818. *The Structure of Limonin: X-Ray Analysis of Epilimonol Iodoacetate.*

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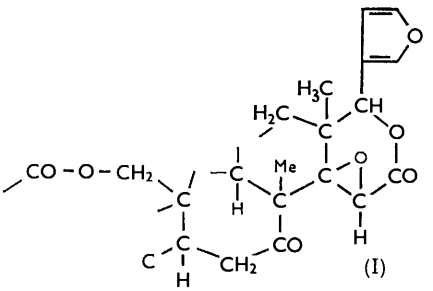
The molecular structure of limonin, $C_{26}H_{30}O_8$, has been determined by an *X*-ray crystallographic study of the heavy atom derivative, epilimonol iodoacetate, $C_{28}H_{33}O_9I$. The iodoacetate crystals are monoclinic, space group C_2^2–$P2_1$, with four molecules per unit cell. The asymmetric crystal unit consists of two chemical molecules. Application of the heavy-atom method and a full three-dimensional analysis with many cycles of refinement have resulted in the complete determination of the molecular structure and stereochemistry of limonin. The chemical behaviour of limonin fully supports the proposed structure.

WHEN our *X*-ray work on limonin, the bitter principle of citrus fruits, began in 1956, knowledge of the chemical structure was limited. The functional groups were known [1] and since vigorous degradation of limonin [2,3] gives 1,2,5-trimethylnaphthalene it could reasonably be expected to contain two six-membered carbocyclic rings. Since that time intensive chemical work,[4] which proceeded independently of our *X*-ray work, has established conclusively the presence of the partial structure (I) in limonin. If the existence of two six-membered rings is assumed, the possibilities are narrowed, and biogenetic arguments may be used to deduce some probable structures.



Even if this chemical knowledge had been available earlier, the complexity of the structural problem, including the unknown stereochemistry, is so great that the only hope of advance seemed to lie in the application of some direct phase-determining method. The most suitable and powerful method was to prepare some derivative with a favourably placed atom or atoms of sufficient scattering power to effect at least partial phase determination [5,6] and enable some of the standard iterative refinement processes to commence. The search for a suitable derivative was actively undertaken by Professor D. H. R. Barton and his collaborators. We examined a number of these compounds, together with various solvates, and elementary crystallographic data have been recorded elsewhere.[7] Epilimonol chloroacetate and iodoacetate appeared to be the most suitable derivatives. Of these the chloroacetate has the smaller unit cell, containing only two molecules related by a screw axis; and much the smaller absorption coefficient. However, the symmetry is such (space group $P2_1$) that the well-known ambiguity of a false symmetry centre would

[1] Melera, Schaffner, Arigoni, and Jeger, *Helv. Chim. Acta*, 1957, **40**, 1420.
[2] Koller and Czerny, *Monatsh.*, 1936, **67**, 248.
[3] Brachvogel, *Arch. Pharm.*, 1952, **57**, 285.
[4] Arigoni, Barton, Corey, and Jeger, in collaboration with Cagliotti, Dev, Ferini, Glazier, Melera, Pradhan, Schaffner, Sternhell, Templeton, and Tobinaga, *Experientia*, 1960, **16**, 41; Barton, Pradhan, Sternhell, and Templeton, *J.*, 1961, 255.
[5] Robertson, *J.*, 1935, 615; 1936, 1195.
[6] Robertson and Woodward, *J.*, 1937, 219; 1940, 36.
[7] Arnott and Robertson, *Acta Cryst.*, 1959, **12**, 75.

have appeared at a later stage of the analysis. In a complex and largely unknown structure this is difficult to resolve, as has been shown for example in our work on isoclovene.[8]

This difficulty does not occur with the iodoacetate because although the space group is the same there are now four molecules in the unit cell, and two in the asymmetric crystal unit. On the other hand, the complexity of the structural problem is greatly increased because we now have to determine the positions of 76 atoms other than hydrogen which are crystallographically unrelated. The choice between these two derivatives was difficult, but two major considerations led us to the iodoacetate. In the first place the greater phase-determining power of the iodine atom would probably be an advantage in a structure of this complexity. Secondly, if the analysis went according to plan, the discovery at a later stage of two crystallographically unrelated but chemically identical molecules would be a most important verification of the work. As it turned out, this factor enabled us to announce the main features of the structure and the stereochemistry of the molecule at a very early stage of the refinement.[9]

The analysis has now been completed, and after some eight cycles of three-dimensional refinement (see Table 1) the discrepancy R over 2927 structure factors is 18·1% (see Table 7).

TABLE 1. *Epilimonol iodoacetate: progress of structure analysis.*

(The first two syntheses and structure factor calculations were carried out on the Manchester University " Mercury " computer. All the later calculations were done on the Glasgow University " Deuce " computer.)

Patterson syntheses and Harker sections. I (2 atoms).

↓

1st F calc. (R = 38%). 1st 3D F_o synthesis.

↓

3D minimum function. 2nd 3D F_o synthesis.
I, I′ + 50 atoms (treated as carbon).

↓

2nd F calc. (R = 26%). 3rd 3D F_o synthesis.
76 atoms of the asymmetric crystal unit.

↓

3rd F calc. (R = 24%). 4th 3D F_o synthesis.

↓

4th F calc. (R = 22%). Least squares refinement for 62 atoms.

↓

5th F calc. (R = 22%). 5th 3D F_o synthesis.

↓

6th F calc. (R = 19·8%). 6th 3D F_o synthesis.

↓

7th F calc. (R = 19·7%). 7th 3D F_o synthesis.

↓

1st 3D F_c synthesis.
Termination of series corrections.

↓

8th F calc. (R = 18·1%). 8th 3D F_o synthesis.

[8] Clunie and Robertson, *Proc. Chem. Soc.*, 1960, 82.
[9] Arnott, Davie, Robertson, Sim, and Watson, *Experientia*, 1960, **16**, 49.

This figure of merit is reasonably satisfactory in view of the complex structure and the great difficulty of collecting accurate data from the poorly formed crystal specimens.

Our final results establish structure (II) for limonin, which is thus shown to be a tetracyclic triterpenoid of the euphol type (III) from which certain carbon atoms in the side chain have been removed and the remainder converted into a furan ring. A full discussion of the chemical implications of this formula and the probable biogenesis has been given by Arigoni, Barton, *et al.*[4]

FIG. 1. *The final three-dimensional electron-density distribution for epilimonol iodoacetate. The superimposed contour sections are drawn parallel to* (010). (Scale, 1 eÅ^{-3} per line.)

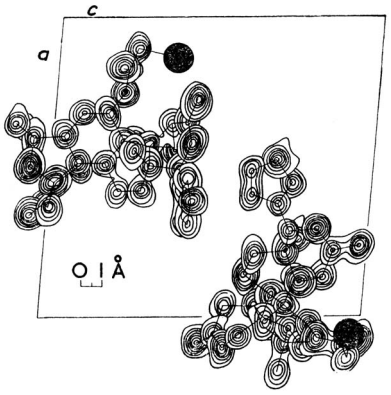
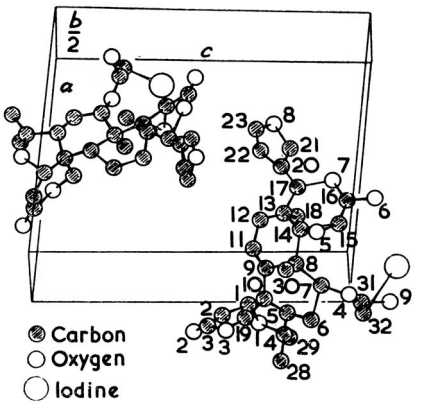
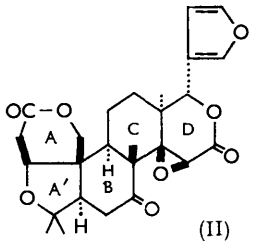


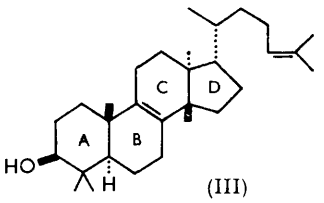
FIG. 2. *The arrangement of the atoms in the crystal asymmetric unit (two chemical molecules).*



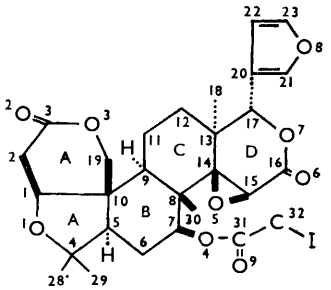
The final electron-density distribution for epilimonol iodoacetate from which our results are deduced is shown in Fig. 1 as superimposed contour sections drawn parallel to (010) and covering the region of two molecules. Fig. 2 gives a roughly perspective



(II)



(III)



(IV)

explanatory drawing. The stereochemistry of epilimonol iodoacetate and also the numbering system used in our subsequent Tables are given in structure (IV). The

7-acetate-oxygen (**4**) is equatorial. This is in agreement with the observation [1,10] that limonin yields epilimonol on reduction with sodium amalgam, a process which yields the more stable of a pair of stereoisomers. Limonol itself must then be the axial isomer.[11] In our structure the 30-methyl group at ring junction B/C is axial. In the *trans*-decalin system ring B has a chair and ring C a boat conformation.

FIG. 3. *Averaged bond lengths.*

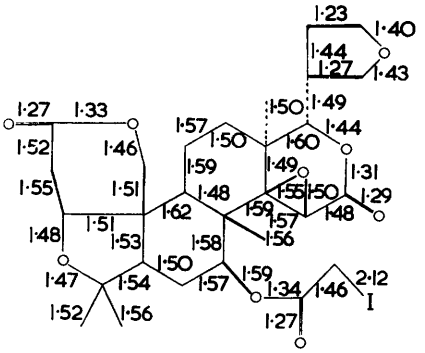


FIG. 4. *Averaged bond angles.*

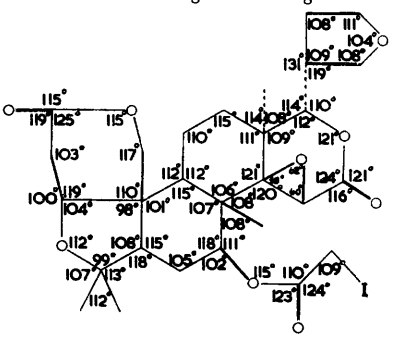


TABLE 2. *Atomic Co-ordinates.*

Atom	*x/a*	*y/b*	*z/c*	Atom	*x/a*	*y/b*	*z/c*
I	0·1360	0·0314	0·3670	C(9′)	0·9300	0·0561	0·7139
I′	1·0622	0·4632	0·9728	C(10′)	1·0047	−0·0245	0·6956
C(1)	0·5489	0·1420	0·0121	C(11′)	0·8610	0·0822	0·6286
C(2)	0·6350	0·0989	0·0028	C(12′)	0·7908	0·1669	0·6501
C(3)	0·6199	−0·0217	−0·0087	C(13′)	0·7832	0·1815	0·7450
C(4)	0·4018	0·0988	−0·0615	C(14′)	0·8777	0·2108	0·7873
C(5)	0·3962	0·1295	0·0338	C(15′)	0·8720	0·2680	0·8723
C(6)	0·3187	0·0999	0·0757	C(16′)	0·7823	0·2884	0·8949
C(7)	0·3256	0·1601	0·1598	C(17′)	0·7200	0·2804	0·7560
C(8)	0·4143	0·1349	0·2154	C(18′)	0·7450	0·0858	0·7847
C(9)	0·4880	0·1661	0·1640	C(19′)	1·0620	0·0217	0·6250
C(10)	0·4884	0·0933	0·0753	C(20′)	0·6259	0·2628	0·7121
C(11)	0·5794	0·1725	0·2129	C(21′)	0·5529	0·2369	0·7520
C(12)	0·5825	0·2711	0·2763	C(22′)	0·5952	0·2918	0·6308
C(13)	0·4957	0·3034	0·3028	C(23′)	0·5166	0·2945	0·6211
C(14)	0·4313	0·2187	0·2982	C(28′)	1·1877	−0·1609	0·7556
C(15)	0·3539	0·2153	0·3606	C(29′)	1·0849	−0·2306	0·8555
C(16)	0·3507	0·3201	0·4080	C(30′)	1·0153	0·2456	0·7062
C(17)	0·5108	0·3473	0·3993	C(31′)	1·0942	0·2320	0·9514
C(18)	0·4508	0·3920	0·2494	C(32′)	1·1440	0·3328	0·9729
C(19)	0·5065	−0·0202	0·0820	O(1)	0·4983	0·1206	−0·0682
C(20)	0·5745	0·4318	0·4159	O(2)	0·6625	−0·0816	−0·0540
C(21)	0·5436	0·5173	0·4255	O(3)	0·5473	−0·0764	0·0217
C(22)	0·6748	0·4372	0·4215	O(4)	0·2519	0·1114	0·2060
C(23)	0·6910	0·5361	0·4403	O(5)	0·4379	0·1436	0·3773
C(28)	0·3524	0·1821	−0·1193	O(6)	0·2769	0·3357	0·4337
C(29)	0·3714	−0·0258	−0·0848	O(7)	0·4224	0·3731	0·4305
C(30)	0·4157	0·0265	0·2514	O(8)	0·6167	0·5965	0·4418
C(31)	0·1733	0·1633	0·2185	O(9)	0·1677	0·2624	0·1947
C(32)	0·1019	0·1050	0·2416	O(1′)	1·0381	−0·2133	0·7063
C(1′)	0·9687	−0·1328	0·6714	O(2′)	1·0913	−0·2103	0·4991
C(2′)	0·9634	−0·1661	0·5701	O(3′)	1·1082	−0·0708	0·5811
C(3′)	1·0588	−0·1425	0·5489	O(4′)	1·0703	0·2263	0·8708
C(4′)	1·0922	−0·1676	0·7772	O(5′)	0·8891	0·3327	0·7992
C(5′)	1·0460	−0·0581	0·7806	O(6′)	0·7678	0·2969	0·9770
C(6′)	1·1008	0·0342	0·8262	O(7′)	0·7098	0·3032	0·8404
C(7′)	1·0269	0·1267	0·8357	O(8′)	0·4798	0·2507	0·6955
C(8′)	0·9575	0·1573	0·7567	O(9′)	1·0861	0·1595	1·0025

A detailed calculation of the bond lengths and bond angles in the two molecules is given in Tables 3 and 4, and the averaged values are shown in Figs. 3 and 4. Standard accuracy

[10] Fujita and Hirose, *J. Pharm. Soc. Japan*, 1954, **74**, 365; 1956, **76**, 129.
[11] Barton, *J.*, 1953. 1027.

calculations are laborious and of limited use in a structure of this degree of complexity. A more realistic idea of the probable errors may be obtained in this case by comparing measurements of corresponding bonds and angles in the two crystallographically independent molecules. These are set out in full in the Tables, and the root mean square deviations

TABLE 3. *Bond lengths* (Å).

