The Stereochemistry of Isocolumbin: *X*-Ray Analysis of the 1-*p*-Iodophenyl-3-phenylpyrazoline Adduct of Isocolumbin

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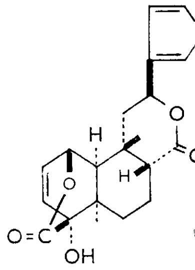
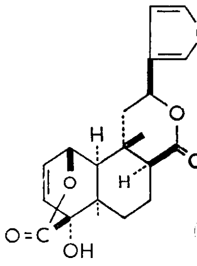
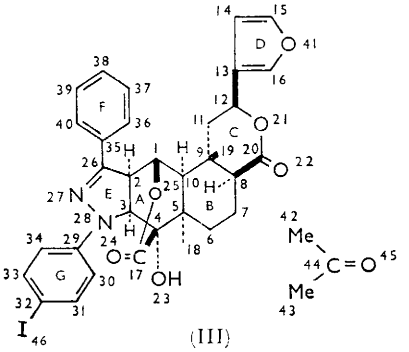
A suitable heavy atom derivative of isocolumbin has now been found in the 1-*p*-iodophenyl-3-phenylpyrazoline adduct (III) which crystallises as a monoacetone solvate in the tetragonal space group $P4_12_12$ with eight molecules of $C_{33}H_{31}O_6N_2I,(CH_3)_2CO$ in a unit cell of dimensions $a = b = 9{\cdot}68$, $c = 70{\cdot}79$ Å. With initial phasing on the iodine atom, the crystal structure was solved by three-dimensional Fourier and least squares calculations. The final value of R over 1860 independent reflections was 9·5%. The results establish structure (III) for this derivative and hence confirm structure (II) for isocolumbin. The molecular dimensions and the stereochemistry of the three boat shaped six-membered rings and the 0-lactone systems are discussed.

The stereochemistry of the Colombo root bitter principles has been fully discussed by Overton, Weir, and Wylie.[1] From chemical, spectroscopic, and optical rotatory dispersion studies they have deduced the stereochemistry and absolute configuration of columbin (I) and isocolumbin (II). Two points of general interest which call for further investigation arise from this work: (1) the biogenetically unexpected stereochemistry of columbin, and (2) the conformational situation that arises in isocolumbin from the fusion of three boat shaped six-membered rings. To confirm the stereochemistry and make a quantitative study of the conformations of the various ring systems, particularly the δ-lactones, with direct measurements of the distances involved, it was decided to carry out a detailed *X*-ray crystal analysis. Very extensive attempts to prepare a simple heavy-atom derivative of columbin or isocolumbin were unsuccessful, but finally a most suitable derivative containing a heavy atom remote from the diterpene framework was found in the 1-*p*-iodophenyl-3-phenylpyrazoline adduct of isocolumbin (III). On crystallisation from acetone a monoacetone solvate was formed, and this compound, $C_{33}H_{31}O_6N_2I,(CH_3)_2CO$, was used in all the *X*-ray work.


(I)


(II)


(III)

The ratio of the iodine structure factor to the average structure factor from all the remaining atoms is about 1·5 to 1, so the situation is favourable for the usual heavy-atom method of phase determination.[2] Four successive three-dimensional electron-density distributions were calculated, and in the last all the atoms including those of the acetone solvate molecule were clearly resolved. At this stage the average discrepancy between the measured and calculated structure amplitudes, R, was 23·3% over 1860 independent reflexions.

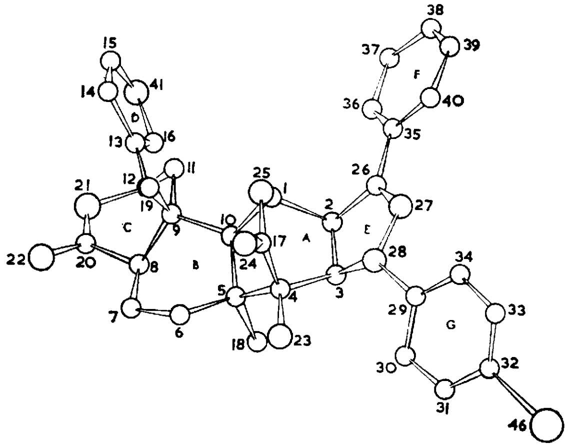


FIGURE 1 Perspective view along the *b* crystal axis of the iodo-derivative of isocolumbin

This was followed by five cycles of least squares isotropic block diagonal refinement and two final cycles when the iodine atom was allowed to refine anisotropically. The final R factor was 9·5%.

Our results establish structure (III) for this derivative and hence confirm structure (II) for isocolumbin. The complicated electron density distributions are not reproduced here, but a perspective view of the molecule, along the *b* axis, is shown in Figure 1. The arrangement of the molecules in a portion of the tetragonal unit cell ($c = 70{\cdot}8$ Å) is shown in Figure 2, where the acetone solvate molecule is indicated by dotted lines.

The final atomic co-ordinates, with estimated standard deviations and temperature factors, are listed in

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[1] K. H. Overton, N. G. Weir, and A. Wylie, *J. Chem. Soc.* (*C*), 1966, 1482.

[2] J. M. Robertson, *Proc. Chem. Soc.*, 1963, 229.

Table 1, bond lengths in Table 2, bond angles in Table 3, a selection of intramolecular non-bonded distances in Table 4, and a selection of intermolecular distances in Table 5.*

Stereochemistry.—The hydrogen atoms at C(8), C(10), and C(12), the methyl groups C(5) and C(9) are all axial, while the hydrogen atom at C(1) and the hydroxyl group at C(4) are quasi-equatorial.

TABLE 1

Fractional atomic co-ordinates, their estimated standard deviations in Å, and isotropic temperature factors ($B = 8\pi^2U_{iso}$). Atoms are numbered as in structure (III). Origin of co-ordinates as in " International Tables for *X*-Ray Crystallography," The Kynoch Press, Birmingham, 1952, vol. 1

Atom	x/a	y/b	z/c	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	U_{iso} (Å^2)
C(1)	0·0508	−0·0284	0·0735	0·016	0·016	0·014	0·030
C(2)	0·2057	−0·0434	0·0658	0·016	0·016	0·016	0·027
C(3)	0·2168	0·0449	0·0479	0·016	0·016	0·015	0·027
C(4)	0·0800	0·0956	0·0424	0·017	0·018	0·016	0·040
C(5)	−0·0309	−0·0218	0·0392	0·017	0·018	0·016	0·040
C(6)	−0·1697	0·0471	0·0325	0·018	0·017	0·016	0·035
C(7)	−0·2934	−0·0506	0·0339	0·016	0·017	0·015	0·035
C(8)	−0·2763	−0·1702	0·0491	0·016	0·017	0·015	0·030
C(9)	−0·2013	−0·1184	0·0668	0·016	0·017	0·016	0·029
C(10)	−0·0484	−0·0971	0·0599	0·017	0·018	0·016	0·041
C(11)	−0·1973	−0·2306	0·0823	0·016	0·016	0·014	0·029
C(12)	−0·2639	−0·3641	0·0753	0·020	0·020	0·018	0·048
C(13)	−0·2989	−0·4658	0·0903	0·017	0·017	0·016	0·039
C(14)	−0·3721	−0·4385	0·1074	0·025	0·024	0·023	0·082
C(15)	−0·3675	−0·5517	0·1181	0·029	0·028	0·026	0·084
C(16)	−0·2532	−0·5932	0·0906	0·022	0·022	0·021	0·061
C(17)	0·0272	0·1902	0·0579	0·018	0·018	0·016	0·038
C(18)	0·0270	−0·1275	0·0241	0·017	0·017	0·015	0·037
C(19)	−0·2676	0·0198	0·0755	0·020	0·020	0·018	0·052
C(20)	−0·4138	−0·2176	0·0557	0·018	0·018	0·017	0·034
O(21)	−0·4125	−0·3205	0·0682	0·012	0·013	0·011	0·046
O(22)	−0·5244	−0·1822	0·0501	0·014	0·015	0·013	0·058
O(23)	0·0835	0·1834	0·0261	0·013	0·013	0·011	0·048
O(24)	−0·0143	0·3085	0·0575	0·014	0·014	0·013	0·056
O(25)	0·0227	0·1169	0·0750	0·011	0·011	0·011	0·036
C(26)	0·3108	0·0232	0·0790	0·015	0·015	0·014	0·022
N(27)	0·3736	0·1269	0·0719	0·013	0·013	0·011	0·029
N(28)	0·3133	0·1502	0·0534	0·013	0·013	0·011	0·026
C(29)	0·4215	0·2105	0·0418	0·017	0·017	0·016	0·036
C(30)	0·3990	0·1977	0·0212	0·018	0·018	0·016	0·041
C(31)	0·5023	0·2575	0·0103	0·021	0·022	0·020	0·061
C(32)	0·6141	0·3334	0·0182	0·018	0·019	0·018	0·050
C(33)	0·6217	0·3508	0·0369	0·019	0·019	0·017	0·051
C(34)	0·5281	0·2846	0·0498	0·021	0·020	0·019	0·055
C(35)	0·3450	−0·0310	0·0986	0·016	0·016	0·014	0·025
C(36)	0·2929	−0·1508	0·1048	0·018	0·019	0·017	0·041
C(37)	0·3324	−0·2059	0·1228	0·020	0·020	0·018	0·046
C(38)	0·4324	−0·1315	0·1336	0·023	0·023	0·021	0·062
C(39)	0·4806	−0·0020	0·1276	0·022	0·021	0·018	0·054
C(40)	0·4367	0·0465	0·1098	0·018	0·018	0·016	0·040
O(41)	−0·3012	−0·6564	0·1075	0·017	0·018	0·016	0·085
C(42)	0·3026	0·4881	0·0806	0·022	0·023	0·021	0·062
C(43)	0·2300	0·5446	0·0464	0·026	0·027	0·024	0·080
C(44)	0·1984	0·5510	0·0673	0·021	0·021	0·019	0·051
O(45)	0·1048	0·6195	0·0742	0·017	0·016	0·015	0·080
I(46)	0·7602	0·4215	−0·0007	0·002	0·002	0·002	—

Anisotropic temperature factors of the iodine atom

U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
0·085	0·083	0·085	0·042	0·065	−0·017

In ring A the atoms C(1), C(2), C(3), C(4), C(5), and C(10) have the boat conformation. This ring is *cis*-fused to ring B and also to ring E.

In the δ-lactone system the five atoms of the lactone group, C(1), O(25), C(17), O(24), and C(4) are as expected almost exactly co-planar. The mean plane * through these atoms has the equation

$$-0{\cdot}9378X - 0{\cdot}2626Y - 0{\cdot}2272Z + 1{\cdot}6344 = 0 \quad (1)$$

and the deviations of the atoms from this plane are +0·064, −0·075, −0·028, +0·055, and −0·017 Å, respectively. The estimated standard deviations (see Table 2) are 0·02 — 0·03 Å, so the small departures of atoms C(1), O(25), and O(24) from plane (1) are possibly significant.

The distances of atoms C(2) and C(3) from plane (1) are −1·18 and −1·22 Å, while the distance of C(5) and C(10) are +1·34 and +1·36 Å, respectively showing that both δ-lactone systems have the half-boat conformation.

The hydroxyl oxygen atom O(23) also lies in plane (1)

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* Intermolecular distances, bond angles, and mean planes were calculated using two Glasgow Algol programmes written by Mr. K. W. Muir and Dr. W. E. Oberhänsli, respectively.

(deviation —0·007 Å). The conformation of the cage structure of ring A is indicated in Figure 3. The intramolecular distance between O(23) and O(24) is 2·70 Å, and that between O(23) and N(28) is 2·97 Å. Both these distances are within the range for hydrogen bonding, and there are no intermolecular approaches between O(23) and other atoms that suggest hydrogen bonding. However, the angles O(24)O(23)C(4) and N(28)O(23)C(4) are only 65·9 and 55·1°, respectively, indicating that the hydrogen atom would lie well off the line joining either O(23) and O(24), or O(23) and N(28).

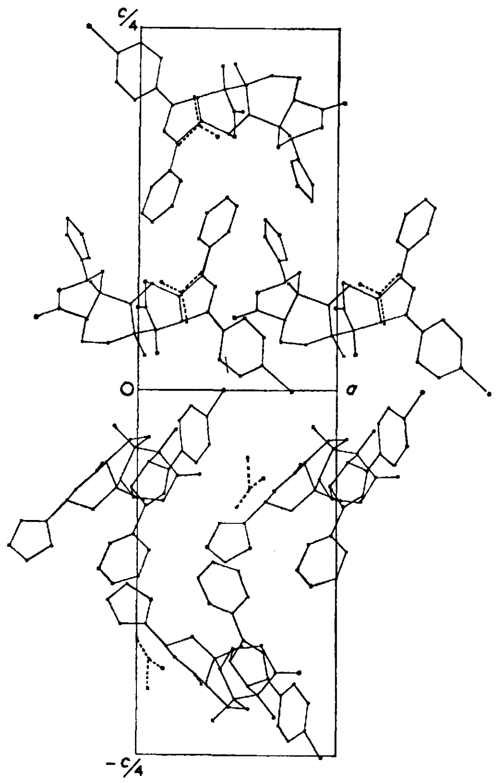

FIGURE 2 The arrangement of the molecules of the iodo-derivative of isocolumbin in half the unit cell, as viewed along the *b* axis

Ring B also has a boat conformation, and it is *trans*-fused to ring C. In the δ-lactone system constituting ring C the lactone group is found to be strictly planar. The mean plane through the five atoms C(8), C(20), O(22), O(21), and C(12) has the equation

$$0\cdot0197X - 0\cdot6950Y - 0\cdot7188Z + 1\cdot4215 = 0 \quad (2)$$

The deviations of these atoms from the plane are +0·015, —0·001, —0·028, +0·028, and —0·014 Å, respectively, all within the limits of the standard deviations. The distances of the atoms C(9) and C(11) from plane (2) are —1·22 and —1·25 Å, indicating the half-boat conformation of ring C.

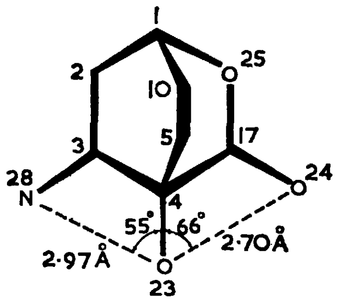

FIGURE 3 Conformation of the cage structure of ring A

The furan ring D is planar, the equation of the mean plane through the five atoms being

$$-0\cdot8533X - 0\cdot2882Y - 0\cdot4305Z - 1\cdot0133 = 0 \quad (3)$$

The deviations of the atoms C(13), C(14), C(15), O(41), and C(16) from this plane are +0·007, +0·017, —0·032, +0·035, and —0·032 Å, respectively.

The pyrazole ring E is also planar, the equation of the mean plane through the five atoms being

$$0\cdot7199X - 0\cdot5864Y - 0\cdot3713Z + 0\cdot0366 = 0 \quad (4)$$

The deviations of the atoms C(2), C(3), N(28), N(27), and C(26) from this plane are —0·015, +0·032, —0·038, +0·028, and —0·007 Å, respectively.

The mean plane through the benzene ring F and atom C(26) has the equation

$$0\cdot7679X - 0\cdot4845Y - 0\cdot4191Z + 0\cdot1954 = 0 \quad (5)$$

The deviations of the atoms C(26) and C(35) to C(40) from this plane are +0·051, —0·023, —0·031, —0·013, +0·061, —0·010, and —0·036 Å, respectively.

