Acta Cryst. (1967). **23**, 654

The Crystal Structure of 2-(*o*-Hydroxyphenyl)-1-phenylpropanesulphonic Acid Sultone (Sultone–B)

By Karin Bjåmer* and George Ferguson

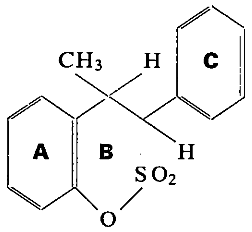
Chemistry Department, University of Glasgow, Glasgow, W.2, Scotland

(*Received* 14 *November* 1966)

The crystal structure of 2-(*o*-hydroxyphenyl)-1-phenylpropanesulphonic acid sultone (sultone-B), $C_{15}H_{14}O_3S$, has been determined from three-dimensional photographic data. The crystals are monoclinic with unit-cell dimensions $a=11{\cdot}18$, $b=10{\cdot}70$, $c=14{\cdot}42$ Å; $\beta=129{\cdot}6°$. The space group is $P2_1/c$. The structure has been refined by least-squares methods and the final residual (R) for 2063 independently measured reflexions is 9·8%. The heterocyclic ring is in the half-chair conformation with the phenyl group axial and *cis* to the methyl group, which is quasi-equatorial. Some bond lengths are: S–O (peripheral) 1·431 ± 0·003, S–O(C) 1·595 ± 0·004, S–C 1·797 ± 0·005 and C^2_{sp}–O 1·426 ± 0·005 Å.

Introduction

The synthesis of 2-(*o*-hydroxyphenyl)-1-phenylpropanesulphonic acid sultone (I) by Timoney (1966) and co-workers yielded equal amounts of two isomers; one with m.p. 106–108 °C (sultone-B) and the other with m.p. 158–160 °C (sultone-C). The former is converted entirely to sultone-C when it is heated under reflux with 10% sodium hydroxide. Timoney suggested that in one of the isomers the methyl group is *cis* to the phenyl group and that in the second isomer they are *trans* to each other.



(I)

It was not possible to distinguish these chemically. The crystals of sultone-B are monoclinic while those of sultone-C are triclinic, and the former were chosen for the crystal structure analysis to determine the configuration and accurate molecular dimensions.

Crystal data

2-(*o*-Hydroxyphenyl)-1-phenylpropanesulphonic acid sultone (sultone-B), $C_{15}H_{14}O_3S$, $M=274{\cdot}3$.
Monoclinic, $a=11{\cdot}18 \pm 0{\cdot}01$, $b=10{\cdot}70 \pm 0{\cdot}01$, $c=14{\cdot}42 \pm 0{\cdot}01$ Å, $\beta=129{\cdot}6 \pm 0{\cdot}2°$.
$U=1329{\cdot}0$ Å^3. $D_m=1{\cdot}374$ g.cm^{-3} (by flotation), $Z=4$, $D_x=1{\cdot}371$ g.cm^{-3}. $F(000)=576$.
Space group $P2_1/c$ (C^5_{2h}) from systematic absences. Absorption coefficient for X-rays ($\lambda=1{\cdot}5418$ Å) $\mu=21{\cdot}1$ cm^{-1}.

Experimental

Oscillation and Weissenberg photographs were taken with Cu $K\alpha$ ($\lambda=1{\cdot}5418$ Å) radiation. The unit-cell parameters were measured from oscillation and zero-layer Weissenberg photographs. The space group was determined uniquely from systematic absences ($0k0$ absent if k is odd, $h0l$ absent if l is odd). The three-dimensional intensity data were obtained from a non-integrating equi-inclination Weissenberg camera with the multiple-film technique. The crystal used was cut to size 0·25 mm × 0·35 mm and 0·6 mm long (in the [010] direction). The reciprocal lattice nets $h0l$–$h9l$ were thus obtained. The 2063 reflexions (72% of the 2872 possible observable reflexions in the copper sphere) were measured visually.

The intensities were corrected for absorption, assuming a cylindrical specimen with a diameter of 0·3 mm ($\mu R=0{\cdot}317$) and for Lorentz, polarization and rotation factors. No allowance was made for unobserved reflexions. Initially the data were put on a common scale by ensuring that $k\Sigma|F_o|=\Sigma|F_c|$ for each layer. The layer scale factors were later refined by least-squares methods.

Structure determination

A three-dimensional Patterson synthesis was computed with the data sharpened to point atom at rest with respect to the sulphur atom. The coordinates for the sulphur atom were deduced from the Harker peaks at $(0,2y-\frac{1}{2},\frac{1}{2})$ and $(2x,\frac{1}{2},2z-\frac{1}{2})$ and the vector peak at $(2x,2y,2z)$.

The heavy-atom technique was used in solving this structure even though the ratio $\sum_H f_H^2/\sum_L f_L^2$ has the low

---

* Present address: Division of Pure Chemistry, National Research Council, Ottawa 2, Canada.

value of 0·34. In the Fourier refinement only data from seven layers (1586 reflexions) were used. The starting overall R index with only the sulphur atom was 66%. In the first electron density map the three oxygen and eight carbon atoms were located. Inclusion of these atoms in the following structure-factor calculation lowered R to 49%. In the subsequent Fourier summation the remaining seven carbon atoms were found and with all the atoms included the R index fell to 30%. Improved coordinates were obtained from another Fourier synthesis and reduced R to 25·5%. In the structure-factor calculations values for the atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962).

Structure refinement

The positional and thermal parameters of the atoms and the individual layer scale factors were refined by least-squares methods. The course of the refinement is outlined in Table 1. The weighting scheme employed was

$$w=1/(2{\cdot}4+|F_o|+0{\cdot}017|F_o|^2+0{\cdot}00038|F_o|^3)\,.$$

After cycle 8 a difference synthesis revealed the hydrogen atoms with peak heights ranging from 0·30 to 0·75 e.Å^{-3}. No shifts were larger than 0·5σ for hydrogen atoms and 0·1σ for the other atoms after the last cycle of refinement.

Atomic scattering factors for bonded hydrogen (Stewart, Davidson & Simpson, 1965) were used and the imaginary and real parts of the anomalous dispersion corrections for sulphur and oxygen were taken from *International Tables for X-ray Crystallography* (1962). Data collected along only one axis lead to degeneracy between the individual layer scale factors and the anisotropic temperature parameters in full-matrix least-squares refinement. This was avoided by using block-diagonal refinement and convergence was obtained without undue difficulty.

The final coordinates and e.s.d.'s for non-hydrogen atoms are listed in Table 2, and their thermal parameters in Table 3. The latter are values of U_{ij} in the expression

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

The hydrogen-atom coordinates, thermal parameters and e.s.d.'s are shown in Table 4. It is felt that all the e.s.d.'s may be underestimated owing to the block-diagonal approximation.

Table 2. *Final fractional coordinates and e.s.d.'s* for non-hydrogen atoms*

The numbering scheme is shown in Figs. 2 and 3.

	x/a	y/b	z/c
S(1)	0·50040 (11)	0·29176 (12)	0·40349 (9)
O(1)	0·62892 (36)	0·35010 (38)	0·42237 (36)
O(2)	0·51868 (47)	0·23897 (43)	0·50304 (31)
O(3)	0·36728 (36)	0·39330 (33)	0·35122 (27)
C(1)	0·30891 (43)	0·45242 (46)	0·24113 (35)
C(2)	0·25463 (49)	0·57154 (48)	0·22755 (41)
C(3)	0·18578 (52)	0·63392 (52)	0·11991 (48)
C(4)	0·17757 (55)	0·57683 (53)	0·03075 (43)
C(5)	0·23482 (51)	0·45783 (49)	0·04666 (39)
C(6)	0·30357 (42)	0·38973 (44)	0·15300 (36)
C(7)	0·37219 (47)	0·26128 (48)	0·16975 (41)
C(8)	0·40686 (42)	0·18787 (45)	0·27752 (37)
C(9)	0·27427 (41)	0·11975 (43)	0·25788 (34)
C(10)	0·12843 (48)	0·17256 (50)	0·19540 (49)
C(11)	0·00730 (51)	0·10502 (57)	0·17374 (52)
C(12)	0·02906 (54)	−0·01530 (55)	0·21259 (42)
C(13)	0·17541 (65)	−0·06923 (53)	0·27670 (51)
C(14)	0·29594 (53)	−0·00181 (47)	0·29812 (43)
C(15)	0·27798 (73)	0·18133 (54)	0·05679 (51)

* The e.s.d.'s were derived from the least-squares residuals.

After an analysis of the rigid-body thermal vibrations (Cruickshank, 1956) the positional parameters were accordingly corrected (Cruickshank, 1961*a*); molecular dimensions deduced from these coordinates are labelled 'corrected'. The hydrogen atoms were excluded from these calculations. The molecular vibration approximated closely to that of a rigid body. The eigenvalues for the vibration tensor **T** are 0·0211, 0·0580, 0·0416 Å^2 and their axes have the following components relative to the crystal axes: (−1·286, 0·140, −1·047), (0·330, 0·402, −0·670) and (0·050, −0·905, −0·456).

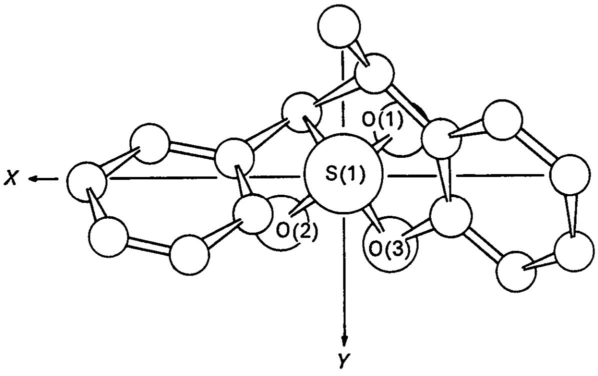


Fig. 1. A view of the sultone-B molecule relative to the inertial X and Y axes.

Table 1. *Progress of refinement*

Parameters refined	Type of refinement	Cycle no.	Final R
x,y,z, U for S,O,C	Full matrix	1–4	21·3 %
x,y,z, $U_{ij}(i,j=1,2,3,)$ for S,O,C	Block diagonal	5–7	11·8
as above $+(\Delta f', \Delta f''$ for S,O)		8	11·7
as above $+x,y,z$, U for H		9–14	9·8

Table 3. *Vibration tensor components, U_{ij} and e.s.d.'s* (Å^2) *for non-hydrogen atoms*

	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
S(1)	0·0291 (5)	0·0497 (8)	0·0372 (5)	−0·0001 (9)	0·0085 (8)	0·0082 (8)
O(1)	0·0316 (15)	0·0531 (25)	0·0799 (26)	−0·0193 (41)	0·0312 (34)	−0·0160 (32)
O(2)	0·0608 (23)	0·0812 (31)	0·0372 (17)	0·0125 (37)	0·0219 (33)	0·0232 (44)
O(3)	0·0430 (15)	0·0546 (23)	0·0380 (15)	0·0054 (28)	0·0399 (27)	0·0216 (29)
C(1)	0·0265 (16)	0·0485 (31)	0·0351 (19)	0·0032 (36)	0·0260 (30)	−0·0001 (34)
C(2)	0·0390 (21)	0·0474 (32)	0·0468 (23)	−0·0021 (42)	0·0433 (39)	0·0118 (40)
C(3)	0·0386 (22)	0·0473 (34)	0·0638 (29)	0·0103 (49)	0·0494 (44)	0·0069 (43)
C(4)	0·0444 (22)	0·0538 (34)	0·0495 (25)	0·0207 (46)	0·0449 (41)	0·0014 (45)
C(5)	0·0417 (20)	0·0515 (32)	0·0410 (21)	0·0005 (42)	0·0450 (37)	−0·0082 (42)
C(6)	0·0267 (16)	0·0473 (30)	0·0390 (19)	−0·0034 (37)	0·0339 (31)	−0·0065 (34)
C(7)	0·0328 (20)	0·0495 (31)	0·0504 (24)	−0·0038 (42)	0·0502 (39)	0·0028 (37)
C(8)	0·0230 (16)	0·0403 (29)	0·0396 (20)	−0·0113 (36)	0·0203 (31)	0·0025 (32)
C(9)	0·0251 (16)	0·0387 (28)	0·0343 (18)	0·0058 (34)	0·0202 (30)	0·0041 (32)
C(10)	0·0287 (19)	0·0418 (33)	0·0691 (30)	0·0390 (48)	0·0381 (41)	0·0169 (38)
C(11)	0·0279 (19)	0·0701 (42)	0·0673 (31)	0·0280 (56)	0·0349 (41)	0·0022 (44)
C(12)	0·0447 (24)	0·0610 (37)	0·0508 (26)	−0·0143 (47)	0·0512 (43)	−0·0355 (46)
C(13)	0·0655 (30)	0·0435 (35)	0·0658 (30)	0·0094 (51)	0·0843 (53)	0·0008 (50)
C(14)	0·0431 (22)	0·0427 (33)	0·0528 (25)	0·0121 (43)	0·0516 (41)	0·0157 (40)
C(15)	0·0781 (36)	0·0534 (37)	0·0579 (29)	−0·0176 (53)	0·0869 (58)	0·0018 (58)

Table 4. *Final fractional coordinates and isotropic temperature parameters* (Å^2) *and e.s.d.'s for the hydrogen atoms*

The hydrogen atoms are numbered according to the carbon atoms to which they are attached.

	x/a	y/b	z/c	U_{iso}
H(2)	0·254 (6)	0·597 (5)	0·286 (5)	0·060 (15)
H(3)	0·151 (7)	0·724 (5)	0·113 (5)	0·074 (17)
H(4)	0·127 (9)	0·607 (7)	−0·050 (7)	0·106 (23)
H(5)	0·239 (6)	0·432 (5)	−0·009 (4)	0·051 (14)
H(7)	0·460 (7)	0·275 (5)	0·176 (6)	0·074 (17)
H(8)	0·490 (5)	0·126 (5)	0·299 (4)	0·050 (13)
H(10)	0·115 (6)	0·255 (5)	0·182 (4)	0·045 (13)
H(11)	−0·093 (6)	0·152 (5)	0·125 (5)	0·063 (16)
H(12)	−0·069 (8)	−0·056 (6)	0·190 (7)	0·101 (21)
H(13)	0·206 (9)	−0·137 (8)	0·317 (7)	0·121 (25)
H(14)	0·408 (7)	−0·027 (6)	0·350 (6)	0·079 (19)
H(15)$_1$	0·332 (6)	0·106 (5)	0·079 (5)	0·060 (15)
H(15)$_2$	0·163 (7)	0·179 (6)	0·018 (5)	0·085 (18)
H(15)$_3$	0·286 (6)	0·201 (5)	−0·002 (5)	0·064 (16)

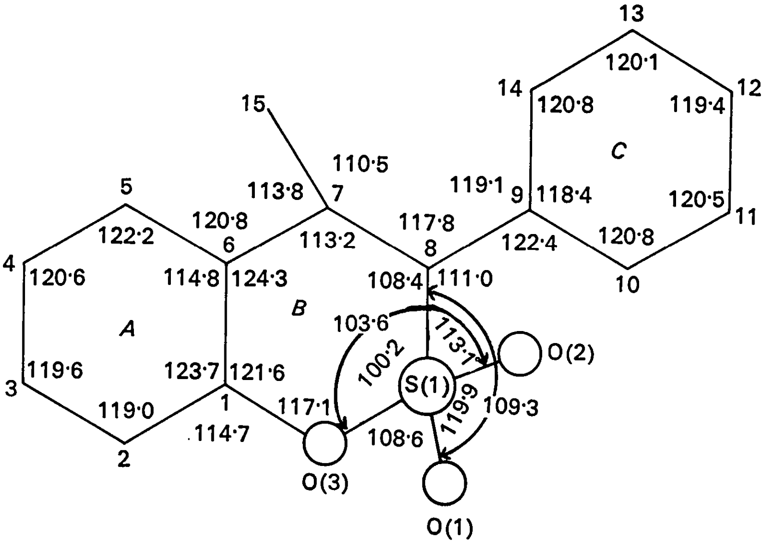


Fig. 2. The bond angles (corrected) in sultone-B.

