streak so often observed. He has also shown from potential energy considerations that the 15*R* structure is the most stable next to 6*H*, but the question still remains how such extremely improbable structures with large identity periods grow into stable forms.

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The Crystal Structure of α_1-Bromopicrotoxinin

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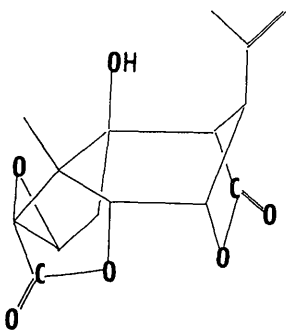
Crystals of α_1-bromopicrotoxinin ($C_{15}H_{15}O_6Br$) are orthorhombic, space group $P2_12_12_1$, with lattice parameters $a = 13{\cdot}40$, $b = 11{\cdot}60$, $c = 8{\cdot}86$ Å, and four molecules in the unit cell. A three-dimensional analysis confirms the structure postulated for picrotoxinin ($C_{15}H_{16}O_6$) by Conroy on chemical evidence. By consideration of the anomalous scattering of X-rays by the bromine atom, the absolute molecular configuration of α_1-bromopicrotoxinin, and hence of picrotoxinin itself, has been established and shown to be the mirror image of the structure as it appears in the previous literature.

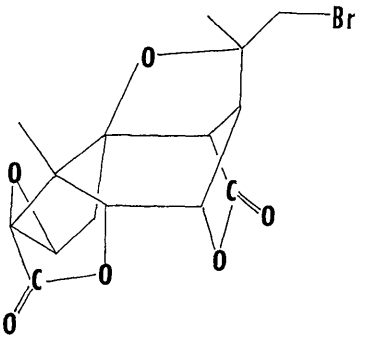
Introduction

Picrotoxinin ($C_{15}H_{16}O_6$) is the physiologically active component which, together with picrotin ($C_{16}H_{18}O_7$) forms the addition compound picrotoxin, a bitter tasting convulsive poison found in the berries of the East Indian creeper, *Anamirta paniculata* and first isolated by Boullay in 1812. These extremely toxic berries have been used by natives to catch fish. Because of its stimulating action on the central nervous system picrotoxin has been used extensively in medicine as an antibarbiturate.

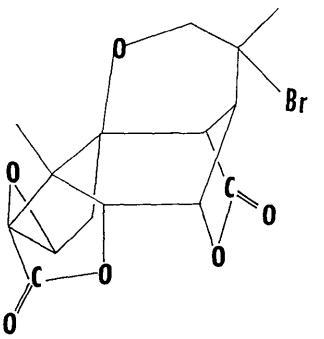
The chemistry of picrotoxinin and picrotin has been a subject of investigations for many years: e.g. by Horrmann (1916), Angelico & Monforte (1923), Robertson, O'Donnel & Harland (1939), Sutter & Schlittler (1949), Slater *et al.* (1956). The structure (I) for picrotoxinin was proposed by Conroy (1951) and subsequently (1957) supported by his conformational analysis.



(I)



(IIa)



(IIb)

The crystal structure studies in this field have been concerned with the halogen substituted derivatives of picrotoxinin ($C_{15}H_{15}O_6X$) for which two structures (II(a)) and (II(b)) were proposed by Conroy, to account for the occurrence of α and β isomers. Conroy did not identify which of the proposed structures he thought was α and which was β.

In the preliminary X-ray diffraction studies of these compounds (Craven, 1958) it was shown that α-bromopicrotoxinin was a mixture of two crystalline modifications here called α_1 and α_2. The relationship between these is still unknown. Crystal structure analysis of both compounds was begun and it was found in a two-dimensional investigation, that both the α_1 and α_2 isomers were suited to crystal structure analysis using the heavy atom technique. The results are now reported of a three-dimensional analysis of α_1-bromopicrotoxinin which confirms the postulated molecular structure (II(a)) except for reversal of sense by mirror reflection.

The crystal data

The crystals were small transparent prisms obtained from 1:10 dioxane-ethyl alcohol solution. The cell parameters were determined from oscillation and Weissenberg photographs taken with Cu $K\alpha$ radiation. The crystal density was measured using the method of flotation in a mixture of chloroform and bromoform.

α_1-Bromopicrotoxinin ($C_{15}H_{15}O_6Br$)
Orthorhombic,

$$a=13{\cdot}40,\ b=11{\cdot}60,\ c=8{\cdot}86\ \text{Å}.$$

M.W. 371

$$U=1378\ \text{Å}^3,\ D_m=1{\cdot}78,\ D_x=1{\cdot}80\ \text{g.cm.}^{-3}.$$

$$Z=4,\ \text{Space group}\ P2_12_12_1.$$

Experimental

The intensity data were recorded on multiple film equi-inclination Weissenberg photographs using Cu $K\alpha$ radiation, 6 layers being photographed about a and 5 layers about both b and c, and the intensities were estimated by eye. Since after 50 hours exposure in the X-ray beam the crystals turned yellow and decomposed, several crystals had to be photographed to obtain all the data. These were chosen as nearly as possible to be cubes with an edge of 0·02 cm. No absorption corrections were made. Of 1710 accessible reflections unrelated by symmetry, 1460 were observed, ranging in relative intensity from 1 to 1200. The 250 intensities too weak to be recorded were all estimated to be half the weakest observable intensity.

The structure determination

All the calculations in this work were carried out on the IBM 650 at the University of Pittsburgh Computation and Data Processing Center using programs designed by Shiono (1957, 1959, 1960) unless otherwise acknowledged.

The bromine atom position as determined from the Patterson projections (Craven, 1958) was confirmed by calculation of the three-dimensional Patterson sections at $u=\frac{1}{2}$, $v=\frac{1}{2}$ and $w=\frac{1}{2}$.

A three-dimensional Fourier synthesis was then computed including in the summation the 1200 reflections for which the amplitude of the bromine contribution amounted to more than one quarter of the corresponding observed structure amplitude. These reflections were assigned the phase angles of the bromine atom contributions. The resulting electron density distribution showed, apart from the bromine peak, about thirty fairly well resolved maxima, large enough to correspond to the twenty-one carbon and oxygen atoms. Of these maxima the eleven largest and most spherical were assumed to be carbon or oxygen positions. When the spatial arrangement of these eleven peaks was compared with models of the molecular structures proposed by Conroy (II), a particular orientation of model (II(a)) was seen to correspond most strikingly to the eleven selected maxima of the calculated electron density map. Approximate atomic positions in the crystal for all oxygen and carbon atoms were then immediately obtained from other existing maxima on the map, and the structure factors calculated from these positions enabled a second Fourier synthesis to be prepared, all but about twenty reflections being included in the summation. The atomic scattering factors used in the structure factor calculations were those of Thomas–Fermi (1935) for bromine, and Berghuis *et al.* (1955) for carbon and oxygen.

In this map all the spurious maxima were found to have disappeared and the molecule was clearly revealed. Calculated structure factors gave a reliability factor $R=0{\cdot}30$. Further refinement was by two cycles of differential syntheses and structure factor calculations, using the following isotropic temperature factors derived from the preliminary two-dimensional investigations, $B_{Br}=3{\cdot}0$ Å^2, $B_C=B_O=2{\cdot}5$ Å^2. An n-shift of 1·7 was used in the differential syntheses.

The reliability index was reduced to $R=0{\cdot}19$, after which it was apparent that in further refinement it would be necessary to consider the anisotropic thermal vibration of the bromine atom which was evident both in the peak shape in the second Fourier synthesis and also in the peak curvatures calculated in the differential syntheses.

A series of three cycles of differential syntheses and structure factors was computed in which the carbon and oxygen parameters were held constant and only the three bromine positional parameters and six bromine anisotropic thermal parameters were allowed to change. The final values obtained for the thermal parameters were $B_{11}=4{\cdot}12$ Å^2, $B_{22}=2{\cdot}38$, $B_{33}=3{\cdot}50$, $B_{23}=-0{\cdot}80$, $B_{12}=1{\cdot}14$, $B_{31}=0{\cdot}30$.

Table 1. *Atomic parameters and standard deviations*

Atom	x	y	z	$\sigma(r)$ (Å)
Br	0·2028	0·9759	0·8327	0·0022
C_1	0·4567	0·6303	0·6803	0·017
C_2	0·3773	0·5360	0·7167	0·017
C_3	0·2720	0·5788	0·7173	0·017
C_4	0·2610	0·7084	0·7647	0·018
C_5	0·3026	0·7607	0·6187	0·017
C_6	0·4145	0·7536	0·6628	0·015
C_7	0·5454	0·6241	0·7812	0·019
C_8	0·3241	0·7674	0·8864	0·018
C_9	0·2751	0·8666	0·9647	0·016
C_{10}	0·3554	0·6874	1·0170	0·018
C_{11}	0·4816	0·8090	0·5422	0·017
C_{12}	0·4958	0·7091	0·4288	0·018
C_{13}	0·4853	0·6018	0·5214	0·018
C_{14}	0·4442	0·4853	0·4830	0·018
C_{15}	0·2644	0·6805	0·5019	0·016
O_1	0·4119	0·8121	0·8021	0·012
O_2	0·5801	0·6361	0·4690	0·013
O_3	0·4608	0·4233	0·3765	0·015
O_4	0·3875	0·4483	0·6026	0·014
O_5	0·2542	0·6951	0·3636	0·014
O_6	0·2350	0·5787	0·5646	0·012

Table 2

(*a*) Bond lengths with standard deviations

	d (Å)	$\sigma(d)$ (Å)		d (Å)	$\sigma(d)$ (Å)
Br–C_9	1·98	0·016	C_{13}–C_{14}	1·50	0·025
C_9–C_8	1·50	0·024	C_5–C_{15}	1·48	0·024
C_8–C_{10}	1·54	0·025	C_8–O_1	1·49	0·021
C_8–C_4	1·53	0·025	C_6–O_1	1·41	0·019
C_1–C_2	1·56	0·024	C_2–O_4	1·44	0·022
C_2–C_3	1·50	0·024	C_3–O_6	1·44	0·021
C_3–C_4	1·57	0·025	C_{12}–O_2	1·46	0·022
C_4–C_5	1·53	0·025	C_{13}–O_2	1·41	0·022
C_5–C_6	1·55	0·023	C_{14}–O_4	1·37	0·023
C_1–C_7	1·49	0·025	C_{15}–O_6	1·36	0·020
C_6–C_{11}	1·54	0·023	C_{14}–O_3	1·21	0·023
C_{11}–C_{12}	1·55	0·025	C_{15}–O_5	1·24	0·021
C_{12}–C_{13}	1·50	0·025			

