136. *The Structure of Geigerin: X-Ray Analysis of Bromogeigerin Acetate.*

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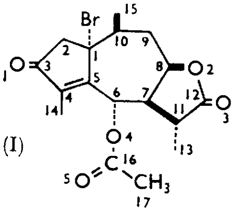
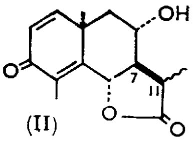
In order to determine the stereochemistry of the sesquiterpenoid lactone geigerin an *X*-ray study of bromogeigerin acetate has been carried out. Bromogeigerin acetate crystallizes in the orthorhombic system, space group $P2_12_12_1$-D_2^4, with four molecules of $C_{17}H_{21}BrO_5$ in the unit cell. Successive three-dimensional Fourier syntheses resulted in the elucidation of the constitution and stereochemistry (apart from absolute configuration) as summarized in formula (I).

The structure of the sesquiterpenoid lactone geigerin has been investigated by Barton and Levisalles[1] who established the constitution and proposed a partial stereochemistry. In order to confirm and extend the assignment we have carried out a crystal-structure analysis of a bromo-derivative of geigerin acetate which Barton and Pinhey[2] prepared by bromination of the acetate with *N*-bromosuccinimide in carbon tetrachloride. After a number of cycles of three-dimensional Fourier syntheses and least-squares refinement of positional and thermal atomic parameters our final results establish the constitution and relative stereochemistry of this derivative to be as in (I). The absolute configuration given in (I) has been defined[2] by a chemical correlation with artemisin (II). Position 2 was the site at which bromination was expected to occur in geigerin acetate. That substitution actually takes place at position 1 has the unfortunate consequence that we cannot infer the configuration of geigerin at position 1 from our results.

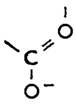
(I)



(II)



(III)



The final electron-density distribution for bromogeigerin acetate is shown in Fig. 1 as superimposed contour sections parallel to (001) and covering the region of one molecule. The atomic arrangement corresponding to this electron-density distribution is illustrated in Fig. 2. The stereochemistry at positions 6, 7, and 8 is the same as that proposed by Barton and Levisalles.[1] The cycloheptane ring has a chair conformation.

The interatomic distances and interbond angles calculated from the final atomic co-ordinates (see Table 1) are listed in Table 2. The standard deviations of the final atomic co-ordinates were estimated in the usual manner from the least-squares residuals

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[1] Barton and Levisalles, *J.*, 1958, 4518.

[2] Barton and Pinhey, *Proc. Chem. Soc.*, 1960, 279.

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)	0·4872	0·0270	0·7184	C(9)	0·3139	0·1670	0·6549	C(17)	0·1111	−0·2225	0·6449
C(2)	0·6497	0·0768	0·7521	C(10)	0·3270	0·0855	0·7275	O(1)	0·9248	0·0396	0·7015
C(3)	0·7761	0·0384	0·6919	C(11)	0·4371	0·1211	0·4602	O(2)	0·2166	0·2117	0·5114
C(4)	0·6912	−0·0003	0·6112	C(12)	0·3236	0·2107	0·4526	O(3)	0·3318	0·2710	0·3928
C(5)	0·5324	−0·0008	0·6259	C(13)	0·4978	0·0759	0·3690	O(4)	0·2658	−0·0830	0·5994
C(6)	0·4028	−0·0286	0·5596	C(14)	0·7864	−0·0327	0·5336	O(5)	0·3794	−0·2248	0·5587
C(7)	0·3199	0·0541	0·5122	C(15)	0·3148	0·1397	0·8209	Br	0·4646	−0·0914	0·7919
C(8)	0·2282	0·1304	0·5715	C(16)	0·2631	−0·1804	0·5999				

* " 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)	1·57	C(8)–C(9)	1·53	C(11)–C(12)	1·54	C(3)–O(1)	1·21
C(2)–C(3)	1·47	C(9)–C(10)	1·58	C(12)–O(2)	1·25	C(6)–O(4)	1·47
C(3)–C(4)	1·51	C(10)–C(1)	1·53	C(12)–O(3)	1·24	C(16)–O(5)	1·29
C(4)–C(5)	1·31	C(1)–C(5)	1·51	C(8)–O(2)	1·45	C(16)–O(4)	1·34
C(5)–C(6)	1·51	C(7)–C(11)	1·54	C(10)–C(15)	1·61	C(16)–C(17)	1·53
C(6)–C(7)	1·51	C(11)–C(13)	1·60	C(4)–C(14)	1·48	C(1)–Br	1·99
C(7)–C(8)	1·57						

Intramolecular non-bonded distances

C(1) ··· C(7)	3·44	C(4) ··· C(11)	3·51	C(6) ··· O(2)	3·71	C(10) ··· O(2)	3·83
C(1) ··· C(8)	3·38	C(4) ··· O(4)	3·64	C(6) ··· O(5)	2·71	C(10) ··· O(4)	3·07
C(1) ··· C(14)	3·81	C(5) ··· C(8)	3·17	C(7) ··· C(10)	3·31	C(11) ··· C(14)	3·71
C(1) ··· C(16)	3·84	C(5) ··· C(9)	2·95	C(7) ··· C(14)	3·98	C(11) ··· O(4)	3·79
C(1) ··· O(1)	3·56	C(5) ··· C(11)	3·13	C(7) ··· C(16)	3·53	C(13) ··· O(2)	3·66
C(1) ··· O(4)	2·97	C(5) ··· C(15)	3·96	C(7) ··· O(3)	3·50	C(13) ··· O(3)	3·03
C(2) ··· C(6)	3·84	C(5) ··· C(16)	3·32	C(7) ··· O(5)	3·93	C(13) ··· C(14)	3·74
C(2) ··· C(9)	3·34	C(5) ··· O(1)	3·43	C(8) ··· C(13)	3·86	C(14) ··· O(1)	2·97
C(2) ··· C(14)	3·82	C(5) ··· O(5)	3·48	C(8) ··· C(15)	3·87	Br ··· C(3)	3·45
C(2) ··· C(15)	3·04	C(6) ··· C(9)	3·14	C(8) ··· O(3)	3·45	Br ··· C(4)	3·54
C(3) ··· C(6)	3·75	C(6) ··· C(10)	3·06	C(8) ··· O(4)	2·98	Br ··· C(6)	3·68
C(3) ··· C(10)	3·74	C(6) ··· C(12)	3·73	C(9) ··· C(11)	3·19	Br ··· C(15)	3·43
C(4) ··· C(7)	3·45	C(6) ··· C(14)	3·14	C(9) ··· C(12)	3·14	Br ··· C(16)	3·57
C(4) ··· C(9)	3·89	C(6) ··· C(13)	3·33	C(9) ··· O(4)	3·57	Br ··· O(4)	3·35
C(4) ··· C(10)	3·64	C(6) ··· C(17)	3·80				

Intermolecular distances ($\leqslant 4$ Å)

C(3) ··· O(3)$_{\mathrm{I}}$	2·96	C(12) ··· O(2)$_{\mathrm{I}}$	3·41	Br ··· C(12)$_{\mathrm{III}}$	3·76	Br ··· C(13)$_{\mathrm{III}}$	3·94
O(1) ··· O(3)$_{\mathrm{I}}$	3·07	O(3) ··· O(2)$_{\mathrm{I}}$	3·45	Br . . . O(3)$_{\mathrm{III}}$	3·78	C(12) ··· C(8)$_{\mathrm{I}}$	3·96
C(11) ··· O(2)$_{\mathrm{I}}$	3·26	O(3) ··· C(8)$_{\mathrm{I}}$	3·53	C(14) ··· O(3)$_{\mathrm{I}}$	3·79	C(15) ··· C(13)$_{\mathrm{III}}$	3·97
C(4) ··· O(3)$_{\mathrm{I}}$	3·36	O(5) ··· C(17)$_{\mathrm{II}}$	3·70	C(17) ··· O(3)$_{\mathrm{III}}$	3·86	C(17) ··· C(15)$_{\mathrm{IV}}$	3·98
C(2) ··· O(3)$_{\mathrm{I}}$	3·38	C(14) ··· O(5)$_{\mathrm{II}}$	3·70	C(13) ··· O(2)$_{\mathrm{I}}$	3·88	C(5) ··· O(3)$_{\mathrm{I}}$	4·00

