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The Structure of Amoxycillin Trihydrate and a Comparison with the Structures of Ampicillin

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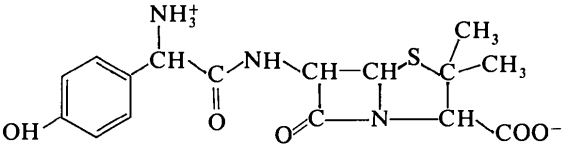
The structure of amoxycillin trihydrate with formula $C_{16}H_{19}N_3O_5S.3H_2O$ has been determined.



It crystallizes in the orthorhombic system with space group $P2_12_12_1$ and four molecules in a unit cell of dimensions a = 15·622 (17), b = 18·785 (14) and c = 6·645 (9) Å. The structure was solved by multi-solution tangent formula refinement from intensity measurements obtained by microdensitometer from equi-inclination Weissenberg films. Least-squares refinement yielded an R value of 0·061 for observed data. The molecular configuration is almost identical to that of ampicillin trihydrate and the crystal packings of these two compounds are very similar with the addition in amoxycillin of H bonding between the aromatic OH and O(12) of a symmetry-related molecule. The structures of amoxycillin and ampicillin are compared in detail and the inability of amoxycillin to crystallize in forms similar to ampicillin anhydrate and delta-form ampicillin is discussed.

Introduction

Amoxycillin [D(−)-α-amino-*p*-hydroxybenzylpenicillin] is a semisynthetic penicillin in common use and is effective as an antibiotic against a wide variety of Gram-positive and Gram-negative organisms. Amoxycillin was first described by Long, Nayler, Smith, Taylor & Ward (1971) and its antibiotic properties have been extensively studied and compared with the structurally related compound ampicillin (Sutherland & Rolinson, 1970; Acred, Hunter, Mizen & Rolinson, 1971; Sutherland, Croydon & Rolinson, 1972; Hunter, Rolinson & Witting, 1973). The minimum inhibitory concentrations of amoxycillin and ampicillin are very similar but the bactericidal effect of amoxycillin is more rapid and more complete than is obtained with the same dose of ampicillin. The superior activity of amoxycillin against experimental infections is apparent when the antibiotic is administered either orally or subcutaneously and is not simply related to the higher blood levels of amoxycillin compared with ampicillin after oral administration.

Considerable attention has been devoted to the structural properties of ampicillin (James, Hall & Hodgkin, 1968; Boles & Girven, 1976*a*). The present report describes the authors' investigation of the crystallographic structure of amoxycillin trihydrate and compares the structure with the known structures of ampicillin.

Experimental

Amoxycillin trihydrate was obtained from Beecham Pharmaceuticals Ltd. A solution was made by adding 2·0 g of starting material to 20 ml 0·25 *N* HCl and warming to 30–40 °C. The solution was then cooled to 15 °C and diluted with 20 ml water and 30 ml ethanol. Neutralization with 5 ml 1·0 *N* NaOH resulted in the commencement of crystallization in about 5 min. Colourless needles of amoxycillin trihydrate were isolated after 4 h.

The unit-cell dimensions were determined from zero-level equi-inclination Weissenberg photographs; the camera radius was determined from high-angle reflexions from an annealed gold wire and the cell dimensions were refined by a least-squares method. The unit-cell dimensions are a = 15·622 (17), b = 18·785 (14), c = 6·645 (9) Å ($\alpha = \beta = \gamma = 90°$), where the standard deviations reflect the internal consistency of the measured 2θ values of the 12 $hk0$ and 10 $0kl$ reflexions used in the refinement. The systematic absences $h00$, $h = 2n + 1$, $0k0$, $k = 2n + 1$, $00l$, $l = 2n + 1$ indicated space group $P2_12_12_1$. Four molecules per unit cell were assumed giving a calculated density of 1429 kg m^{-3}.