(*b*) Bond angles with standard deviations

	β (°)	$\sigma(\beta)$ (°)		β (°)	$\sigma(\beta)$ (°)
Br–C_9–C_8	116	1·2	C_{13}–C_1–C_2	102	1·4
C_9–C_8–C_{10}	104	1·4	C_{13}–C_1–C_7	110	1·5
C_9–C_8–C_4	115	1·5	C_6–C_1–C_7	113	1·4
C_9–C_8–O_1	108	1·4	C_2–C_1–C_7	113	1·4
C_{10}–C_8–C_4	114	1·5	C_{12}–C_{13}–O_2	60	1·1
C_{10}–C_8–O_1	112	1·4	C_{13}–O_2–C_{12}	63	1·1
C_8–C_4–C_3	124	1·5	O_2–C_{12}–C_{13}	57	1·1
C_8–C_4–C_5	102	1·4	C_{11}–C_{12}–O_2	112	1·4
C_3–C_4–C_5	97	1·3	C_1–C_{13}–O_2	118	1·5
C_4–C_5–C_6	97	1·3	C_{12}–C_{13}–C_{14}	131	1·6
C_5–C_6–C_1	115	1·3	O_2–C_{13}–C_{14}	121	1·5
C_6–C_1–C_2	115	1·4	C_1–C_{13}–C_{14}	109	1·4
C_1–C_2–C_3	114	1·4	C_{13}–C_{14}–O_4	108	1·4
C_2–C_3–C_4	103	1·4	C_{13}–C_{14}–O_3	130	1·7
C_4–C_8–O_1	104	1·2	O_4–C_{14}–O_3	121	1·7
C_8–O_1–C_6	107	1·2	C_{14}–O_4–C_2	112	1·4
O_1–C_6–C_5	100	1·3	C_4–C_5–C_{15}	102	1·4
C_5–C_6–C_{11}	111	1·3	C_6–C_5–C_{15}	118	1·4
C_{11}–C_6–O_1	115	1·3	C_5–C_{15}–O_6	111	1·4
C_1–C_6–O_1	111	1·3	C_5–C_{15}–O_5	130	1·6
C_1–C_6–C_{11}	104	1·3	O_6–C_{15}–O_5	119	1·5
O_6–C_{11}–C_{12}	102	1·3	C_{15}–O_6–C_3	106	1·3
C_{11}–C_{12}–C_{13}	105	1·4	O_6–C_3–C_4	103	1·3
C_{12}–C_{13}–C_1	111	1·5	O_6–C_3–C_2	109	1·4

Table 3. *Some intramolecular distances*

	d (Å)	$\sigma(d)$ (Å)		d (Å)	$\sigma(d)$ (Å)
Br–C_4	3·30	0·018	C_{12}–O_5	3·29	0·023
–C_5	3·42	0·017	–C_{15}	3·18	0·024
–C_8	2·98	0·018			
–O_1	3·41	0·012	C_{13}–C_{15}	3·11	0·024
			–O_5	3·56	0·022
C_2–C_{10}	3·18	0·025			
			C_{14}–C_{15}	3·34	0·024
C_7–C_{10}	3·36	0·026	–O_5	3·70	0·022
–O_1	2·86	0·022			
–O_2	2·77	0·023	O_3–O_5	4·29	0·020

Table 4. *Some short intermolecular approaches*

Except for the bromine atom, only those intermolecular approaches are listed which come near or within the sums of the Van der Waals radii as given by Pauling (1960) i.e. carbon (methyl group) 2·0 Å, oxygen 1·4 Å

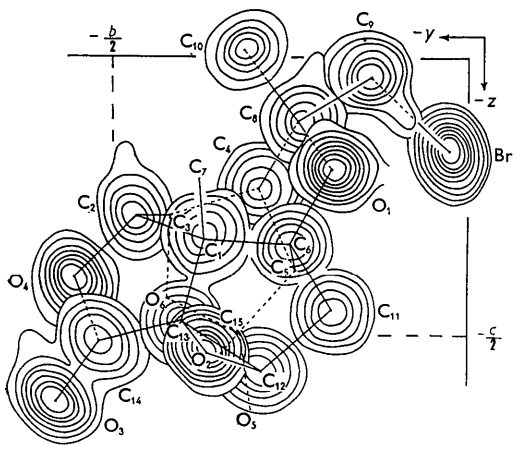
	d (Å)	$\sigma(d)$ (Å)
Br(IV)–O_2 (II)	3·37	0·013
–O_2 (I)	3·88	0·013
–O_3 (II)	3·91	0·015
–C_7 (I)	3·93	0·019
–O_5 (III)	3·93	0·014
–C_{11}(I)	3·99	0·017
C_2 (I)–C_{11}(IV)	3·89	0·024
C_3 (I)–C_{10}(II)	3·98	0·025
–C_{14}(II)	3·79	0·025
–C_{15}(II)	3·97	0·024
C_5 (I)–O_2 (III)	3·31	0·022
C_{10}(I)–O_6 (II)	3·39	0·022
C_{11}(I)–C_{15}(III)	3·81	0·022
C_{12}(I)–C_{15}(III)	3·88	0·024
C_{14}(I)–O_1 (IV)	3·38	0·021
C_{15}(I)–O_2 (III)	3·29	0·021

Molecule	
(I)	x, y, z
(II)	$\frac{1}{2}-x, \bar{y}, \frac{1}{2}+z$
(III)	$\frac{1}{2}+x, \frac{1}{2}-y, \bar{z}$
(IV)	$\bar{x}, \frac{1}{2}+y, \frac{1}{2}+y, \frac{1}{2}-z$

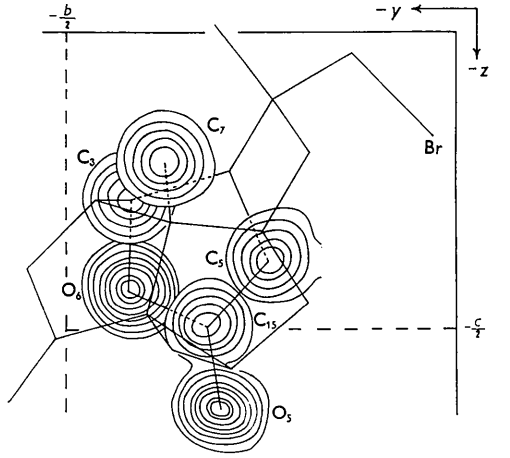
At this stage it was seen that the observed intensities were considerably affected by extinction (see Table 6). In order to limit the effect of extinction on the final atomic parameters, 52 reflections (all those for which $F_o > 50$ up to $\sin\theta = 0{\cdot}35$) were included in the final differential synthesis with the observed structure factor equal to the corresponding calculated value. The final differential synthesis was then carried out involving all carbon and oxygen atoms.

In the final structure factor calculations, the hydrogen atoms were omitted and the light atoms were given the isotropic temperature factors $B_C = 1{\cdot}8$ Å^2 and $B_O = 2{\cdot}5$ Å^2. The larger temperature factor for the oxygen atoms is considered to arise from their lying mostly at the periphery of the molecule.

The final reliability factor is $R = 0{\cdot}17$ for all reflections (unobserved amplitudes included as half the minimum observable value), or $R = 0{\cdot}15$, when the 52 reflections seriously affected by extinction are omitted.



(a)



(b)

Fig. 1(*a*), (*b*). The final three-dimensional electron density distribution.

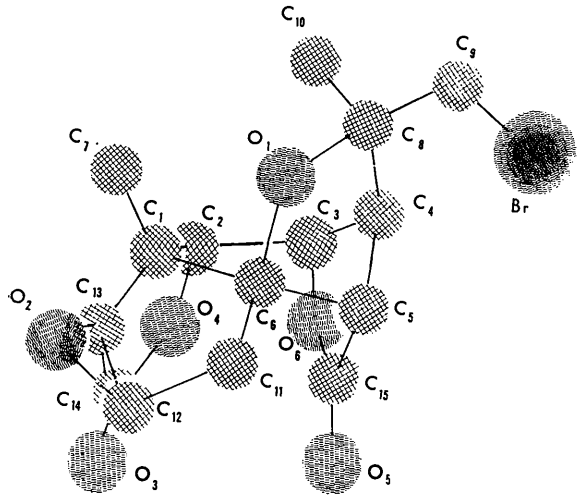


Fig. 2. The absolute molecular configuration of α_1-bromopicrotoxinin.

The final atomic positional parameters are listed in Table 1; the bond lengths and bond angles are in Table 2 and these are illustrated in Figs. 3(*a*) and 3(*b*). Tables 3 and 4 show some intra- and intermolecular non-bonding distances, calculated using the program for the IBM 650 by Templeton (1957). The standard deviations listed in these tables were calculated according to Cruickshank (1949, 1950, 1954). Fig. 1 is a complete electron-density map made from sections of the final three-dimensional Fourier synthesis. The resulting molecular configuration is shown in Fig. 2 in a view corresponding to Conroy's postulated structure (II(*a*)). Fig. 4 is an attempt to show the molecular arrangement in the crystal. Molecule (I) which corresponds to the listed parameters of Table 1 and the map Fig. 1, is shown most heavily shaded. The observed and calculated structure factors are listed in Table 6.

The absolute configuration of the molecule

The anomalous scattering of X-rays has been used to determine absolute configurations of molecules of similar complexity to picrotoxinin since the method was first proposed by Bijvoet, Peerdeman & van Bommel (1951). The procedure in the present work follows that described by Peerdeman & Bijvoet (1956) and applied by Peerdeman (1956) in the case of strychnine hydrobromide.

With Cu K radiation the complex scattering factor for bromine has been given by Dauben & Templeton (1955) as $\Delta f' = -0{\cdot}9$ and $\Delta f'' = 1{\cdot}5$. This gives rise to an anomalous scattering effect large enough to be observed photographically, provided that the small differences in scattered intensities are not masked by absorption effects.