TABLE 2

Bond lengths and their estimated standard deviations

Bond	Length	E.s.d.	Bond	Length	E.s.d.	Bond	Length	E.s.d.	Bond	Length	E.s.d.
C(1)–C(2)	1·600	0·022	C(6)–C(7)	1·529	0·024	C(15)–O(41)	1·415	0·032	C(31)–C(32)	1·421	0·028
C(1)–C(10)	1·510	0·023	C(7)–C(8)	1·590	0·022	C(16)–O(41)	1·417	0·027	C(32)–C(33)	1·333	0·025
C(1)–O(25)	1·436	0·020	C(8)–C(9)	1·533	0·022	C(17)–O(24)	1·213	0·022	C(33)–C(34)	1·440	0·027
C(2)–C(3)	1·531	0·022	C(8)–C(20)	1·483	0·024	C(17)–O(25)	1·400	0·020	C(35)–C(36)	1·337	0·024
C(2)–C(26)	1·524	0·021	C(9)–C(10)	1·571	0·024	C(20)–O(21)	1·332	0·021	C(35)–C(40)	1·405	0·023
C(3)–C(4)	1·464	0·023	C(9)–C(11)	1·542	0·022	C(20)–O(22)	1·192	0·023	C(36)–C(37)	1·432	0·030
C(3)–N(28)	1·437	0·020	C(9)–C(19)	1·604	0·026	C(26)–N(27)	1·275	0·020	C(37)–C(38)	1·429	0·030
C(4)–C(5)	1·579	0·024	C(11)–C(12)	1·524	0·025	C(26)–C(35)	1·521	0·020	C(38)–C(39)	1·403	0·031
C(4)–C(17)	1·516	0·024	C(12)–C(13)	1·487	0·023	N(27)–N(28)	1·451	0·017	C(39)–C(40)	1·408	0·025
C(4)–O(23)	1·438	0·021	C(12)–O(21)	1·581	0·023	N(28)–C(29)	1·455	0·021	C(44)–C(42)	1·505	0·030
C(5)–C(6)	1·573	0·025	C(13)–C(14)	1·423	0·029	C(29)–C(30)	1·478	0·023	C(44)–C(43)	1·514	0·031
C(5)–C(10)	1·648	0·023	C(13)–C(16)	1·310	0·028	C(29)–C(34)	1·379	0·027	C(44)–O(45)	1·224	0·026
C(5)–C(18)	1·581	0·023	C(14)–C(15)	1·333	0·036	C(30)–C(31)	1·387	0·027	C(32)–I(46)	2·125	0·018

TABLE 3

Bond angles and their estimated standard deviations

C(10)C(1)O(25)	110·9°	± 1·3°
C(2)C(1)O(25)	106·9	1·2
C(2)C(1)C(10)	109·9	1·2
C(3)C(2)C(26)	103·0	1·2
C(1)C(2)C(3)	107·2	1·2
C(1)C(2)C(26)	112·3	1·2
C(4)C(3)N(28)	115·0	1·4
C(2)C(3)N(28)	102·5	1·2
C(2)C(3)C(4)	110·1	1·3
C(3)C(4)O(23)	113·0	1·4
C(3)C(4)C(17)	108·3	1·4
C(3)C(4)C(5)	114·3	1·4
C(17)C(4)O(23)	103·6	1·3
C(5)C(4)O(23)	108·9	1·3
C(5)C(4)C(17)	108·1	1·4
C(4)C(5)C(18)	108·8	1·3
C(4)C(5)C(6)	108·6	1·4
C(4)C(5)C(10)	104·9	1·2
C(6)C(5)C(18)	111·9	1·3
C(6)C(5)C(10)	111·7	1·3
C(10)C(5)C(18)	110·6	1·3
C(5)C(6)C(7)	112·8°	± 1·4°
C(6)C(7)C(8)	114·3	1·3
C(7)C(8)C(9)	111·5	1·3
C(7)C(8)C(20)	110·2	1·3
C(9)C(8)C(20)	105·6	1·3
C(8)C(9)C(10)	103·6	1·2
C(10)C(9)C(11)	106·8	1·3
C(10)C(9)C(19)	112·7	1·4
C(8)C(9)C(11)	111·2	1·3
C(8)C(9)C(19)	113·3	1·4
C(11)C(9)C(19)	109·0	1·3
C(1)C(10)C(9)	117·3	1·3
C(5)C(10)C(9)	115·5	1·3
C(1)C(10)C(5)	107·8	1·3
C(9)C(11)C(12)	111·0	1·3
C(11)C(12)C(13)	115·3	1·4
C(13)C(12)O(21)	101·4	1·4
C(11)C(12)O(21)	105·2	1·4
C(14)C(13)C(16)	109·1	1·8
C(12)C(13)C(16)	123·9	1·7
C(12)C(13)C(14)	126·6	1·7
C(13)C(14)C(15)	108·3°	± 2·1°
C(14)C(15)O(41)	107·5	2·1
C(13)C(16)O(41)	108·0	1·8
O(24)C(17)O(25)	119·3	1·5
C(4)C(17)O(24)	131·5	1·6
C(4)C(17)O(25)	109·1	1·4
O(21)C(20)O(22)	116·5	1·6
C(8)C(20)O(22)	127·6	1·6
C(8)C(20)O(21)	115·6	1·5
C(12)O(21)C(20)	114·9	1·3
C(1)O(25)C(17)	115·3	1·2
N(27)C(26)C(25)	121·8	1·3
C(2)C(26)N(27)	114·2	1·2
C(2)C(26)C(35)	124·0	1·3
C(26)N(27)N(28)	106·6	1·2
C(3)N(28)N(27)	113·3	1·1
C(29)N(28)C(3)	126·8	1·2
N(27)N(28)C(29)	106·6	1·2
N(28)C(29)C(34)	120·8	1·5
C(30)C(29)C(34)	124·1	1·6
N(28)C(29)C(30)	114·8°	± 1·4°
C(29)C(30)C(31)	113·9	1·6
C(30)C(31)C(32)	123·1	1·7
C(31)C(32)C(33)	119·7	1·7
C(31)C(32)I(46)	122·5	1·4
C(33)C(32)I(46)	117·8	1·3
C(32)C(33)C(34)	122·7	1·8
C(29)C(34)C(33)	116·0	1·6
C(26)C(35)C(36)	121·0	1·4
C(26)C(35)C(40)	117·8	1·4
C(36)C(35)C(40)	121·2	1·5
C(35)C(36)C(37)	120·8	1·7
C(36)C(37)C(38)	118·0	1·8
C(37)C(38)C(39)	120·8	1·9
C(38)C(39)C(40)	118·0	1·8
C(39)C(40)C(35)	121·0	1·6
C(15)O(41)C(16)	106·7	1·8
C(42)C(44)O(45)	117·7	1·8
C(42)C(44)C(43)	117·2	1·8
C(43)C(44)O(45)	124·3	1·9

TABLE 4

Intramolecular non-bonded distances <3·0 Å

C(1) · · · · C(17)	2·40
C(1) · · · · C(4)	2·52
C(1) · · · · C(3)	2·52
C(1) · · · · C(5)	2·55
C(1) · · · · C(26)	2·59
C(1) · · · · C(9)	2·63
C(2) · · · · N(28)	2·32
C(2) · · · · N(27)	2·35
C(2) · · · · O(25)	2·44
C(2) · · · · C(4)	2·46
C(2) · · · · C(10)	2·55
C(2) · · · · C(35)	2·69
C(2) · · · · C(17)	2·90
C(2) · · · · C(5)	2·97
C(3) · · · · C(26)	2·39
C(3) · · · · N(27)	2·41
C(3) · · · · C(17)	2·42
C(3) · · · · O(23)	2·42
C(3) · · · · C(5)	2·56
C(3) · · · · C(29)	2·59
C(3) · · · · O(25)	2·77
C(3) · · · · C(30)	2·98
C(4) · · · · O(25)	2·38
C(4) · · · · O(24)	2·49
C(4) · · · · C(10)	2·56
C(4) · · · · C(6)	2·56
C(4) · · · · C(18)	2·57
C(5) · · · · O(23)	2·46
C(5) · · · · C(17)	2·51
C(5) · · · · C(7)	2·58
C(5) · · · · C(9)	2·72
C(5) · · · · C(8)	2·86
C(5) · · · · O(25)	2·91
C(6) · · · · C(18)	2·61
C(6) · · · · C(8)	2·62
C(6) · · · · C(10)	2·66
C(6) · · · · O(23)	2·82
C(6) · · · · C(9)	2·93
C(6) · · · · C(17)	2·97
C(7) · · · · C(20)	2·52
C(7) · · · · C(9)	2·58
C(7) · · · · O(22)	2·82
C(8) · · · · O(21)	2·38
C(8) · · · · O(22)	2·40
C(8) · · · · C(10)	2·44
C(8) · · · · C(11)	2·54
C(8) · · · · C(19)	2·62
C(8) · · · · C(12)	2·64
C(9) · · · · C(20)	2·40
C(9) · · · · C(12)	2·53
C(9) · · · · O(21)	2·83
C(10) · · · O(25)	2·43
C(10) · · · C(11)	2·50
C(10) · · · C(19)	2·64
C(10) · · · C(18)	2·66
C(10) · · · C(17)	2·88
C(11) · · · O(21)	2·47
C(11) · · · C(13)	2·54
C(11) . . . C(19)	2·56
C(11) · · · C(20)	2·82
C(12) · · · C(20)	2·46
C(12) · · · C(16)	2·47
C(12) · · · C(14)	2·60
C(13) · · · O(41)	2·21
C(13) · · · C(15)	2·23
C(13) · · · O(21)	2·38
C(14) · · · O(41)	2·22
C(14) · · · C(16)	2·23
C(15) · · · C(16)	2·27
C(17) · · · O(23)	2·32
C(17) · · · N(28)	2·81
C(19) · · · O(25)	2·96
O(21) · · · O(22)	2·15
O(23) · · · O(24)	2·70
O(23) · · · N(28)	2·97
O(24) · · · O(25)	2·26
O(25) · · · C(26)	2·94
C(26) · · · N(28)	2·19
C(26) · · · C(36)	2·49
C(26) · · · C(40)	2·51
N(27) · · · C(29)	2·33
N(27) · · · C(35)	2·45
N(27) · · · C(34)	2·65
N(27) · · · C(40)	2·86
N(28) · · · C(34)	2·46
N(28) · · · C(30)	2·47
C(29) · · · C(33)	2·39
C(29) · · · C(31)	2·40
C(29) · · · C(32)	2·77
C(30) · · · C(32)	2·47
C(30) · · · C(34)	2·52
C(30) · · · C(33)	2·84
C(31) · · · C(33)	2·38
C(31) · · · C(34)	2·82
C(32) · · · C(34)	2·43
C(35) · · · C(37)	2·41
C(35) · · · C(39)	2·45
C(35) · · · C(38)	2·79
C(36) · · · C(40)	2·39
C(36) · · · C(38)	2·45
C(36) · · · C(39)	2·82
C(37) · · · C(39)	2·46
C(37) · · · C(40)	2·80
C(38) · · · C(40)	2·41
C(42) · · · O(45)	2·34
C(42) · · · C(43)	2·58
C(43) · · · O(45)	2·42

TABLE 5

Intermolecular distances <4 Å

O(22) · · · C(2)$_{\mathrm{V}}$	3·14
O(24) · · · C(44)	3·20
O(22) · · · C(26)$_{\mathrm{V}}$	3·27
O(45) · · · C(10)$_{\mathrm{II}}$	3·28
O(45) · · · C(11)$_{\mathrm{II}}$	3·31
O(22) · · · C(3)$_{\mathrm{V}}$	3·33
O(24) · · · C(43)	3·38
O(24) · · · C(16)$_{\mathrm{II}}$	3·43
O(45) · · · O(24)	3·43
C(42) · · · O(21)$_{\mathrm{I}}$	3·44
O(45) · · · C(1)$_{\mathrm{II}}$	3·45
O(45) · · · C(2)$_{\mathrm{II}}$	3·46
O(22) · · · N(27)$_{\mathrm{V}}$	3·51
C(43) · · · O(22)$_{\mathrm{I}}$	3·56
O(45) · · · C(12)$_{\mathrm{II}}$	3·57
C(6) · · · · C(33)$_{\mathrm{V}}$	3·58
C(39) · · · C(16)$_{\mathrm{III}}$	3·58
O(22) · · · I(46)$_{\mathrm{VII}}$	3·58
O(22) · · · N(28)$_{\mathrm{V}}$	3·59
C(39) · · · O(41)$_{\mathrm{III}}$	3·60
C(42) · · · C(38)$_{\mathrm{III}}$	3·60
O(45) · · · C(36)$_{\mathrm{II}}$	3·60
C(42) · · · N(27)	3·61
C(6) · · · · C(32)$_{\mathrm{V}}$	3·61
C(38) · · · C(16)$_{\mathrm{III}}$	3·62
C(38) · · · O(41)$_{\mathrm{III}}$	3·63
C(19) · · · N(27)$_{\mathrm{V}}$	3·63
C(42) · · · C(34)	3·66
C(31) · · · C(31)$_{\mathrm{IV}}$	3·66
O(45) · · · C(39)$_{\mathrm{III}}$	3·70
C(19) · · · C(34)$_{\mathrm{V}}$	3·71
C(43) · · · C(29)	3·74
C(42) · · · C(14)$_{\mathrm{I}}$	3·74
C(40) · · · C(37)$_{\mathrm{III}}$	3·75
C(19) · · · C(40)$_{\mathrm{V}}$	3·76
C(34) · · · C(16)$_{\mathrm{I}}$	3·77
C(7) · · · · C(29)$_{\mathrm{V}}$	3·78
C(42) · · · N(28)	3·79
C(39) · · · C(15)$_{\mathrm{III}}$	3·79
C(14) · · · C(37)$_{\mathrm{V}}$	3·80
C(43) · · · C(34)	3·83
O(24) · · · C(33)$_{\mathrm{V}}$	3·83
C(7) · · · · C(34)$_{\mathrm{V}}$	3·84
O(25) · · · C(38)$_{\mathrm{III}}$	3·84
C(39) · · · C(13)$_{\mathrm{III}}$	3·84
C(40) · · · O(41)$_{\mathrm{I}}$	3·84
O(45) · · · C(38)$_{\mathrm{III}}$	3·86
C(19) · · · O(41)$_{\mathrm{II}}$	3·88
O(22) · · · C(29)$_{\mathrm{V}}$	3·88
C(42) · · · O(24)	3·88
C(33) · · · O(21)$_{\mathrm{I}}$	3·89
C(42) · · · C(36)$_{\mathrm{II}}$	3·89
C(19) · · · C(16)$_{\mathrm{II}}$	3·90
C(20) · · · C(26)$_{\mathrm{V}}$	3·91
C(6) · · · · C(34)$_{\mathrm{V}}$	3·92
C(44) · · · C(17)	3·92
C(44) · · · O(22)$_{\mathrm{I}}$	3·92
C(43) · · · N(28)	3·93
C(44) · · · C(2)$_{\mathrm{II}}$	3·93
C(7) · · · · C(30)$_{\mathrm{V}}$	3·93
O(23) · · · O(23)$_{\mathrm{IV}}$	3·93
O(45) · · · C(9)$_{\mathrm{II}}$	3·93
C(42) · · · C(13)$_{\mathrm{I}}$	3·94
C(30) · · · C(31)$_{\mathrm{IV}}$	3·94
C(7) · · · · C(31)$_{\mathrm{V}}$	3·95
O(22) · · · C(35)$_{\mathrm{V}}$	3·95
C(44) · · · O(21)$_{\mathrm{I}}$	3·97
C(7) · · · · C(33)$_{\mathrm{V}}$	3·97
C(11) · · · C(15)$_{\mathrm{VI}}$	3·97
C(7) · · · · C(32)$_{\mathrm{V}}$	3·98
C(14) · · · C(38)$_{\mathrm{V}}$	3·98
C(39) · · · C(14)$_{\mathrm{III}}$	3·98

The Roman numerals refer to the following equivalent positions:

I; $1 + x, 1 + y, z$
II; $x, 1 + y, z$
III; $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{4} - z$
IV; $y, x, -z$
V; $-1 + x, y, z$
VI; $-\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{4} - z$
VII; $-1 + y, -1 + x, -z$

The equation of the mean plane through N(28), C(29) to C(34) of benzene ring G, and I(46) is

$$-0{\cdot}5461X + 0{\cdot}8370Y - 0{\cdot}0361Z + 0{\cdot}5883 = 0 \quad (6)$$

The deviations of these atoms in order from this plane are +0·013, −0·041, +0·027, −0·007, −0·003, +0·050, −0·025, and −0·013 Å.

