ACTA CHEMICA SCANDINAVICA 23 (1969) 1852–1870

The Crystal and Molecular Structure of 2,4-Diphenyl-thiothiophthene

ASBJØRN HORDVIK, EINAR SLETTEN and JORUNN SLETTEN

Chemical Institute, University of Bergen, Bergen, Norway

An X-ray structure investigation of crystals of 2,4-diphenyl-thiothiophthene has been carried out. The crystals belong to the space group $P\bar{1}$, with the unit cell dimensions: $a=10.224$ Å, $b=8.486$ Å, $c=10.291$ Å, $\alpha=118.45°$, $\beta=94.55°$, and $\gamma=101.40°$. There are two molecules per unit cell.

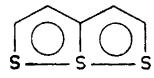
The structure was solved by three-dimensional Patterson synthesis, and refined by least squares methods. The refinement comprises 2011 observed $hk0-hk8$, $h0l$ and $h1l$ reflections.

Unequal S–S distances occur in the linear tri-sulphur sequence of the molecule, *i.e.*, S(1)–S(2)=2.499±0.003 Å, S(2)–S(3)=2.218 ±0.003 Å with the angle S(1)–S(2)–S(3)=178.1±0.1°. The other bond lengths in the thiothiophthene system are, S(1)–C(1)=1.696 ±0.005 Å, S(2)–C(3)=1.741±0.005 Å, S(3)–C(5)=1.669±0.007 Å, C(1)–C(2)=1.374±0.011 Å, C(2)–C(3)=1.398±0.009 Å, C(3)–C(4)=1.448±0.011 Å and C(4)–C(5)=1.360±0.009 Å.

The C–C bonds connecting the phenyl groups to the thiothiophthene system are, C(1)–C(6)=1.500±0.009 Å and C(4)–C(12)=1.482±0.007 Å.

The phenyl groups are planar within the error, and the thiothiophthene system is almost so. The phenyl groups are not co-planar with the thiothiophthene system. Thus, the phenyl group bonded to C(1) is twisted 24.7° about the C(1)–C(6) bond, and the phenyl group bonded to C(4) is twisted 51.6° about the C(4)–C(12) bond.

There are two different kinds of delocalized bonding in the thiothiophthene molecule (I).



(I)

1. There is a delocalized σ-system in the tri-sulphur sequence, equivalent to that in trihalide ions and referred to as a three-center four-electron bond.

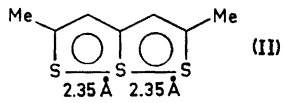
2. There is in addition a delocalized π-system comprising the 10 electrons in p-orbitals perpendicular to the plane of the two fused rings, *i.e.*, a π-system analogous to that in naphthalene.

The electronic structure of the thiothiophthene molecule has been calculated by Giacometti and Rigatti,[1] in terms of sulphur-sulphur bonds made up from both fractional σ and π bonds. From the results of their calculations, the molecule is symmetrical.

The potential energy of the three-center bond in thiothiopthene as a function of the displacement of the central sulphur atom from the symmetrical location toward the terminal sulphur atoms, has recently been calculated by Gleiter and Hoffmann.[2] Two energy curves are given, one for three-center bonds formed by combination of sulphur $3p$-orbitals only, the other for three-center bonds based on combination of $3d$-orbitals as well. The former curve favours an unsymmetrical structure and the latter favours a symmetrical one. It is interesting to note that the energy-minimum of the latter is flat and broad, about 0.3 Å.

As the bonds in the linear sulphur sequence of the thiothiophthene molecule are fractional in both σ- and π-character, one may assume that they are weaker than the other bonds in the molecule and therefore more liable to changes in bond length if the σ-system, the π-system, or both, are perturbed to some degree. It seems likely that intermolecular environment (donating or accepting groups) as well as intramolecular environment (substituents) may cause such perturbation.

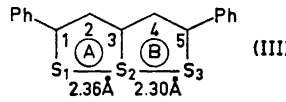
The results from the structure study of the symmetrical derivative (II), by Bezzi *et al.*,[3,4] show that the sulphur-sulphur distances there are equal, *i.e.*,



(II)

2.36 Å (later,[5] 2.35 Å was reported) as compared with the value 2.10 Å for a sulphur-sulphur single bond in a *cis*-planar disulphide group.[6] However, it has been pointed out by different authors [7,8] that the results from (II) could be ambiguous since they may represent the average of twofold disorder of molecules with a "short" and a "long" S—S distance. The molecules lie in special positions, with crystallographic mirror plane passing through the central sulphur and carbon atoms, and the central sulphur atom shows a pronounced anisotropy which may indicate such disorder.

It has recently been shown by one of us (A.H.) [9] that the sulphur-sulphur distances in the symmetrical diphenyl derivative (III) are slightly unequal,



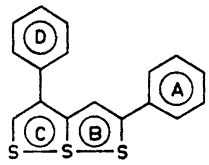
(III)

i.e., S(1)—S(2)=2.355±0.003 Å and S(2)—S(3)=2.297±0.003 Å. However, the difference of 0.06 Å may be caused by intermolecular environment since S(3) approaches the plane of *A* in a symmetry related molecule at a distance

of 3.25 Å, with the bond S(3)—C(5) forming an angle of 77° with this plane. The distance is 0.30 Å shorter than the sum of the van der Waals radius for sulphur and the half-thickness of an aromatic molecule, 3.55 Å,[10] and in fact compatible with a corresponding close contact present in crystals of the benzene-chlorine (1:1) chargetransfer complex.[11] There the molecules are arranged perpendicular to the planes of the benzene molecules, and the distance from a chlorine atom to the nearest benzene plane is 3.28 Å as compared with the corresponding van der Waals distance of 3.50 Å.