Table 5. *Interatomic distances and e.s.d.'s* (Å)*

(*a*) Intramolecular bonded distances not involving hydrogen atoms

Bond	Uncorrected length	Corrected length	E.s.d.
S(1)–O(1)	1·425	1·428	0·004
S(1)–O(2)	1·431	1·434	0·004
S(1)–O(3)	1·592	1·595	0·004
S(1)–C(8)	1·791	1·797	0·005
O(3)–C(1)	1·423	1·426	0·005
C(6)–C(7)	1·517	1·519	0·007
C(8)–C(9)	1·508	1·512	0·006
C(7)–C(8)	1·554	1·557	0·006
C(7)–C(15)	1·519	1·523	0·007
C(1)–C(2)	1·371	1·373	0·007
C(2)–C(3)	1·387	1·390	0·007
C(3)–C(4)	1·373	1·377	0·008
C(4)–C(5)	1·377	1·379	0·008
C(5)–C(6)	1·404	1·406	0·006
C(6)–C(1)	1·404	1·409	0·006
C(9)—C(10)	1·385	1·389	0·006
C(10)–C(11)	1·384	1·386	0·007
C(11)–C(12)	1·363	1·363	0·008
C(12)–C(13)	1·392	1·395	0·007
C(13)–C(14)	1·379	1·382	0·007
C(14)–C(9)	1·381	1·382	0·007

* The e.s.d.'s should probably be increased by 50%.

(*b*) Intramolecular bonded distances involving hydrogen atoms

Bond	Uncorrected length	E.s.d.
C(2)–H(2)	0·89	0·05
C(3)–H(3)	1·02	0·06
C(4)–H(4)	0·97	0·07
C(5)–H(5)	0·88	0·05
C(10)–H(10)	0·90	0·05
C(11)–H(11)	1·00	0·05
C(12)–H(12)	1·02	0·07
C(13)–H(13)	0·86	0·08
C(14)–H(14)	1·00	0·06
C(7)–H(7)	0·94	0·06
C(8)–H(8)	1·01	0·05
C(15)–H(15)$_3$	0·93	0·05
C(15)–H(15)$_2$	1·02	0·06
C(15)–H(15)$_1$	0·94	0·06

(*c*) Some intramolecular non-bonded contacts (corrected)

Heterocyclic ring

O(2)···O(3)	2·383
O(1)···O(3)	2·456
O(1)···O(2)	2·477
O(1)···C(8)	2·639
O(2)···C(8)	2·702
O(1)···C(1)	2·993
O(1)···C(7)	3·024
O(2)···C(9)	3·056
O(3)···C(10)	3·188
S(1)···C(10)	3·479
S(1)···C(14)	3·609

Methyl group

C(15)···C(9)	3·006
C(15)···C(5)	2·989
C(15)···H(5)	2·79
H(15)$_3$··H(5)	2·51

Phenyl group

C(10)···C(1)	3·437
C(10)···C(6)	3·342
H(10)···C(1)	2·74
H(10)···C(6)	2·80
H(14)···H(8)	2·23

Table 5 (*cont.*)

(*d*) Intermolecular contacts (< 3·5 Å) of non-hydrogen atoms (corrected)

O(1)···C(3)$_{\mathrm{I}}$	3·407
O(1)···C(4)$_{\mathrm{I}}$	3·436
O(2)···C(13)$_{\mathrm{II}}$	3·348
O(2)···C(14)$_{\mathrm{II}}$	3·368
O(2)···C(15)$_{\mathrm{III}}$	3·347
O(1)···C(11)$_{\mathrm{IV}}$	3·406

The subscripts refer to the following equivalent positions:

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

Table 6. *Bond angles*

(*a*) Non-hydrogen atoms

Angle	Uncorrected angle	Corrected angle	E.s.d.
O(1)–S(1)–O(2)	120·0°	119·9°	0·2°
O(1)–S(1)–O(3)	108·4	108·6	0·2
O(2)–S(1)–O(3)	103·7	103·6	0·2
O(1)–S(1)–C(8)	109·4	109·3	0·2
O(2)–S(1)–C(8)	113·0	113·1	0·2
O(3)–S(1)–C(8)	100·3	100·2	0·2
S(1)–O(3)–C(1)	117·2	117·1	0·3
O(3)–C(1)–C(2)	114·8	114·7	0·4
O(3)–C(1)–C(6)	121·5	121·6	0·4
C(2)–C(1)–C(6)	123·7	123·7	0·4
C(1)–C(2)–C(3)	119·1	119·0	0·4
C(2)–C(3)–C(4)	119·5	119·6	0·5
C(3)–C(4)–C(5)	120·6	120·6	0·5
C(4)–C(5)–C(6)	122·3	122·2	0·4
C(1)–C(6)–C(5)	114·7	114·8	0·4
C(1)–C(6)–C(7)	124·3	124·3	0·4
C(5)–C(6)–C(7)	120·9	120·8	0·4
C(6)–C(7)–C(8)	113·3	113·2	0·4
C(6)–C(7)–C(15)	113·9	113·8	0·4
C(8)–C(7)–C(15)	110·4	110·5	0·4
S(1)–C(8)–C(7)	108·2	108·4	0·3
S(1)–C(8)–C(9)	111·1	111·0	0·3
C(7)–C(8)–C(9)	117·9	117·8	0·3
C(8)—C(9)—C(10)	122·3	122·4	0·4
C(8)—C(9)—C(14)	119·2	119·1	0·4
C(10)–C(9)—C(14)	118·4	118·4	0·4
C(9)—C(10)–C(11)	120·7	120·8	0·5
C(10)–C(11)–C(12)	120·5	120·5	0·5
C(11)–C(12)–C(13)	119·4	119·4	0·5
C(12)–C(13)–C(14)	120·0	120·1	0·5
C(9)—C(14)–C(13)	120·9	120·8	0·4

* The e.s.d.'s should probably be increased by 50%.

(*b*) Hydrogen atoms

Angle	Uncorrected angle	E.s.d.
H(2)–C(2)–C(1)	115°	3°
H(2)–C(2)–C(3)	125	3
H(3)–C(3)–C(4)	123	3
H(3)–C(3)–C(2)	118	3
H(4)–C(4)–C(5)	112	4
H(4)–C(4)–C(3)	128	4
H(5)–C(5)–C(4)	115	3
H(5)–C(5)–C(6)	122	3

Table 6 (*cont.*)

Angle	Uncorrected angle	E.s.d.
H(10)-C(10)-C(9)	121	3
H(10)-C(10)-C(11)	117	3
H(11)-C(11)-C(12)	127	3
H(11)-C(11)-C(10)	113	3
H(12)-C(12)-C(11)	113	4
H(12)-C(12)-C(13)	127	4
H(13)-C(13)-C(14)	112	5
H(13)-C(13)-C(12)	127	5
H(14)-C(14)-C(9)	112	4
H(14)-C(14)-C(13)	127	4
H(7)-C(7)-C(15)	99	4
H(7)-C(7)-C(6)	105	4
H(7)-C(7)-C(8)	114	4
H(8)-C(8)-C(9)	110	3
H(8)-C(8)-C(7)	103	3
H(8)-C(8)-S(1)	106	3
H(15)$_3$-C(15)-H(15)$_2$	109	5
H(15)$_3$-C(15)-H(15)$_1$	95	5
H(15)$_2$-C(15)-H(15)$_1$	119	5
H(15)$_3$-C(15)-C(7)	116	3
H(15)$_2$-C(15)-C(7)	112	3
H(15)$_1$-C(15)-C(7)	105	3

The eigenvalues for the libration tensor ω are 23·8, 2·3 and 5·7 deg^2. The axis of maximum libration is approximately parallel to the inertial X axis (Fig. 1) and the medium one approximately parallel to the inertial Y axis. The components relative to the crystal axes are (−0·030, 0·951, −0·288) ,(1·106, 0·142, 1·209) and (−0·679, 0·273, 0·375) respectively.

The effect of the thermal vibration corrections was small; most of the bond lengths increased by about 0·5σ – the largest increase was in the S–C(8) bond (1·2σ). The maximum change in any of the angles was about 0·5σ. Corrected and uncorrected interatomic distances and bond angles with their e.d.s.'s are given in Tables 5 and 6 respectively. The corrected bond angles can be more clearly seen in Fig. 2. A list of observed and calculated structure factors is given in Table 7.

Description and discussion of the structure

This crystal structure determination shows that in sultone-B the methyl and the phenyl groups are *cis* to each other. The heterocyclic ring is found to be in the half-chair conformation with the phenyl ring axial and the methyl group quasi-equatorial (Barton & Cookson, 1956). A view of the molecule in the [001] direction and our numbering scheme is given in Fig. 3. The oxygen O(1) is axial and O(2) is equatorial. The atoms of aromatic ring *A* [C(1)···C(6)] are best fitted by the plane with equation $0{\cdot}892X'+0{\cdot}379Y-0{\cdot}246Z'=3{\cdot}903$ Å where X',Y,Z' (in Å) are referred to the orthogonal axes a',b,c. The deviations of the atoms from this plane are not significant; the r.m.s. distance of the ring-*A* carbon atoms is 0·006 Å. The displacements of the hydrogen atoms H(2), H(3), H(4) and H(5) from this plane range from 0·05 to 0·14 Å and are not significant.

Geometrical considerations preclude ring *B* from being planar, and with the presence of hetero atoms it is reasonable to expect the ring to be under some strain. Atoms C(7) and O(3), which might have been expected to be coplanar with aromatic ring *A*, are in fact significantly displaced from it by +0·070 and −0·085 Å; C(8) and S(1) are out of this plane by −0·282 and +0·578 Å respectively. The overall effect is that ring *B* is puckered with atoms C(7), C(8), S and O(3) lying alternately above and below the plane of ring *A*.

The best plane through the atoms [C(9)···C(14)] of the phenyl group C is $0{\cdot}247X'+0{\cdot}332Y+0{\cdot}911Z'=2{\cdot}615$ Å. The r.m.s. distance of these atoms from this

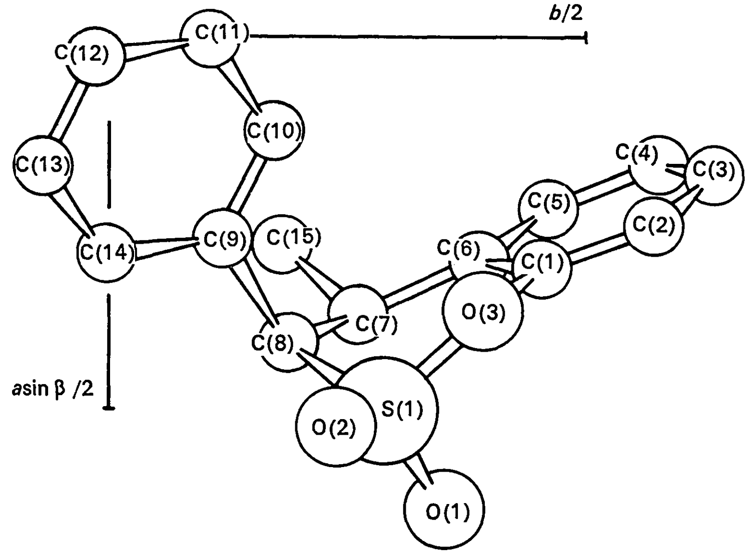


Fig. 3. A view of the sultone-B molecule projected on (001) with our numbering scheme.

plane is 0·004 Å. The dihedral angle between the two aromatic planes is 83 °, and the orientation of the phenyl plane is such that it intersects the C(1)–C(6) bond approximately at its mid point. The deviations of the phenyl hydrogen atoms from the phenyl plane are not significant (ranging from 0·02 to 0·18 Å). However, atom C(8) is significantly out of plane by 0·081 Å. This is almost certainly caused by repulsion between the methyl and the phenyl groups, which can be diminished by bending the phenyl ring towards the oxygen atom O(2), there being more space available at that side of the ring.