A crystal of α_1-bromopicrotoxinin was ground to a sphere of diameter 0·03 cm. and Weissenberg photo-

Table 5. *Comparison of* I_{hkl} *and* $I_{\bar{h}\bar{k}\bar{l}}$

	Calc.		Obs.	
hkl	I_{hkl}	$I_{\bar{h}\bar{k}\bar{l}}$	I_{hkl}	$I_{\bar{h}\bar{k}\bar{l}}$
131	590	620	<	
141	194	220	<	
151	2600	2660	=	
161	372	348	>	
171	525	535	$\leqq$	
181	115	95	$\geqq$	
191	810	840	$\leqq$	
1,10,1	117	141	$\ll$	
1,11,1	352	362	=	
1,12,1	138	160	$\ll$	
1,13,1	308	340	>	(?)
612	196	260	$\ll$	
712	1060	980	>	
812	1340	1031	$\geqq$	
912	225	280	$\ll$	
10,1,2	2420	2750	<	
11,1,2	305	256	>	
12,1,2	730	700	$\geqq$	
13,1,2	225	255	<	

graphs were taken of the layers $hk1$ and $hk2$ using Cu $K\alpha$ radiation. The intensities $1k1$ and $\bar{1}k1$ ($=\bar{1}\bar{k}\bar{1}$), $h12$ and $\bar{h}12$ ($=\bar{h}\bar{1}\bar{2}$) which lie close to each other on the respective films, were compared visually. In Table 5, the observed intensity differences are seen to have approximately the same magnitudes and the same sense as the calculated differences except for the reflection (1,13,1) whose behaviour is not understood. Since both observed and calculated data refer to a right handed set of coordinate axes, the correct absolute configuration of the molecule is taken to be that which, by chance, had been used throughout in the crystal structure determination. A comparison with Conroy's proposed structure (II(a)) as drawn by arbitrary choice, shows it to be the mirror image of the true structure of α_1-bromopicrotoxinin. Conroy (private communication) now has chemical evidence supporting this result.

Discussion

The molecule, excluding the bromine atom, is approximately spherical with complex convolutions, the result of fusing together what may be described as one three-membered, three five-membered, one six-membered and one seven-membered rings. Although all bond lengths lie within three times the calculated standard deviations from their normal accepted values, the bond angles show the strain inherent in a molecule of this nature.

In particular, the six-membered carbon ring (C_1 to C_6) adopts an irregular form somewhere between the normal full and half chair configurations. ($C_2C_3C_5C_6$) are roughly coplanar. The best plane through these atoms was calculated by the least-squares method, using the program of Stewart (1958). The distances of the atoms from the best plane varied from 0·11 Å to 0·13 Å, while C_1 and C_4 lie at 0·19 Å and 0·98 Å respectively on opposite sides of this plane. The bond angles within the ring (see Fig. 3(b)) are 114° or 115° except for the angles at C_4 and C_5 which are 97°.

The five-membered carbon ring ($C_{11}C_{12}C_{13}C_1C_6$) forms with the epoxide O_2 a chair-form six-membered ring in which ($C_{11}C_{12}C_{13}C_1$) are coplanar, (maximum distance from the best plane is 0·03 Å) while O_2 and C_6 are 1·16 Å and 0·96 Å out of this plane in opposite senses.

The five-membered ether ring ($C_8C_4C_5C_6O_1$) has ($C_8C_4C_6O_1$) approximately coplanar (maximum distance from the best plane being 0·10 Å), C_5 being 0·77 Å out of this plane.

The three-membered epoxide ring is symmetrical about the line from O_2 to the midpoint of $C_{12}C_{13}$, within the significance test of three times the cal-

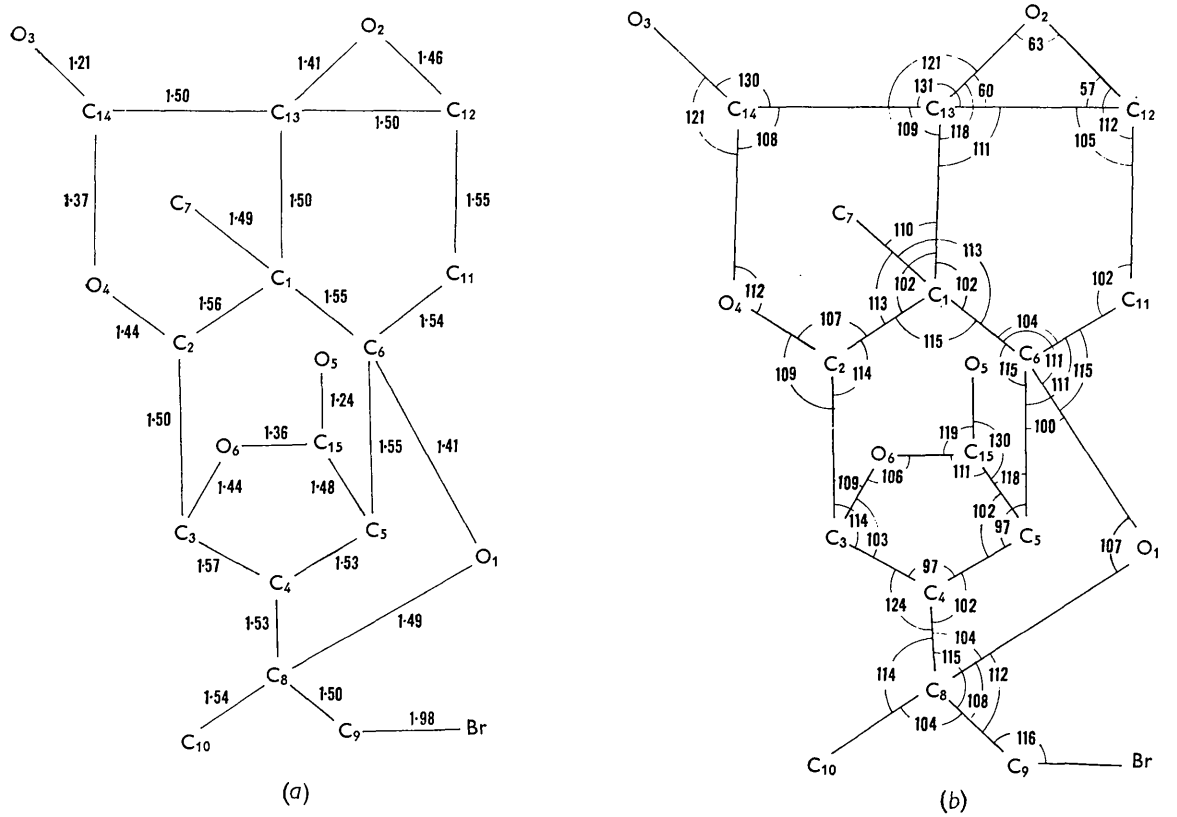


Fig. 3(a), (b). The bond lengths and bond angles in α_1-bromopicrotoxinin.

Table 6. *The observed and calculated structure factors for α_1-bromopicrotoxinin*

(Those reflections marked * are considered to be seriously affected by extinction—see text)