Data for intensity measurement were obtained by the equi-inclination method on a Stoe–Weissenberg camera using Ni-filtered Cu $K\alpha$ radiation (λ = 1·5418 Å) and the multiple-film technique. The crystal of size 0·7 × 0·02 × 0·02 mm was rotated about the c axis with the

long edge of the crystal parallel to the rotation axis. The intensities of the X-ray reflexions were obtained by the Science Research Council microdensitometer at the Atlas Computer Laboratory, Chilton. The intensities of 1352 reflexions were measured. The absorption coefficient, μ, was 1886 m^{-1}; no absorption correction was applied to the intensity measurements.

Structure determination and refinement

The major computations were carried out with the *SHELX* program (Sheldrick, 1975) on the ICL 1906A at the Atlas Computer Laboratory, Chilton. The structure was solved with multisolution tangent refinement (Germain & Woolfson, 1968; Germain, Main & Woolfson, 1971). The starting set (Table 1) was selected from the 256 reflexions with $E > 1{\cdot}20$ and was chosen such that the origin- and enantiomorph-defining reflexions conform to procedures described by Hauptman & Karle (1956) and Karle & Hauptman (1956). Three further reflexions were included in the multisolution starting set so that expansion of the set was uninterrupted. The 'best' eight E maps were generated in the order of increasing RA where $RA = [\Sigma(E_o - kE_c)^2/\Sigma(E_o)^2]^{1/2}$ where k is chosen to minimize RA (Germain, Main & Woolfson, 1970, 1971; Roberts, Petterson, Sheldrick, Isaacs & Kennard, 1973). The lowest RA of 0·058 giving the correct solution was significantly lower than the next lowest of 0·120.

Three cycles of unweighted full-matrix least-squares refinement with the 28 non-hydrogen atom positions obtained from the E map and including interlayer scale-factor refinement produced $R = 0{\cdot}104$, where $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. H atoms were located by an $F_o - F_c$ Fourier synthesis. The final blocked least-squares refinement used anisotropic thermal parameters for the non-hydrogen atoms and the H atom positions were inserted with isotropic $U = 0{\cdot}033$ Å^2 and only their positions were refined. Blocked refinement avoids the matrix singularity which would result

Table 1. *The starting set for phase determination*

	Reflexion $h\,k\,l$	E	Phase
Origin-fixing reflexions	8 5 0	2·10	0°
	11,15,0	1·99	90
	0,20,1	2·31	0
Enantiomorph-defining reflexion	5 5 1	2·29	45 or 135
	10,2,0	2·78	0 or 180
	2 9 0	1·83	0 or 180
	4 9 0	2·62	0 or 180

Table 2. *Final coordinates obtained from least-squares refinement*

Coordinates are given as fractions of cell edges $\times 10^4$. Standard deviations in parentheses are with respect to the last figures given.

	x	y	z
S(1)	3424 (1)	4649 (1)	2639 (3)
C(2)	2993 (4)	3748 (3)	2188 (11)
C(3)	3579 (4)	3244 (3)	3434 (9)
N(4)	3870 (3)	3669 (3)	5138 (9)
C(5)	3596 (4)	4431 (3)	5213 (12)
C(6)	4486 (4)	4598 (3)	6174 (10)
C(7)	4696 (4)	3830 (3)	5650 (11)
O(8)	5333 (3)	3482 (2)	5551 (8)
C(9)	2075 (5)	3722 (4)	2910 (13)
C(10)	3030 (6)	3596 (4)	−31 (13)
C(11)	3143 (4)	2542 (3)	4087 (11)
O(12)	3057 (3)	2416 (2)	5878 (7)
O(13)	2891 (3)	2155 (2)	2662 (7)
N(14)	5009 (4)	5113 (3)	5193 (8)
C(15)	5413 (5)	5639 (3)	6184 (10)
O(16)	5386 (4)	5727 (3)	8005 (8)
C(17)	5832 (4)	6181 (3)	4806 (10)
N(18)	6578 (4)	6496 (3)	5827 (8)
C(19)	5177 (4)	6742 (3)	4272 (10)
C(20)	4770 (4)	6702 (3)	2448 (11)
C(21)	4135 (5)	7173 (4)	1954 (11)
C(22)	3887 (4)	7675 (4)	3342 (11)
C(23)	4293 (5)	7742 (4)	5162 (11)
C(24)	4945 (5)	7270 (4)	5630 (11)
O(25)	7010 (3)	4043 (2)	6261 (9)
O(26)	7469 (4)	4640 (3)	2393 (11)
O(27)	5688 (4)	4817 (3)	1318 (9)
O(28)	3231 (3)	8142 (3)	2966 (8)