	Molecule 1	Molecule 1′	Average		Molecule 1	Molecule 1′	Average
C(1)–C(2)	1·42	1·66	1·55	C(14)–C(8)	1·68	1·49	1·59
C(2)–C(3)	1·51	1·53	1·52	C(8)–C(30)	1·46	1·65	1·56
C(3)–O(2)	1·25	1·28	1·27	C(14)–C(15)	1·60	1·54	1·57
C(3)–O(3)	1·41	1·24	1·33	C(14)–O(5)	1·56	1·53	1·55
O(3)–C(19)	1·37	1·54	1·46	C(15)–O(5)	1·55	1·45	1·50
C(19)–C(10)	1·43	1·58	1·51	C(15)–C(16)	1·50	1·45	1·48
C(10)–C(1)	1·54	1·48	1·51	C(16)–O(6)	1·23	1·35	1·29
C(1)–O(1)	1·45	1·51	1·48	C(16)–O(7)	1·28	1·34	1·31
O(1)–C(4)	1·49	1·45	1·47	O(7)–C(17)	1·49	1·39	1·44
C(4)–C(28)	1·53	1·51	1·52	C(17)–C(13)	1·63	1·57	1·60
C(4)–C(29)	1·64	1·48	1·56	C(13)–C(18)	1·51	1·48	1·50
C(4)–C(5)	1·57	1·52	1·55	C(7)–O(4)	1·51	1·66	1·59
C(5)–C(10)	1·55	1·50	1·53	O(4)–C(31)	1·38	1·30	1·34
C(5)–C(6)	1·44	1·55	1·50	C(31)–O(9)	1·28	1·25	1·27
C(6)–C(7)	1·53	1·61	1·57	C(31)–C(32)	1·37	1·54	1·46
C(7)–C(8)	1·56	1·60	1·58	C(32)–I	2·21	2·02	2·12
C(8)–C(9)	1·49	1·47	1·48	C(17)–C(20)	1·43	1·54	1·49
C(9)–C(10)	1·68	1·55	1·62	C(20)–C(21)	1·17	1·36	1·27
C(9)–C(11)	1·52	1·66	1·59	C(21)–O(8)	1·48	1·37	1·43
C(11)–C(12)	1·58	1·55	1·57	O(8)–C(23)	1·34	1·46	1·40
C(12)–C(13)	1·46	1·54	1·50	C(23)–C(22)	1·28	1·18	1.23
C(13)–C(14)	1·42	1·56	1·49	C(22)–C(20)	1·50	1·38	1·44

TABLE 4. *Bond angles.*

	Mo e-cule 1	Mole-cule 1′	Average		Mole-cule 1	Mole-cule 1′	Average
I–C(32)–C(31)	110°	108°	109°	C(17)–C(20)–C(21)	114°	124°	119°
C(32)–C(31)–O(9)	123	125	124	C(21)–C(20)–C(22)	111	106	109
C(32)–C(31)–O(4)	120	99	110	C(20)–C(22)–C(23)	103	112	108
O(9)–C(31)–O(4)	115	130	123	C(22)–C(23)–O(8)	113	109	111
C(31)–O(4)–C(7)	124	105	115	C(23)–O(8)–C(21)	104	103	104
O(4)–C(7)–C(6)	104	99	102	O(8)–C(21)–C(20)	109	107	108
C(7)–C(6)–C(5)	106	103	105	C(17)–C(13)–C(18)	108	108	108
C(6)–C(5)–C(10)	117	112	115	C(18)–C(13)–C(12)	114	113	114
C(5)–C(10)–C(9)	98	104	101	C(10)–C(5)–C(4)	103	112	108
C(10)–C(9)–C(8)	112	117	115	C(5)–C(4)–O(1)	100	98	99
C(9)–C(8)–C(7)	106	108	107	C(5)–C(4)–C(29)	114	112	113
C(8)–C(7)–C(6)	113	120	117	C(29)–C(4)–C(28)	113	111	112
C(8)–C(7)–O(4)	106	115	111	C(28)–C(4)–O(1)	105	108	107
C(7)–C(8)–C(30)	113	102	107	C(4)–O(1)–C(1)	114	110	112
C(30)–C(8)–C(14)	105	110	108	O(1)–C(1)–C(2)	101	99	100
C(9)–C(8)–C(14)	102	109	106	O(1)–C(1)–C(10)	102	106	104
C(8)–C(14)–C(13)	123	119	121	C(1)–C(10)–C(5)	100	96	98
C(14)–C(13)–C(12)	114	107	111	C(1)–C(10)–C(19)	108	111	110
C(13)–C(12)–C(11)	115	114	115	C(10)–C(19)–O(3)	123	111	117
C(12)–C(11)–C(9)	110	110	110	C(19)–O(3)–C(3)	114	116	115
C(11)–C(9)–C(8)	114	110	112	O(3)–C(3)–C(2)	124	126	125
C(8)–C(14)–O(5)	105	113	109	C(3)–C(2)–C(1)	104	102	103
O(5)–C(14)–C(15)	59	56	58	C(2)–C(3)–O(2)	122	115	119
C(14)–C(15)–O(5)	59	61	60	O(2)–C(3)–O(3)	111	118	115
C(15)–O(5)–C(14)	62	62	62	C(4)–C(5)–C(6)	120	116	118
C(13)–C(14)–C(15)	121	111	116	C(19)–C(10)–C(9)	118	110	114
C(14)–C(15)–C(16)	111	115	113	C(10)–C(9)–C(11)	112	113	113
O(5)–C(15)–C(16)	118	110	114	C(2)–C(1)–C(10)	121	117	119
C(15)–C(16)–O(7)	122	125	124	C(22)–C(20)–C(17)	134	127	131
C(15)–C(16)–O(6)	112	119	116	C(13)–C(14)–O(5)	114	112	113
O(6)–C(16)–O(7)	126	115	121	C(18)–C(13)–C(14)	104	112	108
C(16)–O(7)–C(17)	124	117	121	C(19)–C(10)–C(5)	118	122	120
O(7)–C(17)–C(13)	110	113	112	C(1)–C(10)–C(9)	113	112	112
C(17)–C(13)–C(14)	109	108	109	C(9)–C(8)–C(30)	118	118	118
O(7)–C(17)–C(20)	113	107	110	C(7)–C(8)–C(14)	113	110	111
C(13)–C(17)–C(20)	116	112	114	C(12)–C(13)–C(17)	108	108	108

TABLE 5. *Some intramolecular non-bonded distances* (Å).

	Molecule 1	Molecule 1′	Average		Molecule 1	Molecule 1′	Average
I–O(4)	3·38	3·36	3·37	C(4)–C(9)	3·80	3·76	3·78
I–O(9)	4·02	3·79	3·91	C(4)–C(19)	3·04	3·37	3·21
I–C(7)	4·83	4·70	4·77	C(5)–C(30)	3·68	3·95	3·82
I–C(8)	5·18	5·25	5·22	C(6)–C(19)	3·18	3·21	3·20
I–C(31)	2·96	2·92	2·94	C(6)–C(29)	3·15	3·32	3·24
O(1)–O(2)	3·50	3·47	3·49	C(6)–C(30)	3·17	3·42	3·30
O(2)–C(19)	3·22	3·55	3·49	C(6)–C(31)	3·39	3·16	3·28
O(3)–C(4)	3·27	3·37	3·32	C(6)–C(32)	4·38	4·39	4·39
O(3)–C(5)	3·43	3·40	3·42	C(7)–C(19)	3·81	3·68	3·75
O(4)–C(5)	3·65	3·80	3·73	C(7)–C(31)	2·56	2·39	2·48
O(4)–C(6)	2·39	2·53	2·46	C(7)–C(32)	3·78	3·70	3·74
O(4)–C(8)	2·44	2·51	2·48	C(8)–C(16)	4·01	3·93	3·97
O(4)–C(9)	3·73	3·76	3·75	C(8)–C(18)	3·26	3·38	3·32
O(5)–O(6)	3·56	3·53	3·55	C(8)–C(31)	3·64	3·66	3·65
O(5)–C(9)	3·56	3·75	3·66	C(8)–C(32)	4·76	4·76	4·76
O(5)–C(12)	3·23	3·37	3·30	C(9)–C(15)	3·92	3·79	3·86
O(5)–C(18)	3·70	3·74	3·72	C(9)–C(18)	3·18	3·12	3·15
O(6)–C(14)	3·61	3·73	3·67	C(10)–C(28)	3·72	3·29	3·51
O(6)–C(17)	3·61	3·53	3·57	C(10)–C(29)	3·31	3·72	3·52
O(7)–O(8)	4·01	4·03	4·02	C(10)–C(30)	3·21	3·35	3·28
O(7)–C(12)	3·81	3·76	3·79	C(11)–C(17)	3·89	3·92	3·91
O(7)–C(20)	2·44	2·35	2·40	C(11)–C(18)	3·41	3·17	3·29
O(7)–C(21)	2·56	2·76	2·66	C(12)–C(15)	3·86	3·85	3·86
O(7)–C(22)	3·89	3·62	3·76	C(12)–C(22)	3·30	3·31	3·31
O(7)–C(23)	4·24	4·33	4·29	C(13)–C(20)	2·61	2·57	2·59
O(8)–C(13)	4·52	4·63	4·58	C(13)–C(21)	3·33	3·54	3·44
O(8)–C(17)	3·51	3·67	3·59	C(13)–C(22)	3·55	3·50	3·53
O(9)–C(6)	3·68	3·25	3·47	C(13)–C(23)	4·53	4·52	4·53
O(9)–C(7)	2·79	2·76	2·78	C(14)–C(20)	3·79	3·92	3·86
O(9)–C(8)	4·00	4·21	4·11	C(15)–C(18)	3·24	3·19	3·22
C(1)–C(8)	3·97	3·84	3·91	C(16)–C(20)	3·63	3·59	3·61
C(1)–C(11)	3·21	3·15	3·18	C(16)–C(21)	3·78	4·01	3·90
C(2)–C(9)	3·64	3·64	3·64	C(17)–C(21)	2·19	2·56	2·38
C(2)–C(11)	3·64	3·59	3·62	C(17)–C(22)	2·70	2·62	2·66
C(3)–C(4)	3·63	3·64	3·64	C(17)–C(23)	3·59	3·58	3·59
C(3)–C(5)	3·96	3·86	3·91	C(18)–C(21)	3·39	3·44	3·42
C(4)–C(7)	3·88	3·90	3·89				

TABLE 6. *Some intermolecular distances* (Å).*

O(8′)–C(21$_{VI}$)	3·47	C(28′)–C(12$_{VII}$)	3·63
C(21)–O(5$_{IV}$)	3·50	C(6)–O(9′$_{II}$)	3·66
C(30′)–O(2′$_{V}$)	3·55	C(30)–C(17′$_{VI}$)	3·66
C(31)–O(9′$_{II}$)	3·57	O(8)–C(16$_{IV}$)	3·66
O(6′)–C(29$_{IV}$)	3·58	C(29′)–C(32$_{VI}$)	3·69
C(30)–O(7′$_{VI}$)	3·58	I–O(3′$_{II}$)	3·70
C(17)–C(23′)	3·59	O(2)–C(5$_{III}$)	3·70
C(23′)–C(21$_{VI}$)	3·60	I–O(2′$_{II}$)	3·75
I–O(5′$_{VI}$)	3·60	C(20)–C(22′)	3·82
C(23′)–C(30$_{IV}$)	3·61	C(17)–C(22′)	3·85
O(6)–C(2′$_{IV}$)	3·61	C(20)–C(23′)	3·85
O(1′)–O(9$_{VI}$)	3·61	C(32)–O(9′$_{II}$)	3·85
C(21′)–C(30$_{IV}$)	3·61	I–C(3′$_{II}$)	3·87
O(2)–C(7$_{III}$)	3·62	O(4)–O(9′$_{II}$)	3·95
C(28)–O(3$_{I}$)	3·63	C(16)–C(23′)	4·04

* The subscripts refer to the following positions:

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

are 0·112 Å for the bond lengths and 6·7° for the bond angles. For the averaged dimensions given in Figs. 3 and 4 we may divide by $\sqrt{2}$ and give 0·08 Å and 4·8° as reasonable estimates of the standard deviations.

