Acta Cryst. (1969). B**25**, 665

The Crystal and Molecular Structure of 2-Chlorotropone–Cycloheptatriene Adduct

By Yoshimasa Fukazawa and Sho Itô
Department of Chemistry, Tohoku University, Sendai, Japan

and Yoichi Iitaka
Faculty of Pharmaceutical Sciences, University of Tokyo, Hongo, Tokyo, Japan

(*Received* 6 *May* 1968)

The crystal and molecular structure of the adduct, $C_{14}H_{13}OCl$, formed by thermal reaction of 2-chlorotropone and cycloheptatriene, has been determined by X-ray diffraction methods. The crystals are monoclinic, space group $P2_1/a$ and the unit cell contains four molecules and has dimensions $a=10{\cdot}45$ Å, $b=7{\cdot}65$, $c=13{\cdot}40$ Å and $\beta=105{\cdot}7°$. The structure was solved by the heavy atom method and refined by full-matrix least-squares calculations with anisotropic thermal parameters. The present structure determination confirmed the results of the previous chemical study that the adduct is 7-chloropentacyclo $[7{\cdot}5{\cdot}0{\cdot}0^{2,7}{\cdot}0^{5,13}{\cdot}0^{6,12}]$tetradeca-3,10-diene-8-one having a unique cage structure.

Introduction

It has been shown that tropone and 2-chlorotropone, when heated with cycloheptatriene, afford the adducts, $C_{14}H_{14}O$ and $C_{14}H_{13}OCl$, respectively. Unique cage structures shown in Fig. 1 have been assigned to these adducts on the basis of detailed nuclear magnetic resonance analysis (Ito, Fujise & Woods, 1967). Similar thermal reaction of 2-methoxytropone and cycloheptatriene yielded analogous cage compounds (Sato, Fujise & Ito, 1967). In order to confirm the structure and to obtain more precise information on the stereochemistry of these adducts, the crystal structure analysis of the 2-chlorotropone–cycloheptatriene adduct has been carried out.

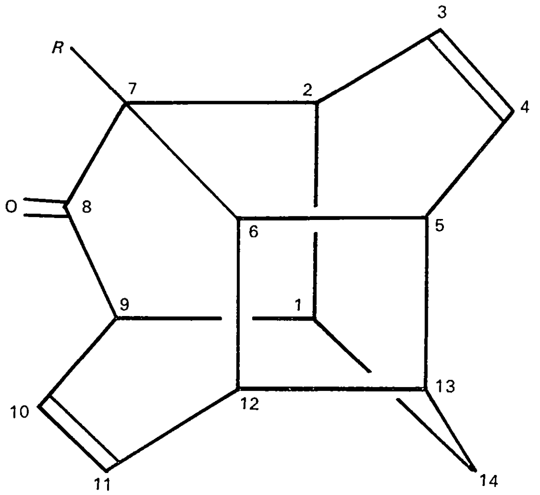


Fig. 1. Chemical structures of the adducts (*a*) tropone + cycloheptatriene, $R=H$ (*b*) 2-chlorotropone + cycloheptatriene, $R=Cl$.

Experimental

2-Chlorotropone–cycloheptatriene adduct, $C_{14}H_{13}OCl$, was prepared by heating a mixture of 2-chlorotropone and cycloheptatriene at 140°C for 5 hours in xylene. The adduct was purified by means of silica gel chromatography and crystallized from the xylene solutions. Recrystallization from xylene afforded well developed crystals of colorless prisms elongated along the *c* axis.

The lattice constants were determined from precession photographs about the three crystallographic axes taken with Cu $K\alpha$ radiation. The density was measured by the flotation method using aqueous solutions of potassium iodide of various concentrations.

Crystal data

2-Chlorotropone–cycloheptatriene adduct (7-chloropentacyclo$[7{\cdot}5{\cdot}0{\cdot}0^{2,7}{\cdot}0^{5,13}{\cdot}0^{6,12}]$tetradeca-3,10-diene-8-one), $C_{14}H_{13}OCl$, m.p. 149 ~ 151 °C, mol.wt. 232·5 .

Monoclinic,

$a=10{\cdot}45\pm0{\cdot}02$, $b=7{\cdot}65\pm0{\cdot}01$, $c=13{\cdot}40\pm0{\cdot}02$ Å,
$\beta=105{\cdot}7°\pm0{\cdot}2°$.

$V=1031{\cdot}3$ Å^3 .
$D_m=1{\cdot}47$ g.cm^{-3}, $D_x=1{\cdot}506$ g.cm^{-3} .
$Z=4$.
$F(000)=492$.
$\mu(\text{Cu } K\alpha)=34{\cdot}0$ cm^{-1} .
Absent spectra, $h0l$ when h is odd, $0k0$ when k is odd.
Space group, $P2_1/a$.

The three-dimensional intensity data were collected from equi-inclination Weissenberg photographs using Cu $K\alpha$ radiation taken for the layer lines: zero to 8th about the *c* axis and zero to 2nd about the *b* axis. The intensities were recorded on multiple-film packs and were measured with a calibrated intensity scale.

The specimen for the c axis photographs was a small prismatic crystal with dimensions 0·18 × 0·25 mm in cross section, and that for the b axis was a cylinder cut and shaped from a larger crystal by means of a thin blade.

The intensity data were corrected for Lorentz and polarization factors and put on a common scale by correlating the equivalent structure factors obtained in various layers. No absorption correction was applied. The total number of independent observed structure factors was 1526.

Determination and refinement of the structure

The coordinates of the chlorine atom were determined from a three-dimensional sharpened Patterson map calculated for the structure with atoms at rest. The crystal contains one chlorine atom in the asymmetric unit and the coordinates were found to be $x=0{\cdot}439$, $y=0{\cdot}355$, $z=0{\cdot}109$. Utilizing the phase angles given by the contribution of the chlorine atoms, the first three-dimensional Fourier synthesis was calculated. The R index was 0·64. Of the twenty-three electron density peaks with noticeable height, apparent in the Fourier synthesis, four peaks were chosen for the next phase angle calculation. A gross feature of the molecular structure suggested by the previous chemical study was of great help in eliminating spurious peaks. Consecutive rounds of structure factor and Fourier or difference Fourier calculations revealed the locations of all the atoms at their approximate positions. The R index at this stage was 0·48.

Refinement of the structure was carried out by least-squares methods. Several cycles of block-matrix least-squares calculations followed by four cycles of full-matrix anisotropic calculations by the *ORFLS* program (Busing, Martin & Levy, 1962) reduced the R value to 0·143 for 1526 observed reflexions. In the latter calculations the weighting scheme was taken as:

$$\sqrt{w}=30/F_o \quad \text{when} \quad F_o>30\,,$$
$$\sqrt{w}=F_o/30 \quad \text{when} \quad F_o\le 30\,.$$

The final positional and thermal parameters are listed in Tables 1 and 2. Averages of the standard deviations of the positional parameters are estimated as follows:

for chlorine atoms, $\sigma(r)=0{\cdot}0024$ Å ,
for oxygen atoms, $\sigma(r)=0{\cdot}0068$ Å ,
and for carbon atoms, $\sigma(r)=0{\cdot}0090$ Å .

The final Fourier and difference Fourier syntheses were then calculated using the parameters shown in Tables 1 and 2. Fig. 2(*a*) and (*b*) show the superimposed contour sections of the electron density map and the view of the molecule along the b axis. All the hydrogen atoms were located on the difference electron density map as shown in Fig. 3. The coordinates and the isotropic temperature factors of the hydrogen atoms were then refined by block-matrix least-squares calculations (program by Okaya & Ashida, 1967) in which the heavy atom parameters were left unchanged. The R index was reduced to 0·139. The refined atomic parameters

Table 1. *Final fractional coordinates and their standard deviations*

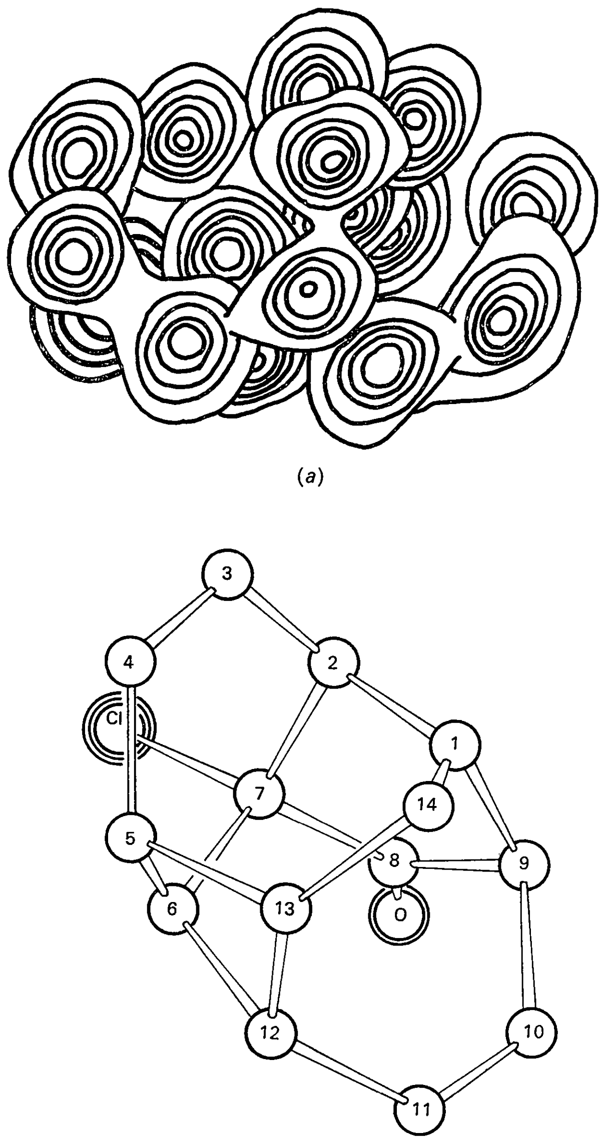
	$x\,[10^4\sigma(x)]$	$y\,[10^4\sigma(y)]$	$z\,[10^4\sigma(z)]$
Cl	−0·0559 (2)	0·1492 (3)	0·1083 (2)
O	0·1033 (7)	0·0705 (8)	0·3278 (6)
C(1)	−0·0429 (8)	0·4781 (10)	0·3415 (7)
C(2)	−0·1133 (7)	0·3943 (11)	0·2352 (6)
C(3)	−0·1855 (10)	0·5079 (13)	0·1493 (8)
C(4)	−0·1155 (10)	0·6012 (14)	0·1036 (8)
C(5)	0·0388 (9)	0·5970 (12)	0·1387 (8)
C(6)	0·1029 (8)	0·4204 (11)	0·1785 (7)
C(7)	0·0020 (8)	0·2991 (10)	0·2109 (6)
C(8)	0·0614 (9)	0·2142 (12)	0·3124 (8)
C(9)	0·0613 (9)	0·3414 (11)	0·3996 (7)
C(10)	0·2036 (9)	0·4013 (12)	0·4374 (8)
C(11)	0·2697 (9)	0·7415 (12)	0·3781 (8)
C(12)	0·2052 (8)	0·5210 (10)	0·2664 (8)
C(13)	0·0998 (9)	0·6709 (12)	0·2521 (7)
C(14)	0·0156 (9)	0·6560 (12)	0·3253 (8)

