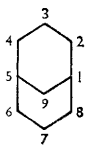
338. *Molecular Conformations. Part I. The Bicyclo[3,3,1]nonane System: X-Ray Analysis of* 1-p-*Bromobenzenesulphonyloxymethyl-5-methylbicyclo[3,3,1]nonan-9-ol* [1]

By W. A. C. Brown, J. Martin, and G. A. Sim

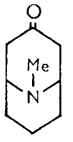
The molecular geometry of 1-*p*-bromobenzenesulphonyloxymethyl-5-methylbicyclo[3,3,1]nonan-9-ol has been determined by *X*-ray crystal analysis. The bicyclo[3,3,1]nonane system of the molecule adopts the twin-chair conformation but each ring deviates significantly from the ideal chair shape and is distinctly flattened. The C(3) · · · C(7) separation is 3·06 Å.

The geometry of the bicyclo[3,3,1]nonane system (I) presents interesting conformational problems [2] which have been investigated by several physical and chemical techniques in recent years. Nitrogen-containing analogues [3] have been studied in various laboratories but the observations have usually led only to deductions of a qualitative nature. In the case of the alkaloid ψ-pelletierine (II) different interpretations of dipole-moment and molecular-polarisability measurements have been proposed.[4,5]

In a systematic survey of the infrared spectra of a series of carbocyclic bicyclo[3,3,1]-nonane derivatives we have attributed absorption bands near 2990 and 1490 cm.$^{-1}$ to C(3),C(7)-methylene interactions present in the twin-chair conformer.[1,6] These infrared bands appear both in solution and in the solid, demonstrating that the molecular conformations are independent of environment and allowing *X*-ray crystallographic results to be extrapolated to molecular conformations in solution. From the results of the spectroscopic investigation and *X*-ray analysis of the derivative (III; R = O·SO_2·C_6H_4Br) we concluded [1] that those bicyclo[3,3,1]nonane compounds which have both three-carbon bridges fully saturated exist in twin-chair conformations both in the solid state and in solution.* The same conclusion has been deduced from analysis of the proton magnetic resonance spectra of certain more complex molecules featuring the bridged bicyclic structure.[8]



(I)



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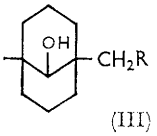

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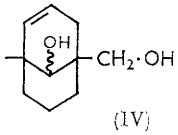

(IV)

The mono-*p*-bromobenzenesulphonyl ester (III; R = O·SO_2·C_6H_4Br) of the diol (III; R = OH) was prepared from the unsaturated diol (IV) [9] by catalytic hydrogenation followed by specific esterification of the primary hydroxyl group. The infrared spectra of the *p*-bromobenzenesulphonyl derivative in solution and in a crystal mull (see Experimental section) exhibit the absorption bands attributed [1] to the " scissoring " vibrational

* As expected, the conformation is very sensitive to non-bonded interactions introduced by various substitution patterns. We are currently studying compounds which possibly feature conformers in equilibrium in solution.[7]

[1] Preliminary communication, W. A. C. Brown, G. Eglinton, J. Martin, W. Parker, and G. A. Sim, *Proc. Chem. Soc.*, 1964, 57.
[2] E. L. Eliel, " Stereochemistry of Carbon Compounds," McGraw-Hill, New York, 1962, p. 295.
[3] H. O. House, H. C. Müller, C. G. Pitt, and P. P. Wickham, *J. Org. Chem.*, 1963, **28**, 2407, and references cited therein.
[4] N. J. Leonard, D. F. Morrow, and M. T. Rogers, *J. Amer. Chem. Soc.*, 1957, **79**, 5476.
[5] J. M. Eckert and R. J. W. Le Fèvre, *J.*, 1964, 358.
[6] G. Eglinton, J. Martin, and W. Parker, *J.*, 1965, 1243.
[7] J. Martin, Ph.D. Thesis, University of Glasgow, 1964.
[8] N. J. W. Pumphrey and M. J. T. Robinson, Paper presented to Congress of I.U.P.A.C., July 1963.
[9] J. Martin, W. Parker, and R. A. Raphael, *J.*, 1964, 289.

mode of the crowded C(3),C(7)-methylene groups in the twin-chair conformer. The crystal structure was elucidated by means of the usual phase-determining heavy-atom method,[10] and the atomic co-ordinates were refined by Fourier and least-squares calculations. At the end of the analysis the value of R, the average discrepancy between measured and calculated structure amplitudes, was 12·9% over 1633 reflexions.

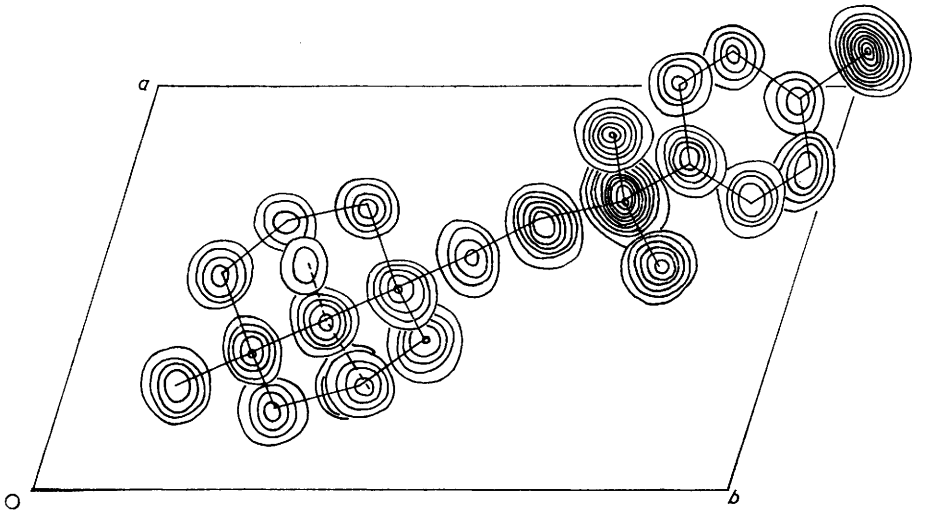

FIG. 1. The final three-dimensional electron-density distribution shown by means of superimposed contour sections drawn parallel to (001). Contour interval 1 eÅ^{-3} for the carbon and oxygen atoms, starting at the 2 eÅ^{-3} level, 2 eÅ^{-3} for the sulphur atom, and 4 eÅ^{-3} for the bromine atom

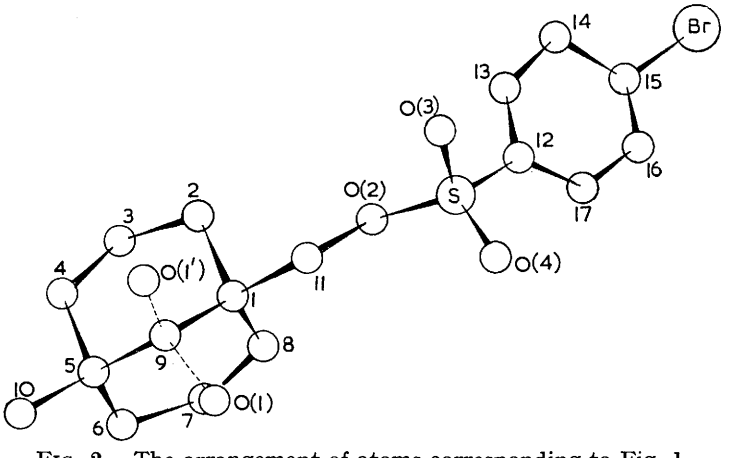

FIG. 2. The arrangement of atoms corresponding to Fig. 1

The final electron-density distribution is shown in Fig. 1 as superimposed contour sections drawn parallel to (001); the corresponding atomic arrangement is explained in Fig. 2. Further views of the molecule as seen in projection on (100) and (010) are shown in Figs. 3 and 4. The final atomic co-ordinates with their estimated standard deviations are given in Table 1, and the interatomic distances and valency angles calculated from these co-ordinates are listed in Table 2. In order to analyse the conformation of the molecule in

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

TABLE 1

Atomic co-ordinates (as fractions of the cell edges) and their estimated standard deviations (Å)

Atom	x/a	y/b	z/c	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	Atom	x/a	y/b	z/c	(σx)	$\sigma(y)$	$\sigma(z)$
C(1)	0·5149	0·4307	0·2186	0·018	0·017	0·018	C(13)	1·0083	0·7481	0·3809	0·020	0·017	0·023
C(2)	0·7011	0·3530	0·1666	0·019	0·019	0·020	C(14)	1·0827	0·8101	0·3434	0·022	0·019	0·022
C(3)	0·6584	0·2407	0·0483	0·023	0·020	0·023	C(15)	0·9715	0·9188	0·3141	0·021	0·017	0·023
C(4)	0·5351	0·1692	0·0572	0·021	0·017	0·020	C(16)	0·7836	0·9651	0·3352	0·021	0·018	0·019
C(5)	0·3523	0·2486	0·1132	0·017	0·016	0·019	C(17)	0·7077	0·9024	0·3764	0·021	0·018	0·018
C(6)	0·1974	0·3103	0·0150	0·018	0·018	0·020	O(1)	0·2564	0·4259	0·2984	0·022	0·021	0·022
C(7)	0·2590	0·4079	−0·0068	0·021	0·017	0·021	O(1′)	0·5677	0·2904	0·3390	0·029	0·024	0·026
C(8)	0·3631	0·4984	0·1238	0·019	0·016	0·018	O(2)	0·6549	0·6126	0·3308	0·012	0·010	0·012
C(9)	0·4206	0·3481	0·2418	0·018	0·015	0·017	O(3)	0·8779	0·6643	0·5518	0·015	0·012	0·013
C(10)	0·2597	0·1645	0·1394	0·020	0·017	0·021	O(4)	0·5613	0·7954	0·5111	0·015	0·014	0·016
C(11)	0·5763	0·5247	0·3525	0·022	0·017	0·021	S	0·7268	0·7162	0·4600	0·005	0·004	0·005
C(12)	0·8198	0·7893	0·3964	0·017	0·015	0·016	Br	1·0781	1·0024	0·2532	0·003	0·002	0·003

detail we calculated the mean planes through various sets of atoms; the deviations of the atoms from the planes are given in Table 3.