In spite of these fairly large uncertainties the overall geometry of the two independently observed molecules is strikingly similar in every detail. Even where there is possibility

of free rotation about single bonds, as in the iodoacetate group and in the junction of the furan ring with ring D, the similarity appears to be closer than might have been expected. It is not easy to see this without examining a three-dimensional model of the electron-density distribution, but the comparison of certain corresponding non-bonded distances

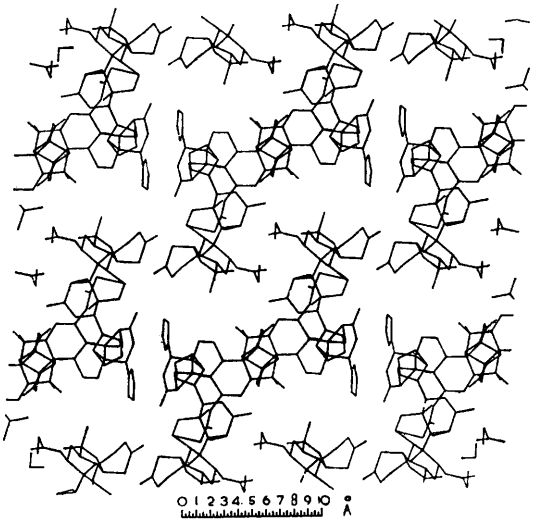


FIG. 5. *Line drawing of molecular framework of several unit cells projected along b.*

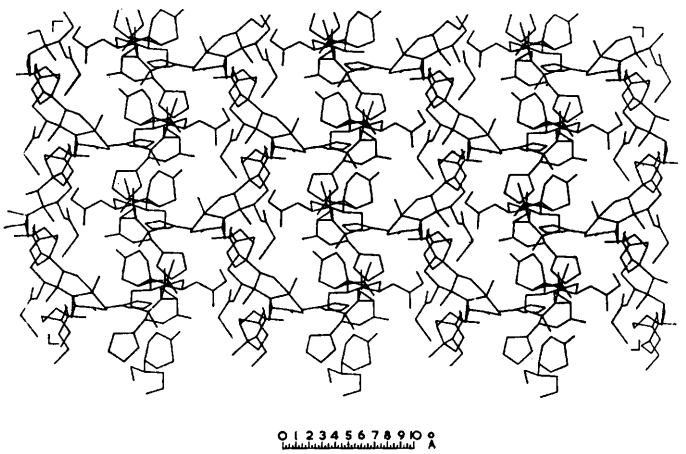


FIG. 6. *Line drawing of molecular framework of several unit cells projected along c. Two-fold screw axis vertical.*

in the two molecules (Table 5) may be used as a test. Thus, in the region of the iodoacetate group we have, for the two molecules, O(4)–C(5) = 3·65 and 3·70 Å, O(4)–C(9) = 3·73 and 3·76 Å, C(31)–C(8) = 3·64 and 3·66 Å, C(32)–C(6) = 4·38 and 4·39 Å, C(32)–C(8) = 4·76 and 4·76 Å. With regard to the orientation of the furan ring and the main skeleton of the molecule we have O(8)–O(7) = 4·01 and 4·03 Å, C(22)–C(13) = 3·55 and 3·50 Å, C(23)–C(13) = 4·53 and 4·52 Å, C(23)–C(17) = 3·59 and 3·58 Å. This very close correspondence of the two crystallographically independent molecules is a phenomenon similar to that observed in the structure of 10-bromo-2-chloro-2-nitrosocamphane [12] and it indicates a somewhat unexpected constancy of molecular shape in different environments in the solid state.

The average carbon–carbon single bond length is 1·52 Å, not significantly different from the value of 1·545 Å in diamond. In the two carbonyl groups the average carbon–oxygen distance is 1·28 Å, and in the two lactone rings the carbon–oxygen single bond

[12] Ferguson, Fritchie, Robertson, and Sim, *J.*, 1961, 1976.

distance in the system –O–C(=O)– is 1·32 Å. These distances compare favourably with the values usually found in carboxylic acids. In the epoxide ring the average carbon–oxygen bond length is 1·53 Å, while the values quoted for ethylene oxide [13] and cyclopentene oxide [14] are 1·44 and 1·47 Å respectively. Comparison of the bond lengths within the furan ring with those cited for furan itself [15] shows that there is no significant deviation from the expected values although the carbon–carbon double bond at 1·25 Å is rather short. In the iodoacetate group the distances are normal and the carbon–iodine bond length of 2·12 Å compares very favourably with the value of 2·14 Å quoted for various alkyl iodides.[16,17]

A reasonable sample of the intermolecular contacts has been calculated (Table 6) and none of these appears to be abnormal, the closest approach being 3·47 Å which occurs between the furan rings of two adjoining molecules. Fig. 1 gives some indication of the mutual arrangement of the two molecules in the asymmetric crystal unit, but it is difficult to provide a complete picture showing all the intermolecular contacts. Projections of the contents of several unit cells are shown in Figs. 5 and 6 by means of line drawings of the superimposed molecular frameworks. In Fig. 6 the effect of the vertical two-fold screw axis is clearly visible.

Experimental

Crystal Data.—Epilimonol iodoacetate, $C_{28}H_{33}O_9I$; *M*, 640·5; m. p. 211—214° (decomp.); *d*, calc. 1·441, found 1·426. Monoclinic, $a = 15{\cdot}03$, $b = 12{\cdot}36$, $c = 15{\cdot}93$ all $\pm 0{\cdot}02$ Å, $\beta = 95° 12' \pm 15'$. Absent spectra, $(0k0)$ when k is odd. Space group, $P2_1$ (C_2^2) or $P2_1/m$ (C_{2h}^2). $P2_1/m$ must be rejected because the ester is optically active. Four molecules per unit cell, two in the asymmetric crystal unit. Volume of the unit cell = 2953 Å^3. Absorption coefficients for *X*-rays, ($\lambda = 1{\cdot}542$ Å) $\mu = 96{\cdot}0$ cm.$^{-1}$, ($\lambda = 0{\cdot}7107$ Å) $\mu = 12{\cdot}3$ cm.$^{-1}$. Total number of electrons per unit cell = $F(000) = 1304$.

Crystals were grown from solutions in acetone–water and could only be obtained in the form of plates never thicker than 0·02 mm., with (100) strongly developed. No other faces could be identified.

Experimental Measurements.—Rotation, oscillation, moving-film, and precession photographs were taken with copper-K_α ($\lambda = 1{\cdot}542$ Å) and molybdenum-K_α ($\lambda = 0{\cdot}7107$ Å) radiation. Cell dimensions were obtained from calibrated equatorial layer line and precession photographs. For the three-dimensional intensity survey molybdenum radiation was used throughout to minimise absorption errors, because no systematic calculation of corrections was feasible. The average size of the crystal specimens employed was about 1·0 × 0·2 × 0·02 mm. Photographic records were obtained by using equi-inclination Weissenberg and precession cameras and exposures of 80—100 hr. were frequently necessary for the weaker reflections. For correlation of strong and weak reflections the multiple-film technique [18] with interleaving sheets of nickel foil, 0·008 in. thick, was used, as well as timed exposures. The reciprocal lattice was explored by recording the intensities of the $(h0l)$–$(h8l)$ and $(0kl)$–$(6kl)$ layers with a Weissenberg camera and the $(hk0)$–$(hk3)$ layers with a precession camera. Intensities were estimated visually by several independent observers using standard spot and step-wedge techniques. The usual correction factors (Lorentz, polarisation, and rotation) were applied and 2927 independent structure factors were finally evaluated by the mosaic crystal formula (Table 7).

Analysis of the Structure.—The positions of the iodine atoms were determined without ambiguity at an early stage in this work by the calculation of two-dimensional Patterson syntheses along the *a* and *b* crystal axes. These projections, prepared from limited data, are shown in Figs. 7 and 8. The iodine co-ordinates were confirmed and refined later by the

[13] Cunningham, Boyd, Myers, Gwinn, and Le Van, *J. Chem. Phys.*, 1951, **19**, 676.
[14] Erlandsson, *Arkiv Fysik*, 1955, **9**, 341.
[15] Bak, Hausen, and Rastrup-Andersen, *Discuss. Faraday Soc.*, 1955, **19**, 30.
[16] Miller, Aamodt, Donsmanis, Townes, and Kraitchman, *J. Chem. Phys.*, 1952, **20**, 1112.
[17] Lister and Sutton, *Trans. Faraday Soc.*, 1941, **37**, 393.
[18] Robertson, *J. Sci. Instr.*, 1943, **20**, 175.

evaluation of various Harker sections and Fourier syntheses. It will be noted (Table 2) that the y co-ordinates of the two iodine atoms in the asymmetric unit differ by only 0·84 Å (in fractional co-ordinates, $\frac{1}{2} + 0{\cdot}0314$ and 0·4632). If these co-ordinates had been equal the phase ambiguity of a false symmetry centre would have recurred. It is of considerable interest to find that even this small separation of 0·84 Å is sufficient to destroy completely any false symmetry, as is shown by our subsequent analysis.

FIG. 7. *Projection of Patterson function along a axis.*

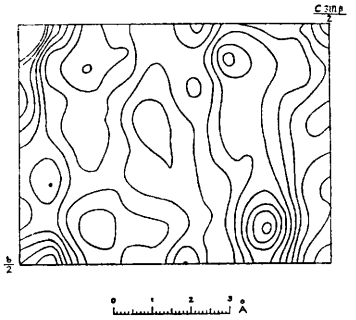


FIG. 8. *Projection of Patterson function along b axis.*

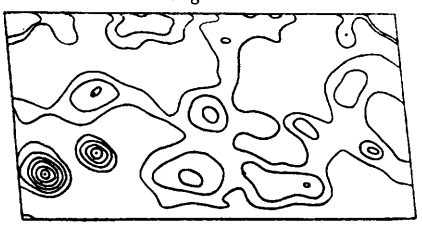


FIG. 9. *Projection of electron density distribution on* (010).

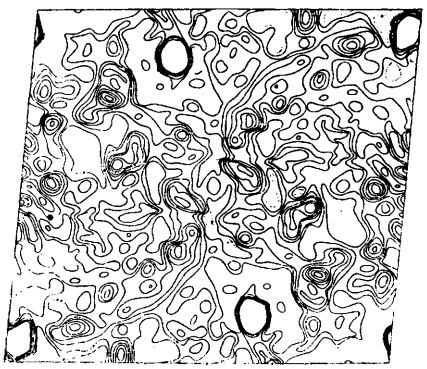
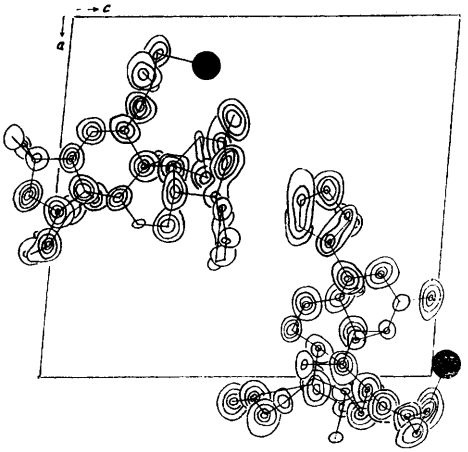


FIG. 10. *The third three-dimensional electron-density distribution shown by means of superimposed contour sections drawn parallel to* (010). *At this stage the bottom right-hand molecule was attached to the wrong iodine atom. Another symmetry-related iodine (not shown) lying closer to the acetate group should have been used. This error does not affect the molecular structure.*



Phase angles calculated from the iodine positions were then associated with the observed structure amplitudes, and electron-density projections on (100) and 010) were calculated, the latter being shown in Fig. 9. The complex overlapping molecular frameworks responsible for this projection are shown in Fig. 5. Interpretation in terms of molecular structure was, of course, quite impossible at this stage, but these projections did provide a useful refinement of the iodine co-ordinates.

All the subsequent work was based on the full three-dimensional data (Table 7). A first Fourier synthesis with phase angles calculated from the iodine contributions alone was evaluated on the "Mercury" computer at Manchester University. Interpretation of the resulting electron distribution proved impossible, but at a later stage it was found that a programme error was responsible for some of the confusion in this synthesis.

A sharpened three-dimensional Patterson synthesis was now computed and from this a