Table 2. *Final thermal parameters and their standard deviations*

Temperature factors are of the form:

$$\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\right].$$

	$\beta_{11}[10^4\sigma(\beta_{11})]$	$\beta_{22}[10^4\sigma(\beta_{22})]$	$\beta_{33}[10^4\sigma(\beta_{33})]$	$\beta_{12}[10^4\sigma(\beta_{12})]$	$\beta_{13}[10^4\sigma(\beta_{13})]$	$\beta_{23}[10^4\sigma(\beta_{23})]$
Cl	0·0111 (3)	0·0158 (4)	0·0074 (2)	−0·0032 (3)	0·0007 (2)	−0·0045 (2)
O	0·0165 (9)	0·0108 (11)	0·0105 (6)	0·0033 (8)	0·0017 (6)	0·0012 (7)
C(1)	0·0097 (10)	0·0089 (14)	0·0087 (8)	−0·0008 (10)	0·0013 (7)	−0·0015 (9)
C(2)	0·0079 (8)	0·0170 (16)	0·0058 (6)	0·0016 (10)	0·0028 (6)	−0·0003 (9)
C(3)	0·0116 (11)	0·0185 (19)	0·0070 (7)	0·0024 (13)	0·0019 (7)	0·0006 (11)
C(4)	0·0131 (12)	0·0206 (22)	0·0081 (8)	0·0076 (15)	−0·0001 (8)	0·0016 (12)
C(5)	0·0126 (11)	0·0149 (17)	0·0095 (9)	0·0035 (12)	0·0011 (8)	0·0004 (11)
C(6)	0·0068 (8)	0·0134 (16)	0·0079 (7)	−0·0008 (9)	0·0012 (6)	0·0011 (9)
C(7)	0·0087 (9)	0·0121 (15)	0·0040 (5)	−0·0023 (9)	0·0027 (5)	−0·0013 (8)
C(8)	0·0089 (10)	0·0149 (18)	0·0083 (8)	−0·0040 (11)	0·0022 (7)	−0·0002 (11)
C(9)	0·0114 (10)	0·0149 (16)	0·0059 (6)	0·0009 (11)	−0·0004 (6)	−0·0039 (10)
C(10)	0·0123 (12)	0·0134 (17)	0·0065 (7)	0·0028 (11)	0·0006 (8)	−0·0020 (10)
C(11)	0·0105 (10)	0·0141 (17)	0·0078 (9)	0·0004 (11)	0·0015 (7)	−0·0045 (10)
C(12)	0·0105 (10)	0·0090 (14)	0·0093 (9)	0·0001 (10)	0·0012 (7)	−0·0012 (9)
C(13)	0·0137 (12)	0·0162 (17)	0·0068 (8)	0·0006 (12)	0·0050 (8)	0·0004 (10)
C(14)	0·0114 (10)	0·0141 (16)	0·0090 (8)	−0·0001 (11)	0·0042 (8)	−0·0008 (10)



(*a*)

(*b*)

Fig. 2. (*a*) Composite projection of the electron density map along the *b* axis. Contours are drawn at 1,2,3 . . . e.Å^{-3} for carbon and oxygen atoms and 3,6,9 . . . e.Å^{-3} for chlorine. (*b*) Molecular structure projected along the same axis as (*a*).

of the hydrogen atoms and their standard deviations are listed in Table 3. A set of structure factors calculated with the final parameters shown in Tables 1, 2 and 3 is given in Table 4, together with the observed structure factors.

Atomic scattering factors used were those given by Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for oxygen and carbon, and those given by Dawson (1960) for chlorine.

Discussion of the structure

Bond lengths and angles found in the adduct molecule are shown in Figs. 4 and 5. The mean values of the estimated standard deviations in bond lengths are 0·008 Å for Cl–C, 0·012 Å for C=O and 0·013 Å for C–C, the mean estimated standard deviation in tetrahedral C–C–C bond angles being 0·8 °. The C–C single bond lengths vary from 1·476 Å in C(2)–C(3) to 1·584 Å in C(5)–C(13), the mean value for the sixteen C–C bonds being 1·531 Å.

The shorter bond lengths found in C(3)–C(4) and C(10)–C(11), the larger valency angles in C(2)–C(3)–C(4), C(3)–C(4)–C(5), C(9)–C(10)–C(11) and C(10)–C(11)–C(12), and the smaller torsion angles of 1·1 ° and 7·7 ° found in C(2)–C(3)–C(4)–C(5) and C(9)–C(10)–C(11)–C(12) respectively, indicate the double bond character of the bonds C(3)–C(4) and C(10)–C(11). The arrangement of hydrogen atoms about the four carbon atoms also supports this view.

The molecular structure of the 2-chlorotropone–cycloheptatriene adduct has thus been determined, without any ambiguity, to be 7-chloropentacyclo-$[7{\cdot}5{\cdot}0{\cdot}0^{2,7}{\cdot}0^{5,13}{\cdot}0^{6,12}]$tetradeca-3,10-diene-8-one, comfirming the previous chemical study (Ito, Fujise & Woods, 1967).

Conformation of the molecule will be discussed by considering each part of the molecule separately. The planarity, bond lengths and valency angles involved in each ring are summarized in Table 5.

The four-membered A ring formed by C(5), C(6), C(12) and C(13).

The bond angles involved in the four-membered cyclobutane ring are all less than a right angle. This

Table 3. *Refined fractional coordinates, temperature factors and standard deviations, of the hydrogen atoms*

	$x[10^3\sigma(x)]$	$y[10^3\sigma(y)]$	$z[10^3\sigma(z)]$	$B\,[\sigma(B)]$
H(1)	−0·131 (10)	0·491 (8)	0·385 (13)	4·8 (2·6) Å^2
H(2)	−0·181 (8)	0·318 (7)	0·266 (10)	2·3 (2·0)
H(3)	−0·292 (10)	0·493 (8)	0·214 (13)	3·5 (2·3)
H(4)	−0·169 (10)	0·665 (8)	0·028 (13)	5·9 (2·7)
H(5)	0·110 (10)	0·641 (8)	0·081 (13)	3·8 (2·5)
H(6)	0·164 (8)	0·365 (7)	0·119 (10)	1·4 (1·8)
H(9)	0·026 (10)	0·303 (8)	0·475 (13)	5·2 (2·6)
H(10)	0·232 (11)	0·379 (8)	0·513 (14)	6·3 (2·8)
H(11)	0·378 (10)	0·501 (8)	0·406 (13)	3·8 (2·5)
H(12)	0·281 (10)	0·564 (8)	0·226 (13)	3·1 (2·3)
H(13)	0·173 (10)	0·822 (8)	0·278 (14)	5·8 (2·7)
H(14)	−0·096 (10)	0·716 (9)	0·297 (14)	6·1 (2·9)
H(15)	0·063 (10)	0·698 (10)	0·415 (14)	4·5 (2·8)