The subscripts refer to the following positions:

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

Interbond angles

C(1)	C(2)	C(3)	103°	C(5)	C(1)	Br	110°	C(6)	C(7)	C(8)	116°	C(12)	C(11)	C(7)	99°
C(2)	C(3)	C(4)	108	C(10)	C(1)	Br	108	C(7)	C(8)	C(9)	119	C(11)	C(7)	C(8)	101
C(3)	C(4)	C(5)	108	C(5)	C(6)	O(4)	112	C(8)	C(9)	C(10)	112	C(7)	C(11)	C(13)	114
C(4)	C(5)	C(1)	114	C(7)	C(6)	O(4)	104	C(9)	C(10)	C(1)	112	C(12)	C(11)	C(13)	115
C(5)	C(1)	C(2)	102	C(6)	O(4)	C(16)	122	C(10)	C(1)	C(5)	115	C(11)	C(12)	O(3)	124
C(2)	C(3)	O(1)	128	O(4)	C(16)	O(5)	117	C(9)	C(10)	C(15)	107	O(2)	C(12)	O(3)	124
C(4)	C(3)	O(1)	124	O(4)	C(16)	C(17)	113	C(1)	C(10)	C(15)	112	C(9)	C(8)	O(2)	107
C(3)	C(4)	C(14)	121	O(5)	C(16)	C(17)	129	C(7)	C(8)	O(2)	101	C(6)	C(7)	C(11)	115
C(5)	C(4)	C(14)	130	C(1)	C(5)	C(6)	121	C(8)	O(2)	C(12)	114	C(4)	C(5)	C(6)	125
C(2)	C(1)	Br	105	C(5)	C(6)	C(7)	116	O(2)	C(12)	C(11)	112	C(2)	C(1)	C(10)	117

(see Experimental section) and are shown in Table 3; from the results the average e.s.d. of a carbon–carbon bond is about 0·03 Å and the average e.s.d. of a valency angle about 2°.

TABLE 3.

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

Atom	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	Atom	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	Atom	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(1)	0·024	0·018	0·019	C(9)	0·026	0·020	0·020	C(17)	0·026	0·020	0·022
C(2)	0·025	0·020	0·020	C(10)	0·023	0·019	0·017	O(1)	0·016	0·014	0·015
C(3)	0·024	0·017	0·019	C(11)	0·024	0·018	0·019	O(2)	0·016	0·013	0·013
C(4)	0·024	0·020	0·020	C(12)	0·026	0·020	0·020	O(3)	0·018	0·015	0·016
C(5)	0·025	0·017	0·017	C(13)	0·022	0·020	0·020	O(4)	0·016	0·013	0·014
C(6)	0·023	0·020	0·022	C(14)	0·027	0·022	0·021	O(5)	0·019	0·015	0·017
C(7)	0·025	0·020	0·020	C(15)	0·027	0·023	0·021	Br	0·003	0·002	0·002
C(8)	0·024	0·020	0·021	C(16)	0·024	0·019	0·021				

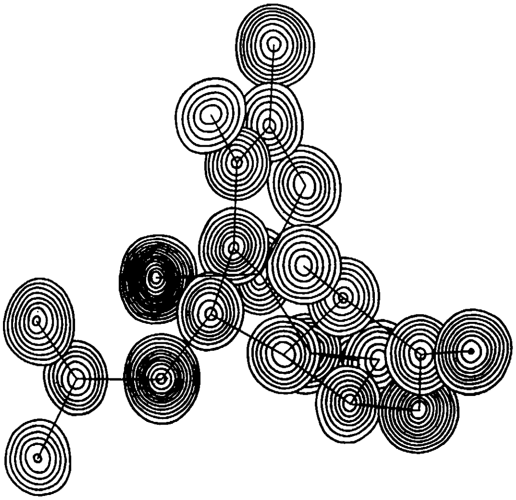

FIG. 1. Final three-dimensional electron-density distribution for bromogeigerin acetate shown by means of superimposed contour sections parallel to (001). Contour interval 1e/Å^3 except around the bromine atom where it is 5e/Å^3.

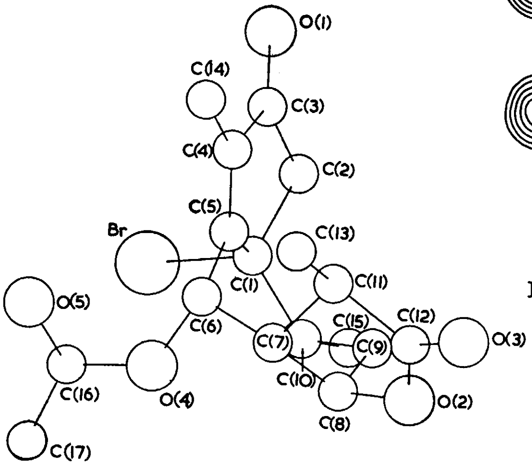

FIG. 2. Atomic arrangement corresponding to Fig. 1.

The average carbon–carbon single bond length of 1·537 Å compares favourably with the value of 1·545 Å in diamond. The carbon–carbon double bond length of 1·31 Å in the cyclopentenone ring does not differ significantly from the values of 1·33 and 1·32 Å reported for ethylene and *p*-benzoquinone,[3] respectively. The carbon–oxygen single bonds fall into two categories; those adjacent to a carbon–oxygen double bond have a mean length of 1·29 Å whereas those not adjacent to a carbon–oxygen double bond have a mean length of 1·46 Å. This difference is highly significant and indicates that in lactone and ester groupings, just as in carboxylic acids, the structure (III) makes an important contribution. The angles around carbon atoms 12 and 16, moreover, conform

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[3] Trotter, *Acta Cryst.*, 1960, **13**, 86.

to the pattern observed in carboxylic acids,[4] the angles C(17)C(16)O(4) and C(11)C(12)O(2) being approximately tetrahedral, whereas the angles C(17)C(16)O(5), O(4)C(16)O(5), C(11)C(12)O(3), and O(2)C(12)O(3) exceed 120°. Similar conclusions were reached in our recent analyses of epilimonol iodoacetate [5] and clerodin bromolactone.[6] Of the three carbonyl carbon–oxygen bonds, C(3)–O(1) is the shortest (1·22 Å). This is not unreasonable, for C(16)–O(5) and C(12)–O(3), unlike C(3)–O(1), are involved in resonance of the type shown in (III) and in consequence have some single-bond character associated with them. The carbon–bromine bond length of 1·99 Å appears to be slightly longer than the average value of 1·94 Å reported for various alkyl bromides.[7] It is perhaps pertinent that the environment of the carbon–halogen bond is not unlike that in isoclovene hydrochloride where a significant lengthening of the carbon–chlorine bond over the accepted value has been attributed to a steric effect.[8] In the cycloheptane ring the angles are consistently greater than tetrahedral, the mean value being 116°. Similar increases in bond angles have been reported for isoclovene hydrochloride,[8] cyclononylamine hydrobromide,[9] and 1,6-*trans*-diaminocyclodecane dihydrochloride.[10]

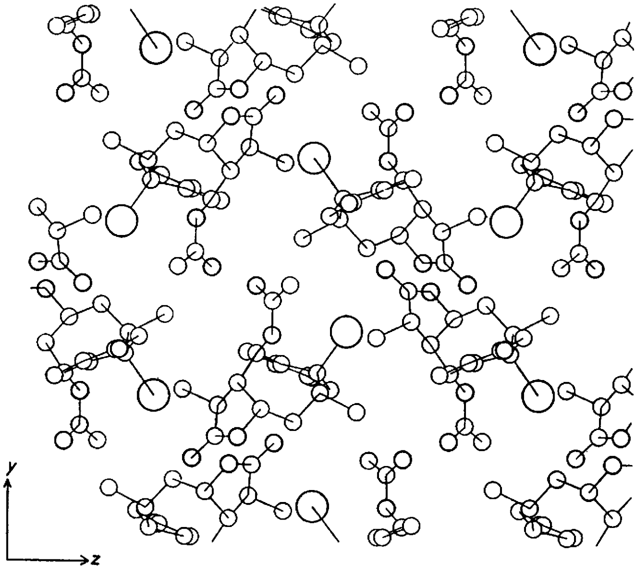

FIG. 3. The arrangement of molecules in the crystal as viewed in projection along the *a*-axis.