The displacement of C(3) from the plane through carbon atoms 1, 2, 4, and 5, 0·51 Å, is distinctly smaller than the displacement of the bridge atom C(9), 0·71 Å. In the other

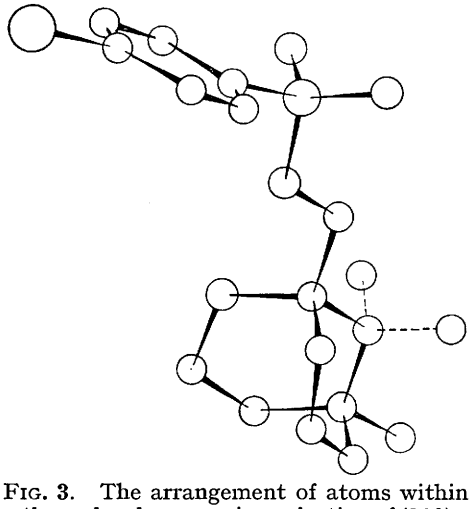

FIG. 3. The arrangement of atoms within the molecule as seen in projection of (100)

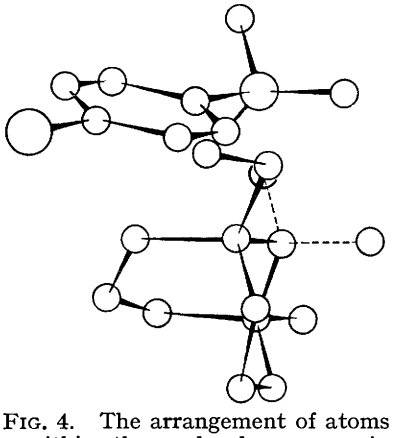

FIG. 4. The arrangement of atoms within the molecule as seen in projection on (010)

ring there is an identical pattern; C(7) is displaced by 0·45 Å from the plane through carbon atoms 1, 5, 6, and 8, whereas C(9) is displaced by 0·72 Å. If each cyclohexane ring adopted the ideal chair conformation with tetrahedral angles, all these displacements would be $1{\cdot}5445 \cos 54° 44' \sin 54° 44' = 0{\cdot}728$ Å. The flattening of the cyclohexane rings is also clearly shown in the displacements of the atoms from the plane through carbon atoms 1, 5, 9, 10, and 11; in the ideal twin-chair conformation the displacements of C(3) and C(7) would not differ from the displacements of C(2), C(4), C(6), and (C(8), whereas, in fact, the average displacement of atoms 3 and 7, 1·52 Å, is considerably greater than the average displacement of atoms 2, 4, 6, and 8, 1·30 Å.

The plane through atoms 2, 3, and 4 and that through atoms 6, 7, and 8 intersect the plane through atoms 1, 5, 9, 10, and 11 at angles of 17 and 18°, respectively; in the ideal twin-chair conformation these planes would be parallel. The angle between the plane through atoms 1, 5, 6, and 8 and the plane through atoms 1, 2, 4, and 5, 113°, is slightly greater than tetrahedral.

Although each cyclohexane ring of the bicyclo[3,3,1]nonane system can be described

TABLE 2

Interatomic distances (Å) and angles

Intramolecular bonded distances

C(1)–C(2)	1·57	C(5)–C(6)	1·54	C(11)–O(2)	1·48	C(15)–C(16)	1·40
C(1)–C(8)	1·55	C(5)–C(9)	1·53	C(12)–S	1·72	C(15)–Br	1·87
C(1)–C(9)	1·51	C(5)–C(10)	1·54	C(12)–C(13)	1·38	C(16)–C(17)	1·34
C(1)–C(11)	1·52	C(6)–C(7)	1·51	C(12)–C(17)	1·43	O(2)–S	1·57
C(2)–C(3)	1·52	C(7)–C(8)	1·57	C(13)–C(14)	1·30	O(3)–S	1·43
C(3)–C(4)	1·48	C(9)–O(1)	1·47	C(14)–C(15)	1·42	O(4)–S	1·45
C(4)–C(5)	1·58	C(9)–O(1′)	1·49				

Intramolecular non-bonded distances

C(1) · · · C(4)	2·97	C(3) · · · C(7)	3·06	C(4) · · · O(1′)	2·92	C(8) · · · · O(1)	2·84
C(1) · · · C(6)	2·97	C(3) · · · C(6)	3·19	C(5) · · · C(8)	2·99	C(8) · · · · O(1′)	3·81
C(1) · · · S	3·86	C(3) · · · C(8)	3·21	C(5) · · · C(11)	3·82	C(10) · · · O(1)	2·97
C(2) · · · C(7)	3·23	C(3) · · · C(9)	2·94	C(6) · · · O(1)	2·91	C(10) · · · O(1′)	2·93
C(2) · · · C(6)	3·65	C(3) · · · O(1′)	3·38	C(6) · · · O(1′)	3·82	C(11) · · · O(3)	2·98
C(2) · · · C(5)	2·99	C(4) · · · C(7)	3·18	C(7) · · · C(9)	2·95	C(11) · · · O(4)	3·06
C(2) · · · O(2)	2·95	C(4) · · · C(8)	3·65	C(7) · · · O(1)	3·39	C(11) · · · O(1)	2·75
C(2) · · · O(1)	3·79	C(4) · · · O(1)	3·85	C(8) · · · O(2)	2·83	C(11) · · · O(1′)	2·79
C(2) · · · O(1′)	2·87						

Intermolecular distances (<4Å)

O(4) · · · · O(1′)$_{I}$	2·81	C(16) · · · C(14)$_{V}$	3·64	O(3) · · · O(3)$_{VI}$	3·73	C(6) · · · C(14)$_{VII}$	3·82
O(3) · · · · O(1)$_{I}$	2·87	C(2) · · · · O(3)$_{VI}$	3·66	O(4) · · · O(1)$_{I}$	3·73	O(3) · · · Br$_{V}$	3·83
C(14) · · · O(4)$_{II}$	3·40	C(11) · · · O(1′)$_{I}$	3·67	C(12) · · Br$_{V}$	3·76	C(7) · · · C(7)$_{VIII}$	3·84
C(10) · · · Br$_{III}$	3·46	C(13) · · · O(1)$_{II}$	3·67	C(6) · · · C(15)$_{VII}$	3·76	C(7) · · · O(2)$_{VII}$	3·84
C(16) · · · O(4)$_{IV}$	3·55	C(15) · · · C(16)$_{V}$	3·67	C(9) · · · O(4)$_{I}$	3·77	C(15) · · C(15)$_{V}$	3·84
C(17) · · · O(4)$_{IV}$	3·56	S · · · · · · O(1′)$_{I}$	3·68	C(15) · · C(17)$_{V}$	3·78	S · · · · · Br$_{V}$	3·94
C(11) · · · O(1)$_{I}$	3·59	S · · · · · · O(1)$_{I}$	3·70	C(9) · · · O(3)$_{I}$	3·78	C(8) · · · C(8)$_{VII}$	3·99
C(10) · · · O(4)$_{I}$	3·62	C(13) · · · O(1′)$_{VI}$	3·71	O(4) · · · Br$_{V}$	3·79	C(7) · · · C(13)$_{VII}$	3·99
C(10) · · · O(3)$_{I}$	3·63	O(3) · · · · O(1′)$_{I}$	3·72	C(17) · · Br$_{V}$	3·80	C(13) · · O(4)$_{II}$	3·99

The subscripts refer to the following positions:

I	$-1-x, -1-y, 1-z$.	IV	$-1-x, -y, 1-z$.	VII	$-1-x, -1-y, -z$.
II	$1+x, y, z$.	V	$-x, -y, 1-z$.	VIII	$-2-x, -1-y, -z$.
III	$-1+x, -1+y, z$.	VI	$-x, -1-y, 1-z$.		

Valency angles

C(1)C(2)C(3)	113°	C(7)C(8)C(1)	112°	C(5)C(9)O(1′)	110°	SC(12)C(13)	121°
C(2)C(3)C(4)	116	C(8)C(1)C(2)	113	O(1)C(9)O(1′)	109	SC(12)C(17)	118
C(3)C(4)C(5)	115	C(8)C(1)C(9)	110	C(1)C(11)O(2)	107	C(17)C(12)C(13)	120
C(4)C(5)C(6)	112	C(8)C(1)C(11)	109	C(11)O(2)S	114	C(12)C(13)C(14)	120
C(4)C(5)C(9)	109	C(2)C(1)C(9)	109	O(2)SC(12)	100	C(13)C(14)C(15)	121
C(4)C(5)C(10)	109	C(2)C(1)C(11)	110	O(2)SO(3)	110	C(14)C(15)C(16)	120
C(10)C(5)C(6)	109	C(9)C(1)C(11)	106	O(2)SO(4)	110	C(14)C(15)Br	120
C(10)C(5)C(9)	110	C(1)C(9)C(5)	111	O(3)SC(12)	111	C(16)C(15)Br	120
C(6)C(5)C(9)	109	C(1)C(9)O(1)	107	O(4)SC(12)	111	C(15)C(16)C(17)	119
C(5)C(6)C(7)	115	C(1)C(9)O(1′)	108	O(3)SO(4)	114	C(16)C(17)C(12)	119
C(6)C(7)C(8)	114	C(5)C(9)O(1)	111				

TABLE 3

Deviations (Å) of atoms from various planes

(*a*) Plane through C(1), C(8), C(6), C(5). (*b*) Plane through C(1), C(2), C(4), C(5). (*c*) Plane through C(11), C(1), C(9), C(5), C(10). (*d*) Plane through C(12), C(13), C(14), C(15), C(16), C(17), S, Br.

	Deviation					Deviation			
Atom	(*a*)	(*b*)	(*c*)	(*d*)	Atom	(*a*)	(*b*)	(*c*)	(*d*)
C(1)	0·00	0·01	0·00		C(12)				−0·06
C(2)		−0·01	1·32		C(13)				−0·01
C(3)		0·51	1·54		C(14)				0·03
C(4)		0·01	1·30		C(15)				0·00
C(5)	0·00	−0·01	0·00		C(16)				0·02
C(6)	0·00		−1·28		C(17)				0·00
C(7)	0·45		−1·50		O(1)			−1·21	
C(8)	0·00		−1·29		O(1′)			1·21	
C(9)	−0·72	−0·71	+0·01		S				0·03
C(10)			0·01		Br				−0·01
C(11)			0·00						

as having a chair conformation, the strong steric interaction between the 3,7-methylene groups is clearly reflected in the increase of the C(3) · · · C(7) non-bonded separation from the ideal twin-chair value of 2·52 Å to the actual value of 3·06 Å and the concomitant flattening of the rings. The C(3) · · · C(7) separation does not differ very greatly from the values reported for distances between axial 1,3-methyl groups in triterpenoids (*e.g.*, 3·04 Å in cedrelone iodoacetate,[11] 3·17 and 3·30 Å in methyl melaleucate iodoacetate [12]). The equality of the distances C(2) · · · C(6) and C(4) · · · C(8), 3·65 Å, is evidence that a sideways (*i.e.*, parallel to the plane through carbon atoms 1, 5, 9, 10, and 11) displacement of the rings is not appreciably involved in the relief of strain.

