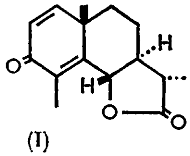
302. *Sesquiterpenoids. Part II.*[1] *The Stereochemistry of Isophoto-santonic Lactone: X-Ray Analysis of 2-Bromodihydroisophoto-α-santonic Lactone Acetate* [2]

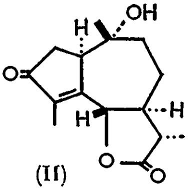
By J. D. M. ASHER and G. A. SIM

Crystals of 2-bromodihydroisophoto-α-santonic lactone acetate belong to the orthorhombic system, space group $P2_12_12_1$–D_2^4, with four molecules of $C_{17}H_{23}BrO_5$ in a unit cell of dimensions $a = 11{\cdot}05$, $b = 19{\cdot}23$, $c = 7{\cdot}93$ Å. A detailed *X*-ray study establishes the molecular stereochemistry shown in (III); it follows that isophoto-α-santonic lactone has the stereochemistry shown in (II). Phase determination was based initially on the bromine atom, and after the calculation of a number of three-dimensional electron-density distributions the atomic parameters were refined by the method of least squares. The final discrepancy, *R*, over the 865 observed reflexions is 12·9%.

THE photochemistry of the sesquiterpenoid lactone α-santonin (I) has been extensively investigated.[3] Irradiation of α-santonin in aqueous acetic acid yields isophoto-α-santonic lactone which was shown [4] to have the constitution (II). Much of the stereochemistry, however, was not established and has been the subject of controversy.[3,5]

Bromination of dihydroisophoto-α-santonic lactone acetate gives a bromo-derivative [6] whose crystal structure we have elucidated. Our results prove that this derivative has the constitution and relative stereochemistry shown in (III); it follows, therefore, that the stereochemistry of isophoto-α-santonic lactone is as in (II), the absolute configuration shown being firmly established chemically.[3]


(I)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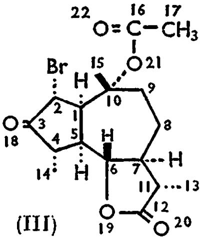
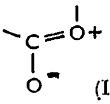

(III)


(IV)

The configuration at C(11), α-methyl, is opposite to that generally accepted hitherto for α-santonin,[7,8] and as epimerisation at C(11) has been shown not to occur in the conversion of α-santonin into 2-bromodihydroisophoto-α-santonic lactone [6] it follows that the configuration of α-santonin must be as in (I). Since our preliminary communication,[2] further proof of the revised configuration at position 11 in α-santonin has been provided by the degradation of α-santonin to (+)-benzoylalanine [9] and by our *X*-ray analysis of 2-bromo-α-santonin.[10]

The crystal structure of 2-bromodihydroisophoto-α-santonic lactone acetate was solved by means of the usual phase-determining heavy-atom method.[11] The arrangement of the

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[1] The Paper by Hamilton, McPhail, and Sim, *J.*, 1962, 708, is regarded as Part I.

[2] Preliminary communication, *Proc. Chem. Soc.*, 1962, 111.

[3] For reviews see Barton, *Proc. Chem. Soc.*, 1958, 61; Barton, *Helv. Chim. Acta*, 1959, **42**, 2604; de Mayo and Reid, *Quart. Rev.*, 1961, **15**, 393.

[4] Barton, de Mayo, and Shafiq, *J.*, 1957, 929.

[5] Djerassi, Osiecki, and Herz, *J. Org. Chem.*, 1957, **22**, 1361; Huffman, *Experientia*, 1960, **16**, 120.

[6] Barton, Miki, Pinhey, and Wells, *Proc. Chem. Soc.*, 1962, 112; Barton, Levisalles, and Pinhey, *J.*, 1962, 3472.

[7] See Cocker and McMurry, *Tetrahedron*, 1960, **8**, 181.

[8] Contrast Miki, *J. Pharm. Soc. Japan*, 1955, **75**, 416.

[9] Nakazaki and Arakawa, *Proc. Chem. Soc.*, 1962, 151.

[10] Asher and Sim, *Proc. Chem. Soc.*, 1962, 335; and unpublished work.

[11] Robertson and Woodward, *J.*, 1937, 219; 1940, 36; Sim, in "Computing Methods and the Phase Problem in X-Ray Crystal Analysis," ed. Pepinsky, Robertson, and Speakman, Pergamon, Oxford, 1961, p. 227.

bromine atoms in the unit cell is approximately centrosymmetrical, and spurious planes of symmetry in the early three-dimensional electron-density distributions made the location of atomic sites difficult. However, the eventual recognition in the electron-density distributions of known molecular features, such as the five- and seven-membered carbocyclic rings, and the incorporation of the corresponding atoms in the phasing calculations, enabled the troublesome pseudo-symmetry to be overcome in the later electron-density distributions.

Thirteen three-dimensional electron-density distributions were evaluated, during which the average discrepancy (*R*) between calculated and measured structure amplitudes dropped from 45·1 to 20·2%. The positional and thermal atomic parameters were then further refined by the method of least squares, and the value of *R* decreased to a final value of 12·9% over the 865 measured structure amplitudes.

The final electron-density distribution over one molecule is shown in Fig. 1 as superimposed contour sections drawn parallel to (001). The atomic arrangement corresponding to this electron-density distribution is explained in Fig. 2. The interatomic distances and valency angles calculated from the final atomic co-ordinates (see Table 1) are listed in Table 2.

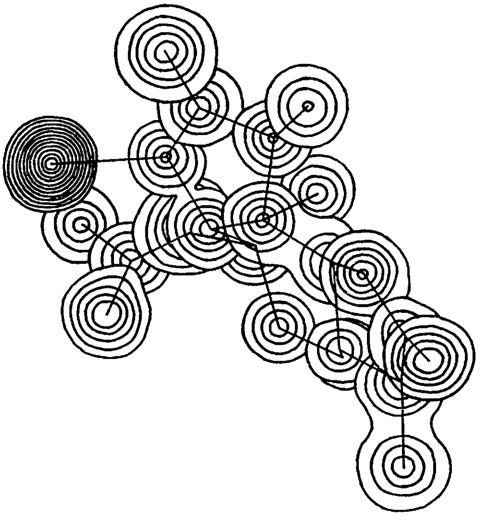

FIG. 1. The final three-dimensional electron-density distribution for bromodihydroisophoto-α-santonic lactone acetate shown by means of superimposed contour sections drawn parallel to (001) and covering the region of one molecule. Contour interval 1 eÅ^{-3}, except around the bromine atom where it is 3 eÅ^{-3}

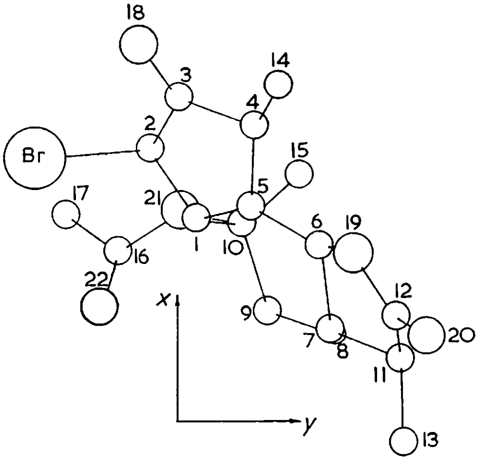

FIG. 2. The arrangement of atoms corresponding to Fig. 1

TABLE 1

Atomic co-ordinates

(Origin of co-ordinates as in " International Tables." *)

Atom	*x/a*	*y/b*	*z/c*	Atom	*x/a*	*y/b*	*z/c*	Atom	*x/a*	*y/b*	*z/c*
C(1)	−0·1819	0·2011	0·2631	C(9)	−0·0316	0·1378	0·4447	C(17)	−0·1712	0·3240	−0·2675
C(2)	−0·2895	0·2463	0·2763	C(10)	−0·1662	0·1655	0·4252	O(18)	−0·4549	0·2587	0·0461
C(3)	−0·3729	0·2225	0·1280	C(11)	0·0378	0·0155	0·0842	O(19)	−0·1291	0·0605	−0·0723
C(4)	−0·3373	0·1510	0·0764	C(12)	−0·0310	0·0175	−0·0866	O(20)	−0·0061	0·0056	−0·2318
C(5)	−0·2003	0·1534	0·1048	C(13)	0·1728	0·0094	0·0556	O(21)	−0·1841	0·2188	−0·4292
C(6)	−0·1416	0·0812	0·0943	C(14)	−0·3852	0·1280	−0·0883	O(22)	−0·0242	0·2911	−0·4832
C(7)	−0·0084	0·0781	0·1596	C(15)	−0·2572	0·1115	0·4872	Br	−0·2576	0·3462	0·2386
C(8)	0·0001	0·0731	0·3521	C(16)	−0·1140	0·2754	−0·4104				

* " International Tables for X-Ray Crystallography," The Kynoch Press, Birmingham, 1952, Vol. I.