Table 4. *Observed and calculated structure factors*

H	K	L	|F(OBS)|	F(CAL)
-6	0	0	29.48	-29.12
2	0	0	82.70	95.74
4	0	0	4.29	0.86
6	0	0	31.42	-29.12
8	0	0	14.84	-14.38
10	0	0	3.18	2.26
12	0	0	5.68	-6.58
-5	1	0	15.15	-16.25
1	1	0	70.23	-74.35
2	1	0	7.66	6.80
3	1	0	23.37	22.08
4	1	0	20.71	24.53
5	1	0	19.53	-16.25
6	1	0	12.59	14.09
7	1	0	7.58	-6.08
8	1	0	8.06	7.15
9	1	0	10.01	-10.08
10	1	0	1.85	1.67
11	1	0	6.52	-6.21
12	1	0	1.28	-1.19
-5	2	0	20.99	-19.22
0	2	0	44.02	44.13
1	2	0	24.61	22.84
2	2	0	64.14	-64.24
3	2	0	2.79	0.41
4	2	0	4.67	-5.13
5	2	0	21.63	19.22
6	2	0	2.29	-3.15
7	2	0	11.58	10.54
8	2	0	3.47	2.19
9	2	0	4.90	-4.10
10	2	0	5.11	6.12
11	2	0	7.35	-6.39
12	2	0	4.56	-4.27
1	3	0	7.48	1.98
2	3	0	13.80	-13.01
3	3	0	1.60	-2.69
4	3	0	21.23	19.22
5	3	0	5.54	-2.17
6	3	0	26.01	24.36
7	3	0	18.96	20.24
8	3	0	13.37	14.35
9	3	0	19.51	19.86
10	3	0	3.30	2.67
11	3	0	5.18	5.21
0	4	0	42.11	-36.32
1	4	0	1.42	-1.37
2	4	0	41.17	-35.40
3	4	0	22.84	-20.25
4	4	0	15.97	16.18
5	4	0	13.94	-11.84
6	4	0	12.96	9.93
7	4	0	10.20	-10.15
8	4	0	1.98	0.13
9	4	0	2.42	-1.05
10	4	0	2.31	2.61
11	4	0	2.46	3.17
1	5	0	1.34	-2.17
2	5	0	29.04	-25.75
3	5	0	10.64	-9.00
4	5	0	27.91	-27.75
5	5	0	15.37	-14.90
6	5	0	1.98	-2.39
7	5	0	4.91	-6.50
8	5	0	1.13	-0.61
9	5	0	2.58	2.28
10	5	0	1.68	2.16
0	6	0	18.37	18.05
1	6	0	9.91	-9.63
2	6	0	10.72	9.35
3	6	0	15.89	-14.59
4	6	0	15.41	14.37
5	6	0	12.75	-10.98
6	6	0	1.95	0.87
7	6	0	8.05	-8.91
8	6	0	9.40	-11.40
9	6	0	3.28	1.98
10	6	0	3.08	-5.68
1	7	0	1.18	2.94
2	7	0	10.07	9.75
3	7	0	8.12	8.72
4	7	0	1.48	-2.08
5	7	0	1.87	-0.58
6	7	0	4.90	5.36
7	7	0	1.69	-1.18
8	7	0	5.10	-4.11
0	8	0	1.11	1.79
1	8	0	1.89	1.26
2	8	0	3.26	-3.77
3	8	0	8.29	9.42
4	8	0	4.23	4.68
5	8	0	7.62	8.07
6	8	0	1.97	4.33
-12	0	1	3.86	3.62
-10	0	1	2.76	-3.55
-8	0	1	16.15	-15.09
-6	0	1	2.09	-2.32
-4	0	1	6.79	-7.20
-2	0	1	47.75	58.13
2	0	1	10.97	9.66
4	0	1	26.05	-26.41
6	0	1	5.67	3.99
8	0	1	4.67	3.33
10	0	1	2.55	-3.04
12	0	1	3.96	-4.99
-12	1	1	4.03	5.59
-11	1	1	3.46	-3.79
-10	1	1	8.96	8.50
-9	1	1	9.44	-10.30
-8	1	1	7.46	6.44
-7	1	1	16.48	-15.60
-6	1	1	12.52	-11.94
-5	1	1	16.99	-15.09
-4	1	1	36.36	-36.92
-3	1	1	47.99	47.15
-2	1	1	60.06	-67.69
-1	1	1	11.32	-8.98
0	1	1	62.21	-63.71
1	1	1	23.83	23.66
2	1	1	1.68	-1.04
3	1	1	14.33	13.94
4	1	1	30.56	30.88
5	1	1	38.40	37.62
6	1	1	32.86	33.59
7	1	1	4.86	-2.60
8	1	1	10.25	10.63
9	1	1	10.50	-11.34
10	1	1	2.45	-1.32
11	1	1	4.13	-4.42
12	1	1	4.06	-4.15
-11	2	1	5.57	6.51
-10	2	1	1.24	1.56
-9	2	1	7.98	7.44
-8	2	1	9.33	-7.37
-7	2	1	9.98	9.58
-6	2	1	2.09	-1.59
-5	2	1	3.40	-1.56
-4	2	1	16.89	-15.72
-3	2	1	63.08	-64.81
-2	2	1	6.72	-4.05
-1	2	1	52.67	-55.53
0	2	1	15.48	-13.71
1	2	1	37.06	-39.84
2	2	1	15.87	13.97
3	2	1	27.76	25.47
4	2	1	0.88	1.41
5	2	1	12.22	12.24
6	2	1	12.85	12.49
7	2	1	12.48	10.48
8	2	1	5.24	5.47
9	2	1	9.98	9.82
10	2	1	3.77	3.19
11	2	1	2.18	2.85
-10	3	1	2.21	-2.82
-9	3	1	8.93	7.61
-8	3	1	4.83	-6.28
-7	3	1	28.65	28.91
-6	3	1	7.55	5.29
-5	3	1	16.76	18.66
-4	3	1	23.37	22.27
-3	3	1	39.56	35.03
-2	3	1	1.62	-0.49
-1	3	1	28.63	-27.89
0	3	1	26.40	-24.11
1	3	1	39.91	-38.86
2	3	1	9.70	9.11
3	3	1	46.07	-49.96
4	3	1	27.61	25.39
5	3	1	23.14	-20.73
6	3	1	28.08	24.66
7	3	1	1.74	1.41
8	3	1	2.05	0.35
9	3	1	7.26	6.37
10	3	1	1.04	-2.14
-10	4	1	4.60	7.64
-9	4	1	3.92	-3.71
-8	4	1	1.18	2.37
-7	4	1	1.17	-0.47
-6	4	1	11.25	10.33
-5	4	1	4.19	-2.16
-4	4	1	7.14	6.90
-3	4	1	2.04	-2.21
-2	4	1	24.51	-20.40
-1	4	1	16.86	15.85
0	4	1	27.47	-25.29
1	4	1	6.29	4.31
2	4	1	24.28	-22.47
3	4	1	8.99	10.30
4	4	1	8.43	-6.57
5	4	1	30.08	-25.13
6	4	1	11.28	8.76
7	4	1	15.74	-14.32
8	4	1	5.26	2.77
9	4	1	3.46	-4.75
10	4	1	6.68	7.43
-10	5	1	6.64	-7.13
-9	5	1	1.04	0.79
-8	5	1	8.57	-9.29
-7	5	1	16.08	15.70
-6	5	1	15.59	14.70
-5	5	1	7.56	-4.76
-4	5	1	17.06	20.36
-3	5	1	7.28	6.52
-2	5	1	11.09	11.55
-1	5	1	6.82	5.44
0	5	1	7.22	-5.83
1	5	1	16.32	16.12
2	5	1	18.06	-15.93
3	5	1	4.04	2.85
4	5	1	9.41	-12.10
5	5	1	21.69	-21.49
6	5	1	10.08	-12.24
7	5	1	12.21	-10.98
8	5	1	10.85	-10.24
9	5	1	3.39	-0.43
10	5	1	2.92	-1.21
-9	6	1	0.87	1.21
-8	6	1	7.26	-9.27
-7	6	1	5.45	6.16
-6	6	1	8.72	-8.47
-5	6	1	9.51	8.15
-4	6	1	8.45	-9.05
-3	6	1	17.80	16.40
-2	6	1	9.77	-12.60
-1	6	1	8.17	7.64
0	6	1	4.74	5.11
1	6	1	9.67	7.07
2	6	1	7.66	6.48
3	6	1	18.02	17.55
4	6	1	6.51	7.15
5	6	1	3.75	2.35
6	6	1	5.17	5.26
7	6	1	7.19	5.56
8	6	1	4.53	-3.62
9	6	1	2.95	-6.60
-8	7	1	2.79	3.97
-7	7	1	3.80	-5.68
-6	7	1	5.04	7.82
-5	7	1	11.05	-14.27
-4	7	1	11.62	-11.94
-3	7	1	3.52	-0.87
-2	7	1	8.50	-8.61
-1	7	1	11.92	12.45
0	7	1	1.20	0.11
1	7	1	29.69	26.81
2	7	1	5.57	-6.47
3	7	1	20.35	21.50
4	7	1	1.15	0.88
5	7	1	5.61	4.48
6	7	1	3.82	-2.77
7	7	1	2.21	-3.30
8	7	1	0.73	0.37
-6	8	1	0.83	1.02
-5	8	1	6.67	-9.04
-4	8	1	7.42	-7.31
-3	8	1	1.81	-0.81
-2	8	1	8.52	-8.36
-1	8	1	8.69	-7.56
0	8	1	3.50	3.57
1	8	1	6.02	-3.60
2	8	1	10.10	10.83
3	8	1	6.05	6.52
4	8	1	6.48	5.32
5	8	1	2.51	2.43
6	8	1	0.75	-1.88
-12	0	2	3.33	3.25
-10	0	2	2.81	-3.10
-8	0	2	10.21	-11.34
-6	0	2	15.92	-17.34
-4	0	2	18.00	-22.29
-2	0	2	96.20	-114.69
2	0	2	36.07	39.92
4	0	2	37.37	42.65
6	0	2	17.65	19.09
8	0	2	7.98	-7.38
10	0	2	19.04	-17.37
-12	1	2	1.99	2.44
-11	1	2	3.42	4.00
-10	1	2	5.28	4.77
-9	1	2	2.02	1.26
-8	1	2	1.18	-1.32
-7	1	2	7.08	-6.30
-6	1	2	12.28	-8.93
-5	1	2	21.53	-20.27
-4	1	2	26.59	-29.37
-3	1	2	65.85	-71.19
-2	1	2	13.87	-14.53
-1	1	2	62.46	61.40
0	1	2	5.77	-6.17
1	1	2	10.88	11.80
2	1	2	40.80	-40.87
3	1	2	65.14	65.99
4	1	2	1.91	0.09
5	1	2	26.48	23.93
6	1	2	1.62	1.47
7	1	2	6.75	6.19
8	1	2	4.76	4.38
9	1	2	6.54	6.98
10	1	2	2.46	-1.78
11	1	2	1.47	-1.53
-11	2	2	4.84	6.21
-10	2	2	2.46	1.40
-9	2	2	19.84	20.59
-8	2	2	9.86	10.12
-7	2	2	19.68	20.09
-6	2	2	24.74	23.35
-5	2	2	13.42	-12.21
-4	2	2	42.87	43.18
-3	2	2	49.01	-46.28
-2	2	2	33.28	33.66
-1	2	2	85.58	-91.81
0	2	2	14.20	-12.82
1	2	2	6.69	3.27
2	2	2	4.07	3.63
3	2	2	10.17	9.37
4	2	2	27.32	-24.75
5	2	2	8.77	8.49
6	2	2	11.74	-10.31
7	2	2	17.67	17.06
8	2	2	5.34	0.92
9	2	2	16.79	16.23
10	2	2	1.75	-1.77
11	2	2	9.95	10.58
-10	3	2	4.76	4.90
-9	3	2	9.13	-11.01
-8	3	2	6.29	5.85
-7	3	2	4.22	-3.25
-6	3	2	10.01	9.56
-5	3	2	23.14	24.77
-4	3	2	2.41	-2.83
-3	3	2	21.98	-18.39
-2	3	2	1.37	-0.42
-1	3	2	5.61	8.14
0	3	2	33.55	29.67
1	3	2	42.44	-40.82
2	3	2	16.52	-14.12
3	3	2	13.91	-15.67
4	3	2	4.40	1.51
5	3	2	5.81	-6.67
6	3	2	7.11	-7.56
7	3	2	16.29	-13.48
8	3	2	5.21	3.83
9	3	2	1.21	0.39
-11	4	2	3.09	-4.52
-10	4	2	4.37	6.19
-9	4	2	2.09	-2.66
-8	4	2	11.96	13.44
-7	4	2	13.16	-10.73
-6	4	2	11.41	9.41
-5	4	2	14.50	-13.78
-4	4	2	10.58	8.25
-3	4	2	6.21	3.77
-2	4	2	31.92	30.16
-1	4	2	4.70	3.57
0	4	2	14.11	-14.64
1	4	2	14.90	12.86
2	4	2	15.98	-15.03
3	4	2	23.68	-21.28
4	4	2	37.07	-33.35
5	4	2	23.56	-21.40
6	4	2	16.88	-14.22
7	4	2	15.82	-11.86
8	4	2	8.90	5.66
9	4	2	2.76	-1.50
-10	5	2	6.65	-7.02
-9	5	2	3.86	4.54
-8	5	2	2.69	-5.02
-7	5	2	1.25	1.09
-6	5	2	3.26	0.60
-5	5	2	16.29	14.73
-4	5	2	1.98	5.07
-3	5	2	4.02	-4.10
-2	5	2	8.08	8.73
-1	5	2	15.87	14.98
0	5	2	36.94	38.51
1	5	2	1.10	0.83
2	5	2	33.40	28.15
3	5	2	2.88	-0.38
4	5	2	17.66	14.28
5	5	2	12.29	10.88
6	5	2	11.04	-12.04