H	K	L	$\lvert F_o\rvert$	$\lvert F_c\rvert$	A_o	B_o
*2	0	0	59	96	59-	0
*4			81	135	81	0
*6			62	62	62-	0
8			18	12	18-	0
10			33	28	33	0
12			17	18	17-	0
14			6	2	6	0
16			4	4	4	0
1	1	0	25	32	0	25
*2			48	70	48-	0
*3			49	69	0	49-
4			26	28	26	0
5			29	28	0	29-
6			18	15	18-	0
7			47	50	0	47
8			12	8	12	0
9			79	91	0	79-
10			19	14	19-	0
11			28	29	0	28
12			6	2	6	0
13			20	17	0	20-
14			24	25	24-	0
15			20	18	0	20
16			4	2	4-	0
17			4	5	0	4
0	2	0	23	40	23	0
			33	47	0	33-
*2			46	52	46-	0
3			9	5	0	9
4			6	3	6	0
5			32	34	0	32-
6			12	12	12	0
7			12	9	0	12
8			33	39	33-	0
9			7	4	0	7
10			25	29	25	0
11			6	4	0	6
12			55	61	55-	0
13			7	2	0	7-
14			6	7	6	0
15			9	10	0	9-
16			5	0	5	0
17			2	0	0	2-
*1	3	0	57	86	0	57
*2			85	117	85	0
*3			106	158	0	106-
4			15	14	15-	0
5			7	4	0	7
6			51	54	51	0
7			23	21	0	23
8			29	24	29-	0
9			8	4	0	8-
10			13	11	13-	0
11			15	17	0	15
12			11	13	11	0
13			14	13	0	14-
14			5	5	5-	0
15			5	2	0	5
16			12	3	12	0
*0	4	0	57	75	58	0
1			3	0	0	3-
*2			69	95	69-	0
3			36	42	0	36
4			13	9	13-	0
5			34	40	0	34-
6			25	25	25	0
7			23	27	0	23
8			28	29	28-	0
9			6	5	0	6-
10			43	48	43	0
11			34	31	0	34-
12			18	14	18-	0
13			20	17	0	20
14			8	9	8	0
15			16	15	0	16-
16			5	12	5	0
1	5	0	43	47	0	43
2			31	29	31	0
3			17	16	0	17-
4			43	39	43	0
5			18	9	0	18
6			51	47	51	0
7			35	42	0	35
8			18	12	18	0
9			38	38	0	38-
10			31	25	31	0
11			42	46	0	42
12			7	4	7	0
13			5	3	0	5-
14			11	10	11	0
15			20	16	0	20
0	6	0	32	31	32	0
1			41	37	0	41
2			26	30	26	0
3			64	73	0	64
4	6	0	6	4	6	0
5			68	72	0	68
6			18	10	18	0
7			20	16	0	20
8			16	11	16	0
9			35	34	0	35
10	6	0	16	9	16	0
11			12	7	0	12
12			20	13	20	0
13			5	4	0	5
14			12	15	12	0
15			12	14	0	12
1	7	0	76	81	0	76
2			38	31	38	0
3			49	47	0	49
4			55	60	55	0
5			46	45	0	46
6			58	52	58	0
7			31	26	0	31
8			23	25	23	0
9			6	9	6	0
10			12	10	12	0
11			10	9	0	10
12			21	23	21	0
13			7	8	0	7
14			19	19	19	0
15			9	9	0	9
0	8	0	8	2	8	0
1			24	19	0	24
2			9	3	9	0
3			54	58	0	54
4			6	0	6	0
5			41	39	0	41
6			12	5	12	0
7			44	39	0	44
8			49	48	49	0
9			6	0	0	6
10			18	14	18	0
11			14	6	0	14
12			21	19	21	0
13			24	23	0	24
14			16	11	16	0
1	9	0	36	32	0	36
2			34	35	34	0
3			13	11	0	13
4			32	30	32	0
5			5	1	0	5
6			41	36	41	0
7			12	9	0	13
8	9	0	29	23	29	0
9			6	4	0	6
10			5	4	5	0
11			9	8	0	9
12			7	3	7	0
13			14	10	0	14
0	10	0	17	15	17	0
1			39	37	0	39
2			8	1	8	0
3			15	8	0	15
4			39	38	39	0
5			30	29	0	30
6			7	3	7	0
7			28	25	0	28
8			25	22	25	0
9			18	10	0	18
10			16	11	16	0
11			4	3	0	4
12			6	7	6	0
1	11	0	22	27	0	22
2			21	19	21	0
3			23	18	0	23
4			32	35	32	0
5			13	9	0	13
6			32	34	32	0
7			15	12	0	15
8			23	24	23	0
9			7	3	0	7
10			15	11	15	0
11			16	11	0	16
0	12	0	14	17	14	0
1			12	10	0	12
2			9	4	9	0
3			29	40	0	29
4			6	3	6	0
5			32	39	0	32
6			7	9	7	0
7			13	16	0	13
8			4	0	4	0
9			20	18	0	20
10			3	4	2	0
1	13	0	5	2	0	5
2			4	3	4	0
3			5	1	0	5
4			19	25	19	0
5			12	11	0	12
6			26	29	26	0
7			12	14	0	12
8			7	11	7	0
0	14	0	16	14	16	0
1			4	3	0	4
2			4	7	4	0
3			11	13	0	11
4			7	12	7	0
5	14	0	9	14	0	9
*1	0	1	46	62	0	46
2			32	49	0	32
3			29	32	0	29
4			14	8	0	14
*5			74	102	0	74
*6			59	72	0	59
7			24	27	0	24
8			14	5	0	14
9			11	7	0	11
10			36	30	0	36
11			6	4	0	6
12			26	27	0	26
13			8	9	0	8
14			21	17	0	21
15			22	22	0	22
16			4	5	0	4
17			11	11	0	11
0	1	1	41	67	0	41
*1			98	114	64	74
*2			55	80	51	19
*3			50	69	49	11
*4			87	121	69	53
*5			50	62	47	18
6			35	45	30	18
7			21	16	20	8
8			46	54	42	20
9			28	27	10	27
10			21	19	19	9
11			6	7	3	5
12			23	22	12	20
13			8	7	5	6
14			19	18	18	2
15			9	11	7	5
16			10	11	8	5
17			10	11	10	2
0	2	1	7	3	7	0
*1			55	79	40	37
*2			69	83	31	62
*3			89	122	74	50
4			32	38	28	16
*5			74	90	30	68
6			16	18	2	15
7			70	79	10	70
8			13	8	11	7
9			20	10	18	8
10			36	35	36	3
11			15	14	2	15
12			16	14	15	3
13			7	10	2	6
14			18	20	7	16
15			7	8	2	7
16			4	3	4	0
17			11	15	1	11
0	3	1	35	44	0	35
1			30	39	8	29
*2			65	87	65	1
*3			44	56	24	37
*4			49	60	48	11
*5			43	53	39	19
*6			35	43	35	3
7			43	54	41	12
8			41	43	40	9
9			25	27	26	9
10			19	19	9	16
11			17	15	9	14
12			21	22	14	15
13			15	15	10	12
14			12	12	12	1
15			4	1	2	3
16			12	12	12	2
0	4	1	30	30	30	0
1			24	24	16	18
*2			53	65	31	42
3			24	26	21	12
4			22	21	9	21
5			61	69	15	59
6			39	41	24	31
7			40	42	20	35
8			49	55	23	43
9			14	11	10	9
10			18	21	18	0
11			21	19	13	17
12			17	17	15	7
13			12	13	6	10
14			5	6	1	6
15			14	16	12	7
16			11	9	4	10
0	5	1	45	52	0	45
*1			60	85	20	56
2			17	19	14	10
3			42	43	11	41
4			43	45	43	6
5			32	29	31	9
6			39	42	29	2
7			29	25	3	29
8			30	26	17	25
9			25	23	8	24
10	5	1	21	19	19	10
11			18	16	1	18
12			8	7	1	8
13			12	11	8	8
14			18	17	16	8
15			11	7	10	5
0	6	1	70	88	70	0
1			29	30	29	6
2			19	20	17	7
3			48	58	26	40
4			36	35	19	31
5			41	40	4	41
6			31	28	15	27
7			36	33	4	36
8			40	37	38	13
9			35	33	30	19
10			30	26	29	8
11			9	4	7	5
12			18	15	15	9
13			5	2	5	1
14			5	2	3	3
15			10	8	2	10
0	7	1	50	47	0	50
1			33	35	2	33
2			26	24	25	7
3			14	13	2	14
4			20	20	12	15
5			17	16	16	5
6			32	30	32	5
7			23	21	12	20
8			26	23	8	25
9			28	25	10	27
10			26	20	0	26
11			26	23	8	25
12			16	14	8	13
13			16	16	8	13
14			11	11	5	9
15			4	2	4	0
0	8	1	52	52	52	0
1			20	18	18	7
2			45	46	45	1
3			28	31	20	20
4			32	28	31	8
5			6	4	6	2
6			12	4	5	10
7			22	17	17	15
8			21	20	21	0
9			33	30	17	29
10			20	18	19	4
11			16	15	16	1
12			12	15	11	5
13			16	14	13	9
14			11	10	4	10
0	9	1	39	40	0	39
1			42	45	8	41
2			36	38	6	36
3			26	26	12	24
4			42	45	2	42
5			20	14	14	14
6			15	13	14	4
7			26	21	15	22
8			17	12	13	10
9			32	29	10	31
10			14	13	9	11
11			24	21	8	23
12			9	8	8	3
13			14	12	5	13
0	10	1	38	37	38	0
1			15	17	13	7
2			42	45	42	6
3			10	8	7	7
4			24	22	23	8
5			13	10	13	1
6			18	20	11	14
7			27	24	27	4
8			24	21	24	4
9			13	9	5	12
10			29	27	28	8
11			20	20	19	5
12			10	9	9	3
0	11	1	25	17	0	25
1			30	28	7	29
2			32	32	12	30
3			17	18	4	16
4			8	9	8	0
5			9	8	7	5
6			17	12	9	14
7			9	8	5	7
8			20	18	3	19
9			16	17	5	15
10			4	6	2	4
11			16	18	3	15
0	12	1	31	31	31	0
1			19	18	18	5
2			21b	22	21	1
3			15	15	10	11
4			21	19	20	8
5			12	12	8	8
6			6	5	4	4
7			9	6	8	4
8	13	1	13	11	11	6
9			13	12	13	1
10			11	10	11	0
0	13	1	8	9	0	8
1			26	26	7	25
2			12	11	12	0
3			8	8	3	7
4			13	12	13	1
5			9	7	9	1
6			7	5	5	4
7			3	7	1	3
8			12	12	3	11
0	14	1	16	18	16	0
1			12	11	10	6
2			15	15	13	7
3			9	11	3	8
4			13	12	10	1
5			16	16	6	14
0	0	2	10	1	10	0
*1			52	76	52	0
*2			45	60	45	0
*3			69	92	69	0
4			23	18	23	0
5			18	19	18	0
6			21	14	21	0
7			37	42	37	0
8			34	34	34	0
9			29	28	29	0
10			21	17	21	0
11			17	13	17	0
12			25	25	25	0
13			7	8	7	0
14			8	10	8	0
15			6	0	6	0
16			5	1	5	0
*0	1	2	72	126	0	72
1			35	51	5	35
*2			83	115	11	82
3			15	19	4	14
4			35	38	35	5
5			21	22	16	14
6			18	14	17	2
7			29	30	8	28
8			35	36	32	14
9			19	17	8	17
10			46	48	18	42
11			23	14	9	22
12			28	25	4	28
13			21	16	19	1
14			10	9	2	10
15			8	7	8	1
16			5	4	4	0
0	2	2	46	52	46	0
1			44	45	44	3
2			42	49	41	9
3			29	30	26	14
4			62	59	14	60
5			41	42	12	39
6			42	44	41	2
7			57	63	54	19
8			28	22	4	28
9			51	47	51	6
10			25	24	25	4
11			43	44	34	27
12			22	20	21	9
13			30	29	27	15
14			10	11	9	3
15			14	15	11	8
16			3	5	0	3
*0	3	2	90	107	0	90
1			40	50	20	35
*2			62	74	7	61
3			33	33	3	33
4			42	42	7	41
5			57	64	47	31
6			18	16	18	1
7			23	22	3	23
8			5	4	1	5
9			25	27	11	23
10			31	33	8	30
11			11	12	0	11
12			20	19	11	16
13			10	13	8	6
14			8	7	2	6
15			4	2	4	1
16			8	8	5	6
*0	4	2	66	62	66	0
*1			64	75	55	34
*2			65	74	52	40
3			64	67	41	49
4			44	45	10	43
5			25	20	25	5
6			20	15	19	6
7			42	37	31	29
8			34	35	3	34
9			20	20	14	13
10			31	30	30	10
11			12	13	11	5
12			11	9	4	10
13			10	10	9	4
14			14	14	11	8

culated standard deviations in bond lengths and angles.

The calculated bond lengths and angles in the two β lactone bridges are almost the same. The small angles C_{13}–C_{14}–O_4, and C_5–C_{15}–O_6 (108° and 111°) for the lactone carbon atoms C_{14} and C_{15} must be a consequence of the strain in forming the β bridges. The asymmetry of the carbonyl bond angles C_{13}–C_{14}–O_3, O_3–C_{14}–O_4 (130° and 121°) and C_5–C_{15}–O_5, O_5–C_{15}–O_6 (130° and 119°) is believed to result from the proximity of C_{12} (3·44 Å and 3·29 Å from O_3 and O_5 respectively —see Fig. 2).