The cyclopentenone system is non-planar. The mean plane through atoms C(1), C(3), C(4), C(5), C(6), and C(14) was calculated by the method of Schomaker *et al.*,[11] and the deviations of the atoms of the cyclopentenone system from this plane are listed in Table 4. The displacement, 0·32 Å, of C(2) is highly significant.

The arrangement of the molecules in the crystal as viewed in projection along the *a*-axis is shown in Fig. 3. The shorter intermolecular contacts are listed in Table 2; none appears to be abnormal.

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[4] Cochran, *Acta Cryst.*, 1953, **6**, 260; Ahmed and Cruickshank, *ibid.*, 1953, **6**, 385; Broadley, Cruickshank, Morrison, Robertson, and Shearer, *Proc. Roy. Soc.*, 1959, **251**, *A*, 441; Ferguson and Sim, *Acta Cryst.*, 1961, **14**, 1262.

[5] Arnott, Davie, Robertson, Sim, and Watson, *J.*, 1961, 4183.

[6] Paul, Sim, Hamor, and Robertson, unpublished work.

[7] Sutton *et al.*, " Tables of Interatomic Distances and Configuration in Molecules and Ions," *Chem. Soc. Special Publ.* No. 11, 1958.

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

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

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

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

TABLE 4.

Displacements (Å) of the atoms of the cyclopentenone system from the mean plane through C(1), C(3), C(4), C(5), C(6), and C(14).

C(1)	0·076	C(3)	−0·066	C(5)	−0·010	C(14)	0·061
C(2)	−0·321	C(4)	−0·014	C(6)	−0·048	O(1)	0·040

EXPERIMENTAL

Crystal Data.—Bromogeigerin acetate, $C_{17}H_{21}BrO_5$; $M = 385·3$; m. p. 147—153° (decomp.). Orthorhombic, $a = 8·11$, $b = 13·77$, $c = 15·24$ Å, $U = 1702$ Å^3, $D_m = 1·512$ (by flotation), $Z = 4$, $D_c = 1·505$, $F(000) = 792$, space group $P2_12_12_1$-D_2^4. Absorption coefficient for X-rays ($\lambda = 1·542$ Å) $\mu = 36·5$ cm.$^{-1}$.

Experimental Measurements.—Rotation, oscillation, Weissenberg, and precession photographs were taken with copper K_α ($\lambda = 1·542$ Å) and molybdenum K_α ($\lambda = 0·7107$ Å) radiation. Cell dimensions were obtained from rotation and precession photographs. The space group was determined uniquely from the systematic halvings in the reflexions. For the intensity measurements small crystals were employed, completely bathed in a uniform X-ray beam. No corrections for absorption were applied. The multiple-film technique[12] with visual intensity estimation was applied to equatorial and equi-inclination upper-layer Weissenberg photographs taken from crystals rotated about the a- and c-crystal axes. The intensities were corrected for Lorentz, polarization, and the rotation factors appropriate to upper layers,[13] and values of $|F_o|$ were then evaluated by the mosaic-crystal formula. The various layers were put on the same relative scale by comparison of common reflexions on different photographs. The absolute scale was obtained at a later stage by correlation with the final calculated structure amplitudes, $|F_c|$. In all 1625 independent structure amplitudes were measured (Table 5). The crystal density was determined by flotation in zinc chloride solution.

Structure Analysis.—Because of the presence of the bromine atom there was no need to postulate trial structures with assumptions as to the stereochemistry of the molecule, the analysis proceeding directly on the basis of the usual phase-determining heavy-atom method.[14] The position of the bromine atom was determined initially from two-dimensional Patterson syntheses along the a- and c-crystal axes (see Fig. 4) and was confirmed by a study of appropriate sections of the three-dimensional Patterson function.

The unit-cell dimensions indicated that there would be considerable overlap of atoms in projection along the crystallographic axes and in consequence no attempt was made to elucidate the crystal structure further by two-dimensional methods.

The first three-dimensional Fourier synthesis was evaluated with the measured values of the structure amplitudes and phase constants appropriate to the bromine atom alone. When the resulting electron-density distribution was drawn various significant peaks were apparent and could be attributed to carbon atoms (1), (2), (3), (4), (5), (6), (7), (8), (9), (10), (16), and (17), and oxygen atoms (1), (2), (4), and (5). Co-ordinates were assigned to these atoms and they were then included (all as carbon atoms) in the calculation of a second set of structure amplitudes and phase constants. An overall isotropic temperature factor B of 3·0 Å^2 was assumed. The value of R, the average discrepancy between the calculated and measured structure amplitudes, was 0·280.

The improved phase constants were employed in the computation of a second three-dimensional Fourier synthesis in which it was possible to locate without ambiguity the remaining carbon and oxygen atoms other than C(14). When structure factors were recalculated with the inclusion of these additional atoms the value of R fell to 0·230. A third three-dimensional electron-density distribution based on the revised phase constants was then calculated. All the atoms (other than hydrogen) were clearly revealed and it proved possible to distinguish between carbon and oxygen atoms on the basis of peak heights and interatomic distances. On calculating a further set of structure factors, with each atom assigned its correct

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[12] Robertson, *J. Sci. Instr.*, 1943, **20**, 175.

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

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

TABLE 5.

Measured and calculated values of the structure factors.