Table 7. *Observed and calculated structure factors*

H	K	L	|Fo|	|Fc|
0	0	2	66.6	70.7
0	0	4	28.0	23.3
0	0	6	16.5	16.4
0	0	8	6.9	7.4
0	0	10	7.3	7.4
0	0	12	9.2	9.1
1	0	0	92.0	103.6
1	0	2	96.3	105.0
1	0	4	17.7	16.2
1	0	6	9.1	8.7
1	0	8	22.5	23.6
1	0	10	10.0	10.3
1	0	12	12.8	13.9
2	0	0	41.2	38.2
2	0	2	27.0	24.3
2	0	4	13.3	12.6
2	0	6	2.2	2.4
2	0	10	19.5	20.5
2	0	12	6.0	7.1
3	0	0	3.5	3.9
3	0	2	56.2	53.1
3	0	4	12.9	13.2
3	0	6	14.3	15.2
3	0	10	14.5	15.9
4	0	0	45.5	43.9
4	0	2	13.7	13.8
4	0	4	19.4	19.7
4	0	6	21.8	24.5
4	0	8	11.2	11.8
4	0	10	8.0	11.3
5	0	0	23.5	22.2
5	0	2	3.2	3.4
5	0	4	7.2	7.1
5	0	6	13.4	15.9
5	0	8	7.7	8.9
6	0	0	48.0	45.1
6	0	2	9.7	9.9
6	0	4	25.9	28.3
6	0	6	8.8	8.3
7	0	0	13.0	12.9
7	0	2	29.4	32.7
7	0	4	23.2	25.8
7	0	6	10.1	11.2
8	0	0	32.2	32.9
8	0	2	3.7	3.9
8	0	4	9.6	11.1
9	0	0	26.4	28.6
9	0	2	5.7	5.6
10	0	0	18.6	21.2
11	0	0	4.4	5.3
1	0	-2	24.0	24.4
1	0	-4	78.1	87.3
1	0	-6	35.4	37.7
1	0	-8	6.8	6.6
1	0	-10	15.7	16.2
1	0	-12	15.9	17.6
2	0	-2	57.0	56.6
2	0	-4	83.7	95.8
2	0	-6	20.2	19.3
2	0	-8	9.7	6.6
2	0	-10	58.6	59.0
2	0	-12	14.0	14.6
2	0	-14	2.1	2.5
3	0	-2	103.6	109.5
3	0	-4	34.3	33.3
3	0	-6	21.3	22.6
3	0	-8	42.2	39.8
3	0	-10	51.9	54.3
3	0	-12	16.3	15.9
3	0	-14	6.4	5.8
3	0	-16	5.1	5.7
4	0	-2	22.4	21.9
4	0	-4	17.8	15.0
4	0	-6	20.1	19.9
4	0	-10	21.0	19.6
4	0	-12	10.9	10.0
4	0	-16	2.1	2.3
5	0	-2	12.8	13.5
5	0	-4	11.9	11.4
5	0	-6	36.8	34.1
5	0	-8	12.5	10.8
5	0	-10	10.0	10.9
5	0	-12	32.3	31.3
5	0	-14	2.4	2.3
5	0	-16	1.9	2.1
6	0	-2	20.5	19.6
6	0	-4	15.3	15.3
6	0	-6	21.7	20.2
6	0	-8	4.4	3.3
6	0	-10	26.0	23.1
6	0	-12	26.6	25.0
6	0	-14	8.0	7.4
6	0	-16	8.9	7.1
7	0	-2	35.6	33.3
7	0	-4	33.4	33.1
7	0	-6	8.3	7.5
7	0	-8	3.3	1.7
7	0	-12	12.3	11.7
7	0	-14	8.4	7.3
7	0	-16	10.5	8.7
8	0	-2	10.4	10.0
8	0	-4	18.3	17.5
8	0	-6	15.1	12.7
8	0	-8	2.8	2.3
8	0	-10	8.9	8.0
8	0	-12	15.6	13.1
8	0	-14	3.5	3.1
8	0	-18	1.3	1.4
9	0	-2	2.5	2.4
9	0	-4	4.6	3.3
9	0	-6	48.8	47.5
9	0	-8	30.7	31.2
9	0	-10	11.6	11.2
9	0	-12	3.5	2.5
9	0	-14	4.2	3.7
9	0	-16	4.4	2.9
10	0	-2	2.8	2.8
10	0	-4	23.7	23.6
10	0	-8	5.9	1.3
10	0	-10	17.9	16.2
10	0	-14	1.7	2.4
10	0	-18	4.5	3.2
11	0	-2	4.9	4.8
11	0	-4	3.5	3.5
11	0	-6	9.4	8.6
11	0	-10	18.5	17.8
11	0	-14	2.7	3.2
11	0	-16	2.6	2.4
12	0	-6	10.1	9.0
12	0	-8	2.2	2.3
12	0	-10	9.0	7.7
12	0	-12	5.4	4.0
12	0	-14	8.4	7.1
12	0	-16	2.8	2.1
13	0	-6	8.5	9.2
13	0	-10	5.8	5.5
13	0	-12	2.9	2.9
13	0	-16	2.7	2.3
14	0	-10	2.3	2.2
14	0	-12	1.3	1.4
14	0	-14	3.8	3.3
0	1	1	16.5	12.9
0	1	2	36.3	38.3
0	1	3	17.8	16.7
0	1	4	52.9	51.5
0	1	5	35.8	34.8
0	1	6	29.7	28.3
0	1	7	7.3	6.4
0	1	8	37.9	36.4
0	1	9	9.1	9.3
0	1	10	8.2	7.9
0	1	11	15.9	14.7
0	1	12	7.6	5.2
0	1	14	9.6	8.0
1	1	0	52.5	52.2
1	1	1	17.9	15.8
1	1	2	35.6	34.8
1	1	3	3.4	3.1
1	1	4	18.5	18.8
1	1	5	25.2	24.4
1	1	6	7.1	5.8
1	1	7	3.6	1.7
1	1	8	28.2	31.8
1	1	9	18.4	17.9
1	1	10	14.9	12.6
1	1	11	7.4	5.7
1	1	12	12.3	9.2
2	1	0	52.2	52.6
2	1	1	32.3	31.9
2	1	2	9.0	9.9
2	1	3	5.8	5.2
2	1	4	22.8	19.6
2	1	5	34.0	31.1
2	1	7	13.0	12.7
2	1	8	11.4	12.5
2	1	10	14.1	13.0
2	1	12	6.5	5.6
3	1	0	3.2	3.8
3	1	1	13.0	10.1
3	1	2	3.5	2.2
3	1	4	12.9	12.1
3	1	5	6.1	5.9
3	1	6	8.7	8.1
3	1	8	13.5	13.1
3	1	9	3.6	4.1
3	1	11	5.7	4.5
4	1	0	41.0	35.9
4	1	1	9.0	8.1
4	1	2	9.9	8.6
4	1	3	11.5	9.8
4	1	4	4.2	3.2
4	1	5	8.2	8.2
4	1	6	12.1	12.6
4	1	7	6.5	7.1
4	1	8	23.7	22.3
4	1	9	6.6	6.0
5	1	0	5.2	2.2
5	1	1	11.5	10.0
5	1	2	17.8	17.2
5	1	3	13.5	11.7
5	1	4	11.3	11.9
5	1	5	14.6	15.9
5	1	6	18.3	21.3
5	1	7	4.0	3.0
5	1	8	14.1	12.5
5	1	9	7.3	6.1
6	1	0	11.8	10.8
6	1	1	6.4	6.4
6	1	2	18.5	18.3
6	1	3	2.3	2.1
6	1	4	21.4	22.4
6	1	5	3.7	3.3
6	1	6	19.7	20.0
7	1	0	15.3	15.5
7	1	1	7.6	7.1
7	1	2	27.7	28.3
7	1	3	9.0	9.2
7	1	4	7.0	6.5
7	1	5	5.3	5.1
7	1	6	10.5	9.8
8	1	0	10.9	11.3
8	1	1	3.5	3.6
8	1	2	14.7	16.3
8	1	3	4.8	4.7
8	1	4	7.6	7.2
8	1	5	4.4	4.9
9	1	1	5.8	6.1
9	1	2	14.0	15.0
10	1	0	6.3	6.4
11	1	0	3.0	3.9
1	1	-1	30.6	30.6
1	1	-2	92.5	110.1
1	1	-3	56.0	66.2
1	1	-4	5.6	5.8
1	1	-5	55.7	59.3
1	1	-6	20.5	21.9
1	1	-7	11.5	12.0
1	1	-8	22.1	27.3
1	1	-9	17.1	21.4
1	1	-10	7.3	9.0
1	1	-11	2.3	3.0
1	1	-12	2.2	2.3
1	1	-14	7.0	6.7
2	1	-1	82.1	88.9
2	1	-2	82.6	89.3
2	1	-3	87.6	95.0
2	1	-4	47.3	52.5
2	1	-5	11.1	10.4
2	1	-6	15.0	14.7
2	1	-7	22.8	23.0
2	1	-8	32.0	36.4
2	1	-9	20.1	21.3
2	1	-10	6.7	7.1
2	1	-11	13.0	16.4
2	1	-12	5.1	5.2
2	1	-14	5.1	4.4
2	1	-15	3.5	3.2
3	1	-1	62.7	62.5
3	1	-2	99.8	113.1
3	1	-3	3.7	3.4
3	1	-4	33.9	37.3
3	1	-5	33.8	37.3
3	1	-6	46.0	48.4
3	1	-7	3.2	2.1
3	1	-8	5.9	6.9
3	1	-9	5.3	4.8
3	1	-10	11.4	12.3
3	1	-11	5.7	5.7
3	1	-12	16.4	16.6
3	1	-13	2.2	3.1
3	1	-14	5.4	4.9
4	1	-1	21.8	19.7
4	1	-2	24.0	25.6
4	1	-3	27.5	27.5
4	1	-4	12.4	11.8
4	1	-5	8.9	8.1
4	1	-6	2.9	3.1
4	1	-7	11.0	10.8
4	1	-8	16.1	19.3
4	1	-9	1.9	1.7
4	1	-10	9.4	10.6
4	1	-11	21.4	22.9
4	1	-12	19.8	20.5
4	1	-13	6.2	6.7
4	1	-14	7.0	6.9
4	1	-15	4.7	3.6
4	1	-16	1.9	2.2
5	1	-1	3.1	1.6
5	1	-2	12.3	10.0
5	1	-3	34.0	32.8
5	1	-4	6.0	5.7
5	1	-6	19.7	18.7
5	1	-7	4.2	3.5
5	1	-8	15.4	15.7
5	1	-9	6.1	5.8
5	1	-10	7.3	6.7
5	1	-11	3.4	2.7
5	1	-12	21.8	21.2
5	1	-13	10.0	10.3
5	1	-14	10.1	10.3
5	1	-15	4.8	4.1
5	1	-16	6.2	5.0
5	1	-17	1.2	1.3
6	1	-1	5.4	5.7
6	1	-2	28.5	27.8
6	1	-3	15.6	16.7
6	1	-4	39.8	38.7
6	1	-5	5.3	5.7
6	1	-7	24.3	24.2
6	1	-8	1.3	1.3
6	1	-9	6.4	6.1
6	1	-10	5.6	5.1
6	1	-12	15.4	17.8
6	1	-13	4.6	4.5
6	1	-16	2.9	2.6
7	1	-1	6.2	5.1
7	1	-2	36.4	34.1
7	1	-3	3.4	2.4
7	1	-4	27.4	26.1
7	1	-5	7.5	7.9
7	1	-6	1.3	1.7
7	1	-7	8.3	7.5
7	1	-8	7.4	7.6
7	1	-9	19.5	17.9
7	1	-10	6.8	6.2
7	1	-11	8.3	8.3
7	1	-12	2.3	1.9
7	1	-13	19.4	15.4
7	1	-14	2.8	2.3
7	1	-15	6.7	6.1
7	1	-16	1.9	2.2
8	1	-1	30.0	29.0
8	1	-2	12.8	13.2
8	1	-3	28.6	29.2
8	1	-4	30.7	30.3
8	1	-5	7.7	7.2
8	1	-6	17.3	17.5
8	1	-7	5.6	4.8
8	1	-8	17.5	17.2
8	1	-11	3.2	2.9
8	1	-13	3.6	3.4
8	1	-14	14.0	12.5
8	1	-16	3.9	3.8
8	1	-17	6.5	5.4
8	1	-18	3.3	2.6
9	1	-1	3.5	2.3
9	1	-2	21.6	23.3
9	1	-3	4.6	4.4
9	1	-4	17.5	16.5
9	1	-5	14.3	15.0
9	1	-6	15.7	14.6
9	1	-7	4.9	4.5
9	1	-8	12.0	11.9
9	1	-9	7.7	7.4
9	1	-10	3.9	4.2
9	1	-11	3.6	3.5
9	1	-12	10.9	10.0
9	1	-13	7.0	6.6
9	1	-14	11.6	11.0
9	1	-15	3.0	2.9
9	1	-17	3.4	2.7
9	1	-18	1.5	1.5
10	1	-2	10.2	12.5
10	1	-3	5.6	5.5
10	1	-4	12.1	12.5
10	1	-5	15.4	16.0
10	1	-6	6.7	6.5
10	1	-7	4.0	4.2
10	1	-8	16.2	15.3
10	1	-10	10.6	10.9
10	1	-11	11.0	10.6
10	1	-12	4.2	4.1
10	1	-13	3.5	3.4
10	1	-14	4.0	3.4
10	1	-16	3.4	2.4
10	1	-17	2.2	2.2
10	1	-18	2.5	2.1
11	1	-1	4.2	4.0
11	1	-2	11.7	13.2
11	1	-3	3.7	3.5
11	1	-4	2.0	2.7
11	1	-5	6.5	6.6
11	1	-6	5.1	4.9
11	1	-7	7.5	7.4
11	1	-8	7.6	7.2
11	1	-9	16.5	17.7
11	1	-11	5.0	4.4
11	1	-12	3.8	3.8
11	1	-14	5.0	4.5
12	1	-3	5.2	6.1
12	1	-5	2.4	2.2
12	1	-6	2.6	1.8
12	1	-7	2.8	2.6
12	1	-8	8.4	7.1
12	1	-10	2.1	1.5
12	1	-12	9.5	8.5
12	1	-13	3.9	1.2
13	1	-5	4.1	5.1
13	1	-6	1.6	1.4
13	1	-7	2.3	2.6
13	1	-8	3.6	2.8
13	1	-10	2.1	1.3
13	1	-12	3.2	3.0
13	1	-14	4.9	4.5
13	1	-15	3.8	3.3
13	1	-16	2.8	2.7
14	1	-9	4.5	5.2
14	1	-10	4.6	4.3
14	1	-11	3.3	3.5
14	1	-12	4.0	4.3
0	2	1	5.6	4.4
0	2	2	80.1	75.3
0	2	3	48.0	52.1
0	2	4	39.6	42.7
0	2	5	24.3	22.8
0	2	6	3.2	2.1
0	2	7	10.7	9.1
0	2	8	3.6	4.2
0	2	9	4.9	4.5
0	2	10	23.0	22.0
0	2	11	7.6	6.6
0	2	12	17.1	12.9
1	2	0	114.5	112.8
1	2	1	84.4	73.2
1	2	2	48.4	49.9
1	2	3	42.9	41.1
1	2	4	17.7	15.6
1	2	5	15.2	13.3
1	2	6	14.0	12.9
1	2	7	3.6	2.6
1	2	8	4.9	4.3
1	2	9	3.2	2.9
1	2	10	11.5	10.7
1	2	12	12.2	9.8
1	2	13	3.7	3.0
2	2	0	110.1	106.5
2	2	1	24.0	27.2
2	2	2	33.0	31.7
2	2	3	26.2	24.2
2	2	4	17.4	14.8
2	2	5	14.5	12.5
2	2	6	17.1	16.3
2	2	7	6.4	5.8
2	2	8	16.8	19.2
2	2	9	9.4	9.2
2	2	10	8.8	7.6
2	2	11	5.1	4.7
2	2	12	9.7	8.5
3	2	0	66.4	66.9
3	2	1	19.4	17.0
3	2	2	5.2	5.9
3	2	3	19.3	16.1
3	2	4	3.8	4.4
3	2	5	17.7	15.1
3	2	6	14.0	13.5
3	2	8	16.1	14.9
3	2	9	17.9	15.4
3	2	10	16.7	13.2
3	2	11	2.0	2.4
4	2	0	4.7	3.6
4	2	1	6.8	5.5
4	2	2	37.3	34.6
4	2	4	22.9	21.6
4	2	6	15.7	15.3
4	2	8	8.2	7.1
4	2	9	7.4	6.0
4	2	10	12.7	11.2
5	2	0	28.7	29.0
5	2	1	3.8	4.0
5	2	2	15.1	14.7
5	2	3	17.5	16.5
5	2	4	5.5	5.3
5	2	5	10.9	10.1
5	2	6	11.7	12.9
5	2	7	9.0	8.5
6	2	0	18.8	18.5
6	2	1	19.0	18.7
6	2	2	18.6	18.5
6	2	3	6.2	6.2
6	2	4	13.0	15.1
6	2	5	6.4	7.4
6	2	7	22.4	20.8
7	2	0	27.3	27.5
7	2	1	28.1	28.3
7	2	3	11.2	12.9
7	2	4	7.8	7.8
7	2	6	7.3	6.9
8	2	0	25.3	27.0
8	2	1	5.4	5.9
8	2	3	16.2	17.0
8	2	4	8.2	8.7
9	2	0	13.6	15.8
9	2	1	2.7	2.8
9	2	2	6.6	7.9
10	2	0	8.6	9.5
10	2	1	3.0	3.3
1	2	-1	26.2	26.9
1	2	-3	65.9	72.5
1	2	-4	26.7	30.7
1	2	-5	30.2	36.6
1	2	-6	5.0	8.2
1	2	-7	5.1	5.6
1	2	-8	6.1	7.2
1	2	-9	6.3	6.7
1	2	-10	23.9	31.3
1	2	-12	4.0	4.8
1	2	-13	10.9	10.8
1	2	-14	1.9	2.1
2	2	-1	54.2	50.6
2	2	-2	5.2	4.9
2	2	-3	11.2	14.0
2	2	-4	13.4	12.9
2	2	-5	41.8	48.4
2	2	-6	17.8	17.7
2	2	-7	47.4	54.3
2	2	-9	5.5	6.4
2	2	-10	21.2	24.0
2	2	-11	5.5	6.0
2	2	-12	4.8	5.9
2	2	-13	3.9	4.7
2	2	-14	2.8	2.6
2	2	-15	4.2	3.7
3	2	-1	9.6	7.3
3	2	-2	28.3	28.5
3	2	-3	5.0	3.1
3	2	-4	8.3	8.9
3	2	-5	14.7	15.4
3	2	-6	5.8	6.1
3	2	-7	38.2	39.6
3	2	-8	3.3	1.2
3	2	-9	7.7	8.6
3	2	-10	27.5	30.8
3	2	-11	3.1	3.0
3	2	-12	8.0	9.1
3	2	-13	4.1	4.7
3	2	-15	2.0	1.8
4	2	-1	43.1	40.3
4	2	-2	56.1	59.7
4	2	-3	24.1	24.8
4	2	-4	11.2	12.3
4	2	-5	4.0	3.2
4	2	-6	2.7	1.5
4	2	-7	16.0	15.5
4	2	-8	30.1	32.9
4	2	-9	23.7	22.5
4	2	-10	12.1	13.0
4	2	-11	22.7	23.1
4	2	-12	14.7	15.8
4	2	-14	[illegible]	5.0
4	2	-15	2.6	2.8
5	2	-1	19.4	19.5
5	2	-2	60.0	62.2
5	2	-4	29.5	29.7
5	2	-5	2.8	2.7
5	2	-6	12.0	11.5
5	2	-7	4.7	4.6
5	2	-9	5.2	4.7
5	2	-10	19.2	20.3
5	2	-13	7.1	7.2
5	2	-16	6.9	5.7
5	2	-17	5.3	5.0
6	2	-1	21.5	20.1
6	2	-2	25.2	25.1
6	2	-3	33.8	35.2
6	2	-4	16.5	18.1
6	2	-5	19.4	17.2
6	2	-6	32.3	33.9
6	2	-8	16.4	16.5
6	2	-10	9.6	9.3
6	2	-11	15.5	15.3
6	2	-12	6.5	5.9
6	2	-13	8.3	8.0
6	2	-14	4.1	4.3
6	2	-16	5.1	4.0
6	2	-17	5.9	4.7
7	2	-1	16.2	16.2
7	2	-2	6.5	5.4
7	2	-3	3.1	3.3
7	2	-4	15.5	15.2
7	2	-5	13.1	12.3
7	2	-6	11.0	10.3
7	2	-7	6.5	5.6
7	2	-8	14.4	14.4
7	2	-9	16.0	14.2
7	2	-10	13.3	11.8
7	2	-11	4.1	4.2
7	2	-12	16.2	15.6
7	2	-13	9.3	9.0
7	2	-16	1.8	2.0
7	2	-17	3.7	2.9
8	2	-3	42.3	42.6
8	2	-5	2.6	3.1
8	2	-6	8.5	7.7
8	2	-7	11.6	11.5
8	2	-8	4.9	4.0
8	2	-9	6.5	6.0
8	2	-10	11.6	11.8
8	2	-11	8.2	7.1
8	2	-12	11.4	10.9
8	2	-13	5.1	4.9
8	2	-14	11.3	10.2
8	2	-15	2.1	2.1
8	2	-16	3.2	3.0
9	2	-1	6.2	6.7
9	2	-2	5.5	5.8
9	2	-3	21.8	23.7
9	2	-4	8.9	9.5
9	2	-5	24.3	23.6
9	2	-6	7.2	5.6
9	2	-7	6.2	6.9
9	2	-8	7.8	6.5
9	2	-9	12.0	11.1
9	2	-10	9.4	9.3
9	2	-11	6.6	6.1
9	2	-12	7.3	7.2
9	2	-13	6.1	5.6
9	2	-14	10.4	9.8
9	2	-15	6.3	6.4
10	2	-1	1.9	0.6
10	2	-6	17.1	17.3
10	2	-7	14.1	14.5
10	2	-8	5.6	4.9
10	2	-10	7.3	7.8
10	2	-11	8.9	9.4
10	2	-12	7.4	7.1
10	2	-13	3.5	3.6
10	2	-15	3.1	2.9
10	2	-18	3.7	3.4
11	2	-1	5.4	6.8
11	2	-3	2.9	3.1
11	2	-4	3.1	3.8
11	2	-5	2.9	2.7
11	2	-6	8.9	9.1
11	2	-7	6.3	7.5
11	2	-9	7.6	7.9
11	2	-10	13.7	14.2
11	2	-11	4.1	3.7
11	2	-13	11.7	11.7
11	2	-14	3.4	3.0
11	2	-15	9.9	8.5
11	2	-16	5.3	4.1
12	2	-3	2.1	2.6
12	2	-5	3.7	3.5
12	2	-6	7.7	7.6
12	2	-7	1.9	1.9
12	2	-8	4.1	4.9
12	2	-9	6.1	6.1
12	2	-10	7.7	8.0
12	2	-12	4.1	4.5
12	2	-13	3.2	3.0
12	2	-14	2.8	2.4
12	2	-15	5.6	5.3
12	2	-16	6.2	5.5
13	2	-9	7.9	7.6
13	2	-10	3.1	2.2
13	2	-11	3.5	4.1
13	2	-15	4.4	3.7
14	2	-11	1.5	2.4
14	2	-12	1.9	1.8
0	3	1	80.2	73.9
0	3	2	6.3	7.1
0	3	3	2.3	1.7
0	3	4	15.7	15.5
0	3	5	22.5	23.1
0	3	6	7.6	7.4
0	3	7	22.4	20.6
0	3	8	19.2	20.0
0	3	9	4.9	4.6
0	3	10	6.7	5.9
0	3	11	10.9	10.1
0	3	12	6.3	4.6
0	3	13	5.7	3.2
1	3	0	15.1	14.7
1	3	1	61.6	49.8
1	3	2	1.6	1.6
1	3	3	4.2	3.9
1	3	4	37.7	37.9
1	3	5	5.8	4.1
1	3	6	11.2	10.3
1	3	7	13.7	12.8
1	3	8	25.9	27.5
1	3	9	8.7	8.3
1	3	10	16.6	13.0
1	3	11	2.7	2.4
2	3	0	51.7	42.9
2	3	1	8.4	7.8
2	3	2	33.1	33.2
2	3	3	5.6	5.0
2	3	4	6.9	6.4
2	3	5	15.7	14.6