The two lactone bridges C_5 to C_3 and C_{13} to C_2 are each approximately coplanar and almost parallel,

Table 6 (*cont.*)

H	K	L	$\lvert F_o \rvert$	$\lvert F_c \rvert$	A_c	B_c
0	5	2	19	20	0	19
1			46	52	23	40
2			19	15	1	19
3			33	31	33	0
4			29	26	9	28
5			37	39	37	1
6			21	17	16	14
7			43	47	43	1
8			39	42	2	39
9			34	30	6	34
10			33	33	25	22
11			8	5	3	0
12			26	23	16	21
13			20	20	8	18
14			11	10	9	6
15			18	15	16	5
0	6	2	17	10	17	0
1			65	65	64	13
2			43	44	16	40
3			46	47	40	22
4			37	34	8	36
5			27	24	22	16
6			50	47	9	49
7			25	24	22	12
8			28	24	6	28
9			30	26	28	12
10			28	24	24	15
11			22	17	17	14
12			17	13	10	14
13			13	12	10	8
14			13	10	2	13
15			15	12	11	10
0	7	2	29	32	0	29
1			19	12	1	19
2			11	11	7	8
3			47	45	40	25
4			51	52	34	37
5			57	51	56	8
6			31	22	30	10
7			23	21	20	13
8			12	10	10	6
9			7	2	7	1
10			12	7	0	12
11			10	7	2	10
12			17	14	1	17
13			16	12	12	9
14			4	7	1	4
0	8	2	21	15	21	0
1			28	24	11	26
2			31	29	5	31
3			28	26	26	10
4			42	39	13	40
5			24	25	15	19
6			41	36	17	37
7			7	6	7	0
8			17	12	6	15
9			7	4	1	7
10			7	3	7	0
11			18	13	14	10
12			18	17	11	13
13			20	16	10	17
14			13	11	0	13
0	9	2	7	5	0	7
1			21	14	21	5
2			10	7	1	10
3			39	37	39	6
4			39	38	37	12
5			36	31	36	5
6			29	26	29	4
7			43	36	40	15
8			29	26	28	7
9			22	17	22	2
10			6	5	3	5
11			16	13	9	13
12			7	4	2	7
13			15	9	11	10
0	10	2	7	4	7	0
1			28	23	19	21
2			29	27	22	19
3			22	22	6	22
4			31	29	0	31
5			21	20	14	15
6			35	34	20	29
7			24	24	18	16
8			21	20	6	20
9			5	2	3	4
10			4	3	2	4
11			8	10	1	9
0	11	2	16	11	0	16
1			23	20	3	23
2			10	11	10	1
3			28	28	28	2
4			22	18	22	4
5			29	26	27	11
6			23	22	21	11
7			22	18	18	13
8			11	7	9	6
9			9	9	8	3
10			4	7	4	1
11			4	5	2	4

H	K	L	$\lvert F_o \rvert$	$\lvert F_c \rvert$	A_c	B_c
0	12	2	13	8	13	0
1			20	15	19	5
2			20	11	4	19
3			21	19	20	5
4			23	24	1	23
5			11	9	2	11
6			25	28	1	25
7			12	12	1	12
8			9	8	3	8
9			16	12	15	3
0	13	2	17	11	0	17
1			16	16	14	6
2			8	8	7	2
3			16	16	16	0
4			16	16	13	9
5			16	19	15	2
6			13	11	11	7
7			21	18	20	5
0	14	2	4	4	4	0
1			11	9	10	2
2			8	8	3	8
3			15	17	8	12
4			14	15	9	10
1	0	3	39	44	0	39
2			75	103	0	75
3			26	32	0	26
4			65	70	0	65
5			19	6	0	19
6			69	72	0	69
7			27	22	0	27
8			45	46	0	45
10			17	16	0	17
11			23	25	0	23
12			6	1	0	6
13			21	16	0	21
14			22	23	0	22
15			4	6	0	4
16			9	10	0	9
0	1	3	27	31	0	27
1			20	23	19	3
2			33	31	1	33
3			47	53	47	6
4			28	25	25	13
5			68	81	68	3
6			27	28	27	6
7			57	73	49	29
8			9	8	2	9
9			21	18	18	12
10			25	23	18	18
11			15	13	12	9
12			18	14	15	8
13			8	6	7	2
14			5	5	3	3
15			20	19	20	1
16			7	6	7	2
0	2	3	53	55	53	0
1			38	41	38	1
2			45	50	5	45
3			38	32	35	15
4			65	75	1	65
5			26	24	4	26
6			53	58	6	52
7			17	15	12	11
8			33	38	6	32
9			25	23	22	12
10			21	20	1	20
11			12	9	2	12
12			11	16	2	11
13			11	10	7	8
14			5	8	1	5
15			3	2	2	2
16			3	3	2	2
0	3	3	64	70	0	64
1			43	37	42	8
2			17	14	10	13
3			46	50	45	11
4			30	26	28	12
5			62	75	46	40
6			8	3	4	7
7			36	36	36	2
8			19	19	3	18
9			17	17	0	17
10			24	23	14	20
11			13	13	2	12
12			5	6	1	5
13			16	16	16	2
14			5	8	5	2
15			12	15	11	3
16			3	2	2	2
0	4	3	39	36	39	0
1			31	32	25	19
2			22	20	3	22
3			52	56	50	12
4			79	93	50	61
5			13	9	12	4
6			27	24	12	24
7			23	22	15	18
8			34	36	7	34
9			32	33	29	14
10			19	17	16	9

H	K	L	$\lvert F_o \rvert$	$\lvert F_c \rvert$	A_c	B_c
11	6	3	14	16	13	3
12			20	23	10	17
13			18	20	15	9
14			4	8	1	6
15			7	7	6	3
16			11	22	2	11
0	5	3	40	37	0	40
1			9	1	7	5
2			50	50	10	49
3			42	38	35	23
4			31	25	3	31
5			49	52	49	3
6			34	31	1	34
7			50	53	46	20
8			27	21	17	21
9			17	12	14	8
10			10	12	1	10
11			20	16	18	6
12			21	23	8	20
13			10	12	10	1
14			5	4	4	3
15			3	5	3	0
0	6	3	39	28	39	0
1			57	62	56	5
2			18	16	16	7
3			42	43	41	11
4			46	47	5	46
5			25	19	11	23
6			21	18	14	15
7			19	15	17	7
8			31	29	31	7
9			28	20	26	10
10			15	12	12	8
11			31	25	26	18
12			5	6	4	3
13			10	7	8	6
14			12	9	3	11
15			9	9	6	6
0	7	3	57	55	0	57
1			44	43	31	31
2			44	45	7	43
3			25	25	23	10
4			21	16	1	21
5			22	20	22	0
6			18	19	17	4
7			36	35	35	8
8			35	30	4	35
9			20	13	20	1
10			37	38	6	37
11			9	11	9	1
12			13	13	6	11
13			10	5	8	6
14			12	10	11	5
0	8	3	9	2	9	0
1			49	48	49	0
2			24	19	9	23
3			26	20	13	23
4			14	12	14	2
5			23	23	9	22
6			31	29	9	30
7			32	28	30	11
8			36	29	5	36
9			32	26	30	12
10			5	4	0	5
11			28	24	28	1
12			9	10	9	0
13			15	14	12	8
0	9	3	22	21	0	22
1			25	25	20	16
2			53	53	10	52
3			12	9	1	12
4			15	11	14	4
5			12	10	7	9
6			19	13	2	18
7			14	11	12	7
8			25	22	10	23
9			21	16	17	12
10			17	18	3	16
11			13	11	8	10
12			15	12	5	14
13			2	3	2	1
0	10	3	29	26	29	0
1			41	40	41	5
2			21	16	1	21
3			17	14	15	7
4			19	16	7	17
5			16	14	16	0
6			7	7	5	5
7			23	24	23	3
8			5	2	4	3
9			16	18	15	4
10			10	6	3	9
11			16	15	16	1
0	11	3	37	35	0	37
1			17	14	9	14
2			25	24	4	25
3			12	11	9	8
4			17	15	6	15
5			6	2	4	4
6			9	8	5	7

H	K	L	$\lvert F_o \rvert$	$\lvert F_c \rvert$	A_c	B_c
7	11	3	7	6	6	3
8			17	17	1	17
9			4	3	4	1
10			24	30	4	24
0	12	3	9	9	9	0
1			32	36	32	6
2			9	10	2	9
3			8	13	8	0
4			10	6	7	6
5			11	6	7	8
6			5	6	0	5
7			4	7	4	1
8			16	10	14	7
9			15	14	15	1
0	13	3	18	21	0	18
1			8	3	4	6
2			22	26	6	22
3			8	7	7	2
4			21	21	4	20
5			16	17	16	2
6			13	13	12	5
7			3	7	3	1
0	14	3	14	14	14	0
1			15	16	14	5
2			8	10	4	7
3			15	20	14	5
0	0	4	17	18	17	0
1			74	88	74	0
2			43	41	43	0
3			42	44	43	0
4			7	1	7	0
5			9	7	9	0
6			29	20	29	0
7			8	2	8	0
8			32	28	32	0
9			32	33	32	0
11			28	22	28	0
12			19	13	19	0
13			5	3	5	0
14			8	10	8	0
15			14	11	14	0
0	1	4	58	76	0	58
1			36	37	10	35
2			30	27	1	30
3			45	47	11	43
4			13	9	7	11
5			19	15	15	10
6			19	12	1	19
7			20	18	16	11
8			50	40	0	50
9			20	15	10	17
10			56	43	37	42
11			17	9	2	17
12			26	23	11	24
13			15	13	4	14
14			15	14	10	11
15			4	5	1	4
16			3	2	2	2
0	2	4	18	25	18	0
1			56	70	45	33
2			21	18	20	5
3			41	47	40	8
4			33	31	8	32
5			20	13	0	20
6			4	4	2	3
7			22	22	22	0
8			5	9	2	4
9			28	34	28	1
10			17	15	9	14
11			21	26	20	8
12			11	13	11	1
13			12	9	11	3
14			4	5	4	2
15			3	7	1	3
0	3	4	32	45	0	32
1			36	35	16	32
2			78	93	33	71
3			38	44	38	2
4			38	35	28	26
5			32	35	17	27
6			31	26	29	12
7			30	32	29	9
8			22	22	9	20
9			24	25	17	17
10			13	14	9	9
11			15	16	12	9
12			26	31	14	23
13			10	11	3	9
14			5	7	2	4
15			4	3	4	1
0	4	4	64	65	64	0
1			52	52	48	19
2			35	33	33	12
3			52	60	41	32
4			18	16	16	6
5			26	25	13	23
6			24	29	1	24
7			30	30	25	18
8			16	15	15	2
9			13	14	11	7
10			27	27	24	12