h	*k*	*l*	$\lvert F_o\rvert$	$\lvert F_c\rvert$	α°
0	0	2	51	60	180
		4	41	41	0
		6	18	21	180
		8	22	28	180
		10	60	61	0
		12	54	58	180
		14	19	21	0
		16	6	8	180
0	1	1	72	69	270
		2	77	83	90
		3	14	14	90
		4	98	89	90
		5	85	92	270
		6	41	44	270
		7	32	29	90
		8	8	13	90
		9	42	44	270
		10	7	6	270
		11	23	15	90
		12	< 4	8	90
		13	< 4	4	90
		14	19	18	90
		15	10	13	270
		16	10	14	270
		17	8	10	90
		18	14	16	90
		19	9	10	270
0	2	0	116	118	0
		1	47	57	180
		2	103	120	180
		3	38	27	180
		4	44	49	0
		5	31	26	180
		6	4	8	180
		7	8	7	180
		8	13	13	180
		9	31	33	0
		10	37	38	0
		11	54	53	180
		12	36	35	180
		13	39	40	0
		14	8	9	0
		15	8	11	180
		16	< 4	2	0
		17	15	15	0
		18	< 3	5	180
		19	6	10	0
0	3	1	36	36	270
		2	31	38	90
		3	120	114	90
		4	45	38	270
		5	121	131	270
		6	65	63	90
		7	99	91	90
		8	14	11	270
		9	46	36	270
		10	27	23	90
		11	34	32	90
		12	< 4	8	90
		13	< 4	2	90
		14	18	19	270
		15	20	32	270
		16	8	4	270
		17	15	22	90
		18	< 3	0	270
		19	4	10	270
0	4	0	19	9	0
		1	43	36	180
		2	89	90	0
		3	25	16	180
		4	53	42	180
		5	34	26	0
		6	15	9	0
		7	21	11	180
		8	24	29	0
		9	30	26	0
		10	14	17	180
		11	38	33	180
		12	< 4	7	0
		13	18	18	0
		14	18	15	180
		15	7	14	180
		16	15	15	0
		17	< 3	3	0
		18	< 3	2	0
		19	3	2	0
0	5	1	< 2	2	270
		2	120	127	90
		3	61	61	90
		4	73	72	270
		5	77	70	270
		6	60	64	90
		7	20	17	90
		8	39	35	270
		9	43	45	270
		10	39	41	90
		11	18	18	270
		12	4	7	90
		13	18	15	90
		14	18	21	270
		15	9	12	270
		16	16	19	90
		17	< 3	3	90
		18	9	13	270
0	6	0	101	105	180
		1	34	35	0
		2	68	68	0
		3	76	73	180
		4	58	55	180
		5	< 3	3	0
		6	28	22	0
		7	7	4	180
		8	13	13	0
		9	< 4	12	180
		10	42	37	180
		11	37	40	0
		12	31	30	0
		13	< 4	0	180
		14	18	21	180
		15	18	16	0
		16	5	8	0
		17	8	1	180
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		2	19	16	90
		3	11	7	270
		4	48	42	270
		5	13	10	90
		6	19	21	90
		7	53	44	270
		8	20	22	270
		9	27	25	90
		10	4	8	90
		11	23	21	270
		12	7	10	270
		13	< 4	10	270
		14	30	27	270
		15	< 4	1	90
		16	11	11	90
		17	5	9	270
		18	5	9	270
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		1	96	100	0
		2	14	17	0
		3	41	39	180
		4	23	22	180
		5	5	3	180
		6	19	13	180
		7	24	33	0
		8	6	5	180
		9	39	44	180
		10	21	15	180
		11	33	31	0
		12	7	6	180
		13	32	38	180
		14	< 4	3	180
		15	14	14	0
		16	< 3	0	360
		17	5	5	180
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		2	10	7	90
		3	20	18	270
		4	30	33	90
		5	55	57	90
		6	49	47	270
		7	32	24	270
		8	16	12	90
		9	11	10	90
		10	17	9	270
		11	13	16	270
		12	13	2	90
		13	6	5	270
		14	10	1	270
		15	18	15	90
0	10	0	54	51	0
		1	30	35	0
		2	30	25	180
		3	12	12	180
		4	12	13	0
		5	24	20	180
		6	19	17	180
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		8	4	3	0
		9	47	42	180
		10	15	19	0
		11	26	35	0
		12	21	25	180
		13	< 4	1	180
		14	14	11	0
		15	< 3	5	0
		16	7	10	180
0	11	1	< 4	5	270
		2	21	24	270
		3	10	6	270
		4	31	34	90
		5	4	8	270
		6	49	56	270
		7	14	13	90
		8	27	22	90
		9	11	10	270
		10	14	12	270
		11	6	2	270
		12	< 4	2	90
		13	< 3	1	270
		14	10	13	90
		15	7	5	90
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		1	22	22	180
		2	43	46	180
		3	7	6	0
		4	16	16	180
		5	20	17	180
		6	4	12	180
		7	6	1	0
		8	7	10	0
		9	4	8	0
		10	22	19	0
		11	16	16	180
		12	15	13	180
		13	5	3	0
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		2	7	4	90
		3	35	42	90
		4	20	25	90
		5	29	32	270
		6	21	26	270
		7	17	21	90
		8	4	7	90
		9	16	14	270
		10	15	9	90
		11	14	11	90
		12	8	7	270
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		1	19	26	180
		2	15	13	0
		3	< 4	7	0
		4	14	12	180
		5	7	7	180
		6	9	2	0
		7	6	9	180
		8	3	11	0
		9	20	22	0
		10	22	6	180
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		2	< 4	3	270
		3	11	13	90
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		5	15	16	270
		6	3	2	90
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		9	6	12	270
		10	12	12	90
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		4	16	17	180
		5	9	8	0
		6	< 3	1	180
		7	6	5	180
		8	9	15	0
0	17	1	< 3	2	90
		2	11	13	90
		3	< 2	5	270
		4	10	11	270
		5	5	8	90
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		2	17	8	180
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		4	61	68	180
		5	54	51	90
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		10	8	8	0
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		12	16	18	0
		13	30	34	270
		14	26	29	180
		15	18	20	90
		16	5	6	180
		17	4	4	270
		18	5	8	180
		19	3	4	90
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		2	30	32	102
		3	72	86	324
		4	88	93	178
		5	99	107	191
		6	30	27	346
		7	72	79	23
		8	41	33	195
		9	25	30	152
		10	21	18	30
		11	18	17	220
		12	9	8	137
		13	14	14	40
		14	13	15	89
		15	15	20	149
		16	13	17	333
		17	21	23	23
		18	8	12	141
		19	9	14	142
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		1	76	82	75
		2	84	90	296
		3	51	44	343
		4	56	46	126
		5	61	56	11
		6	22	21	335
		7	17	16	267
		8	50	48	284
		9	6	4	328
		10	57	60	86
		11	33	33	135
		12	32	36	280
		13	22	20	315
		14	29	29	98
		15	7	8	49
		16	13	12	314
1	3	0	141	145	270
		1	20	23	326
		2	27	34	342
		3	58	60	120
		4	55	60	170
		5	13	7	156
		6	106	92	352
		7	5	6	85
		8	69	67	170
		9	32	31	32
		10	27	26	356
		11	17	16	246
		12	< 5	3	58
		13	8	6	352
		14	< 5	10	145
		15	< 5	6	336
		16	13	17	4
		17	< 4	4	311
		18	11	15	175
		19	5	6	78
1	4	0	61	59	90
		1	65	65	269