TABLE 7. *Observed and calculated structure factors.*

h	k	l	$\|F_o\|$	$\|F_c\|$	α°	h	k	l	$\|F_o\|$	$\|F_c\|$	α°	h	k	l	$\|F_o\|$	$\|F_c\|$	α°	h	k	l	$\|F_o\|$	$\|F_c\|$	α°
0	0	3	219	195	0			2	72	79	277			17	24	21	180			−1	180	209	258
		4	48	50	0			3	29	21	311			−1	132	216	0			−2	55	54	152
		5	207	198	0			4	98	102	260			−2	76	81	180			−3	30	30	222
		6	25	2	0			5	54	57	343			−3	17	3	0			−4	109	99	254
		7	12	2	180			6	32	30	315			−5	26	17	180			−5	52	51	151
		8	55	46	0			7	52	41	254			−6	236	205	0			−6	112	118	253
		10	29	17	180			8	42	36	37			−7	85	65	180			−7	78	64	265
		11	21	19	0			10	19	29	256			−9	53	32	0			−8	87	78	195
		12	68	46	180			11	44	35	66			−10	88	65	180			−9	90	76	272
		14	41	25	0			14	24	15	103			−11	10	5	180			−10	37	32	289
		15	75	52	180			15	13	12	175			−12	55	38	180			−11	28	26	174
		17	14	12	180			16	24	22	95			−13	77	58	180			−12	21	30	279
		18	37	23	180	0	7	1	35	38	146			−14	16	1	0			−13	35	28	177
0	1	2	107	115	26			2	73	80	5			−15	37	31	180			−14	18	16	243
		3	16	22	77			3	8	14	250			−16	24	21	180			−15	16	19	299
		4	173	147	55			4	15	17	327			−18	29	22	180			−16	24	18	115
		5	64	56	265			5	74	60	0	1	1	3	78	83	93	1	4	0	112	120	6
		6	204	174	94			6	32	23	46			4	77	68	299			1	105	127	292
		7	49	38	70			7	59	49	359			5	78	68	106			2	120	132	344
		8	25	24	50			8	45	38	4			6	73	62	69			3	105	109	296
		9	144	133	70			9	21	11	333			7	34	29	303			4	59	62	241
		11	92	72	77			10	49	41	357			8	95	84	42			5	90	88	1
		12	69	47	82			11	22	19	36			9	66	51	82			6	34	39	303
		14	48	37	91			12	25	20	349			10	15	21	32			7	27	31	283
		15	13	16	122			13	46	37	338			11	60	56	79			8	29	36	8
		17	19	15	91			14	19	9	226			12	32	33	54			9	62	53	214
0	2	0	49	32	63			16	16	16	356			13	18	16	44			10	14	17	343
		1	57	50	72	0	8	1	69	69	270			14	18	29	102			12	53	44	211
		2	153	201	334			2	49	56	253			15	17	11	324			13	16	10	90
		3	142	148	355			4	65	64	267			17	18	16	97			15	16	13	237
		4	70	56	239			5	30	22	181			−3	161	153	256			16	21	17	71
		5	121	109	359			6	11	14	103			−4	171	150	285			17	19	14	193
		6	27	21	84			7	26	25	259			−5	62	51	216			−1	47	63	356
		7	119	98	238			8	49	43	101			−6	122	104	254			−2	77	88	295
		8	59	46	57			9	27	23	267			−7	85	85	267			−3	58	56	37
		10	46	37	201			10	15	9	255			−8	16	12	254			−4	70	66	255
		11	77	54	51			11	40	28	77			−9	82	73	262			−5	28	33	274
		12	24	18	219			14	16	14	87			−10	37	40	256			−6	64	63	57
		13	46	31	106	0	9	1	26	26	183			−11	23	20	158			−7	80	65	228
		14	29	22	97			2	43	46	334			−12	54	50	277			−8	35	29	86
		15	38	29	165			4	28	27	333			−13	15	13	116			−10	44	36	171
		16	15	14	106			5	57	49	3			−15	17	17	255			−11	54	46	87
		17	15	15	145			6	11	24	223			−16	18	18	109			−12	38	30	146
		18	27	19	157			7	41	40	318	1	2	0	79	119	326			−13	15	19	174
0	3	1	103	116	155			8	47	38	338			1	121	154	266			−14	33	29	71
		2	91	100	301			9	21	23	221			2	30	42	311			−15	15	18	168
		3	66	68	109			10	29	28	0			3	144	170	17			−16	18	11	138
		4	78	65	76			11	20	11	281			4	102	100	224			−17	18	17	89
		5	45	44	53			13	14	18	3			5	138	128	4	1	5	0	57	69	122
		6	36	34	106	0	10	0	30	35	161			6	100	89	294			1	41	55	204
		7	105	81	355			1	39	40	267			7	42	36	44			2	46	63	340
		8	47	43	16			2	18	13	246			8	103	89	35			3	52	63	125
		9	77	67	89			3	14	22	174			9	79	69	217			4	65	66	344
		10	19	17	35			4	41	39	263			10	29	29	1			5	33	39	339
		11	23	20	2			5	45	32	140			11	16	18	3			6	77	73	126
		12	36	32	58			6	17	16	226			12	41	29	169			7	90	81	2
		13	21	19	347			7	35	25	247			13	34	28	15			8	51	51	80
		15	15	8	337			8	13	7	139			14	20	16	205			9	37	28	26
		17	16	17	129		11	2	35	32	35			15	38	29	203			10	63	56	6
0	4	0	91	87	33			3	26	27	316			16	18	10	90			11	46	37	103
		1	152	145	269			4	24	26	221			17	18	14	155			12	45	32	23
		2	59	68	253			5	27	23	352			−1	101	69	183			13	30	27	36
		3	90	94	344			6	29	26	278			−2	83	119	229			−1	32	43	21
		4	105	93	247			8	19	18	338			−3	114	145	349			−2	51	70	183
		5	58	41	209			9	28	25	260			−4	41	40	293			−3	59	72	191
		6	68	54	345			10	27	18	342			−6	82	88	36			−4	68	74	236
		7	63	45	279			11	17	13	258			−7	57	58	192			−5	76	89	180
		8	70	52	14	0	12	0	24	29	148			−8	24	25	76			−6	18	26	211
		9	48	41	216			1	24	23	258			−9	19	14	119			−7	36	31	211
		11	51	44	68			2	21	17	209			−10	63	57	187			−8	55	63	157
		12	59	40	198			3	29	24	191			−11	34	34	58			−9	35	38	261
		13	25	21	127			4	13	17	283			−12	56	50	166			−10	29	27	232
		14	24	21	93			6	20	17	212			−13	41	32	178			−11	30	36	193
		15	36	28	193	0	13	3	19	14	268			−14	10	18	129			−12	37	33	284
		16	24	22	89			6	27	24	253			−15	45	42	168			−13	14	17	195
		18	29	18	168			8	24	8	312			−16	18	17	156			−14	14	10	359
0	5	1	48	41	59	0	14	0	24	23	159			−18	20	17	191			−15	21	21	288
		2	59	58	327			1	23	10	218	1	3	0	119	127	188	1	6	0	43	42	11
		3	66	67	79			3	21	14	162			1	41	55	248			1	40	65	268
		4	7	14	71	0	15	7	22	5	228			2	39	40	289			2	63	66	29
		5	86	93	352	0	16	0	24	19	183			3	91	102	112			3	38	25	327
		6	76	61	97	1	0	2	32	52	0			4	47	55	271			4	118	126	296
		7	65	60	15			3	78	61	0			5	26	24	359			5	32	27	6
		8	67	59	9			4	57	47	0			6	59	51	73			6	48	44	281
		9	62	48	74			5	146	138	0			7	49	47	9			7	54	48	260
		10	76	68	350			6	27	20	0			8	45	38	48			9	39	38	271
		11	27	28	8			7	25	21	0			9	99	70	73			10	16	8	134
		12	26	18	67			8	108	112	0			10	38	42	354			11	37	34	55
		13	32	27	348			9	24	16	180			11	56	49	88			12	31	25	249
		14	16	21	50			10	39	35	0			12	41	35	52			13	38	29	81
		16	19	13	332			12	68	52	180			13	29	26	345			14	19	9	109
0	6	0	82	90	293			14	19	16	180			14	54	41	78			−1	27	38	297
		1	137	139	264			15	37	28	180			15	21	18	12			−2	79	95	254

TABLE 7. (*Continued.*)

h	k	l	$\|F_o\|$	$\|F_c\|$	α°
		−3	71	84	27
		−4	63	60	286
		−5	16	18	247
		−6	24	21	83
		−7	55	55	258
		−8	19	16	55
		−9	20	18	50
		−10	26	20	228
		−11	50	39	91
		−12	18	7	73
		−14	30	30	87
		−15	23	14	101
1	7	0	31	40	198
		1	12	25	54
		2	12	31	300
		3	59	61	168
		4	54	48	14
		5	43	27	20
		6	12	13	121
		7	51	52	327
		8	15	8	54
		9	19	14	331
		10	40	40	358
		11	27	14	83
		12	27	22	344
		13	33	30	353
		15	22	20	22
		−2	66	77	187
		−3	40	43	192
		−5	88	84	164
		−6	51	36	185
		−7	66	58	201
		−8	84	75	183
		−9	15	5	162
		−10	27	25	171
		−11	37	32	188
		−12	18	13	303
		−13	40	32	150
1	8	0	30	29	214
		1	77	84	264
		2	10	13	224
		3	40	36	250
		4	85	83	256
		5	23	20	220
		6	45	43	282
		7	32	28	277
		8	13	7	76
		9	54	49	272
		10	27	17	139
		11	29	17	110
		13	21	16	86
		−1	14	19	254
		−2	30	30	254
		−3	19	25	134
		−4	46	41	216
		−5	27	32	273
		−6	33	32	60
		−7	30	29	275
		−8	24	14	94
		−9	41	33	103
		−11	42	33	82
		−12	21	18	83
		−13	17	12	75
		−14	39	30	100
1	9	0	54	62	163
		2	31	30	19
		7	38	34	345
		8	29	18	317
		9	27	22	333
		10	38	30	344
		11	18	21	280
		12	23	21	337
		13	23	20	349
		−1	18	22	38
		−2	43	38	166
		−3	43	42	141
		−4	15	26	68
		−5	77	72	198
		−6	40	34	157
		−7	16	20	120
		−8	63	55	167
		−9	16	15	166
		−10	17	21	182
		−11	39	30	149
1	10	0	37	39	188
		1	40	42	239
		2	32	34	199
		3	46	38	238
		4	54	48	258
		5	24	30	159
		6	26	29	250
		7	25	21	260
		8	17	12	135
		13	19	15	115
		−1	28	25	224
		−2	40	38	266
		−3	19	21	190
		−4	24	25	262
		−6	16	12	94
		−11	29	23	96
1	11	0	28	27	215
		1	18	18	73
		2	19	26	21
		3	24	32	223
		7	32	25	9
		10	34	24	1
		−2	22	15	162
		−3	24	11	133
		−4	16	24	74
		−5	38	33	172
		−6	19	21	81
		−8	27	19	190
		−9	20	17	98
		−12	24	14	80
1	12	0	24	23	173
		1	24	21	256
		2	23	26	202
		4	28	23	265
		5	18	17	155
		6	18	12	220
		−1	21	19	206
		−2	18	14	262
		−6	20	22	143
1	13	0	18	17	199
		−1	18	23	72
		−2	18	13	176
		−3	18	12	96
		−5	19	8	165
1	14	0	18	16	176
		2	25	20	165
		5	19	22	167
		−3	19	19	157
1	16	0	16	11	159
2	0	0	190	244	0
		1	30	21	0
		2	156	142	0
		3	99	123	0
		4	107	102	0
		5	91	85	0
		7	142	118	0
		8	93	91	0
		10	73	71	0
		11	21	17	180
		12	27	23	180
		13	60	49	0
		16	15	0	0
		17	25	18	180
		−1	40	68	0
		−2	38	58	180
		−3	133	128	0
		−5	125	108	180
		−6	38	27	0
		−8	120	115	180
		−9	28	37	180
		−10	37	39	180
		−11	24	22	180
		−13	37	35	180
		−14	12	11	180
		−15	21	21	180
		−16	15	12	180
		−17	14	7	0
		−18	26	14	180
2	1	1	90	147	240
		3	74	68	136
		4	33	29	250
		5	102	101	103
		6	72	71	92
		7	27	22	273
		8	88	88	104
		9	52	39	84
		10	30	30	97
		11	65	61	102
		12	13	16	88
		13	37	36	99
		14	34	35	89
		16	32	31	87
		−3	82	78	198
		−4	118	126	259
		−5	48	41	188
		−6	61	62	205
		−7	93	100	260
		−8	33	29	125
		−9	51	49	280
		−10	33	35	277
		−11	28	25	149
		−12	36	33	273
		−14	25	20	69
		−15	16	13	293
		−16	15	15	84
2	2	0	23	59	322
		1	45	44	328
		2	158	233	17
		3	67	74	236
		4	42	41	334
		5	148	159	334
		6	46	45	280
		7	59	66	5
		8	34	38	357
		9	60	60	262
		10	54	54	334
		11	15	19	15
		12	13	15	6
		13	37	33	42
		14	46	36	200
		16	15	13	70
		17	23	19	173
		−1	159	210	36
		−2	61	81	214
		−3	84	88	4
		−4	46	55	344
		−5	56	47	134
		−6	26	23	88
		−7	66	55	179
		−8	18	25	175
		−9	48	41	34
		−10	96	78	177
		−11	27	30	158
		−12	34	30	81
		−13	84	61	179
		−14	19	18	95
		−15	17	12	162
		−16	47	34	176
		−17	18	14	89
		−18	16	15	158
2	3	0	13	15	152
		1	51	59	269
		2	33	37	14
		3	76	73	101
		4	102	121	308
		5	62	59	86
		6	45	44	136
		7	51	55	338
		8	73	70	103
		9	25	28	324
		11	52	49	83
		12	24	26	330
		13	35	32	70
		14	29	22	70
		15	26	26	352
		16	21	20	78
		−1	86	100	270
		−2	118	148	240
		−3	77	84	164
		−4	152	168	266
		−5	63	75	228
		−6	80	92	168
		−7	109	108	259
		−8	99	90	176
		−9	52	56	241
		−10	68	57	257
		−11	57	38	121
		−12	28	30	249
		−14	19	11	148
		−15	26	20	294
		−16	22	8	80
2	4	0	69	85	290
		1	103	122	268
		2	29	34	356
		3	80	83	267
		4	101	101	305
		5	62	52	295
		6	80	80	266
		7	79	66	324
		8	51	41	315
		9	59	64	273
		10	48	47	16
		11	16	16	255
		12	38	29	225
		13	15	26	53
		15	17	15	117
		−1	61	63	308
		−2	96	113	235
		−3	70	79	32
		−5	104	98	228
		−6	52	47	46
		−7	22	18	148
		−8	55	41	180
		−9	31	32	115
		−10	27	23	195
		−11	41	36	131
		−12	37	35	114
		−13	43	33	200
		−14	31	24	86
		−15	17	16	131
		−16	19	16	165
		−17	21	18	87
		−18	26	16	173
2	5	0	70	119	171
		1	49	66	307
		2	23	19	169
		3	15	8	106
		4	94	93	323
		5	47	45	160
		6	49	39	41
		7	77	76	337
		9	54	49	350
		10	48	38	4
		11	12	10	46
		12	51	46	351
		13	22	24	31
		14	19	16	31
		15	23	26	357
		−1	43	45	242
		−2	18	16	350
		−3	88	95	189
		−4	46	43	263
		−5	63	67	199
		−6	43	49	191
		−7	32	44	274
		−8	60	65	162
		−9	10	14	213
		−11	51	50	161
		−12	19	21	288
		−14	27	18	106
		−15	16	19	359
2	6	0	39	48	271
		1	59	76	265
		3	81	94	265
		4	46	54	257
		5	51	48	15
		6	70	92	267
		8	26	28	306
		9	31	35	280
		10	19	20	13
		11	30	29	277
		12	12	6	273
		14	20	18	276
		−1	14	26	32
		−2	74	86	276
		−3	15	17	254
		−4	10	18	33
		−5	27	39	284
		−6	58	59	78
		−7	36	34	151
		−8	33	24	165
		−9	65	60	88
		−11	38	30	96
		−12	40	33	97
		−13	20	18	219
		−14	33	24	106
		−15	30	22	84
2	7	0	54	76	170
		1	23	24	222
		2	68	61	185
		3	34	42	179
		4	40	45	4
		5	21	24	134
		7	36	33	344
		8	18	8	151
		9	43	41	14
		10	31	33	5
		11	30	23	107
		12	40	40	356
		13	20	11	31
		14	23	9	62
		15	19	25	1
		−1	37	32	279
		−2	61	57	211
		−3	83	98	166
		−4	21	27	331
		−5	65	61	160
		−6	65	58	169
		−7	18	23	3
		−8	43	38	187
		−9	16	16	183
		−11	33	33	148
		−12	19	13	317
		−13	20	10	157
		−14	18	12	151
		−15	22	18	345
2	8	0	30	40	308
		1	41	63	274