TABLE 2

Interatomic distances (Å) and angles

Intramolecular bonded distances

C(1)–C(2)	1·48	C(4)–C(5)	1·53	C(7)–C(11)	1·44	C(11)–C(13)	1·51
C(1)–C(5)	1·57	C(4)–C(14)	1·48	C(8)–C(9)	1·49	C(12)–O(19)	1·37
C(1)–C(10)	1·47	C(5)–C(6)	1·53	C(9)–C(10)	1·59	C(12)–O(20)	1·26
C(2)–Br	1·98	C(6)–C(7)	1·56	C(10)–C(15)	1·53	C(16)–C(17)	1·60
C(2)–C(3)	1·56	C(6)–O(19)	1·39	C(10)–O(21)	1·56	C(16)–O(21)	1·34
C(3)–C(4)	1·49	C(7)–C(8)	1·53	C(11)–C(12)	1·55	C(16)–O(22)	1·19
C(3)–O(18)	1·31						

Intramolecular non-bonded distances

C(1) ··· C(7)	3·15	C(3) ··· C(10)	3·46	C(6) ··· C(9)	3·22	C(9) ··· C(16)	3·03
C(1) ··· C(8)	3·26	C(3) ··· C(15)	3·78	C(6) ··· C(10)	3·10	C(9) ··· O(22)	3·00
C(1) ··· C(14)	3·85	C(4) ··· C(7)	3·95	C(6) ··· C(13)	3·75	C(10) ··· Br	3·91
C(1) ··· C(16)	3·05	C(4) ··· C(10)	3·36	C(6) ··· C(14)	3·19	C(10) ··· C(17)	3·90
C(1) ··· O(18)	3·65	C(4) ··· C(15)	3·46	C(6) ··· C(15)	3·42	C(10) ··· O(22)	2·97
C(1) ··· O(19)	3·84	C(4) ··· O(19)	3·12	C(6) ··· C(20)	3·42	C(13) ··· O(19)	3·62
C(1) ··· O(22)	3·17	C(5) ··· Br	3·91	C(7) ··· C(10)	3·21	C(14) ··· O(19)	3·12
C(2) ··· C(6)	3·85	C(5) ··· C(8)	3·34	C(7) ··· C(15)	3·84	C(15) ··· C(16)	3·62
C(2) ··· C(9)	3·78	C(5) ··· C(9)	3·29	C(7) ··· O(20)	3·50	C(16) ··· Br	3·48
C(2) ··· C(14)	3·83	C(5) ··· C(11)	3·74	C(8) ··· C(12)	3·66	O(18) ··· Br	3·15
C(2) ··· C(15)	3·11	C(5) ··· C(12)	3·55	C(8) ··· C(15)	3·13	O(19) ··· O(20)	2·25
C(2) ··· C(16)	3·20	C(5) ··· C(15)	3·20	C(8) ··· O(19)	3·66	O(21) ··· Br	3·69
C(2) ··· O(21)	2·66	C(5) ··· O(18)	3·50	C(8) ··· O(21)	3·87	O(21) ··· O(22)	2·29
C(2) ··· O(22)	3·60	C(5) ··· O(21)	3·91	C(9) ··· C(11)	3·78	O(22) ··· Br	3·56
C(3) ··· C(6)	3·74						

Intermolecular distances (<4 Å)

O(22) ··· C(2)$_{\mathrm{I}}$	3·15	C(15) ··· O(19)$_{\mathrm{III}}$	3·57	O(22) ··· O(18)$_{\mathrm{I}}$	3·68	C(6) ··· O(18)$_{\mathrm{II}}$	3·87
O(22) ··· C(3)$_{\mathrm{I}}$	3·29	C(7) ··· O(18)$_{\mathrm{II}}$	3·58	C(13) ··· Br$_{\mathrm{II}}$	3·71	C(15) ··· O(19)$_{\mathrm{V}}$	3·89
C(17) ··· O(18)$_{\mathrm{I}}$	3·37	C(17) ··· C(3)$_{\mathrm{I}}$	3·59	C(15) ··· O(20)$_{\mathrm{III}}$	3·74	O(21) ··· C(14)$_{\mathrm{V}}$	3·91
C(5) ··· O(18)$_{\mathrm{II}}$	3·41	O(18) ··· C(1)$_{\mathrm{IV}}$	3·59	C(16) ··· C(2)$_{\mathrm{I}}$	3·76	C(9) ··· Br$_{\mathrm{I}}$	3·95
C(16) ··· O(18)$_{\mathrm{I}}$	3·44	C(4) ··· O(20)$_{\mathrm{III}}$	3·62	C(9) ··· O(20)$_{\mathrm{V}}$	3·78	C(15) ··· C(6)$_{\mathrm{III}}$	3·96
C(15) ··· C(12)$_{\mathrm{III}}$	3·46	C(8) ··· O(20)$_{\mathrm{V}}$	3·63	C(17) ··· O(20)$_{\mathrm{VI}}$	3·82	O(19) ··· O(18)$_{\mathrm{II}}$	3·98
C(16) ··· C(3)$_{\mathrm{I}}$	3·48	C(1) ··· C(13)$_{\mathrm{VI}}$	3·66	O(22) ··· O(21)$_{\mathrm{I}}$	3·83	O(21) ··· O(18)$_{\mathrm{I}}$	3·98
O(22) ··· C(15)$_{\mathrm{I}}$	3·50	C(15) ··· C(14)$_{\mathrm{V}}$	3·66	C(14) ··· O(20)$_{\mathrm{III}}$	3·87	O(22) ··· C(4)$_{\mathrm{I}}$	3·99

The subscripts refer to the following positions:

I $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z.$
II $\frac{1}{2} + x, \frac{1}{2} - y, -z.$
III $-\frac{1}{2} - x, -y, \frac{1}{2} + z.$
IV $-\frac{1}{2} + x, \frac{1}{2} - y, -z.$
V $x, y, 1 + z.$
VI $-x, \frac{1}{2} + y, \frac{1}{2} - z.$

Valency angles

C(2)C(1)C(5)	107°	C(3)C(4)C(14)	115°	C(8)C(7)C(11)	110°	C(7)C(11)C(13)	119°
C(2)C(1)C(10)	108	C(5)C(4)C(14)	120	C(7)C(8)C(9)	115	C(12)C(11)C(13)	111
C(5)C(1)C(10)	116	C(1)C(5)C(4)	105	C(8)C(9)C(10)	117	C(11)C(12)O(19)	109
C(1)C(2)Br	115	C(1)C(5)C(6)	121	C(1)C(10)C(9)	111	C(11)C(12)O(20)	133
C(1)C(2)C(3)	105	C(4)C(5)C(6)	113	C(1)C(10)O(21)	109	O(19)C(12)O(20)	118
C(3)C(2)Br	106	C(5)C(6)C(7)	115	C(9)C(10)O(21)	106	C(17)C(16)O(22)	122
C(2)C(3)C(4)	109	C(5)C(6)O(19)	111	C(15)C(10)C(1)	121	O(21)C(16)C(17)	109
C(2)C(3)O(18)	129	C(7)C(6)O(19)	102	C(15)C(10)C(9)	111	O(21)C(16)O(22)	129
C(4)C(3)O(18)	123	C(6)C(7)C(8)	113	C(15)C(10)O(21)	114	C(6)O(19)C(12)	109
C(3)C(4)C(5)	101	C(6)C(7)C(11)	103	C(7)C(11)C(12)	100	C(10)O(21)C(16)	123