H	K	L	$\lvert F_o \rvert$	$\lvert F_c \rvert$	A_c	B_c
11	4	4	23	24	23	1
12			21	21	0	21
13			18	19	17	5
14			5	8	4	3
15			5	7	2	5
0	5	4	62	72	0	62
1			47	47	25	39
2			29	25	25	16
3			37	38	37	1
4			31	29	6	31
5			23	25	22	8
6			11	11	8	8
7			20	19	14	14
8			15	11	10	10
9			20	18	18	7
10			32	32	12	30
11			10	9	10	1
12			8	11	3	7
13			16	18	7	14
14			14	14	9	10
15			5	8	4	3
0	6	4	26	28	26	0
1			37	36	35	11
2			20	20	18	7
3			7	13	3	6
4			44	39	18	40
5			23	21	5	23
6			32	37	1	32
7			21	18	7	20
8			7	10	7	2
9			28	24	28	5
10			8	10	6	5
11			15	16	11	10
12			4	2	3	2
13			4	5	3	2
14			15	19	4	14
0	7	4	38	33	0	38
1			10	5	2	10
2			51	49	42	29
3			31	30	30	7
4			32	27	32	5
5			36	35	36	7
6			23	18	16	18
7			7	8	7	2
8			21	20	17	13
9			5	10	4	2
10			6	2	5	33
11			7	8	3	6
12			17	11	8	14
13			21	20	20	4
14			2	7	2	0
0	8	4	6	1	6	0
1			18	15	5	17
2			25	29	19	17
3			30	27	26	15
4			38	34	2	38
5			35	39	10	34
6			33	38	6	32
7			25	22	3	25
8			20	20	2	19
9			10	12	10	1
10			6	6	6	0
11			9	9	9	2
12			6	4	5	3
13			11	11	10	4
0	9	4	8	10	0	8
1			35	30	35	7
2			10	11	8	6
3			25	28	25	4
4			26	27	26	7
5			15	17	14	3
6			5	3	4	3
7			23	23	23	2
8			9	14	9	0
9			4	8	4	1
10			4	6	2	3
11			14	10	13	5
12			9	8	5	7
0	10	4	26	23	26	0
1			17	14	16	6
2			21	21	1	21
3			8	9	3	7
4			29	31	2	29
5			21	21	11	18
6			25	30	7	24
7			16	15	6	15
8			27	29	5	27
9			11	10	5	10
10			8	6	8	2
11			11	14	2	10
0	11	4	9	1	0	9
1			7	3	5	5
2			9	10	9	2
3			20	20	19	6
4			20	22	19	4
5			23	27	22	7
6			13	11	8	10
7			13	14	8	10
8			10	12	10	1
9			10	12	6	8

projecting from the same side of the carbon ring (C_1–C_6) but slightly divergent from their points of attachment. The maximum atomic distance from the best lactone planes are 0·10 Å and 0·03 Å, and the angle between the lactone plane normals is 17° 30′.

Together the two lactone bridges form a cage surrounding C_{12} on the reverse side from the epoxide O_2. In Table 3 are listed some intramolecular distances giving the approximate size of the cage, e.g., distance $C_{12} \cdots C_{15}$ is 3·18 Å. It is of great significance in explaining the unusual chemical properties of picrotoxinin that there should be steric hindrance to a rearward nucleophilic attack at the epoxide ring (Conroy, 1957). The inertness of this epoxide ring

Table 6 (*cont.*)

H	K	L	$\|F_o\|$	$\|F_c\|$	A_o	B_o
0	12	4	6	2	6-	0
1			10	10	2	10
2			10	13	6-	8
3			11	9	2	10-
4			21	23	2	21 -
5			10	11	6 -	8
6			20	20	4	19
7			13	17	1	13 -
0	13	4	13	11	0	13 -
1			8	4	7 -	4
2			3	2	3 -	0 -
3			18	19	18 -	0
4			4	5	3	2
5			10	14	9	3 -
1	0	5	12	9	0	12
2			13	5	0	13
3			35	28	0	35 -
4			35	34	0	35 -
5			63	67	0	63
6			55	48	0	55
7			30	24	0	30 -
8			6	2	0	6
9			34	26	0	34
10			14	9	9	14
11			17	16	0	17 -
12			6	2	0	6 -
13			14	14	0	14 -
14			9	8	0	9 -
15			3	9	0	3
0	1	5	12	6	0	12 -
1			12	13	4	11 -
2			35	30	31 -	16
3			38	37	25 -	28
4			35	52	35	1 -
5			37	34	25	28 -
6			55	48	55 -	2
7			31	29	3 -	31
8			29	23	29	1 -
9			28	20	0	28 -
10			13	9	12 -	4 -
11			13	11	9	9
12			6	1	5 -	3
13			21	19	18 -	10 -
14			15	13	12	8 -
15			8	6	7	2 -
0	2	5	22	17	22	0
1			44	40	26	36
2			57	54	49 -	29
3			52	46	15	49 -
4			35	32	19	30 -
5			43	44	20	38
6			33	29	7 -	32
7			35	28	10	34 -
8			22	19	4	22 -
9			16	12	4	14
10			6	6	2	5
11			21	20	16	13
12			5	3	2 -	5 -
13			7	11	1 -	7 -
14			8	11	4 -	7 -
15			13	15	4 -	12
0	3	5	29	29	0	29
1			18	13	17	2
2			36	34	27 -	25
3			31	26	28 -	14 -
4			42	35	42	2 -
5			19	15	17	6
6			37	34	36 -	7 -
7			42	41	36 -	22
8			40	39	35	20
9			19	16	18	2
10			7	7	6	2
11			13	13	9	9
12			5	4	2 -	5 -
13			4	6	2 -	3 -
14			12	7	12 -	2 -
15			9	9	1 -	9 -
0	4	5	57	46	57	0
1			27	28	27	1 -
2			32	24	14 -	29
3			30	31	3	30 -
4			28	21	12	26 -
5			29	21	15 -	25
6			24	21	22	11
7			29	24	11	27 -
8			21	17	8 -	19 -
9			15	14	9	11
10			26	25	24	11 -
11			15	14	14	4 -
12			8	9	8 -	1
13			6	6	1 -	6 -
14			10	13	9	3 -
0	5	5	8	2	0	8
1			53	38	6	52
2			26	24	20 -	16
3			26	23	23 -	13 -
4			34	26	14	31 -
5			43	39	43	4
6			26	25	26 -	6 -
7			20	16	1 -	20
8			21	17	2	20
9			38	38	17	34 -
10	6	5	9	8	6 -	6 -
11			16	14	3	15
12			10	10	10 -	0
13			4	6	4	0
14			4	6	2	3 -
0	6	5	64	56	64	0
1			25	17	25	3
2			68	57	68	7
3			34	26	5	34 -
4			24	15	18	16 -
5			31	27	16 -	27
6			32	30	22 -	23
7			29	25	15 -	25 -
8			21	19	20 -	4 -
9			34	30	27 -	21
10			13	12	10	8
11			10	14	10	2
12			7	10	6 -	3
13			4	2	4 -	1
0	7	5	40	26	0	40
1			39	28	21 -	33
2			18	11	18 -	1
3			37	31	19 -	32 -
4			13	14	12	4 -
5			20	17	17	9 -
6			28	29	27 -	8 -
7			24	18	19 -	15
8			21	20	10	19
9			19	22	10	16 -
10			11	13	10 -	5 -
11			12	14	0 -	12
12			8	9	6	6 -
0	8	5	21	21	21	0
1			35	28	32	15 -
2			35	27	12 -	33
3			20	17	16 -	12 -
4			12	11	1	12
5			15	14	10 -	11
6			23	19	21	10
7			6	2	6 -	0 -
8			19	19	18 -	6
9			16	13	8 -	13
10			17	20	17	1 -
11			10	12	6	8 -
12			11	18	10 -	2
0	9	5	12	10	0	12 -
1			49	41	3 -	49
2			21	16	1	21
3			33	34	9 -	32 -
4			28	23	6 -	28 -
5			28	19	17	23
6			12	10	6 -	10 -
7			15	13	11 -	9
8			15	16	3	14
9			11	12	5	9 -
10			8	10	5 -	6
11			10	13	1 -	10
0	10	5	32	28	32	0
1			13	13	11	6
2			19	13	18 -	6
3			9	10	7 -	5
4			12	6	7	10 -
5			8	7	8 -	1 -
6			15	17	11	10
7			15	13	12	8 -
8			16	19	14 -	6 -
9			6	7	3 -	5
10			11	15	9	6
0	11	5	25	28	0	25 -
1			20	16	3 -	19
2			11	9	1 -	11
3			15	17	2 -	15 -
4			11	9	6 -	9 -
5			7	4	4	5 -
6			4	9	4	1
7			4	8	0	4
8			9	9	2	9
0	12	5	20	23	20	0
1			17	13	16	4 -
2			13	14	13 -	0
3			18	17	8 -	16
4			12	13	11	5 -
0	13	5	14	11	0	14 -
1			19	23	1	19
2			9	12	8	4
3			10	15	2	10 -
0	0	6	55	66	55	0
1			21	19	21 -	0
2			31	28	31 -	0
3			10	4	10 -	0
4			13	13	13	0
5			25	23	25 -	0
6			5	3	5 -	0
7			11	12	11 -	0
8			38	36	38 -	0
9			33	30	33 -	0
10			24	36	24	0
11			9	3	9	0
12			17	14	17 -	0
13			8	6	8 -	0
14			8	12	8	0
0	1	6	24	23	0	24 -
1			61	63	9	60
2			29	21	15 -	25
3			46	37	2	46 -
4			29	25	21 -	20 -
5			13	7	12 -	3 -
6			8	5	8 -	2
7			27	26	27 -	2 -
8			14	11	0 -	14 -
9			34	29	3	34 -
10			5	6	4 -	3
11			25	24	1	25
12			8	7	7	3
13			11	8	3	10 -
14			8	6	8 -	1
0	2	6	45	37	45	0
1			21	17	5 -	21 -
2			48	41	36 -	32
3			20	15	2	19
4			35	33	34	9
5			21	16	11 -	18 -
6			31	26	31	3
7			14	11	10	10
8			37	34	33 -	17 -
9			3	4	2	2 -
10			21	24	21	3
11			12	7	11 -	4
12			16	18	14 -	6 -
13			2	6	2	1
14			3	8	3	1
0	3	6	6	4	0	6
1			58	55	3	58
2			27	18	18	21 -
3			39	33	7	39 -
4			16	14	16 -	0 -
5			9	8	9	0
6			21	15	9	19 -
7			16	14	6 -	14
8			14	15	13 -	3
9			17	17	14 -	8 -
10			12	13	11 -	5 -
11			17	22	5 -	16
12			3	1	2	2
13			9	11	1	8 -
14			3	5	3 -	1
0	4	6	38	30	38	0
1			22	19	12	18 -
2			39	33	39 -	2 -
3			21	20	17	13
4			18	12	12	13 -
5			27	31	19	20 -
6			7	7	6 -	3 -
7			20	18	13	15
8			23	23	23 -	1
9			14	15	3 -	13 -
10			20	18	16	12
11			13	13	2	13
12			10	14	9 -	4
13			5	7	2 -	5
0	5	6	7	7	0	7
1			25	21	12 -	22
2			25	20	21	13
3			15	14	10	10 -
4			42	42	33 -	26
5			19	18	18	4
6			35	36	34	10
7			12	15	2	11
8			11	7	2 -	11
9			13	17	2 -	13 -
10			8	6	7	4 -
11			17	22	0 -	17
12			3	2	3	0 -
13			5	5	3 -	4 -
0	6	6	43	47	43	0
1			14	11	14	1
2			42	37	42 -	4
3			42	41	3 -	42
4			23	20	21	9 -
5			25	27	11	23 -
6			14	21	4 -	13 -
0	7	6	11	10	0	11
1			12	14	8 -	8
2			22	19	21	8
3			18	15	4	17 -
4			18	16	17 -	3
5			8	9	2	8 -
6			30	36	27	13 -
0	8	6	22	23	22	0
1			21	20	17	11 -
2			12	12	1	12
3			32	34	24 -	22
4			9	11	8	4
5			21	27	5	20 -
6			9	9	9 -	1
0	9	6	10	2	0	11
1			9	6	1 -	9
2			21	22	21	6
3			11	16	1 -	11 -
4			28	31	28 -	2 -
5			12	11	11	5
6			19	22	18	3 -
0	10	6	8	4	8	0
1			10	5	1	10 -
2			3	7	2 -	2
3			20	20	5	19
4			6	12	1 -	6
5			17	22	2 -	16 -
6			8	12	1 -	8
0	11	6	18	18	0	18
1			7	10	4 -	5 -
2			18	17	17	5
3			4	2	2 -	4 -
4			15	17	15 -	1 -
5			7	6	4 -	6
6			14	19	13	3
0	12	6	4	1	4	0
1			7	4	4	6 -
2			4	8	4	1
3			12	16	5	11
4			5	4	1	5
1	0	7	8	6	0	8 -
2			17	11	0	17
3			57	59	0	57
4			18	11	0	18 -
5			41	23	0	41 -
6			26	31	0	26
7			29	25	0	29
8			30	23	0	30 -
9			5	4	0	5 -
10			5	0	0	5 -
11			8	5	0	8 -
12			4	0	0	4 -
13			11	10	0	11
0	1	7	15	8	0	15
1			8	6	2 -	8 -
2			27	17	26	10 -
3			39	34	33 -	21 -
4			31	23	31 -	4
5			26	21	8	25 -
6			49	45	41	27 -
7			15	8	8 -	12
8			13	5	12 -	4 -
9			15	16	14	3
10			4	5	2 -	3 -
11			4	7	3	3 -
12			14	12	12	7
13			7	5	4 -	5 -
0	2	7	27	22	27 -	0
1			20	20	2 -	19 -
2			5	8	5	1
3			31	26	24 -	20
4			21	12	4 -	21 -
5			15	13	5 -	18 -
6			21	21	6	20
7			24	25	2 -	24
8			10	12	6	7
9			12	16	3 -	11 -
10			7	4	7 -	0
11			3	4	2	2 -
12			4	2	3 -	3
13			5	8	4	3
0	3	7	35	33	0	35 -
1			35	33	1	35 -
2			43	35	15	40
3			24	20	18 -	16
4			40	39	38 -	12 -
5			21	17	20	2
6			28	32	20	20
7			19	16	16 -	10 -
8			19	16	19 -	2
9			15	15	12	8
10			8	10	8	0
11			9	12	7 -	6 -
12			5	5	3	4
13			2	5	2 -	1
0	4	7	5	2	5	0
1			21	20	10 -	19 -
2			30	23	26	16
3			36	29	20	30
4			6	9	6 -	2 -
5			45	50	20 -	40 -
6			21	23	10 -	18
7			21	25	14	16
8			23	24	21	11 -
9			21	24	19 -	7 -
10			3	5	3 -	1 -
11			11	13	9	6
12			8	11	7	4 -
0	5	7	35	34	0	35 -
1			21	21	20	9 -
2			20	13	1	19
3			6	6	0	6
4			43	44	40 -	14 -
5			12	14	5	11
6			14	13	13	4
7			5	5	3	4
8			18	22	18 -	1
9			9	12	4	8
10			4	1	4 -	1 -
11			8	9	4	7 -
12			5	8	1	5
0	6	7	29	20	29 -	0
1			22	21	19	12 -
2			9	6	9	1 -
3			25	22	25 -	4
4			12	15	11 -	5 -
5			14	16	14	8 -
6			11	15	10 -	5
0	7	7	25	20	0	25 -
1			27	27	8 -	26 -
2			19	14	18	6 -
3			13	14	3 -	12
4			4	5	2 -	3
5			4	5	3	3
6			15	16	6	13 -
0	8	7	23	24	23 -	0
1			19	17	17	7 -
2			21	22	21	0
3			18	16	17 -	3
4			7	7	6 -	3 -
5			13	15	5	11 -
6			11	13	9 -	5 -
0	9	7	6	8	0	6 -
1			23	27	6 -	23 -
2			38	19	11	14
3			10	6	7	6
4			6	7	6 -	1 -
5			3	9	1	3 -
6			8	7	6	5
0	10	7	15	13	15 -	0
1			14	14	12	6
2			21	24	21	1 -
3			13	14	12 -	3
4			6	10	4 -	4
5			3	1	2	2 -
6			6	6	5	3 -
0	11	7	4	6	0	4 -
1			19	19	5 -	18 -
2			6	8	6 -	0 -
3			9	9	3	8
0	0	8	19	4	19 -	0
1			34	35	34	0
2			11	2	11 -	0
3			37	31	37 -	0
4			7	3	7 -	0
5			5	6	5	0
6			7	3	7 -	0
7			5	1	5	0
8			14	7	14	0
9			13	8	13 -	0
10			19	19	19 -	0
11			15	17	15	0
12			4	3	4	0
0	1	8	28	17	0	28 -
1			30	29	21 -	22 -
2			28	22	2 -	28
3			8	4	8 -	2
4			32	34	24	21 -
5			5	7	4 -	3 -
6			16	16	4 -	15 -
7			13	11	8 -	10 -
8			18	16	5	17
9			11	7	3	10
10			14	10	2 -	14 -
11			11	9	6 -	0 -
12			10	11	5	9
0	2	8	31	22	31 -	0
1			41	43	41	5
2			51	48	51	0 -
3			4	5	0 -	4
4			15	14	12 -	9
5			4	5	4	2
6			8	9	1 -	8 -
7			4	2	3	3
8			4	6	4	0 -
9			14	15	10 -	9 -
10			2	2	2 -	0 -
11			8	11	8	1
12			4	5	3	3 -
0	3	8	20	11	0	20 -
1			6	3	1 -	7
2			20	19	7	18
3			6	2	4	5 -
4			9	9	1	9
5			17	14	16 -	5
6			4	4	4	1 -
7			19	21	1	19 -
8			11	16	4	10
9			13	16	3	12
10			12	19	1	12 -
11			9	12	0 -	8 -
0	4	8	9	3	9 -	0
1			20	20	15	12
2			13	10	2	13 -
3			11	7	3 -	10 -
4			24	23	3 -	24
5			6	2	1 -	6 -
6			9	13	4 -	8 -
7			11	10	7	8 -
8			7	10	1 -	7
9			7	9	7 -	0