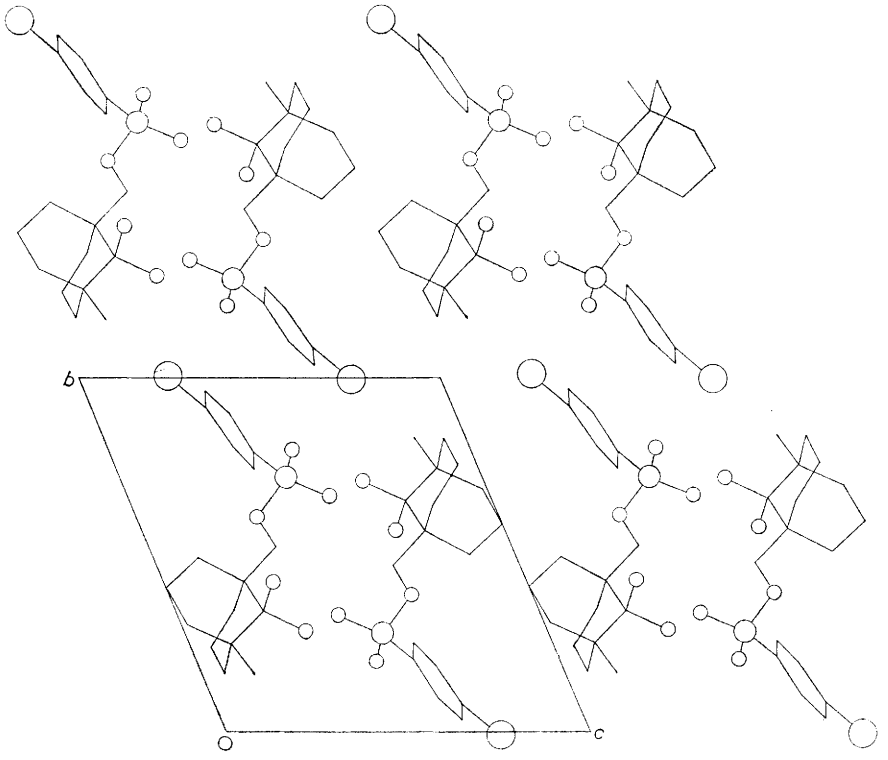

FIG. 5. The crystal structure as viewed in projection on (100)

The valency angles at the carbon atoms involved in the flattening of the rings, *i.e.*, atoms 2, 3, 4, 6, 7, and 8, are necessarily greater than tetrahedral; the average value is 114°. The angles C(4)C(5)C(6), 112°, and C(2)C(1)C(8), 113°, are also rather greater than tetrahedral. The other valency angles at carbon atoms 1 and 5 are close to tetrahedral.

In an attempt to locate the hydrogen atoms we evaluated a three-dimensional difference electron-density distribution, using Fourier coefficients ($F_o - F_c$), where F_c are structure factors calculated on the basis of the atoms other than hydrogen. Peaks were located in stereochemically acceptable regions but we were unable to assign accurate co-ordinates; the overcrowded hydrogen atoms associated with the C(3),C(7)-methylene groups were only diffusely visible.

In the ideal twin-chair conformation with all angles tetrahedral the H · · · H separation across the molecule at the C(3),C(7)-methylene groups would be only 0·75 Å, an impossibly low value. Provided the HCH angles remain tetrahedral with the C–H bonds in their

[11] I. J. Grant, J. A. Hamilton, T. A. Hamor, J. M. Robertson, and G. A. Sim, *J.*, 1963, 2506.
[12] C. S. Chopra, N. W. Fuller, K. J. L. Thieberg, D. C. Shaw, D. E. White, S. R. Hall, and E. N. Maslen, *Tetrahedron Letters*, 1963, 1847; S. R. Hall and E. N. Maslen, *Acta Cryst.*, 1965, **18**, 265.

ideal positions, the carbon framework found in this analysis corresponds to an H · · · H separation at the C(3),C(7)-methylene groups of about 1·7 Å. It is possible, of course, that the HCH angles are not tetrahedral, for the CCC angles at atoms 3 and 7 are about 115°. If the bonding orbitals of an atom A in the system AX_2Y_2 point along the internuclear directions and are orthogonal *s*,*p*-hybrids constructed from one *s*-orbital and three *p*-orbitals, the angles XAX (2α) and YAY (2θ) are related by the equation

$$\tan \alpha = (1 - \cot^2 \theta)^{-\frac{1}{2}}.$$

It follows that the HCH angles at carbon atoms 3 and 7 may be about 105° and the H · · · H separation at the C(3),C(7)-methylene groups about 1·8 Å. Transannular H · · · H contacts of from 1·8 Å upwards have been reported in cyclononylammonium bromide [13] and 1,6-*trans*-diaminocyclodecane dihydrochloride.[14]

The molecular dimensions obtained by Dobler and Dunitz for 3-azabicyclo[3,3,1]-nonane hydrobromide [15] are in good accord with the results described above; it can be concluded that substitution of N^+ for C at position 3 causes no significant change in the conformation of the bicyclo-system.

The oxygen atom of the secondary hydroxyl group persistently appeared in the electron-density distributions as two distinct peaks, one on either side of the plane through carbon atoms 1, 5, 9, 10, and 11 (see Fig. 1). The appearance of the oxygen atom in both stereochemically acceptable sites indicates that in the crystal the (+)- and (−)-molecules do not occupy uniquely their respective positions *x*, *y*, *z* and $\bar{x}$, $\bar{y}$, $\bar{z}$, but are distributed randomly over both sites. Packing disorder is by no means rare; somewhat similar examples occur in the crystal structures of, for example, 2-amino-4-chloro-6-methylpyrimidine,[16] di-indenyliron,[17] acepleiadylene,[18] azulene,[19] and *p*-bromochlorobenzene.[20] The disorder did not lead to any marked ellipticity of atomic peaks in the electron-density distributions (see Fig. 1), demonstrating that the (+)- and (−)-molecules in the racemate are very closely superposable except for the oxygen substituent on C(9), the only asymmetric centre in the molecule. The close superposition of the carbon frameworks of the (+)- and (−)-molecules is, of course, intimately connected with our conclusion that a sideways displacement of the rings is not an important factor in the molecular geometry.

The arrangement of the molecules in the crystal as seen in projection on (100) is shown in Fig. 5. The shortest approach distances between molecules, $O(4) \cdots O(1')_I$, 2·81 Å, $O(3) \cdots O(1)_I$, 2·87 Å, represent hydrogen-bonded contacts. All other approach distances (see Table 2) appear to be normal van der Waals interactions.

Experimental

1-Hydroxymethyl-5-methylbicyclo[3,3,1]*nonan-9-ol* (III; R = OH). A solution of the unsaturated diol (IV) [9] (0·42 g.) in ethyl acetate (5 ml.) was hydrogenated over 10% palladium–charcoal (0·1 g.). The hydrogen uptake after 1 hr. at room temperature corresponded to 1 mol. equivalent. The *diol* (III; R = OH) (0·40 g.) recovered from the reaction mixture crystallised from benzene–light petroleum as prisms, m. p. 105—106° (Found: C, 71·55; H, 10·75. $C_{11}H_{20}O_2$ requires C, 71·7; H, 10·95%).

The primary *mono*-p-*bromobenzenesulphonate* (III; R = $O{\cdot}SO_2{\cdot}C_6H_4Br$), prepared in the usual manner from the diol with *p*-bromobenzenesulphonyl chloride in pyridine, crystallised from ether as prisms, m. p. 116—118° (Found: C, 50·9; H, 5·85; Br, 19·8. $C_{17}H_{23}BrO_4S$ requires C, 50·6; H, 5·75; Br, 19·8%; $\nu_{max.}$ (Nujol) 3536 (inter-bonded OH), 2994, and 1486

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cm.$^{-1}$ (crowded-methylene bands); (in CCl_4 at high dilution) 3638 (free OH), 3583 (intra-bonded hydroxyl), 2992 and 1491 cm.$^{-1}$ (crowded-methylene bands).

Crystal Data.—1-*p*-Bromobenzenesulphonyloxymethyl-5-methyl-bicyclo[3,3,1]nonan-9-ol, $C_{17}H_{23}BrO_4S$; $M = 403{\cdot}3$. Triclinic, $a = 7{\cdot}40$, $b = 12{\cdot}06$, $c = 11{\cdot}34$ Å, $\alpha = 112°\ 30'$, $\beta = 109°\ 31'$, $\gamma = 72°\ 41'$, $U = 862$ Å^3, $D_m = 1{\cdot}53$, $Z = 2$, $D_c = 1{\cdot}55$ g.cm.$^{-3}$. Space group $P\bar{1}$. $F(000) = 416$. Absorption coefficient for *X*-rays ($\lambda = 1{\cdot}542$ Å) $\mu = 48{\cdot}1$ cm.$^{-1}$.

Crystallographic Measurements.—The unit-cell parameters were evaluated from precession photographs taken with molybdenum K_α radiation ($\lambda = 0{\cdot}7107$ Å). For the intensity measurements small crystals were employed, completely bathed in a uniform *X*-ray beam; absorption effects were neglected. The intensities were estimated visually from multiple-fim equi-inclination Weissenberg photographs of the 0*kl*, 1*kl*, and *hk*0—*hk*7 layers, taken with copper K_α radiation, and from a timed series of precession photographs of the *h*0*l* zone, taken with molybdenum K_α radiation. The various layers were placed on the same relative scale by comparison of common reflexions on different photographs. The absolute scale was obtained at a later stage by correlation of the measured structure amplitudes, F_o, with the calculated values, F_c. In all, 1633 independent structure amplitudes were derived from the intensity measurements (Table 5). The crystal density was determined by flotation in aqueous potassium iodide.

TABLE 4

Anisotropic temperature-factor parameters ($b_{ij} \times 10^5$)

	b_{11}	b_{22}	b_{33}	b_{23}	b_{13}	b_{12}		b_{11}	b_{22}	b_{33}	b_{23}	b_{13}	b_{12}
C(1)	3753	1178	1144	−277	1340	−2268	C(13)	3317	1085	2854	1404	456	−1459
C(2)	3104	1469	1648	516	1164	−1071	C(14)	4329	1038	2111	720	2002	157
C(3)	5153	1289	2062	−307	2796	−1427	C(15)	5108	942	2293	198	268	−3291
C(4)	3475	1040	1800	1113	1072	533	C(16)	5047	1580	1525	1996	1590	−987
C(5)	2490	1156	1540	962	371	−1088	C(17)	4727	1403	1121	1183	1028	−1245
C(6)	2685	1574	1906	1316	459	−1748	O(1)	2633	976	1066	221	1269	−984
C(7)	4961	1113	1764	946	437	−1674	O(1′)	5029	1250	1321	929	−346	−1224
C(8)	3941	1103	941	179	1009	−1809	O(2)	5522	1200	1434	373	905	−3745
C(9)	3362	858	923	356	−73	−1238	O(3)	5234	1644	1438	1242	332	−2228
C(10)	4035	917	1893	223	860	−1549	O(4)	4486	1702	2451	414	2403	−1283
C(11)	6490	1039	1659	401	491	−3207	S	3780	1049	1031	162	865	−1910
C(12)	3146	868	1060	745	1637	−530	Br	5388	1458	1668	679	1485	−2743

Structure Analysis.—Initial co-ordinates for the bromine and sulphur atoms were derived from inspection of two- and three-dimensional Patterson syntheses. The positions of the carbon atoms directly bonded to the bromine and sulphur atoms were estimated by assuming standard lengths for the carbon–sulphur and carbon–bromine bonds. The value of *R* for structure factors calculated on the basis of these four atoms was 41·4%.