TABLE 3

Standard deviations of the final atomic co-ordinates (Å)

Atom	σ(x)	σ(y)	σ(z)	Atom	σ(x)	σ(y)	σ(z)	Atom	σ(x)	σ(y)	σ(z)
C(1)	0·020	0·018	0·030	C(9)	0·026	0·025	0·041	C(17)	0·026	0·028	0·040
C(2)	0·021	0·021	0·033	C(10)	0·024	0·023	0·032	O(18)	0·019	0·018	0·024
C(3)	0·025	0·026	0·035	C(11)	0·027	0·024	0·032	O(19)	0·017	0·017	0·023
C(4)	0·024	0·024	0·032	C(12)	0·028	0·025	0·034	O(20)	0·020	0·018	0·022
C(5)	0·025	0·024	0·038	C(13)	0·026	0·026	0·036	O(21)	0·018	0·015	0·023
C(6)	0·027	0·024	0·031	C(14)	0·031	0·032	0·040	O(22)	0·017	0·019	0·027
C(7)	0·025	0·023	0·033	C(15)	0·026	0·022	0·030	Br	0·003	0·002	0·005
C(8)	0·026	0·025	0·032	C(16)	0·023	0·028	0·031				

The standard deviations of the final atomic co-ordinates were estimated in the usual manner from the least-squares residuals (see Experimental section) and are given in Table 3; from the results the average estimated standard deviation (e.s.d.) of a carbon–carbon or carbon–oxygen bond length is about 0·04 Å and the average e.s.d. of a valency angle about 2°.

The average carbon–carbon single bond length is 1·52 Å, not significantly different from the accepted value of 1·545 Å. The average sp^3-carbon–oxygen single-bond length is 1·47 Å, whereas the average sp^2-carbon–oxygen single-bond length is only 1·35 Å; the difference demonstrates that in lactone and ester groups, just as in carboxylic acids, the structure (IV) makes an important contribution. This contribution implies that the atoms C(6), O(19), C(12), O(20), and C(11) of the γ-lactone ring should be coplanar. Accordingly, the mean plane through these atoms was calculated by the method of Schomaker *et al.*[12] The atomic deviations from this plane (see Table 4) prove that the atoms C(6), O(19), C(12), O(20), and C(11) are indeed closely coplanar but that the γ-lactone ring is nevertheless significantly non-planar, since the atom C(7) is displaced by 0·60 Å from the plane. Comparable results have been obtained for the γ-lactone rings in himbacine hydrobromide,[13] bromogeigerin acetate,[1] and 2-bromo-α-santonin.[10] As expected, the five atoms C(10), O(21), C(16), O(22), and C(17) of the ester group are also closely coplanar.

TABLE 4

Deviations (Å) of the atoms from planes connected with the five-membered rings

Plane through C(6), C(11), C(12), O(19), O(20)			
C(6)	−0·018	C(12)	−0·041
C(7)	0·595	O(19)	0·033
C(11)	0·020	O(20)	0·005

Plane through C(1), C(2), C(3), C(5)			
C(1)	−0·012	C(4)	0·541
C(2)	0·012	C(5)	0·008
C(3)	−0·007		

The angles around carbon atoms 12 and 16 conform to the pattern noted in carboxylic acids,[14] the angles C(17)C(16)O(21) and C(11)C(12)O(19) being approximately tetrahedral, whereas the angles C(17)C(16)O(22), O(21)C(16)O(22), C(11)C(12)O(20), and O(19)C(12)O(20) exceed 120°.

The cycloheptane ring adopts a chair conformation but is somewhat flattened, since the valency angles are consistently greater than tetrahedral, the mean value being 115°. Similar large values for the valency angles in seven- and higher-membered rings have been noted in bromogeigerin acetate,[1] isoclovene hydrochloride,[15] cyclononylamine hydrobromide,[16] and 1,6-*trans*-diaminocyclodecane dihydrochloride.[17] In the five-membered rings the average valency angle (105°) is distinctly less than tetrahedral and in good agreement with the average values for the five-membered rings in other molecules, *e.g.*, hydroxy-L-proline (106°),[18] clerodin bromolactone (106°),[19] and isoclovene hydrochloride (105°).[15] These values are consistent with the non-planarity and consequent angle deformations in cyclopentane rings.[20] In accord with this, the ring C(1)C(2)C(3)C(4)C(5) is distinctly non-planar; atoms C(1), C(2), C(3), and C(5) are closely coplanar but C(4) deviates by 0·54 Å from the mean plane through these atoms (see Table 4).

Fig. 3 shows the arrangement of the molecules in the crystal as viewed in projection along the *a*- and *c*-axes. The intermolecular contacts (Table 2) are all greater than 3 Å and correspond to normal van der Waals interactions; the shortest separations involve oxygen atoms.

[12] Schomaker, Waser, Marsh, and Bergman, *Acta Cryst.*, 1959, **12**, 600.

[13] Fridrichsons and Mathieson, *Acta Cryst.*, 1962, **15**, 119.

[14] Cochran, *Acta Cryst.*, 1953, **6**, 260; Ahmed and Cruickshank, *ibid.*, 385; Ferguson and Sim, *ibid.*, 1961, **14**, 1262; *J.*, 1962, 1767.

[15] Clunie and Robertson, *J.*, 1961, 4382.

[16] Bryan and Dunitz, *Helv. Chim. Acta*, 1960, **43**, 3.

[17] Huber-Buser and Dunitz, *Helv. Chim. Acta*, 1960, **43**, 760.

[18] Donohue and Trueblood, *Acta. Cryst.*, 1952, **5**, 419.

[19] Paul, Sim, Hamor, and Robertson, *J.*, 1962, 4133.

[20] Pitzer and Donath, *J. Amer. Chem. Soc.*, 1959, **81**, 3213; Brutcher, Roberts, Barr, and Pearson, *ibid.*, p. 4915.