		2	68	92	300
		3	72	79	92
		4	31	29	34
		5	< 3	7	69
		6	46	34	150
		7	16	15	223
		8	51	55	248
		9	29	34	64
		10	36	35	89
		11	24	27	252
		12	33	31	281
		13	10	11	85
		14	15	18	96
		15	13	16	257
		16	7	10	223
		17	7	4	115
		18	5	2	254
		19	3	4	111
1	5	0	34	29	270
		1	74	73	292
		2	42	40	106
		3	62	59	193
		4	35	34	141
		5	62	63	12
		6	38	32	45
		7	81	73	179
		8	< 4	9	124
		9	47	39	38
		10	27	28	286
		11	19	19	142
		12	17	15	68
		13	8	10	133
		14	< 5	2	48
		15	19	22	26
		16	9	8	54
		17	10	13	188
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		1	58	55	266
		2	45	35	79
		3	88	90	59
		4	24	20	326
		5	58	54	304
		6	9	5	181
		7	49	42	327
		8	19	15	281
		9	41	42	88
		10	< 5	6	311
		11	28	28	279
		12	11	8	48
		13	13	18	83
		14	16	16	291
		15	12	15	254
		16	13	11	124
		17	< 4	3	39
		18	9	9	338
1	7	0	56	44	270
		1	20	18	16
		2	40	36	230
		3	52	46	169
		4	52	56	349
		5	39	36	1
		6	48	48	187
		7	26	24	175
		8	47	40	344
		9	29	30	29
		10	19	17	232
		11	10	6	121
		12	13	11	76
		13	12	12	181
		14	7	12	339
		15	16	14	32
		16	10	14	165
		17	12	14	199
		18	5	11	331
1	8	0	58	58	270
		1	8	7	22
		2	52	50	63
		3	13	10	47
		4	36	32	273
		5	50	48	193
		6	19	19	178
		7	28	23	330
		8	21	21	99
		9	10	9	152
		10	39	40	287
		11	7	1	340
		12	28	28	64
		13	7	8	300
		14	23	22	292
		15	7	6	176
		16	17	15	119
		17	4	3	197
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		1	22	16	80
		2	32	31	179
		3	21	23	34
		4	54	51	37
		5	26	21	157
		6	50	52	179
		7	12	8	316
		8	30	33	329
		9	26	19	197
		10	9	10	247
		11	8	3	51
		12	9	8	232
		13	15	12	165
		14	16	15	24
		15	5	2	173
		16	10	12	173
1	10	0	13	11	270
		1	50	52	84
		2	27	22	87
		3	44	42	310
		4	15	15	291
		5	19	18	160
		6	16	12	274
		7	11	4	326
		8	17	15	83
		9	15	19	293
		10	18	17	295
		11	25	28	83
		12	19	17	113
		13	21	22	266
		14	< 4	6	234
		15	12	11	117
1	11	0	5	9	90
		1	8	7	219
		2	28	24	261
		3	8	7	6
		4	12	11	198
		5	45	41	178
		6	11	12	219
		7	41	42	342
		8	15	11	190
		9	12	10	180
		10	23	21	62
		11	7	6	35
		12	15	10	223
		13	< 4	6	73
		14	11	6	351
		15	7	10	181
1	12	0	18	22	90
		1	35	42	89
		2	18	21	250
		3	16	21	240
		4	13	8	69
		5	< 5	6	67
		6	< 5	4	136
		7	14	11	106
		8	< 5	2	264
		9	14	13	280
		10	7	7	77
		11	22	22	107
		12	7	8	258
		13	11	13	263
		14	9	10	104
1	13	0	5	9	90
		1	9	7	259
		2	12	19	333
		3	< 5	4	306
		4	21	25	173
		5	12	14	190
		6	34	35	15
		7	10	10	8
		8	25	24	208
		9	8	10	80
		10	15	13	10
		11	8	8	240
		12	6	6	233
		13	8	6	310
1	14	0	28	29	90
		1	12	14	191
		2	9	11	277
		3	12	13	164
		4	11	13	70
		5	19	15	341
		6	5	10	178
		7	9	6	152
		8	15	15	254
		9	< 4	4	339
		10	15	15	108
		11	4	7	162
		12	8	15	267
1	15	0	5	1	90
		1	5	8	336
		2	8	11	39
		3	9	16	203
		4	18	21	171
		5	8	11	348
		6	17	19	19
		7	7	6	178
		8	7	13	203
		9	15	14	3
		10	4	6	43
1	16	0	< 4	1	270
		1	19	18	282
		2	< 4	2	122
		3	16	15	92
		4	< 4	3	349
		5	16	15	279
		6	9	10	136
		7	7	10	221
		8	6	6	309
2	0	0	18	12	180
		1	44	71	90
		2	111	147	180
		3	61	57	90
		4	32	41	0
		5	16	21	270
		6	5	8	0
		7	13	14	270
		8	25	37	180
		9	29	24	270
		10	17	18	0
		11	8	9	270
		12	49	62	180
		13	9	1	270
		14	25	38	0
		15	10	12	270
2	1	0	25	19	0
		1	81	72	172
		2	100	95	268
		3	69	74	61
		4	50	46	155
		5	31	28	235
		6	47	57	293
		7	51	58	127
		8	37	42	73
		9	37	40	325
		10	22	26	246
		11	19	24	166
		12	13	15	277
		13	19	22	44
		14	9	11	151
		15	13	15	193
		16	11	15	260
		17	7	12	76
		18	6	14	85
2	2	0	111	89	0
		1	134	116	185
		2	38	35	156
		3	68	64	11
		4	81	74	276
		5	45	48	111
		6	16	20	69
		7	38	42	159
		8	36	36	260
		9	19	21	352
		10	28	28	49
		11	30	27	170
		12	22	22	195
		13	22	33	18
		14	16	18	7
		15	12	16	173
		16	9	12	98
2	3	0	57	35	180
		1	104	81	278
		2	62	55	41
		3	80	67	89
		4	24	19	358
		5	59	70	245
		6	26	22	19
		7	56	57	119
		8	22	23	260
		9	42	46	266
		10	13	13	122
		11	11	12	101
		12	21	17	319
		13	16	19	76
		14	10	6	134
		15	10	12	265
		16	< 7	6	58
		17	16	19	100
		18	< 5	4	293
		19	5	13	280
2	4	0	55	44	180
		1	76	64	154
		2	14	16	58
		3	48	47	348
		4	62	54	215
		5	28	28	116
		6	65	64	103
		7	42	48	259
		8	22	24	284
		9	24	23	356
		10	25	28	151
		11	29	26	173
		12	29	29	8
		13	25	26	348
		14	8	8	230
		15	8	10	110
		16	13	10	42
		17	< 6	4	208
		18	5	1	251
2	5	0	7	9	180
		1	32	32	358
		2	63	59	99
		3	58	43	137
		4	57	53	259
		5	11	14	213
		6	68	64	86
		7	22	19	95
		8	56	53	287
		9	< 8	9	213
		10	33	31	58
		11	26	26	22
		12	15	15	263
		13	15	14	179
		14	20	20	282
		15	13	13	326
		16	10	15	95
		17	6	9	113
2	6	0	57	60	180
		1	39	37	344
		2	62	55	12
		3	29	23	241
		4	36	33	243
		5	45	45	54
		6	25	24	225
		7	16	15	296
		8	32	32	359
		9	10	10	196
		10	27	26	186
		11	6	8	330
		12	30	30	26
		13	13	16	179
		14	14	17	173
		15	13	10	69
		16	< 7	7	26
		17	17	18	226
		18	6	2	26
2	7	0	30	27	0
		1	34	25	32
		2	63	55	129
		3	32	30	286
		4	57	46	290
		5	41	45	75
		6	26	22	72
		7	24	24	251
		8	30	32	248
		9	25	22	145
		10	22	28	52
		11	27	23	352
		12	21	16	114
		13	8	10	203
		14	15	14	331
		15	9	14	69
		16	16	15	101
2	8	0	15	13	0
		1	62	61	353
		2	18	19	314
		3	59	50	167
		4	25	25	68
		5	29	22	356
		6	16	8	255
		7	6	7	325
		8	34	27	52
		9	19	15	205
		10	12	11	260
		11	39	38	359
		12	8	5	80
		13	27	25	190
		14	< 7	3	187
		15	19	18	5
		16	8	7	202
		17	3	5	213
2	9	0	29	30	0
		1	24	21	86
		2	28	32	182
		3	45	44	285
		4	8	11	44
		5	14	15	162
		6	29	26	289
		7	38	34	289
		8	23	21	93
		9	19	23	60