Table 7 (*cont.*)

H	K	L	|Fo|	|Fc|
2	3	6	11.6	11.0
2	3	7	8.9	7.6
2	3	8	22.6	24.8
2	3	9	6.1	5.4
2	3	10	3.4	3.6
2	3	11	11.9	9.5
2	3	12	7.2	6.2
3	3	0	10.9	9.0
3	3	1	6.7	6.6
3	3	2	13.6	14.0
3	3	3	7.4	7.3
3	3	4	10.7	11.2
3	3	5	14.8	12.5
3	3	6	13.6	14.2
3	3	7	7.3	7.6
3	3	8	18.8	17.2
3	3	10	8.0	7.5
3	3	11	9.4	9.1
4	3	0	38.2	35.3
4	3	1	17.9	16.6
4	3	2	23.6	23.2
4	3	3	4.7	4.1
4	3	4	13.0	7.3
4	3	5	17.1	17.2
4	3	6	16.2	14.5
4	3	8	4.7	4.6
5	3	0	13.4	11.7
5	3	1	15.0	14.4
5	3	2	15.3	13.9
5	3	3	12.7	11.9
5	3	4	5.0	5.1
5	3	5	6.8	7.1
5	3	6	14.4	13.6
5	3	7	14.1	12.6
5	3	8	4.1	3.9
6	3	0	17.7	17.5
6	3	1	16.7	16.7
6	3	2	19.3	19.1
6	3	3	14.9	15.4
6	3	4	6.1	6.2
6	3	5	7.9	8.0
6	3	6	5.2	3.6
6	3	7	10.3	9.7
7	3	0	5.4	4.5
7	3	2	6.8	6.9
7	3	4	12.2	12.3
7	3	5	16.4	16.5
7	3	6	2.4	3.0
8	3	0	5.0	5.5
8	3	1	15.1	15.6
8	3	3	2.7	2.4
8	3	4	8.9	8.7
9	3	1	8.3	9.0
9	3	2	3.6	4.3
9	3	3	6.2	6.7
10	3	0	4.8	5.3
1	3	-1	24.2	23.8
1	3	-2	4.9	5.7
1	3	-3	8.7	10.4
1	3	-4	14.0	18.2
1	3	-5	88.8	96.2
1	3	-7	9.1	11.6
1	3	-8	12.4	16.0
1	3	-9	5.2	6.3
1	3	-10	6.9	8.7
1	3	-11	10.6	13.4
1	3	-12	11.7	13.0
1	3	-13	1.8	1.9
1	3	-14	2.2	3.0
2	3	-1	14.1	11.5
2	3	-2	23.2	23.7
2	3	-3	30.4	30.9
2	3	-4	25.3	25.3
2	3	-5	87.1	89.3
2	3	-6	7.1	6.4
2	3	-7	40.1	45.5
2	3	-8	2.6	1.1
2	3	-9	5.6	6.1
2	3	-10	8.9	9.0
2	3	-11	13.8	15.8
2	3	-12	6.6	7.4
2	3	-14	9.0	7.9
2	3	-15	6.0	5.6
3	3	-1	10.9	8.3
3	3	-2	34.7	33.4
3	3	-3	1.8	1.9
3	3	-4	6.9	6.9
3	3	-5	9.7	10.2
3	3	-6	2.5	2.4
3	3	-7	7.5	8.7
3	3	-8	14.7	16.8
3	3	-9	25.5	26.0
3	3	-14	10.3	8.6
3	3	-15	10.6	9.7
4	3	-1	2.1	3.1
4	3	-2	86.5	82.9
4	3	-3	21.3	21.2
4	3	-4	2.3	1.8
4	3	-5	11.0	10.8
4	3	-6	12.9	13.6
4	3	-7	27.5	27.8
4	3	-8	4.6	3.8
4	3	-9	10.6	10.9
4	3	-10	5.0	5.4
4	3	-11	4.4	3.7
4	3	-12	6.9	6.8
4	3	-14	5.8	5.8
4	3	-15	12.5	11.3
4	3	-16	1.3	2.0
5	3	-1	15.6	15.4
5	3	-2	48.2	51.6
5	3	-3	25.1	25.1
5	3	-4	30.1	33.2
5	3	-5	2.2	1.7
5	3	-6	20.6	21.5
5	3	-7	24.4	27.3
5	3	-8	2.8	2.8
5	3	-9	7.6	7.2
5	3	-11	11.3	10.8
5	3	-12	7.4	7.3
5	3	-13	4.2	3.2
5	3	-14	2.1	1.5
5	3	-16	3.3	2.9
6	3	-2	11.3	10.8
6	3	-3	2.8	2.0
6	3	-4	5.6	5.7
6	3	-5	6.8	7.2
6	3	-6	7.8	8.3
6	3	-7	6.9	7.6
6	3	-8	16.6	16.8
6	3	-9	4.4	4.4
6	3	-10	5.8	1.4
6	3	-11	12.8	11.7
6	3	-12	7.4	7.2
6	3	-13	3.9	4.1
6	3	-14	5.9	6.4
6	3	-15	4.4	4.1
6	3	-16	2.9	2.2
6	3	-17	3.1	2.8
7	3	-1	29.7	31.6
7	3	-2	13.9	12.8
7	3	-4	30.0	28.7
7	3	-5	17.3	18.3
7	3	-6	4.7	4.1
7	3	-7	3.1	2.8
7	3	-8	22.4	22.3
7	3	-10	6.5	5.3
7	3	-11	6.7	6.4
7	3	-12	19.0	19.4
7	3	-13	2.7	2.3
7	3	-15	6.4	6.1
7	3	-17	7.3	5.8
8	3	-1	22.4	22.7
8	3	-2	10.1	10.3
8	3	-3	12.4	14.3
8	3	-4	30.6	30.2
8	3	-5	15.2	15.7
8	3	-6	2.6	2.4
8	3	-7	8.8	8.5
8	3	-8	6.6	6.5
8	3	-9	6.4	5.9
8	3	-11	1.6	1.7
8	3	-12	12.0	11.9
8	3	-14	1.5	2.0
8	3	-15	4.9	4.3
8	3	-16	2.2	2.2
9	3	-1	5.2	5.0
9	3	-2	9.5	10.4
9	3	-3	8.4	8.5
9	3	-4	6.3	6.6
9	3	-5	12.1	13.0
9	3	-6	3.5	3.6
9	3	-7	9.9	9.4
9	3	-8	14.1	13.7
9	3	-9	9.3	8.5
9	3	-10	1.6	2.0
9	3	-11	4.5	4.8
9	3	-12	1.6	1.5
9	3	-14	4.2	3.7
9	3	-15	3.1	2.6
9	3	-16	2.5	2.6
9	3	-17	4.5	3.3
10	3	-1	5.6	5.9
10	3	-2	12.5	12.9
10	3	-3	4.4	4.5
10	3	-4	3.7	3.9
10	3	-5	11.6	12.4
10	3	-6	2.2	1.9
10	3	-7	2.2	1.7
10	3	-9	5.0	5.8
10	3	-11	12.1	12.4
10	3	-13	3.4	3.3
10	3	-14	14.4	14.5
10	3	-15	2.3	2.6
10	3	-17	1.7	1.7
11	3	-1	3.5	4.3
11	3	-2	7.5	8.7
11	3	-3	3.0	2.6
11	3	-4	9.1	8.7
11	3	-5	8.0	9.1
11	3	-6	2.1	1.8
11	3	-8	5.4	5.7
11	3	-9	5.5	4.8
11	3	-11	7.8	8.2
11	3	-12	1.5	1.2
11	3	-13	7.9	8.1
11	3	-14	9.3	8.3
11	3	-15	1.7	1.5
11	3	-16	5.1	5.1
12	3	-3	1.3	1.4
12	3	-5	7.4	8.1
12	3	-7	5.1	5.0
12	3	-8	3.0	2.1
12	3	-10	6.4	6.7
12	3	-13	2.8	2.9
12	3	-14	4.1	2.0
12	3	-15	5.9	5.0
12	3	-16	2.9	2.7
13	3	-8	9.1	9.5
13	3	-9	4.1	3.2
13	3	-10	1.5	1.5
13	3	-11	5.1	5.2
13	3	-12	7.6	7.8
13	3	-14	2.1	2.0
13	3	-15	2.4	2.7
0	4	1	32.8	25.8
0	4	2	21.6	18.0
0	4	3	57.2	46.5
0	4	4	32.6	27.0
0	4	5	6.5	7.2
0	4	6	3.4	0.9
0	4	7	14.5	15.0
0	4	8	3.3	2.7
0	4	9	14.4	14.0
0	4	10	20.6	20.9
0	4	11	5.1	5.2
0	4	13	18.0	12.7
1	4	0	46.6	32.0
1	4	1	28.3	24.2
1	4	2	4.7	4.4
1	4	3	43.5	40.8
1	4	4	5.0	6.3
1	4	6	7.1	6.1
1	4	7	12.6	13.9
1	4	8	7.6	8.6
1	4	9	26.4	23.7
1	4	10	15.5	12.6
1	4	11	7.8	6.2
1	4	12	2.2	2.7
2	4	0	64.6	53.9
2	4	1	13.3	13.2
2	4	2	17.9	14.6
2	4	3	70.7	56.6
2	4	4	16.1	14.4
2	4	5	2.6	2.0
2	4	6	15.7	15.9
2	4	7	8.0	6.8
2	4	8	21.6	21.7
2	4	9	8.9	8.5
2	4	10	8.1	6.2
3	4	0	34.1	30.6
3	4	1	11.1	11.4
3	4	2	35.3	33.2
3	4	3	22.4	20.4
3	4	4	30.4	27.4
3	4	5	10.5	9.8
3	4	6	4.9	5.1
3	4	7	14.8	14.1
3	4	8	9.1	8.6
3	4	9	2.8	2.0
4	4	0	31.9	33.0
4	4	1	25.5	23.4
4	4	2	24.7	22.7
4	4	3	2.7	1.8
4	4	4	12.4	12.6
4	4	5	12.6	11.0
4	4	6	4.5	4.0
4	4	7	11.5	10.7
4	4	8	3.4	3.3
4	4	9	12.6	10.7
5	4	0	26.4	25.9
5	4	1	23.8	22.3
5	4	2	11.5	12.6
5	4	3	20.6	20.4
5	4	7	13.5	12.1
6	4	0	6.7	6.7
6	4	1	17.0	17.6
6	4	2	8.6	7.8
6	4	3	16.8	17.8
6	4	4	15.8	16.2
6	4	5	6.1	6.3
6	4	6	12.6	11.0
6	4	7	4.9	5.1
7	4	0	11.9	12.8
7	4	1	12.4	13.3
7	4	2	7.3	6.3
7	4	3	4.4	4.2
7	4	5	5.6	5.8
8	4	0	5.1	4.5
8	4	1	13.0	14.9
8	4	2	9.7	10.7
8	4	3	5.8	6.7
8	4	4	6.9	7.7
9	4	0	3.0	3.4
9	4	1	3.9	3.8
9	4	2	6.1	7.3
10	4	0	2.7	3.4
10	4	1	2.9	4.9
1	4	-1	13.8	14.0
1	4	-2	32.1	33.9
1	4	-3	16.5	20.1
1	4	-4	10.6	11.8
1	4	-5	10.2	11.1
1	4	-6	6.0	7.6
1	4	-7	16.8	21.3
1	4	-9	8.2	9.7
1	4	-10	9.4	12.7
1	4	-11	4.5	5.1
1	4	-12	10.0	10.7
1	4	-13	10.9	10.5
1	4	-14	6.5	7.2
2	4	-1	40.3	35.7
2	4	-2	2.4	3.1
2	4	-3	20.9	24.0
2	4	-4	22.1	20.9
2	4	-5	3.5	4.3
2	4	-6	2.7	1.1
2	4	-7	15.1	15.6
2	4	-8	6.3	6.7
2	4	-9	8.6	9.3
2	4	-10	5.1	5.5
2	4	-12	7.2	7.9
2	4	-13	6.7	7.4
3	4	-1	45.0	37.7
3	4	-2	11.6	13.5
3	4	-3	29.1	31.6
3	4	-4	6.6	6.7
3	4	-6	8.3	7.7
3	4	-7	15.7	18.1
3	4	-8	3.2	2.5
3	4	-9	10.0	9.1
3	4	-11	3.0	3.9
3	4	-12	13.3	15.1
3	4	-13	10.0	10.1
3	4	-14	2.4	2.3
4	4	-1	51.1	46.6
4	4	-2	34.9	31.6