The first three-dimensional electron-density distribution revealed all the atoms, apart from hydrogen, and it was immediately obvious that the molecule did indeed adopt the anticipated chair–chair conformation and that the rings were distinctly flattened.

Co-ordinates were assigned to all the atoms by numerical interpolation in the electron-density values, and these co-ordinates were then utilized in the calculation of a second set of structure factors. The value of *R* was 27·2%.

Four further rounds of structure-factor and Fourier calculations, with back-shift corrections for termination of series derived at each stage from an F_c synthesis, reduced the value of *R* to 20·1%. During the refinement the relative scattering factors of atoms O(1) and O(1′) were varied (the sum of these scattering factors was kept equal to the scattering factor of an oxygen atom) in an attempt to determine whether or not there was a completely random distribution of the oxygen atom over the two sites. From a comparison of the peak heights of these atoms in the F_o and F_c syntheses we concluded that both sites were equally occupied.

The refinement was concluded by least-squares adjustment of the positional and anisotropic thermal parameters of the atoms; the programme employed was that devised by Dr. J. S. Rollett.[21] After six cycles of calculations the value of *R* fell to 12·9%. The weighting scheme was then altered and two further rounds of calculations were completed; the value of *R* remained constant at 12·9% and no significant changes of co-ordinates occurred.

[21] J. S. Rollett, in " Computing Methods and the Phase Problem in X-Ray Crystal Analysis," ed. R. Pepinsky, J. M. Robertson, and J. C. Speakman, Pergamon, Oxford, 1961, p. 87.

TABLE 5

Measured and calculated values of the structure factors

h	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c
0	0	2	31.2	- 34.4			1	2.5	2.0	0	- 7	2	24.1	- 22.7
		3	32.0	29.1			2	19.9	- 19.2			3	2.2	3.2
		4	31.6	33.8			3	5.3	4.4			4	32.3	31.2
		5	15.9	- 12.5			4	7.0	- 4.6			5	8.6	6.8
		6	6.6	- 8.3			5	8.4	8.2			6	14.9	- 15.5
		7	2.3	0.3			6	16.2	- 17.7			7	5.1	5.9
		8	19.1	19.5			7	10.0	8.8			8	12.2	11.0
		9	12.7	11.0			- 1	10.9	8.7			9	9.7	7.8
		10	19.1	- 18.8			- 3	8.0	- 5.4			10	13.4	- 13.1
		11	3.4	3.2	0	9	0	9.6	7.2			11	6.4	6.5
		12	2.0	0.7			1	5.3	- 4.1	0	- 8	2	24.8	- 23.7
0	1	1	28.4	- 22.2			2	23.5	- 21.8			4	18.7	21.9
		2	123.0	-128.9			3	2.7	0.1			5	17.3	- 16.5
		3	23.8	18.3			4	13.0	14.1			6	6.9	- 6.1
		4	21.8	23.3			5	2.5	2.0			7	11.1	- 12.3
		5	17.5	- 17.3			6	7.0	- 8.6			8	25.2	26.3
		6	31.8	- 33.2			- 1	8.5	5.9			9	6.6	- 6.5
		7	6.1	3.2	0	10	0	16.8	17.2			10	8.5	- 8.4
		8	6.4	8.2			1	18.8	- 16.1			11	2.3	2.9
		9	11.1	- 9.4			2	4.6	3.5	0	- 9	2	20.0	- 18.2
		10	12.6	- 13.9			3	5.2	4.3			3	2.6	1.9
0	2	0	5.0	- 1.8			4	9.9	9.3			4	17.4	15.5
		1	43.8	53.6	0	11	0	13.0	14.2			5	14.7	13.7
		2	111.7	-126.4			1	2.6	1.5			6	22.3	- 26.0
		3	10.9	12.1			2	7.6	- 9.3			7	2.7	- 2.8
		4	17.7	20.6			3	4.0	3.1			8	13.7	14.7
		5	4.1	- 4.6			- 1	14.6	- 16.7			9	2.6	- 4.8
		6	14.3	- 14.7	0	11	- 3	10.7	- 10.1			10	2.4	- 3.2
		7	2.5	- 3.8	0	12	0	10.3	10.1	0	-10	1	2.7	0.5
		8	19.6	20.4			1	7.3	5.0			2	16.1	- 16.2
		9	4.7	- 4.7			2	8.6	- 9.7			3	2.7	4.9
		10	2.5	- 1.9			- 1	12.9	13.4			4	12.4	11.0
		- 1	13.0	- 13.5	0	13	2	6.2	- 5.7			5	9.6	6.3
		- 3	41.9	42.9	0	- 1	2	28.6	26.6			6	8.0	- 10.6
0	3	0	57.2	59.5			4	35.4	37.3			7	7.1	7.6
		1	19.7	- 13.5			5	2.5	5.0			8	2.6	3.6
		2	41.6	- 42.9			6	27.0	- 32.3			9	5.5	3.8
		3	8.4	8.3			7	6.9	- 6.6			10	3.9	- 4.3
		4	72.8	86.2			8	26.3	26.3	0	-11	2	9.1	- 10.0
		5	18.7	- 16.8			9	12.8	- 13.9			4	12.5	11.5
		6	10.4	- 12.6			10	3.8	- 3.0			5	5.7	- 4.7
		7	8.2	8.1			11	4.4	- 3.3			6	6.4	- 6.5
		8	9.3	8.8			12	7.7	8.2			7	7.4	- 6.8
		9	3.7	3.2	0	- 2	2	80.1	- 96.3			9	3.2	- 4.8
		10	5.4	- 8.5			4	33.7	42.9			10	11.3	- 9.2
		- 1	21.4	- 23.5			5	2.9	- 1.6	0	-12	2	8.0	- 9.1
		- 3	6.2	- 6.1			6	28.1	- 32.9			3	6.7	- 5.2
0	4	0	64.1	58.5			7	7.4	8.5			4	5.0	6.6
		1	11.7	- 9.3			8	16.5	18.6			5	2.5	- 0.1
		2	6.5	- 8.3			9	3.7	0.8			6	7.9	- 8.5
		3	5.1	- 2.8			10	7.2	- 6.6	0	-13	1	5.2	3.3
		4	18.3	19.6			11	3.6	- 3.2			2	2.2	- 1.8
		5	2.3	- 0.4			12	9.9	9.2			4	2.2	0.9
		6	26.6	- 32.3	0	- 3	2	51.6	- 53.7			6	7.6	- 9.5
		7	8.4	8.6			4	25.0	26.6	0	-14	4	10.7	9.3
		8	8.1	8.2			5	28.0	27.3	1	0	0	4.4	8.3
		9	2.5	1.2			6	28.0	- 33.8			1	43.3	- 35.0
		10	10.6	- 10.4			7	7.7	8.3			2	99.9	- 89.9
		- 1	18.5	- 18.0			8	9.0	8.7			3	63.9	56.0
		- 3	9.0	- 9.7			9	10.9	9.8			4	24.1	23.3
0	5	0	46.1	40.0			10	25.2	- 24.1			5	10.5	- 8.5
		1	25.5	21.1			11	10.6	10.6			6	16.6	- 14.5
		2	50.0	- 52.7			12	4.5	4.9			7	41.3	38.1
		3	25.7	23.3	0	- 4	2	28.5	- 32.7			9	5.9	- 6.5
		4	12.3	13.4			4	26.3	25.7			10	10.3	- 10.5
		5	2.5	1.9			5	23.3	- 21.2			- 1	22.3	- 16.8
		6	15.5	- 18.5			6	15.9	- 18.9			- 2	7.2	- 0.4
		7	4.7	2.9			7	8.0	- 9.2			- 3	25.8	- 20.9
		8	8.3	9.1			8	30.2	27.8			- 4	34.3	33.0
		9	2.4	- 1.3			9	2.7	0.9			- 5	20.6	25.2
		- 1	6.1	- 4.8			10	8.9	- 7.1			- 6	43.0	- 43.5
		- 3	8.0	- 5.1			11	2.6	- 1.4			- 7	14.2	- 13.7
0	6	0	30.5	29.1			12	5.9	5.2			- 8	21.5	22.3
		1	10.9	8.9	0	- 5	2	34.7	- 33.7			-12	10.1	9.0
		2	34.6	- 31.2			4	44.7	53.0	1	1	0	11.6	16.6
		3	19.1	- 16.1			5	7.9	- 6.0			1	49.1	46.0
		4	30.8	31.7			6	14.8	- 18.4			2	109.1	-114.9
		5	16.3	- 16.8			7	11.1	- 11.7			3	44.8	52.0
		6	6.6	- 5.4			8	20.8	18.2			4	29.2	33.5
		7	2.7	- 0.6			9	14.7	- 15.0			5	17.1	- 15.8
		8	18.1	15.5			10	2.7	- 0.2			6	8.8	- 8.1
		- 3	9.8	- 9.4			11	4.4	- 2.9			7	8.5	- 9.5
0	7	0	28.4	24.0			12	14.4	13.9			8	11.6	12.9
		1	12.5	- 9.1	0	- 6	1	2.7	1.5			9	10.2	- 10.7
		2	10.5	- 10.4			2	19.1	- 22.3			- 1	10.9	14.2
		3	4.6	- 2.6			4	5.9	5.5			- 2	114.6	-119.9
		4	26.4	29.0			5	18.4	18.5			- 3	15.4	- 9.4
		5	7.6	- 5.2	0	- 6	6	29.7	- 35.3			- 4	13.2	2.9
		6	8.5	- 8.6			7	7.1	6.3			- 5	21.3	18.5
		7	2.5	2.0			8	12.0	10.1			- 6	41.2	- 36.9
		8	2.2	1.9			9	8.1	7.7			- 7	9.2	- 11.3
		- 1	12.2	11.2			10	29.3	- 27.2			- 8	10.7	15.5
0	8	0	17.2	16.1			11	2.5	1.2			- 9	5.1	2.4