The acetone solvent molecule is also planar within experimental limits. The equation of the mean plane through the four atoms is

$$-0{\cdot}5462X - 0{\cdot}8371Y - 0{\cdot}0302Z + 5{\cdot}7082 = 0 \quad (7)$$

The deviations of the atoms C(42), C(43), C(44), and O(45) from this plane are −0·016, −0·017, +0·054, and −0·021 Å, respectively.

TABLE 6

Dimensions of the furan ring

	Furan [a]	3,4-Furandicarboxylic acid [b]	Cedrelone iodoacetate	This structure
C–C	1·440 ± 0·016	1·454 ± 0·005	1·46 ± 0·09	1·423 ± 0·029
C=C	1·354 ± 0·016	1·346 ± 0·003	1·32 ± 0·09	1·333 ± 0·036
			1·22 ± 0·09	1·310 ± 0·036
C–O	1·371 ± 0·016	1·356 ± 0·003	1·34 ± 0·09	1·415 ± 0·032
				1·417 ± 0·027
C–O–C	106·0 ± 1·6°	107·2 ± 0·3°	114°	106·7 ± 1·8°
C=C–O	110·9 ± 0·7°	110·8 ± 0·2°	110°	107·5 ± 2·1°
			100°	108·0 ± 1·8°
C–C=C	106·1 ± 0·6°	105·7 ± 0·2°	93°	109·1 ± 1·8°
			108°	108·3 ± 2·1°

[a] B. Bak, L. Hansen, and J. Rastrup-Anderson, *Discuss. Faraday Soc.*, 1955, **19**, 30; [b] D. E. Williams and R. E. Rundle, *J. Amer. Chem. Soc.*, 1964, **86**, 1660; [c] I. J. Grant, J. A. Hamilton, T. A. Hamor, J. M. Robertson, and G. A. Sim, *J. Chem. Soc.*, 1963, 2506.

Dihedral Angles.—The dihedral angle between the furan ring and the plane of the δ-lactone system of ring C is 60·5°. The furan ring is also tilted slightly upwards, that is, atom 13 is displaced towards O(21) and towards the plane of C(9)C(11)O(21). This is shown by the angle C(13)C(12)C(8) being 164° instead of the 180° that might be expected in a regular structure.

The dihedral angle between the pyrazoline ring E and the benzene ring F is 7·9°, while between ring E and the benzene ring G the dihedral angle is 28·7°. This larger rotation of the benzene ring G permits a greater clearance between C(30) and the hydroxyl group O(23) (3·08 Å) than would apply if the benzene ring were coplanar with the pyrazoline ring.

Molecular dimensions.—With a few exceptions the bond lengths and valency angles are close to the accepted standard values. The largest departures, which are probably due to the accumulation of random errors, are 1·581 Å for C(12)—O(21), 1·648 Å for C(5)—C(10), 1·478 Å for C(29)—C(30), and 1·464 Å for C(3)—C(4). The simple mean over the fifteen C(sp^3)—C(sp^3) single bonds is 1·558 Å. The mean of the twelve C–C bonds in the two benzene rings is 1·396 Å.

The value of 2·125 Å for C(32)—I(46) is longer than the value of 2·05 (Tables of Interatomic Distances [3]), although a value of 2·19 Å has been reported in 5-iodo-2-phthalimidobenzoic acid monohydrate.[4] The value of 1·437 Å for C(3)—N(28) is shorter, though not significantly, than the values of 1·472 ± 0·005 Å (Tables of Interatomic Distances [3]) and the very accurate value of 1·476 ± 0·002 Å obtained by Becka and Cruickshank.[5] The value of 1·455 Å for N(28)—C(29) is not significantly larger than 1·426 Å in acetanilide,[6] and 1·43 Å in 5-iodo-2-phthalimidobenzoic acid monohydrate.[4] The value of 1·451 Å for N(27)—N(28) compares well with the single bond N–N distance of 1·468 Å in pyrazoline hydrochloride,[7] 1·46 Å in hydrazine,[8] 1·46 and 1·447 Å in lithium hydrazinium sulphate [9,10] and 1·47 Å in byssochlamic acid bis-*p*-bromophenylhydrazide.[11] The value of 1·275 Å for C(26)—N(27) compares well with the normal C=N double bond length: 1·27 Å in 2-(2-hydroxyethylamino)-2-thiazoline,[12] 1·255 Å in pyrazoline hydrochloride,[7] 1·267 Å in 2-amino-7-methyl-5-propyl-*s*-triazolo[2,3-*c*]pyrimidine hydrochloride,[13] and a mean value of 1·269 Å in oximes and amine oxides.[14]

The dimensions found for the furan ring in this structure are compared with results for furan itself and two other structures in Table 6.

The bond lengths in the δ-lactone systems (IV) found in this structure are compared with other determinations in Table 7. The expected shortening of bond p as compared with q is found in all cases, but in ring A of this structure the difference is hardly significant, while in ring C the bond q is anomalously long.

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

[4] R. M. Mayer and M. R. A. Pratt, *Acta Cryst.*, 1963, **16**, 1086.

[5] L. N. Becka and D. W. J. Cruickshank, *Proc. Roy. Soc.*, 1963, *A*, **273**, 435.

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[13] P. G. Owston and J. M. Rowe, *Acta Cryst.*, 1962, **15**, 231.

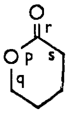
[14] K. Folting, W. N. Lipscomb, and B. Jerslev, *Acta Cryst.*, 1964, **17**, 1263.

The acetone solvate molecule is associated with a high thermal vibration. Its C–C distances of 1·505 and 1·514 Å and C=O distance of 1·224 Å do not differ significantly from the values found in acetone itself by electron diffraction[15] (1·55, 1·52, and 1·22, 1·23 Å). This solvate molecule appears to play an important part in the cohesive forces that bind the molecules together in the unit cell. Only van der Waals forces are operative, but it is significant that out of 72 intermolecular distances less than 4 Å (Table 5) no fewer than 29 involve the atoms of the acetone molecule.

TABLE 7

Bond lengths in δ-lactone systems

Compound	p	q	r	s
Dihydro-β-erythroidine hydrobromide[a]	1·33	1·43	1·21	1·54
Jacobine[b]	1·36	1·44	1·19	1·52
	1·39	1·48	1·20	1·51
Isoiridomyrmecin[c]	1·361	1·488	1·180	1·506
,, [d]	1·365	1·428	1·317	1·502
α-Bromoisotutin[e]	1·35	1·44	1·12	1·56
α-Bromoisotutinone[f]	1·37	1·46	1·20	1·48
Simarolide derivatives[g]	1·28	1·44	1·34	1·39
	1·41	1·63	1·26	1·43
Epilimonol iodoacetate[h]	1·31	1·44	1·29	1·48
	1·33	1·46	1·27	1·52
Swietenine[i]	1·41	1·52	1·18	1·45
This structure ring A	1·400	1·436	1·213	1·516
This structure ring C	1·332	1·581	1·192	1·483



(IV)

[a] A. W. Hanson, *Acta Cryst.*, 1963, **16**, 939; [b] A. McL. Mathieson and J. C. Taylor, *Acta Cryst.*, 1963, **16**, 524; [c] B. P. Schoenborn and J. F. McConnell, *Acta Cryst.*, 1962, **15**, 779; [d] J. F. McConnell, A. McL. Mathieson, and B. P. Schoenborn, *Acta Cryst.*, 1964, **17**, 472; [e] B. M. Craven, *Acta Cryst.*, 1964, **17**, 396; [f] M. F. Mackay and A. McL. Mathieson, *Acta Cryst.*, 1965, **19**, 417; [g] W. A. C. Brown, Ph.D. Thesis, University of Glasgow, 1964; [h] S. Arnott, A. W. Davie, J. M. Robertson, G. A. Sim, and D. G. Watson, *J. Chem. Soc.*, 1961, 4183; [i] A. T. McPhail and G. A. Sim, *J. Chem. Soc.* (*B*), 1966, 318.

A number of the shortest intermolecular contacts involve oxygen atoms. The carbonyl oxygen O(24) is 3·20 Å from atom C(44) and 3·38 Å from atom C(43) of the acetone molecule, while the carbonyl oxygen atom O(45) of the acetone molecule is 3·28 Å from atom C(10) and 3·31 Å from atom C(11). The carbonyl oxygen atom O(22) is 3·14 Å from C(2), 3·27 Å from C(26) and 3·33 Å from C(3) all on the neighbouring molecule at (−1 + x, y, z).

The only intermolecular distance less than 4 Å involving the iodine atom is a value of 3·58 Å for O(22) · · · $I(46)_{VII}$. This is just greater than the sum of the van der Waals radii for the oxygen and iodine atoms.

EXPERIMENTAL

Preparation of Adduct (III) *of Isocolumbin and Iododiphenylnitrilimine.*—N′-(p-*Iodophenyl*)*benzhydrazide*. *p*-Iodophenylhydrazine (m. p. 101—103°) was prepared in 65% yield by reduction with stannous chloride of *p*-iodoaniline according to the method of Bullock and Hand.[16]

To an ice-cooled solution of *p*-iodophenylhydrazine (1·5 g.) in dry ether (20 ml.) was added, dropwise and with stirring, redistilled benzoyl chloride (0·35 ml.) in dry ether (5 ml.). The precipitated solid was collected after 2 hr., and boiled with water to remove *p*-iodophenylhydrazine hydrochloride. The insoluble residue crystallised from ethanol to afford N′-(p-*iodophenyl*)*benzhydrazide* (853 mg.) m. p. 169—170° (Found: C, 46·35; H, 3·55; N, 8·41. $C_{13}H_{11}IN_2O$ requires C, 46·15; H, 3·25; N, 8·45%).

N-α-*Chlorobenzylidene*-N′-p-*iodophenylhydrazine*. *N′*-(*p*-Iodophenyl)benzhydrazide (3·62 g.) and phosphorus pentachloride (5·07 g.) were refluxed in dry ether (10 ml.) for 16 hr. Phenol (7·9 g.) in dry ether (10 ml.) was then run into this solution and, after cooling, methanol (72 ml.) added. The combined solvents were removed until the residue boiled at 60—70° at atmospheric pressure. The solid which separated on refrigeration was cystallised from ethanol affording the required *enol chloride* (1·87 g.), m. p. 112—113° (Found: C, 43·7; H, 3·25; N, 8·15. $C_{13}H_{10}ClIN_2$ requires C, 43·8; H, 2·8; N, 7·9%).

Adduct of isocolumbin and iododiphenylnitrilimine.[17] The reagent was prepared *in situ* in presence of isocolumbin as follows: Isocolumbin (100 mg.; m. p. 180—183°; $[\alpha]_D$ +75°), the above enol chloride (400 mg.), and redistilled triethylamine (1·5 ml.) were refluxed in dry AnalaR acetone (5 ml.) under nitrogen for 5 hr. The product, obtained in the usual way, was chromatographed over silica gel (B.D.H., 5 g.). Benzene–chloroform (7 : 3) eluted the *adduct* (III) (160 mg.) which slowly crystallised from acetone in flat prisms, m. p. softens with decomposition near 165° but does not completely melt below 340° (Found: C, 58·8; H, 4·75; N, 3·85. $C_{33}H_{31}O_6N_2I$ requires C, 58·5; H, 4·6; N, 4·15%).

Crystal Data.—1-*p*-Iodophenyl-3-phenylpyrazoline adduct of isocolumbin, monoacetone solvate, $C_{33}H_{31}O_6N_2I,(CH_3)_2CO$; M, 724·6, m. p. >340° (decomp.) Tetragonal, $a = b = 9{\cdot}68$, $c = 70{\cdot}79$ Å, $U = 6626$ Å^3, $D_m = \sim 1{\cdot}43$ g. cm.$^{-3}$, $Z = 8$, $D_c = 1{\cdot}452$ g. cm.$^{-3}$. Space group $P4_12_12 - D_4^4$ (or the enantiomorphous $P4_32_12 - D_4^8$). Absent spectra, $h00$ when h is odd, $0k0$ when k is odd, $00l$ when $l \neq 4n$. Absorption coefficient for X-rays ($\lambda = 1{\cdot}542$ Å) $\mu = 81{\cdot}6$ cm.$^{-1}$ Total number of electrons per unit cell = F(000) = 2960. Σf^2 (heavy atom)/Σf^2 (light atoms) = 1·5 at $\sin\theta = 0$.

Crystallographic Measurements.—Rotation, Weissenberg,

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[17] R. Huisgen, M. Seidel, G. Wallbillich, and H. Knupfer, *Tetrahedron*, 1962, **17**, 3.