4	4	-3	17.9	14.3
4	4	-4	1.5	1.6
4	4	-5	4.4	5.6
4	4	-6	13.6	16.0
4	4	-7	34.9	38.7
4	4	-9	3.4	2.5
4	4	-10	19.5	21.6
4	4	-13	12.6	12.6
4	4	-14	6.1	6.2
4	4	-16	5.3	5.2
5	4	-1	8.2	6.6
5	4	-2	17.2	16.7
5	4	-3	3.5	3.7
5	4	-4	23.0	25.8
5	4	-5	4.3	4.6
5	4	-6	15.2	16.0
5	4	-7	24.8	25.6
5	4	-8	18.0	18.3
5	4	-9	2.2	0.6
5	4	-10	12.8	13.3
5	4	-11	13.0	14.0
5	4	-13	3.4	3.5
5	4	-14	2.2	2.4
5	4	-15	4.8	4.4
6	4	-1	6.4	5.6
6	4	-2	14.8	12.9
6	4	-3	44.7	45.7
6	4	-4	16.1	15.5
6	4	-5	3.6	3.1
6	4	-6	10.1	10.2
6	4	-7	8.2	5.8
6	4	-8	19.7	21.2
6	4	-9	7.1	6.1
6	4	-10	9.6	9.9
6	4	-11	7.9	7.9
6	4	-12	2.4	2.7
6	4	-16	3.4	3.2
6	4	-17	4.3	4.3
7	4	-1	20.0	20.0
7	4	-2	12.1	12.0
7	4	-3	24.2	24.7
7	4	-4	3.5	2.4
7	4	-5	10.9	11.3
7	4	-6	8.0	6.9
7	4	-7	12.8	13.6
7	4	-8	14.8	16.3
7	4	-9	21.1	21.7
7	4	-11	4.8	4.8
7	4	-12	11.2	11.4
7	4	-13	2.9	3.7
7	4	-14	7.5	6.4
7	4	-15	2.1	2.3
7	4	-17	4.3	3.3
8	4	-1	14.7	16.3
8	4	-2	5.2	4.7
8	4	-3	4.2	4.6
8	4	-4	7.3	6.8
8	4	-6	2.8	3.1
8	4	-7	20.2	22.1
8	4	-8	4.9	4.4
8	4	-9	12.3	12.5
8	4	-10	8.7	8.6
8	4	-13	8.1	7.8
8	4	-15	5.4	5.8
8	4	-16	4.5	3.6
8	4	-17	3.0	2.3
9	4	-1	6.0	5.2
9	4	-2	7.3	8.2
9	4	-3	9.7	11.2
9	4	-4	3.0	3.5
9	4	-5	2.4	3.3
9	4	-6	6.9	8.2
9	4	-8	12.7	13.7
9	4	-9	7.5	7.8
9	4	-10	8.8	8.7
9	4	-11	4.6	5.3
9	4	-12	7.0	7.3
9	4	-13	17.5	15.5
9	4	-15	3.6	3.1
9	4	-16	3.8	3.1
10	4	-1	5.1	5.1
10	4	-2	2.1	1.3
10	4	-3	12.7	12.5
10	4	-4	12.2	11.8
10	4	-6	7.2	6.0
10	4	-7	3.8	4.2
10	4	-8	7.3	7.0
10	4	-9	9.8	7.9
10	4	-10	11.7	12.0
10	4	-11	2.4	1.9
10	4	-12	2.9	3.2
10	4	-13	6.0	5.1
10	4	-14	4.3	4.6
10	4	-15	3.7	3.7
10	4	-17	3.8	3.2
11	4	-1	3.0	3.7
11	4	-2	3.2	4.2
11	4	-3	11.5	12.9
11	4	-5	9.2	9.3
11	4	-6	11.3	11.4
11	4	-7	6.1	6.0
11	4	-8	6.8	6.7
11	4	-9	11.1	9.9
11	4	-10	2.3	2.2
11	4	-11	5.2	4.6
11	4	-14	3.4	2.6
11	4	-15	2.2	2.2
11	4	-16	1.5	1.5
11	4	-17	1.3	1.6
12	4	-4	6.9	8.6
12	4	-5	2.9	3.5
12	4	-6	6.1	5.6
12	4	-7	2.6	3.2
12	4	-8	3.6	4.1
12	4	-11	3.7	3.7
12	4	-13	2.9	2.7
12	4	-14	5.1	4.7
12	4	-15	3.4	3.6
13	4	-7	7.6	9.9
13	4	-9	5.6	6.1
13	4	-10	1.9	2.0
13	4	-11	4.5	4.3
13	4	-12	1.8	1.5
13	4	-13	4.8	5.2
13	4	-14	4.2	5.2
0	5	1	34.8	31.6
0	5	2	17.1	15.5
0	5	3	9.9	9.9
0	5	4	1.7	1.5
0	5	5	23.9	25.5
0	5	6	4.5	2.0
0	5	7	18.0	18.4
0	5	9	12.5	13.1
0	5	11	5.5	4.7
0	5	12	7.8	5.2
0	5	13	2.3	1.3
1	5	0	2.3	1.3
1	5	1	58.2	40.8
1	5	2	5.5	5.5
1	5	3	10.1	9.3
1	5	4	7.4	5.9
1	5	5	44.8	41.3
1	5	6	14.5	15.0
1	5	7	4.6	5.0
1	5	8	4.4	5.1
1	5	11	18.7	14.8
1	5	12	2.0	2.0
2	5	0	30.8	24.1
2	5	1	24.6	20.6
2	5	2	4.5	5.4
2	5	3	7.1	8.1
2	5	4	9.1	8.1
2	5	5	29.7	30.1
2	5	6	6.0	5.6
2	5	7	10.2	11.6
2	5	8	13.2	12.0
2	5	9	13.5	11.7
2	5	10	2.8	1.4
2	5	11	10.3	7.7
3	5	0	5.2	4.5
3	5	1	42.7	37.7
3	5	2	6.3	6.5
3	5	3	29.1	29.1
3	5	5	10.8	11.6
3	5	6	4.0	4.3
3	5	7	12.7	11.9
3	5	8	4.0	3.3
3	5	9	10.8	9.0
3	5	10	4.4	3.7
4	5	0	4.8	4.0
4	5	1	16.0	15.9
4	5	2	18.1	16.9
4	5	3	13.7	12.8
4	5	4	13.0	11.0
4	5	5	14.4	13.4
4	5	6	2.5	2.5
4	5	7	12.1	11.4
4	5	8	10.7	8.4
4	5	9	10.9	10.7
5	5	0	11.8	12.3
5	5	1	19.6	18.5
5	5	2	20.1	22.6
5	5	3	19.8	19.5
5	5	5	11.7	11.2
5	5	7	6.3	5.7
5	5	8	5.2	5.5
6	5	0	17.3	17.0
6	5	1	17.8	17.5
6	5	2	4.4	3.6
6	5	3	2.5	2.3
6	5	4	8.4	8.2
6	5	5	14.2	15.6
7	5	0	1.8	2.6
7	5	1	6.6	7.5
7	5	2	15.8	15.8
7	5	3	2.7	2.1
7	5	5	11.7	11.9
8	5	0	10.0	9.2
8	5	2	2.0	2.9
9	5	1	7.5	6.6
9	5	2	3.5	5.2
1	5	-1	29.3	22.8
1	5	-2	32.4	33.8
1	5	-3	5.5	5.2
1	5	-4	28.5	32.3
1	5	-5	24.0	30.7
1	5	-6	9.6	10.0
1	5	-7	13.6	16.8
1	5	-8	2.9	1.5
1	5	-11	10.0	11.4
1	5	-13	2.6	3.3
2	5	-1	22.1	16.2
2	5	-2	24.4	22.1
2	5	-3	4.7	5.7
2	5	-4	3.9	6.4
2	5	-5	16.9	19.7
2	5	-6	4.7	4.6
2	5	-7	15.0	17.0
2	5	-8	2.3	0.5
2	5	-9	5.9	7.4
2	5	-11	14.4	19.3
2	5	-12	2.4	3.0
2	5	-14	2.0	2.4
3	5	-1	30.2	26.7
3	5	-2	21.0	19.0
3	5	-3	14.9	15.3
3	5	-4	13.6	14.4
3	5	-5	17.5	18.5
3	5	-6	9.3	9.6
3	5	-8	2.7	2.4
3	5	-9	3.7	3.5
3	5	-10	3.1	3.4
3	5	-11	9.5	11.1
3	5	-13	3.2	3.2
3	5	-14	4.6	4.6
3	5	-15	7.3	7.1
4	5	-1	39.6	33.3
4	5	-2	19.8	17.5
4	5	-3	23.4	21.8
4	5	-4	20.4	22.8
4	5	-5	31.4	34.4
4	5	-6	3.9	3.1
4	5	-8	4.8	5.8
4	5	-9	6.1	5.6
4	5	-10	3.5	3.2
4	5	-14	4.5	4.4
4	5	-15	5.5	4.5
5	5	-1	24.1	25.7
5	5	-2	17.9	18.2
5	5	-3	14.7	14.5
5	5	-4	7.6	8.0
5	5	-5	30.5	32.8
5	5	-6	10.8	13.2
5	5	-7	3.3	3.2
5	5	-9	5.4	5.9
5	5	-10	3.9	3.6
5	5	-11	2.5	1.6
5	5	-12	5.7	5.9
5	5	-13	8.1	10.0
5	5	-15	8.2	8.3
6	5	-1	16.7	15.5
6	5	-2	11.0	10.3
6	5	-3	9.0	6.7
6	5	-4	3.0	2.1
6	5	-5	13.6	15.1
6	5	-6	6.8	6.9
6	5	-7	21.7	23.9
6	5	-8	2.3	2.3
6	5	-9	22.8	25.0
6	5	-10	3.9	4.3
6	5	-11	2.5	2.6
6	5	-12	9.3	9.1
6	5	-15	5.0	4.3
6	5	-16	2.6	2.4
7	5	-1	8.4	8.1
7	5	-2	6.1	5.0
7	5	-3	9.6	10.4
7	5	-5	12.4	13.0
7	5	-6	6.1	6.6
7	5	-7	16.7	17.2
7	5	-8	17.3	18.5
7	5	-9	10.8	11.9
7	5	-10	7.5	8.0
7	5	-11	12.0	12.0
7	5	-13	8.5	8.4
7	5	-14	6.1	5.2
7	5	-15	3.6	2.6
7	5	-16	3.6	2.8
8	5	-1	8.7	9.4
8	5	-2	3.1	3.9
8	5	-4	8.9	10.2
8	5	-5	6.8	6.4
8	5	-6	4.3	4.6
8	5	-7	14.4	15.2
8	5	-8	10.1	10.2
8	5	-9	10.9	11.6
8	5	-10	11.3	11.7
8	5	-11	13.9	15.3
8	5	-12	3.6	3.3
8	5	-13	12.3	11.1
8	5	-17	2.4	2.7
9	5	-1	13.5	13.7
9	5	-2	8.6	8.6
9	5	-4	15.4	14.5
9	5	-5	15.2	14.1
9	5	-7	3.1	2.8
9	5	-8	14.0	13.3
9	5	-9	4.7	4.4
9	5	-10	3.1	2.8
9	5	-11	3.1	2.8
9	5	-13	2.4	1.7
9	5	-15	8.6	7.9
9	5	-16	4.0	3.0
10	5	-1	10.1	10.9
10	5	-2	3.8	4.1
10	5	-3	9.9	9.6
10	5	-4	16.9	16.4
10	5	-5	14.8	14.2
10	5	-6	7.1	6.5
10	5	-8	11.9	12.1
10	5	-9	5.5	4.4
10	5	-10	13.7	12.6
10	5	-11	6.9	5.6
10	5	-13	2.2	1.9
10	5	-15	2.3	2.3
11	5	-2	6.2	8.9
11	5	-3	6.5	7.4
11	5	-5	6.3	5.8
11	5	-8	7.2	6.1
11	5	-11	9.0	8.3
11	5	-13	4.6	4.8
11	5	-14	4.0	3.6
11	5	-15	3.9	3.[illegible]
12	5	-5	6.2	7.1
12	5	-6	2.0	2.7
12	5	-9	4.3	4.0
12	5	-10	2.4	1.9
12	5	-11	5.7	1.8
12	5	-13	2.5	3.1
12	5	-14	5.9	5.7
12	5	-15	3.1	3.3
13	5	-8	1.8	2.4
13	5	-9	1.6	2.5