TABLE 5 (*Continued*)

h	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c
		-10	10.6	- 12.4			-10	12.9	- 11.9			- 1	18.7	15.2
		-11	10.2	- 12.5			-11	7.2	7.9			- 2	41.6	- 42.1
1	2	0	51.3	70.8	1	7	0	24.5	21.1			- 3	16.0	- 13.0
		1	12.2	- 12.4			1	18.0	- 15.2			- 4	40.7	43.7
		2	25.5	- 31.9			2	20.3	- 21.5			- 5	8.7	1.0
		3	42.9	40.4			3	13.5	14.1			- 6	11.1	- 8.8
		4	85.7	87.7			6	13.4	- 15.3			- 8	15.8	18.7
		5	53.3	- 50.1			7	9.3	10.8			-9	19.1	21.5
		6	9.4	- 11.6			- 1	24.8	22.1			-10	10.4	- 12.8
		7	13.2	15.8			- 2	28.1	- 26.6	1	- 2	0	27.4	26.7
		8	5.9	4.9			- 3	15.5	- 18.2			1	12.5	- 13.3
		- 1	27.8	23.8			- 4	30.0	28.4			2	36.5	- 30.9
		- 2	13.4	8.6			- 5	8.3	- 7.4			3	35.9	- 36.9
		- 3	22.6	- 26.3			- 6	7.9	- 8.3			4	20.9	23.9
		- 4	61.6	61.8			- 7	22.8	- 22.5			5	48.2	- 43.4
		- 5	11.9	13.1			- 8	43.1	41.7			6	7.0	- 5.5
		- 6	38.0	- 27.8			- 9	5.3	- 5.5			7	10.3	- 10.1
		- 7	14.2	- 13.5			-10	9.1	- 7.0			8	17.8	16.3
		- 8	10.5	10.9	1	8	1	14.7	- 13.9			9	14.4	- 18.3
		- 9	18.5	18.0			2	21.9	- 25.4			- 1	39.3	37.6
		-10	21.5	- 22.0			3	10.7	13.1			- 2	51.6	- 65.9
1	3	0	47.5	53.5			4	12.1	14.4			- 3	14.6	- 12.0
		1	21.7	- 23.0			6	7.3	- 9.4			- 4	24.0	25.0
		2	27.4	26.4			- 1	10.9	7.6			- 5	17.5	17.9
		3	47.4	- 39.8			- 2	24.3	- 19.3			- 6	34.0	- 38.6
		4	18.7	24.4			- 3	19.5	- 17.9			- 7	10.3	6.3
		5	18.7	- 21.3			- 4	13.9	12.9			- 8	10.3	14.7
		6	36.9	- 39.5			- 5	27.3	26.7			- 9	5.3	- 5.4
		7	18.8	23.3			- 6	36.8	- 40.7			-10	8.8	- 12.7
		- 1	7.1	5.4			- 7	30.2	- 29.1	1	- 3	0	58.9	66.7
		- 2	24.4	- 20.8			- 8	21.2	19.3			1	23.4	17.5
		- 3	61.5	- 65.8			- 9	7.5	- 7.6			2	36.2	- 39.3
		- 4	62.6	63.5	1	9	0	29.8	27.0			3	71.8	75.6
		- 5	14.1	16.9			1	21.0	- 16.6			4	37.0	34.4
		- 6	19.2	- 20.0			2	16.2	14.6			5	48.7	- 41.8
		- 7	19.8	- 19.7			3	10.9	10.9			6	24.9	- 22.2
		- 8	31.0	27.5			4	9.4	9.6			7	11.9	14.4
		- 9	8.8	4.9			5	10.8	- 11.2			8	8.0	5.8
		-10	16.6	- 16.2			- 1	18.7	16.5			10	8.2	- 6.4
1	4	0	24.2	25.7			- 2	22.3	- 20.9			- 1	8.2	8.7
		1	11.3	- 11.7			- 3	10.6	- 9.2			- 2	23.5	- 29.2
		2	69.4	- 76.9			- 4	23.7	21.8			- 3	25.4	- 7.3
		3	19.9	23.8			- 5	23.5	22.3			- 4	17.2	21.5
		4	11.1	15.1			- 6	18.7	- 22.3			- 5	21.6	24.7
		6	17.7	- 19.7			- 7	18.7	21.2			- 7	12.8	- 16.9
		7	11.3	14.6			- 8	7.5	- 6.1			- 8	12.9	17.8
		8	5.8	9.8			-10	8.7	- 6.2	1	- 4	0	41.1	42.1
		9	7.6	- 9.9	1	10	0	15.6	16.2			1	26.9	- 32.8
		- 1	45.6	45.5			2	13.6	- 15.2			2	37.9	- 39.3
		- 2	52.3	- 54.3			- 1	7.2	5.5			3	40.3	41.8
		- 3	6.0	2.2			- 2	5.9	- 6.6			4	18.4	19.4
		- 4	25.1	23.1			- 3	20.6	- 19.1			5	32.1	26.9
		- 5	21.3	24.2			- 4	27.6	24.6			6	29.3	- 27.9
		- 6	17.5	- 21.8			- 5	5.2	3.7			7	12.3	13.5
		- 7	17.1	- 17.6			- 6	12.6	- 13.8			8	5.7	4.2
		- 8	35.0	29.2			- 8	10.5	13.5			9	10.2	- 0.3
		- 9	10.2	- 9.7			-10	8.2	- 8.7			10	18.1	- 17.0
		-11	7.3	- 7.0			-11	6.0	- 5.1			- 1	5.4	2.2
1	5	0	18.8	17.2	1	11	1	6.1	- 5.1			- 2	18.1	- 21.3
		1	9.6	- 8.0			2	15.1	- 15.2			- 3	21.5	- 21.4
		2	26.8	- 29.4			3	9.4	9.6			- 4	37.4	50.1
		3	23.4	24.5			4	4.5	- 2.7			- 5	9.5	- 9.5
		4	14.9	18.6			- 1	20.5	19.6			- 6	15.1	- 20.6
		5	9.0	- 13.6			- 2	18.2	- 17.0			- 7	12.7	- 17.0
		6	8.6	- 9.2			- 3	15.7	- 13.2			- 8	7.5	12.8
		8	13.7	17.1			- 4	19.1	15.9			- 9	5.0	2.2
		9	12.1	- 11.2			- 7	6.5	- 5.3			-10	7.7	- 8.8
		- 1	29.3	24.1			- 8	10.0	10.2	1	- 5	0	41.6	34.6
		- 2	51.9	- 47.1			- 9	4.7	3.3			1	27.3	- 31.1
		- 3	20.6	- 20.8	1	12	0	4.6	7.5			2	16.6	- 19.9
		- 4	8.4	11.5			1	10.2	- 8.7			3	11.8	10.9
		- 5	33.5	35.0			- 1	5.9	5.3			4	15.3	15.8
		- 6	40.9	- 42.5			- 2	8.3	- 10.7			5	16.0	- 15.4
		- 7	5.6	- 4.7			- 3	9.3	7.7			8	21.1	21.6
		- 8	17.5	18.3			- 5	9.4	8.1			9	5.9	- 5.8
		- 9	18.6	14.9			- 6	12.1	- 10.5	1	- 5	- 1	12.8	9.4
		-10	30.2	- 27.5	1	13	0	5.5	7.4			- 2	46.6	- 45.8
		-11	7.3	- 5.8			- 3	14.5	- 10.7			- 5	9.2	9.2
1	6	0	43.5	33.5			- 4	9.3	8.2			- 6	18.7	- 21.2
		1	29.5	- 27.5			- 5	4.6	2.9			- 9	4.7	5.3
		2	6.2	- 6.3			- 6	11.5	- 9.8	1	- 6	0	15.8	14.6
		4	25.3	26.7	1	14	- 3	9.7	- 6.3			1	18.9	- 21.6
		5	9.5	- 11.2	1	- 1	0	157.8	164.9			2	20.1	- 24.7
		6	6.6	- 8.5			1	80.4	- 95.6			3	9.3	4.1
		7	5.7	6.9			2	57.0	- 57.9			4	19.0	23.4
		- 1	23.5	22.6			3	27.1	25.2			5	13.4	- 15.7
		- 2	10.3	- 12.7			4	27.1	25.7			6	11.8	- 13.7
		- 3	27.0	- 32.7			5	17.2	- 17.2			7	15.0	3.7
		- 4	38.5	40.4			6	24.1	- 24.4			8	11.7	11.0
		- 5	24.2	21.0			7	14.2	14.6			9	10.2	- 10.0
		- 6	24.4	- 25.5			8	17.9	20.0			- 1	19.3	19.3
		- 8	9.9	8.9			9	8.3	5.0			- 2	32.9	- 34.2
		- 9	18.9	17.3			10	8.1	- 10.2			- 3	10.8	12.0

TABLE 5 (*Continued*)