Table 3. *Coordinates and bond lengths of hydrogen atoms*

Coordinates are given as fractions of cell edges $\times 10^4$. The heavy atom associated with each hydrogen atom is also given. Standard deviations in parentheses are with respect to the last figures given.

	x	y	z	Bond length (Å) (H—heavy atom)
H(C3)	4084 (55)	3127 (43)	2748 (147)	0·955 (90)
H(C5)	2966 (57)	4568 (46)	6136 (132)	1·197 (89)
H(C6)	4381 (55)	4717 (44)	7870 (153)	1·162 (101)
H(C9)(1)	1896 (62)	3959 (51)	2339 (161)	0·640 (100)
H(C9)(2)	1728 (52)	3149 (46)	2547 (142)	1·219 (86)
H(C9)(3)	2064 (53)	3764 (46)	4093 (148)	0·810 (99)
H(C10)(1)	2771 (53)	3136 (49)	9552 (143)	0·995 (91)
H(C10)(2)	2646 (56)	4030 (48)	9104 (151)	1·168 (93)
H(C10)(3)	3551 (63)	3638 (49)	9426 (148)	0·880 (100)
H(N14)	5176 (58)	5125 (46)	3864 (141)	0·933 (94)
H(C17)	6043 (55)	5928 (44)	3414 (141)	1·075 (91)
H(N18)(1)	6778 (54)	6123 (47)	6260 (142)	0·817 (88)
H(N18)(2)	6309 (54)	6713 (46)	7177 (140)	1·073 (91)
H(N18)(3)	6763 (49)	6867 (45)	5061 (145)	0·911 (88)
H(C20)	4993 (54)	6265 (48)	1364 (129)	1·154 (88)
H(C21)	3857 (51)	7068 (47)	640 (138)	0·989 (90)
H(C23)	4058 (52)	8075 (46)	6190 (139)	1·004 (89)
H(C24)	5159 (52)	7219 (45)	7192 (143)	1·106 (94)
H(O25)(1)	6693 (72)	3719 (63)	5182 (180)	1·069 (118)
H(O25)(2)	7319 (77)	3694 (63)	6515 (175)	0·826 (119)
H(O26)(1)	7444 (74)	4355 (59)	3882 (193)	1·114 (125)
H(O26)(2)	7676 (74)	5279 (61)	2455 (182)	1·260 (114)
H(O27)(1)	6231 (77)	4709 (61)	1210 (185)	0·888 (121)
H(O27)(2)	5732 (72)	5112 (59)	387 (183)	0·836 (117)
H(O28)	7283 (56)	7145 (49)	6785 (138)	1·605 (115)

from simultaneous full-matrix refinement of interlayer scale factors and all U_{33}, but results in U_{33} values which may be systematically in error. The final R factor is 0·061. 11 reflexions showing signs of being affected by extinction were omitted from the refinement.*

Discussion of the structure

The final coordinates of the non-hydrogen atoms are given in Table 2. The bond distances and angles are listed with their standard deviations in Tables 4 and 5.