7	5	2	1.24	-2.24
8	5	2	8.09	-7.70
9	5	2	2.42	0.53
-8	6	2	3.77	-3.81
-7	6	2	10.20	-8.97
-6	6	2	16.75	-16.66
-5	6	2	5.71	-5.64
-4	6	2	22.47	-21.54
-3	6	2	6.22	3.93
-2	6	2	1.79	0.33
-1	6	2	18.80	16.36
0	6	2	1.20	-1.46
1	6	2	19.68	20.13
2	6	2	16.46	14.98
3	6	2	3.56	3.61
4	6	2	6.21	5.04
5	6	2	3.55	3.23
6	6	2	5.68	4.27
7	6	2	7.92	-6.08
8	6	2	3.65	3.50
-7	7	2	8.15	-10.13
-6	7	2	1.13	0.40
-5	7	2	4.74	-6.96
-4	7	2	1.22	1.27
-3	7	2	3.03	-4.36
-2	7	2	4.83	-3.82
-1	7	2	11.96	10.44
0	7	2	4.74	-2.96
1	7	2	4.00	1.39
2	7	2	5.03	-2.88
3	7	2	1.24	0.94
4	7	2	1.21	1.64
5	7	2	3.96	7.67
6	7	2	3.30	-3.66
-5	8	2	4.20	3.97
-4	8	2	1.07	0.47
-3	8	2	5.26	-6.86
-2	8	2	3.65	3.43
-1	8	2	13.89	-14.19
0	8	2	4.04	3.06
1	8	2	8.94	-10.20
2	8	2	4.22	4.06
3	8	2	1.08	-3.49
4	8	2	1.01	-4.03
5	8	2	7.75	10.96
-12	0	3	2.41	3.70
-10	0	3	2.56	-2.82
-8	0	3	5.11	4.70
-6	0	3	20.44	-22.82
-4	0	3	15.61	-20.41
-2	0	3	31.45	-37.83
2	0	3	40.48	43.25
4	0	3	24.95	24.51
6	0	3	3.43	6.46
8	0	3	2.04	1.16
10	0	3	5.70	-4.51
-12	1	3	1.44	-3.79
-11	1	3	9.23	9.33
-10	1	3	2.18	2.12
-9	1	3	5.88	7.86
-8	1	3	18.59	20.15
-7	1	3	5.85	-6.73
-6	1	3	25.88	24.47
-5	1	3	41.09	-39.03
-4	1	3	23.03	23.91
-3	1	3	58.46	-62.05
-2	1	3	11.92	12.28
-1	1	3	5.81	5.55
0	1	3	36.97	-35.62
1	1	3	26.73	29.30
2	1	3	26.35	-27.16
3	1	3	17.57	-14.75
4	1	3	28.71	-30.49
5	1	3	13.40	11.33
6	1	3	5.17	5.15
7	1	3	5.60	5.97
8	1	3	14.38	14.83
9	1	3	12.13	12.48
10	1	3	9.94	10.60
-11	2	3	5.20	6.33
-10	2	3	4.30	-4.08
-9	2	3	1.94	0.36
-8	2	3	9.63	9.20
-7	2	3	2.25	-1.95
-6	2	3	6.86	-5.89
-5	2	3	22.50	19.90
-4	2	3	32.56	29.50
-3	2	3	19.31	19.61
-2	2	3	3.52	4.55
-1	2	3	10.01	-11.31
0	2	3	4.37	2.65
1	2	3	60.00	-67.15
2	2	3	6.38	7.24
3	2	3	43.55	-43.53
4	2	3	8.50	-8.45
5	2	3	2.11	1.82
6	2	3	1.41	-1.61
7	2	3	1.89	0.62
8	2	3	3.19	0.69
9	2	3	5.31	5.16
10	2	3	1.14	0.41
-10	3	3	4.59	4.17
-9	3	3	6.07	-5.97
-8	3	3	13.56	13.74
-7	3	3	14.50	14.43
-6	3	3	5.16	-6.47
-5	3	3	24.95	25.07
-4	3	3	18.87	-18.70
-3	3	3	21.79	21.23
-2	3	3	10.74	9.00
-1	3	3	31.08	32.33
0	3	3	10.67	-10.41
1	3	3	15.85	-14.75
2	3	3	8.63	6.25
3	3	3	27.43	-26.78
4	3	3	30.32	-31.87
5	3	3	18.84	-21.81
6	3	3	6.02	-3.61
7	3	3	19.67	-20.59
8	3	3	4.63	3.62
9	3	3	3.38	2.98
10	3	3	2.88	2.12
-10	4	3	8.26	-9.20
-9	4	3	7.89	-8.34
-8	4	3	1.92	-0.45
-7	4	3	11.52	-11.08
-6	4	3	15.84	-15.62
-5	4	3	3.55	3.97
-4	4	3	25.42	22.30
-3	4	3	14.91	13.98
-2	4	3	10.95	7.92
-1	4	3	12.52	10.16
0	4	3	29.37	27.05
1	4	3	7.42	-7.27
2	4	3	14.30	13.59
3	4	3	3.15	-2.53
4	4	3	17.43	-15.13
5	4	3	21.56	19.76
6	4	3	12.11	-8.56
7	4	3	3.18	4.36
8	4	3	10.01	-10.77
9	4	3	2.69	-1.51
-10	5	3	3.53	-3.02
-9	5	3	4.26	-4.35
-8	5	3	7.75	-9.54
-7	5	3	4.20	3.39
-6	5	3	14.67	-16.53
-5	5	3	10.15	9.14
-4	5	3	9.13	-8.56
-3	5	3	1.14	0.87
-2	5	3	27.07	23.86
-1	5	3	2.85	0.68
0	5	3	26.87	23.35
1	5	3	18.06	-17.52
2	5	3	31.79	33.72
3	5	3	9.20	8.53
4	5	3	1.27	3.22
5	5	3	11.15	9.47
6	5	3	1.30	-0.98
7	5	3	2.48	-4.84
8	5	3	7.92	-10.20
9	5	3	1.88	-3.08
-10	6	3	2.38	2.13
-9	6	3	3.87	-5.87
-8	6	3	5.57	6.70
-7	6	3	4.67	-3.15
-6	6	3	8.13	-9.20
-5	6	3	5.53	-5.80
-4	6	3	1.27	2.68
-3	6	3	9.64	8.70
-2	6	3	9.70	-10.29
-1	6	3	10.74	11.34
0	6	3	6.48	6.05
1	6	3	9.16	8.69
2	6	3	14.30	16.26
3	6	3	5.18	-7.02
4	6	3	10.37	8.62
5	6	3	5.40	-6.55
6	6	3	8.87	9.48
7	6	3	4.39	-2.83
8	6	3	2.28	1.65
-8	7	3	2.85	-4.06
-7	7	3	2.58	0.36
-6	7	3	1.15	2.49
-5	7	3	1.22	-2.86
-4	7	3	2.52	2.04
-3	7	3	17.06	-21.11
-2	7	3	5.64	-5.75
-1	7	3	21.09	-23.70
0	7	3	17.84	-17.13
1	7	3	21.52	-23.53
2	7	3	1.28	-0.78
3	7	3	2.51	1.32
4	7	3	5.67	6.46
5	7	3	9.73	12.43
6	7	3	9.95	13.68
7	7	3	4.81	5.83
8	7	3	2.68	4.90
-7	8	3	7.18	11.52
-6	8	3	0.90	-3.10
-5	8	3	1.01	2.42
-4	8	3	3.42	4.91
-3	8	3	1.14	1.15
-2	8	3	2.32	1.26
-1	8	3	1.18	-1.33
0	8	3	2.88	-4.18
1	8	3	2.83	1.88
2	8	3	2.25	-0.91
3	8	3	3.36	-4.46
4	8	3	2.78	2.81
5	8	3	3.65	-5.03
6	8	3	3.80	6.05
-12	0	4	2.96	4.31
-10	0	4	16.24	17.62
-8	0	4	16.76	18.82
-6	0	4	3.82	2.94
-4	0	4	29.75	-32.43
-2	0	4	40.99	-51.87
0	0	4	86.11	-83.12
2	0	4	28.41	-31.66
4	0	4	1.32	3.92
6	0	4	22.87	23.31
8	0	4	15.55	16.87
10	0	4	12.09	13.10
-12	1	4	2.91	-2.81
-11	1	4	4.56	4.45
-10	1	4	9.27	9.48
-9	1	4	6.04	-8.08
-8	1	4	26.80	26.97
-7	1	4	4.00	1.56
-6	1	4	43.62	44.51
-5	1	4	11.19	10.12
-4	1	4	27.02	27.76
-3	1	4	39.82	36.28
-2	1	4	15.58	-15.61
-1	1	4	18.59	14.54
0	1	4	17.22	15.61
1	1	4	24.28	-28.59
2	1	4	8.17	4.33
3	1	4	15.07	-15.33
4	1	4	7.79	-9.82
5	1	4	1.11	-0.83
6	1	4	9.23	-9.92
7	1	4	16.72	16.38
8	1	4	3.19	2.75
9	1	4	4.27	-1.43
10	1	4	8.63	9.72
-11	2	4	4.41	-3.67
-10	2	4	3.57	-4.06
-9	2	4	10.57	-10.51
-8	2	4	11.41	-9.82
-7	2	4	4.44	0.67
-6	2	4	10.38	-6.26
-5	2	4	24.75	23.08
-4	2	4	15.54	-14.14
-3	2	4	40.02	39.24
-2	2	4	7.38	7.67
-1	2	4	26.22	-28.91
0	2	4	33.00	-31.09
1	2	4	91.45	-93.46
2	2	4	4.20	-5.05
3	2	4	32.29	-33.08
4	2	4	5.58	4.61
5	2	4	8.20	-6.61
6	2	4	2.11	-1.43
7	2	4	8.55	6.82
8	2	4	4.73	-3.80
9	2	4	1.75	-1.30
10	2	4	1.04	0.18
-12	3	4	3.26	-5.61
-11	3	4	1.05	2.00
-10	3	4	4.14	2.70
-9	3	4	3.99	-1.57
-8	3	4	6.86	7.02
-7	3	4	2.93	-2.39
-6	3	4	14.63	13.28
-5	3	4	1.04	0.50
-4	3	4	1.92	3.02
-3	3	4	24.65	27.96
-2	3	4	23.63	-25.22
-1	3	4	23.47	25.67
0	3	4	12.90	9.58
1	3	4	39.16	35.80
2	3	4	0.94	-0.22
3	3	4	4.06	-1.39
4	3	4	14.54	-11.22
5	3	4	16.55	-15.97
6	3	4	20.68	-21.48
7	3	4	4.73	-4.64
8	3	4	8.99	-9.25
9	3	4	13.15	-11.34
10	3	4	2.21	2.92
-10	4	4	7.53	-9.06
-9	4	4	6.69	-7.77
-8	4	4	12.38	-12.33
-7	4	4	5.87	-5.01
-6	4	4	6.14	6.53
-5	4	4	2.81	1.72
-4	4	4	7.02	4.24
-3	4	4	3.57	2.90
-2	4	4	39.65	39.10
-1	4	4	15.72	15.99
0	4	4	22.96	20.13
1	4	4	14.80	14.11
2	4	4	14.70	10.91
3	4	4	46.74	44.96
4	4	4	8.55	5.56
5	4	4	23.03	20.53
6	4	4	10.58	-10.58
7	4	4	4.97	5.28
8	4	4	4.32	-5.52
9	4	4	7.82	-8.46
-9	5	4	2.25	-0.97
-8	5	4	5.18	-4.52
-7	5	4	4.71	5.25
-6	5	4	10.68	-13.94
-5	5	4	2.46	1.93
-4	5	4	7.76	-9.53
-3	5	4	2.85	1.96
-2	5	4	14.60	-14.10
-1	5	4	18.64	-17.01
0	5	4	8.19	-7.48
1	5	4	10.84	9.96
2	5	4	2.91	1.17
3	5	4	3.86	-1.10
4	5	4	6.86	9.20
5	5	4	6.45	-5.61
6	5	4	5.26	4.23
7	5	4	2.32	-1.76
8	5	4	1.03	-0.60
9	5	4	6.42	-7.16
-10	6	4	5.37	7.87
-9	6	4	3.01	-3.91
-8	6	4	4.37	4.05
-7	6	4	1.20	-1.88
-6	6	4	3.05	1.54
-5	6	4	4.37	-1.83
-4	6	4	17.13	-17.66
-3	6	4	21.21	-21.42
-2	6	4	9.80	-9.47
-1	6	4	4.64	-1.88
0	6	4	9.78	-8.75
1	6	4	5.00	-3.23
2	6	4	1.25	-3.04
3	6	4	15.79	13.24
4	6	4	7.08	7.66
5	6	4	6.41	7.03
6	6	4	4.23	5.51
7	6	4	1.99	4.37
8	6	4	6.98	9.83
-9	7	4	2.41	3.99
-8	7	4	1.74	1.68
-7	7	4	5.16	6.89
-6	7	4	1.11	1.00
-5	7	4	4.09	-3.82
-4	7	4	6.45	7.46
-3	7	4	10.52	-10.69
-2	7	4	1.25	3.01
-1	7	4	16.63	-16.30
0	7	4	4.69	-6.25
1	7	4	4.32	4.57
2	7	4	1.22	-1.28
3	7	4	6.32	5.73
4	7	4	3.22	2.94
5	7	4	5.92	6.42
6	7	4	5.35	7.84
7	7	4	2.92	5.61
-7	8	4	2.39	3.94
-6	8	4	1.71	-1.14
-5	8	4	4.29	7.17
-4	8	4	4.12	-4.26
-3	8	4	2.15	-1.53
-2	8	4	5.18	-6.11
-1	8	4	7.58	9.26
0	8	4	4.97	-6.73
1	8	4	5.77	-6.71
2	8	4	2.56	-4.41
3	8	4	7.75	-7.83
4	8	4	0.88	-2.30
5	8	4	8.08	-9.77
-12	0	5	2.41	1.81
-10	0	5	6.91	8.30
-8	0	5	17.09	16.59
-6	0	5	33.01	35.40
-4	0	5	19.14	-18.66
-2	0	5	26.42	-31.81
0	0	5	43.58	-44.97