Table 7 (*cont.*)

H	K	L	|Fo|	|Fc|
13	5	-11	1.4	1.1
13	5	-13	2.6	3.0
0	6	1	32.1	29.3
0	6	2	3.0	1.7
0	6	3	7.2	8.2
0	6	4	5.4	6.5
0	6	5	13.0	14.1
0	6	6	16.2	17.6
0	6	7	20.8	21.5
0	6	8	14.2	14.8
0	6	9	8.5	7.9
0	6	10	9.4	8.2
0	6	11	4.3	3.7
0	6	12	10.2	7.2
1	6	0	11.1	8.2
1	6	1	6.1	5.0
1	6	2	4.9	4.5
1	6	3	25.6	22.4
1	6	4	10.3	10.4
1	6	5	8.9	9.2
1	6	6	16.5	16.9
1	6	7	16.9	20.0
1	6	8	6.0	5.5
1	6	9	8.4	7.1
1	6	10	6.2	5.2
1	6	11	6.1	4.9
2	6	0	3.1	2.2
2	6	1	17.5	15.6
2	6	2	21.6	18.7
2	6	3	37.7	32.8
2	6	4	32.7	28.5
2	6	5	19.1	20.2
2	6	6	14.4	15.4
2	6	7	6.0	5.4
2	6	8	10.2	8.4
2	6	9	16.4	15.0
2	6	10	6.0	4.5
2	6	11	5.4	6.2
3	6	0	12.0	11.6
3	6	1	18.2	16.9
3	6	2	7.3	6.0
3	6	3	58.3	47.0
3	6	4	19.9	19.7
3	6	5	13.9	14.2
3	6	6	7.9	8.0
3	6	7	12.4	10.9
3	6	9	11.6	9.8
4	6	0	9.5	9.8
4	6	1	22.3	22.1
4	6	2	4.0	2.9
4	6	3	20.8	20.2
4	6	4	13.8	14.3
4	6	5	11.3	10.7
4	6	7	14.9	13.5
4	6	8	3.8	2.4
5	6	0	4.7	3.7
5	6	1	11.4	11.7
5	6	2	7.0	7.0
5	6	3	3.6	3.0
5	6	5	9.8	10.2
5	6	6	4.8	3.8
5	6	7	13.4	12.9
6	6	0	7.3	7.2
6	6	1	20.2	21.3
6	6	2	7.3	6.4
6	6	5	7.4	5.9
7	6	0	5.1	5.9
7	6	1	6.2	5.2
7	6	2	3.3	2.5
7	6	3	15.0	16.6
7	6	4	5.3	5.0
7	6	5	1.6	2.0
8	6	2	6.3	6.2
8	6	3	10.1	10.9
9	6	0	2.9	2.4
9	6	1	5.0	5.3
1	6	-1	1.7	1.3
1	6	-2	1.9	2.7
1	6	-3	14.1	16.5
1	6	-4	14.1	14.1
1	6	-5	2.6	3.0
1	6	-6	8.5	9.7
1	6	-7	10.1	11.6
1	6	-8	3.1	3.7
1	6	-9	9.5	12.3
1	6	-13	7.0	7.0
2	6	-1	6.9	5.9
2	6	-2	18.9	19.3
2	6	-3	19.0	20.2
2	6	-4	23.1	23.6
2	6	-5	3.2	2.7
2	6	-7	2.8	2.4
2	6	-8	4.5	4.4
2	6	-9	12.3	14.5
2	6	-10	2.3	2.7
2	6	-13	9.5	8.9
3	6	-1	17.7	14.5
3	6	-2	30.0	24.7
3	6	-3	29.8	29.9
3	6	-4	19.5	20.1
3	6	-6	7.0	7.7
3	6	-7	12.6	14.1
3	6	-8	3.9	4.1
3	6	-9	5.8	5.9
3	6	-10	9.1	11.3
3	6	-12	4.0	4.6
3	6	-13	8.2	8.3
3	6	-14	4.6	3.5
4	6	-1	41.9	37.3
4	6	-2	8.0	6.5
4	6	-3	17.5	18.8
4	6	-4	1.6	0.3
4	6	-5	1.7	1.9
4	6	-6	9.1	10.3
4	6	-7	10.6	11.1
4	6	-9	10.7	10.6
4	6	-10	6.2	6.5
4	6	-12	2.7	2.6
4	6	-13	5.2	5.3
4	6	-15	2.1	1.6
5	6	-1	15.2	15.6
5	6	-3	10.9	13.8
5	6	-4	8.6	10.2
5	6	-5	17.0	18.0
5	6	-6	8.2	8.8
5	6	-7	37.4	35.9
5	6	-8	17.5	19.1
5	6	-9	5.3	6.0
5	6	-11	2.3	1.9
5	6	-12	7.1	7.3
5	6	-13	7.0	7.0
5	6	-14	2.3	1.9
5	6	-15	3.3	2.7
6	6	-1	8.3	7.9
6	6	-3	19.5	20.9
6	6	-4	5.5	5.2
6	6	-5	2.5	2.8
6	6	-6	9.8	9.8
6	6	-7	26.0	26.1
6	6	-8	5.2	6.0
6	6	-9	8.4	9.2
6	6	-10	7.8	7.7
6	6	-11	11.3	13.0
6	6	-12	6.6	6.3
6	6	-13	4.8	4.2
6	6	-16	3.9	4.1
7	6	-1	5.4	4.0
7	6	-2	5.8	6.8
7	6	-3	16.6	17.5
7	6	-4	2.7	2.1
7	6	-5	3.1	3.7
7	6	-6	9.7	10.8
7	6	-7	10.1	11.1
7	6	-8	14.7	17.2
7	6	-9	2.7	3.1
7	6	-10	10.4	10.6
7	6	-11	7.7	8.5
7	6	-12	5.9	6.0
7	6	-14	5.9	5.4
7	6	-16	2.1	1.6
8	6	-1	7.5	7.1
8	6	-3	15.0	14.5
8	6	-4	4.8	4.1
8	6	-5	4.8	4.5
8	6	-6	7.4	7.9
8	6	-7	5.1	4.6
8	6	-8	3.2	3.6
8	6	-9	13.2	14.0
8	6	-11	2.8	2.2
8	6	-12	4.1	4.8
8	6	-13	9.1	7.9
8	6	-14	2.7	2.4
8	6	-16	1.7	1.5
9	6	-1	9.5	8.9
9	6	-2	5.8	5.3
9	6	-3	3.4	2.5
9	6	-6	9.1	9.1
9	6	-7	7.4	7.2
9	6	-9	18.1	15.9
9	6	-11	3.5	3.7
9	6	-13	7.6	6.6
9	6	-14	2.3	2.2
9	6	-15	2.3	2.2
9	6	-16	2.1	2.2
10	6	-1	3.8	4.8
10	6	-2	4.8	5.0
10	6	-3	5.0	4.2
10	6	-4	2.4	2.2
10	6	-5	3.6	3.9
10	6	-7	2.6	1.8
10	6	-9	8.1	6.9
10	6	-11	5.7	5.4
10	6	-13	7.1	6.0
11	6	-3	7.9	9.5
11	6	-5	5.0	5.7
11	6	-6	4.2	3.5
11	6	-7	6.1	5.6
11	6	-10	2.7	2.5
11	6	-13	3.2	2.4
12	6	-6	3.5	4.0
12	6	-7	7.3	8.4
12	6	-9	2.4	1.9
12	6	-10	1.9	2.1
12	6	-11	2.9	2.8
12	6	-13	2.1	2.0
12	6	-14	1.1	1.1
0	7	1	2.1	1.8
0	7	2	4.1	2.3
0	7	3	12.0	10.0
0	7	4	10.5	8.9
0	7	5	35.0	27.5
0	7	6	3.9	4.0
0	7	8	4.6	4.4
0	7	9	3.8	4.6
0	7	10	3.5	3.8
0	7	11	9.6	9.3
0	7	12	4.5	3.8
1	7	0	2.5	2.0
1	7	1	4.9	2.5
1	7	2	19.3	15.8
1	7	3	4.2	4.6
1	7	4	19.7	18.2
1	7	5	23.5	21.5
1	7	6	6.7	6.2
1	7	7	11.4	12.3
1	7	8	9.6	9.3
1	7	9	6.2	7.3
1	7	11	7.8	6.8
2	7	0	22.9	18.0
2	7	1	3.0	1.9
2	7	2	19.2	14.4
2	7	4	22.5	19.3
2	7	5	18.3	19.0
2	7	7	1.9	1.6
2	7	9	6.9	6.3
2	7	10	6.1	5.6
3	7	0	9.5	6.1
3	7	1	24.6	19.2
3	7	2	16.4	15.2
3	7	3	24.0	21.5
3	7	4	17.2	16.1
3	7	5	16.8	16.2
3	7	6	7.4	6.2
3	7	7	11.4	10.0
3	7	9	7.7	8.9
4	7	0	19.2	14.7
4	7	1	41.1	34.1
4	7	2	6.9	5.1
4	7	3	18.1	17.1
4	7	4	7.0	6.4
4	7	5	14.2	13.3
4	7	7	11.9	11.2
5	7	0	5.1	4.6
5	7	1	11.3	10.6
5	7	2	14.6	12.6
5	7	3	3.2	3.0
5	7	4	5.8	4.2
5	7	5	9.9	9.0
5	7	6	4.0	3.7
5	7	7	6.4	7.1
6	7	0	9.9	9.7
6	7	1	5.8	5.7
6	7	2	3.8	3.2
6	7	4	5.9	4.9
6	7	5	11.9	12.6
7	7	0	1.9	2.3
7	7	1	12.7	10.5
7	7	4	4.7	4.4
8	7	1	7.2	6.4
9	7	0	6.1	6.1
1	7	-1	30.7	28.0
1	7	-2	6.0	6.5
1	7	-3	10.7	13.3
1	7	-4	6.9	9.1
1	7	-5	2.5	2.1
1	7	-6	8.8	12.8
1	7	-7	4.6	6.4
1	7	-9	4.5	7.8
1	7	-11	3.0	5.2
1	7	-12	8.0	9.0
2	7	-1	32.6	29.4
2	7	-2	16.3	15.9
2	7	-4	11.3	12.3
2	7	-5	14.1	17.9
2	7	-6	19.3	26.7
2	7	-7	12.2	16.2
2	7	-8	7.6	9.9
3	7	-1	9.2	9.3
3	7	-2	5.1	5.2
3	7	-3	4.1	4.2
3	7	-5	40.8	42.3
3	7	-6	5.4	6.5
3	7	-7	3.8	4.6
3	7	-8	8.9	11.6
3	7	-11	6.8	8.4
4	7	-1	4.3	3.4
4	7	-2	11.7	11.0
4	7	-3	5.0	4.4
4	7	-4	10.6	10.7
4	7	-5	23.6	25.8
4	7	-6	19.8	19.4
4	7	-7	1.7	1.4
4	7	-8	1.8	1.7
4	7	-9	5.2	5.5
4	7	-11	7.8	8.7
4	7	-12	4.5	5.3
5	7	-1	29.4	25.4
5	7	-2	9.6	8.6
5	7	-3	21.9	19.3
5	7	-4	17.4	17.0
5	7	-5	1.9	1.5
5	7	-6	22.9	24.6
5	7	-7	23.2	26.0
5	7	-8	11.4	12.2
5	7	-9	8.5	9.9
5	7	-10	6.6	6.5
5	7	-11	6.2	6.7
5	7	-13	4.2	3.9
5	7	-15	4.5	4.0
6	7	-1	17.8	15.0
6	7	-2	10.8	10.3
6	7	-3	22.4	19.2
6	7	-4	6.7	5.3
6	7	-5	19.7	19.1
6	7	-6	5.6	7.0
6	7	-7	9.8	10.7
6	7	-8	7.6	8.9
6	7	-9	6.9	7.8
6	7	-10	5.3	6.7
6	7	-11	7.3	8.6
6	7	-14	1.8	2.5
6	7	-15	5.4	4.8
7	7	-3	7.2	6.4
7	7	-4	5.3	4.5
7	7	-5	11.4	10.6
7	7	-6	5.2	4.7
7	7	-8	17.6	18.6
7	7	-9	7.4	8.8
7	7	-11	7.4	8.0
7	7	-12	2.2	2.5
7	7	-14	2.7	2.9
7	7	-15	4.3	3.9
8	7	-3	5.3	5.5
8	7	-5	10.3	10.6
8	7	-6	4.3	4.3
8	7	-7	2.8	2.5
8	7	-8	7.9	8.2
8	7	-9	4.5	5.0
8	7	-10	2.2	1.6
8	7	-11	1.5	1.2
8	7	-12	3.5	4.0
8	7	-14	3.4	3.4
8	7	-15	5.4	5.4
9	7	-1	11.2	11.1
9	7	-2	5.2	4.2
9	7	-3	4.3	4.0
9	7	-5	10.1	8.2
9	7	-6	4.4	3.9
9	7	-7	7.0	6.1
9	7	-9	4.4	3.5
9	7	-11	6.0	5.0
9	7	-12	5.9	4.7
9	7	-13	5.5	4.6
10	7	-1	6.3	9.0
10	7	-3	2.4	2.8
10	7	-4	1.8	1.5
10	7	-5	7.0	5.5
10	7	-6	1.4	2.0
10	7	-7	2.3	2.3
10	7	-8	2.5	2.4
10	7	-9	5.8	5.4
10	7	-10	4.7	4.0
10	7	-13	3.6	2.4
10	7	-15	1.5	1.2
11	7	-4	2.9	3.8
11	7	-5	5.2	6.2
11	7	-7	2.9	2.5
11	7	-8	5.5	5.6
11	7	-9	2.1	2.3
11	7	-10	3.0	3.0
11	7	-11	6.1	5.2
11	7	-12	4.2	3.7