TABLE 8

Measured and calculated structure factors

h	k	l	F_o	F_c	α
0	0	8	239.6	225.2	180
0	0	12	155.7	177.5	0
0	0	16	462.9	526.2	0
0	0	20	145.3	154.6	0
0	0	24	273.5	266.0	0
0	0	28	317.1	343.3	0
0	0	32	67.4	76.8	0
0	0	36	166.6	181.4	0
0	0	40	268.7	307.4	0
0	0	44	143.8	147.2	0
0	0	48	88.2	92.0	0
0	0	52	93.2	105.8	0
0	0	56	117.5	140.9	0
0	0	60	26.3	34.4	0
0	0	64	51.9	51.5	0
0	0	68	28.8	29.6	0
0	1	1	18.4	8.1	225
0	1	2	25.1	15.6	180
0	1	3	12.1	12.5	315
0	1	4	129.0	139.4	90
0	1	5	22.2	14.9	225
0	1	6	154.5	201.7	180
0	1	7	33.0	16.5	135
0	1	8	93.4	101.7	90
0	1	9	131.2	145.5	225
0	1	10	338.8	369.2	180
0	1	11	22.3	19.9	315
0	1	12	37.4	32.4	90
0	1	13	208.0	212.5	225
0	1	14	63.6	65.3	180
0	1	15	56.8	68.0	315
0	1	16	74.9	77.4	270
0	1	17	13.1	16.5	45
0	1	18	257.3	225.6	180
0	1	19	63.4	87.9	315
0	1	20	13.4	5.6	90
0	1	21	40.9	37.3	225
0	1	22	205.1	193.6	180
0	1	23	63.1	78.5	315
0	1	24	54.5	56.8	90
0	1	25	39.3	44.7	225
0	1	26	97.3	87.6	180
0	1	27	73.8	86.2	315
0	1	28	14.7	21.1	90
0	1	29	64.1	61.5	225
0	1	30	11.4	9.7	180
0	1	31	112.3	126.2	315
0	1	32	77.0	69.2	90
0	1	33	49.1	36.8	225
0	1	34	67.2	76.5	180
0	1	35	49.1	49.3	315
0	1	36	79.1	74.3	270
0	1	37	75.2	72.4	225
0	1	38	21.0	25.3	180
0	1	39	28.3	25.6	135
0	1	40	28.2	27.3	90
0	1	41	28.0	25.3	45
0	1	42	56.4	52.7	180
0	1	43	65.7	62.7	315
0	1	44	11.8	18.8	90
0	1	46	73.6	86.5	180
0	1	47	55.4	52.9	315
0	1	48	19.5	18.9	90
0	1	49	48.7	36.0	45
0	1	51	46.3	54.4	315
0	1	52	31.0	24.8	270
0	1	53	30.2	30.8	45
0	1	54	28.4	33.3	180
0	1	55	23.2	21.8	315
0	1	56	17.8	16.6	270
0	1	57	52.7	57.2	225
0	1	58	69.8	80.3	180
0	1	59	43.8	38.5	315
0	1	62	17.8	40.3	180
0	1	63	46.7	48.6	315
0	1	64	52.4	44.4	90
0	2	0	36.0	30.3	180
0	2	1	96.7	100.8	315
0	2	2	138.1	127.8	90
0	2	3	29.2	30.2	225
0	2	4	90.9	96.6	180
0	2	5	115.6	114.3	315
0	2	6	145.5	159.0	270
0	2	7	191.4	202.1	225
0	2	8	48.4	60.2	180
0	2	9	230.7	258.5	315
0	2	10	153.5	163.8	270
0	2	11	30.8	24.5	225
0	2	12	123.6	121.1	180
0	2	13	46.6	41.9	315
0	2	14	115.9	113.3	90
0	2	15	137.0	145.4	225
0	2	16	30.4	42.5	180
0	2	17	47.5	51.7	315
0	2	18	99.1	98.1	270
0	2	19	168.1	164.4	225
0	2	20	146.3	130.0	180
0	2	21	209.9	223.1	315
0	2	22	173.9	168.0	270
0	2	23	116.8	108.3	225
0	2	24	36.0	33.9	180
0	2	25	25.7	31.4	315
0	2	26	51.6	63.1	270
0	2	27	36.6	35.1	225
0	2	28	17.9	8.3	0
0	2	30	76.0	73.4	90
0	2	31	134.1	151.0	225
0	2	32	33.4	27.0	180
0	2	33	78.6	84.0	315
0	2	34	49.3	39.9	90
0	2	35	92.9	91.6	225
0	2	36	73.6	76.2	180
0	2	37	94.4	95.7	315
0	2	38	22.7	28.7	270
0	2	39	60.1	54.0	225
0	2	40	50.4	43.6	180
0	2	42	34.2	34.2	270
0	2	43	48.4	47.7	225
0	2	44	26.1	25.4	180
0	2	47	73.6	74.9	225
0	2	48	33.7	36.4	180
0	2	49	65.0	71.3	315
0	2	51	59.0	59.0	225
0	2	52	22.0	24.5	0
0	2	54	53.0	49.1	270
0	2	55	44.5	42.0	225
0	2	56	18.1	26.2	180
0	2	59	70.4	59.8	225
0	2	60	58.2	53.0	180
0	2	61	41.1	47.4	315
0	3	1	258.8	281.2	45
0	3	2	20.3	17.1	0
0	3	3	100.1	110.1	135
0	3	4	113.7	120.2	270
0	3	5	185.0	208.9	45
0	3	6	171.0	164.5	180
0	3	7	153.7	174.4	135
0	3	8	14.4	19.3	90
0	3	9	277.8	261.5	45
0	3	10	39.3	41.7	0
0	3	11	219.0	229.4	135
0	3	12	42.0	28.7	270
0	3	13	224.5	253.4	45
0	3	14	50.4	59.1	180
0	3	15	73.4	87.4	135
0	3	16	47.2	40.1	90
0	3	17	146.5	162.7	45
0	3	18	68.8	61.0	180
0	3	19	64.7	92.7	135
0	3	20	23.0	18.6	270
0	3	21	128.2	119.2	45
0	3	22	117.7	117.7	180
0	3	23	228.4	237.4	135
0	3	24	24.6	21.1	270
0	3	25	198.2	206.1	45
0	3	26	39.4	36.5	0
0	3	27	242.7	248.9	135
0	3	28	30.1	20.5	90
0	3	29	89.1	106.0	45
0	3	30	20.0	21.5	0
0	3	31	57.4	60.9	135
0	3	32	118.3	109.6	90
0	3	33	109.7	113.3	45
0	3	34	53.6	59.5	180
0	3	35	122.0	134.8	135
0	3	36	45.1	45.8	90
0	3	37	105.6	107.7	45
0	3	38	34.5	31.6	180
0	3	39	153.8	157.7	135
0	3	40	7.9	13.5	270
0	3	41	122.8	121.6	45
0	3	42	64.3	64.4	180
0	3	43	109.9	114.1	135
0	3	44	25.7	29.0	270
0	3	45	91.9	96.4	45
0	3	46	86.9	74.0	180
0	3	47	13.6	25.8	135
0	3	48	41.7	36.3	90
0	3	49	68.4	58.8	45
0	3	50	35.0	32.7	0
0	3	51	71.6	66.4	135
0	3	53	88.2	85.2	45
0	3	54	58.3	52.8	0
0	3	55	50.4	49.6	135
0	3	56	39.6	33.5	270
0	3	57	60.3	66.9	45
0	3	58	17.7	28.1	180
0	3	60	44.4	41.7	270
0	3	62	28.9	28.6	180
0	3	63	48.1	52.6	135
0	4	0	139.9	126.2	0
0	4	1	29.3	18.0	135
0	4	2	66.4	54.2	270
0	4	3	45.1	53.9	225
0	4	4	81.3	75.0	0
0	4	5	121.7	121.9	315
0	4	6	66.8	66.3	270
0	4	7	88.9	90.7	225
0	4	8	18.4	20.0	0
0	4	9	35.7	36.3	315
0	4	10	57.3	97.1	90
0	4	11	84.3	81.1	225
0	4	12	73.9	63.1	0
0	4	13	74.6	79.6	315
0	4	14	76.1	70.2	90
0	4	15	67.6	53.1	45
0	4	16	78.4	82.8	0
0	4	17	146.0	144.5	315
0	4	18	17.5	12.0	90
0	4	19	25.8	1.4	225
0	4	20	144.7	129.4	0
0	4	21	24.2	23.8	315
0	4	22	27.4	17.7	270
0	4	23	94.4	85.1	225
0	4	24	152.1	146.0	0
0	4	25	28.7	31.2	135
0	4	26	40.4	40.3	90
0	4	27	79.3	75.2	225
0	4	28	20.1	24.3	180
0	4	29	48.0	38.4	315
0	4	30	17.3	18.0	90
0	4	31	109.8	106.5	225
0	4	32	41.1	39.9	0
0	4	33	84.1	92.2	315
0	4	34	77.9	67.5	90
0	4	36	57.5	65.6	0
0	4	37	18.2	6.3	135
0	4	38	35.7	41.1	90
0	4	39	13.0	17.3	45
0	4	40	45.2	35.0	0
0	4	41	17.8	17.6	315
0	4	42	20.9	17.6	90
0	4	44	19.0	7.5	180
0	4	45	76.9	80.4	315
0	4	46	21.3	7.7	270
0	4	47	55.2	57.7	225
0	4	48	33.2	31.8	0
0	4	50	27.7	32.9	90
0	4	52	14.2	7.1	0
0	4	53	55.2	59.7	315
0	4	54	79.2	37.6	90
0	5	1	25.0	21.7	225
0	5	2	150.9	153.5	0
0	5	3	46.7	44.6	315
0	5	4	24.3	23.0	270
0	5	5	28.5	22.7	45
0	5	6	83.7	100.1	0
0	5	7	18.4	12.1	135
0	5	8	38.9	31.8	270
0	5	9	45.2	44.4	45
0	5	10	61.6	62.4	0
0	5	11	88.2	88.0	315
0	5	12	72.1	70.2	90
0	5	13	44.5	38.6	225
0	5	14	96.4	104.5	0
0	5	15	34.6	32.1	315
0	5	16	62.1	55.7	270
0	5	17	25.4	30.4	225
0	5	18	88.0	90.7	0
0	5	19	25.8	28.3	315
0	5	20	97.0	101.9	270
0	5	21	25.1	24.8	225
0	5	22	48.7	56.7	0
0	5	23	11.9	3.4	315
0	5	24	18.3	3.6	90
0	5	25	12.8	10.3	225
0	5	26	84.5	77.6	0
0	5	27	23.6	20.1	135
0	5	30	90.1	86.1	0
0	5	31	46.0	53.8	315
0	5	32	55.1	63.1	270
0	5	33	35.1	38.3	225
0	5	34	64.3	73.9	0
0	5	36	59.0	73.6	270
0	5	37	14.9	25.4	45
0	5	38	34.0	40.6	0
0	5	39	14.3	12.9	315
0	5	41	14.0	6.5	225
0	5	42	59.9	64.4	0
0	5	43	11.4	15.5	135
0	5	44	22.7	15.0	90
0	5	46	61.3	65.1	0
0	5	48	29.2	41.2	270
0	5	50	63.5	65.5	0
0	5	51	32.8	27.2	315
0	5	52	19.2	31.1	270
0	5	53	16.8	20.4	225
0	5	54	46.3	43.2	0
0	5	55	35.1	35.5	315
0	5	56	7.9	14.8	270
0	5	58	36.5	32.7	0
0	5	59	24.7	29.9	315
0	6	0	84.2	61.5	180
0	6	1	36.9	30.3	315
0	6	2	45.3	40.2	90
0	6	4	157.9	166.2	180
0	6	5	42.3	34.2	315
0	6	6	47.0	44.5	90
0	6	7	12.5	11.2	225
0	6	8	148.2	149.3	180
0	6	9	28.6	18.0	135
0	6	10	10.6	5.7	90
0	6	11	83.0	78.2	225
0	6	12	150.8	157.1	180
0	6	15	38.8	51.4	225
0	6	16	112.1	113.5	180
0	6	17	73.5	77.7	315
0	6	18	12.4	2.1	90
0	6	19	19.2	22.1	225
0	6	20	109.2	121.3	180
0	6	21	20.5	22.8	315
0	6	22	9.1	9.6	270
0	6	23	52.2	62.2	225
0	6	24	117.8	125.2	180
0	6	25	43.1	47.3	315
0	6	26	33.2	27.8	270
0	6	27	17.3	18.2	225
0	6	28	121.5	131.6	180
0	6	29	17.7	19.3	315
0	6	30	16.9	10.6	90
0	6	32	88.2	90.8	180
0	6	33	32.0	34.9	315
0	6	34	33.8	38.5	270
0	6	35	18.8	23.3	225
0	6	36	75.1	71.3	180
0	6	38	43.5	43.4	270
0	6	39	70.1	65.1	225
0	6	40	41.1	41.5	180
0	6	42	16.3	18.6	270
0	6	43	60.0	66.1	45
0	6	44	111.3	115.9	180
0	6	45	20.6	13.2	315
0	6	49	55.5	63.3	180
0	6	51	59.6	65.6	225
0	6	52	47.0	48.9	180
0	6	55	26.0	27.2	225
0	6	56	19.2	29.7	180
0	6	57	17.1	20.3	315
0	6	58	19.5	16.0	90
0	6	59	13.0	12.2	225
0	6	60	19.1	21.8	180
0	6	64	33.5	30.3	180
0	7	1	70.3	60.3	45
0	7	2	21.1	21.7	0
0	7	5	16.5	13.7	45
0	7	7	51.3	51.0	135
0	7	9	13.2	11.1	225
0	7	10	35.2	33.0	0
0	7	11	61.1	57.9	135
0	7	12	18.1	14.2	270
0	7	14	41.8	40.1	0
0	7	15	63.2	55.4	135
0	7	16	15.5	19.5	90
0	7	17	25.8	26.8	45
0	7	18	35.2	33.2	0
0	7	19	21.7	16.5	135
0	7	20	25.2	29.8	90
0	7	21	16.9	19.9	225
0	7	22	25.0	30.5	0
0	7	23	9.7	9.1	135
0	7	24	18.4	20.3	90
0	7	25	9.3	3.3	45
0	7	27	7.4	5.4	135
0	7	28	21.3	0.9	270
0	7	29	7.9	9.6	225
0	7	30	24.9	22.6	0
0	7	31	10.4	10.7	315
0	7	38	48.2	49.4	0
0	7	39	79.3	91.8	135
0	7	40	27.2	21.8	270
0	8	0	24.5	29.3	180
0	8	1	24.5	22.8	135
0	8	2	20.0	23.3	90
0	8	3	14.1	11.1	225
0	8	5	69.3	57.9	135
0	8	7	21.9	25.1	45
0	8	8	26.9	21.0	0
0	8	9	28.3	29.0	135
0	8	10	34.8	34.9	270
0	8	11	84.8	84.7	45
0	8	12	51.4	53.9	0
0	8	13	35.5	29.6	135
0	8	14	20.2	19.6	90
0	8	15	81.8	79.2	45
0	8	17	22.2	23.4	135
0	8	19	37.4	31.6	45
0	8	20	20.4	26.0	180
0	8	21	22.3	12.4	135
0	8	22	30.3	28.3	270
0	8	23	24.1	28.0	45
0	8	24	30.4	26.0	0
0	9	1	53.5	50.0	225
0	9	3	59.7	51.2	315
0	9	5	39.9	39.6	225
0	9	7	36.7	34.4	315
0	9	9	53.4	41.3	225
0	9	13	14.4	7.6	225
1	1	0	126.7	107.9	0
1	1	1	190.9	223.2	90
1	1	2	54.4	43.6	0
1	1	3	145.5	159.5	90
1	1	4	136.5	121.4	0
1	1	5	56.3	32.8	90
1	1	6	282.7	256.9	0
1	1	7	24.4	7.1	90
1	1	8	145.4	130.9	180
1	1	9	236.8	221.9	90
1	1	10	344.1	345.9	0
1	1	11	286.4	275.6	90
1	1	12	11.4	11.9	180
1	1	13	40.2	26.2	270
1	1	14	47.8	44.7	0
1	1	15	214.1	203.9	90
1	1	16	111.4	102.1	180
1	1	17	246.6	243.8	90
1	1	18	200.7	179.9	90
1	1	19	226.7	219.6	90
1	1	20	97.5	76.5	180
1	1	21	142.3	160.4	90
1	1	22	135.9	128.8	0
1	1	23	130.9	125.3	90
1	1	24	33.5	29.2	180
1	1	25	63.6	58.9	90
1	1	26	176.9	120.2	0
1	1	27	67.5	67.6	90
1	1	28	45.1	52.7	0
1	1	29	66.0	55.8	90
1	1	30	167.0	159.3	0
1	1	31	141.1	136.0	90
1	1	32	9.3	5.5	180
1	1	33	117.1	133.5	90
1	1	34	77.3	84.3	0
1	1	35	76.9	78.6	90
1	1	36	28.6	26.2	180
1	1	37	151.6	168.5	90
1	1	38	37.8	47.7	0
1	1	39	68.8	58.2	90
1	1	40	54.4	46.9	180
1	1	41	156.5	157.4	90
1	1	42	31.2	37.6	0
1	1	43	31.5	33.6	90
1	1	44	11.6	13.3	0
1	1	45	89.4	87.2	90
1	1	46	61.9	66.9	0
1	1	47	54.5	50.6	90
1	1	49	37.6	41.0	90
1	1	50	56.8	62.4	0
1	1	51	35.6	41.1	90
1	1	53	56.1	60.2	90
1	1	54	29.8	43.7	0
1	1	55	39.1	48.8	90
1	1	57	26.0	30.5	90
1	1	58	53.6	45.6	0
1	1	59	27.9	27.1	90
1	1	60	42.0	29.8	180
1	1	61	14.2	12.2	90
1	1	62	14.1	22.7	0
1	1	63	8.9	6.0	90
1	1	65	38.6	37.7	90
1	1	66	49.0	52.8	0
1	1	69	40.3	38.8	90
1	1	70	13.6	5.6	0