TABLE 5. (*Continued*).

h	*k*	*l*	$\lvert F_o\rvert$	$\lvert F_c\rvert$	α°
		10	30	23	339
		11	6	7	75
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		13	7	10	288
		14	< 7	3	290
		15	15	16	89
2	10	0	39	39	0
		1	18	25	325
		2	39	37	195
		3	45	42	159
		4	12	17	328
		5	14	16	269
		6	38	33	284
		7	17	14	22
		8	12	15	200
		9	17	19	266
		10	13	14	358
		11	13	11	359
		12	24	24	164
		13	17	18	178
		14	16	14	11
		15	7	11	301
2	11	0	6	3	180
		1	12	13	134
		2	29	27	255
		3	19	19	1
		4	35	35	92
		5	6	9	267
		6	25	25	264
		7	6	8	122
		8	38	36	94
		9	8	4	298
		10	6	8	263
		11	17	13	109
		12	< 7	2	179
		13	15	11	329
		14	7	8	148
		15	7	12	148
2	12	0	26	29	0
		1	17	15	240
		2	12	12	182
		3	21	19	6
		4	21	19	327
		5	12	13	248
		6	25	20	41
		7	20	16	100
		8	20	16	211
		9	5	7	340
		10	19	16	359
		11	5	6	157
		12	18	16	192
		13	22	17	43
		14	7	12	355
2	13	0	15	19	180
		1	19	21	269
		2	12	11	297
		3	8	9	107
		4	12	13	119
		5	20	21	260
		6	< 8	4	268
		7	24	26	100
		8	10	14	61
		9	22	22	284
		10	< 6	7	208
		11	4	4	145
		12	11	9	346
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		1	20	24	196
		2	10	11	82
		3	19	21	20
		4	22	20	223
		5	< 8	2	335
		6	5	10	39
		7	5	7	238
		8	5	7	271
		9	4	9	12
		10	4	7	98
		11	10	13	201
2	15	0	7	10	180
		1	14	17	356
		2	5	9	86
		3	12	17	81
		4	15	18	237
		5	15	16	305
		6	4	10	101
		7	7	12	93
		8	6	8	301
2	16	0	11	13	180
		1	< 6	6	226
		2	12	18	348
		3	< 6	4	303
		4	4	8	184
		5	9	10	84
		6	3	7	19
		7	4	3	159
2	17	0	< 4	3	180
		1	7	11	337
		2	< 4	6	82
		3	< 4	4	163
		4	11	14	285
3	0	1	40	45	90
		2	58	48	0
		3	37	36	270
		4	43	39	180
		5	19	18	90
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		7	23	20	90
		8	52	52	180
		9	40	38	270
		10	27	26	0
		11	45	46	90
		12	6	12	0
		13	35	34	270
		14	< 4	1	0
		15	11	16	90
		16	10	14	0
		17	< 3	5	270
		18	8	11	180
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		2	57	51	336
		3	33	27	24
		4	38	38	88
		5	56	51	208
		6	48	46	10
		7	48	55	322
		8	47	43	221
		9	27	29	185
		10	6	6	39
		11	9	6	254
		12	27	31	291
		13	15	16	86
		14	15	15	138
		15	8	12	229
		16	5	7	291
		17	7	11	347
		18	4	6	180
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		1	24	17	55
		2	108	90	285
		3	72	63	223
		4	72	62	121
		5	22	23	3
		6	14	16	58
		7	49	47	145
		8	31	29	229
		9	39	39	322
		10	15	19	75
		11	6	6	106
		12	15	18	250
		13	19	23	231
		14	13	16	119
		15	7	8	94
		16	7	10	308
		17	8	11	189
		18	4	5	186
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		1	62	53	268
		2	34	31	75
		3	71	54	109
		4	87	83	184
		5	39	33	289
		6	47	46	356
		7	27	25	237
		8	49	45	185
		9	17	20	96
		10	8	5	131
		11	29	31	225
		12	13	12	90
		13	23	23	86
		14	12	13	200
		15	8	8	271
		16	11	16	8
		17	4	7	147
		18	9	13	177
3	4	0	71	59	90
		1	33	24	6
		2	65	60	205
		3	44	38	132
		4	64	59	103
		5	36	33	328
		6	44	40	191
		7	15	16	206
		8	25	19	351
		9	32	35	63
		10	33	34	96
		11	11	12	266
		12	13	13	314
		13	15	17	93
		14	4	10	61
		15	13	14	311
		16	< 4	3	214
		17	5	7	173
		18	7	11	347
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		1	22	22	301
		2	43	40	62
		3	44	43	171
		4	44	37	263
		5	63	61	349
		6	22	23	304
		7	35	38	193
		8	13	15	174
		9	48	47	0
		10	25	26	271
		11	11	10	244
		12	23	23	90
		13	6	7	91
		14	15	15	238
		15	8	12	339
		16	8	7	87
		17	9	13	190
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		1	51	55	225
		2	42	36	123
		3	26	22	122
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		5	31	35	160
		6	36	39	173
		7	24	20	285
		8	29	29	5
		9	17	18	89
		10	22	22	234
		11	32	29	299
		12	6	9	138
		13	12	14	88
		14	8	9	307
		15	6	10	267
		16	8	8	155
		17	5	7	67
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		1	40	34	64
		2	40	41	130
		3	25	22	232
		4	23	21	15
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		6	21	23	187
		7	36	41	181
		8	30	25	55
		9	6	12	273
		10	13	9	195
		11	13	16	113
		12	11	12	107
		13	12	13	233
		14	12	14	281
		15	9	10	38
		16	8	11	188
		17	4	5	217
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		1	13	14	134
		2	67	63	100
		3	24	23	8
		4	40	37	284
		5	21	21	187
		6	12	13	74
		7	40	41	334
		8	20	23	63
		9	24	21	134
		10	15	15	277
		11	9	8	334
		12	26	26	110
		13	< 4	4	359
		14	16	16	266
		15	7	7	127
		16	5	6	93
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		1	17	17	63
		2	21	16	243
		3	39	45	279
		4	14	14	3
		5	14	14	204
		6	31	28	194
		7	31	29	12
		8	10	12	335
		9	9	8	249
		10	4	7	174
		11	18	18	57
		12	13	12	226
		13	8	12	288
		14	10	9	22
		15	6	6	101
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		1	40	41	137
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		4	22	23	235
		5	6	5	79
		6	21	19	19
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		8	21	19	151
		9	8	10	233
		10	16	15	311
		11	13	14	99
		12	< 4	5	61
		13	11	14	273
		14	4	6	244
		15	11	12	124
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		1	13	14	196
		2	22	25	289
		3	32	36	340
		4	21	22	91
		5	28	28	205
		6	11	9	42
		7	27	28	358
		8	8	10	261
		9	21	19	193
		10	15	16	60
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		12	17	14	232
		13	4	5	24
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		1	10	13	26
		2	21	20	338
		3	13	17	238
		4	15	15	172
		5	6	8	336
		6	12	10	320
		7	16	15	132
		8	10	13	193
		9	20	18	299
		10	4	6	61
		11	15	14	106
		12	8	11	284
		13	9	11	286
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		1	9	10	253
		2	6	10	306
		3	18	21	81
		4	14	19	181
		5	14	15	193
		6	13	14	8
		7	9	16	347
		8	19	19	193
		9	3	5	95
		10	13	13	34
		11	6	7	256
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		1	6	10	285
		2	17	22	270
		3	15	16	135
		4	8	11	72
		5	8	11	337
		6	< 4	1	75
		7	11	14	160
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		9	11	14	2