TABLE 7. (*Continued.*)

h	k	l	$\|F_o\|$	$\|F_c\|$	α°	h	k	l	$\|F_o\|$	$\|F_c\|$	α°	h	k	l	F_o	$\|F_c\|$	α°	h	k	l	$\|F_o\|$	$\|F_c\|$	α°
		2	13	7	216			−1	46	33	0			−15	14	18	358			12	19	17	338
		3	55	57	269			−3	142	113	180			−17	24	19	72			13	19	5	102
		4	33	37	249			−4	72	64	0	3	4	0	65	77	252			14	20	22	328
		6	50	51	255			−5	134	149	180			1	56	67	319			−3	92	96	178
		7	13	8	269			−8	150	134	180			2	53	57	12			−4	24	34	196
		8	13	7	212			−10	74	80	180			3	95	105	284			−5	21	17	163
		9	46	44	268			−11	66	62	180			4	81	84	359			−6	82	83	169
		10	15	10	187			−13	51	40	180			5	30	39	313			−7	13	10	239
		11	16	16	274			−15	58	15	180			6	73	75	265			−8	46	37	184
		12	19	19	296			−16	40	33	180			7	62	56	33			−9	34	30	165
		13	18	12	98			−17	23	10	0			8	69	68	304			−10	22	19	328
		−1	27	12	204			−18	14	5	0			9	28	28	295			−11	33	29	157
		−2	46	59	270	3	1	0	73	73	147			10	48	42	346			−13	23	21	10
		−3	13	12	66			1	163	238	257			11	25	31	260	3	8	0	37	59	261
		−4	28	24	101			2	44	42	203			13	23	20	0			1	8	17	196
		−5	19	16	219			3	140	162	223			−2	52	53	231			2	18	16	241
		−6	41	36	94			4	80	121	259			−3	17	10	261			3	67	79	272
		−9	47	41	93			5	59	46	131			−4	74	78	72			5	43	47	246
		−10	25	20	117			6	64	70	256			−5	59	47	195			6	65	62	269
		−11	32	28	91			7	67	46	233			−6	36	42	117			8	34	30	265
		−12	37	32	80			8	60	50	101			−7	60	51	86			9	45	40	257
		−14	20	21	89			9	24	27	287			−8	66	52	193			10	21	15	192
2	9	0	59	68	181			10	34	27	60			−9	62	56	103			11	32	33	276
		1	15	17	354			11	28	25	82			−10	30	33	133			−1	18	31	77
		3	35	43	189			13	55	48	88			−11	33	32	151			−2	15	29	258
		4	29	31	354			14	24	22	22			−12	57	50	87			−3	13	11	1
		7	32	38	17			−3	29	30	186			−13	19	25	160			−4	47	46	86
		9	31	26	332			−4	63	129	292			−14	16	11	143			−5	16	19	322
		10	16	18	315			−5	51	54	271			−15	27	23	83			−6	22	29	35
		12	23	23	354			−6	23	26	234			−16	16	15	206			−7	31	27	87
		−1	34	45	129			−7	96	89	264			−17	17	11	88			−9	41	34	71
		−2	46	48	140			−8	32	34	239	3	5	0	55	69	160			−10	32	23	34
		−3	52	49	168			−10	54	58	306			1	77	82	246			−11	18	5	350
		−5	33	31	178			−11	34	28	100			2	89	88	184			−12	30	28	82
		−6	34	32	162			−14	50	42	77			3	22	29	257			−13	17	11	354
		−7	32	33	71	3	2	0	29	51	287			4	51	53	258	3	9	0	44	53	166
		−8	38	38	151			1	49	65	1			5	59	59	177			1	21	33	50
		−11	19	25	203			2	60	54	18			7	42	37	25			2	48	51	170
		−12	19	16	50			3	66	75	278			8	51	34	107			3	23	25	148
2	10	0	26	28	206			4	78	102	353			9	23	28	329			5	35	42	194
		1	15	18	211			5	82	75	332			10	22	22	88			6	33	31	35
		2	28	27	158			6	65	61	327			11	15	10	56			7	20	9	56
		3	33	36	251			7	85	85	17			12	51	50	350			9	34	38	337
		4	18	20	222			8	32	33	262			13	18	14	117			12	31	26	344
		5	30	35	169			9	37	33	299			14	24	19	27			−3	61	62	170
		6	32	32	255			10	77	64	0			15	13	19	3			−4	18	20	93
		7	27	23	211			11	35	27	251			16	21	16	84			−5	37	29	222
		9	16	19	244			12	16	17	349			−1	80	81	205			−6	68	62	183
		−1	18	26	181			13	24	23	338			−3	89	98	155			−7	26	21	44
		−2	36	43	283			14	44	32	242			−4	32	43	222			−8	17	23	158
		−5	19	16	305			−1	130	129	21			−5	61	66	238			−9	30	24	203
		−6	19	16	123			−2	27	32	267			−6	75	76	167			−10	22	11	343
		−9	16	21	81			−3	62	53	198			−7	41	48	270			−13	23	16	12
		−12	21	19	77			−4	118	127	32			−8	19	18	156	3	10	0	25	28	282
2	11	0	23	26	176			−5	53	65	167			−9	31	36	201			1	18	17	269
		1	21	28	90			−6	62	56	142			−10	25	26	331			2	15	12	183
		4	26	30	53			−7	43	44	120			−11	29	31	117			3	29	31	265
		5	19	17	172			−8	103	89	151			−13	21	25	331			4	26	26	219
		6	16	15	321			−9	58	60	61			−14	26	23	78			5	19	16	251
		7	18	16	345			−10	35	34	127	3	6	0	54	68	255			6	35	32	268
		−3	45	41	159			−11	70	63	165			1	40	48	262			7	17	20	174
		−4	40	41	56			−12	19	22	108			2	23	17	36			9	18	20	244
		−5	19	21	126			−13	30	35	165			3	60	70	247			−3	28	30	281
		−6	43	32	162			−14	22	27	157			4	36	32	298			−4	16	23	107
		−7	20	23	57			−16	22	18	197			5	66	69	269			−6	17	18	48
2	12	0	21	18	222	3	3	0	74	113	206			6	57	56	263			−7	18	14	78
		1	27	24	252			1	79	111	273			7	29	33	321			−8	18	22	324
		2	22	24	152			2	66	90	189			8	48	52	264			−9	27	24	79
		3	26	22	252			3	40	45	239			9	19	22	302	3	11	0	36	36	177
		4	21	12	230			4	90	104	249			11	23	30	252			1	19	22	80
		6	25	16	249			5	68	72	138			14	19	20	259			2	16	20	186
		−6	20	14	104			6	52	61	269			−1	57	67	69			4	19	20	89
2	13	0	18	12	174			8	45	48	126			−3	13	18	237			5	20	28	203
		1	21	27	72			9	46	50	343			−4	41	41	100			9	21	19	32
		−1	21	23	96			10	47	44	65			−5	13	18	201			−3	26	26	162
		−2	19	17	82			11	29	28	84			−6	24	27	70			−4	19	22	95
		−3	21	11	176			12	34	32	33			−7	49	45	67			−5	20	26	106
		−4	21	22	95			13	30	31	87			−8	19	15	162			−6	18	10	173
2	14	2	25	19	165			14	20	17	359			−9	51	46	89	3	12	2	16	20	186
		−1	19	21	184			16	23	18	98			−10	35	32	88			3	18	15	278
3	0	0	51	54	180			−1	33	48	208			−12	43	39	84			4	19	22	181
		1	47	73	0			−3	59	75	203			−14	27	19	128			−4	19	22	105
		2	187	212	0			−4	41	55	233			−15	38	31	87	3	13	0	16	18	126
		3	47	40	0			−5	48	54	251	3	7	0	52	61	181	3	14	2	22	18	165
		4	95	87	0			−6	57	48	187			1	15	12	177	4	0	0	77	112	180
		5	49	56	0			−7	94	81	295			2	59	66	166			1	84	126	0
		6	22	11	0			−8	48	44	226			3	37	40	150			2	59	51	180
		7	187	191	0			−9	34	31	157			4	17	30	21			3	16	17	0
		9	35	26	0			−10	67	64	293			5	54	70	150			4	85	82	0
		10	77	68	0			−11	21	17	175			7	19	13	233			5	82	66	180
		12	29	31	0			−12	19	11	94			8	41	40	157			6	48	43	0
		13	19	21	0			−13	18	16	312			9	45	49	358			7	93	79	0
		15	19	23	0			−14	26	22	115			11	26	13	97			8	43	39	180

TABLE 7. (*Continued.*)