h	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c
		- 4	8.4	9.5			7	17.7	19.5			2	9.8	- 11.9
		- 5	11.7	13.4			- 1	20.5	27.6			3	9.8	10.3
		- 6	10.4	- 13.3			- 2	26.3	- 22.8			- 1	8.3	8.5
		- 7	5.2	- 3.6			- 3	48.2	- 34.8			- 3	37.3	- 36.9
		- 8	6.8	7.0			- 4	37.5	23.0			- 4	25.7	22.3
		- 9	8.3	8.0			- 5	48.3	30.0			- 5	13.0	14.1
1	- 7	0	17.5	17.2			- 6	11.1	- 11.5			- 6	18.4	- 17.8
		1	8.1	- 7.8			- 7	20.8	- 19.5			- 7	7.5	- 9.2
		2	15.9	- 19.2	2	2	0	37.3	37.4	2	10	0	9.9	- 9.2
		3	8.0	8.9			1	32.6	- 31.1			1	13.5	- 11.0
		4	8.4	9.1			4	18.8	17.1			2	9.6	- 10.0
		5	12.1	3.9			5	8.8	- 7.9			3	13.3	14.1
		6	21.8	- 25.6			6	16.9	- 19.6			- 1	35.3	32.5
		7	9.7	11.8			7	29.1	30.3			- 2	18.7	- 19.6
		10	9.4	- 8.1			- 1	25.9	28.6			- 3	34.5	- 32.0
		- 1	16.8	14.5			- 2	2.7	- 2.4			- 4	19.5	15.8
		- 2	8.7	- 10.4			- 3	92.9	- 82.4			- 5	7.4	- 9.3
		- 3	19.8	- 19.6			- 4	51.1	37.3			- 7	19.1	- 18.4
		- 4	22.0	25.7			- 5	42.4	30.2	2	11	0	11.0	12.0
		- 5	5.3	- 7.3			- 6	11.6	- 8.4			1	16.7	- 16.4
1	- 8	0	22.0	21.6			- 7	31.0	- 27.7			3	8.4	9.0
		1	6.7	- 3.7	2	3	0	2.8	2.7			- 1	22.6	21.8
		2	11.0	- 11.4			2	53.6	- 48.4			- 2	14.8	- 14.5
		3	7.6	- 7.4			3	37.6	37.7			- 3	10.4	11.3
		4	21.0	24.6			6	17.7	- 19.3			- 4	6.3	5.7
		5	5.7	- 6.5			7	13.4	18.3			- 7	8.1	- 8.7
		6	5.8	- 4.5			- 1	60.9	64.0	2	12	0	10.1	10.3
		7	7.9	9.7			- 2	39.4	- 44.6			1	15.0	- 14.0
		8	8.2	5.8			- 3	10.1	6.4			- 2	5.8	- 4.2
		10	7.2	- 7.3			- 4	9.9	- 4.6			- 4	6.2	5.0
		- 1	10.1	0.7			- 5	31.5	39.1			- 5	19.9	20.3
		- 2	7.0	- 3.8			- 6	26.5	- 24.4			- 6	6.1	- 6.0
		- 3	7.2	- 8.1			- 7	20.7	- 25.8			- 7	7.9	- 9.3
		- 4	12.2	16.5	2	4	0	4.3	4.6	2	13	0	3.9	5.5
1	- 9	0	9.9	9.7			1	18.0	- 18.5			- 7	5.1	- 9.1
		1	6.0	- 4.1			2	9.7	- 11.1	2	- 1	0	34.9	28.8
		2	11.3	- 11.9			3	31.6	32.2			1	3.7	- 7.1
		3	8.2	4.4			5	14.1	- 17.3			2	34.6	- 31.8
		4	6.0	6.4			- 1	43.0	48.8			3	53.8	49.9
		5	19.3	- 23.8			- 2	62.3	- 59.6			4	15.5	16.4
		8	5.6	7.9			- 3	39.2	- 43.2			6	12.8	- 11.6
		10	4.7	- 7.4			- 4	5.4	9.9			7	38.2	40.4
1	- 9	- 1	7.4	2.9			- 5	44.5	58.2			- 1	65.5	51.3
		- 2	15.3	- 15.3			- 6	26.3	- 34.8			- 2	52.4	39.1
		- 3	6.1	0.0			- 7	10.6	- 14.1			- 3	33.4	- 31.1
		- 6	9.5	- 11.0	2	5	0	29.4	30.8			- 4	59.5	49.0
1	-10	0	7.0	7.4			1	50.5	- 50.6			- 5	15.0	10.0
		1	9.5	7.8			4	20.7	19.7			- 6	7.7	- 10.2
		2	21.6	- 19.4			5	10.5	- 12.9			- 7	19.1	- 19.0
		3	17.5	16.8			- 1	22.9	25.6	2	- 2	0	34.1	28.6
		4	9.6	9.5			- 2	49.8	38.8			1	24.0	- 28.5
		5	5.4	3.0			- 3	82.5	- 89.7			2	23.9	- 22.3
		6	10.8	- 12.2			- 4	33.1	29.5			3	34.0	34.0
		- 1	9.3	7.8			- 5	9.8	13.8			4	29.7	29.7
		- 2	7.8	- 9.9			- 6	29.8	- 33.8			5	21.6	- 21.4
		- 3	4.9	0.4			- 7	12.3	- 12.4			6	11.5	- 11.4
		- 4	6.4	7.4	2	6	0	21.3	23.8			7	23.4	22.3
1	-11	0	11.6	11.1			1	25.0	- 28.0			- 1	40.0	41.1
		3	7.0	8.3			3	25.8	32.8			- 2	26.5	- 28.1
		4	5.6	4.3			5	10.9	- 10.7			- 3	53.0	- 47.4
		- 1	8.8	- 7.6			6	7.2	- 8.2			- 4	14.8	16.8
1	-12	0	5.9	5.6			- 1	25.5	29.9			- 5	20.0	20.4
		4	11.5	8.2			- 2	15.2	- 17.7			- 6	24.4	- 24.9
1	-13	0	4.6	3.7			- 3	8.1	- 13.2			- 7	17.7	- 17.8
2	0	0	9.3	- 1.5			- 4	53.1	49.5	2	- 3	0	4.4	- 3.8
		1	51.2	- 58.0			- 6	7.7	- 6.9			1	26.2	- 25.6
		2	13.9	- 17.9			- 7	20.1	- 28.3			2	22.6	- 26.7
		3	23.3	24.7	2	7	0	12.9	- 11.8			3	15.1	- 9.7
		4	13.9	12.8			1	13.1	- 14.3			4	18.6	24.6
		5	24.0	- 24.9			2	25.5	- 23.6			5	36.5	- 33.0
		6	20.0	- 21.6			3	17.4	20.4			6	8.4	9.0
		7	17.7	- 16.0			5	7.8	- 10.5			- 1	34.8	32.6
		8	14.3	10.3			- 1	43.2	48.9			- 2	36.6	- 38.6
		9	14.4	- 13.3			- 2	30.7	- 32.9			- 3	21.6	- 26.7
		- 1	45.5	47.7			- 3	18.7	- 24.6			- 4	6.4	- 6.2
		- 2	82.9	- 86.2			- 4	6.6	6.9			- 5	25.2	21.1
		- 3	18.1	- 18.7			- 5	37.1	49.7			- 6	42.8	- 48.4
		- 4	39.0	36.7			- 6	22.5	- 24.8			- 7	10.0	7.4
		- 5	30.5	27.4			- 7	28.4	- 35.7	2	- 4	0	32.5	32.2
		- 6	19.7	- 20.1	2	8	0	11.9	12.0			1	13.7	- 15.8
		- 7	13.1	- 13.5			1	30.4	- 29.2			2	24.6	- 29.2
		- 8	9.6	8.7			2	11.5	12.5			3	48.0	46.8
		- 9	9.9	10.1			3	6.9	10.9			5	26.5	- 25.6
		-10	10.1	- 8.3			4	6.3	4.2			6	8.7	- 7.2
		-11	20.4	- 16.9			- 1	25.4	22.5			7	12.6	13.8
		-12	14.3	11.4			- 2	18.7	- 17.7			- 1	6.8	4.6
2		0	38.6	41.2			- 3	20.7	- 23.4			- 2	12.4	- 13.3
		1	47.6	- 36.0			- 4	21.4	17.3			- 3	19.7	- 20.3
		2	22.5	- 20.0			- 5	35.0	31.6			- 4	5.1	8.6
		3	25.7	24.4			- 6	16.7	- 16.5			- 5	25.0	23.1
		4	33.4	36.4			- 7	19.0	22.8			- 6	21.5	21.0
		5	54.9	- 60.2	2	9	0	19.5	21.8			- 7	10.7	- 10.9
		6	6.0	3.4			1	21.0	- 15.7	2	- 5	0	18.5	18.2

TABLE 5 (*Continued*)

h	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c
		1	27.5	- 27.4	3	3	1	31.3	- 28.2			- 7	10.0	- 13.5
		2	14.8	- 14.4			3	23.3	22.7	3	12	0	6.2	6.7
		3	17.7	19.1			4	17.6	17.5			1	11.2	- 11.3
2	- 5	5	9.5	- 10.0			5	27.3	- 26.7			- 1	5.8	4.8
		6	17.2	- 14.0			6	7.1	10.0			- 4	6.2	- 5.4
		- 1	11.9	12.6			- 1	46.7	44.4			- 6	8.5	6.0
		- 3	24.4	- 27.3			- 2	6.3	- 9.6			- 7	12.5	- 16.3
		- 4	38.6	42.8			- 3	29.0	- 34.0	3	13	0	3.7	- 2.0
		- 5	7.2	7.8			- 4	21.2	- 18.0			- 5	13.0	13.3
		- 7	13.6	- 17.6			- 5	39.1	53.3			- 7	10.3	- 12.2
2	- 6	0	18.8	16.2			- 6	25.6	- 19.4	3	- 1	0	18.7	- 19.4
		1	13.5	- 18.1			- 7	16.7	- 17.8			1	33.9	- 42.4
		3	5.2	8.4	3	4	0	3.5	4.6			2	17.9	19.0
		4	20.6	20.5			1	40.6	- 41.2			3	16.9	15.7
		5	14.3	- 16.3			2	13.4	13.5			5	13.5	- 12.2
		- 1	13.0	12.9			3	10.3	11.8			6	11.6	- 11.9
		- 5	10.8	13.6			4	5.8	3.0			- 1	45.3	42.5
		- 6	14.5	- 18.4			5	18.5	- 20.6			- 2	59.6	- 54.0
2	- 7	0	4.6	5.1			- 1	27.3	29.4			- 3	49.6	- 47.1
		1	5.7	- 6.5			- 2	60.9	50.7			- 4	29.3	27.1
		2	13.6	- 14.5			- 3	61.2	- 67.3			- 5	33.3	27.6
		3	9.6	14.6			- 4	18.3	15.1			- 6	7.5	- 10.5
		- 1	23.0	22.6			- 5	19.1	23.3			- 7	11.0	- 12.0
		- 2	14.8	- 15.2			- 6	37.2	- 20.5	3	- 2	0	6.0	- 5.6
		- 4	8.9	- 6.0			- 7	24.5	- 23.9			1	11.8	- 17.9
		- 5	11.0	12.9	3	5	1	35.5	- 30.8			2	10.3	- 10.2
		- 6	10.1	- 12.2			3	23.2	27.6			3	37.1	34.5
2	- 8	0	6.9	5.3			4	8.5	- 8.2			6	6.7	- 4.8
		2	8.3	- 9.3			- 1	26.2	30.8			7	18.9	22.4
		3	15.5	20.7			- 2	8.3	- 8.3			- 1	11.4	14.6
		- 2	9.7	- 10.3	3	5	- 3	19.3	- 25.2			- 2	35.5	30.4
2	- 9	0	11.1	9.6			- 4	69.1	52.1			- 3	24.3	- 27.1
		1	12.5	- 12.8			- 5	17.0	25.3			- 4	36.0	32.9
		2	6.1	- 5.5			- 6	12.0	15.6			- 6	11.8	11.0
		3	8.5	- 7.8			- 7	19.1	- 20.4			- 7	13.9	- 17.3
		4	12.6	17.9	3	6	0	34.2	- 37.6	3	- 3	0	11.7	12.1
		5	7.8	- 8.3			1	21.0	- 19.2			1	33.9	- 54.7
2	-10	0	4.8	5.4			2	10.8	- 12.8			2	5.4	- 5.3
		2	5.9	- 5.2			3	21.2	23.2			3	9.9	11.9
		5	10.6	- 13.5			5	11.1	- 13.4			4	5.4	3.4
		- 1	6.4	4.5			- 1	57.9	64.7			5	22.8	- 20.9
		- 2	6.4	- 8.6			- 2	21.7	- 32.7			7	7.8	5.9
2	-11	2	7.8	- 9.2			- 3	39.4	- 37.7			- 1	33.4	29.2
		3	9.4	11.3			- 4	21.8	- 20.8			- 2	5.2	2.9
		- 1	8.1	7.3			- 5	43.1	48.9			- 3	22.5	- 24.6
3	0	0	16.6	19.3			- 6	11.2	- 7.7			- 4	5.1	3.7
		1	37.5	- 38.9			- 7	21.4	- 23.2			- 5	21.5	24.6
		2	4.8	2.4	3	7	0	6.1	- 5.4			- 7	10.6	- 14.9
		3	13.1	14.1			1	27.5	- 29.0	3	- 4	0	9.8	9.7
		4	8.0	7.1			2	23.9	24.7			4	5.6	4.6
		5	36.4	- 39.4			3	9.7	11.2			5	19.2	- 17.0
		6	15.1	12.8			5	10.9	- 13.1			7	7.8	9.4
		7	12.6	11.4			- 1	19.0	19.5			- 1	44.4	41.5
		8	10.2	6.9			- 2	20.0	- 19.3			- 2	9.9	- 14.4
		- 1	48.3	48.3			- 3	28.2	- 31.1			- 5	17.6	18.3
		- 2	24.6	- 24.1			- 5	8.4	14.0			- 6	18.6	- 24.4
		- 3	15.9	- 18.2			- 6	9.4	- 6.5	3	- 5	0	13.7	- 13.2
		- 4	46.5	- 39.1			- 7	12.4	- 11.5			3	33.2	31.1
		- 5	32.5	29.5	3	8	0	48.3	48.1			7	7.9	9.2
		- 6	8.4	- 9.7			1	29.0	- 26.2			- 1	28.5	30.9
		- 7	14.0	- 11.0			3	12.1	16.5			- 2	6.2	- 9.8
		- 8	9.6	- 6.7			- 1	14.5	18.3			- 3	15.8	- 16.9
		- 9	22.2	24.2			- 2	16.8	18.1			- 6	7.2	7.6
		-10	14.3	- 10.8			- 3	30.7	- 36.4	3	- 6	1	15.4	- 17.9
		-13	9.6	5.5			- 4	9.3	9.8			5	10.9	- 9.5
3	1	0	17.7	18.1			- 5	14.9	16.2			- 1	6.3	7.5
		1	32.8	- 36.2			- 7	25.6	- 27.0			- 3	33.3	- 38.1
		2	4.8	4.2	3	9	0	28.0	- 25.6			- 4	13.9	15.4
		3	40.2	41.9			1	21.2	- 17.2	3	- 7	1	10.6	- 13.5
		4	7.3	- 6.7			2	6.9	- 5.7			4	6.2	8.5
		6	6.8	- 4.6			3	13.7	14.8			5	11.1	- 13.8
		7	17.4	18.5			4	8.3	- 7.2			- 1	11.6	- 9.6
		- 1	29.8	35.1			- 1	39.7	39.3			- 2	9.7	9.2
		- 2	4.0	5.0			- 2	8.1	6.1			- 3	7.0	- 6.8
		- 3	33.5	- 27.9			- 3	23.1	- 26.0			- 5	15.3	18.1
		- 4	27.9	25.1			- 4	8.2	8.2	3	- 8	3	6.0	8.0
		- 5	27.7	22.4			- 5	13.1	12.8			- 1	17.3	17.7
		- 6	5.8	5.2			- 7	20.9	- 23.1	3	- 9	3	8.2	11.4
		- 7	42.6	- 31.5	3	10	0	7.0	- 3.5			- 1	6.5	8.4
3	2	0	8.3	6.7			1	15.0	- 15.6	4	0	0	7.3	5.0
		1	33.5	- 32.6			3	11.0	11.0			1	22.8	- 26.4
		2	18.1	- 16.8			- 1	22.6	22.4			2	12.6	13.8
		3	32.0	33.7			- 2	18.8	- 19.6			3	15.9	13.7
		4	19.5	- 19.4			- 3	5.9	6.1			6	7.2	5.2
		5	10.0	- 9.1			- 5	17.0	15.4			7	12.8	12.3
		6	6.9	- 8.1			- 7	9.8	- 11.9			- 1	16.0	19.7
		7	13.6	15.0	3	11	0	6.8	7.4			- 2	10.7	9.8
		- 1	37.2	37.0			2	8.6	8.6			- 3	17.6	- 21.0
		- 2	11.0	- 9.1			3	7.8	7.5			- 4	12.0	11.5
		- 3	42.6	- 41.3			- 1	6.2	5.0			- 5	13.5	12.1
		- 4	7.8	- 2.7	3	10	- 3	15.7	- 17.0			- 6	17.3	15.8
		- 5	42.8	29.7			- 5	24.8	25.9			- 7	20.7	- 23.9
		- 7	48.5	- 28.4			- 6	8.7	3.5			-10	10.2	5.3