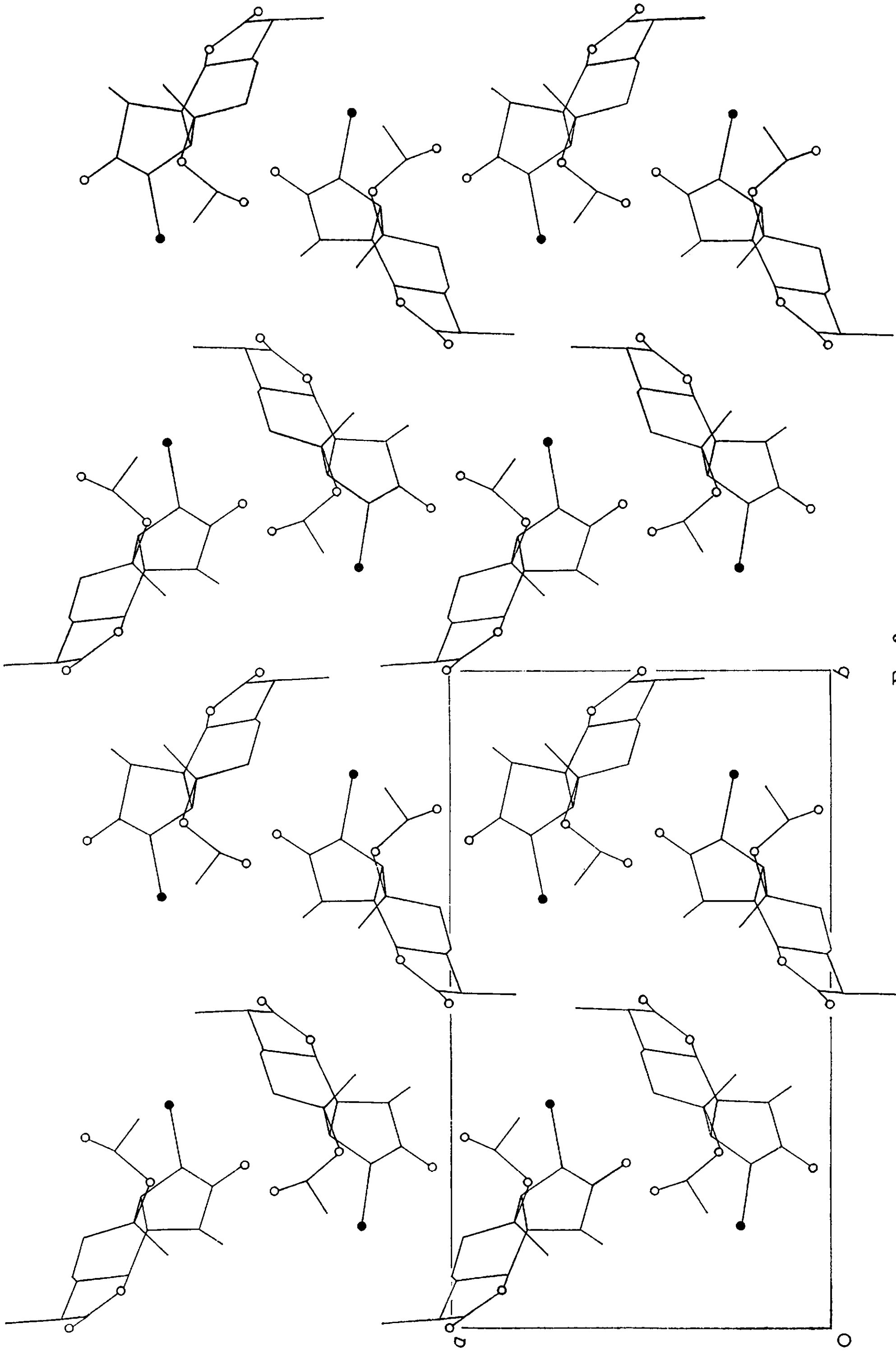

FIG 3*a*

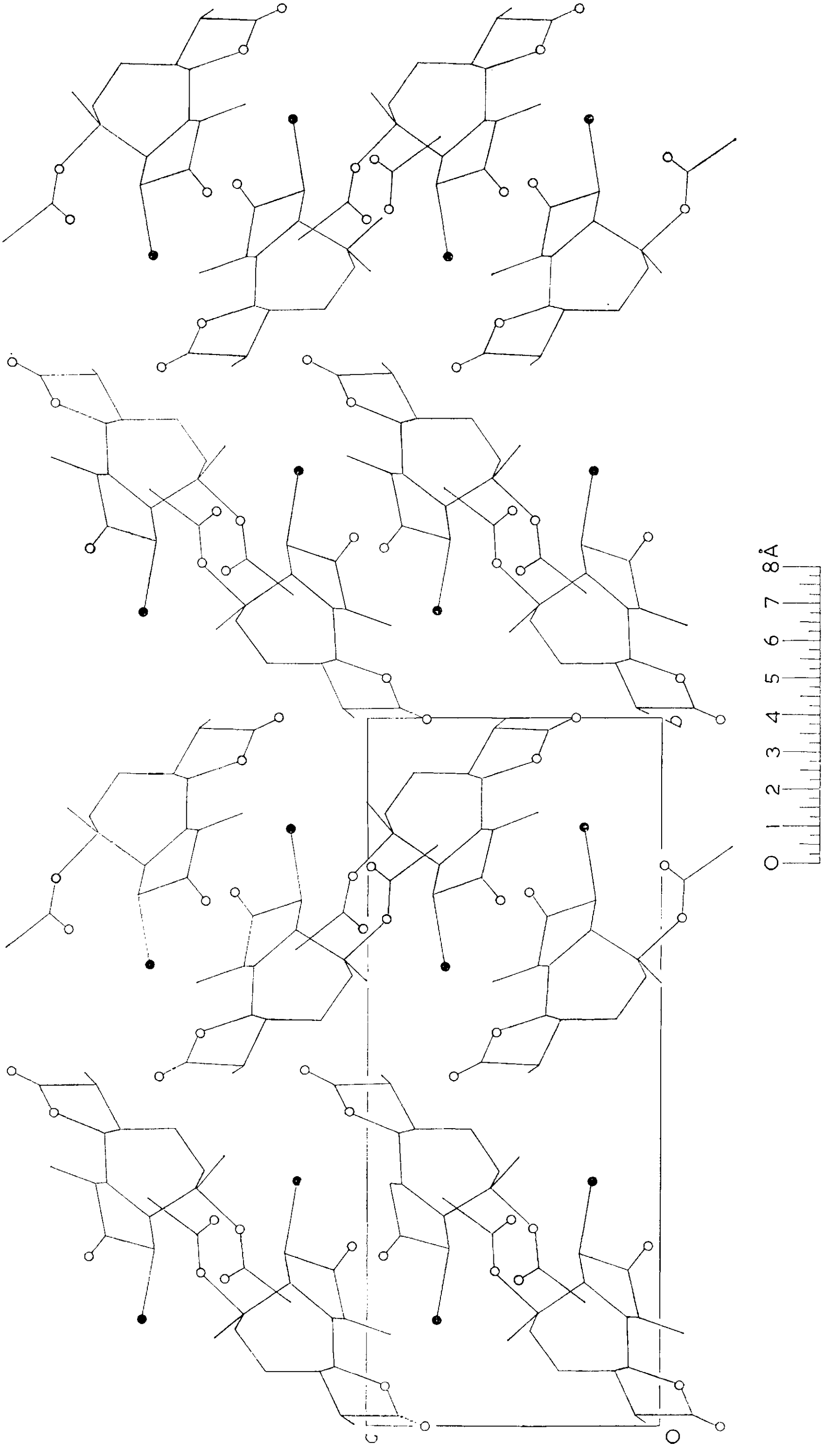

FIG. 3*b*

FIG. 3. The arrangement of molecules in the crystal as viewed along (*a*) the *c*-axis, (*b*) the *a*-axis

EXPERIMENTAL

Crystal Data.—2-Bromodihydroisophoto-α-santonic lactone acetate, $C_{17}H_{23}BrO_5$; $M =$ 387·3; m. p. 117—118° (decomp.). Orthorhombic, $a = 11{\cdot}05$, $b = 19{\cdot}23$, $c = 7{\cdot}93$ Å, $U =$ 1685 Å,^{3} $Z = 4$, $D_c = 1{\cdot}526$ g.cm.$^{-3}$, $F(000) = 800$, space group $P2_12_12_1$-$D_2{}^4$. Absorption coefficient for *X*-rays ($\lambda = 1{\cdot}542$ Å) $\mu = 38{\cdot}7$ cm.$^{-1}$.

Experimental Measurements.—Rotation, oscillation, and Weissenberg photographs were taken with copper K_α ($\lambda = 1{\cdot}542$ Å) radiation; precession photographs were taken with molybdenum K_α ($\lambda = 0{\cdot}7107$ Å) radiation. The unit-cell dimensions were obtained from the rotation and precession photographs. The space group was determined uniquely from the systematic halvings in the *X*-ray spectra. For the intensity measurements small crystals were employed, completely bathed in a uniform *X*-ray beam, and no corrections for absorption were applied.