Table 4 (*cont.*)

2	0	5	30.63	-35.14	-8	7	5	1.89	-0.05	-5	5	6	6.25	-6.75	-6	4	7	7.25	-4.19	-6	3	8	4.40	-4.29	-2	1	10	6.57	-0.31
4	0	5	20.25	21.16	-7	7	5	3.13	3.99	-4	5	6	10.11	-10.58	-5	4	7	9.39	9.94	-5	3	8	23.20	-25.02	-1	1	10	19.25	20.08
6	0	5	10.08	-10.90	-6	7	5	2.99	-3.69	-3	5	6	6.04	5.31	-4	4	7	18.27	-16.95	-4	3	8	6.91	-5.76	1	1	10	3.66	2.82
8	0	5	13.22	14.18	-5	7	5	4.86	6.51	-2	5	6	3.90	-2.46	-3	4	7	6.22	-4.05	-3	3	8	17.82	-17.65	2	1	10	4.94	-6.92
10	0	5	8.64	10.01	-4	7	5	5.71	5.50	-1	5	6	11.01	-12.20	-2	4	7	24.31	-23.93	-2	3	8	13.16	-12.80	3	1	10	1.34	-1.30
-12	1	5	2.51	-0.63	-3	7	5	2.75	-0.72	0	5	6	13.20	-15.11	-1	4	7	8.66	-7.76	-1	3	8	11.31	-10.20	4	1	10	1.31	0.18
-11	1	5	4.22	-1.87	-2	7	5	4.39	2.57	1	5	6	3.25	1.64	0	4	7	6.22	-5.29	0	3	8	5.91	2.95	5	1	10	6.15	7.15
-10	1	5	4.86	-4.13	-1	7	5	1.40	-0.87	2	5	6	4.06	-3.69	1	4	7	3.70	0.70	1	3	8	4.06	-4.63	6	1	10	7.56	6.28
-9	1	5	15.11	14.32	0	7	5	10.55	10.38	3	5	6	5.82	3.97	2	4	7	11.22	8.07	2	3	8	15.20	13.47	-10	2	10	2.86	1.74
-8	1	5	1.60	0.53	1	7	5	1.37	-2.57	4	5	6	3.36	3.25	3	4	7	1.71	2.95	3	3	8	3.65	1.20	-9	2	10	3.16	4.23
-7	1	5	3.05	0.62	2	7	5	7.11	-9.87	5	5	6	7.32	6.62	4	4	7	2.46	-0.43	4	3	8	1.88	0.92	-8	2	10	8.82	4.50
-6	1	5	4.71	4.35	3	7	5	5.78	-8.48	6	5	6	5.73	8.09	5	4	7	7.69	6.17	5	3	8	3.75	5.25	-7	2	10	2.76	3.97
-5	1	5	26.90	24.72	4	7	5	5.94	-4.95	7	5	6	4.69	4.30	6	4	7	6.94	6.23	6	3	8	3.57	3.15	-6	2	10	4.77	-5.41
-4	1	5	2.98	3.06	5	7	5	2.68	-1.39	8	5	6	5.77	6.94	7	4	7	2.08	0.63	7	3	8	6.01	6.02	-5	2	10	9.21	-6.70
-3	1	5	1.31	-0.59	6	7	5	2.61	-2.64	-8	6	6	5.61	6.39	-10	5	7	3.02	3.09	8	3	8	5.58	-5.41	-4	2	10	10.71	-12.28
-2	1	5	11.91	13.47	-7	8	5	3.56	5.64	-7	6	6	1.54	0.36	-9	5	7	3.59	-3.26	-10	4	8	7.48	7.04	-3	2	10	33.68	-31.31
-1	1	5	45.67	-52.95	-6	8	5	2.59	2.08	-6	6	6	13.39	16.18	-8	5	7	8.56	9.29	-9	4	8	5.45	7.21	-2	2	10	10.55	-10.86
0	1	5	5.91	6.21	-5	8	5	3.59	3.70	-5	6	6	5.77	6.67	-7	5	7	1.71	-0.38	-8	4	8	4.50	4.30	-1	2	10	21.29	-22.98
1	1	5	8.84	-7.74	-4	8	5	3.16	-4.59	-4	6	6	13.46	15.06	-6	5	7	6.95	6.77	-7	4	8	14.83	15.11	1	2	10	4.32	-1.49
2	1	5	9.58	-11.16	-3	8	5	6.61	8.64	-3	6	6	4.13	2.68	-5	5	7	8.84	7.52	-6	4	8	7.95	6.91	2	2	10	5.53	-4.52
3	1	5	14.88	-16.28	-2	8	5	2.39	2.49	-2	6	6	3.36	-3.45	-4	5	7	1.72	-1.85	-5	4	8	7.33	4.58	3	2	10	11.05	11.84
4	1	5	27.70	-29.79	-1	8	5	5.13	-5.69	-1	6	6	3.36	-5.43	-3	5	7	18.74	17.93	-4	4	8	5.06	-1.67	4	2	10	3.82	-2.26
5	1	5	7.59	7.07	0	8	5	2.93	-4.75	0	6	6	12.60	-12.43	-2	5	7	23.20	-22.47	-3	4	8	1.75	-0.10	5	2	10	15.55	14.00
6	1	5	25.38	-27.06	1	8	5	5.94	-6.64	1	6	6	1.68	-2.72	-1	5	7	15.79	15.84	-2	4	8	27.40	-31.71	6	2	10	1.14	0.48
7	1	5	13.37	11.82	2	8	5	5.65	-7.28	2	6	6	13.42	-14.28	0	5	7	28.27	-28.17	-1	4	8	5.47	-5.78	-10	0	11	1.82	-1.79
8	1	5	9.03	-7.50	3	8	5	4.57	-6.47	3	6	6	5.71	-5.98	1	5	7	9.09	-8.90	0	4	8	12.43	-9.18	-8	0	11	2.99	2.78
9	1	5	1.69	0.18	4	8	5	3.59	-5.08	4	6	6	3.18	-1.01	2	5	7	21.25	-20.77	1	4	8	13.24	-10.40	-6	0	11	8.12	-8.44
10	1	5	1.00	0.69	5	8	5	2.96	-5.86	5	6	6	4.67	2.09	3	5	7	2.45	0.51	2	4	8	8.67	-7.83	-4	0	11	8.77	-9.62
-11	2	5	6.45	-5.44	-12	0	6	1.68	2.03	6	6	6	3.20	2.36	4	5	7	2.41	-1.86	3	4	8	9.95	-7.07	-2	0	11	19.78	-22.00
-10	2	5	4.74	-4.94	-10	0	6	7.75	-7.46	7	6	6	6.01	5.84	5	5	7	3.25	1.98	4	4	8	12.15	10.42	2	0	11	1.54	1.94
-9	2	5	2.31	2.58	-8	0	6	10.78	9.20	-6	7	6	2.82	-0.46	6	5	7	3.60	5.87	5	4	8	6.76	-4.69	4	0	11	4.60	4.06
-8	2	5	1.67	-1.38	-6	0	6	10.95	10.85	-5	7	6	3.69	5.49	7	5	7	4.98	7.17	6	4	8	5.75	6.05	6	0	11	2.32	1.37
-7	2	5	4.23	4.93	-4	0	6	32.53	35.16	-4	7	6	6.65	-10.40	-9	6	7	2.91	5.26	7	4	8	2.85	4.09	-10	1	11	8.03	4.89
-6	2	5	9.87	10.14	-2	0	6	16.22	16.51	-3	7	6	4.53	5.58	-8	6	7	5.24	6.88	-9	5	8	3.05	2.66	-9	1	11	6.21	-3.34
-5	2	5	15.25	13.93	0	0	6	15.45	-16.12	-2	7	6	7.25	8.03	-7	6	7	3.09	1.94	-8	5	8	4.17	4.22	-8	1	11	5.17	1.64
-4	2	5	22.96	-21.06	2	0	6	4.87	2.62	-1	7	6	5.63	-6.64	-6	6	7	6.96	8.08	-7	5	8	3.63	3.11	-7	1	11	1.32	-2.60
-3	2	5	20.14	18.73	4	0	6	16.08	-16.06	0	7	6	4.56	-4.59	-5	6	7	6.79	5.28	-6	5	8	5.28	4.77	-6	1	11	3.77	-3.98
-2	2	5	3.59	-0.64	6	0	6	6.65	7.16	1	7	6	11.44	-12.99	-4	6	7	5.97	-5.53	-5	5	8	1.88	-3.09	-5	1	11	4.19	-3.74
-1	2	5	29.27	28.63	8	0	6	1.47	0.58	2	7	6	3.08	-1.27	-3	6	7	3.47	-2.79	-4	5	8	4.60	6.23	-4	1	11	11.45	-12.65
0	2	5	8.73	9.56	10	0	6	6.96	6.14	3	7	6	11.31	-14.63	-2	6	7	14.54	-13.96	-3	5	8	10.57	10.04	-3	1	11	1.85	-1.31
1	2	5	28.55	28.29	-12	1	6	3.76	-3.47	4	7	6	6.29	-5.62	-1	6	7	3.47	-0.48	-2	5	8	1.87	0.40	-2	1	11	17.69	-18.26
2	2	5	6.67	-5.68	-11	1	6	4.26	3.02	5	7	6	6.21	-7.62	0	6	7	4.24	-3.99	-1	5	8	5.27	4.73	-1	1	11	1.31	-1.39
3	2	5	5.85	-6.94	-10	1	6	11.49	-12.06	6	7	6	4.51	-5.36	1	6	7	11.45	-10.02	0	5	8	1.87	-1.56	1	1	11	6.24	6.22
4	2	5	10.75	10.94	-9	1	6	12.06	12.04	-5	8	6	5.21	6.57	2	6	7	4.16	2.59	1	5	8	8.42	-8.42	2	1	11	10.48	-10.45
5	2	5	2.32	0.01	-8	1	6	24.00	-25.68	-4	8	6	5.92	7.56	3	6	7	4.03	-3.37	2	5	8	3.76	-3.06	3	1	11	11.39	10.64
6	2	5	23.40	-23.24	-7	1	6	11.61	11.21	-3	8	6	5.63	7.69	4	6	7	7.92	-9.35	3	5	8	5.24	-3.83	4	1	11	2.49	0.09
7	2	5	5.55	-6.41	-6	1	6	16.26	-15.95	-2	8	6	1.35	1.72	5	6	7	2.82	0.49	4	5	8	5.63	-6.60	5	1	11	10.48	9.30
8	2	5	5.92	-5.69	-5	1	6	7.22	-5.22	-1	8	6	1.37	0.97	6	6	7	5.61	-6.43	5	5	8	1.65	0.11	-9	2	11	4.57	3.79
9	2	5	3.39	-2.87	-4	1	6	15.35	15.78	0	8	6	1.91	-0.30	-8	7	7	1.03	2.92	6	5	8	1.44	1.73	-8	2	11	5.26	3.69
10	2	5	3.89	-3.88	-3	1	6	15.70	-10.03	1	8	6	1.30	3.47	-7	7	7	1.24	1.53	7	5	8	6.17	7.59	-7	2	11	11.07	9.88
-11	3	5	5.73	-6.87	-2	1	6	25.65	26.44	2	8	6	2.44	-1.29	-6	7	7	2.79	3.64	-1	6	8	8.74	-9.75	-6	2	11	1.38	0.35
-10	3	5	3.77	-3.03	-1	1	6	8.73	-8.24	3	8	6	3.79	-7.07	-5	7	7	4.23	3.09	0	6	8	12.29	11.65	-5	2	11	3.92	-1.33
-9	3	5	9.97	-10.57	0	1	6	1.79	0.48	-12	0	7	4.30	-3.59	-4	7	7	6.99	10.42	1	6	8	6.34	-6.85	-4	2	11	6.75	8.80
-8	3	5	2.82	4.86	1	1	6	2.73	-0.46	-10	0	7	2.21	1.77	-3	7	7	7.53	8.85	2	6	8	6.65	-8.61	-3	2	11	11.64	-9.13
-7	3	5	33.82	-32.97	2	1	6	22.87	-22.22	-8	0	7	20.21	-20.26	-2	7	7	1.62	2.22	3	6	8	7.18	-8.33	-2	2	11	1.37	3.10
-6	3	5	9.36	10.42	3	1	6	29.05	-29.08	-6	0	7	5.21	3.50	-1	7	7	3.97	5.41	4	6	8	9.60	-11.18	-1	2	11	13.47	-13.00
-5	3	5	3.35	-3.39	4	1	6	17.39	-14.09	-4	0	7	24.21	27.03	0	7	7	3.22	3.64	5	6	8	6.08	-7.20	1	2	11	2.76	0.23
-4	3	5	8.83	8.69	5	1	6	17.62	-17.35	-2	0	7	18.27	19.98	1	7	7	3.85	-3.97	6	6	8	8.36	-11.75	2	2	11	3.36	-1.85
-3	3	5	10.48	11.13	6	1	6	6.58	-6.23	0	0	7	6.45	5.99	2	7	7	3.01	-2.75	-3	7	8	8.49	10.08	3	2	11	10.68	8.13
-2	3	5	7.58	6.41	7	1	6	10.71	-10.61	2	0	7	39.91	-44.39	3	7	7	4.43	-5.80	-2	7	8	3.36	3.84	4	2	11	3.56	0.06
-1	3	5	26.25	25.14	8	1	6	8.82	-8.13	4	0	7	23.71	-22.46	4	7	7	4.34	-7.13	-1	7	8	11.88	11.33	5	2	11	4.26	3.01
0	3	5	5.82	5.85	9	1	6	7.35	5.17	6	0	7	3.08	-0.75	5	7	7	4.87	-10.45	0	7	8	5.71	6.20	-10	0	12	7.93	5.67
1	3	5	49.01	46.87	-11	2	6	5.80	-6.50	8	0	7	7.31	-7.56	-5	8	7	3.50	-3.80	1	7	8	3.19	2.96	-8	0	12	9.73	-8.47