The H atom coordinates and the bond lengths to the associated heavy atoms are given in Table 3; the average heavy atom–hydrogen distance is 1·027 Å with a standard deviation of 0·156 Å. Table 6 gives the donor–acceptor distances and donor–H–acceptor bond angles for those atoms involved in hydrogen bonding. Fig. 1 shows the amoxycillin trihydrate structure viewed along **c** drawn in the orientation required for direct comparison with the structure of ampicillin trihydrate shown in Fig. 2 and ampicillin anhydrate shown in Fig. 3 (Boles & Girven, 1976*a*,*b*).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33000 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 4. *Bond distances and their standard deviations (Å) after final least-squares refinement*

S(1)–C(2)	1·843 (6)	C(6)–N(14)	1·426 (8)
S(1)–C(5)	1·775 (8)	N(14)–C(15)	1·346 (8)
C(2)–C(3)	1·559 (9)	C(15)–O(16)	1·219 (8)
C(3)–N(4)	1·456 (8)	C(15)–C(17)	1·516 (9)
N(4)–C(5)	1·492 (8)	C(17)–N(18)	1·481 (8)
C(5)–C(6)	1·575 (10)	C(17)–C(19)	1·514 (9)
C(6)–C(7)	1·515 (9)	C(19)–C(20)	1·372 (10)
N(4)–C(7)	1·381 (9)	C(20)–C(21)	1·375 (10)
C(2)–C(9)	1·527 (10)	C(21)–C(22)	1·372 (10)
C(2)–C(10)	1·499 (11)	C(22)–C(23)	1·372 (10)
C(3)–C(11)	1·545 (9)	C(23)–C(24)	1·391 (10)
C(11)–O(12)	1·218 (9)	C(19)–C(24)	1·386 (9)
C(11)–O(13)	1·255 (8)	C(22)–O(28)	1·379 (8)
C(7)–O(8)	1·200 (8)		

Table 5. *Bond angles and their standard deviations (°)*

C(3)–C(2)–S(1)	104·3 (0·4)	O(13)–C(11)–C(3)	114·9 (0·6)
N(4)–C(3)–C(2)	105·6 (0·5)	O(13)–C(11)–O(12)	125·9 (0·6)
N(4)–C(5)–S(1)	103·4 (0·5)	N(14)–C(15)–C(17)	113·7 (0·5)
C(5)–S(1)–C(2)	90·1 (0·3)	N(14)–C(6)–C(5)	117·8 (0·5)
C(5)–N(4)–C(3)	117·1 (0·5)	N(14)–C(6)–C(7)	114·1 (0·6)
C(6)–C(5)–S(1)	118·6 (0·5)	C(15)–N(14)–C(6)	123·1 (0·6)
C(6)–C(5)–N(4)	86·8 (0·5)	C(15)–C(17)–N(18)	109·7 (0·5)
C(6)–C(7)–N(4)	93·3 (0·5)	C(15)–C(17)–C(19)	107·9 (0·5)
C(7)–N(4)–C(3)	127·5 (0·5)	O(16)–C(15)–C(17)	121·4 (0·6)
C(7)–N(4)–C(5)	93·3 (0·5)	O(16)–C(15)–N(14)	124·5 (0·6)
C(7)–C(6)–C(5)	85·1 (0·5)	C(19)–C(17)–N(18)	112·0 (0·5)
O(8)–C(7)–N(4)	131·2 (0·6)	C(20)–C(19)–C(17)	119·2 (0·6)
O(8)–C(7)–C(6)	135·3 (0·6)	C(21)–C(20)–C(19)	121·1 (0·6)
C(9)–C(2)–C(3)	112·2 (0·6)	C(22)–C(21)–C(20)	119·2 (0·7)
C(9)–C(2)–S(1)	109·2 (0·5)	C(23)–C(22)–C(21)	121·3 (0·7)
C(10)–C(2)–S(1)	108·6 (0·5)	C(23)–C(24)–C(19)	120·3 (0·7)
C(10)–C(2)–C(3)	112·4 (0·6)	C(24)–C(23)–C(22)	118·9 (0·7)
C(10)–C(2)–C(9)	109·8 (0·6)	C(24)–C(19)–C(17)	121·5 (0·6)
C(11)–C(3)–C(2)	113·5 (0·5)	C(24)–C(19)–C(20)	119·2 (0·6)
C(11)–C(3)–N(4)	112·8 (0·5)	O(28)–C(22)–C(21)	121·8 (0·7)
O(12)–C(11)–C(3)	119·2 (0·6)	O(28)–C(22)–C(23)	116·9 (0·6)

Table 6. *Distances and angles between donor–H–acceptor atoms involved in hydrogen bonding in amoxycillin trihydrate*

Standard deviations in parentheses are with respect to last figures given.