h	k	l	$\|F_o\|$	$\|F_c\|$	α°
		9	79	68	0
		10	54	45	0
		11	16	14	0
		12	79	62	0
		13	17	10	0
		−1	42	52	180
		−2	55	38	180
		−3	90	89	180
		−4	51	38	0
		−5	94	91	180
		−6	100	93	180
		−7	46	33	0
		−8	107	103	180
		−9	67	65	180
		−11	96	86	180
		−13	30	13	0
		−14	60	45	180
		−15	18	10	0
		−17	21	11	180
4	1	0	49	69	257
		1	84	119	261
		2	63	72	148
		3	94	95	239
		4	59	59	303
		5	32	27	104
		6	63	72	298
		7	38	39	137
		8	30	19	160
		9	26	31	289
		10	37	34	80
		11	23	10	169
		12	22	16	111
		13	52	43	68
		14	21	15	2
		15	24	19	88
		16	19	22	98
		−1	71	77	168
		−2	140	164	257
		−3	100	128	252
		−4	21	13	278
		−5	85	94	264
		−6	52	48	123
		−7	12	20	271
		−8	47	47	235
		−9	78	71	115
		−10	32	25	331
		−11	27	21	77
		−12	37	37	100
		−14	33	31	93
		−17	20	23	100
4	2	0	86	94	209
		1	65	87	34
		2	31	43	239
		3	77	78	224
		4	57	65	5
		5	59	56	275
		6	62	58	345
		7	58	53	330
		8	40	43	269
		9	64	64	348
		10	48	42	349
		11	35	32	232
		12	30	32	10
		15	30	28	8
		−1	52	56	18
		−2	24	26	32
		−3	122	136	207
		−4	56	57	32
		−5	68	75	146
		−6	80	80	196
		−7	24	28	153
		−8	82	73	197
		−9	26	33	191
		−10	30	32	89
		−11	54	54	187
		−12	36	32	130
		−13	32	31	181
		−14	34	27	159
		−15	34	25	53
		−16	17	15	191
		−17	10	9	175
		−18	23	13	16
4	3	0	76	96	264
		1	71	99	249
		2	29	49	180
		3	55	64	295
		4	48	45	244
		5	19	31	117
		6	65	80	262
		7	26	27	174
		8	22	20	338
		9	47	45	311
		10	32	33	109
4	4	0	65	61	207
		1	41	62	10
		2	35	44	357
		3	31	40	305
		4	91	98	6
		5	54	61	269
		6	47	42	304
		7	77	71	11
		8	76	71	268
		9	52	47	2
		10	43	35	332
		11	40	34	267
		12	17	21	4
		13	18	20	301
		−1	66	66	111
		−2	43	49	112
		−3	42	40	206
		−4	37	44	66
		−5	52	59	139
		−6	62	59	197
		−7	62	67	75
		−8	66	60	182
		−9	43	39	116
		−10	51	57	93
		−11	54	47	201
		−12	18	25	103
		−14	23	22	186
		−15	19	19	85
4	5	0	26	31	213
		1	32	32	258
		2	111	123	158
		3	51	62	285
		4	47	35	209
		5	54	51	194
		6	41	30	359
		7	12	17	217
		9	18	21	318
		10	24	17	94
		11	22	20	337
		12	35	35	345
		13	38	27	87
		14	32	27	4
		15	24	16	51
		16	23	12	62
		17	27	21	347
		−1	93	106	166
		−2	98	107	272
		−3	66	69	195
		−4	21	37	197
		−5	45	45	275
		−6	39	41	147
		−7	15	16	301
		−10	27	32	335
		−12	17	13	80
		−13	23	29	322
		−14	13	14	53
		−17	21	15	92
4	6	0	59	64	269
		3	34	47	268
		4	31	31	20
		5	37	41	262
		6	49	51	276
		7	40	35	355
		8	39	49	252
		10	28	26	231
		11	54	53	274
		12	17	16	25
		13	22	20	253
		−1	12	13	155
		−2	44	55	55
		−3	22	33	223
		−4	82	90	116
		−5	26	18	162
		−6	35	43	143
		−7	87	83	75
		−8	19	20	140
		−9	34	35	127
		−10	39	43	97
		−11	19	21	146
		−12	45	37	80
		−15	38	29	85
4	7	0	46	49	203
		1	26	23	222
		2	77	95	167
		3	14	15	286
		4	18	29	163
		5	53	56	193
		6	21	24	323
		7	35	48	173
		8	18	20	224
		9	24	22	345
		10	17	20	177
		11	22	14	59
		12	19	15	346
		−1	65	73	153
		−2	16	16	145
		−3	64	76	149
		−4	36	45	188
		−5	12	9	148
		−6	63	61	157
		−7	18	10	101
		−8	21	25	353
		−9	40	41	172
		−10	30	23	18
		−12	18	7	176
		−13	34	27	3
4	8	0	20	31	283
		1	24	44	90
		2	22	28	272
		3	25	21	450
		4	19	26	153
		5	36	41	266
		6	17	15	245
		7	16	13	229
		8	57	47	264
		9	15	7	236
		10	30	22	270
		11	29	28	255
		−1	20	25	133
		−2	34	41	99
		−3	12	12	265
		−4	58	64	82
		−5	30	27	88
		−7	59	60	96
		−8	26	22	117
		−9	37	31	88
		−10	39	36	69
		−12	24	21	78
		−13	17	18	76
4	9	0	27	28	126
		1	17	18	131
		2	52	52	175
		4	21	19	182
		5	29	27	164
		7	26	28	170
		9	18	13	345
		−1	63	65	183
		−3	37	34	179
		−4	38	48	160
		−6	40	44	202
		−8	24	21	44
		−10	18	21	340
		−13	20	21	3
4	10	0	25	29	292
		1	16	19	94
		2	16	14	179
		3	30	39	254
		4	17	12	135
		5	32	33	264
		6	24	20	216
		8	29	27	268
		9	19	13	186
		−2	23	20	86
		−4	30	27	105
		−5	17	18	25
		−6	21	24	18
		−7	46	41	81
		−10	26	27	73
4	11	0	18	16	110
		1	18	26	106
		2	24	21	196
		3	38	40	69
		4	19	16	106
		5	21	19	187
		−1	23	28	171
		−2	19	19	88
		−3	22	30	164
		−9	20	21	207
		−10	19	12	17
4	12	0	14	17	294
		1	18	26	161
		4	19	18	176
		5	19	16	224
		6	19	13	219
		7	20	20	186
		−2	15	8	104
		−3	18	23	345
		−4	22	19	99
		−5	25	20	30
		−6	19	15	5
		−8	20	22	354
		−10	21	12	65
4	13	0	19	17	113
		1	20	23	73
		2	19	11	159
		3	26	21	98
		−1	19	10	119
		−2	28	27	76
		−3	19	10	72
4	14	0	21	10	353
		−5	21	13	335
4	15	−2	25	19	91
5	0	0	97	112	180
		1	30	39	0
		2	84	82	180
		3	94	91	0
		4	118	103	0
		5	63	68	180
		6	79	80	0
		7	75	61	0
		8	37	29	0
		9	90	85	0
		11	45	52	0
		12	29	29	0
		13	28	20	180
		15	19	24	0
		−1	83	98	180
		−2	23	32	180
		−3	58	79	180
		−5	18	1	0
		−6	129	133	180
		−7	26	35	180
		−8	58	55	180
		−9	41	43	180
		−10	30	14	0
		−11	70	51	180
		−12	30	26	180
		−13	16	13	0
		−14	35	31	180
5	1	0	86	127	281
		1	40	60	240
		2	54	67	229
		3	96	122	279
		4	43	40	276
		5	18	31	259
		6	55	60	245
		7	35	37	179
		8	37	39	296
		9	24	30	268
		10	37	38	119
		11	13	22	258
		12	22	10	76
		13	20	13	66
		15	21	24	87
		16	21	5	139
		−1	27	19	35
		−2	52	49	280
		−3	67	77	241
		−4	48	41	93
		−5	65	56	290
		−6	12	10	359
		−7	24	33	119
		−8	45	45	296
		−9	33	33	117
		−11	35	28	330
		−12	39	39	94
		−13	20	20	94
		−14	23	21	109
		−15	40	36	93
5	2	0	40	48	150
		1	42	46	342
		2	51	68	213
		4	49	64	11
		5	17	27	237
		6	107	115	358
		7	48	38	329
		8	19	28	306
		9	87	86	3
		10	24	23	313
		11	45	39	266
		12	48	47	351
		13	30	23	321
		14	21	26	359

TABLE 7. (*Continued.*)

h	k	l	$\|F_o\|$	$\|F_c\|$	α°
		−2	43	63	45
		−3	134	160	195
		−4	56	54	136
		−5	77	86	117
		−6	94	100	173
		−7	38	29	84
		−8	36	37	163
		−9	88	82	178
		−10	26	23	95
		−11	55	51	170
		−12	21	24	209
		−14	38	36	198
		−15	21	12	4
		−17	24	18	224
		−18	23	16	24
5	3	0	91	110	247
		1	42	49	225
		2	43	62	221
		3	103	124	267
		4	70	77	204
		5	59	70	226
		6	80	85	263
		7	43	40	154
		8	21	29	238
		9	14	16	119
		11	55	47	266
		12	37	35	130
		13	20	13	82
		15	21	12	102
		−1	71	88	201
		−2	68	79	247
		−3	36	54	240
		−4	65	76	121
		−5	40	49	228
		−6	16	15	199
		−7	16	24	117
		−8	44	44	284
		−9	45	40	93
		−10	19	16	51
		−11	30	26	36
		−12	41	39	101
		−13	37	28	340
		−14	23	28	32
		−15	29	31	74
5	4	0	50	52	219
		1	73	112	35
		2	53	70	201
		3	20	31	23
		4	31	35	17
		5	63	77	247
		6	35	35	320
		7	38	35	302
		8	35	43	266
		9	29	29	350
		10	61	59	296
		11	44	37	321
		12	34	33	351
		13	29	30	254
		14	18	18	312
		15	23	16	333
		16	13	16	255
		17	18	16	22
		−1	72	73	153
		−2	116	130	78
		−3	18	43	152
		−4	38	44	84
		−5	66	84	95
		−6	70	72	163
		−7	94	101	91
		−8	36	40	129
		−9	43	44	152
		−10	52	57	78
		−11	22	24	149
		−12	20	20	127
		−13	32	27	32
		−15	23	18	66
5	5	0	27	25	264
		1	80	95	190
		2	83	85	184
		3	27	30	252
		4	70	72	171
		5	21	15	212
		6	33	31	278
		7	73	72	155
		8	31	29	244
		9	16	14	213
		10	24	28	132
		11	23	29	326
		−1	48	77	179
		−2	33	40	206
		−3	18	33	229
		−4	56	67	148
		−5	33	44	302
		−6	31	28	163
		−7	48	46	130
		−8	33	35	351
		−9	22	19	107
		−10	17	6	53
		−11	23	35	347
		−12	24	22	88
		−13	22	27	15
		−14	19	26	0
		−15	22	12	92
5	6	1	19	38	82
		2	30	48	235
		3	18	12	80
		5	62	76	256
		7	51	35	252
		8	48	53	267
		9	29	11	8
		10	29	42	259
		11	28	29	251
		12	21	14	53
		13	22	28	266
		−1	14	8	48
		−2	55	73	82
		−3	18	30	275
		−4	48	63	73
		−5	45	56	105
		−6	52	47	212
		−7	57	61	96
		−8	48	49	74
		−10	41	43	94
		−11	23	21	203
		−12	28	23	85
		−13	33	29	90
		−14	19	11	228
5	7	0	23	31	310
		1	37	53	193
		2	71	80	172
		3	16	13	309
		4	45	53	184
		5	37	37	157
		7	42	40	178
		9	19	1	71
		−1	49	54	176
		−2	13	19	161
		−4	43	56	172
		−5	20	27	345
		−6	15	4	258
		−7	21	16	140
		−8	34	37	4
		−9	16	11	177
		−11	24	21	7
		−12	19	11	149
		−13	24	23	358
		−14	25	21	354
5	8	0	11	19	357
		1	33	48	96
		2	27	38	273
		4	12	9	53
		5	30	30	273
		6	24	21	234
		7	32	26	291
		8	26	30	266
		10	21	21	267
		11	20	18	266
		12	22	7	274
		13	27	30	261
		−1	13	8	142
		−2	38	37	94
		−4	32	34	78
		−5	42	45	62
		−6	35	34	334
		−8	16	20	125
		−9	18	19	85
		−10	45	36	73
		−13	21	17	88
5	9	0	31	40	113
		1	34	45	164
		2	34	38	164
		3	18	14	133
		4	37	46	157
		5	29	26	177
		7	30	35	163
		−1	32	32	159
		−4	18	16	189
		−5	40	41	30
		−8	19	23	354
		−11	23	22	349
		−12	20	11	274
5	10	0	19	20	300
		1	23	30	109
		2	19	17	302
		5	25	31	282
		−2	34	37	57
		−3	19	24	316
		−4	19	18	76
		−5	27	26	61
		−6	20	22	354
		−7	35	38	54
		−8	21	24	72
5	11	0	19	19	91
		1	20	25	166
		2	21	15	129
		3	23	26	80
		4	21	23	172
		5	21	16	140
		6	21	18	62
		7	20	12	182
		−1	20	24	158
		−2	31	28	85
		−3	27	34	65
		−4	20	26	186
		−5	21	17	75
		−7	21	2	261
		−8	24	21	19
5	13	0	15	20	88
5	15	0	15	16	78
6	0	0	65	69	180
		1	36	44	0
		2	13	49	180
		3	29	42	0
		4	103	98	180
		5	88	79	180
		6	46	51	0
		7	29	36	180
		8	21	26	180
		9	33	33	0
		10	13	23	180
		11	42	41	0
		12	16	20	0
		14	27	35	0
		17	21	21	0
		−1	117	150	180
		−2	15	35	180
		−3	16	30	180
		−4	113	121	180
		−6	59	79	180
		−7	26	36	180
		−8	22	17	180
		−9	102	97	180
		−10	38	27	0
		−11	14	0	180
		−13	62	49	0
		−14	33	23	180
		−16	21	16	0
6	1	0	83	112	272
		1	26	31	149
		2	67	76	243
		3	56	75	293
		4	18	5	270
		5	94	109	261
		6	73	64	237
		7	26	24	246
		8	65	66	268
		9	24	17	188
		10	17	23	274
		11	38	39	293
		12	15	9	123
		15	18	19	105
		−1	20	11	135
		−3	101	117	304
		−4	65	73	73
		−5	47	35	200
		−6	12	22	344
		−7	65	60	80
		−9	35	36	76
		−10	63	58	93
		−11	18	22	32
		−13	35	38	65
		−14	18	5	271
		−15	10	24	116
6	2	0	18	18	103
		2	42	53	173
		3	51	54	46
		4	35	30	321
		5	47	56	223
		6	67	72	13
		7	19	24	208
		8	17	16	326
		9	54	51	353
		10	34	36	238
		11	36	36	11
		12	34	25	3
		13	17	14	281
		14	34	36	8
		15	18	13	306
		16	16	9	326
		−1	45	66	184
		−2	77	93	57
		−3	78	92	218
		−4	96	110	190
		−5	49	44	116
		−6	105	105	182
		−7	29	47	149
		−8	32	26	24
		−9	68	61	179
		−10	12	5	68
		−11	15	17	147
		−12	41	29	160
		−13	18	18	21
		−15	23	11	264
		−16	17	18	4
		−17	17	9	232
6	3	0	66	85	281
		1	21	34	154
		2	80	93	248
		3	75	78	243
		4	78	86	184
		5	71	76	274
		6	25	17	204
		7	66	66	212
		8	43	50	285
		9	16	22	240
		10	17	17	241
		12	22	18	94
		15	19	18	104
		−1	29	39	240
		−2	43	55	211
		−3	61	68	261
		−4	19	17	4
		−5	13	13	10
		−6	41	42	265
		−7	33	32	122
		−8	61	63	5
		−9	15	21	40
		−10	52	48	111
		−11	52	40	324
		−12	48	38	82
		−13	21	26	70
		−15	16	28	76
6	4	0	19	29	94
		1	46	59	119
		2	48	58	200
		3	59	69	79
		4	15	27	326
		5	10	6	253
		6	40	44	27
		7	12	25	229
		9	60	56	10
		10	46	35	300
		11	37	35	336
		12	15	9	276
		13	15	22	278
		−1	87	90	196
		−2	96	102	99
		−3	54	61	113
		−4	43	57	165
		−5	62	82	64
		−6	55	58	184
		−7	43	42	113
		−8	46	49	113
		−9	30	22	201
		−10	48	42	79
		−11	22	22	106
		−12	27	25	198
		−13	25	21	70
6	5	0	39	50	276
		1	58	68	166
		2	46	44	199
		3	18	33	205
		4	64	67	182
		5	75	68	246
		6	45	44	187
		7	53	52	188
		8	34	28	254
		9	43	40	154
		10	14	4	138
		12	22	28	175
		−2	18	32	208
		−3	37	48	341
		−4	29	34	159
		−5	26	14	326
		−6	69	72	332
		−7	13	21	105
		−9	23	24	355
		−10	50	48	60
		−11	41	45	359
		−12	18	17	48
		−13	16	14	71
		−14	20	28	7