2	3	5	1.11	3.38	-10	2	6	6.70	-5.15	-11	1	7	6.49	-6.54	-4	8	7	1.21	2.03	2	7	8	1.50	2.42	-6	0	12	13.81	-15.28
3	3	5	21.51	19.16	-9	2	6	2.63	-0.35	-10	1	7	9.37	-7.89	-3	8	7	3.13	-3.60	3	7	8	2.73	0.88	-4	0	12	34.59	-33.37
4	3	5	10.72	-10.68	-8	2	6	2.22	-1.08	-9	1	7	3.02	-1.81	-2	8	7	2.62	1.27	-10	0	9	4.04	-2.19	-2	0	12	5.31	-4.42
5	3	5	5.78	4.99	-7	2	6	10.27	12.63	-8	1	7	10.95	-9.26	-1	8	7	7.65	10.02	-8	0	9	2.16	0.27	2	0	12	6.68	4.66
6	3	5	18.40	-17.02	-6	2	6	16.39	-14.02	-7	1	7	22.06	19.65	0	8	7	4.06	4.72	-6	0	9	2.92	-3.34	4	0	12	10.88	9.95
7	3	5	1.41	-5.84	-5	2	6	14.30	11.38	-6	1	7	2.91	2.94	1	8	7	3.45	3.27	-4	0	9	3.32	5.10	-9	1	12	6.41	3.29
8	3	5	2.63	-3.79	-4	2	6	10.44	-9.45	-5	1	7	10.62	9.49	2	8	7	2.22	2.68	-2	0	9	20.81	23.41	-8	1	12	2.46	5.01
-10	4	5	3.03	0.77	-3	2	6	39.58	38.38	-4	1	7	15.41	15.01	3	8	7	3.28	5.09	2	0	9	24.10	31.62	-7	1	12	5.16	4.62
-9	4	5	5.78	5.05	-2	2	6	13.05	10.72	-3	1	7	27.32	27.31	-12	0	8	7.75	-6.72	4	0	9	1.54	1.21	-6	1	12	1.87	1.42
-8	4	5	3.46	1.76	-1	2	6	51.61	60.81	-2	1	7	18.63	19.02	-10	0	8	4.88	-3.93	6	0	9	1.45	-0.01	-5	1	12	1.88	0.62
-7	4	5	2.81	-0.73	0	2	6	25.73	21.93	-1	1	7	17.37	17.73	-8	0	8	3.85	-5.02	8	0	9	3.73	-3.88	-4	1	12	2.66	-4.10
-6	4	5	4.30	3.14	1	2	6	39.21	38.09	0	1	7	11.25	10.19	-6	0	8	6.55	0.78	-11	1	9	1.08	2.19	-3	1	12	3.77	-1.18
-5	4	5	15.50	-15.93	2	2	6	19.14	19.23	1	1	7	14.83	-14.37	-4	0	8	23.43	25.52	-10	1	9	1.22	0.76	-2	1	12	4.22	-3.34
-4	4	5	3.03	2.40	3	2	6	2.88	3.96	2	1	7	9.84	9.49	-2	0	8	17.45	18.43	-9	1	9	9.44	-8.17	-1	1	12	15.20	-13.72
-3	4	5	17.23	-16.20	4	2	6	4.10	-5.07	3	1	7	4.63	4.48	0	0	8	17.94	18.70	-8	1	9	9.97	-8.44	1	1	12	4.16	2.39
-2	4	5	22.25	24.36	5	2	6	25.75	-26.56	4	1	7	12.23	10.80	2	0	8	20.84	-19.60	-7	1	9	11.01	-6.81	2	1	12	1.82	-2.69
-1	4	5	9.03	-8.31	6	2	6	2.88	2.88	5	1	7	26.76	-27.51	4	0	8	22.32	-22.04	-6	1	9	14.10	-12.92	3	1	12	2.45	1.64
0	4	5	15.72	17.22	7	2	6	15.17	-15.93	6	1	7	2.14	-2.85	6	0	8	14.04	-15.20	-5	1	9	11.82	-11.35	4	1	12	4.77	-4.42
1	4	5	16.19	15.16	8	2	6	6.25	-4.76	7	1	7	2.24	-4.05	8	0	8	4.12	-5.01	-4	1	9	17.06	-17.12	-9	2	12	4.20	2.57
2	4	5	4.66	2.50	9	2	6	6.20	-6.34	8	1	7	4.09	-5.86	-11	1	8	6.57	-6.47	-3	1	9	5.23	1.08	-8	2	12	3.50	-0.26
3	4	5	4.53	-3.28	-10	3	6	3.13	2.06	-11	2	7	3.39	-5.12	-10	1	8	1.72	1.21	-2	1	9	1.15	2.88	-7	2	12	4.53	1.13
4	4	5	9.47	7.64	-9	3	6	4.09	-2.06	-10	2	7	8.57	9.09	-9	1	8	3.77	-2.18	-1	1	9	12.11	10.06	-6	2	12	9.74	10.53
5	4	5	3.45	1.62	-8	3	6	9.20	-9.41	-9	2	7	2.18	-2.56	-8	1	8	2.72	4.54	1	1	9	6.92	6.75	-5	2	12	10.08	9.76
6	4	5	1.41	-1.98	-7	3	6	4.60	-4.08	-8	2	7	11.78	-9.91	-7	1	8	2.54	5.76	2	1	9	13.17	16.37	-4	2	12	2.76	1.25
7	4	5	3.82	-0.76	-6	3	6	7.22	-5.61	-7	2	7	11.59	-10.36	-6	1	8	7.06	-4.68	3	1	9	1.85	-0.76	-3	2	12	3.92	2.96
8	4	5	3.85	-0.00	-5	3	6	1.44	-1.16	-6	2	7	8.89	-4.99	-5	1	8	8.26	10.32	4	1	9	16.63	17.62	-2	2	12	11.56	10.98
-10	5	5	3.39	3.77	-4	3	6	10.30	9.55	-5	2	7	2.18	-1.12	-4	1	8	11.04	-11.71	5	1	9	2.63	-2.29	1	2	12	10.97	-12.40
-9	5	5	3.53	3.67	-3	3	6	8.49	7.60	-4	2	7	4.33	5.00	-3	1	8	3.01	-2.96	6	1	9	3.06	3.82	2	2	12	3.69	-3.43
-8	5	5	1.35	-0.08	-2	3	6	10.20	12.07	-3	2	7	1.38	0.06	-2	1	8	11.74	-9.97	7	1	9	7.89	-6.62	3	2	12	6.74	-6.98
-7	5	5	6.31	-6.49	-1	3	6	15.99	-15.54	-2	2	7	1.68	2.85	-1	1	8	9.36	-7.79	-10	2	9	4.61	-2.37	4	2	12	2.24	-0.41
-6	5	5	11.14	-13.00	0	3	6	9.73	-5.22	-1	2	7	8.55	7.34	0	1	8	22.62	18.15	-9	2	9	1.34	0.69	-8	0	13	1.88	0.03
-5	5	5	7.11	-7.28	1	3	6	10.85	10.96	0	2	7	9.36	6.85	1	1	8	10.44	7.72	-8	2	9	5.53	3.64	-6	0	13	5.08	-2.76
-4	5	5	18.04	-18.16	2	3	6	7.66	-6.47	1	2	7	20.22	23.59	2	1	8	33.77	31.31	-7	2	9	10.13	-7.80	-4	0	13	1.51	-0.78
-3	5	5	23.91	-27.06	3	3	6	12.86	15.16	2	2	7	6.89	-5.52	3	1	8	13.63	14.09	-6	2	9	1.91	-1.39	-2	0	13	4.29	-3.89
-2	5	5	19.82	-17.83	4	3	6	6.74	5.93	3	2	7	2.83	4.40	4	1	8	13.34	15.20	-5	2	9	11.88	-10.15	2	0	13	4.77	-5.01
-1	5	5	11.34	-13.84	5	3	6	13.70	14.11	4	2	7	9.10	6.87	5	1	8	6.42	5.55	-4	2	9	8.27	-7.42	4	0	13	1.13	-0.78
0	5	5	9.44	7.62	6	3	6	12.83	12.78	5	2	7	3.93	-3.92	6	1	8	5.63	5.68	-3	2	9	3.96	-1.76	-9	1	13	6.28	3.35
1	5	5	3.26	1.85	7	3	6	3.99	0.90	6	2	7	20.64	18.47	7	1	8	10.33	-9.22	-2	2	9	4.64	-6.05	-8	1	13	9.07	8.95
2	5	5	16.71	14.19	8	3	6	1.48	1.37	7	2	7	9.46	-8.24	8	1	8	4.44	-4.23	-1	2	9	4.33	-5.01	-7	1	13	1.71	-1.47
3	5	5	5.23	6.06	-10	4	6	2.89	-3.36	8	2	7	4.41	4.63	-11	2	8	6.15	4.51	1	2	9	1.31	-0.40	-6	1	13	16.75	12.96
4	5	5	20.52	22.39	-9	4	6	5.55	2.57	9	2	7	4.69	-4.30	-10	2	8	5.51	3.48	2	2	9	6.01	6.13	-5	1	13	1.30	1.72
5	5	5	5.63	7.73	-8	4	6	7.11	-7.18	-11	3	7	4.36	4.75	-9	2	8	9.09	-10.49	3	2	9	2.75	0.59	-4	1	13	10.45	10.17
6	5	5	10.27	12.64	-7	4	6	1.68	-2.97	-10	3	7	5.53	-4.58	-8	2	8	3.20	0.58	4	2	9	6.49	-6.36	-3	1	13	5.24	-4.45
7	5	5	3.02	3.69	-6	4	6	17.02	-16.53	-9	3	7	1.71	-0.85	-7	2	8	18.47	-18.45	5	2	9	1.91	3.15	-2	1	13	1.31	1.07
8	5	5	4.91	3.41	-5	4	6	14.26	-14.96	-8	3	7	6.51	-6.58	-6	2	8	8.17	5.07	6	2	9	1.27	-1.48	-1	1	13	1.30	-2.44
-9	6	5	2.56	-0.39	-4	4	6	23.30	-21.73	-7	3	7	3.40	-4.53	-5	2	8	28.28	-23.65	7	2	9	6.81	7.69	1	1	13	4.64	-4.70
-8	6	5	1.21	0.63	-3	4	6	16.62	-16.94	-6	3	7	3.25	-1.68	-4	2	8	14.00	12.67	-10	0	10	5.13	-6.03	2	1	13	6.67	-4.93
-7	6	5	2.63	1.32	-2	4	6	5.71	-3.82	-5	3	7	23.93	-26.35	-3	2	8	9.17	-5.64	-8	0	10	4.84	-0.29	-8	2	13	2.25	0.72
-6	6	5	3.39	2.34	-1	4	6	10.44	-9.12	-4	3	7	2.89	1.31	-2	2	8	8.23	-8.43	-6	0	10	10.87	0.96	-7	2	13	2.44	-0.24
-5	6	5	10.74	-10.05	0	4	6	19.54	19.51	-3	3	7	26.50	-24.28	-1	2	8	18.41	18.77	-4	0	10	5.75	0.29	-5	2	13	4.16	0.46
-4	6	5	3.47	4.53	1	4	6	10.07	-8.93	-2	3	7	5.68	4.54	0	2	8	7.36	8.56	-2	0	10	11.64	14.18	-4	2	13	2.66	-1.73
-3	6	5	11.82	-9.92	2	4	6	18.00	16.97	-1	3	7	6.82	-4.66	1	2	8	17.02	15.06	2	0	10	20.52	22.53	-3	2	13	9.67	7.26
-2	6	5	11.25	12.65	3	4	6	5.57	-2.18	0	3	7	5.50	-2.53	2	2	8	1.72	1.17	4	0	10	6.81	7.84	1	2	13	10.33	-8.93
-1	6	5	19.04	-19.02	4	4	6	12.68	11.33	1	3	7	4.07	-2.45	3	2	8	14.18	13.12	6	0	10	7.56	-7.19	2	2	13	4.40	-3.08
0	6	5	10.14	-9.67	5	4	6	3.38	2.23	2	3	7	10.13	6.01	4	2	8	11.36	10.51	-11	1	10	2.01	1.12	3	2	13	7.99	-7.14
1	6	5	2.82	1.97	6	4	6	4.03	4.67	3	3	7	25.79	28.56	5	2	8	3.42	2.52	-10	1	10	3.32	2.01	-8	0	14	7.83	5.64
2	6	5	18.80	-17.78	7	4	6	10.40	7.88	4	3	7	21.72	18.50	6	2	8	3.13	2.81	-9	1	10	8.83	-5.43	-6	0	14	13.32	13.58
3	6	5	6.61	6.98	8	4	6	6.22	-4.59	5	3	7	7.38	8.09	7	2	8	2.14	-1.14	-8	1	10	1.32	-0.98	-4	0	14	9.49	8.70
4	6	5	7.79	-6.65	-10	5	6	1.24	3.35	6	3	7	3.43	[illegible].35	8	2	8	2.07	-0.08	-7	1	10	18.36	-19.09	-2	0	14	11.08	-11.05
5	6	5	6.68	6.58	-9	5	6	1.45	-1.40	7	3	7	5.54	3.34	-10	3	8	6.74	-6.52	-6	1	10	3.22	-4.68	2	0	14	5.60	-3.26
6	6	5	3.38	2.03	-8	5	6	3.19	3.08	-9	4	7	3.26	3.32	-9	3	8	3.66	1.76	-5	1	10	6.04	1.01	1	1	14	4.41	-3.48
7	6	5	3.55	4.86	-7	5	6	7.08	-7.21	-8	4	7	1.72	-1.31	-8	3	8	5.33	1.30	-4	1	10	4.37	3.52	-5	2	14	8.86	3.93
-9	7	5	5.23	8.78	-6	5	6	3.38	1.98	-7	4	7	1.74	-0.48	-7	3	8	9.50	-8.65	-3	1	10	4.32	0.19	-4	2	14	4.93	2.65