Donor (*D*)	Acceptor (*A*)	*D*–*A* distance	*D*–H · · · *A* angle
O(25)	O(8)	2·863 (7) Å	119·6°
O(25)	O(13)	2·733 (7)	171·5
O(26)	O(25)	2·894 (9)	158·6
O(26)	O(25)	2·711 (8)	147·4
O(27)	O(26)	2·892 (8)	151·6
O(27)	O(16)	2·827 (9)	159·1
O(28)	O(12)	2·659 (8)	107·2
N(18)	O(26)	2·802 (10)	165·6
N(18)	O(12)	2·847 (8)	124·8
N(18)	O(13)	2·755 (8)	144·4

Table 7. *Equations expressed as $Px + Qy + Rz = S$ in direct space*

P	*Q*	*R*	*S*	Deviations (Å) of atoms from planes	
(*a*) Planarity of the thiazolidine ring					
12·509	5·160	−3·609	4·897	C(2)	−0·006
				C(3)	0·012
				N(4)	−0·012
				C(5)	0·007
				*S(1)	0·832
(*b*) Pyramidal nature of N(4)					
6·493	7·933	−5·350	3·058	C(3)	0·000
				C(5)	0·000
				C(7)	0·000
				*N(4)	−0·382
(*c*) Deviation of O(8) from the plane of the β-lactam					
4·655	3·881	−6·184	0·113	N(4)	−0·064
				C(5)	0·055
				C(6)	−0·054
				C(7)	0·062
				*O(8)	0·297

* Atoms not used to define the planes.

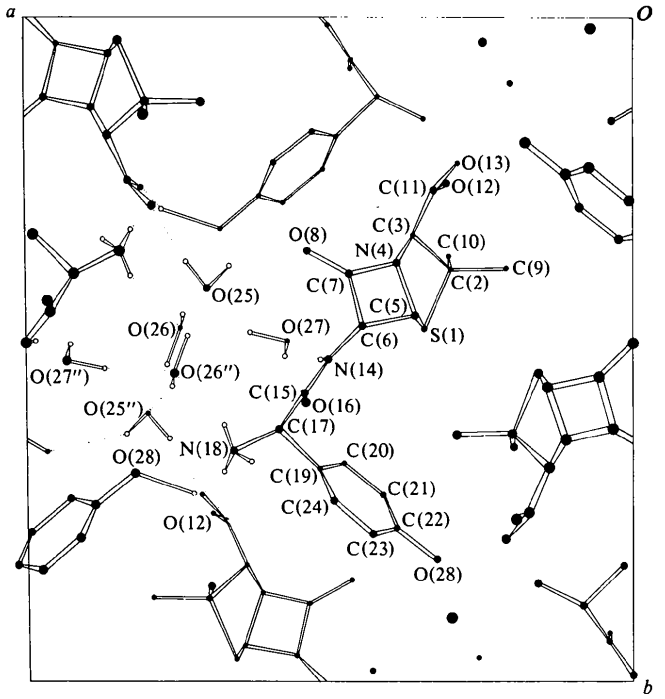


Fig. 1. Amoxycillin trihydrate viewed along **c**. Hydrogen atoms involved in hydrogen bonding are shown and the bonds indicated by dotted lines.