11	7	-14	2.1	1.9
12	7	-8	4.1	4.3
12	7	-10	2.7	2.7
12	7	-11	6.8	7.3
0	8	1	8.9	7.2
0	8	2	5.8	5.3
0	8	3	18.5	15.5
0	8	4	12.4	11.9
0	8	5	18.0	19.5
0	8	6	19.6	19.9
0	8	7	6.6	7.2
0	8	9	6.1	6.8
1	8	0	1.5	0.9
1	8	1	27.5	20.9
1	8	3	24.3	19.9
1	8	4	4.2	3.6
1	8	5	5.5	5.4
1	8	6	2.7	1.9
1	8	7	8.4	9.2
1	8	10	7.4	6.5
2	8	1	15.8	12.8
2	8	2	6.7	4.9
2	8	3	9.6	8.8
2	8	4	15.3	15.3
2	8	5	7.3	7.4
2	8	6	9.2	9.6
2	8	7	11.7	10.6
3	8	0	31.3	24.6
3	8	1	23.0	18.2
3	8	2	16.2	14.0
3	8	3	4.5	4.9
3	8	4	3.8	3.3
3	8	6	12.7	10.6
3	8	7	11.9	10.5
4	8	0	5.5	4.7
4	8	2	8.0	6.9
4	8	3	23.2	20.5
4	8	4	12.5	9.8
4	8	5	5.5	4.6
4	8	6	9.4	7.2
4	8	7	9.4	9.4
5	8	0	5.3	4.2
5	8	1	6.6	6.4
5	8	2	7.5	7.3
5	8	3	17.6	13.7
5	8	4	10.6	8.6
5	8	5	4.8	4.2
5	8	6	4.8	4.7
6	8	2	12.7	9.8
6	8	3	13.5	11.9
6	8	4	6.4	6.1
7	8	0	3.0	2.4
7	8	1	8.1	6.5
7	8	3	3.9	3.7
8	8	0	4.4	3.4
8	8	1	6.5	6.6
1	8	-1	7.7	4.6
1	8	-2	2.9	3.8
1	8	-3	5.1	5.7
1	8	-4	7.6	9.6
1	8	-5	3.6	5.4
1	8	-6	16.1	27.9
1	8	-9	2.6	5.0
1	8	-11	4.5	6.0
2	8	-1	5.0	2.3
2	8	-2	6.4	6.4
2	8	-3	15.1	17.0
2	8	-4	20.1	20.2
2	8	-5	14.0	17.7
2	8	-7	7.0	11.0
2	8	-8	3.8	5.9
2	8	-9	2.7	4.1
2	8	-12	6.5	4.7
2	8	-13	3.1	4.6
3	8	-1	14.3	12.7
3	8	-2	6.7	5.6
3	8	-3	12.0	10.8
3	8	-4	8.4	8.0
3	8	-5	12.6	14.5
3	8	-6	14.0	16.7
3	8	-7	15.4	19.0
3	8	-8	9.5	13.3
3	8	-9	8.2	12.5
3	8	-10	4.9	7.8
3	8	-11	2.5	3.0
3	8	-13	2.0	3.1
4	8	-1	8.9	7.7
4	8	-2	22.1	17.4
4	8	-3	21.0	18.5
4	8	-4	25.8	23.8
4	8	-5	5.6	6.4
4	8	-7	14.9	17.8
4	8	-8	10.9	13.7
4	8	-9	5.0	5.8
4	8	-11	3.1	3.8
4	8	-13	4.4	5.3
4	8	-14	3.6	3.2
5	8	-1	10.9	9.7
5	8	-3	25.0	21.2
5	8	-4	24.1	19.5
5	8	-5	4.9	4.6
5	8	-6	9.8	10.1
5	8	-9	8.7	10.8
5	8	-10	2.3	2.0
5	8	-12	4.5	5.1
5	8	-13	5.8	6.0
5	8	-14	4.9	4.6
6	8	-1	18.1	15.4
6	8	-2	5.3	4.3
6	8	-3	5.2	4.7
6	8	-4	6.4	4.7
6	8	-5	2.6	3.1
6	8	-6	24.4	20.3
6	8	-7	15.0	13.2
6	8	-8	16.9	15.0
6	8	-9	7.1	8.4
6	8	-12	2.7	3.6
6	8	-13	3.8	3.6
6	8	-14	2.7	3.2
7	8	-1	12.4	9.3
7	8	-2	1.9	2.4
7	8	-3	4.6	4.5
7	8	-4	6.0	5.4
7	8	-5	11.0	10.3
7	8	-6	6.0	5.9
7	8	-7	6.6	6.0
7	8	-8	1.9	1.5
7	8	-9	3.5	3.4
7	8	-10	8.1	8.0
7	8	-12	4.8	3.9
7	8	-13	6.3	6.5
7	8	-14	4.5	4.1
8	8	-1	2.8	2.9
8	8	-3	3.6	2.7
8	8	-4	5.2	5.0
8	8	-6	3.7	3.1
8	8	-7	6.2	5.8
8	8	-10	2.6	2.9
8	8	-12	2.8	2.2
8	8	-13	5.1	4.7
9	8	-3	11.9	10.9
9	8	-7	5.6	4.6
9	8	-9	2.5	1.7
9	8	-12	4.6	3.4
10	8	-3	6.7	7.5
10	8	-4	1.6	1.5
10	8	-5	1.7	1.8
10	8	-6	8.2	7.0
10	8	-7	6.6	5.1
10	8	-8	3.3	2.8
10	8	-9	4.4	4.2
10	8	-10	1.9	1.9
10	8	-11	1.8	2.1
10	8	-12	5.4	4.5
10	8	-14	1.6	2.0
11	8	-6	3.9	4.1
11	8	-8	5.0	4.5
11	8	-9	7.0	7.0
11	8	-13	2.4	3.2
0	9	1	5.6	4.8
0	9	2	9.8	7.3
0	9	3	2.5	1.7
0	9	4	5.1	3.4
0	9	5	7.4	7.3
0	9	6	1.8	0.6
0	9	7	3.0	3.9
0	9	8	2.0	1.7
0	9	9	6.4	6.7
1	9	0	1.9	1.6
1	9	1	2.4	2.6
1	9	2	18.8	19.1
1	9	3	17.2	14.5
1	9	4	12.1	11.6
1	9	5	2.1	0.7
1	9	7	8.2	7.4
1	9	8	9.4	9.1
1	9	9	6.6	6.7
2	9	0	6.9	5.7
2	9	1	11.2	9.4
2	9	2	20.8	15.7
2	9	3	25.2	21.6
2	9	4	11.4	10.2
2	9	5	3.4	2.5
2	9	6	11.6	9.2
2	9	7	10.8	7.7
2	9	8	7.6	7.3
3	9	0	14.6	12.1
3	9	1	5.0	4.2
3	9	2	2.9	2.8
3	9	3	6.1	6.1
3	9	4	16.3	19.9
3	9	5	15.4	14.2
3	9	6	5.9	5.3
3	9	7	3.8	3.7
4	9	0	1.8	1.5
4	9	2	3.0	2.6
4	9	3	6.3	4.7
4	9	4	10.7	9.2
4	9	5	12.2	10.3
4	9	6	2.9	2.8
5	9	1	12.6	11.2
5	9	2	9.1	7.6
5	9	3	3.0	2.6
5	9	4	9.3	7.4
5	9	5	10.6	12.7
6	9	0	4.2	3.7
6	9	1	10.0	8.6
6	9	2	7.3	6.5
6	9	3	7.7	6.5
6	9	4	6.4	8.5
7	9	1	8.9	8.0
7	9	2	5.5	6.0
8	9	0	3.6	4.0
1	9	-1	7.3	6.3
1	9	-2	8.8	8.5
1	9	-3	6.3	7.4
1	9	-4	5.6	5.9
1	9	-5	18.8	26.7
1	9	-8	5.2	8.8
2	9	-1	10.5	9.5
2	9	-2	7.2	5.1
2	9	-4	3.5	2.8
2	9	-5	14.1	16.3
2	9	-6	4.7	5.3
2	9	-8	4.3	7.0
2	9	-9	3.0	4.3
2	9	-12	3.7	4.4
3	9	-1	25.5	22.5
3	9	-2	17.2	15.7
3	9	-3	7.3	6.5
3	9	-4	4.6	5.1
3	9	-5	4.6	5.2
3	9	-6	6.0	7.1
3	9	-7	6.3	7.6
3	9	-8	5.0	6.0
3	9	-9	2.6	3.8
3	9	-11	1.8	2.3
3	9	-12	4.6	5.3
4	9	-1	21.1	18.2
4	9	-2	13.3	12.8
4	9	-3	18.5	17.0
4	9	-4	15.2	14.8
4	9	-6	2.0	2.2
4	9	-7	13.9	16.1
4	9	-8	14.4	17.0
4	9	-11	7.2	9.5
4	9	-13	2.1	3.0
5	9	-1	15.4	13.3
5	9	-2	8.7	9.0
5	9	-3	11.2	10.3
5	9	-4	2.5	2.3
5	9	-5	19.3	19.3
5	9	-6	15.9	15.9
5	9	-7	3.3	3.0
5	9	-8	7.9	9.3
5	9	-9	1.8	2.2
5	9	-11	6.8	7.4
5	9	-12	3.5	3.9
6	9	-2	2.1	2.2
6	9	-4	8.5	8.0
6	9	-5	8.4	8.0
6	9	-6	12.3	12.6
6	9	-8	5.2	5.1
6	9	-10	3.6	3.9
6	9	-11	6.3	5.7
6	9	-12	7.1	7.5
7	9	-1	5.9	4.6
7	9	-2	8.9	8.0
7	9	-3	3.3	3.2
7	9	-4	6.2	5.0
7	9	-5	5.2	4.5
7	9	-6	4.8	4.8
7	9	-7	4.3	5.3
7	9	-9	2.9	3.8
7	9	-10	4.0	4.0
7	9	-12	2.1	2.3
8	9	-1	8.7	9.0
8	9	-2	6.4	5.5
8	9	-3	9.5	8.4
8	9	-4	2.0	2.0
8	9	-5	6.4	5.6
8	9	-6	5.6	4.5
8	9	-8	8.4	8.3
8	9	-12	2.0	1.8
8	9	-13	2.1	2.6
9	9	-2	2.7	2.5
9	9	-4	2.5	2.4
9	9	-5	6.6	5.7
9	9	-6	3.2	2.9
9	9	-9	3.6	2.9
9	9	-10	1.8	1.6
9	9	-11	2.0	2.2
9	9	-12	3.0	3.1
9	9	-13	1.5	1.9
10	9	-4	3.0	4.7
10	9	-5	4.6	4.9
10	9	-7	3.2	3.3
10	9	-8	5.8	5.4
10	9	-9	3.9	3.1
10	9	-11	2.1	2.2
10	9	-12	1.6	1.9

The hydrogen atoms of the methyl group are not completely staggered. This can be seen by looking down the C(7)–C(15) bond (Fig. 4) and the cause is repulsion between H(5) and $H(15)_3$ and between $H(15)_2$ and the aromatic ring *C*.

The aromatic C–C bond lengths vary from 1·363 ± 0·008 to 1·409 ± 0·006 Å; two of them, C(1)–C(2) and C(11)–C(12), being somewhat short (1·373 ± 0·007 and 1·363 ± 0·008 Å). Of the two $C_{sp^3}-C_{sp^3}$ bonds, C(7)–C(8) and C(7)–C(15) (1·557 ± 0·006 and 1·523 ± 0·007Å) the former is slightly long. It is probable that the e.s.d.'s are underestimated and none of the above mentioned distances are really significantly different from the mean values (1·394 ± 0·005 and 1·537 ± 0·005 Å respectively) given by Sutton (1965). (A block-diagonal approximation was used in the final least-squares calculation and no correlations between atoms were taken into account when bond-length e.s.d.'s were calculated.)

The two $C_{sp^2}-C_{sp^3}$ bonds, C(9)–C(8) and C(6)–C(7), have a mean value of 1·516 ± 0·005 Å which agrees with the value (1·505 ± 0·005 Å) given by Sutton (1965). The C_{sp^2}–O bond length of 1·426 ± 0·005 Å is comparable to that found in phenoxthionine (1·39 ± 0·04Å) (Hosoya, 1966) and is very close to the accepted C_{sp^3}–O distance (1·43 Å).

The C–H bond lengths vary from 0·86 ± 0·08 to 1·02 ± 0·06 Å with a mean value of 0·96 Å.