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		2	7	9	29
		3	8	11	143
		4	6	8	223
		5	7	9	1
		6	15	16	9
		7	8	8	212
		8	4	6	121
3	16	0	< 3	6	90
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		2	4	7	306
		3	9	11	124
		4	13	14	11
		5	4	5	281
		6	3	7	213
4	0	0	76	76	0
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		2	31	32	180
		3	53	50	90
		4	4	5	0
		5	35	32	270
		6	11	6	0
		7	32	28	90
		8	9	8	180
		9	35	36	270
		10	53	50	0
		11	15	17	90
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		13	< 4	1	90
		14	13	13	0
		15	4	4	270
		16	3	8	180
		17	8	13	90
4	1	0	39	43	180
		1	49	53	208
		2	53	44	355
		3	54	45	12
		4	34	38	87
		5	44	43	244
		6	29	31	275
		7	13	14	87
		8	17	20	34
		9	37	36	2
		10	14	14	216
		11	17	21	154
		12	15	16	28
		13	22	26	351
		14	14	14	122
		15	13	17	241
		16	7	12	295
4	2	0	69	57	0
		1	77	59	181
		2	44	38	124
		3	34	30	336
		4	55	50	245
		5	41	43	268
		6	41	44	90
		7	16	13	0
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		9	19	21	313
		10	14	9	49
		11	23	27	185
		12	21	23	163
		13	< 4	8	339
		14	10	13	293
		15	7	9	231
		16	8	13	101
		17	3	5	44
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		1	25	20	174
		2	52	53	7
		3	48	48	96
		4	57	47	208
		5	39	37	298
		6	22	22	170
		7	39	36	85
		8	11	12	350
		9	16	19	295
		10	32	33	158
		11	9	9	53
		12	15	15	350
		13	9	11	114
		14	18	26	189
		15	15	16	288
		16	< 3	5	5
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		1	56	47	187
		2	26	25	77
		3	37	31	292
		4	52	44	282
		5	42	36	110
		6	26	26	51
		7	34	31	255
		8	43	38	306
		9	37	30	90
		10	13	11	148
		11	15	15	198
		12	10	11	10
		13	10	13	36
		14	10	16	220
		15	< 3	10	123
		16	6	11	73
		17	5	9	248
4	5	0	14	13	180
		1	42	42	344
		2	27	22	132
		3	45	39	157
		4	47	46	264
		5	16	16	243
		6	33	34	69
		7	10	7	113
		8	20	22	283
		9	20	21	180
		10	22	26	113
		11	15	16	14
		12	6	9	33
		13	16	18	172
		14	7	10	243
		15	14	15	328
		16	8	10	103
4	6	0	22	19	180
		1	36	29	49
		2	22	23	343
		3	38	36	252
		4	17	15	126
		5	41	38	75
		6	11	10	153
		7	52	45	285
		8	28	26	48
		9	36	32	95
		10	21	24	163
		11	< 4	4	321
		12	15	18	8
		13	12	12	213
		14	8	11	192
		15	7	9	62
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		1	32	28	25
		2	57	52	144
		3	25	26	207
		4	32	28	313
		5	15	18	63
		6	14	15	119
		7	32	30	332
		8	11	10	240
		9	13	11	118
		10	25	26	15
		11	10	10	330
		12	16	21	200
		13	9	11	195
		14	15	16	315
		15	< 3	5	90
		16	10	11	134
4	8	0	8	9	180
		1	50	47	23
		2	23	18	261
		3	27	27	184
		4	24	23	65
		5	10	11	46
		6	28	27	260
		7	10	10	226
		8	11	10	84
		9	21	24	192
		10	15	14	221
		11	18	17	12
		12	4	4	45
		13	16	13	204
		14	6	7	65
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		1	18	15	200
		2	28	30	203
		3	15	14	296
		4	25	25	16
		5	29	30	104
		6	11	11	259
		7	< 4	8	179
		8	21	21	150
		9	8	11	73
		10	11	12	345
		11	4	9	213
		12	21	25	176
		13	10	8	297
		14	11	13	7
4	10	0	25	26	0
		1	39	37	358
		2	13	14	209
		3	25	28	76
		4	< 4	4	38
		5	21	21	294
		6	< 4	4	254
		7	33	31	90
		8	15	14	92
		9	16	13	230
		10	12	17	1
		11	16	13	66
		12	13	12	218
		13	4	6	161
		14	10	13	48
4	11	0	11	11	0
		1	22	25	200
		2	10	5	21
		3	19	17	357
		4	25	26	86
		5	< 4	3	228
		6	32	32	294
		7	10	10	146
		8	7	9	91
		9	12	14	330
		10	7	8	310
		11	17	19	180
		12	< 3	1	255
		13	7	8	323
4	12	0	20	20	0
		1	17	16	232
		2	9	10	141
		3	17	19	88
		4	13	13	217
		5	16	21	255
		6	5	8	96
		7	13	17	97
		8	13	16	250
		9	6	11	294
		10	16	15	48
		11	5	5	126
		12	8	12	177
4	13	0	18	20	180
		1	< 4	4	106
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		3	6	12	69
		4	14	13	142
		5	15	19	254
		6	8	9	278
		7	14	16	122
		8	< 3	3	30
		9	9	8	295
4	14	0	< 3	4	180
		1	13	15	177
		2	8	9	30
		3	7	8	353
		4	11	14	240
		5	< 3	6	131
		6	14	18	78
		7	< 3	2	34
		8	5	8	290
		9	9	10	23
4	15	0	10	15	180
		1	10	11	313
		2	12	13	45
		3	9	12	149
		4	< 3	4	171
		5	10	10	290
		6	5	6	50
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		1	5	8	128
		2	10	13	33
		3	7	9	286
5	0	1	59	53	90
		2	26	21	0
		3	55	46	270
		4	82	68	180
		5	24	23	270
		6	73	67	0
		7	8	6	270
		8	15	18	180
		9	16	16	270
		10	31	27	0
		11	25	22	90
		12	5	9	180
		13	5	7	270
		14	11	15	180
		15	10	11	90
		16	11	15	0
		17	4	7	270
5	1	0	52	49	90
		1	30	32	266
		2	33	31	254
		3	41	32	110
		4	31	23	132
		5	28	26	177
		6	38	32	318
		7	27	25	347
		8	16	15	233
		9	27	26	46
		10	19	21	85
		11	20	20	253
		12	14	17	245
		13	8	12	51
		14	7	11	104
		15	11	12	236
		16	9	10	290
		17	5	8	47
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		1	39	28	57
		2	37	29	346
		3	25	22	194
		4	25	23	164
		5	50	49	19
		6	30	25	344
		7	42	36	192
		8	16	17	205
		9	14	14	321
		10	20	25	57
		11	25	24	119
		12	9	13	321
		13	5	7	200
		14	8	9	146
		15	10	14	15
		16	6	6	340
		17	5	11	188
5	3	0	37	32	270
		1	97	74	279
		2	19	16	350
		3	19	19	109
		4	23	21	189
		5	22	17	302
		6	46	39	5
		7	17	17	255
		8	10	10	186
		9	34	34	52
		10	23	24	324
		11	37	39	276
		12	4	6	68
		13	19	22	91
		14	< 3	2	286
		15	10	13	289
		16	6	8	4
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		1	16	16	261
		2	28	23	252
		3	23	22	175
		4	27	24	346
		5	35	31	321
		6	37	31	181
		7	37	33	223
		8	30	30	310
		9	9	10	52
		10	24	23	169
		11	10	12	208
		12	7	7	298
		13	5	7	179
		14	3	3	298
		15	8	7	358
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		1	11	10	272
		2	37	36	84
		3	38	38	155
		4	19	18	270
		5	17	15	9
		6	31	30	9
		7	19	18	201
		8	22	25	116
		9	20	21	72
		10	9	12	259
		11	11	11	244
		12	23	26	90
		13	9	10	132
		14	11	12	263
		15	6	7	317
		16	3	5	62
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		2	17	17	195
		3	24	27	62
		4	43	39	335
		5	22	23	225
		6	53	51	163
		7	28	23	329
		8	31	30	26
		9	11	13	99