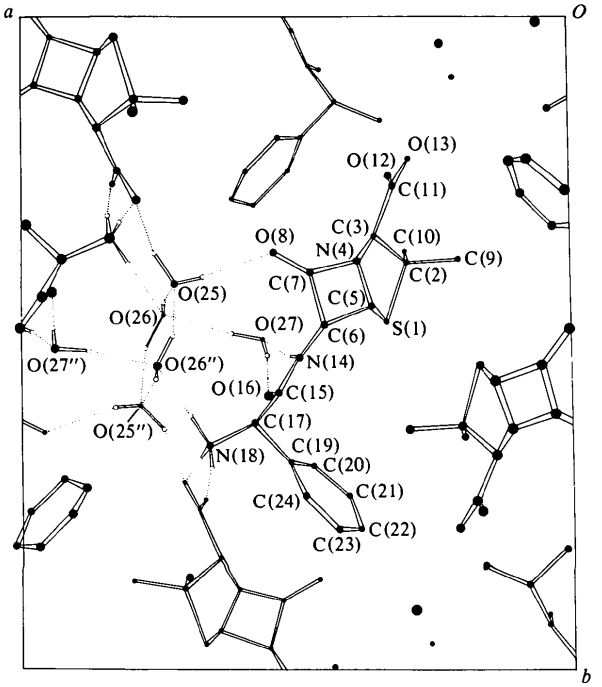


Fig. 2. Ampicillin trihydrate viewed along **c**.

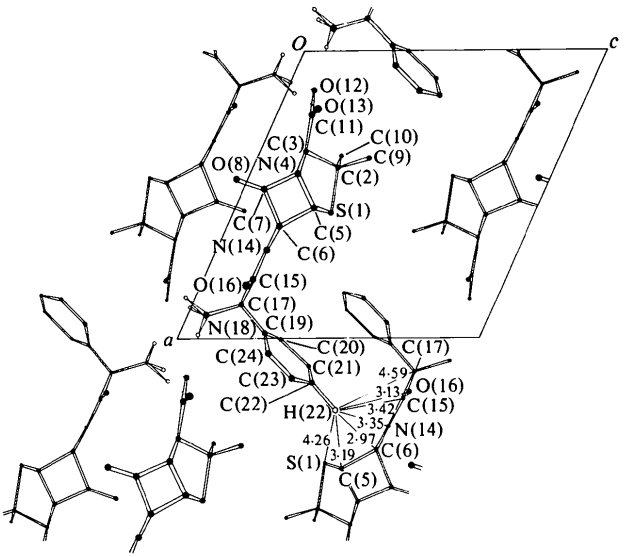


Fig. 3. Ampicillin anhydrate viewed along **b**. [Distances from H(22) to atoms on the symmetry-related molecule are shown in Å.]

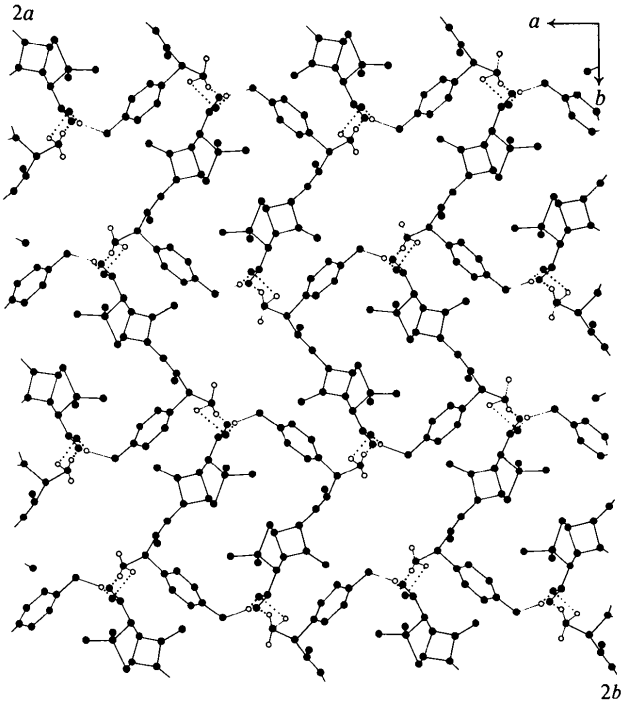


Fig. 4. Amoxycillin trihydrate, with the water molecules omitted, viewed along **c**.