1	1	73	39.1	41.2	90
1	2	0	256.0	279.8	180
1	2	1	148.8	145.6	240
1	2	2	285.4	293.5	347
1	2	3	161.3	166.1	159
1	2	4	168.7	170.4	165
1	2	5	134.2	182.5	336
1	2	6	228.2	229.8	310
1	2	7	191.0	195.4	224
1	2	8	176.3	167.4	138
1	2	9	259.4	250.1	306
1	2	10	127.4	127.2	49
1	2	11	51.8	43.7	209
1	2	12	74.5	66.3	239
1	2	13	96.4	91.8	312
1	2	14	159.2	158.8	20
1	2	15	60.6	55.9	265
1	2	16	90.6	89.0	191
1	2	17	89.1	85.6	305
1	2	18	130.2	117.4	359
1	2	19	81.6	83.0	248
1	2	20	143.7	133.8	162
1	2	21	112.3	113.1	353
1	2	22	181.5	158.0	355
1	2	23	119.3	124.3	202
1	2	24	128.7	123.0	167
1	2	25	88.4	91.3	351
1	2	26	142.5	138.9	7
1	2	27	68.3	62.8	229
1	2	28	65.9	66.6	139
1	2	29	89.5	90.3	288
1	2	30	79.7	79.3	353
1	2	31	122.8	120.3	223
1	2	32	82.1	82.6	136
1	2	33	145.3	135.9	308
1	2	34	52.1	41.8	51
1	2	35	64.1	58.4	248
1	2	36	83.1	83.9	167
1	2	37	112.0	106.4	343
1	2	38	54.4	53.7	39
1	2	39	36.8	37.6	201
1	2	40	58.5	53.5	198
1	2	41	69.7	65.6	331
1	2	42	94.7	97.3	15
1	2	43	42.6	45.3	215
1	2	44	65.7	67.9	142
1	2	45	54.0	56.1	304
1	2	46	87.0	86.0	344
1	2	47	43.5	46.6	258
1	2	48	51.7	53.3	176
1	2	49	29.4	30.1	13
1	2	50	52.9	48.6	40
1	2	51	36.4	37.4	259
1	2	52	32.2	30.9	163
1	2	53	54.5	54.0	333
1	2	54	66.3	70.1	21
1	2	55	51.0	54.2	251
1	2	56	46.4	46.7	242
1	2	57	66.8	68.0	316
1	2	58	21.8	28.9	333
1	2	60	34.0	39.0	143
1	3	0	37.8	41.2	0
1	3	1	86.2	84.8	327
1	3	2	72.1	68.7	4
1	3	3	118.4	119.7	322
1	3	4	71.7	68.5	44
1	3	5	123.1	122.1	338
1	3	6	101.5	96.6	332
1	3	7	107.4	112.4	311
1	3	8	85.8	85.3	197
1	3	9	99.7	94.8	275
1	3	10	75.2	80.7	18
1	3	11	113.6	118.2	202
1	3	12	51.8	44.4	35
1	3	13	69.8	70.2	330
1	3	14	140.0	135.4	321
1	3	15	136.8	141.9	201
1	3	16	44.2	44.8	338
1	3	17	167.2	157.0	332
1	3	18	58.3	57.5	298
1	3	19	32.4	33.7	220
1	3	20	58.9	53.6	10
1	3	21	101.2	92.9	326
1	3	22	113.4	105.7	3
1	3	23	81.0	84.6	195
1	3	24	84.2	73.5	313
1	3	25	64.8	67.5	205
1	3	26	70.3	70.1	44
1	3	27	61.6	55.2	211
1	3	28	71.6	84.5	291
1	3	29	38.3	39.9	274
1	3	30	31.8	38.6	320
1	3	31	79.8	72.0	237
1	3	32	26.6	26.4	221
1	3	33	96.5	101.6	304
1	3	34	47.1	49.1	333
1	3	35	103.6	110.6	193
1	3	37	55.2	52.2	296
1	3	38	48.8	53.9	24
1	3	39	33.8	35.5	243
1	3	40	66.1	63.6	322
1	3	41	31.9	33.6	320
1	3	42	62.1	51.9	313
1	3	43	24.4	21.5	182
1	3	44	50.4	44.7	262
1	3	45	39.7	39.1	294
1	3	47	29.2	25.7	177
1	3	48	10.6	14.0	205
1	3	49	56.1	48.8	278
1	3	50	16.5	29.2	342
1	3	51	51.6	48.8	175
1	3	52	55.4	48.7	4
1	3	53	29.2	30.1	296
1	3	54	35.7	37.2	290
1	3	56	14.5	18.6	347
1	3	57	40.6	38.8	321
1	3	58	33.8	35.1	338
1	3	61	33.7	31.2	12
1	3	62	48.5	40.4	359
1	4	0	94.4	103.0	180
1	4	1	74.2	77.6	139
1	4	2	102.7	116.7	187
1	4	4	166.0	167.8	204
1	4	5	55.4	57.2	154
1	4	6	127.8	130.5	143
1	4	7	37.8	41.3	348
1	4	8	96.9	102.7	189
1	4	9	109.1	112.1	156
1	4	10	132.3	143.5	170
1	4	11	53.1	56.8	47
1	4	12	78.3	81.6	179
1	4	13	114.4	124.5	120
1	4	14	109.4	108.1	168
1	4	15	104.7	100.8	30
1	4	16	148.9	146.7	190
1	4	17	108.9	113.8	169
1	4	18	82.9	90.7	195
1	4	19	115.4	114.9	25
1	4	20	121.6	124.6	202
1	4	21	58.6	52.3	157
1	4	22	120.3	114.7	160
1	4	23	80.0	81.4	23
1	4	24	140.0	148.1	172
1	4	25	30.0	34.8	179
1	4	26	56.5	54.9	156
1	4	27	55.1	52.6	53
1	4	28	87.3	90.7	206
1	4	29	54.1	54.6	150
1	4	30	68.9	61.1	202
1	4	31	64.8	58.1	67
1	4	32	85.5	91.3	187
1	4	33	60.2	63.8	170
1	4	34	88.2	88.7	197
1	4	35	44.0	49.2	45
1	4	36	64.8	67.7	170
1	4	37	48.6	51.6	222
1	4	38	72.5	67.5	157
1	4	39	63.0	56.9	28
1	4	40	80.0	75.7	195
1	4	41	21.0	22.5	19
1	4	42	77.4	71.5	153
1	4	43	42.6	42.4	22
1	4	44	67.4	68.4	196
1	4	45	30.0	17.7	157
1	4	46	47.9	47.8	146
1	4	47	26.1	26.7	9
1	4	48	25.2	23.3	199
1	4	49	42.0	43.9	150
1	4	50	50.5	48.6	203
1	4	51	19.5	10.8	111
1	4	52	49.3	44.0	205
1	4	54	28.3	27.6	146
1	4	55	15.0	16.1	15
1	4	56	35.4	37.8	223
1	4	59	26.8	25.0	16
1	4	60	34.6	36.8	171
1	4	61	22.9	22.5	190
1	4	62	30.0	21.6	150
1	4	63	13.9	13.1	48
1	4	64	40.1	37.9	188
1	4	65	42.9	47.0	171
1	4	68	32.2	31.6	200
1	5	0	39.7	43.4	180
1	5	1	41.6	49.2	177
1	5	2	135.3	134.9	345
1	5	3	92.8	92.6	26
1	5	4	46.6	52.8	119
1	5	5	75.2	74.0	165
1	5	6	94.0	92.5	358
1	5	7	79.8	74.7	16
1	5	8	49.3	43.8	143
1	5	9	85.4	89.0	169
1	5	10	70.2	69.2	24
1	5	11	57.4	58.6	352
1	5	12	65.7	75.6	316
1	5	13	93.8	103.3	173
1	5	14	144.8	157.5	7
1	5	15	50.7	51.3	0
1	5	16	44.4	42.1	146
1	5	17	54.9	51.7	221
1	5	18	110.1	109.4	5
1	5	19	37.3	40.3	41
1	5	20	65.1	70.8	139
1	5	21	93.6	96.5	187
1	5	22	81.1	78.3	4
1	5	23	34.3	40.7	351
1	5	24	36.9	35.0	219
1	5	25	55.0	56.9	166
1	5	26	49.3	51.2	3
1	5	27	72.4	63.6	347
1	5	28	28.9	24.8	353
1	5	29	44.0	41.2	151
1	5	30	89.3	85.9	15
1	5	31	55.3	54.7	20
1	5	32	35.0	33.4	124
1	5	33	73.4	69.2	167
1	5	34	66.0	67.4	21
1	5	35	25.3	26.0	356
1	5	36	42.1	37.3	117
1	5	37	64.1	80.7	180
1	5	38	55.0	57.0	20
1	5	39	32.4	37.0	32
1	5	40	44.0	37.4	220
1	5	41	42.7	44.6	141
1	5	42	102.8	99.1	14
1	5	43	25.9	23.0	9
1	5	44	45.4	49.0	64
1	5	45	46.9	41.6	174
1	5	46	39.2	37.7	28
1	5	47	41.6	40.6	54
1	5	48	32.9	32.0	178
1	5	49	37.2	28.5	210
1	5	50	21.8	13.2	37
1	5	51	29.1	25.1	138
1	5	52	20.5	25.2	143
1	5	53	35.4	38.7	152
1	5	54	29.3	25.2	354
1	5	56	16.2	6.2	125
1	5	57	34.0	30.4	118
1	5	58	44.4	40.2	359
1	5	61	34.4	32.8	165
1	5	62	31.1	35.1	39
1	6	0	20.9	18.6	180
1	6	1	58.7	57.8	99
1	6	2	66.8	72.1	37
1	6	3	38.0	39.4	77
1	6	4	35.9	39.3	276
1	6	5	58.0	56.7	32
1	6	6	59.6	66.1	338
1	6	7	22.6	22.0	102
1	6	8	24.3	20.6	73
1	6	9	23.0	23.1	160
1	6	10	87.0	92.6	317
1	6	11	15.2	17.5	132
1	6	12	22.3	25.9	63
1	6	13	95.1	97.8	118
1	6	14	53.2	51.4	350
1	6	15	61.6	52.6	26
1	6	16	12.8	13.1	253
1	6	17	61.6	59.9	75
1	6	18	44.7	41.8	23
1	6	19	80.6	71.2	44
1	6	20	19.0	23.8	111
1	6	22	35.2	42.6	351
1	6	23	52.8	52.0	79
1	6	24	11.4	12.5	115
1	6	25	17.5	17.5	220
1	6	26	55.0	59.6	315
1	6	27	23.0	23.3	21
1	6	28	12.3	11.3	89
1	6	29	42.2	46.3	36
1	6	30	51.2	51.1	352
1	6	31	64.6	65.7	15
1	6	32	36.7	44.8	347
1	6	33	46.4	52.7	44
1	6	34	51.0	53.4	10
1	6	35	52.7	54.4	70
1	6	36	25.8	33.2	137
1	6	37	27.5	31.1	123
1	6	38	42.9	39.7	321
1	6	39	49.7	53.6	85
1	6	40	17.7	19.5	119
1	6	41	40.2	42.8	35
1	6	42	39.2	29.8	335
1	7	0	15.3	19.2	0
1	7	1	49.7	47.3	247
1	7	2	28.5	27.7	175
1	7	3	49.6	50.9	279
1	7	4	37.8	45.4	357
1	7	5	80.2	80.5	246
1	7	6	46.2	48.0	154
1	7	7	83.2	90.4	260
1	7	8	41.2	46.7	328
1	7	9	65.8	71.7	251
1	7	10	58.3	50.3	207
1	7	11	38.9	45.5	268
1	7	13	50.8	54.5	250
1	7	14	50.3	39.4	172
1	7	15	35.1	29.2	304
1	7	16	27.4	19.7	95
1	7	17	53.1	50.6	252
1	7	18	26.1	17.2	196
1	7	19	36.0	37.6	300
1	7	20	22.3	21.5	46
1	7	21	54.1	45.2	247
1	7	22	57.7	65.2	182
1	7	23	55.6	54.3	270
1	7	24	19.5	23.4	329
1	7	25	66.4	68.8	282
1	7	26	38.5	32.1	199
1	7	27	44.3	46.3	281
1	7	28	39.9	34.8	2
1	7	29	76.0	79.6	290
1	7	30	19.3	21.0	261
1	7	32	27.5	28.5	83
1	7	33	47.1	42.2	244
1	7	34	44.1	42.1	171
1	7	35	39.9	41.7	265
1	7	36	46.9	41.7	35
1	7	37	28.2	23.3	182
1	7	38	42.1	37.3	223
1	7	39	20.0	11.1	337
1	7	41	57.7	58.5	238
1	7	42	16.7	23.0	267
1	7	43	24.4	29.4	303
1	7	44	16.5	12.5	292
1	7	45	66.1	61.9	262
1	7	47	29.2	29.0	260
1	7	48	27.7	25.5	309
1	7	49	25.1	24.4	266
1	7	50	20.2	12.8	163
1	7	51	32.6	30.7	265
1	7	52	23.2	31.4	18
1	7	53	23.5	27.5	310
1	7	54	30.6	25.6	178
1	8	1	46.0	44.8	164
1	8	2	48.0	54.3	148
1	8	3	46.0	44.6	22
1	8	4	24.0	32.6	77
1	8	5	19.6	19.5	163
1	8	6	27.6	34.6	162
1	8	7	31.1	27.1	67
1	8	8	32.4	35.6	359
1	8	9	46.2	49.8	164
1	8	10	45.0	44.0	249
1	8	11	45.0	40.7	100
1	8	13	48.5	51.1	139
1	8	14	31.3	26.3	190
1	8	15	42.1	46.9	24
1	8	16	14.0	25.1	232
1	8	18	42.2	36.5	174
1	8	19	46.3	42.1	358
1	9	3	20.0	22.8	14
1	9	4	35.8	33.5	231
1	9	5	8.9	17.2	23
1	9	6	21.9	20.7	108
1	9	8	20.5	13.4	209
1	9	9	43.4	40.7	173
1	9	11	17.8	11.1	38
1	9	12	21.8	16.8	115
1	9	13	41.6	30.1	164
1	9	15	8.9	11.5	50
1	9	17	14.0	7.7	267
1	9	18	8.9	10.9	168
2	2	0	446.0	443.7	180
2	2	1	141.7	138.6	90
2	2	2	15.3	19.9	180
2	2	3	58.7	53.5	90
2	2	4	327.6	309.7	180
2	2	5	25.3	31.3	90
2	2	6	88.2	73.9	0
2	2	7	175.8	179.0	90
2	2	8	57.2	44.3	0
2	2	9	137.2	134.0	90
2	2	10	28.8	26.3	180
2	2	11	155.7	142.4	90
2	2	12	209.5	192.9	180
2	2	13	201.7	184.3	90
2	2	14	174.7	140.6	180
2	2	15	44.1	54.1	90
2	2	16	175.2	161.0	180
2	2	17	96.5	90.0	90
2	2	18	30.6	23.2	180
2	2	19	106.9	99.9	90
2	2	20	205.0	199.2	180
2	2	21	65.0	64.5	90
2	2	22	47.5	39.3	180
2	2	23	129.2	118.5	90
2	2	24	119.4	96.9	180
2	2	25	63.1	50.3	90
2	2	26	14.0	15.3	180
2	2	27	232.1	218.2	90
2	2	28	77.8	65.5	180
2	2	29	113.6	100.3	90
2	2	30	97.4	89.2	180
2	2	31	48.2	45.6	90
2	2	32	128.3	115.7	180
2	2	33	77.2	77.1	90
2	2	35	28.5	27.6	90
2	2	36	119.1	114.2	180
2	2	37	7.6	8.1	90
2	2	38	36.5	26.0	180
2	2	39	12.4	19.0	90
2	2	40	88.2	86.9	180
2	2	41	58.7	54.2	90
2	2	42	32.4	35.5	180
2	2	43	89.0	67.1	90
2	2	44	96.8	84.1	180
2	2	45	54.4	51.5	90
2	2	46	42.2	43.0	180
2	2	47	92.4	87.6	90
2	2	49	54.3	46.7	90
2	2	50	16.7	15.1	180
2	2	52	35.9	42.5	180
2	2	54	31.6	33.3	0
2	2	55	43.0	41.6	90
2	2	57	34.4	37.4	90
2	2	58	9.8	15.9	0
2	2	59	45.4	42.7	90
2	2	60	57.2	58.7	180
2	2	64	42.9	41.0	180
2	2	65	32.6	31.7	90
2	2	66	27.6	25.9	180
2	3	0	109.6	106.9	0
2	3	1	120.5	106.8	181
2	3	2	48.6	46.5	83
2	3	3	90.7	89.8	309
2	3	4	91.4	85.9	344
2	3	5	88.5	88.8	219
2	3	6	71.3	67.3	137
2	3	7	91.1	80.1	261
2	3	8	148.0	142.0	15
2	3	9	57.7	51.3	250
2	3	10	55.1	50.8	336
2	3	11	27.0	18.8	243
2	3	12	169.4	155.7	342
2	3	13	119.2	100.4	142
2	3	14	100.5	80.3	80
2	3	15	110.1	119.7	30
2	3	16	126.4	130.1	337
2	3	17	49.8	44.9	155
2	3	18	90.7	77.6	94
2	3	19	40.5	37.8	278
2	3	20	145.8	134.5	35
2	3	21	71.3	61.7	230
2	3	22	87.8	74.3	329
2	3	23	87.8	79.2	342
2	3	24	137.5	113.8	23
2	3	25	23.0	20.9	242
2	3	26	44.3	41.5	328
2	3	27	51.8	44.0	53
2	3	28	105.1	95.1	343
2	3	29	31.1	22.1	155
2	3	30	64.9	62.6	120
2	3	31	45.8	48.4	174
2	3	32	97.5	90.1	7
2	3	33	53.6	48.7	251
2	3	34	22.7	17.2	152
2	3	35	49.9	47.1	280
2	3	36	115.6	105.1	27