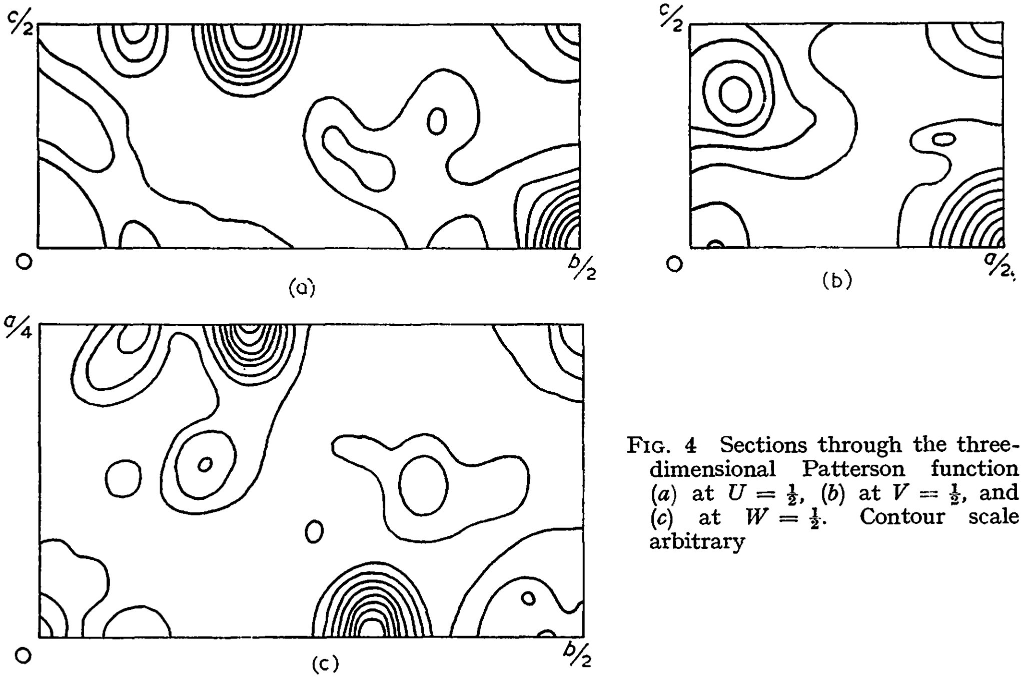

FIG. 4 Sections through the three-dimensional Patterson function (*a*) at $U = \frac{1}{2}$, (*b*) at $V = \frac{1}{2}$, and (*c*) at $W = \frac{1}{2}$. Contour scale arbitrary

The *hk*0—*hk*4 spectra were recorded photographically by means of an equi-inclination Weissenberg camera, whilst the 0*kl* and *h*0*l* spectra were obtained with a precession camera. The intensities were estimated visually and were corrected by the usual factors (Lorentz, polarization, and rotation [21]). The various layers were placed on the same relative scale by comparison of common reflections on different photographs; the absolute scale was obtained at a later stage by correlation with the calculated structure amplitudes, $|F_c|$. In all, 865 independent structure amplitudes, $|F_o|$, were evaluated.

The crystals, stored at room temperature, decomposed in a few months to a black resinous gum, and so we have no experimental value for their density.

Structure Analysis.—The position of the bromine atom was determined from two-dimensional Patterson syntheses and sections through the three-dimensional Patterson synthesis $P(UVW)$ at $U = \frac{1}{2}$, $V = \frac{1}{2}$, and $W = \frac{1}{2}$. These sections are shown in Fig. 4. The co-ordinates obtained for the bromine atom were $x = 0{\cdot}242$, $y = 0{\cdot}152$, $z = 0{\cdot}233$.

Structure factors based on the bromine atom alone were calculated; the value of R, the average discrepancy between measured and calculated structure amplitudes, was 45·1%. With the measured $|F|$ values and the calculated phase angles we then evaluated a three-dimensional electron-density distribution. The approximately centrosymmetrical arrangement of the bromine atoms in the unit cell led to spurious planes of symmetry perpendicular to the *a*- and *c*-axes in the Fourier synthesis, but, since the *x*- and *z*-co-ordinates of the bromine atom are not

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[21] Tunell, *Amer. Min.*, 1939, **24**, 448.

exactly $\frac{1}{4}$, the peak heights within each set of four pseudo-symmetrically related peaks were not exactly identical. We selected three peaks which were rather higher than their "ghosts" and included them (as carbon atoms) with the bromine atom in the calculation of a set of structure factors. The value of R was 42·0%.

With the revised phase constants we evaluated a second three-dimensional electron-density distribution, and from this selected a further five atoms for inclusion in the calculation of a further set of structure factors. The value of R fell to 38·9%. In the subsequent two rounds of Fourier and structure-factor calculation four more peaks were designated as atoms and the value of R was reduced to 36·9%. Prolonged attempts to recognise known chemical features (five- and seven-membered rings, etc.) in the fifth electron-density distribution failed and we were forced to conclude that some of the peaks we had selected as atoms were spurious, *i.e.*, "ghosts" of genuine peaks.

During the five rounds of calculations the x-co-ordinate of the bromine atom had moved slightly further away from the special value of $\frac{1}{4}$, and we therefore decided to recalculate structure factors based solely on the bromine atom; the value of R was 43·7%. The subsequent Fourier synthesis with phases determined by the bromine atom in its revised position was carefully examined from the viewpoint of fitting the five- and seven-membered rings and their substituents into the unit cell without having abnormal contacts. We were able, eventually, to postulate positions for the atoms of the five-membered ring which carries the bromine atom. These five atoms were included with the bromine atom in the calculation of a set of structure amplitudes (R = 40·5%) and phase angles which were then used to derive an electron-density distribution. In this we were able to discern the seven-membered carbocyclic ring and the two substituent atoms at C(10). The lactone ring was nebulously defined, but its constituent atoms were not sufficiently well resolved to merit inclusion in the eighth structure-factor calculation, which was based, therefore, on twelve light atoms (weighted as carbon) and the bromine. The value of R was 36·6%.

Three more rounds of Fourier and structure-factor calculations, with increasing numbers of atoms included in the structure-factor calculations at each stage, enabled us to place the remaining atoms apart from C(13) and to distinguish between oxygen and carbon atoms. The value of R was then 24·8%.

Three further stages of refinement were carried out by Fourier methods, and C(13) was unambiguously located β-oriented to C(11). Both F_o and F_c syntheses were computed, and atomic co-ordinates obtained by numerical interpolation in the results; errors due to termination of series were corrected and individual isotropic temperature factors assigned. The value of R fell to 20·2%.

In the final stages of the analysis Rollett's least-squares programme [22] for DEUCE was used to refine positional and anisotropic thermal parameters for all atoms other than hydrogen. The weighting scheme employed was:

$$\sqrt{w} = |F_o|/15 \text{ if } |F_o| < 15, \sqrt{w} = 15/|F_o| \text{ if } |F_o| > 15.$$

After four cycles of calculations the value of R was 14·6%.

Examination of the bond lengths at this stage showed that C(7)–C(11) was about 0·1 Å shorter than expected, whereas C(10)–O(21) was about 0·1 Å longer than expected. We thought that these results might not be inherent in the intensity data, but might be due to neglect of correlation between temperature-factor and positional parameters in the least-squares calculations. We therefore derived from the anisotropic thermal parameters (b_{ij}) for each atom an isotropic temperature factor (B), adjusted the co-ordinates of C(7), C(11), C(10), and O(21) to give more acceptable bond lengths, and began further least-squares refinement. The value of R rose initially to 21·4%, fell to 16·4% at the end of the next cycle, and thereafter fell steadily to the final value of 12·9%.

The theoretical atomic scattering factors used in all the structure-factor calculations were those of Berghuis *et al.*[23] for carbon and oxygen, and the Thomas–Fermi values for bromine.[24] The final calculated structure amplitudes and phase constants are listed with the measured values of the structure amplitudes in Table 5. The phase constants of Table 5 and the measured

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[22] Rollett, in "Computing Methods and the Phase Problem in X-Ray Crystal Analysis," ed. Pepinsky, Robertson, and Speakman, Pergamon, Oxford, 1961, p. 87.

[23] Berghuis, Haanappel, Potters, Loopstra, MacGillavry, and Veenendaal, *Acta Cryst.*, 1955, **8**, 478.