Comparison of Figs. 1 and 2 shows that the molecular packings of the trihydrates of amoxycillin and ampicillin are very similar; this was expected from a comparison of the zero-level Weissenberg films. This similarity is also apparent in the molecular conformation; for example, the thiazolidine ring of amoxycillin consists of four atoms in an approximately planar arrangement with S(1) being out of this plane by 0·832 Å (0·841 Å in ampicillin trihydrate) and the distance of N(4) from the plane defined by C(3), C(5), C(7) is 0·382 Å, the same as in ampicillin trihydrate. Therefore, the configuration of this N atom conforms to the pyramidal arrangement which is a common

feature of biologically active β-lactam antibiotics (Sweet & Dahl, 1970; Vijayan, Anderson & Hodgkin, 1973). The β-lactam carboxyl O(8) is 0·297 Å from the plane of the β-lactam ring. The results of these calculations are shown in Table 7.

The major difference between the structures of amoxycillin and ampicillin trihydrates is the hydrogen bonding between the H of O(28) and the O(12) of a symmetry-related molecule. This O—H bond distance of 1·605 Å is considerably longer than the average heavy atom–hydrogen distance of 1·027 Å because of the electron-donating effect of the benzene ring, resulting in a strong hydrogen bond between O(28) and O(12). Both of the carboxyl O atoms, O(12) and O(13), are hydrogen bonded to two donor atoms.

A recent paper has described three known crystalline forms of ampicillin (Boles & Girven, 1976*b*). The authors have not succeeded in crystallizing forms of amoxycillin other than the trihydrate and it is of interest to examine the possible steric or structural reasons for this. Fig. 3 shows the distances from H(22) of ampicillin anhydrate to a number of atoms on the symmetry-related molecule. There are two important steric considerations involved in the replacement of H(22) by the —OH group in this structure: (*a*) The stability of ampicilllin anhydrate in the **c** crystallographic direction is enhanced by the van der Waals forces due to the partial overlapping of the benzene rings of symmetry-related molecules. The inclusion of the —OH would certainly reduce this overlap. (*b*) H bonding between OH···O(16) which would contribute to the stability of the proposed anhydrate is highly unlikely since the H(22)···O(16) distance of 3·13 Å in ampicillin anhydrate is longer than the expected donor–acceptor distance (average donor–acceptor distance in amoxycillin trihydrate = 2·806 Å), and replacement of H(22) by OH increases this distance. It seems unlikely, therefore, that hydrogen bonding could replace the stability lost by displacement of the benzene rings.

The delta form of ampicillin anhydrate reported by Parker & Stanniforth (1975) is prepared by rapidly heating purified ampicillin trihydrate under specified conditions to a temperature of 120°C. Similar treatment of amoxycillin trihydrate results in loss of crystallinity and apparent decomposition, but it has not been possible to produce the equivalent delta form of amoxycillin. The crystal structure of delta-form ampicillin is not known but analysis of the X-ray powder pattern is consistent with an overall increase in the cell dimensions compared with ampicillin trihydrate, as shown in Table 8. This is a surprising observation since the unit-cell content of delta-form ampicillin is less than that of the trihydrate. It appears that the trihydrate lattice expands to allow the escape of the water molecules and does not return to a close-packed structure. It seems likely that the extra OH···O(12) bonding between symmetry-related molecules in amoxycillin prevents the expansion of the lattice in this manner. Figs. 4 and 5 show the projections down **c** of the trihydrates of amoxycillin and ampicillin, respectively, with the water molecules excluded from the diagrams. Fig. 5 shows that in the absence of the bonding due to the water molecules the ampicillin structure contains no strong bonds in the **a** direction and is therefore comparatively free to move in the *bc* plane. Table 8 shows that the increase in the *b* and *c* cell dimensions in the trihydrate–delta form transformation is large, requiring considerable freedom of movement in this plane. Fig. 4 shows clearly the strong hydrogen bonding between OH···O(12) of amoxycillin, which is predominantly in the **a** direction, thus considerably restricting molecular rearrangement in the *bc* plane and preventing the formation of the form of amoxycillin equivalent to delta-form ampicillin.