2	3	37	48.4	46.7	244
2	3	38	33.1	26.6	133
2	3	39	46.3	50.3	354
2	3	40	76.3	68.7	4
2	3	41	21.1	13.4	184
2	3	42	58.3	53.7	97
2	3	43	55.1	51.1	28
2	3	44	76.3	69.0	348
2	3	45	28.7	29.0	241
2	3	46	13.7	10.5	127
2	3	47	14.2	17.6	9
2	3	48	54.4	46.2	66
2	3	49	77.7	67.8	235
2	3	52	9.6	12.7	201
2	3	54	9.4	19.6	100
2	3	55	46.7	44.7	76
2	3	56	50.9	45.6	355
2	3	57	31.1	30.0	94
2	3	58	48.6	48.2	77
2	3	60	71.3	68.3	26
2	4	0	108.3	122.5	0
2	4	1	130.6	132.7	207
2	4	2	50.2	46.5	208
2	4	3	92.4	90.3	357
2	4	4	113.9	117.2	11
2	4	5	132.3	132.5	170
2	4	6	40.5	45.4	302
2	4	7	93.7	96.4	27
2	4	9	109.8	101.0	161
2	4	10	90.7	77.0	19
2	4	11	153.0	149.5	2
2	4	12	86.6	86.4	317
2	4	13	53.8	55.9	128
2	4	14	72.6	60.7	296
2	4	15	115.5	115.7	21
2	4	16	108.0	102.0	3
2	4	17	56.1	48.9	148
2	4	18	47.0	50.3	253
2	4	19	73.8	70.7	29
2	4	20	77.0	84.5	351
2	4	21	131.5	129.9	169
2	4	22	101.8	97.2	112
2	4	23	69.4	64.5	33
2	4	24	111.9	105.7	322
2	4	25	85.0	81.3	165
2	4	26	45.4	42.8	4
2	4	27	125.2	132.0	353
2	4	28	92.4	87.7	334
2	4	30	22.6	24.1	343
2	4	31	99.7	98.5	13
2	4	32	90.8	82.3	343
2	4	33	26.0	23.2	163
2	4	34	40.5	34.4	342
2	4	35	64.2	55.5	2
2	4	36	30.8	23.7	10
2	4	37	81.1	76.2	157
2	4	39	65.4	64.8	2
2	4	40	51.2	48.4	24
2	4	41	40.5	33.6	161
2	4	42	25.3	20.8	10
2	4	43	64.1	60.3	6
2	4	44	60.4	59.7	8
2	4	45	43.1	45.6	217
2	4	46	14.8	15.7	322
2	4	47	30.9	18.8	6
2	4	48	53.3	49.4	339
2	4	49	49.4	49.5	199
2	4	51	67.0	72.5	24
2	4	52	29.7	33.8	307
2	4	53	25.4	29.0	83
2	4	54	31.1	36.5	242
2	4	55	62.3	58.0	8
2	4	56	62.3	64.7	339
2	4	57	15.1	7.8	100
2	4	58	9.9	14.0	327
2	5	0	42.2	49.0	180
2	5	1	59.5	65.7	262
2	5	2	80.0	83.6	160
2	5	3	77.9	93.2	293
2	5	4	62.3	63.1	184
2	5	5	82.4	88.9	249
2	5	6	20.3	15.7	94
2	5	7	79.1	85.0	265
2	5	8	36.0	37.6	181
2	5	9	73.6	70.9	254
2	5	10	73.3	76.4	152
2	5	11	71.9	78.2	226
2	5	12	54.2	59.2	175
2	5	13	116.0	123.4	241
2	5	14	100.7	111.4	182
2	5	15	65.2	58.7	277
2	5	16	68.2	80.1	157
2	5	17	38.5	39.4	165
2	5	18	51.1	51.3	271
2	5	19	74.6	69.6	295
2	5	20	96.4	101.2	198
2	5	21	69.6	69.6	245
2	5	22	52.4	49.6	173
2	5	23	66.5	69.0	270
2	5	24	64.5	70.4	177
2	5	25	72.9	76.5	245
2	5	26	49.2	49.8	210
2	5	27	82.0	83.8	324
2	5	28	10.4	12.3	197
2	5	29	46.7	46.4	252
2	5	30	24.7	27.3	210
2	5	31	68.1	61.7	315
2	5	32	57.4	53.1	186
2	5	33	64.7	58.2	267
2	5	34	48.8	41.6	187
2	5	35	38.3	38.7	271
2	5	36	39.1	34.4	157
2	5	37	60.9	61.7	204
2	5	38	41.5	52.8	143
2	5	39	58.4	56.5	284
2	5	40	50.7	53.2	160
2	5	41	27.9	30.9	223
2	5	42	18.1	23.6	246
2	5	43	59.6	60.3	287
2	5	44	37.6	36.3	225
2	5	45	19.0	11.0	299
2	5	46	30.3	29.7	264
2	5	47	45.4	44.3	270
2	5	48	34.0	32.5	211
2	5	49	62.7	65.0	268
2	5	50	56.8	52.7	177
2	5	51	55.8	58.4	288
2	5	52	23.4	21.8	168
2	5	53	51.9	50.2	236
2	5	54	37.1	35.7	197
2	5	55	31.1	29.6	268
2	5	56	30.3	29.3	97
2	6	0	37.9	44.4	0
2	6	1	34.1	26.8	16
2	6	2	41.7	41.4	195
2	6	3	66.5	65.9	164
2	6	4	68.1	57.3	36
2	6	5	39.2	42.0	0
2	6	6	11.0	7.6	304
2	6	7	79.1	71.1	119
2	6	8	37.1	33.3	27
2	6	9	50.0	49.2	97
2	6	10	14.9	17.8	196
2	6	11	81.5	79.5	163
2	6	12	37.4	33.4	297
2	6	13	56.4	46.1	80
2	6	14	57.7	54.3	217
2	6	15	70.8	65.0	142
2	6	16	34.7	27.7	80
2	6	17	24.3	13.2	51
2	6	18	22.7	20.4	185
2	6	19	64.9	64.2	167
2	6	20	10.5	14.9	126
2	6	21	20.2	22.2	59
2	6	22	19.5	23.1	148
2	6	23	32.4	29.7	162
2	6	24	18.3	20.2	335
2	6	25	42.9	41.9	30
2	6	26	8.9	8.7	125
2	6	27	25.3	24.6	161
2	6	28	34.3	26.3	28
2	6	29	24.3	21.2	64
2	6	30	38.2	39.1	233
2	6	31	58.8	60.8	111
2	6	32	66.1	68.3	21
2	6	33	35.3	30.6	83
2	6	35	61.5	56.6	139
2	6	37	42.6	31.2	90
2	6	39	38.7	41.0	207
2	6	40	16.4	11.5	10
2	6	41	29.8	23.5	99
2	6	42	38.3	34.0	223
2	6	43	26.7	20.2	130
2	6	44	26.8	28.2	340
2	6	46	19.2	13.4	212
2	6	47	32.9	32.4	123
2	7	0	55.7	58.3	0
2	7	1	40.9	39.1	40
2	7	2	66.3	68.0	166
2	7	3	17.9	13.7	74
2	7	4	46.7	48.3	22
2	7	5	28.9	21.3	15
2	7	6	42.7	40.2	162
2	7	7	45.7	49.7	120
2	7	8	37.6	34.0	19
2	7	9	36.6	36.9	102
2	7	10	56.7	56.3	240
2	7	11	51.0	59.6	130
2	7	12	27.8	34.2	326
2	7	13	61.7	51.3	61
2	7	14	61.6	57.8	203
2	7	15	29.2	30.0	29
2	7	17	61.6	57.1	37
2	7	18	118.1	114.3	167
2	7	19	18.7	19.9	82
2	7	20	32.4	21.0	54
2	7	21	16.5	19.5	333
2	7	22	55.7	54.8	163
2	7	23	31.8	34.2	110
2	7	24	42.5	40.6	18
2	7	25	19.2	18.4	159
2	7	26	19.3	18.7	163
2	7	27	13.0	26.2	354
2	7	28	23.5	26.5	359
2	7	29	66.5	61.9	41
2	7	30	59.5	59.2	153
2	7	31	17.3	21.5	28
2	7	32	34.8	35.7	48
2	7	33	42.2	38.7	43
2	7	34	34.7	33.2	154
2	7	35	57.2	59.0	105
2	7	36	15.9	12.2	355
2	7	37	21.4	20.4	331
2	7	38	51.5	48.2	121
2	7	39	22.5	22.0	126
2	7	40	14.6	7.7	345
2	7	41	32.4	27.5	44
2	7	42	45.6	48.6	172
2	7	43	14.6	21.8	44
2	7	44	39.6	36.8	20
2	7	45	32.7	34.8	45
2	7	46	35.2	40.7	177
2	7	47	26.9	24.9	106
2	7	48	26.2	23.0	42
2	7	49	26.6	29.3	91
2	7	50	27.3	30.5	165
2	7	51	10.4	9.2	102
2	7	52	9.2	13.7	90
2	7	53	16.0	19.4	48
2	7	54	18.8	21.0	163
2	8	1	43.5	45.2	288
2	8	2	26.7	30.9	10
2	8	3	53.3	53.4	287
2	8	4	40.7	41.1	13
2	8	8	77.2	73.7	4
2	8	9	21.9	29.8	205
2	8	10	23.9	24.8	32
2	8	11	15.4	28.4	199
2	8	12	21.9	25.2	330
2	8	14	38.0	30.3	28
2	8	15	22.0	23.2	338
2	8	16	30.8	36.9	344
2	8	17	9.8	12.9	14
2	8	19	56.2	46.6	328
2	8	20	39.5	37.3	8
2	8	23	58.4	54.1	239
2	8	24	57.6	56.7	353
2	8	25	35.0	40.4	278
2	8	26	44.2	43.8	3
2	8	27	15.6	9.8	151
2	8	28	34.9	33.1	15
2	8	29	22.1	17.5	320
2	8	30	28.7	27.6	37
2	8	31	38.1	34.9	293
2	8	32	45.0	42.7	338
2	8	33	9.8	11.5	147
2	8	34	21.1	26.6	56
2	8	35	23.8	21.6	307
2	8	36	21.7	15.7	8
2	8	38	21.5	18.0	10
2	8	40	45.0	37.9	12
2	8	42	14.7	17.2	7
2	8	43	18.5	17.3	306
2	8	44	34.4	32.6	342
2	8	45	14.3	18.1	291
2	8	46	20.0	25.7	37
2	8	47	19.8	20.3	299
3	3	0	41.6	41.9	180
3	3	1	90.8	76.5	270
3	3	2	188.1	218.0	180
3	3	3	151.2	145.0	270
3	3	4	34.7	32.1	0
3	3	5	48.7	49.1	270
3	3	6	163.2	178.8	180
3	3	7	130.5	134.3	270
3	3	8	36.6	35.3	180
3	3	9	51.9	50.5	90
3	3	10	236.0	259.6	180
3	3	11	51.9	62.1	90
3	3	12	12.7	17.9	0
3	3	13	27.9	22.6	270
3	3	14	202.0	197.6	180
3	3	15	26.7	26.4	90
3	3	16	59.3	55.1	180
3	3	17	112.9	99.4	90
3	3	18	275.0	262.2	180
3	3	19	68.0	65.4	270
3	3	20	44.7	36.6	0
3	3	21	97.8	78.4	270
3	3	22	200.7	206.6	180
3	3	23	29.5	24.4	270
3	3	24	15.0	15.2	180
3	3	25	29.5	31.3	90
3	3	26	194.1	189.6	180
3	3	27	15.6	19.4	90
3	3	28	65.8	61.3	0
3	3	30	157.0	148.3	180
3	3	31	45.1	48.7	270
3	3	32	23.5	22.1	180
3	3	33	35.0	36.7	270
3	3	34	143.0	131.5	180
3	3	35	72.5	62.1	270
3	3	36	23.7	22.9	180
3	3	37	66.7	61.2	270
3	3	38	98.6	104.8	180
3	3	39	57.2	52.5	90
3	3	40	48.3	45.0	180
3	3	41	73.3	67.3	270
3	3	42	87.0	84.3	180
3	3	45	30.3	31.9	270
3	3	46	122.5	124.1	180
3	3	47	91.9	78.2	270
3	3	49	18.5	11.9	270
3	3	50	73.8	69.3	180
3	3	51	36.4	35.3	270
3	3	52	39.5	31.5	0
3	3	54	65.1	57.3	180
3	3	56	26.8	25.4	180
3	3	57	27.8	26.5	270
3	3	58	48.8	48.2	180
3	3	60	21.4	17.1	0
3	4	0	34.9	38.4	0
3	4	1	37.2	41.0	250
3	4	2	52.9	56.0	138
3	4	3	29.0	30.8	30
3	4	4	103.6	103.1	348
3	4	5	53.8	55.1	98
3	4	6	71.4	79.7	91
3	4	7	35.9	38.4	28
3	4	8	105.4	108.4	350
3	4	9	120.2	124.8	120
3	4	10	40.5	31.3	65
3	4	11	78.0	85.0	104
3	4	12	65.5	57.9	8
3	4	13	65.4	53.1	103
3	4	14	37.9	20.8	57
3	4	15	35.5	29.2	110
3	4	16	27.2	18.0	15
3	4	17	23.8	18.7	173
3	4	18	25.5	20.2	195
3	4	19	39.9	29.7	117
3	4	20	70.5	71.1	341
3	4	21	94.1	87.0	149
3	4	22	30.5	25.2	279
3	4	23	22.0	24.2	112
3	4	24	27.8	22.8	61
3	4	25	56.0	51.2	127
3	4	26	34.9	25.5	43
3	4	27	16.3	13.4	73
3	4	28	35.7	31.0	5
3	4	29	35.3	30.0	112
3	4	30	20.9	21.3	259
3	4	31	37.5	23.6	79
3	4	32	38.2	29.3	23
3	4	33	59.7	54.7	132
3	4	34	20.3	20.4	35
3	4	35	26.0	20.6	254
3	4	36	58.4	50.9	29
3	4	37	97.3	86.9	138
3	4	38	16.3	22.5	136
3	4	39	40.5	25.4	148
3	4	40	67.1	67.6	350
3	4	41	49.9	41.1	157
3	4	42	14.4	11.9	87
3	4	43	37.2	23.7	135
3	4	44	36.4	35.3	8
3	4	45	46.3	44.3	71
3	4	46	20.8	24.0	267
3	4	47	22.7	27.0	66
3	4	48	9.3	17.7	339
3	4	49	45.7	41.4	152
3	4	50	28.3	20.7	350
3	4	51	28.6	32.7	131
3	4	52	21.4	27.4	50
3	4	53	39.5	31.7	137
3	5	0	16.2	20.3	180
3	5	1	102.7	114.3	11
3	5	2	10.9	9.4	280
3	5	4	13.0	12.2	355
3	5	5	32.9	33.7	36
3	5	6	47.3	50.5	297
3	5	7	63.1	65.7	102
3	5	8	13.7	14.7	239
3	5	9	66.2	60.8	39
3	5	10	60.2	61.0	2
3	5	11	66.5	71.9	107
3	5	12	56.9	56.4	155
3	5	13	97.8	100.8	55
3	5	14	44.4	38.1	18
3	5	15	41.9	37.3	123
3	5	16	31.8	28.9	181
3	5	17	53.1	48.3	67
3	5	18	20.3	19.5	106
3	5	19	25.0	32.0	148
3	5	20	39.8	37.9	7
3	5	21	66.3	53.4	3
3	5	22	44.4	26.5	297
3	5	23	41.9	44.3	116
3	5	24	15.6	18.5	266
3	5	25	83.2	74.6	43
3	5	26	24.3	20.8	320
3	5	27	29.1	23.4	103
3	5	28	37.4	33.8	152
3	5	29	86.4	72.6	29
3	5	30	43.9	33.0	50
3	5	31	21.0	13.6	37
3	5	32	20.3	12.5	60
3	5	33	71.4	62.7	38
3	5	34	26.4	24.9	29
3	5	35	46.7	35.7	82
3	5	36	19.7	24.7	124
3	5	37	92.3	81.0	33
3	5	38	20.2	21.0	325
3	5	39	15.5	21.0	105
3	5	40	41.9	26.3	131
3	5	41	90.7	80.3	34
3	5	42	20.4	23.8	8
3	5	43	28.3	20.6	41
3	5	44	25.3	22.2	92
3	5	45	44.5	37.2	45
3	5	46	17.2	13.6	315
3	5	47	41.8	34.3	33
3	5	49	22.8	19.5	91
3	5	50	9.6	7.1	276
3	5	51	38.4	31.3	189
3	5	52	38.0	27.4	148
3	5	53	15.3	30.4	71
3	5	54	15.4	18.8	96
3	6	0	49.5	45.7	0
3	6	1	90.8	85.8	310
3	6	2	57.0	57.2	357
3	6	3	101.3	93.6	247
3	6	4	60.9	60.5	53
3	6	5	86.1	81.6	311
3	6	6	26.4	26.7	360
3	6	7	81.3	77.3	217
3	6	8	35.3	36.9	290
3	6	9	85.3	80.1	316
3	6	10	51.2	49.9	290
3	6	11	97.7	95.6	212
3	6	12	64.8	58.3	339
3	6	13	95.3	90.8	301
3	6	14	21.1	17.1	214
3	6	15	50.4	52.8	209
3	6	16	39.9	33.3	44
3	6	17	89.4	89.7	349
3	6	18	14.6	16.0	30
3	6	19	100.6	97.7	216
3	6	20	32.1	33.1	57
3	6	21	68.9	66.0	333
3	6	22	42.0	39.1	337
3	6	23	106.1	98.7	224
3	6	24	53.4	57.9	346
3	6	25	64.3	57.6	286
3	6	26	14.0	13.3	273
3	6	27	58.6	53.3	232
3	6	28	49.7	52.7	30
3	6	29	79.8	80.5	312
3	6	30	42.9	49.1	71
3	6	31	59.3	60.5	262
3	6	32	20.8	17.9	11
3	6	33	65.7	68.1	321
3	6	34	18.0	19.6	29
3	6	35	58.4	42.9	217
3	6	36	14.0	18.0	345
3	6	37	29.1	33.2	338