[24] "Internationale Tabellen zur Bestimmung von Kristallstrukturen," Borntraeger, Berlin, 1935, vol. II, p. 572.

TABLE 5

Measured and calculated values of the structure factors

h	*k*	*l*	F_o	F_c	α
0	1	3	28	33	270
0	2	0	75	100	180
		1	12	23	0
		2	21	18	0
		3	76	90	180
		4	31	35	180
0	3	1	59	67	90
		2	14	14	90
		3	16	15	270
0	4	0	21	15	180
		1	40	44	180
		2	50	58	0
		3	36	50	0
		4	15	16	180
0	5	1	30	31	90
		2	5	6	90
		3	37	33	90
		4	10	19	90
0	6	0	81	92	0
		1	66	71	180
		2	77	88	180
		3	51	60	0
		4	50	54	0
0	7	1	17	17	90
		2	10	11	270
		3	9	5	270
		4	24	22	90
0	8	0	38	45	0
		1	91	87	0
		2	9	9	0
		3	75	72	180
		4	16	16	0
0	9	1	36	38	90
		2	25	19	90
		3	4	5	90
0	10	0	72	69	180
		2	53	48	0
		3	23	20	0
		4	19	14	180
0	11	1	38	32	270
		2	10	9	90
		4	10	11	90
0	12	0	64	58	0
		1	50	49	180
		2	42	42	180
		3	46	42	0
		4	9	11	0
0	13	1	22	20	90
		2	7	7	90
		3	10	8	90
		4	11	15	90
0	14	0	79	65	0
		1	25	23	0
		2	14	15	180
		3	43	45	180
0	15	1	8	9	90
		2	5	2	270
		3	12	13	270
0	16	0	42	39	180
		2	21	23	0
0	16	3	8	11	180
0	17	1	15	15	90
		2	12	10	270
		3	9	11	90
0	18	0	8	7	0
		1	46	31	180
		2	7	7	0
		3	21	26	0
0	19	3	5	1	270
0	20	0	23	20	0
		1	12	12	0
		2	27	24	180
		3	7	9	180
0	21	1	8	7	90
		3	4	0	270
0	22	0	24	21	180
		1	18	12	0
		2	16	12	0
		3	4	7	180
0	23	1	3	2	90
0	24	1	11	9	180
1	0	2	26	38	0
		3	11	11	90
		4	7	9	180
1	1	0	11	7	270
		1	112	147	70
		2	64	88	274
		3	45	66	267
		4	29	39	81
1	2	0	52	63	90
		1	35	30	57
		2	64	60	179
		3	52	54	98
		4	29	32	210
1	3	0	12	27	270
		1	82	65	8
		2	125	131	90
		3	12	11	138
		4	52	54	275
1	4	0	88	88	90
		1	64	70	287
		2	23	22	104
		3	28	30	260
		4	28	31	314
1	5	0	48	37	90
		1	117	116	294
		2	28	20	82
		3	17	18	47
		4	5	7	93
1	6	0	18	13	90
		1	24	26	275
		2	6	5	133
		3	43	40	88
		4	32	32	204
1	7	0	98	106	90
		1	37	42	138
		2	78	74	277
		3	31	29	221
		4	37	43	86
1	8	0	21	19	90
1	8	1	31	27	282
		2	39	25	94
		3	19	22	36
1	9	0	58	58	270
		1	50	60	72
		2	58	66	81
		3	37	35	283
		4	39	40	285
1	10	0	22	23	270
		1	19	16	174
		2	13	11	137
		3	13	12	149
		4	11	11	55
1	11	1	70	72	281
		2	28	26	122
		3	25	20	116
		4	19	21	223
1	12	0	10	9	270
		1	17	16	186
		2	8	6	28
		3	16	19	113
		4	15	9	213
1	13	0	48	43	90
		1	7	9	25
		2	47	47	265
		3	11	12	98
		4	30	39	83
1	14	0	13	15	90
		1	7	6	153
		2	17	17	34
		3	5	4	43
1	15	0	16	17	270
		1	45	42	88
		2	23	21	108
		3	27	25	274
1	16	0	16	14	90
		1	7	6	10
		2	25	21	187
		3	8	7	51
1	17	0	22	21	270
		1	22	20	275
		2	26	18	93
		4	13	15	279
1	18	0	10	10	90
		1	11	10	272
		2	13	11	75
1	19	0	22	17	90
		1	32	22	268
		2	29	23	269
1	20	1	4	6	211
		3	6	2	348
1	21	1	33	26	92
		3	12	18	267
1	22	1	4	2	320
1	23	0	14	13	270
		1	9	9	246
2	0	0	60	78	180
		1	82	87	90
		2	91	107	0
		3	14	12	90
2	0	4	52	56	180
2	1	0	34	45	0
		1	12	10	6
		2	26	23	301
		3	26	22	155
		4	18	14	270
2	2	0	119	121	0
		1	112	131	218
		2	92	101	186
		3	58	63	359
		4	14	13	325
2	3	0	10	16	0
		1	67	61	191
		2	8	7	1
		3	18	20	120
		4	36	34	106
2	4	0	116	137	0
		1	41	40	58
		2	37	35	165
		3	40	32	171
		4	37	44	354
2	5	0	21	20	0
		1	44	43	94
		2	25	21	321
		3	34	32	204
		4	13	13	229
2	6	0	75	93	180
		1	55	46	35
		2	54	61	332
		3	45	46	179
		4	24	19	201
2	7	0	41	35	0
		1	35	25	289
		3	12	6	318
2	8	0	8	4	0
		1	68	74	147
		2	20	17	286
		3	79	74	5
		4	19	18	279
2	9	0	47	52	180
		1	18	18	151
		2	16	18	271
		3	17	14	74
		4	9	8	160
2	10	0	75	81	0
		1	20	18	17
		2	71	69	184
		3	14	11	184
		4	35	27	8
2	11	1	17	16	55
		2	15	17	134
		3	7	10	274
		4	15	20	40
2	12	0	26	24	180
		1	45	50	352
		2	18	18	14
		3	27	27	164
		4	17	16	171
2	13	0	17	15	180
		1	20	20	91
2	13	2	9	8	218
		3	9	7	84
		4	15	14	246
2	14	0	10	8	180
		1	33	38	171
		2	17	16	347
		3	40	36	357
2	15	1	10	9	263
		2	7	8	329
		3	8	6	183
2	16	0	45	44	0
		1	19	19	209
		2	27	26	176
		3	7	4	31
		4	9	11	3
2	17	0	5	4	180
		1	7	6	150
		2	4	6	181
		3	8	7	280
		4	9	8	73
2	18	0	13	12	180
		1	26	25	14
		2	9	9	193
		3	17	20	186
2	19	0	4	5	0
		1	8	8	87
		3	6	7	160
2	20	0	35	28	180
		1	14	12	196
		2	23	21	344
		3	6	9	11
2	21	3	4	4	86
2	22	0	21	16	0
		1	11	8	159
		2	14	14	195
		3	8	10	1
3	0	1	32	32	90
		2	16	16	180
		3	16	11	90
3	1	0	68	77	270
		1	76	95	269
		2	51	53	67
		3	55	55	96
		4	21	16	299
3	2	0	8	5	270
		1	49	55	324
		2	28	32	194
		3	13	12	114
		4	24	24	216
3	3	0	58	69	90
		1	39	40	211
		2	77	86	266
		3	25	22	207
		4	33	34	101
3	4	0	30	35	90
		1	26	22	283
		2	26	27	353
		3	29	27	64
		4	8	6	128
3	5	0	16	18	90
3	5	1	83	97	56
		2	28	22	63
		3	31	49	277
		4	18	15	319
3	6	0	38	36	270
		1	35	36	294
		2	48	44	214
		3	13	10	258
		4	16	15	275
3	7	0	80	81	270
		1	30	35	257
		2	46	53	111
		3	17	15	149
		4	46	42	251
3	8	0	7	5	90
		1	37	28	218
		2	25	26	92
		3	12	11	199
		4	8	8	84
3	9	0	78	76	90
		1	72	59	297
		2	22	25	290
		3	28	26	107
		4	31	30	87
3	10	0	11	7	90
		1	26	29	283
		2	19	19	55
		3	17	15	76
		4	13	11	231
3	11	0	25	19	90
		1	55	54	98
		2	16	17	290
		3	38	42	266
		4	15	16	32
3	12	1	7	8	117
		3	22	25	299
3	13	0	50	46	270
		2	36	38	87
		3	5	2	167
		4	24	25	263
3	14	0	4	4	90
		3	18	20	119
3	15	1	33	32	252
		2	15	17	280
		3	21	22	105
3	16	1	5	5	265
		2	8	7	80
3	17	0	30	23	90
		1	21	17	102
		2	19	19	285
		3	9	13	267
3	18	0	26	17	90
3	19	0	21	17	270
		1	16	13	81
		2	22	21	92
		3	13	13	270
3	20	0	13	11	270
		1	6	8	327
		3	3	1	20
3	21	1	24	23	257
3	21	3	7	11	101
3	22	2	8	7	66
3	23	2	14	12	273
3	24	1	10	2	246
4	0	0	89	107	0
		1	13	10	90
		2	73	82	180
		3	11	11	90
		4	61	66	0
4	1	0	27	27	180
		1	36	36	231
		2	14	14	211