The S–O(1) and S–O(2) bond lengths are equal within experimental errors with a mean value of 1·431 ± 0·003 Å. The S–O(3) bond is shorter than the formal S–O single bond (1·69 Å) and has a π-bond order of about 0·25 (Cruickshank, 1961*b*), the same as in potassium ethyl sulphate. The bond lengths and valency angles associated with the sulphur atom are compared with values found in some related compounds in Table 8. There is some variation in the C–S bond lengths available in the literature. A value for the C–S single bond of 1·82 Å is given by Abrahams (1956), a mean

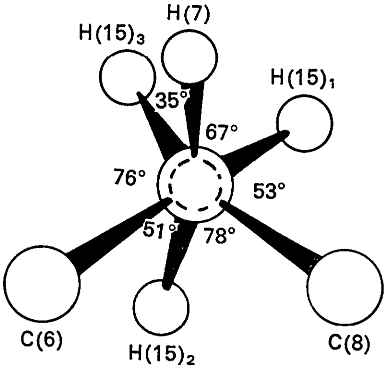


Fig. 4. View down the C(7)–C(15) bond showing the orientation of the hydrogen atoms of the methyl group.

Table 8. *Some bond lengths and valency angles in sultone B compared with values in related compounds*

Feature in sultone B		Compared with		in	
O(1)–S–O(2)	119·9 ± 0·2°	O=S=O	119·3 ± 0·9°	$(CH_3SO_2)CH$	(Silverton, Gibson & Abrahams, 1965)
			120·4 ± 0·4	$(p\text{-}Cl.C_6H_4)_2SO_2$	(Sime & Abrahams, 1960)
			131 ± 3	$(p\text{-}Br.C_6H_4)_2SO_2$	(Abrahams, 1956)*
			113 ± 1·5	$\beta\text{-}CH_3.C_4H_5SO_2$	(Abrahams, 1956)
O(1)–S–O(3)	108·6 ± 0·2	O=S–O	106·0, 108·5, 101·5 (± 0·4)	$KSO_3.C_2H_5$	(Truter, 1958)
O(2)–S–O(3)	103·6 ± 0·2				
O(1)–S–C(8)	109·3 ± 0·2	O=S–C	107, 106, 107, 111 (± 1°)	$(CH_3SO_2)_3CH$	
O(2)–S–C(8)	113·1 ± 0·2		107·3, 108·0 (± 0·3)	$(p\text{-}Cl.C_6H_4)_2SO_2$	
			109 ± 5°	$(p\text{-}Br.C_6H_4)_2SO_2$	
			105 ± 3	$(CH_3)_2SO_2$	(Abrahams, 1956)
			100 ± 1·5	$\beta\text{-}CH_3.C_4H_5SO_2$	
C(1)–O(3)–S	117·1 ± 0·3	C–O–S	115·3 ± 0·6	$KSO_3C_2H_5$	
C(8)–S–O(3)	100·2 ± 0·2	C–S–C	105 ± 1	$(CH_3SO_2)_3CH$	
			104·8 ± 0·4	$(p\text{-}Cl.C_6H_4)_2SO_2$	
			100 ± 5	$(p\text{-}Br.C_6H_4)_2SO_2$	
			98 ± 1·5	$\beta\text{-}CH_3.C_4H_5SO_2$	
S–O(1)	1·428 ± 0·004 Å	S=O	1·435 ± 0·010 Å	$(CH_3SO_2)_3CH$	
S–O(2)	1·434 ± 0·004		1·432 ± 0·005	$(p\text{-}Cl.C_6H_4)_2SO_2$	
			1·436 ± 0·017	$\beta\text{-}CH_3.C_4H_5SO_2$	
			1·43 ± 0·02	$(CH_3)_2SO_2$	
S–O(3)	1·595 ± 0·004	S–O	1·604 ± 0·004	$KSO_3C_2H_5$	
S–C(8)	1·797 ± 0·005	$S\text{–}C_{sp^3}$	1·83 ± 0·01	$(CH_3SO_2)_3CH$	
			1·73 ± 0·02		
			1·75 ± 0·02	$\beta\text{-}CH_3.C_4H_5SO_2$	
			1·80 ± 0·02	$(CH_3)_2SO_2$	
		$S\text{–}C_{sp^2}$	1·765 ± 0·006	$(p\text{-}Cl.C_6H_4)_2SO_2$	

* A number of the structures reviewed by Abrahams (1956) were examined with limited data and the results are not accurate.

value based on six compounds and Cox & Jeffrey (1951) give a mean value of 1·812 Å from eleven compounds. The length of the S–C(8) bond in sultone B is on the short side of both these mean values. It is also shorter than the central carbon–sulphur bond in $(CH_3SO_2)_3CH$ (Table 8) but considerably longer than the methyl–sulphur bond in the same compound. It is also longer than the S–C_{sp^2} bond in $(p\text{-Cl.}C_6H_4)_2SO_2$ where overlap of the sulphur 3*d* orbitals and the adjacent carbon atoms 2*p* orbitals occur.

The atoms around the sulphur atom are arranged in a distorted tetrahedron with angles ranging from 100 to 120°. The C(8)–S–O(3) angle is close to the C–S–C angles found in sulphones (Table 8). The angles O(1)–S–C(8) and O(1)–S–O(3) are comparable as might be expected. However, the close approach of carbon C(9) of the phenyl ring to O(2) may be the cause of an increase in the O(2)–S–C(8) angle and a consequent decrease in the O(2)–S–O(3) angle. As a result of this displacement the non-bonded distance O(2)···O(3) (2·383 Å) is shorter than O(1)···O(3) (2·456 Å). Both of these distances are shorter than the O(1)···O(2) distance (2·477 Å) which is in agreement with McDonald & Cruickshank's (1967) findings that the O···O distance in a tetrahedron is longer for shorter S–O bond lengths.

The average of the angles in the aromatic rings is 120·1°, but there are several angles which deviate significantly from 120°. Deviations of the angles in ring *A* are caused by strain due to the long S–C(8) bond in the heterocyclic ring. Distortions of the angles around C(9) are probably caused by close contacts between the hydrogen [H(10)] of the phenyl ring and the carbon atoms C(1) and C(6) as well as between H(14) and H(8). There are also significant differences of the angles around C(7) and C(8) from the tetrahedral angle of 109·5° caused by interactions of the methyl group with C(5) and with the phenyl ring as well as interactions of H(10) with C(1) and C(6).

The C–C–H angles vary from 112 ± 4° to 128 ± 4° with a mean value of 120° in the aromatic part and from 95 ± 5° to 119 ± 5° with an average of 108° for the tetrahedrally arranged hydrogen atoms.

Intermolecular distances

The twelve contacts, shorter than 3·5 Å, which each molecule makes with seven neighbouring molecules (Fig. 5) are listed in Table 5; there are fifty contacts less than 4·0 Å with fourteen neighbouring molecules. Three of the oxygen–carbon contacts, O(2)···C(15), O(2)···C(13) and O(2)···C(14) (3·347, 3·348 and 3·368 Å), are shorter than the oxygen–carbon van der Waals distance (3·4 Å), but the corresponding oxygen–hydrogen contacts O(2)···$H(15)_3$, O(2)···H(13) and O(2)···H(14) (2·64, 2·68 and 2·84 Å) are equal to or larger than the oxygen–hydrogen van der Waals distance (2·6 Å) so there are no hydrogen bonds.

We wish to thank Professor R. F. Timoney for sending us the crystals and Professor J. M. Robertson, C.B.E., F.R.S., for his interest. The computing was

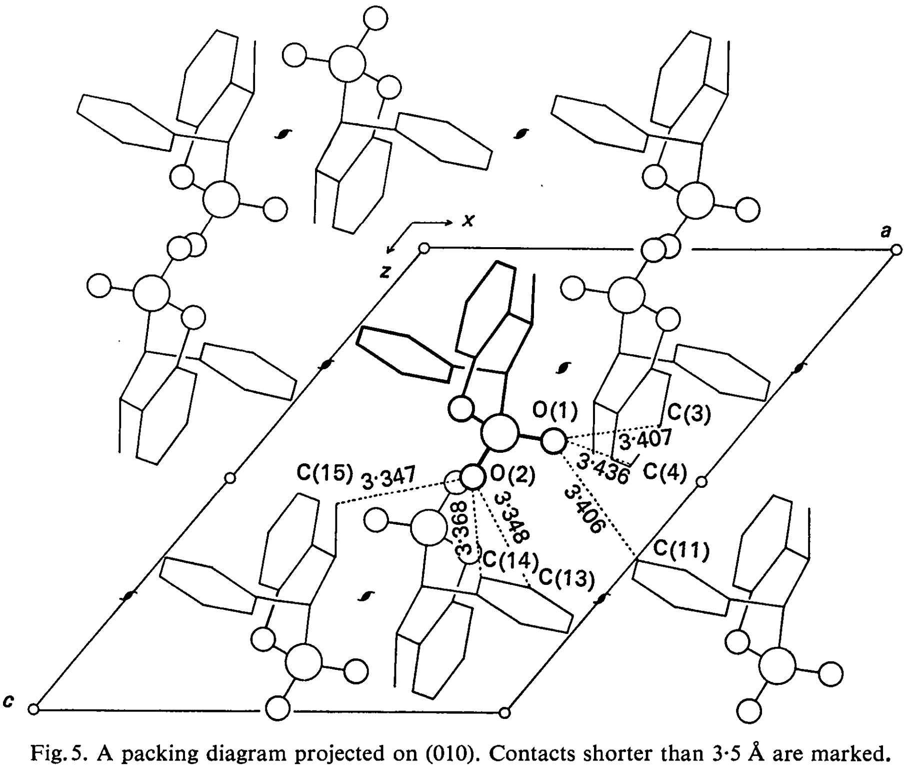


Fig. 5. A packing diagram projected on (010). Contacts shorter than 3·5 Å are marked.

carried out on the Glasgow University KDF9 computer. Programs made available by D. McGregor, K. W. Muir, R. Pollard and Dr J. G. Sime were used in the Fourier refinement. The least-squares program was devised by Professor D. W. J. Cruickshank and J. G. F. Smith and the rigid-body thermal vibration analysis program by Dr W. S. McDonald and K. W. Muir. We are grateful to the referee for useful comments.

References

ABRAHAMS, S. C. (1956). *Quart. Rev. Chem. Soc. Lond.* **10**, 407.

BARTON, D. H. R. & COOKSON, R. C. (1956). *Quart. Rev. Chem. Soc. Lond.* **10**, 44.

COX, E. G. & JEFFREY, G. A. (1951). *Proc. Roy. Soc.* A, **207**, 110.

CRUICKSHANK, D. W. J. (1956). *Acta Cryst.* **9**, 754.

CRUICKSHANK, D. W. J. (1961*a*). *Acta Cryst.* **14**, 896.

CRUICKSHANK, D. W. J. (1961*b*). *J. Chem. Soc.* p. 5486.

HOSOYA, S. (1966). *Acta Cryst.* **20**, 429.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

MCDONALD, W. S. & CRUICKSHANK, D. W. J. (1967). *Acta Cryst.* **22**, 37.

SILVERTON, J. V., GIBSON, D. T. & ABRAHAMS, S. C. (1965). *Acta Cryst.* **19**, 651.

SIME, J. G. & ABRAHAMS, S. C. (1960). *Acta Cryst.* **13**, 1.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175.

SUTTON, L. E. (1965). Special Publication No. 18. London: The Chemical Society.

TRUTER, M. R. (1958). *Acta Cryst.* **11**, 680.

TIMONEY, R. F. (1966). Private communication.

Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

Acta Cryst. (1967). **23**, 664

Inclusion of secondary extinction in least-squares calculations*.

By ALLEN C. LARSON, *University of California, Los Alamos Scientific Laboratory, Los Alamos, New Mexico, U.S.A.*

(*Received* 13 *March* 1967)

A procedure for including secondary extinction as a parameter in crystal structure least-squares analysis is described and the effect of its use on the data for chrysoberyl is given.

Several years ago at this laboratory we began including the secondary extinction as a parameter in our least-squares refinements of crystal structure models. Several papers have now been published in which the results of such calculations have been included, as well as the equations used to define the least-squares model (*e.g.* Cromer, Larson & Roof, 1964). It is the purpose of this note to call attention to the technique and to present in support of its use some results on the refinement of chrysoberyl (Åsbrink & Werner, 1966), the data for which are quite strongly affected by extinction.

Common practice when severe extinction is evident is to omit the more greatly affected data from the final refinement. It is obviously undesirable, however, to remove the stronger and more heavily weighted data points. Less commonly the observed data are corrected by such methods as those of Hamilton (1957) or Zachariasen (1963). Correcting the observed data convolutes those data with a crystal 'parameter' which is most easily determined after the structure is known. Furthermore, the observed data published in a paper should be given as observed, not as corrected by some function which is a parameter of the crystal.

Starting with equation (13) of Zachariasen's (1963) paper and using his notation,

$$R/I_0 \approx R_0/I_0(1-\alpha R_0/I_0)\,. \tag{1}$$

Substituting for R/I_0 and R_0/I_0 one obtains

$$F_o^2 \approx F_c^2(1-g\beta F_c^2)\,, \tag{2}$$

where

$$\beta = \left(\frac{e^2N}{mc^2}\right)^2 \frac{p_2}{p_1}\,\frac{A\,dA^*/d\mu}{\sin 2\theta}\,.$$

Three approximations for the application of (2) to least-squares calculation will be described along with the derivatives needed. Results of tests on chrysoberyl (Åsbrink & Werner, 1966) are given below. The least-squares calculations were carried out by minimizing the quantity $\Sigma w(F_o-F_c^*)^2$, with

$$F_c^* = KF_c[1+g\beta(2\theta)F_c^2]^{-\frac{1}{2}} \tag{3}$$

$$F_c^* = KF_c[1-g\beta(2\theta)F_c^2] \tag{4}$$

and

$$F_c^* = KF_c[1-g\beta(2\theta)F_c^2]^{\frac{1}{2}}\,, \tag{5}$$

where g is the secondary extinction parameter, K is a scale factor, and

$$\beta(2\theta) = \frac{p_2}{p_1}\,\frac{A\,dA^*/d\mu}{\sin 2\theta}\,. \tag{6}$$

Equation (3)

Use of equation (3) is tantamount to assuming that the higher order terms of Zachariasen's (1963) equation (9), which were neglected in equation (2), are equal to $(-g\beta F_c^2)^n$, a fairly reasonable assumption.

The derivatives of F_c^* are

$$\frac{\partial F_c^*}{\partial \xi_i} = \left(\frac{\partial F_c}{\partial \xi_i}\right) K\,[1+g\beta(2\theta)F_c^2]^{-3/2} \tag{7}$$

$$\frac{\partial F_c^*}{\partial K} = F_c\,[1+g\beta(2\theta)F_c^2]^{-\frac{1}{2}} \tag{8}$$

---

* Work performed under the auspices of the U.S. Atomic Energy Commission.