TABLE 5 (*Continued*)

h	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c
		-13	13.6	8.6			3	13.3	13.7			6	7.0	8.9
4	1	1	21.8	- 21.9			- 1	14.1	15.6			- 1	9.7	10.6
		3	36.5	29.9			- 2	15.6	- 18.5			- 2	5.3	7.2
		4	18.8	- 20.1			- 4	19.8	- 24.9			- 4	33.3	- 31.1
		7	7.7	7.4			- 5	17.1	22.0			- 5	22.6	21.2
		- 1	22.1	25.7			- 7	12.7	- 15.5			- 6	6.5	2.4
		- 2	28.5	22.4	4	10	0	5.0	2.2	5	2	0	12.9	- 13.1
		- 3	38.2	- 39.1			1	17.1	- 16.7			1	11.1	- 10.4
		- 4	6.4	- 4.6			2	15.8	15.3			2	17.4	18.9
		- 5	15.1	11.4			3	10.4	9.5			5	7.9	- 9.3
		- 6	26.9	18.0			- 1	11.0	10.4			6	12.1	8.6
		- 7	27.3	- 19.6			- 2	22.6	20.8			- 1	9.6	10.9
4	2	0	10.1	- 13.9			- 3	25.4	- 22.5			- 2	21.5	21.0
		1	22.0	- 24.0			- 5	17.8	19.5			- 3	13.6	- 14.8
		3	20.0	16.0			- 7	11.7	- 13.8			- 4	17.4	- 19.0
		4	5.9	5.1	4	11	1	10.6	- 9.3			- 5	4.7	8.3
		5	24.9	- 26.7			2	9.9	8.8	5	3	0	16.8	- 17.5
		6	10.2	12.9			- 1	6.2	6.5			1	6.5	- 5.2
		- 1	14.4	14.6			- 2	12.2	9.9			2	9.7	10.0
		- 2	13.6	13.1			- 3	16.0	- 17.3			3	10.1	10.3
		- 4	26.5	- 24.5			- 5	7.8	5.3			4	9.2	- 9.3
		- 5	51.2	41.2			- 7	14.4	- 15.1			- 1	11.2	10.6
		- 7	16.3	- 12.9	4	12	0	9.5	- 6.4			- 2	21.6	23.5
4	3	0	17.7	- 18.9			- 1	11.3	10.9			- 3	14.7	- 14.3
		1	25.3	- 22.6			- 4	10.7	- 10.5			- 4	5.4	6.3
		2	19.3	16.7			- 5	17.3	15.6			- 5	4.7	4.8
		3	6.6	5.6			- 6	6.1	6.3			- 6	27.2	30.7
		5	17.8	- 17.2			- 7	9.8	- 11.5			- 7	33.5	- 38.5
		6	7.4	10.9	4	13	0	7.7	- 5.1	5	4	0	12.4	- 11.0
		- 1	23.0	25.2			- 5	13.0	13.7			2	12.0	11.6
		- 2	9.0	11.7	4	- 1	0	5.7	- 7.0			3	10.1	11.0
		- 3	36.6	- 39.5			1	23.1	- 27.9			4	12.9	- 14.4
		- 4	18.7	- 16.2			2	13.5	11.9			5	7.7	- 8.4
		- 5	19.3	24.8			3	6.4	5.2			- 1	11.4	10.7
		- 7	11.8	- 11.9			4	8.6	- 8.6			- 2	17.0	17.2
4	4	0	18.3	- 19.4			5	20.7	- 29.6			- 3	7.5	- 9.1
		1	18.5	- 18.7			6	10.5	10.6			- 4	32.7	- 32.7
		3	21.6	22.6			- 1	26.0	21.7			- 5	28.5	32.0
		4	9.0	- 7.0			- 4	35.8	- 29.4			- 6	38.5	38.6
		- 1	22.0	23.0			- 5	17.5	19.3			- 7	11.5	- 13.5
		- 2	10.4	12.7			- 7	7.3	- 5.5	5	5	0	19.6	- 21.8
		- 3	30.7	- 33.4	4	- 2	0	8.5	- 8.5			1	21.2	- 19.8
		- 4	9.3	12.1			2	6.2	- 2.8			2	17.2	17.0
		- 5	6.8	8.4			3	29.6	27.7			3	14.4	- 14.4
		- 6	20.4	23.0			4	8.7	- 6.4			4	6.4	- 6.6
		- 7	36.0	- 36.8			- 1	46.1	39.5			- 1	14.3	14.0
4	5	0	17.6	- 20.2			- 2	8.3	- 6.4			- 2	14.2	13.1
		1	6.1	- 4.9			- 3	19.1	- 18.8			- 3	20.0	- 16.6
		2	13.2	13.7			- 5	22.9	21.8			- 4	33.5	- 32.3
		3	22.3	25.1	4	- 3	2	9.1	9.1			- 5	17.9	19.6
		4	6.5	- 6.7			3	30.4	25.4			- 6	17.9	- 16.9
		5	13.8	- 15.5			4	8.9	- 10.3			- 7	5.2	2.8
		- 1	45.3	38.1			- 1	6.0	- 4.1	5	6	0	8.6	- 5.9
		- 2	6.9	5.7			- 2	8.8	8.2			1	15.4	- 14.0
		- 3	27.9	- 26.3			- 3	16.9	- 16.3			2	10.0	9.4
		- 4	29.0	- 30.7			- 6	10.1	9.7			3	12.3	13.2
		- 5	41.0	42.8			- 7	11.4	- 16.0			4	8.7	- 6.5
		- 6	10.3	11.4	4	- 4	1	42.6	- 58.2			5	8.3	- 1.7
		- 7	23.6	- 26.6			2	17.7	18.0			- 1	6.0	4.6
4	6	0	10.2	- 10.7			3	6.9	5.8			- 2	27.6	25.8
		1	26.2	- 23.7			- 3	11.7	- 12.2			- 3	23.1	- 22.9
		2	27.8	26.1	4	- 5	1	6.7	- 8.7			- 4	13.0	- 14.7
		3	7.1	- 7.2			3	7.1	- 6.8			- 6	15.0	16.0
		4	9.2	- 8.5			- 1	24.3	25.6			- 7	23.2	- 21.3
		5	11.0	- 11.6			- 5	8.0	9.5	5	7	0	24.3	- 26.2
		- 1	31.4	29.0	4	- 6	0	14.4	- 17.5			2	9.9	9.8
		- 3	22.6	- 22.9			1	6.4	6.6			3	13.8	13.4
		- 4	10.3	- 13.8			3	7.2	7.7			4	10.0	- 12.8
		- 5	16.4	18.9			4	6.5	- 9.9			- 1	19.6	17.7
		- 7	15.9	- 13.9			- 1	21.7	22.7			- 2	26.4	27.7
4	7	0	21.3	24.5	4	- 7	- 1	6.9	6.7			- 3	15.2	- 12.7
		1	38.4	- 34.9			- 2	7.0	7.2			- 4	8.5	- 5.3
		2	7.0	5.1	4	- 8	- 1	6.6	- 4.0			- 5	10.3	12.4
		3	14.3	13.7	4	- 9	- 1	5.9	5.8			- 6	15.4	14.3
		4	9.0	- 8.9	5	0	0	15.3	- 16.1			- 7	18.6	- 15.6
		- 1	18.3	18.3			1	6.5	- 4.6	5	8	0	16.1	- 13.6
		- 2	24.4	28.2			2	6.8	7.8			1	9.7	- 9.1
		- 3	31.4	- 24.9			3	22.5	22.7			2	6.8	9.4
		- 5	12.4	13.6			4	15.4	- 12.5			4	5.3	- 7.4
		- 6	26.9	27.6			- 1	12.1	12.2			- 1	12.2	12.0
		- 7	27.7	- 24.2			- 2	8.4	7.0			- 2	6.1	- 7.5
4	8	0	33.0	- 37.1			- 3	7.8	- 8.9			- 4	25.8	- 24.8
		1	15.4	- 15.3			- 4	8.7	- 8.5			- 5	14.1	17.8
		3	12.2	16.7			- 5	8.3	6.5			- 6	12.1	9.8
		4	10.4	- 12.2			- 6	16.8	14.6			- 7	9.9	- 7.8
		- 1	19.2	19.6			- 7	10.5	- 11.1	5	9	0	5.0	- 4.4
		- 2	14.9	16.3			-10	10.6	9.1			1	9.4	- 9.9
		- 3	18.1	- 18.8			-11	14.8	- 9.4			2	14.3	13.6
		- 4	10.2	- 9.8	5	1	0	14.4	- 15.3			- 2	22.6	26.5
		- 5	22.9	28.2			1	6.4	- 4.9			- 3	16.3	- 16.2
		- 7	25.6	- 25.0			3	7.0	6.2			- 4	9.1	- 10.8
4	9	0	11.4	- 9.3			4	9.2	- 8.8			- 5	12.3	15.9
		1	6.9	- 7.0			5	7.9	- 8.0			- 6	8.8	9.0