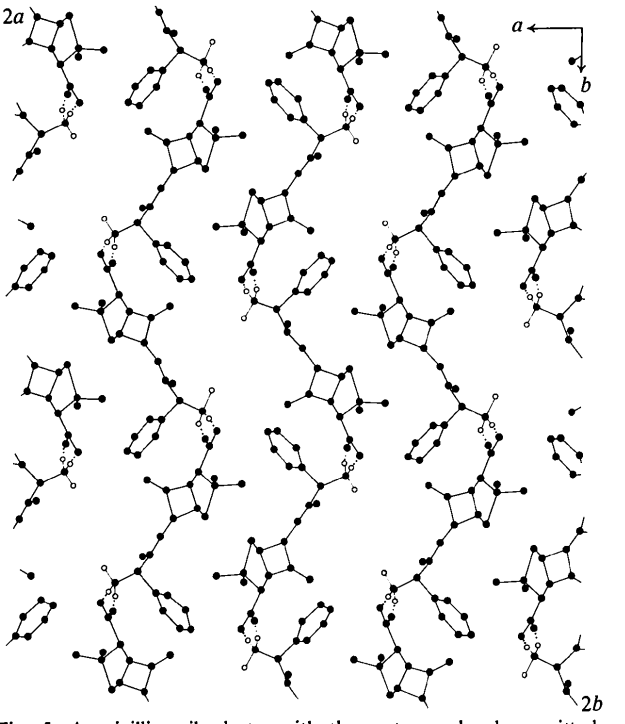


Fig. 5. Ampicillin trihydrate, with the water molecules omitted, viewed along **c**.

Table 8. *Comparison of cell dimensions* (Å) *of ampicillin trihydrate and delta-form ampicillin anhydrate*

	Ampicillin trihydrate	Delta-form ampicillin anhydrate	Percent increase
a	15·49	16·12	4·06
b	18·891	21·96	16·24
c	6·662	7·92	18·88

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The Structure of the Molecular Compound Decachloropyrene: Benzene, $C_{16}Cl_{10}:C_6H_6$

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The molecular compound between decachloropyrene and benzene is monoclinic, space group $P2_1/c$, with a = 18·043 (9), b = 7·180 (4), c = 17·579 (8) Å, β = 98·75 (0·2)°, Z = 4. Least-squares refinement with 3601 reflexions [$I > 3\sigma(I)$] gave R = 0·046 and R_w = 0·053 for 314 parameters. The molecule is saddle-shaped, the maximum deviations from planarity being *ca* 1 Å for Cl and *ca* 0·5 Å for C. The mean nearest-neighbour Cl···Cl distance in the molecule is 3·025 (1) Å, the mean C—Cl distance is 1·721 (1) Å. The geometry is similar to that found in decachloropyrene itself. The molecules pack in columns of alternating decachloropyrene and benzene molecules parallel to **b**.

Introduction

Decachloropyrene is an overcrowded molecule. The strain is relieved by a splaying of angles and by large out-of-plane displacements (Hazell & Jagner, 1976) to give a saddle-shaped molecule. Packing forces cause the decachloropyrene molecule to deviate from the ideal *mm*2 symmetry. The structure of decachloropyrene:benzene has been determined to compare the geometries of the decachloropyrene molecule in different environments. The decachloropyrene molecules will be referred to as DCP and DCPB in the two compounds.

Crystal data

$C_{16}Cl_{10}:C_6H_6$, M_r = 624·8, monoclinic, a = 18·043 (9), b = 7·180 (4), c = 17·579 (8) Å, β = 98·75 (0·2)°, U = 2251 Å^3, Z = 4, D_c = 1·84 g cm^{-3}, space group $P2_1/c$. Packing coefficient 0·75, μ(Mo $K\alpha$) 12·6 cm^{-1}.