TABLE 5 (*Continued*)

h	k	l	F_o	F_c	α
		3	21	19	307
		4	18	13	66
4	2	0	2	2	180
		1	48	63	347
		2	6	6	231
		3	64	60	190
		4	30	25	191
4	3	0	26	24	0
		1	45	44	303
		2	13	12	40
		3	32	34	198
4	4	0	34	39	180
		1	69	84	183
		2	32	35	1
		3	22	21	359
		4	25	17	182
4	5	0	13	14	180
		1	18	19	186
		2	15	14	215
		3	31	26	44
		4	13	12	139
4	6	0	58	69	0
		1	34	35	220
		2	42	48	188
		3	24	30	347
		4	29	25	338
4	7	0	9	6	180
		1	25	23	149
		2	20	21	342
		3	12	11	195
		4	8	6	119
4	8	0	32	35	0
		1	52	58	11
		2	6	9	129
		3	47	46	182
		4	19	23	30
4	9	0	21	20	0
		1	25	24	35
		2	22	19	197
		3	9	8	357
		4	12	12	310
4	10	0	33	35	180
		1	9	10	234
		2	47	52	345
		3	12	14	5
		4	22	22	202
4	11	0	9	8	180
		1	13	15	201
4	11	2	27	25	358
		3	12	13	175
		4	11	11	191
4	12	0	32	35	0
		1	35	33	185
		2	38	37	180
		3	29	27	351
		4	9	8	338
4	13	1	19	22	93
		2	14	12	232
4	14	0	20	19	0
		1	28	33	354
		2	27	25	185
		4	12	11	7
4	15	0	5	3	0
4	16	0	26	24	180
		1	7	7	75
		2	23	20	345
		4	14	17	202
4	17	0	5	4	0
		1	7	8	346
		2	8	9	45
		3	5	6	114
4	18	0	9	6	180
		1	32	28	196
		3	14	16	353
4	19	0	4	5	180
		1	7	6	183
4	20	0	19	20	0
		1	8	9	22
		2	17	14	187
		3	8	8	186
4	21	0	4	4	0
4	22	0	21	16	180
		1	11	9	0
		2	9	7	1
5	0	1	8	10	270
		2	8	7	180
		3	42	34	90
		4	10	12	180
5	1	0	54	51	90
		1	53	61	82
		2	24	26	258
		3	36	36	266
5	2	0	48	50	90
		1	36	34	167
		2	14	13	264
		3	10	11	54
5	3	0	89	89	270
		1	18	16	62
		2	32	44	82
		3	19	18	280
		4	27	27	264
5	4	0	18	21	270
		1	28	18	206
		2	8	10	160
		3	23	19	167
		4	11	12	348
5	5	0	29	24	90
		1	44	53	278
5	5	2	16	14	140
		3	50	51	91
		4	18	13	111
5	6	1	25	28	308
		3	20	18	62
		4	10	14	220
5	7	0	17	15	90
		1	34	28	43
		2	51	56	267
		3	31	30	259
		4	15	15	106
5	8	0	5	6	90
		1	22	24	265
		2	9	11	167
		3	9	8	37
		4	11	11	128
5	9	0	40	41	270
		1	37	38	73
		2	18	19	82
		3	24	24	279
		4	19	19	294
5	10	0	9	10	90
		1	12	10	113
		2	19	18	129
		3	13	17	262
		4	9	6	279
5	11	0	31	27	270
		1	45	44	282
		2	11	11	102
		3	31	28	89
5	12	0	18	15	270
		1	17	17	322
		2	10	9	58
		3	17	20	105
5	13	0	54	47	90
		1	5	5	188
		2	21	19	272
		3	14	16	251
		4	15	14	65
5	14	0	18	16	90
		1	7	7	100
		3	5	6	38
5	15	0	13	10	270
		1	24	18	87
		3	23	27	278
		4	11	3	202
5	16	0	9	11	90
		3	5	2	332
5	17	0	26	21	270
		1	16	14	259
		2	20	17	80
		3	10	10	96
		4	12	12	284
5	18	0	6	6	270
		2	10	10	69
5	19	0	20	19	90
		1	6	7	263
		2	14	15	265
5	20	0	5	5	90
		1	4	4	297
5	20	2	4	5	328
5	21	1	15	12	88
6	0	0	27	24	0
		1	26	24	90
		2	38	40	0
		3	28	21	90
		4	27	29	180
6	1	1	14	14	295
		2	22	18	27
		3	11	11	158
		4	13	12	153
6	2	0	20	20	0
		1	73	77	180
		2	23	21	166
		3	26	28	329
6	3	0	6	7	180
		1	30	36	144
		2	13	11	253
		3	10	11	70
6	4	0	48	53	0
		1	38	39	21
		2	35	28	200
		3	27	30	162
		4	30	33	351
6	5	0	21	26	180
		2	20	22	178
		3	15	13	224
		4	11	10	25
6	6	0	15	20	180
		1	21	20	330
		2	41	39	17
		3	5	8	273
6	7	0	4	5	0
		1	4	7	297
		3	8	8	159
6	8	0	21	19	180
		1	39	45	191
		2	12	13	291
		3	16	16	19
		4	9	10	237
6	9	0	12	14	180
		1	17	19	167
		2	14	15	23
		3	7	8	145
		4	22	22	136
6	10	0	40	41	0
		1	21	19	0
		2	39	39	181
		3	5	4	280
		4	25	27	357
6	11	0	29	27	0
		1	6	7	158
		2	9	6	230
		4	8	10	355
6	12	1	24	23	358
		2	21	23	313
		3	12	11	179
		4	15	14	213
6	13	0	20	19	180
		1	11	10	47
6	13	2	10	9	93
6	14	1	29	25	169
		2	18	17	8
		3	8	8	25
6	15	2	9	9	342
6	16	0	20	16	0
		1	17	12	159
		2	31	25	201
6	18	1	16	15	355
		3	11	12	185
6	19	1	8	7	346
6	20	0	15	13	180
		1	5	6	170
		2	11	10	7
6	21	0	7	6	180
7	0	1	14	14	90
		2	4	5	180
		3	11	10	270
		4	7	8	0
7	1	0	18	19	270
		1	21	24	282
		2	28	30	72
		3	33	33	86
		4	9	9	302
7	2	0	9	8	270
		1	8	8	252
		2	15	14	121
		3	13	14	104
		4	18	17	213
7	3	0	57	56	90
		1	18	15	335
		2	18	24	273
		3	13	13	99
		4	16	14	62
7	4	0	37	43	90
		1	15	15	268
		2	9	9	189
		4	9	8	214
7	5	0	11	10	270
		1	34	32	89
		2	5	6	107
		3	40	40	273
7	6	0	4	5	90
		1	13	13	215
		2	12	15	203
		3	6	6	327
		4	17	8	191
7	7	0	32	35	270
		1	25	23	231
		2	20	19	74
		3	7	5	91
7	8	0	14	15	270
		1	8	5	330
		2	16	17	107
		3	13	13	191
		4	9	3	237
7	9	0	25	30	90
		1	20	18	279
		2	12	10	316
		3	17	19	92
7	10	0	19	16	270
		1	18	14	225
		2	9	7	248
		3	20	19	70
7	11	0	17	18	90
		1	22	18	76
		2	7	8	222
		3	32	37	265
7	12	0	10	8	90
		1	18	13	51
		2	8	4	196
		3	8	5	347
7	13	0	31	31	270
		2	18	14	103
		3	5	2	304
7	14	0	12	10	270
		3	6	6	189
7	15	0	8	9	90
		1	22	19	288
		3	5	6	77
7	16	3	5	5	96
7	17	0	24	20	90
		1	13	11	94
		2	13	10	256
		3	9	10	269
7	18	0	10	10	90
		1	9	8	214
7	19	0	11	12	270
		2	11	11	86
		3	5	7	280
8	0	0	38	39	0
		1	23	23	90
		2	21	23	180
		3	10	9	90
8	1	0	21	25	0
		1	7	9	167
		2	15	16	228
		3	19	13	264
		4	9	15	344
8	2	0	11	10	0
		1	30	33	337
		2	10	12	285
		3	12	19	187
		4	19	10	283
8	3	0	4	5	0
		1	5	5	61
		3	9	10	166
8	4	0	33	29	180
		1	25	28	176
		2	14	13	24
		3	24	23	3
		4	13	14	172
8	5	0	9	13	180
		1	6	5	149
		3	11	9	161
8	6	1	19	19	145
		2	28	26	170
		3	5	4	159
		4	21	22	7
8	7	0	10	6	0
8	7	1	16	16	166
		2	5	6	253
		4	8	8	69
8	8	1	34	36	13
		2	22	18	236
		3	5	2	315
		4	12	13	320
8	9	1	7	7	304
		2	5	9	164
8	10	0	19	19	180
		1	9	8	217
		2	27	20	349
		3	9	4	106
8	11	0	7	6	180
		1	5	6	314
		2	16	15	22
8	12	0	12	9	0
		1	11	10	166
		2	19	16	179
		3	12	13	359
8	13	1	12	10	217
		2	5	7	212
8	14	0	18	15	0
		1	8	8	19