has proved a stumbling block for chemical workers for many years.

The intermolecular approaches are normal although the distance 3·37 Å between Br(IV) and O_2(II) is of interest. (These particular atoms are chosen since they are conveniently near the centre of Fig. 4.) The approach is close to the sum of the van der Waals radii for bromine and oxygen atoms (3·35 Å) given by Pauling (1960). The atoms are also close to making tetrahedral angles, e.g. the angle C_9(IV)–Br(IV) $\cdots$ O_2(II) is 117° and the line from Br(IV) to the mid-point of C_{12}(II)–C_{13}(II) makes an angle of 83° with the bond C_{12}(II)–C_{13}(II). It is possible that there is an extremely weak intermolecular bonding interaction.

The determination of the absolute configuration of α_1-bromopicrotoxinin establishes the absolute con-

Table 6 (*cont.*)

H	K	L	$\|F_o\|$	$\|F_c\|$	A_o	B_o
10	4	8	10	13	9 -	3 -
11			9	13	8	4 -
0	5	8	12	6	0	12 -
1			34	31	10 -	33 -
2			26	28	6 -	26
3			22	23	17	15
4			14	15	14	2 -
5			18	17	10 -	15 -
6			10	12	10 -	1
7			19	21	17	8
8			4	4	3	3
9			4	6	4 -	0 -
10			7	9	3	6 -
0	6	8	21	15	21 -	0
1			12	10	12	5
2			18	15	2 -	18 -
3			9	8	5 -	7 -
4			17	17	1 -	17
5			12	13	5	11
6			18	22	11 -	14 -
0	7	8	17	15	0	17 -
1			14	11	6 -	12 -
2			15	14	1 -	15
3			21	22	17	13
4			13	12	9	9 -
5			17	19	16 -	4
6			5	6	4 -	3
0	8	8	13	11	13 -	0
1			5	4	4	3
2			17	13	14	10
3			8	9	5 -	6 -
4			19	19	6 -	18
5			3	6	2	2
6			10	12	5	8 -
0	9	8	13	11	0	13 -
1			9	2	3 -	8 -
2			8	6	8 -	2 -
3			15	13	13	8
4			10	11	8	6 -
5			11	14	10 -	3 -
6			4	8	3 -	2

H	K	L	$\|F_o\|$	$\|F_c\|$	A_o	B_o
0	10	8	7	4	7 -	0
1			11	12	5 -	10
2			8	9	1	8 -
3			9	11	0	9 -
1	0	9	10	10	0	10 -
2			18	7	0	18 -
3			5	3	0	5
4			21	22	0	21
5			21	23	0	21
6			12	12	0	12 -
7			6	7	0	6
8			4	3	0	4
9			9	13	0	9
10			11	12	0	11
0	1	9	16	14	0	16 -
1			16	16	16 -	1 -
2			21	20	0 -	20 -
3			26	24	26	0
4			6	3	1	6
5			22	21	22 -	6 -
6			6	4	5 -	2 -
7			20	18	19	6 -
8			5	4	5 -	1 -
9			15	16	15 -	2
10			5	7	3 -	4
0	2	9	12	10	12 -	0
1			9	10	7 -	6 -
2			11	12	10	3
3			5	7	3	4 -
4			23	28	2 -	23
5			5	5	2	5 -
6			18	22	6	17 -
7			3	3	3 -	0
8			6	13	1	6
9			2	5	2	1 -
10			3	6	3	1
0	3	9	19	13	0	19
1			14	17	14 -	2 -
2			10	14	5 -	8 -
3			17	19	15	6 -
4			7	9	7 -	0

