in $ZnCl_2$ solution) = 1·08, D_c = 1·07 Mg m^{-3}. The central P_4 ring is non-planar with P–P distances of 2·212 (2) Å and P–P–P–P torsion angles of 24·5°. The *tert*-butyl groups are on alternate sides of the ring such that the molecule very nearly possesses $\bar{4}2m$ symmetry. The final R was 0·037 for the 1981 observed reflections.

Introduction. The crystal structures of four cyclopolyphosphane molecules have been reported: the tetramer $(PCF_3)_4$ (Palenik & Donohue, 1962), pentamers $(PCF_3)_5$ (Spencer & Lipscomb, 1961, 1962) and $(PPh)_5$ (Daly, 1964) and the hexamer $(PPh)_6$ (Daly, 1965, 1966). The present paper reports the structure of $[PC(CH_3)_3]_4$ which was determined in order to study the effects of the bulky *tert*-butyl groups on the phosphorus ring conformation and, through

0567-7408/81/081631-04$01.00