clearly indicates the puckered conformation of the ring. The planarity of the ring may be seen from Table 5. The angle between the normals of the two planes formed by C(5), C(6), C(12) and C(12), C(13), C(5) is 33° and that between the planes C(6), C(12), C(13) and C(13), C(5), C(6) is 34°. The exocyclic bond angles are all significantly larger than a normal tetrahedral angle, the largest being 131° at C(11)–C(12)–C(6).

The structure of the four-membered ring may be compared with that of octachlorocyclobutane, C_4Cl_8 (Owen & Hoard, 1951). In this compound, the cyclobutane ring has a mirror plane, involving a diagonal of the ring, and perpendicular to the molecular plane. The bond lengths and endocyclic bond angles were found to be 1·60 Å, 1·58 Å, 87° and 89°, respectively. The ring is puckered, the angle between the normals of the two planes related by the mirror plane being about 22°. It should be noted that the bond lengths found in every cyclobutane ring are slightly longer than the normal C–C single bond length and the bond angles are appreciably smaller than the tetrahedral angle.

The five-membered B ring formed by C(1), C(2), C(7), C(8) and C(9).

As is usually observed in five-membered rings, the four atoms C(1), C(2), C(8) and C(9) are situated nearly on a plane while C(7) is displaced by 0·667 Å from the plane. The bond angles involved in the ring are approximately 100° except the angles subtended at C(8) and C(1), the former being the sp^2 carbon.

The six-membered C ring formed by C(2), C(3), C(4), C(5), C(6) and C(7).

As shown in Table 5, this six-membered ring contains a double bond at C(3)=C(4) and takes a deformed boat conformation, the chlorine atom being in an axial position with respect to the ring.

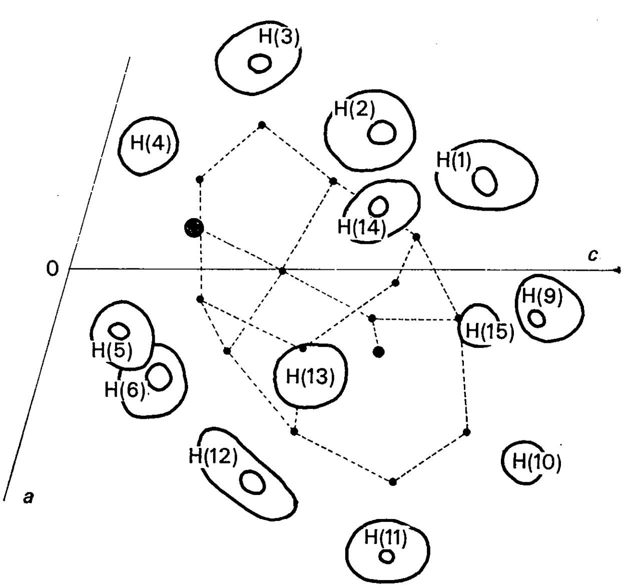


Fig. 3. Composite projection of the difference electron density map along the *b* axis showing the peaks of hydrogen atoms. Contours are drawn at 0·25, 0·5, ... e.Å^{-3}.

Table 5. *Planarity, bond lengths*

	Four-membered *A* ring		Five-membered *B* ring		Six-membered *C* ring	
Best plane formed by	C(5)	0·163 Å	C(1)	0·074	C(3)*	0·072
	C(6)	−0·165	C(2)	−0·050	C(4)*	−0·069
	C(12)	0·162	C(8)*	0·050	C(6)	0·057
	C(13)	−0·160	C(9)	−0·074	C(7)	−0·060
Perpendicular distances from the best plane			C(7)	0·667	C(2)	0·581
					C(5)	0·306
Endo-cyclic bond angles	C(5)—C(6)—C(12)	88°	C(1)—C(2)—C(7)	101	C(2)—C(3)*=C(4)*	117
	C(6)—C(12)-C(13)	87	C(2)—C(7)—C(8)*	100	C(3)*=C(4)*-C(5)	121
	C(12)-C(13)-C(5)	87	C(7)—C(8)*-C(9)	110	C(4)*-C(5)—C(6)	116†
	C(13)-C(5)—C(6)	88	C(8)*-C(9)—C(1)	101	C(5)—C(6)—C(7)	111†
			C(9)—C(1)—C(2)	106	C(6)—C(7)—C(2)	114
					C(7)—C(2)—C(3)*	113
Bond lengths	C(5)—C(6)	1·56 Å	C(1)—C(2)	1·55	C(2)—C(3)*	1·48
	C(6)—C(12)	1·56	C(2)—C(7)	1·52	C(3)*=C(4)*	1·29
	C(12)-C(13)	1·57	C(7)—C(8)*	1·48	C(4)*-C(5)	1·55
	C(13)-C(5)	1·58	C(8)*-C(9)	1·52	C(5)—C(6)	1·54
			C(9)—C(1)	1·56	C(6)—C(7)	1·55
					C(7)—C(2)	1·52

The seven-membered D ring formed by C(1), C(9), C(10), C(11), C(12), C(13) and C(14).

As seen in Fig. 2(*b*), this ring takes a deformed boat conformation. The perpendicular distances of the atoms from the best plane formed by the four atoms are shown in Table 5. A rather large bond angle of 119° is found at the C(1)–C(14)–C(13) bond, indicating an appreciable strain.

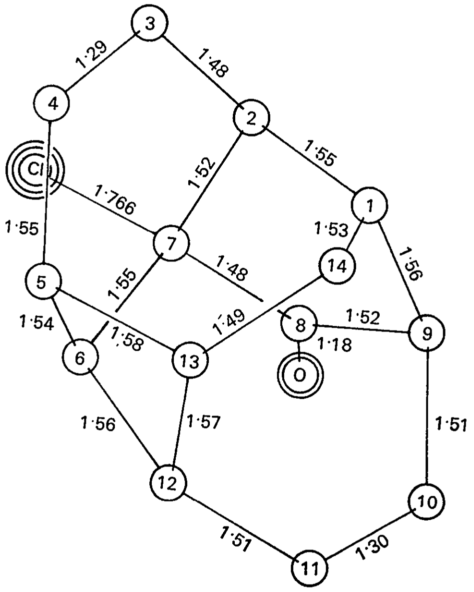


Fig. 4. Bond lengths (Å).

The seven-membered E ring formed by C(6), C(7), C(8), C(9), C(10), C(11) and C(12).

This part of the molecule is originated from 2-chlorotropone, and contains a double bond at C(10)=C(11). A chlorine atom and a ketone group are attached to C(7) and C(8), respectively. Conformation of the ring cannot be easily understood but may be described as being composed of a planar group formed by C(10)=C(11)–C(12)–C(6)–C(7) and a carbon chain C(8)–C(9) attached to the planar group at C(10) and C(7). As shown in Table 5, the carbon atoms C(8) and C(9) are situated at the opposite sides of the best plane formed by the planar group.

The seven-membered F ring formed by C(1), C(2), C(3), C(4), C(5), C(13) and C(14).

This part of the molecule is originated from cycloheptatriene. One of the three double bonds involved in cycloheptatriene is still maintained in C(3)=C(4). The conformation of the ring may be considered to be one of the boat forms; C(3)=C(4) and C(14) are situated away from the seven-membered *E* ring described above.

The projection of the crystal structure viewed along the *a* axis is shown in Fig. 6. Fig. 7 is another projection of the structure viewed along the *b* axis, in which all of the intermolecular short contacts of less than 3·8 Å are shown. All the packing distances are normal van der Waals separations.

and valency angles of the rings

Seven-membered *D* ring		Seven-membered *E* ring		Seven-membered *F* ring	
C(1)	−0·129	C(6)	−0·069	C(1)	−0·004
C(9)	0·113	C(7)	0·000	C(2)	0·004
C(12)	−0·100	C(10)*	0·073	C(5)	−0·004
C(13)	0·125	C(11)*	−0·162	C(13)	0·004
		C(12)	0·158		
C(10)*	1·504	C(8)*	−0·530	C(3)*	1·109
C(11)*	0·919	C(9)	0·528	C(4)*	1·106
C(14)	0·581			C(14)	0·546
C(1)—C(9)—C(10)*	117	C(6)—C(7)—C(8)*	112	C(1)—C(2)—C(3)*	119
C(9)—C(10)*-C(11)*	124	C(7)—C(8)*—C(9)	110	C(2)—C(3)*-C(4)*	117
C(10)*-C(11)*-C(12)	123	C(8)*—C(9)—C(10)*	104	C(3)*-C(4)*-C(5)	121
C(11)*-C(12)—C(13)	115†	C(9)—C(10)*-C(11)*	124	C(4)*-C(5)—C(13)	113†
C(12)—C(13)—C(14)	113†	C(10)*-C(11)*-C(12)	123	C(5)—C(13)-C(14)	117†
C(13)—C(14)—C(1)	119	C(11)*-C(12)—C(6)	131†	C(13)-C(14)-C(1)	119
C(14)—C(1)—C(9)	115	C(12)-C(6)—C(7)	118†	C(14)-C(1)—C(2)	110
C(1)—C(9)	1·56	C(6)—C(7)	1·55	C(1)—C(2)	1·55
C(9)—C(10)*	1·51	C(7)—C(8)*	1·48	C(2)—C(3)*	1·48
C(10)*=C(11)*	1·30	C(8)*—C(9)	1·52	C(3)*=C(4)*	1·29
C(11)*-C(12)	1·51	C(9)—C(10)*	1·51	C(4)*—C(5)	1·55
C(12)—C(13)	1·57	C(10)*=C(11)*	1·30	C(5)—C(13)	1·58
C(13)—C(14)	1·49	C(11)*-C(12)	1·51	C(13)—C(14)	1·49
C(14)—C(1)	1·53	C(12)—C(6)	1·56	C(14)—C(1)	1·53

* sp^2 configuration.