TABLE 5. (*Continued*).

h	*k*	*l*	$\|F_o\|$	$\|F_c\|$	α°
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		15	3	6	246
5	7	0	28	30	270
		1	22	21	84
		2	31	30	78
		3	32	26	261
		4	16	14	1
		5	20	19	64
		6	11	8	193
		7	< 4	9	130
		8	11	11	346
		9	8	14	332
		10	21	23	266
		11	14	16	67
		12	8	7	114
		13	16	16	258
		14	< 2	5	339
		15	8	11	60
5	8	0	40	42	270
		1	15	17	177
		2	11	12	88
		3	27	28	4
		4	18	17	322
		5	34	36	193
		6	15	13	225
		7	19	22	355
		8	6	5	54
		9	22	21	160
		10	14	15	289
		11	6	4	65
		12	10	6	107
		13	6	7	342
		14	6	8	281
5	9	0	18	21	90
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		2	10	9	276
		3	10	10	243
		4	28	28	35
		5	15	14	153
		6	7	10	158
		7	5	7	333
		8	7	11	332
		9	19	20	257
		10	9	13	65
		11	19	18	94
		12	9	8	247
		13	14	15	248
		14	5	9	68
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		1	20	22	45
		2	10	11	17
		3	15	14	353
		4	23	24	202
		5	20	21	180
		6	17	18	23
		7	10	11	322
		8	26	23	140
		9	10	9	268
		10	17	14	333
		11	9	9	105
		12	11	10	133
		13	9	10	311
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		1	12	12	354
		2	17	25	275
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		4	11	14	81
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		6	< 3	3	355
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		10	19	20	77
		11	< 2	4	43
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		1	15	17	107
		2	10	10	330
		3	7	6	127
		4	17	18	171
		5	17	18	18
		6	14	14	6
		7	10	10	158
		8	8	11	188
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		10	5	8	40
		11	7	10	141
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		1	25	25	288
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		3	< 3	6	105
		4	8	10	157
		5	12	12	228
		6	13	14	8
		7	6	7	5
		8	6	6	240
		9	7	10	93
5	14	0	< 3	3	90
		1	4	3	21
		2	6	8	252
		3	9	13	166
		4	< 2	2	25
		5	17	18	341
		6	24	10	158
		7	6	11	181
5	15	0	6	7	270
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		2	6	8	86
		3	6	8	105
		4	6	6	205
6	0	0	21	18	0
		1	15	17	270
		2	26	26	180
		3	27	24	90
		4	8	3	0
		5	35	33	270
		6	10	6	0
		7	36	36	90
		8	16	17	0
		9	24	25	270
		10	< 4	0	180
6	0	11	5	4	90
		12	5	7	180
		13	< 3	1	90
		14	< 3	2	0
		15	14	18	270
6	1	0	30	28	180
		1	47	53	198
		2	26	26	308
		3	58	48	17
		4	13	12	148
		5	17	14	131
		6	10	12	256
		7	19	17	170
		8	10	9	112
		9	24	25	339
		10	25	25	152
		11	24	25	172
		12	9	11	331
		13	5	11	3
		14	16	17	140
		15	9	12	174
		16	4	7	309
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		1	6	4	35
		2	20	15	118
		3	37	32	74
		4	55	48	277
		5	11	9	51
		6	39	37	85
		7	11	12	133
		8	35	34	300
		9	16	14	320
		10	14	13	81
		11	8	11	134
		12	5	5	295
		13	14	14	13
		14	5	10	302
		15	5	4	264
6	3	0	42	36	180
		1	17	13	347
		2	38	33	9
		3	4	2	89
		4	13	12	163
		5	18	20	213
		6	13	14	341
		7	23	17	172
		8	23	24	346
		9	14	13	263
		10	22	23	173
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		12	20	24	3
		13	7	6	231
		14	12	14	169
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		1	14	14	193
		2	35	29	46
		3	26	26	273
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		5	45	44	57
		6	42	37	110
		7	31	29	264
		8	23	24	293
		9	15	15	37
		10	9	10	95
		11	7	9	193
		12	8	5	94
		13	4	6	7
		14	7	10	241
		15	4	7	94
6	5	0	9	8	180
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		2	16	14	62
		3	29	22	188
		4	12	12	271
		5	14	13	18
		6	20	16	219
		7	7	3	111
		8	10	12	264
		9	12	15	181
		10	4	4	146
		11	23	27	7
		12	11	8	35
		13	14	16	183
		14	4	3	294
		15	4	7	4
6	6	0	14	15	180
		1	3	4	63
		2	11	10	11
		3	25	21	290
		4	13	13	141
		5	44	42	85
		6	< 4	6	13
		7	36	35	250
		8	5	3	60
		9	7	10	96
		10	17	16	256
		11	< 3	5	227
		12	3	5	16
		13	< 3	1	280
		14	5	5	258
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		5	5	7	94
		6	< 4	2	311
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		9	15	15	142
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		11	15	16	12
		12	13	13	177
		13	13	12	206
		14	11	16	338
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		1	10	8	273
		2	7	10	245
		3	< 4	6	97
		4	24	24	119
		5	13	13	82
		6	24	29	263
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		8	21	24	85
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		12	5	3	132
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		1	17	13	104
		2	22	24	166
		3	25	26	327
		4	7	10	12
		5	< 4	5	85
		6	6	9	311
		7	7	9	294
		8	20	16	202
		9	< 3	8	24
		10	27	24	8
		11	4	3	98
		12	11	15	162
6	10	0	10	10	0
		1	10	14	266
		2	9	13	227
		3	16	17	86
		4	11	11	74
		5	23	24	263
		6	16	15	254
		7	12	13	64
		8	7	6	195
		9	22	20	273
		10	4	6	291
		11	7	5	69
6	11	0	< 4	5	180
		1	18	21	168
		2	< 4	1	174
		3	12	14	343
		4	7	5	206
		5	18	18	182
		6	< 3	3	85
		7	9	9	127
		8	11	10	145
		9	13	14	18
6	12	0	13	17	0
		1	< 3	7	240
		2	10	11	144
		3	17	17	78
		4	13	15	277
		5	17	17	286
		6	10	11	78
		7	10	12	91
		8	11	8	261
		9	5	9	275
6	13	0	14	17	180
		1	4	7	221
		2	12	14	10
		3	8	9	41
		4	7	8	176
		5	< 2	3	248
		6	< 2	3	0
		7	8	10	111
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		1	6	8	289
		2	7	10	90
		3	< 2	2	4
		4	6	11	259

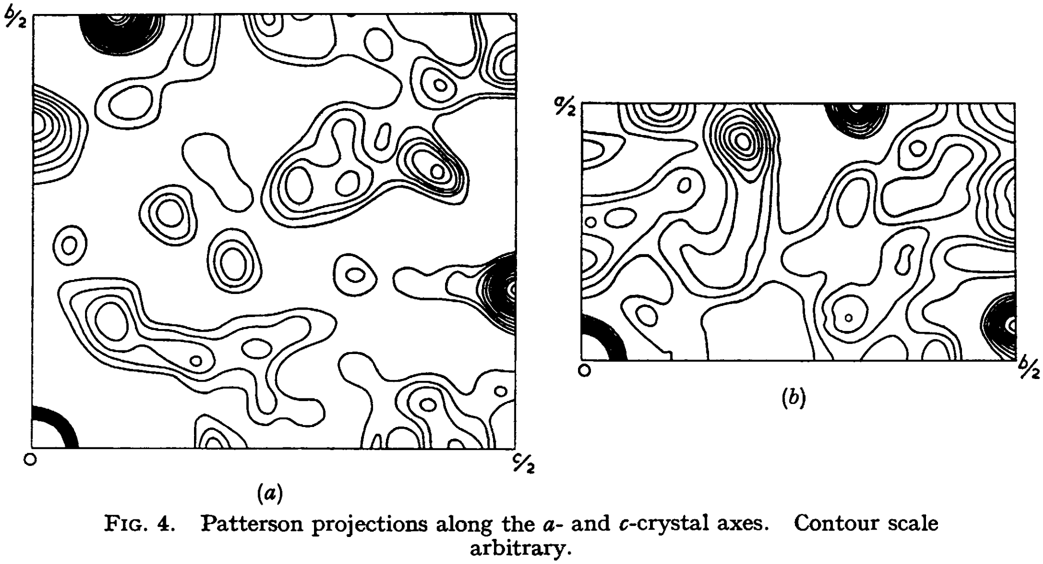

FIG. 4. Patterson projections along the *a*- and *c*-crystal axes. Contour scale arbitrary.

chemical type, the value of R was 0·206. A fourth F_o synthesis was then computed and the co-ordinates from this were corrected for termination-of-series errors by applying back-shift corrections based on a three-dimensional F_c synthesis. This was followed by two cycles of least-squares adjustment [15] of the positional and thermal parameters of the bromine, carbon, and oxygen atoms.

On the basis of the final phase constants (see Table 5) a fifth and final three-dimensional

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[15] Rollett, cf. ref. 14, p. 87.

TABLE 6.

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

	b_{11}	b_{22}	b_{33}	b_{12}	b_{23}	b_{13}		b_{11}	b_{22}	b_{33}	b_{12}	b_{23}	b_{13}
C(1)	1854	509	505	219	165	638	C(12)	2280	461	437	−460	−79	−213
C(2)	1986	635	401	−461	162	1	C(13)	1417	702	582	42	311	17
C(3)	2206	352	459	274	424	−176	C(14)	1617	754	590	512	207	−131
C(4)	864	599	609	637	−55	−215	C(15)	1828	766	526	26	26	128
C(5)	1282	323	496	178	30	−358	C(16)	1643	430	590	−183	6	32
C(6)	1067	496	658	242	−191	9	C(17)	1733	494	766	−243	14	−242
C(7)	2029	557	391	178	212	−132	O(1)	1351	821	692	−65	270	−477
C(8)	1318	437	600	346	−186	3	O(2)	1215	603	532	278	307	−74
C(9)	2177	582	476	310	183	−341	O(3)	2128	705	750	−36	341	−417
C(10)	1566	461	449	274	223	227	O(4)	1954	443	596	−130	354	449
C(11)	1257	499	441	−20	52	−207	O(5)	2785	552	920	180	−272	490
							Br	1594	599	542	4	308	187

electron-density distribution was evaluated and is shown in Fig. 1 by means of superimposed contour sections drawn parallel to (001). All the atoms are very clearly resolved and the oxygen atoms are represented by distinctly higher peaks than the carbon atoms.

For the structure-factor calculations theoretical atomic scattering factors were used; those of Berghuis *et al.*[16] for carbon and oxygen and the Thomas–Fermi values [17] for bromine were chosen. The final value for the discrepancy R is 0·154 over all the observed structure amplitudes. The atomic co-ordinates, molecular dimensions and some non-bonded distances are shown in Tables 1 and 2. The final anisotropic temperature-factor parameters given by the least-squares refinement are shown 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 application 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 3.

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

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