TABLE 5 (*Continued*)

h	*k*	*l*	F_o	F_c	α
		2	15	12	193
		3	8	9	167
8	15	0	4	2	0
		3	4	2	205
8	16	0	11	9	180
		2	11	14	353
8	17	3	5	3	135
8	18	1	14	12	175
8	19	1	5	5	167
8	20	1	5	5	32
9	0	1	12	12	270
		3	9	9	90
9	1	1	17	16	83
		2	18	16	245
		4	12	11	140
9	2	0	9	9	90
		1	6	6	87
		2	14	15	241
		3	5	6	264
9	3	0	26	30	270
		1	12	14	149
		2	21	18	88
		3	9	11	248
9	4	0	8	6	90
		2	7	8	75
9	5	0	18	18	90
		1	20	19	269
		2	10	9	36
		3	18	15	90
9	6	0	11	11	270
		1	24	20	332
		2	9	7	158
		3	7	5	50
9	7	0	13	13	90
		1	5	6	113
		2	18	17	280
9	7	3	13	13	261
9	8	0	16	16	90
		1	13	13	208
		2	5	4	271
		3	6	7	35
9	9	0	8	9	270
		1	9	12	122
		2	14	13	112
		3	10	10	264
9	10	0	24	20	270
		1	6	4	69
		3	7	8	250
9	11	0	10	7	270
		1	16	14	254
		2	5	7	56
		3	8	9	101
9	12	0	11	7	270
		2	7	6	43
		3	8	3	136
9	13	0	13	13	90
		1	7	3	349
		2	9	10	263
		3	5	3	227
9	14	2	11	8	267
9	15	1	16	12	102
9	17	0	15	11	270
		1	6	6	261
9	18	1	3	2	299
10	0	0	15	15	180
10	1	0	9	10	180
		1	5	5	300
		2	16	16	1
		3	7	5	109
10	2	1	17	19	209
		2	5	6	160
		3	12	13	355
10	3	0	8	8	0
		1	9	9	128
		2	7	5	251
		3	6	4	123
10	4	0	16	15	0
		1	8	8	15
		2	11	12	177
10	5	0	11	10	0
		1	10	10	243
		2	5	5	166
		3	5	6	326
10	6	0	17	16	180
		2	13	15	329
10	7	0	5	2	180
		1	6	7	5
		2	9	8	18
10	8	1	10	11	159
10	9	1	4	4	195
10	10	0	11	13	0
		2	13	13	214
11	0	1	16	12	90
11	1	0	9	10	270
		1	6	8	244
		2	9	8	112
11	2	1	9	8	326
		2	6	4	144
11	3	0	8	7	90
		2	14	14	281
11	4	1	5	4	223
		2	9	6	307
11	5	1	12	11	93
11	6	0	8	6	90
		1	10	5	10
11	7	0	12	12	270
		1	4	7	312
11	8	0	13	11	270
11	9	0	9	9	90
12	0	2	9	8	180
12	1	0	15	14	0
12	2	2	5	2	355
12	3	0	6	5	180
12	4	0	13	11	180
12	5	0	6	5	180
12	6	0	10	11	0
12	8	0	7	3	180

TABLE 6

Anisotropic temperature factors ($b_{ij} \times 10^5$)

	b_{11}	b_{22}	b_{33}	b_{12}	b_{23}	b_{13}
C(1)	1130	286	1820	−168	423	−2019
C(2)	1308	308	3305	68	444	−1423
C(3)	1483	450	3970	423	−704	−271
C(4)	1001	499	3208	425	−630	−862
C(5)	1268	397	4381	−48	191	878
C(6)	1954	452	2016	−619	243	1256
C(7)	1436	350	3689	127	146	82
C(8)	1655	461	2272	410	−822	29
C(9)	1271	398	7279	265	122	−821
C(10)	1228	412	1965	152	844	486
C(11)	1831	373	2447	−37	−62	−181
C(12)	2286	421	2613	159	479	1127
C(13)	1313	562	4398	438	1535	1489
C(14)	1908	714	4754	23	−622	−2856
C(15)	1441	422	2328	−143	−75	1805
C(16)	1308	606	2316	7	−154	−1030
C(17)	1508	642	3944	604	−562	998
O(18)	1824	604	3799	530	302	−1372
O(19)	1437	437	4157	150	258	601
O(20)	2556	668	1756	613	200	1523
O(21)	1646	300	5036	67	−70	−83
O(22)	1194	615	5869	−191	−547	−208
Br	1945	329	5460	70	161	−378
($B = 6$ Å^2	1767	584	3456	0	0	0)

structure amplitudes were used to derive a final three-dimensional electron-density distribution which is shown in Fig. 1 by means of superimposed contour sections drawn parallel to (001). All the atoms are well resolved, and the oxygen atoms are represented by distinctly higher peaks than the carbon atoms. The final atomic co-ordinates, molecular dimensions, and some non-bonded distances are given in Tables 1 and 2. The parameters defining the anisotropic thermal vibrations are in Table 6; they are values of b_{ij} in the equation:

$$\exp(-B \sin^2 \theta/\lambda^2) = 2^{-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{23}kl + b_{13}hl)}$$

The standard deviations of the final atomic co-ordinates were derived from the least-squares residuals by means of the equation:

$$\sigma^2(x_i) = \sum_j w_j(\Delta F)^2/[(n - s) \sum w_j(\partial F_j/\partial x_i)^2].$$

The results are listed in Table 3.

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