TABLE 7. (*Continued.*)

h	k	l	$\|F_o\|$	$\|F_c\|$	α°
		−15	18	15	94
6	6	0	24	42	85
		1	16	24	113
		2	20	28	204
		3	31	44	85
		4	15	14	152
		5	22	24	182
		6	27	36	82
		7	46	48	286
		8	14	15	6
		9	33	9	101
		10	31	30	244
		13	19	28	261
		−1	13	20	163
		−2	63	79	93
		−3	29	42	138
		−4	30	35	135
		−5	63	78	81
		−6	15	26	142
		−7	22	25	100
		−8	57	63	91
		−10	29	20	81
		−12	19	18	252
6	7	0	14	23	312
		1	62	85	181
		2	16	12	140
		3	16	15	235
		4	56	64	174
		6	30	29	194
		7	48	48	157
		8	15	12	219
		9	21	23	188
		10	18	21	188
		12	19	19	139
		−1	23	26	152
		−2	45	48	161
		−3	12	21	294
		−4	34	38	170
		−6	14	17	346
		−7	16	13	168
		−8	45	44	350
		−9	13	17	342
		−10	16	14	188
		−11	38	41	353
6	8	0	25	25	84
		1	16	22	69
		2	22	31	262
		3	12	15	48
		5	25	32	272
		6	13	18	80
		7	26	32	248
		8	18	19	253
		10	36	39	276
		11	20	9	240
		12	24	22	277
		13	19	20	274
		−1	16	11	50
		−2	43	50	81
		−4	15	16	22
		−5	73	76	96
		−6	14	12	23
		−7	23	28	69
		−10	21	24	64
		−11	18	9	12
		−12	20	13	275
6	9	0	23	29	65
		1	39	52	188
		2	17	13	173
		3	29	29	122
		4	47	46	175
		6	19	28	174
		7	30	26	181
		9	18	23	175
		−1	43	47	177
		−2	28	27	187
		−4	34	45	193
		−7	16	20	176
		−8	15	14	337
		−11	25	27	5
		−14	18	16	6
6	10	0	9	16	72
		1	25	23	47
		2	16	17	356
		5	15	20	283
		8	16	11	254
		10	20	24	264
		−2	40	43	73
		−3	30	37	51
		−4	20	14	36
		−5	35	38	90
		−6	15	17	355
		−7	17	19	65
		−8	32	29	86
		−9	18	16	331
		−10	15	14	82
		−11	17	10	41
6	11	0	14	16	73
		1	19	21	180
		2	16	19	78
		3	15	19	111
		4	29	31	177
		5	18	20	81
		6	16	20	131
		−1	19	15	175
		−2	19	19	169
		−3	17	20	50
		−7	15	16	252
		−11	18	18	353
6	12	0	18	14	28
		1	15	11	83
		−1	22	25	4
		−2	20	17	88
		−4	15	19	355
		−6	16	19	355
6	13	0	15	19	69
7	0	0	26	27	180
		1	81	97	180
		2	106	115	180
		3	21	22	180
		4	47	56	180
		5	40	40	0
		6	14	14	0
		7	20	25	180
		8	40	30	0
		9	22	19	0
		11	63	61	0
		14	35	32	0
		−1	129	167	180
		−2	56	75	180
		−4	91	92	180
		−5	21	17	0
		−6	36	47	180
		−7	54	63	180
		−9	32	33	180
		−10	37	42	180
		−12	50	38	180
		−13	12	11	0
		−15	38	20	180
7	1	0	70	79	236
		1	24	39	148
		2	74	90	288
		3	42	43	264
		4	27	35	244
		5	91	89	262
		7	32	40	275
		8	61	57	278
		10	34	44	248
		−1	76	85	277
		−2	37	38	98
		−3	50	54	311
		−4	29	33	339
		−5	41	51	74
		−6	45	40	249
		−7	63	71	87
		−8	31	39	78
		−10	71	69	96
		−13	55	49	94
7	2	0	22	31	63
		1	49	78	183
		2	33	44	187
		3	23	30	39
		4	77	96	186
		6	41	43	19
		7	40	49	236
		8	29	39	14
		9	24	23	329
		12	30	25	279
		14	21	23	338
		−1	94	115	184
		−2	21	35	151
		−3	39	47	101
		−4	71	82	178
		−5	38	49	113
		−6	40	44	171
		−7	44	45	209
		−8	52	44	41
		−9	44	39	188
		−10	19	21	168
		−11	20	17	3
		−12	35	31	227
		−15	18	13	235
		−16	22	20	353
7	3	0	14	17	334
		1	39	62	187
		2	40	56	270
		3	29	45	186
		5	37	53	272
		6	18	30	184
		7	29	33	249
		8	35	37	260
		10	29	32	276
		11	21	22	271
		12	19	23	151
		−2	73	90	138
		−3	13	14	271
		−4	47	45	86
		−5	55	60	93
		−6	38	51	351
		−7	29	41	94
		−8	37	34	60
		−9	33	47	24
		−10	58	60	80
		−13	21	20	89
		−14	24	23	3
7	4	0	46	72	92
		1	50	63	118
		2	57	70	173
		3	31	41	55
		4	18	28	268
		5	13	22	95
		6	23	28	116
		7	27	40	236
		10	45	42	259
		11	18	22	21
		−1	49	65	175
		−2	46	54	103
		−3	41	62	111
		−4	48	64	186
		−5	37	51	101
		−7	18	29	168
		−8	49	45	82
		−9	9	18	189
		−10	23	25	239
		−11	40	32	50
		−13	17	16	355
		−16	22	18	26
7	5	0	12	24	192
		1	55	62	156
		2	45	38	254
		3	51	46	186
		4	71	61	185
		5	56	37	278
		6	41	37	193
		7	59	42	205
		8	45	34	221
		9	44	40	160
		11	31	23	215
		−1	46	44	283
		−2	59	58	162
		−4	34	22	85
		−5	53	41	103
		−6	39	44	345
		−7	54	44	90
		−8	26	34	2
		−9	9	30	355
		−10	24	18	120
		−11	34	37	6
		−14	11	21	352
7	6	0	60	77	90
		1	21	19	150
		2	18	21	98
		3	55	76	71
		6	15	12	16
		7	29	37	259
		8	19	1	183
		−1	18	23	120
		−2	59	70	90
		−3	36	52	117
		−4	26	33	127
		−5	36	49	110
		−6	21	25	101
		−8	28	37	101
		−10	27	14	89
		−11	31	26	69
7	7	1	35	45	174
		3	15	21	177
		4	49	47	181
		5	16	7	187
		6	35	47	149
		7	16	7	200
		8	16	9	286
		9	42	49	182
		10	18	13	278
		12	17	17	156
		−2	48	56	143
		−3	26	40	341
		−6	44	61	334
		−7	19	20	89
		−8	25	21	34
		−9	43	43	338
		−11	43	31	343
		−12	27	27	354
		−14	20	20	346
7	8	0	46	60	75
		2	14	7	169
		3	40	52	81
		5	13	24	89
		7	20	23	279
		8	23	19	64
		10	23	22	271
		−1	19	16	97
		−2	43	43	90
		−3	12	22	75
		−4	21	25	305
		−5	37	42	84
		−6	13	19	70
		−8	26	34	87
		−9	15	7	5
		−11	19	17	91
		−12	20	15	275
7	9	1	19	35	192
		2	21	29	50
		−1	23	33	26
		−2	16	19	193
7	10	0	30	38	91
		−2	17	19	88
7	11	0	14	11	64
		2	18	13	110
		−2	18	19	205
7	12	0	15	13	87
		−1	21	25	347
		−2	18	20	35
8	0	0	11	5	180
		1	77	89	180
		3	18	27	180
		4	46	53	180
		7	40	43	180
		9	22	20	180
		11	29	29	0
		−1	26	31	180
		−2	76	77	180
		−3	39	33	0
		−4	113	117	180
		−5	31	46	180
		−6	19	19	0
		−7	66	69	180
		−10	16	20	0
		−11	30	31	0
		−14	39	32	0
8	1	0	15	26	131
		1	38	49	31
		2	69	77	260
		4	40	45	251
		5	48	53	268
		7	62	71	277
		8	24	24	263
		9	34	30	212
		10	40	50	263
		−1	27	33	257
		−2	55	60	112
		−3	51	46	55
		−4	24	26	79
		−5	65	71	120
		−7	21	32	75
		−8	75	70	79
		−9	16	12	313
		−10	29	33	93
		−11	27	38	97
		−13	51	45	61
8	2	0	38	50	94
		1	46	74	167
		2	15	22	111
		3	26	37	146
		4	52	72	186
		5	12	22	82
		6	18	27	211
		7	12	28	185
		8	15	21	12
		10	19	16	300
		11	26	26	323
		−1	34	40	162
		−2	80	98	161
		−4	50	68	149
		−5	25	38	147
		−6	23	29	64
		−7	52	64	195
		−8	28	23	356
		−9	12	16	54
		−10	34	35	221
		−11	57	48	36
		−12	23	23	343
		−13	9	21	328

TABLE 7. (*Continued.*)

h	k	l	$\|F_o\|$	$\|F_c\|$	α°	h	k	l	$\|F_o\|$	$\|F_c\|$	α°	h	k	l	$\|F_o\|$	$\|F_c\|$	α°	h	k	l	$\|F_o\|$	$\|F_c\|$	α°
		−14	29	28	355			−1	30	48	0			−1	30	42	9			3	89	101	180
8	3	0	33	47	115			−2	39	31	150			−3	60	72	79			4	29	30	180
		2	35	46	294			−4	21	36	351			−4	33	47	6			5	27	34	180
		3	23	39	141			−6	32	37	4			−5	55	65	61			6	51	59	180
		4	21	34	245			−7	27	41	333			−6	18	32	42			9	53	47	180
		5	36	40	268			−8	16	18	68			−7	16	27	21			−1	32	29	0
		6	19	33	170			−9	29	32	355			−8	53	57	80			−2	33	38	180
		7	42	59	243			−11	19	10	18			−9	16	15	352			−3	12	4	180
		8	30	28	214			−12	21	28	345			−11	23	23	95			−5	54	52	180
		9	13	21	194	8	8	0	16	23	68			−12	19	20	4			−6	16	25	0
		10	33	37	270			2	20	27	79	9	4	1	30	50	159			−8	53	39	180
		11	29	34	187			3	22	31	76			2	37	52	82			−9	38	44	0
		13	21	25	272			5	15	19	112			3	41	55	184			−12	48	43	0
		−1	45	60	332			8	16	23	99			4	19	25	130	10	1	0	45	60	76
		−2	51	70	98			9	19	19	272			5	40	39	88			1	32	34	348
		−3	10	17	204			−1	23	30	46			6	32	39	176			2	26	40	110
		−4	40	59	4			−2	16	13	71			8	27	34	121			4	32	28	292
		−5	60	71	94			−3	23	38	78			9	23	29	187			5	23	31	102
		−6	27	32	359			−4	16	19	323			−1	51	68	101			7	27	25	287
		−7	16	31	58			−6	28	38	75			−2	19	27	194			9	27	33	278
		−8	50	59	63			−7	15	11	318			−3	20	27	53			11	19	9	235
		−9	30	35	42			−8	17	14	112			−5	37	41	198			−1	21	26	68
		−10	47	44	70			−9	15	6	135			−6	39	42	70			−3	74	84	97
		−11	43	35	33			−10	26	22	287			−7	24	29	255			−4	20	25	49
		−13	29	29	92			−12	18	12	264			−9	48	41	23			−5	31	38	56
8	4	0	77	89	89	8	9	0	21	22	166			−10	10	20	230			−6	49	60	96
		1	46	65	180	8	10	0	29	30	49			−12	18	19	349			−8	41	46	81
		2	43	60	124			2	18	20	68			−13	27	25	256			−9	26	35	42
		3	49	55	86	8	11	2	17	17	81	9	5	0	15	25	120			−10	26	28	6
		4	39	43	151	8	12	0	15	14	72			1	10	22	338			−11	35	33	88
		5	60	70	85	9	0	0	26	35	180			4	15	24	303	10	2	0	43	55	172
		8	32	36	60			1	60	90	180			5	22	25	188			1	22	42	140
		10	16	16	23			3	43	58	180			7	26	23	257			2	36	58	86
		11	25	27	27			4	15	30	180			8	34	37	166			3	43	55	171
		−1	39	54	118			6	65	69	180			9	19	12	243			4	16	39	121
		−3	53	65	67			8	12	18	0			10	19	17	207			5	13	29	156
		−4	30	43	192			9	32	37	180			11	30	28	161			6	52	62	179
		−5	31	42	136			−2	57	76	180			−1	45	50	2			7	18	28	74
		−6	35	49	74			−4	21	36	180			−2	22	16	121			8	17	26	199
		−7	23	28	217			−5	24	34	180			−3	40	40	91			10	16	19	10
		−8	23	24	47			−6	50	52	0			−4	41	48	343			−1	43	48	74
		−9	25	20	60			−7	21	23	180			−5	21	30	68			−2	35	43	175
		−10	25	27	224			−9	42	43	0			−6	21	20	57			−3	12	27	138
		−11	18	17	316			−12	37	30	0			−7	26	36	349			−4	21	31	48
		−15	28	19	238			−13	38	22	180			−8	23	21	85			−5	36	38	217
8	5	0	14	8	305	9	1	0	21	38	117			−9	31	28	23			−6	21	23	343
		1	27	26	218			1	23	34	324			−12	25	22	331			−7	23	28	340
		2	55	57	285			2	22	31	322	9	6	0	40	46	102			−8	32	31	235
		3	57	58	142			3	13	20	113			2	25	38	89			−9	39	40	5
		4	10	21	253			4	40	58	273			4	27	23	114			−10	34	31	254
		5	55	32	248			7	42	52	285			5	38	49	92			−12	43	38	6
		6	60	53	182			8	26	20	233			−1	44	58	92	10	3	0	49	63	104
		7	30	24	250			9	25	24	256			−2	35	39	199			1	13	23	290
		8	44	32	195			10	37	35	285			−3	33	41	110			2	18	25	43
		9	35	35	174			12	19	31	273			−6	33	34	63			3	8	15	91
		10	35	26	256			−1	27	32	319			−8	25	26	259			5	33	33	106
		11	29	26	178			−2	22	25	72			−9	30	12	67			7	20	20	279
		12	26	20	174			−3	49	59	64			−10	33	34	274			−1	16	22	71
		14	15	18	170			−4	45	39	323	9	7	0	16	25	158			−2	14	30	13
		−1	57	56	312			−5	38	46	83			1	15	26	353			−3	33	51	89
		−2	23	16	197			−6	25	39	93			2	15	5	290			−4	25	36	13
		−3	48	43	352			−8	52	60	78			3	18	23	175			−5	21	33	44
		−4	41	42	343			−9	29	21	3			4	22	25	303			−7	23	34	6
		−5	33	24	67			−11	32	33	104			6	16	22	159			−8	35	41	68
		−6	53	50	340			−12	27	17	232			8	27	35	178			−9	26	28	86
		−7	29	28	35	9	2	0	12	30	153			−1	16	27	334			−10	23	22	333
		−8	32	27	114			1	56	83	185			−2	15	17	58	10	4	0	22	32	143
		−9	57	59	343			3	41	59	168			−3	15	17	273			1	21	34	120
		−10	15	21	63			4	47	52	174			−4	37	53	357			2	26	44	112
		−11	12	17	70			5	24	32	60			−6	18	28	342			3	36	46	198
		−12	18	27	356			6	32	48	180			−7	30	40	7			4	33	47	100
		−13	13	22	100			7	9	14	111	9	8	0	26	36	96			5	23	22	99
8	6	0	26	37	96			8	22	28	118			1	28	13	77			6	21	21	194
		2	25	41	84			9	11	26	182			2	58	68	101			7	10	26	70
		3	18	26	99			−1	27	31	171			5	26	35	80			8	18	21	124
		4	28	24	217			−2	77	95	186			6	15	8	13			9	15	17	173
		5	36	50	107			−3	21	28	94			7	16	20	78			−1	33	42	86
		6	24	23	87			−4	18	23	195			−1	26	33	97			−2	16	18	236
		−1	19	33	80			−5	39	43	192			−2	20	22	84			−4	19	25	21
		−3	42	64	82			−6	48	45	53			−3	21	33	94			−5	32	40	221
		−5	24	32	77			−9	21	19	8			−4	13	13	37			−6	24	24	28
		−6	30	36	90			−10	33	26	230			−6	15	13	96			−9	27	27	358
		−7	27	27	230			−11	25	22	29			−7	15	4	329			−10	10	19	311
		−8	29	30	99			−12	20	25	358			−8	15	3	317			−12	18	15	1
		−10	24	17	265			−13	15	13	257			−9	16	11	64	10	5	0	19	21	58
8	7	0	15	19	195			−14	27	28	355			−10	22	28	258			1	35	36	345
		1	18	18	184	9	3	0	42	61	94	9	9	−1	23	22	347			2	27	25	65
		2	20	29	359			3	24	33	160	9	10	0	21	25	68			3	36	28	104
		3	46	61	185			5	30	36	195			2	20	29	83			4	21	22	296
		4	30	30	210			6	16	21	162			−1	17	22	73			8	28	28	182
		6	16	21	157			7	42	47	267	9	13	0	18	16	248			−1	18	17	23
		8	28	32	177			8	13	19	172	10	0	0	19	42	180			−2	26	37	6
		9	24	25	181			10	24	26	257			1	32	48	180			−3	31	25	79