† Exocyclic bond angles of the four-membered ring.

The magnitudes and direction cosines of the principal axes of thermal ellipsoids are listed in Table 6.

Table 6. *Magnitudes and orientations of the principal axes of thermal ellipsoids*

R.m.s. displacement (Å) is directed along the *j*th axis of the ellipsoid where q_{1j}, q_{2j} and q_{3j} are the angles between the *j*th axis and the *a*, *c** and *b* axes, respectively.

	axis	r.m.s.$_j$ (Å)	q_{1j}	q_{2j}	q_{3j}
Cl	1	0·162	−0·3353	−0·7400	−0·5831
	2	0·255	−0·8464	0·5084	−0·1586
	3	0·286	0·4138	0·4403	−0·7968
O	1	0·169	−0·2112	0·9669	−0·1433
	2	0·283	0·6048	0·2444	0·7580
	3	0·322	−0·7679	−0·0734	0·6364
C(1)	1	0·158	0·1169	0·9767	0·1787
	2	0·223	0·8979	−0·1808	0·4012
	3	0·282	−0·4243	−0·1136	0·8984
C(2)	1	0·178	−0·8782	−0·3466	0·3297
	2	0·225	−0·2221	−0·3150	−0·9228
	3	0·231	−0·4237	−0·8835	−0·1996
C(3)	1	0·217	−0·5398	0·7598	−0·3625
	2	0·245	−0·1116	0·3622	0·9254
	3	0·260	0·8344	0·5400	−0·1108
C(4)	1	0·175	−0·5897	0·6672	−0·4551
	2	0·271	−0·0207	−0·5758	−0·8173
	3	0·322	−0·8073	−0·4726	0·3534
C(5)	1	0·193	0·4252	−0·8852	0·1885
	2	0·258	−0·6377	−0·4408	−0·6317
	3	0·305	0·6423	0·1484	−0·7520
C(6)	1	0·182	0·8648	0·4604	0·2006
	2	0·199	−0·3829	0·8629	−0·3297
	3	0·267	−0·3249	0·2083	0·9225
C(7)	1	0·165	0·4331	−0·2488	−0·8664
	2	0·174	−0·5670	−0·8223	−0·0473
	3	0·229	−0·7007	0·5117	−0·4972
C(8)	1	0·171	−0·6838	−0·7232	−0·0967
	2	0·246	−0·6749	0·6773	−0·2929
	3	0·267	0·2773	−0·1350	−0·9512
C(9)	1	0·164	0·1167	0·7101	0·6944
	2	0·222	0·6733	−0·5705	0·4702
	3	0·293	−0·7301	−0·4127	0·5447
C(10)	1	0·181	−0·2610	0·9244	0·2780
	2	0·222	0·5224	−0·1069	0·8460
	3	0·287	−0·8118	−0·3660	0·4550
C(11)	1	0·167	0·1038	−0·8658	−0·4895
	2	0·230	−0·9173	0·1069	−0·3836
	3	0·290	0·3844	0·4889	−0·7831
C(12)	1	0·162	−0·0164	0·9943	0·1053
	2	0·229	0·8894	−0·0352	0·4557
	3	0·294	−0·4568	−0·1003	0·8839
C(13)	1	0·202	−0·6688	−0·0495	0·7418
	2	0·218	−0·1061	0·9939	−0·0294
	3	0·279	0·7358	−0·0983	0·6700
C(14)	1	0·202	−0·1729	0·9728	0·1541
	2	0·226	0·9466	0·2073	−0·2467
	3	0·281	0·2720	−0·1032	0·9567

A perspective drawing of the molecule along the *b* axis is shown in Fig. 8 which is drawn by a CALCOMP plotter with the use of the *ORTEP* program (Johnson, 1965). In the Figure, the atoms are represented by ellipsoids of thermal displacement including 50% probability.

The authors are greatly indebted to C. Itoh Electronic Computing Service for use of a CDC 3600. This work was supported by a Research Grant from Hohansha.

References

Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.

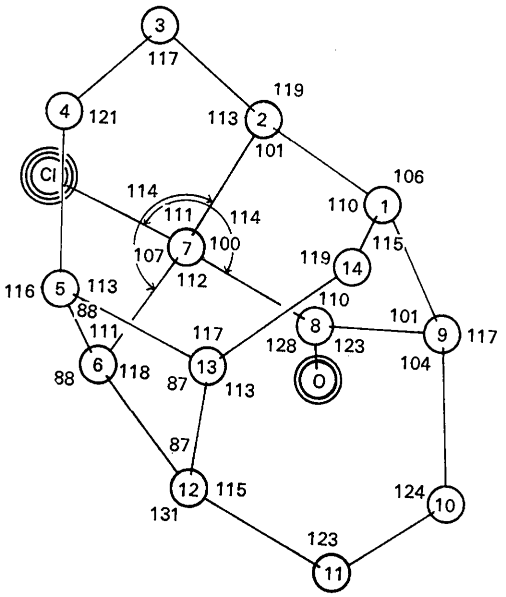


Fig. 5. Bond angles (°).

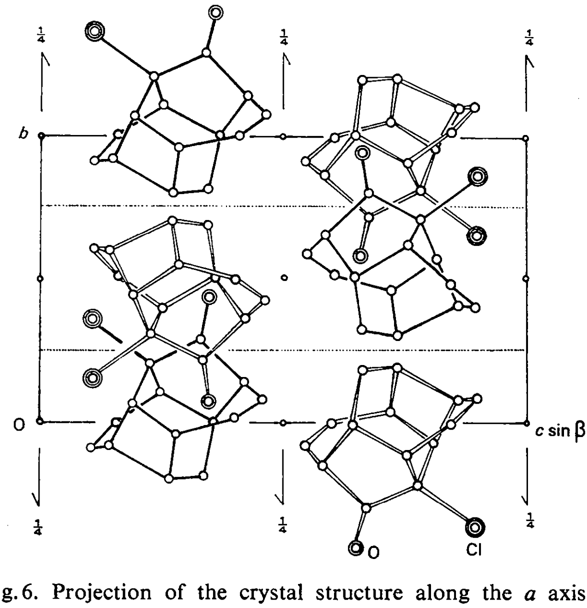


Fig. 6. Projection of the crystal structure along the *a* axis.

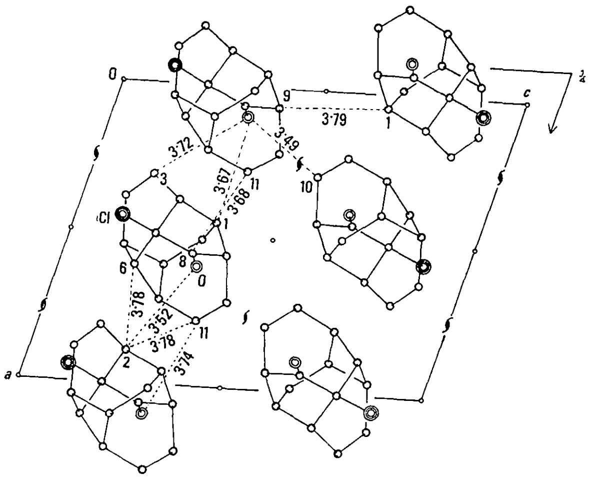


Fig. 7. Projection of the crystal structure along the *b* axis. Intermolecular short distances less than 3·8 Å are shown by broken lines.

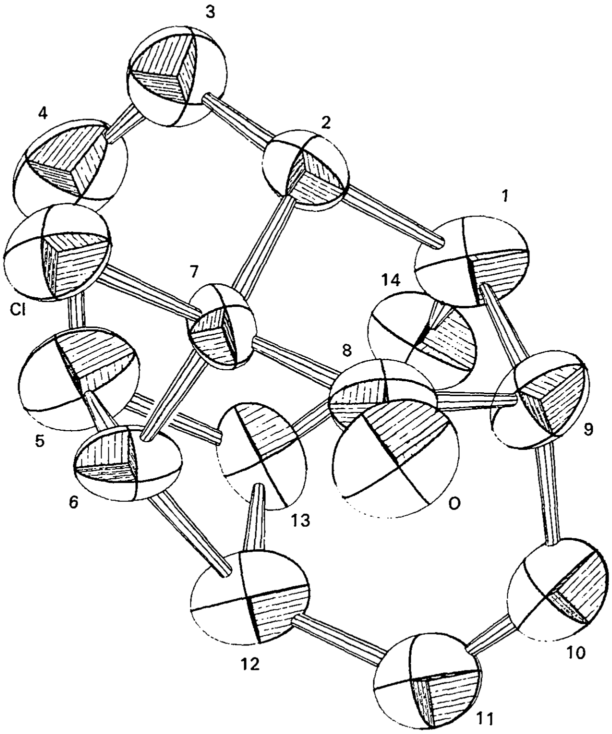


Fig. 8. Projection of the molecular structure along the *b* axis showing the ellipsoids of thermal vibration. The centres of atoms are included in the ellipsoids with 50% probability.

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Dawson, B. (1960). *Acta Cryst.* **13**, 403.

Ito, S., Fujise, Y. & Woods, M. C. (1967). *Tetrahedron Letters*, 1059.

Johnson, C. K. (1965). ORTEP, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Okaya, Y. & Ashida, T. (1967). *HBLS* IV, *The Universal Crystallographic Computing System* (I), p. 65. Japanese Crystallographic Association.

Owen, T. B. & Hoard, J. L. (1951). *Acta Cryst.* **4**, 172.

Sato, M., Fujise, Y. & Ito, S. (1967). Presented at the Third Symposium on Organic Mass Spectrometry, Oct. 12th, 1967 at Kyoto. Abstract p.25.

Acta Cryst. (1969). B**25**, 673

Crystallography of Pyrrhotite

By Bibhuti Mukherjee

Geological Survey of India, Calcutta 13, *India*

(*Received* 8 *May* 1968)

Pyrrhotite crystals (Fe_7S_8) from the sulphide ore of the Mosaboni mine, India, are found to have a monoclinic supercell, $a_0 = 11{\cdot}90$, $b_0 = 6{\cdot}87$, $c_0 = 22{\cdot}88$ Å, $\beta = 90°30'$, $Z = 8$ with density 4·60 g.cm^{-3}, and the space group $C2/c$ (or Cc). The hexagonal supercell, $a_0 = 6{\cdot}88$, $c_0 = 22{\cdot}90$ Å, $Z = 4$ with density 4·58 g.cm^{-3}, is also observed in pyrrhotite crystals, and the space group $P6/mcc$ (or $P6cc$) has been assigned to the hexagonal phase.

Buerger (1947) proposed a hexagonal superstructure for pyrrhotite with $a = 6{\cdot}87$, $c = 22{\cdot}7$ Å; this supercell has 16 times the volume of the Ni–As-type cell. Moreover, he suggested that the observed diffraction effect in precession photographs is probably due to twinned aggregates and that the true symmetry is perhaps monoclinic or orthorhombic. Bertaut (1953) postulated that the pyrrhotite twin is composed of two monoclinic crystals, related by a 60° rotation about the pseudo-hexagonal *c* axis and he proposed a unit cell with the parameters 11·9, 6·865, 22·72 Å, $\beta = 90{\cdot}45°$ (Byström, 1945) and the space group $F2/d$. Wuensch (1963) observed that the pyrrhotite lattice is at least dimensionally monoclinic (with $\beta = 91{\cdot}79°$) and is twinned by a