TABLE 5 (*Continued*)

h	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c	*h*	*k*	*l*	F_o	F_c
		- 7	8.3	- 7.4			- 6	20.0	22.6			- 2	8.7	8.3
5	10	0	8.3	- 7.4			- 7	8.2	- 5.4	7	2	0	18.0	- 15.1
		2	9.9	9.5	6	4	0	8.8	- 9.2			1	14.6	13.5
		- 2	19.3	17.3			2	12.2	13.9			2	6.3	3.6
		- 3	19.7	- 21.6			3	9.7	- 10.0			- 1	6.3	6.3
		- 6	16.7	15.5			- 1	6.3	5.3			- 2	6.2	7.5
		- 7	10.2	- 6.5			- 2	19.0	14.7			- 3	12.3	11.4
5	11	0	15.9	- 13.3			- 4	22.9	- 23.4			- 4	9.1	- 11.3
		2	4.8	- 4.6			- 5	13.0	13.4	7	3	- 2	6.2	6.1
		- 1	15.3	12.6			- 6	16.3	- 18.2			- 3	6.2	6.2
		- 2	5.8	2.2	6	5	0	8.8	- 8.2			- 4	6.5	- 7.6
		- 4	16.7	- 15.9			1	9.8	- 7.6			- 5	5.5	3.9
		- 5	7.7	5.8			2	9.8	7.2			- 7	5.8	7.9
		- 6	13.9	9.3			4	5.5	- 5.9	7	4	0	8.5	- 8.4
5	12	0	11.7	- 8.3			- 1	6.4	- 4.8			1	15.7	- 13.1
		- 1	5.1	5.2			- 2	29.3	28.0			2	6.2	8.0
		- 2	5.3	4.8			- 3	13.4	- 11.8			- 1	12.6	- 13.0
		- 4	13.1	- 13.6			- 4	10.8	- 13.3			- 2	16.3	15.8
		- 5	11.4	11.2			- 5	9.3	10.5			- 3	6.2	- 4.2
		- 6	5.9	5.8			- 6	12.2	14.4			- 4	6.5	- 7.7
5	- 1	1	14.7	- 15.1			- 7	8.1	- 8.1			- 6	8.9	10.0
		2	6.8	7.5	6	6	0	23.8	- 24.3	7	5	0	12.8	- 12.6
		3	7.1	- 6.3			1	11.8	9.3			1	16.1	15.8
		4	9.1	6.5			2	6.7	6.6			2	5.9	5.4
		- 3	14.6	- 15.2			4	9.0	- 9.3			- 1	6.2	- 6.0
		- 6	14.2	15.9			- 1	6.4	6.1			- 2	10.6	11.3
		- 7	8.0	- 10.9			- 2	29.6	27.0			- 3	6.1	- 2.2
5	- 2	0	6.9	- 7.3			- 3	10.5	- 7.2			- 5	5.5	- 6.6
		1	9.5	- 10.4			- 4	6.3	6.1			- 6	8.9	11.4
		2	6.9	8.0			- 5	5.4	- 4.4	7	6	0	10.5	- 11.0
		4	9.2	- 9.7			- 6	16.6	19.0			1	6.0	4.9
		5	15.8	- 16.5	6	7	0	15.9	- 18.7			2	5.6	6.8
		6	9.9	12.7			1	6.7	- 3.9			- 1	10.6	10.8
		- 3	11.8	- 11.8			2	11.2	14.4			- 4	6.4	- 8.0
		- 4	12.4	- 11.0			4	4.7	- 5.5			- 6	6.3	7.3
		- 5	7.8	9.0			- 1	9.0	7.4			- 7	8.2	8.3
5	- 3	0	7.0	- 6.5			- 2	6.2	- 8.5	7	7	1	5.7	- 4.2
		2	7.0	- 5.9			- 4	11.1	- 12.6			2	8.9	9.4
		3	23.0	20.4			- 5	5.5	6.0			- 2	8.4	8.5
		4	9.1	- 10.6			- 6	10.8	10.9			- 3	8.5	8.4
		- 1	21.2	18.0	6	8	1	6.4	- 5.5			- 4	16.8	- 18.8
		- 4	6.4	- 8.0			2	10.4	11.9			- 5	5.5	- 8.0
		- 5	7.9	8.7			- 2	16.3	15.5			- 6	8.8	7.2
5	- 4	3	10.1	9.1			- 4	29.2	- 25.8			- 7	5.8	4.7
		4	11.0	- 11.6			- 6	12.6	10.5	7	8	2	6.3	7.1
5	- 4	- 4	6.5	- 7.3	6	9	0	8.0	- 7.4			- 2	8.1	8.1
5	- 5	1	15.5	- 19.8			2	7.6	8.7			- 3	5.8	3.7
		2	15.7	17.7			- 2	15.5	17.7			- 5	5.3	- 6.2
5	- 6	- 1	9.6	9.1			- 6	16.9	15.4			- 6	13.7	14.4
5	- 7	0	4.7	- 6.4			- 7	5.9	- 3.8	7	9	0	6.6	- 5.8
5	- 8	0	4.2	0.5	6	10	0	13.3	- 12.7			- 2	5.3	6.7
6	0	0	12.2	- 11.6			- 2	5.7	5.1			- 4	11.9	- 12.8
		2	7.0	2.6			- 4	13.9	- 13.9			- 6	11.8	12.8
		4	8.4	- 7.8			- 6	10.7	12.3	7	10	0	6.4	- 6.2
		- 1	13.8	11.7	6	11	0	11.5	- 8.8			- 2	6.9	5.8
		- 2	10.1	13.5			- 2	7.4	4.5			- 3	7.2	7.7
		- 4	20.2	- 20.0			- 4	13.1	- 14.7			- 4	9.6	- 11.4
		- 5	14.3	10.5			- 5	5.1	4.5			- 6	5.6	3.4
		- 8	10.6	- 7.8			- 6	5.9	7.8	7	11	- 4	5.0	- 4.9
6	1	0	7.2	- 7.9	6	12	- 4	7.5	- 6.9			- 5	6.2	- 8.1
		1	9.8	- 7.6			- 5	6.6	- 7.3			- 6	5.1	10.2
		2	12.2	12.2			- 6	13.1	12.5	7	- 1	0	6.7	- 7.5
		- 1	16.1	- 15.5	6	- 1	0	7.2	- 8.0	8	3	- 1	5.4	- 8.7
		- 2	19.0	21.1			1	9.8	6.9			- 2	5.4	5.1
		- 6	6.2	7.5			2	7.0	7.8			- 5	5.0	- 6.6
6	2	0	10.1	- 10.7			3	9.9	9.3	8	4	- 1	5.3	- 4.8
		2	7.0	7.0			4	6.1	- 6.7			- 4	5.8	- 5.2
		4	8.6	- 8.0			- 1	9.0	8.8			- 6	5.7	8.1
		- 2	8.5	8.8			- 4	9.1	- 8.8	8	5	- 1	7.4	6.4
		- 3	15.5	- 18.4	6	- 2	0	5.1	- 5.9			- 3	7.7	9.0
		- 6	20.2	22.0			1	6.9	- 4.4			- 4	5.8	- 5.2
6	3	0	18.4	- 18.0			4	5.9	3.1	8	6	- 1	8.7	- 8.5
		1	9.8	8.4			- 1	6.4	- 7.0			- 2	5.1	4.3
		2	7.0	8.0			- 4	6.5	- 6.7			- 4	5.7	- 7.0
		3	7.0	7.1	6	- 4	- 1	8.7	7.9			- 5	5.0	- 5.5
		4	8.5	- 7.8	7	0	2	6.2	5.8	8	7	- 2	6.9	5.1
		- 1	6.2	7.5			- 1	18.4	- 14.3			- 5	6.8	- 8.1
		- 2	8.5	10.4			- 2	9.6	8.3			- 6	7.8	8.6
		- 4	30.1	- 31.8	7	1	2	6.3	5.0	8	8	- 5	4.6	- 4.8
												- 6	7.5	6.6

The final values of the structure factors are listed in Table 5. These structure factors were used to compute the final three-dimensional electron-density distribution shown in Fig. 1.

The final anisotropic temperature factors given by the least-squares refinement are shown in Table 4; 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:

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

The results are listed in Table 1.

The extensive calculations were performed on the Glasgow University DEUCE computer with programmes devised by Dr. J. S. Rollett and Dr. J. G. Sime. We are indebted to Professor J. M. Robertson, F.R.S., and Dr. W. Parker for their advice and interest.

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[*Received, August 6th*, 1964.]