TABLE 7. (*Continued.*)

h	k	l	|Fo|	|Fc|	α°	h	k	l	|Fo|	|Fc|	α°	h	k	l	|Fo|	|Fc|	α°	h	k		|Fo|	|Fc|	α°
		−4	30	29	30			1	18	27	335			−5	18	12	291	13	2	0	12	18	17
		−5	29	33	6			2	32	46	86			−6	18	27	73			1	9	15	258
		−6	27	26	84			3	32	39	10			−7	27	23	96			2	21	28	201
		−7	41	42	9			−1	27	43	81	12	2	0	16	21	169			3	16	24	36
		−9	24	21	61			−2	29	37	340			1	36	41	55			5	25	29	187
		−10	27	26	355			−3	33	40	80			2	30	40	186			8	14	18	188
10	6	2	29	49	94			−4	34	47	47			3	18	17	169			−2	30	36	33
		4	31	45	77			−5	32	37	351			4	11	16	90			−3	15	15	293
		5	31	38	87			−6	29	36	106			5	29	45	178			−4	21	31	310
		7	27	35	84			−9	23	25	110			8	23	30	177			−5	33	44	9
		−1	48	62	68			−10	24	20	288			−2	19	25	333			−6	15	26	260
		−2	30	37	207	11	4	0	16	25	225			−3	24	30	250			−7	14	23	2
		−4	23	25	60			3	39	51	213			−4	21	30	12			−8	15	24	328
		−6	27	20	20			4	27	36	77			−6	15	16	237			−10	30	28	14
10	7	1	21	36	352			5	24	30	142			−7	32	40	338	13	3	0	19	29	321
		4	16	18	334			6	21	25	163			−8	18	22	287			2	18	22	84
		8	19	27	158			7	19	24	83			−10	12	25	6			3	19	28	344
		−1	24	26	17			8	30	35	182	12	3	0	20	32	16			7	27	25	78
		−2	27	40	354			9	27	24	140			1	30	31	29			−1	19	29	90
		−4	40	50	354			11	23	19	198			2	45	59	63			−4	21	29	65
		−5	28	38	351			12	24	19	73			−1	46	49	90			−7	20	23	92
		−7	35	45	335			−1	18	16	44			−2	25	32	21	13	4	0	14	18	355
		−10	24	31	333			−2	18	12	300			−3	27	38	6			−1	17	27	231
10	8	0	15	4	24			−3	30	40	253			−5	26	31	348	13	5	0	27	32	344
		1	18	18	66			−4	32	26	81			−7	23	27	83			4	18	20	75
		2	21	36	62			−5	12	20	233			−8	20	27	297			5	24	28	6
		3	17	11	286			−8	29	35	243			−9	20	19	116			6	11	17	8
		5	27	29	91			−11	26	33	261	12	4	0	15	20	217			−2	21	19	35
		7	19	28	81	11	5	1	48	47	349			1	16	24	72			−3	23	25	2
		−1	16	24	45			3	22	24	292			2	16	25	201			−4	26	22	124
		−2	16	5	291			5	20	15	87			4	29	42	89			−6	16	19	17
		−3	15	17	15			6	25	27	335			−2	16	24	3	13	6	−1	23	27	725
		−4	16	18	97			−1	23	24	77			−4	20	23	344			−6	38	45	262
		−5	16	22	260			−2	45	53	355			−6	24	33	279	13	7	0	23	38	358
		−6	17	20	116			−3	33	35	60			−7	23	30	341			2	18	17	334
		−8	16	16	256			−6	35	32	98			−11	23	23	275			3	19	30	352
		−10	17	14	280			−7	29	25	1	12	5	0	30	41	10	13	8	0	15	3	338
10	9	−1	19	21	4			−9	28	18	133			1	38	19	57	13	9	0	15	16	339
		−2	26	26	350	11	6	0	15	15	205			2	17	19	48	14	0	0	27	34	0
10	10	2	17	15	71			1	25	37	104			3	17	26	7			3	36	39	0
		−1	23	26	92			4	21	34	93			4	29	27	105			4	34	28	180
10	11	0	15	16	247			−1	30	33	64			6	20	19	4			5	21	13	180
11	0	0	42	54	180			−5	30	26	270			−1	41	36	116			−2	19	27	0
		1	11	10	0			−8	36	42	258			−2	29	32	15			−8	15	39	0
		3	54	59	180	11	7	0	18	28	342			−3	27	27	5			−11	30	22	0
		−3	10	26	180			1	18	31	349			−4	10	25	95	14	1	1	23	33	76
		−4	54	58	0			−1	19	11	64			−5	39	39	324			−2	18	29	101
		−7	49	46	0			−2	23	33	349			−6	10	14	168	14	2	3	17	22	25
		−9	62	54	0			−4	16	22	3			−8	31	28	328			−1	19	21	250
		−10	40	42	0			−5	25	41	0	12	7	0	19	29	353			−2	17	24	340
		−12	44	41	0	11	8	0	15	15	290			1	18	22	52			−3	19	31	12
11	1	0	45	51	57			2	16	27	91			2	18	9	91			−5	24	24	347
		1	15	14	13			3	15	6	337			3	18	34	355	14	3	1	20	30	76
		2	31	47	104			4	16	26	88			−2	17	23	336			−3	19	25	314
		5	18	31	72			5	16	11	69			−5	26	28	358	14	4	0	24	25	1
		9	28	27	255			7	17	25	77	12	8	1	18	24	95	14	5	0	16	19	16
		−1	56	64	84			−1	19	24	110			7	18	17	80			1	24	21	94
		−3	35	44	84			−2	15	11	127			−1	17	14	308			2	11	20	19
		−4	16	41	103			−3	17	18	286			−3	20	29	261			3	19	19	20
		−5	19	21	1			−4	15	16	62			−4	16	7	101			−3	22	20	342
		−6	34	41	70			−5	15	17	281	12	9	0	22	24	341	14	6	0	26	13	25
		−10	30	23	305			−8	19	26	272	13	0	0	14	4	180	15	0	2	19	27	0
11	2	0	33	47	194	11	9	−2	29	34	0			1	24	38	0			−3	35	47	0
		1	15	27	51	11	12	0	15	16	328			5	35	31	180			−8	24	23	0
		2	16	19	100	12	0	1	11	24	0			−2	61	70	0	15	1	1	19	25	74
		3	18	31	161			5	35	48	180			−5	27	40	0	15	2	0	20	34	2
		5	26	36	168			8	35	35	180			−6	26	32	0			3	21	22	6
		6	24	31	151			11	21	21	180			−7	40	45	0			−1	16	20	300
		7	28	38	81			−3	30	20	180			−10	34	30	0			−2	15	15	298
		8	26	40	178			−5	17	34	0			−11	27	23	0			−3	21	32	354
		10	12	14	84			−8	32	7	180			−13	24	17	0	15	4	0	14	19	354
		−2	18	22	139			−10	45	49	0	13	1	0	20	18	113	15	5	2	30	29	356
		−3	8	18	163			−11	20	9	180			1	30	45	93	16	0	0	14	19	0
		−4	34	46	23	12	1	1	16	35	103			4	34	48	96	16	2	0	18	27	4
		−6	16	19	1			2	22	33	111			−1	48	50	122			−1	13	21	342
		−7	53	55	7			3	14	16	315			−2	33	36	37			−3	23	26	3
		−9	21	24	17			4	34	36	44			−3	18	10	291	17	2	0	14	11	295
		−10	24	33	15			−1	52	60	51			−4	40	44	107	17	4	0	14	12	311
		−12	23	24	338			−2	26	16	344			−7	18	21	111						
11	3	0	12	20	83			−4	44	53	93			−8	15	23	275						

minimum function [19] was prepared. At the same time a second three-dimensional Fourier synthesis based on the iodine phase angles and with coefficients weighted according to the method proposed by Sim [20] was evauated. In this and many later syntheses the axial subdivisions adopted were *a*/48, *b*/48, and *c*/48, the function thus being evaluated at 55,296 points

[19] Buerger, *Acta Cryst.*, 1951, **4**, 531.
[20] Sim, *Acta Cryst.*, 1959, **12**, 813.

in the unit cell. The three-dimensional functions were displayed as contoured sections drawn on sheets of glass or Perspex and stacked in metal frames.

The second electron-density distribution was then thoroughly compared with the minimum function, and strong peaks which were well represented in both functions were considered to represent genuine atoms. Over the whole asymmetric unit it proved possible in this way to assign co-ordinates to 50 " atoms " other than the two iodine atoms. At this stage relative peak heights did not justify any distinction between carbon and oxygen.

These 50 peaks were now treated as carbon, and, by use of the scattering factor of Berghuis *et al.*[21] and a temperature factor of $B = 4{\cdot}9$ Å^2, they were included in the phasing calculations for the third Fourier synthesis. When this was drawn out (Fig. 10) it showed a considerable diminution of background detail and two distinct and well-separated molecules were clearly visible. Although some of the atoms were weak and barely resolved, a total of 76 could now be counted, and these were found to group themselves into two chemically identical but differently oriented molecules. With this very important double confirmation of each atom it was now possible to assign the structure (IV) to epilimonol iodoacetate.

From this point the refinement of the structure proceeded in a relatively straightforward way. Another Fourier synthesis gave better definition to the peaks, and the fifth F_c calculation reduced the discrepancy R to 22%. A least-squares refinement for 62 atoms was then carried out, followed by several further three-dimensional Fourier syntheses, for which it is not necessary to give details. The course of the refinement is summarised in Table 1. Termination of series errors were taken into account near the end of the refinement by applying back-shift corrections based on a three-dimensional F_c synthesis. The eighth and final three-dimensional Fourier synthesis is shown in Fig. 1 by means of superimposed contour sections drawn parallel to (010). All the atoms are now well resolved and the oxygen atoms are represented by distinctly higher peaks than the carbon atoms.

For the final F_c calculation the atomic scattering factors used for carbon and oxygen were those of Berghuis *et al.*[21] and for iodine that of James and Brindley [22] with a uniform isotropic temperature factor of $B = 4{\cdot}94$ Å^2. The final value for the discrepancy R is 18·1% over all structure factors, and we do not think the refinement can usefully be carried further with the existing data. The atomic co-ordinates, molecular dimensions, and some non-bonded distances are given in Tables 2—6.

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[21] Berghuis, Haanappel, Potters, Loopstra, MacGillavry, and Veenendaal, *Acta Cryst.*, 1955, **8**, 478.
[22] James and Brindley, *Z. Krist.*, 1931, **78**, 470.