3	6	38	41.5	43.1	310
3	6	39	25.2	23.7	208
3	6	40	45.4	39.8	1
3	6	41	26.7	25.6	310
3	6	42	17.1	15.0	116
3	6	43	56.7	49.8	260
3	6	45	47.9	42.5	340
3	6	46	14.4	10.0	5
3	6	47	56.6	54.8	217
3	6	49	37.2	33.4	305
3	6	50	33.5	30.5	340
3	6	51	26.5	27.4	234
3	6	52	13.6	15.3	322
3	6	53	48.7	42.7	274
3	6	55	30.9	28.4	228
3	6	56	18.3	18.5	348
3	6	57	30.8	36.1	275
3	7	1	17.7	19.2	21
3	7	2	44.3	42.9	169
3	7	3	11.4	10.4	80
3	7	4	19.7	17.9	108
3	7	5	23.5	24.1	92
3	7	6	14.0	20.9	162
3	7	7	21.0	22.0	178
3	7	8	33.1	30.9	188
3	7	9	18.2	24.2	132
3	7	10	20.7	28.2	210
3	7	11	40.8	31.8	105
3	7	12	37.6	27.1	285
3	7	13	42.4	45.8	39
3	7	14	30.9	35.8	181
3	7	15	76.7	69.5	87
3	7	16	32.4	33.7	253
3	7	17	32.1	26.7	13
3	7	18	49.5	42.3	228
3	7	19	28.5	25.5	217
3	8	1	42.7	46.5	202
3	8	2	15.1	17.3	31
3	8	4	21.3	20.2	159
3	8	6	15.1	21.4	115
3	8	7	26.2	10.4	231
3	8	8	71.0	58.7	167
3	8	10	15.1	17.6	212
3	8	11	30.3	26.5	351
3	8	12	84.0	79.7	178
3	8	13	15.1	26.1	116
3	8	15	26.3	31.5	80
4	4	0	10.4	20.4	180
4	4	1	58.2	67.8	270
4	4	2	44.1	49.2	0
4	4	3	123.2	137.2	270
4	4	4	33.0	34.2	180
4	4	5	57.1	56.1	270
4	4	6	27.8	23.9	180
4	4	7	129.4	152.9	270
4	4	8	67.1	80.4	180
4	4	9	70.5	67.2	270
4	4	10	37.8	39.5	0
4	4	11	79.1	81.8	270
4	4	12	65.9	67.2	180
4	4	13	87.0	87.6	270
4	4	14	58.6	56.4	0
4	4	15	47.9	39.4	270
4	4	16	22.5	28.2	180
4	4	17	83.6	85.6	270
4	4	18	89.6	88.7	0
4	4	19	78.2	80.4	270
4	4	20	73.0	75.9	180
4	4	21	23.6	25.3	270
4	4	22	16.9	15.7	0
4	4	23	57.3	80.6	270
4	4	25	69.3	64.4	270
4	4	26	19.3	16.2	180
4	4	27	107.7	98.2	270
4	4	28	25.4	22.1	180
4	4	29	88.8	87.2	270
4	4	30	12.3	16.2	0
4	4	31	133.4	108.2	270
4	4	32	59.6	52.1	180
4	4	33	66.5	63.1	270
4	4	34	19.1	15.3	0
4	4	35	45.4	41.3	270
4	4	36	70.5	69.4	180
4	4	37	48.4	42.1	270
4	4	38	55.7	54.8	0
4	4	39	22.0	23.3	90
4	4	40	47.6	46.0	180
4	4	41	50.1	46.2	270
4	4	42	74.4	66.2	0
4	4	43	58.7	54.7	270
4	4	45	21.0	20.4	270
4	4	46	31.2	30.3	0
4	4	47	113.5	104.0	270
4	4	48	28.4	27.6	180
4	4	50	45.3	42.6	180
4	4	51	59.7	49.2	270
4	4	53	35.7	44.1	270
4	4	55	60.2	53.2	270
4	4	57	47.2	42.6	270
4	4	59	35.1	29.4	270
4	5	0	73.1	59.9	180
4	5	1	54.6	53.2	45
4	5	2	28.3	32.3	14
4	5	3	61.7	65.6	145
4	5	4	62.6	70.6	187
4	5	5	37.3	38.7	30
4	5	6	30.2	27.6	351
4	5	7	59.8	62.9	99
4	5	8	61.8	66.6	195
4	5	9	21.9	21.6	27
4	5	10	13.3	15.3	15
4	5	11	75.2	63.3	132
4	5	12	62.7	76.6	195
4	5	13	24.5	23.5	55
4	5	14	21.5	18.8	295
4	5	15	63.2	63.4	148
4	5	16	49.7	47.9	123
4	5	17	51.2	53.5	52
4	5	18	68.1	77.9	328
4	5	19	60.6	56.9	166
4	5	20	65.4	65.2	204
4	5	21	37.2	34.9	69
4	5	22	59.8	60.1	6
4	5	23	67.2	60.1	161
4	5	24	45.6	41.0	208
4	5	25	10.6	9.8	43
4	5	26	33.7	35.2	18
4	5	27	58.1	48.8	172
4	5	28	93.3	95.5	183
4	5	30	11.2	14.7	343
4	5	31	40.3	35.5	121
4	5	32	67.2	69.0	167
4	5	33	55.0	41.7	113
4	5	34	49.9	47.3	287
4	5	35	35.9	25.0	77
4	5	36	17.5	23.9	196
4	5	37	79.5	64.8	95
4	5	38	28.6	27.8	305
4	5	39	39.3	40.0	136
4	5	40	37.4	26.8	264
4	5	41	34.9	19.8	56
4	5	43	59.6	62.2	177
4	5	44	41.3	41.6	193
4	6	1	46.1	49.3	50
4	6	3	35.7	30.0	140
4	6	4	57.1	60.2	168
4	6	5	37.5	35.0	2
4	6	6	38.6	43.0	164
4	6	7	22.4	21.1	60
4	6	8	29.2	34.2	201
4	6	9	35.0	32.7	10
4	6	11	24.9	21.9	135
4	6	12	46.9	47.9	197
4	6	13	32.3	38.7	51
4	6	14	27.7	32.4	241
4	6	15	17.0	21.6	131
4	6	16	24.6	25.2	211
4	6	17	28.7	31.4	94
4	6	18	21.7	14.9	172
4	6	19	28.0	26.1	48
4	6	20	25.6	23.1	180
4	6	21	29.7	27.7	227
4	6	22	36.7	32.5	115
4	6	23	19.0	14.3	63
4	6	24	54.4	50.2	91
4	6	25	22.8	29.6	331
4	6	26	29.2	24.3	146
4	6	27	20.6	27.3	200
4	6	28	61.4	58.8	119
4	6	29	29.0	26.2	52
4	6	30	49.9	35.9	143
4	6	31	37.7	38.9	97
4	6	32	28.6	27.4	163
4	6	33	30.3	32.9	41
4	6	34	36.0	32.8	194
4	6	35	23.2	20.1	100
4	6	36	24.6	25.0	193
4	6	37	33.3	31.1	343
4	6	38	14.8	17.2	87
4	6	39	20.1	21.0	120
4	6	43	30.2	19.0	103
4	6	44	46.8	43.4	136
4	7	0	28.2	26.0	0
4	7	1	31.1	35.5	187
4	7	2	65.8	69.7	355
4	7	3	36.9	43.9	333
4	7	4	16.3	16.8	35
4	7	5	50.8	45.9	201
4	7	6	49.2	57.9	338
4	7	7	24.4	25.0	294
4	7	8	42.2	41.5	14
4	7	9	35.9	36.4	168
4	7	10	31.1	35.5	45
4	7	11	31.0	31.8	32
4	7	12	48.1	47.9	11
4	7	13	17.4	13.4	132
4	7	14	39.1	34.4	357
4	7	15	14.6	18.2	60
4	7	16	53.7	55.2	357
4	7	17	24.0	19.3	65
4	7	18	33.6	36.4	19
4	7	19	38.7	40.8	20
4	7	20	53.7	59.0	357
4	7	21	23.4	29.0	205
4	7	22	39.0	44.8	15
4	7	23	23.3	19.1	286
4	7	24	32.1	32.5	26
4	7	25	30.6	25.0	244
4	7	26	47.4	43.5	1
4	7	28	47.5	43.3	351
4	7	29	28.8	34.9	172
4	7	30	49.2	43.8	36
4	7	32	34.7	27.7	351
4	7	34	41.1	43.0	359
4	7	35	27.2	28.7	14
4	7	36	37.1	23.6	310
4	7	37	15.3	18.8	182
4	7	38	26.2	25.2	318
4	7	39	32.6	32.6	46
4	7	40	29.8	29.4	1
4	7	42	27.9	25.7	3
4	7	43	25.2	22.0	13
4	7	44	29.1	24.9	1
4	7	46	39.3	33.3	358
4	8	0	37.0	35.9	180
4	8	1	21.4	16.7	163
4	8	2	21.4	17.5	67
4	8	3	23.4	11.1	129
4	8	4	30.2	34.8	221
4	8	5	21.4	22.8	182
4	8	6	15.1	5.3	264
4	8	7	52.4	54.0	325
4	8	8	50.4	35.9	283
4	8	9	23.4	28.8	211
4	8	10	19.1	10.5	295
4	8	11	23.4	31.5	331
4	8	12	41.2	42.3	161
4	8	13	30.2	25.0	180
4	8	14	28.7	34.4	90
4	8	15	21.3	13.5	330
4	8	16	21.3	24.5	155
4	8	17	19.1	4.9	254
4	8	18	15.1	22.3	39
4	8	19	30.1	24.1	358
4	8	20	21.2	27.4	190
4	8	22	49.8	46.3	288
4	8	23	45.0	35.5	339
4	8	24	37.9	45.6	211
4	8	25	14.9	12.5	177
4	8	27	18.7	15.5	2
4	8	28	42.9	40.1	223
4	8	29	28.0	26.7	269
4	8	31	27.8	26.5	307
4	8	32	20.5	19.7	131
5	5	0	16.0	13.6	180
5	5	1	62.2	52.9	270
5	5	2	55.7	68.3	0
5	5	3	14.7	13.5	270
5	5	4	47.7	56.3	0
5	5	5	50.2	49.1	270
5	5	6	48.3	52.2	0
5	5	8	56.4	56.5	0
5	5	9	14.9	15.6	270
5	5	10	63.6	66.8	0
5	5	11	93.9	105.5	270
5	5	13	30.3	40.9	270
5	5	14	95.3	115.5	0
5	5	15	36.0	41.0	270
5	5	16	15.4	17.6	180
5	5	17	36.3	39.7	270
5	5	18	35.6	41.3	0
5	5	19	30.2	26.7	90
5	5	20	86.1	97.7	0
5	5	21	42.1	47.2	270
5	5	22	39.2	45.5	0
5	5	23	32.2	29.6	270
5	5	24	57.5	52.2	0
5	5	25	46.1	48.1	270
5	5	26	73.1	73.6	0
5	5	27	38.1	37.2	270
5	5	28	1.8	6.5	0
5	5	29	42.5	41.8	270
5	5	30	38.3	38.3	0
5	5	31	11.9	16.1	90
5	5	32	18.6	15.5	0
5	5	34	23.0	27.9	0
5	5	36	40.6	40.5	0
5	5	38	45.1	45.8	0
5	5	39	12.4	15.5	270
5	5	32	18.6	18.5	0
5	5	34	23.0	27.9	0
5	5	36	40.6	42.5	0
5	5	38	46.1	45.8	0
5	5	41	39.2	34.1	270
5	5	42	47.1	45.6	0
5	5	44	30.4	18.7	0
5	5	46	30.3	35.1	0
5	5	47	23.4	22.2	270
5	5	48	57.6	49.1	0
5	5	49	39.4	33.6	270
5	5	50	36.4	31.9	0
5	5	51	31.9	21.7	270
5	5	52	34.7	33.2	0
5	5	53	16.7	21.3	270
5	5	54	37.8	33.4	0
5	6	0	14.5	14.9	180
5	6	2	76.9	83.3	217
5	6	3	21.7	16.2	165
5	6	4	31.6	34.1	133
5	6	5	37.3	33.9	141
5	6	6	16.8	15.5	173
5	6	7	35.1	45.6	62
5	6	8	23.9	22.6	122
5	6	9	42.9	48.0	221
5	6	10	47.0	47.7	193
5	6	11	25.5	25.5	4
5	6	12	43.3	46.3	315
5	6	13	27.5	24.5	243
5	6	14	44.4	43.0	182
5	6	15	22.7	21.7	343
5	6	16	18.3	15.7	15
5	6	17	20.9	16.6	37
5	6	18	38.6	36.0	193
5	6	19	16.2	10.6	321
5	6	20	38.2	32.7	[illegible]
5	6	22	38.0	35.1	201
5	6	23	28.2	28.7	317
5	6	24	28.3	23.7	234
5	6	25	21.3	25.3	232
5	6	26	33.6	41.9	152
5	6	28	39.8	37.2	319
5	6	29	27.1	24.7	266
5	6	30	26.8	24.8	241
5	6	31	26.5	23.7	76
5	6	32	19.2	17.4	296
5	6	33	18.4	19.6	159
5	6	34	50.0	47.0	222
5	6	35	33.7	25.2	14
5	6	36	26.7	19.0	156
5	6	38	76.9	69.8	193
5	6	39	24.4	24.5	332
5	6	40	43.8	36.8	249
5	6	42	43.1	35.7	232
5	6	44	19.1	13.4	253
5	7	0	14.8	16.6	180
5	7	1	27.9	26.1	162
5	7	2	28.5	32.3	174
5	7	3	58.9	61.7	339
5	7	5	26.7	27.0	213
5	7	0	14.8	16.6	180
5	7	1	27.9	26.1	162
5	7	2	28.5	32.3	174
5	7	3	58.9	61.7	339
5	7	5	26.7	27.0	213
5	7	7	30.7	28.5	236
5	7	8	17.3	19.2	227
5	7	9	39.2	41.7	172
5	7	10	40.7	45.8	178
5	7	11	36.5	40.2	233
5	7	12	17.4	12.0	254
5	7	13	22.1	22.0	179
5	7	14	25.7	21.7	96
5	7	16	21.4	20.2	93
5	7	17	39.9	38.8	195
5	7	18	34.6	29.5	116
5	7	19	45.0	54.6	12
5	7	20	24.8	10.0	166
5	7	21	67.0	63.8	188
5	7	22	22.0	30.0	197
5	7	23	13.5	27.1	339
5	7	25	43.3	46.8	207
5	7	26	27.9	25.7	204
5	7	27	36.0	29.4	222
5	7	28	33.7	31.7	58
5	7	29	23.4	10.7	281
5	7	31	16.6	21.3	327
5	7	33	35.3	31.2	194
5	7	34	33.7	32.9	138
5	7	36	17.1	19.6	197
5	7	37	36.7	32.7	154
5	7	38	21.4	19.6	154
5	7	39	17.8	8.9	185
5	7	40	15.9	19.4	140
5	7	41	19.4	22.6	330
5	7	42	25.4	22.1	145
5	7	45	16.4	19.9	229
5	7	46	42.6	34.3	140
5	7	48	15.0	10.0	125
5	7	50	21.4	16.2	131
5	7	51	15.7	14.3	245
5	7	52	18.2	14.2	97
5	7	53	16.7	20.7	139
5	7	56	25.4	17.2	117
5	7	57	17.7	23.1	235
5	7	58	17.1	6.1	143
5	8	0	24.7	19.8	0
5	8	3	32.7	35.4	80
5	8	6	27.6	17.7	14
5	8	7	30.3	28.2	62
5	8	8	24.7	16.0	156
5	8	9	21.4	31.0	111
5	8	10	17.4	11.9	357
5	8	11	19.0	20.4	91
5	8	12	24.6	20.2	20
5	8	13	38.9	35.2	110
5	8	15	21.2	26.8	44
5	8	16	29.0	33.7	13
5	8	17	35.4	36.9	89
5	8	19	27.2	29.3	94
5	8	21	23.0	27.3	55
5	8	22	15.3	19.7	6
5	8	23	20.9	27.0	54
5	8	24	48.0	28.3	334
5	8	25	18.5	18.4	132
5	8	27	29.9	16.9	152
5	8	30	23.3	20.8	329
5	8	31	16.4	21.6	96
5	8	32	16.2	9.5	177
5	8	33	44.2	35.9	114
5	8	37	26.9	25.0	73
6	6	0	62.8	63.7	0
6	6	3	20.4	25.0	90
6	6	4	43.9	42.2	0
6	6	6	18.7	24.6	180
6	6	8	41.9	51.1	0
6	6	9	20.5	23.2	90
6	6	10	18.8	9.5	0
6	6	11	32.6	41.5	90
6	6	12	37.8	30.6	0
6	6	13	50.1	41.3	90
6	6	14	20.5	29.0	0
6	6	15	13.4	20.8	90
6	6	16	57.1	59.2	0
6	6	17	27.3	22.5	90
6	6	19	46.9	24.3	90
6	6	20	89.2	94.8	0
6	6	22	28.5	34.0	180
6	6	23	21.1	16.5	90
6	6	24	61.1	66.7	0
6	6	26	36.2	38.2	0
6	6	27	30.5	28.7	90
6	6	28	23.7	25.1	0
6	6	29	17.3	24.0	90
6	6	30	49.3	59.2	0
6	6	31	19.3	20.9	90
6	6	32	45.3	52.3	0
6	6	33	21.1	30.9	90
6	6	35	30.4	30.7	90
6	6	36	49.5	43.4	0
6	6	37	23.3	25.2	90
6	6	38	13.4	2.4	180
6	6	40	31.4	31.0	0
6	6	42	13.1	6.9	180
6	6	43	13.0	15.6	90
6	6	44	12.9	16.2	0
6	6	45	12.7	6.7	90
6	6	48	30.1	37.9	0
6	6	52	25.7	39.5	0
6	7	0	25.8	38.9	0
6	7	2	23.5	28.5	52
6	7	3	17.9	20.3	315
6	7	4	14.7	12.1	177
6	7	6	35.2	31.6	167
6	7	7	39.6	43.6	276
6	7	8	27.3	25.5	142
6	7	9	33.9	26.9	225
6	7	10	35.9	38.9	228
6	7	11	33.7	35.9	355
7	7	0	18.0	19.2	180
7	7	1	33.0	35.4	90
7	7	2	18.0	20.8	0
7	7	3	18.0	22.9	90