H	K	L	$\|F_o\|$	$\|F_c\|$	A_o	B_o
5	3	9	15	15	14 -	3 -
6			5	6	0 -	5 -
7			11	15	11	0 -
8			4	6	4	0 -
9			3	5	3 -	0 -
10			7	13	4	6
0	4	9	5	5	5	0
1			17	16	16 -	5
2			17	15	7	15 -
3			8	5	7	2 -
4			14	15	0 -	14
5			5	7	5 -	0
6			16	21	4	15 -
7			6	8	5 -	2 -
8			8	12	0	8
9			4	9	4	2 -
0	5	9	12	11	0	12
1			9	9	8 -	3 -
2			10	11	4 -	9 -
3			11	10	10	4 -
4			7	5	4	5
5			9	9	9 -	0 -
6			10	10	2 -	10
7			10	14	9	3 -
8			10	12	5 -	8 -
0	6	9	5	7	5	0
1			18	17	16 -	6 -
2			13	10	9	9 -
3			8	7	8	1 -
4			13	13	1 -	13
5			5	7	2 -	5
6			13	18	6 -	11 -
0	7	9	21	21	0	21
1			9	8	6	6
2			14	15	1 -	14 -
3			5	7	4	2
4			3	6	2	2
5			7	7	6 -	3 -
6			5	8	5 -	1 -
0	8	9	4	6	4 -	0
1			14	15	14 -	1

H	K	L	$\|F_o\|$	$\|F_c\|$	A_o	B_o
2	8	9	6	7	4 -	4 -
3			13	15	6	11 -
4			5	7	4 -	3
5			3	3	1	3
0	9	9	16	17	0	16
1			9	12	4	8 -
0	0	10	5	1	5 -	0
1			16	14	16 -	0
2			12	10	12	0
3			4	2	4	0
4			16	15	16 -	0
5			8	11	8	0
6			3	1	3	0
7			11	8	11 -	0
8			4	1	4 -	0
0	1	10	24	23	0	24
1			14	14	1 -	14 -
2			15	8	10 -	11 -
3			5	4	5 -	1
4			9	12	5	7
5			5	7	5 -	1 -
6			5	4	1 -	5 -
7			12	12	12 -	1 -
8			14	20	2	14 -
0	2	10	6	2	6 -	0
1			19	20	19 -	0 -
2			12	18	9	8
3			13	15	12	4 -
4			4	3	3 -	3 -
5			3	6	0	3 -
6			12	10	5 -	11
7			2	5	2	1 -
8			3	9	3	1 -
0	3	10	14	12	0	14
1			8	9	1	8 -
2			13	13	0 -	13 -
3			4	4	4 -	1 -
4			4	5	1	4
5			5	6	5	1 -
6			11	12	5 -	9
7			2	3	1 -	1

H	K	L	$\|F_o\|$	$\|F_c\|$	A_o	B_o
0	4	10	8	4	8 -	0
1			14	13	13 -	4
2			7	7	0	7
3			12	16	12	2 -
4			6	7	5 -	3 -
5			8	7	4	7
6			7	15	1	7
0	5	10	15	11	0	15
1			3	6	3 -	1 -
2			15	18	1 -	15 -
3			8	11	8 -	1
4			16	19	12	10
5			3	4	3	0 -
6			6	11	5 -	2 -
0	6	10	7	0	7 -	0
1			11	12	9 -	5
2			7	9	2	7
3			6	7	0	6 -
4			9	10	3 -	8 -
0	7	10	13	13	0	13
1			7	8	4 -	5 -
1	0	11	10	9	0	10
2			5	4	0	5
3			9	12	0	9 -
4			7	7	0	7 -
5			13	23	0	13
0	1	11	4	0	0	4 -
1			4	2	2 -	3 -
2			8	6	8 -	0
3			11	11	11 -	0 -
4			9	9	7	5
0	2	11	2	1	2 -	0
1			3	2	3	1
2			6	7	4	4
3			7	13	2	6 -
4			3	9	2	2 -
0	3	11	3	1	0	3 -
1			9	11	7	6
2			3	8	0	3
3			5	9	1 -	5 -
0	4	11	13	18	13	0

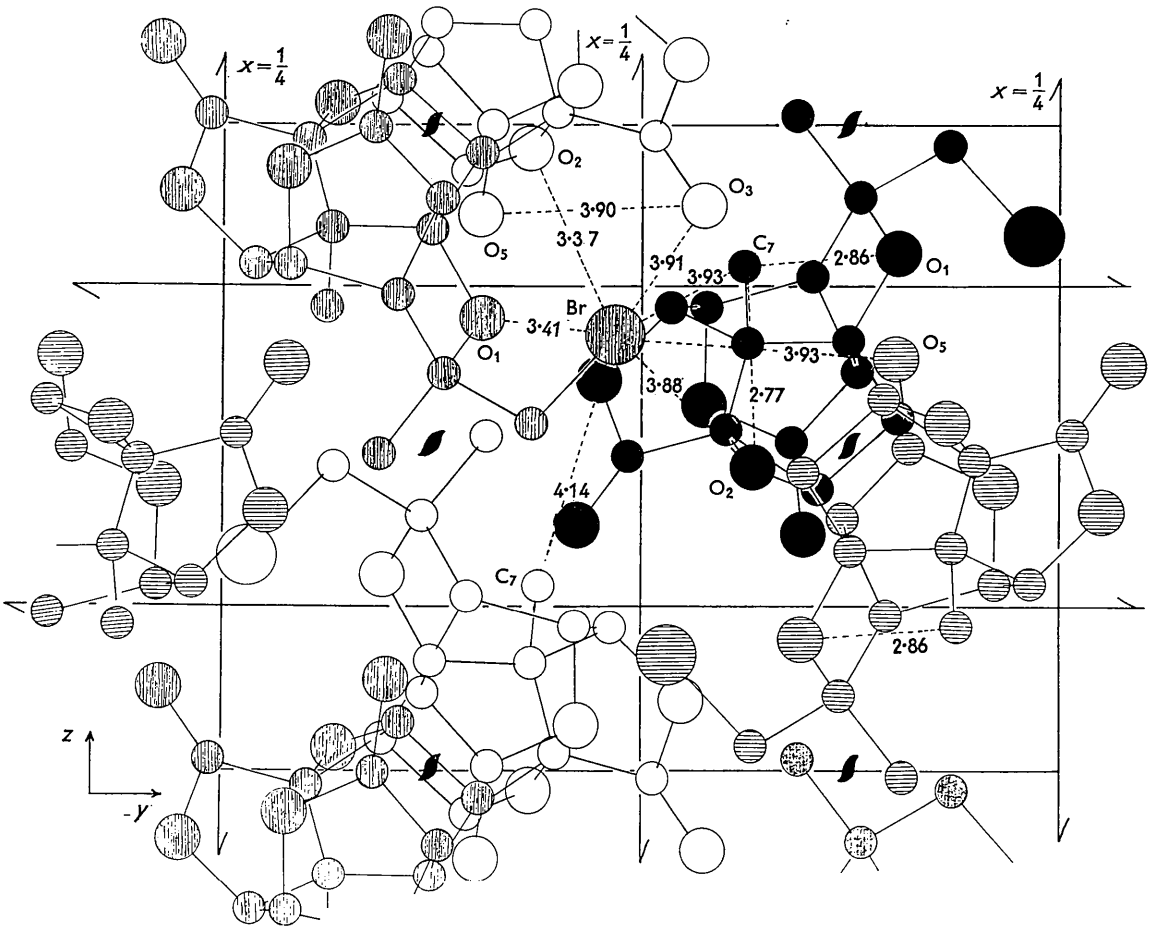
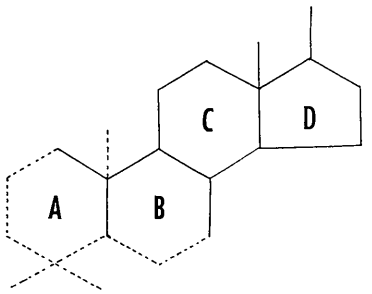
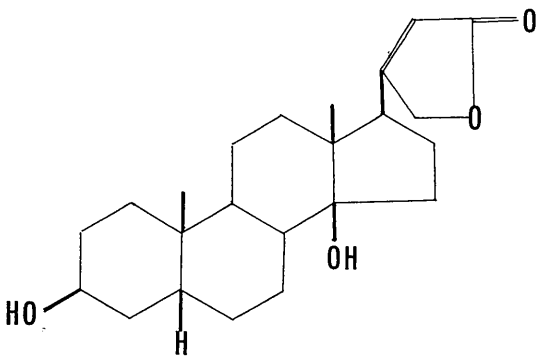


Fig. 4. The molecular packing of α_1-bromopicrotoxinin in the crystal structure. The heavily shaded molecule corresponds to the atomic coordinates listed in Table 1.

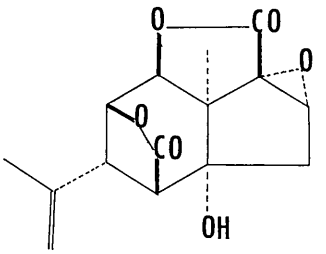
figuration of picrotoxinin itself as the mirror image of the structure (I) which is shown by arbitrary choice in Conroy's papers. The absolute configuration is of importance in any consideration of the biosynthesis of picrotoxinin, e.g., Conroy has pointed out the structural similarities between picrotoxinin and the steroid series. His conformational analysis and the



present X-ray analysis show that the carbon rings of picrotoxinin which correspond to the C and D rings of the steroids are *cis* fused, as in the cardiac glycosides and aglycones, e.g. digitoxigenin (III).



(III)



(IV)

The absolute configuration of picrotoxinin at the 'C/D' ring junction (IV) is now found to be of the *opposite* sense to the C/D junction in the *cis* fused steroids, which is known following the determination of absolute configuration in the steroid family by the chemical method of asymmetrical synthesis (Dauben *et al.*, 1953). Thus, when the evidence of absolute configuration is considered, a distinct dissimilarity in the carbon framework appears, implying that there is a considerable difference in the course of biogenesis in the two series.

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