and precession photographs were taken with Cu-Kα and Mo-Kα radiation. The cell dimensions were obtained from precession photographs. Three-dimensional intensity data were recorded on zero layer and equi-inclination Weissenberg photographs about the 9·68 Å axis, using a small crystal completely bathed in the X-ray beam. No absorption corrections were applied. The layers $0kl$, $1kl \cdots 7kl$ were recorded visually and reduced to structure amplitudes by applying the Lorentz, polarisation, and Tunell rotation factors. A total of 2068 independent reflections were observed, but for those of highest order (l about 60 or larger) it was impossible to estimate the intensities accurately with the available equipment. These were therefore excluded from the analysis, which is based on a total 1860 $|F_o|$ values, as listed in Table 8.

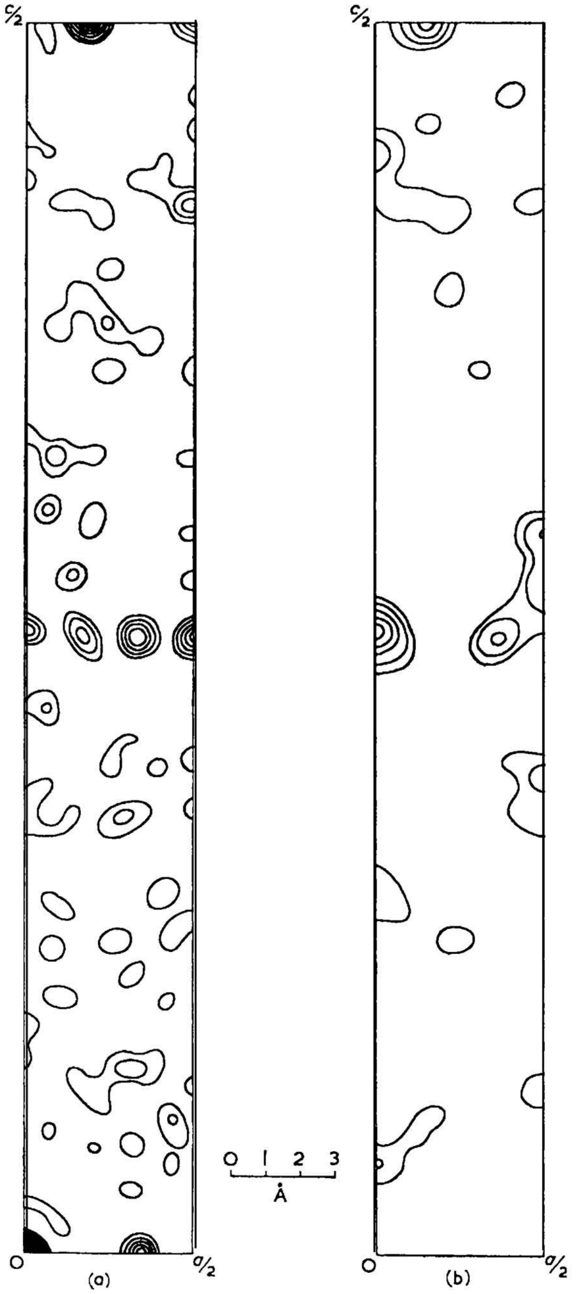

FIGURE 4 (a) Patterson projection along the b-axis. (b) Section at $V = \frac{1}{2}$ through the three-dimensional Patterson synthesis. Contour scale arbitrary

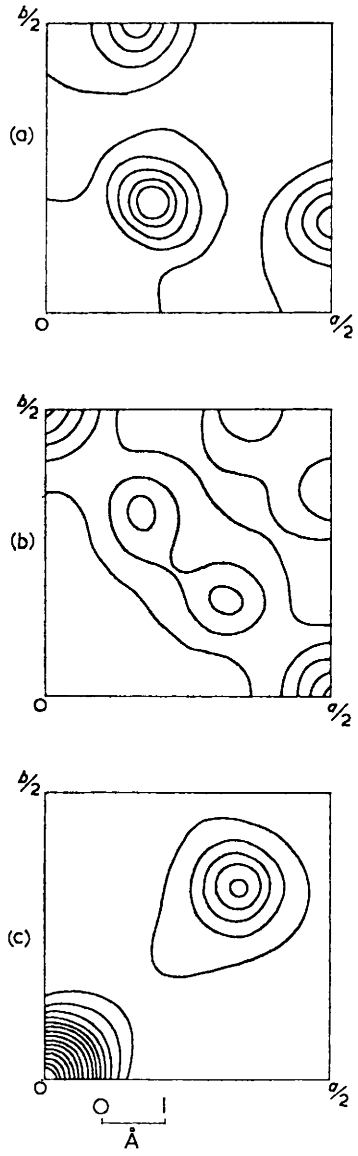

FIGURE 5 Three-dimensional Patterson synthesis. Contour scale arbitrary. (a) Section P(UV$\frac{1}{2}$). (b) Section P(UV$\frac{1}{4}$). (c) Section P(UVO)

Analysis of the Structure.—The position of the iodine atom was first determined from a Patterson vector analysis. The projection P(UW) is shown in Figure 4 (a) and the concentration of heavy peaks on the lines $z = 0$, $\frac{1}{4}$, and $\frac{1}{2}$ immediately suggests that the z co-ordinate of the iodine atom is at a special position very near to these lines. The vectors between the 8 symmetry related iodine atoms in this space group give rise to a total of 28 peaks in the three-dimensional Patterson function. However, a complete solution is possible[18] from a study of the heavier peaks appearing on the section P(U$\frac{1}{2}$W) shown in Figure 4 (b), and on the sections P(UV$\frac{1}{2}$), P(UV$\frac{1}{4}$), and P(UVO) which are shown in Figure 5.

To overcome difficulties with the Fourier and structure

[18] K. K. Cheung, Ph.D. Thesis, University of Glasgow, 1965.

factor programmes [19] for the DEUCE computer in space group $P4_12_12$, the unit cell was treated as if it belonged to the orthorhombic space group $P2_12_12_1$, with two molecules in the asymmetric crystal unit. This involved a shift of origin to $(\frac{1}{4}, 0, \frac{3}{8})$.

After the fourth set of structure factor and Fourier calculations, when all the atoms had been clearly resolved and the value of R was 23·3%, the space group was transformed back to $P4_12_12$ and least squares refinement commenced. This was carried out on the Glasgow University KDF9 computer using the S.F.L.S. programme for which we are indebted to Professor D. W. J. Cruickshank and Mr. J. G. F. Smith. The weighting scheme [20] employed was

$$w = 1/(a + |F_o| + c|F_o|^2)$$

where

$$a = 2|F_o| \text{ min.} = 14{\cdot}4$$

$$c = 2/|F_o| \text{ max.} = 0{\cdot}006$$

During the last two cycles the iodine atom was allowed to refine anisotropically using the temperature factor

$$\exp\,[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{31}lhc^*a^* + 2U_{12}hka^*b^*)]$$

where, for example, U_{11} is the mean square amplitude of vibration of the atom parallel to the reciprocal axis a^*. The values found for these parameters are given in Table 1.

The final atomic co-ordinates, bond lengths, and angles are given in Tables 1—5. The estimated standard deviations of the atomic parameters were also calculated by the Glasgow S.F.L.S. programme using the equation [21]

$$\sigma_i^2 = (c^{-1})_{ii}\,\frac{\Sigma w\Delta^2}{m - n}$$

where $(c^{-1})_{ii}$ is the diagonal element of the matrix inverse to the block-diagonal c_{ij} matrix in the normal equations; m is the number of observations and n is the number of parameters.

The bond length standard deviations, $\sigma(l)$, between two atoms with co-ordinates $(x_1\ y_1\ z_1)$ and $(x_2\ y_2\ z_2)$ were calculated from the equation

$$\sigma(l) = \frac{1}{l}\{(x_1 - x_2)^2[\sigma^2(x_1) + \sigma^2(x_2)]^2 + (y_1 - y_2)^2[\sigma^2(y_1) + \sigma^2(y_2)]^2 + (z_1 - z_2)^2[\sigma^2(z_1) + \sigma^2(z_2)]^2\}^{\frac{1}{2}}$$

The bond angle standard deviations were calculated from the equation given by Darlow.[22] These were calculated by using a Glasgow Algol programme written by Dr. W. S. McDonald.

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GLASGOW W.2. [6/349 *Received, March 18th*, 1966.]

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[20] D. W. J. Cruickshank, D. E. Pilling, A. Bujosa, F. M. Lovell, and M. R. Truter, in ref. 19, p. 32.

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[22] S. F. Darlow, *Acta Cryst.*, 1960, **13**, 683.