It seems likely, therefore, that in crystals of (III) there is a transfer of charge from the π-system of the molecule towards S(3) of a symmetry related molecule, thus making S(3) less electronegative than S(1). This may, if analogy is drawn between the bonding in the sulphur sequence and that in trihalide ions,[12,13] explain why S(1)—S(2) in (III) is found to be somewhat longer than S(2)—S(3). Hence one may assume that the S—S distances in any completely symmetrical thiothiophthene derivative are exactly equal in an isolated molecule.

The present investigation of 2,4-diphenyl-thiothiophthene (IV) was carried out in order to find to which degree phenyl-substituents in unsymmetrical positions influence the bonding in the sulphur sequence.



(IV)

EXPERIMENTAL

Crystal data on 2,4-diphenyl-thiothiophthene have been reported earlier.[14] The compound crystallizes from benzene as rather irregularly shaped orange crystals. The crystals are triclinic, with $a=10.224$ Å, $b=8.486$ Å, $c=10.291$ Å, $\alpha=118.45°$, $\beta=94.55°$, and $\gamma=101.40°$. The experimental error is estimated to be within 0.2 %. There are two molecules per unit cell; density, calc. 1.380, found 1.387 g/cm^3. The "average test" and the "zero moment test",[15,16] based on 213 $h0l$ reflections, show that the space group is $P\bar{1}$.

The intensities of the $hk0-hk8$, $h0l$ and $h1l$ reflections were estimated visually from Weissenberg photographs taken with Cu$K\alpha$ radiation ($\mu=40.3$ cm^{-1}). Small crystals of cross-section 0.06×0.06 mm were used in order to minimize absorption effects, and no absorption correction was applied. 2011 of the 2566 reflections obtainable with Cu$K\alpha$ radiation were observed. The intensities were corrected in the usual way to give sets of relative structure factors. Common reflections in $hk0-hk8$, $h0l$ and $h1l$ were used to put all the reflections on the same scale. The calculated structure factors in Table 6 are based on the atomic scattering curves for sulphur, carbon and hydrogen given in the *International Tables*,[17] the first set of the listed scattering factors for carbon being used.

DETERMINATION OF THE STRUCTURE

A brief account of the structure determination has been reported earlier,[18] and a more detailed description is given here.

Approximate coordinates for the sulphur atoms and the carbon atoms of the thiothiophthene system were found from a three-dimensional Patterson

synthesis. Furthermore, by taking packing and intramolecular non-bonding atomic distances into account, it seemed likely that the angle between the plane of ring *D* and the plane of the thiothiophthene system was greater than the angle between the plane of ring *A* and the plane of the thiothiophthene system. However, the twist direction of the phenyl groups about the respective connecting bonds were ambiguous, and it seemed reasonable, therefore, to try a structure with *D* perpendicular to and *A* coplanar with the plane of the thiothiophthene system. This trial structure refined, and it is interesting to note that the rings *A* and *D*, without distortion, turned 25 and 38° about the respective connection bonds during the refinement.

The atomic parameters were refined by least squares methods on an IBM 1620 II computer, using a block-diagonal program designed by Mair.[19] Weighting scheme No. 3, recommended by Mair was used with $F_{min}=0.75$. The refinement comprises the 2011 observed *hk*0—*hk*8, *h*0*l* and *h*1*l* reflections. The unobserved reflections were neglected in order to save computer time.

Anisotropic temperature factors were applied to sulphur and carbon, and isotropic to hydrogen. The strong low order reflections were corrected

Table 1. Atomic coordinates in fractions of corresponding cell edges.

Atom	*x*	*y*	*z*
S(1)	0.32494	0.16258	0.25631
S(2)	0.12776	−0.10566	0.07678
S(3)	−0.04498	−0.34401	−0.08707
C(1)	0.37638	0.18880	0.11336
C(2)	0.30203	0.06846	−0.03058
C(3)	0.18833	−0.07789	−0.06649
C(4)	0.11246	−0.20484	−0.21927
C(5)	−0.00047	−0.33392	−0.23609
C(6)	0.49856	0.34196	0.14434
C(7)	0.57109	0.32368	0.03253
C(8)	0.68267	0.46644	0.05740
C(9)	0.72381	0.62721	0.19898
C(10)	0.65391	0.64509	0.30942
C(11)	0.54167	0.50159	0.28470
C(12)	0.15674	−0.19482	−0.34944
C(13)	0.06713	−0.18171	−0.45057
C(14)	0.10815	−0.17585	−0.57265
C(15)	0.23639	−0.18168	−0.59910
C(16)	0.32552	−0.19477	−0.49899
C(17)	0.28710	−0.20193	−0.37578
H(2)	0.3240	0.0965	−0.1050
H(5)	−0.0536	−0.4078	−0.3177
H(7)	0.5245	0.1965	−0.0781
H(8)	0.7209	0.4658	−0.0270
H(9)	0.8037	0.7191	0.1962
H(10)	0.6786	0.7751	0.4202
H(11)	0.4869	0.5092	0.3462
H(13)	−0.0272	−0.1822	−0.4242
H(14)	0.0547	−0.1534	−0.6248
H(15)	0.2795	−0.1793	−0.6894
H(16)	0.4211	−0.2061	−0.5149
H(17)	0.3467	−0.2096	−0.3091

Table 2. Temperature parameters β_{ji} for sulphur and carbon, and B for hydrogen. The expressions used are $\exp-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+hk\beta_{12}+kl\beta_{23}+hl\beta_{13})$ for sulphur and carbon, and $\exp-[B(\sin^2\theta/\lambda^2)]$ for hydrogen.

	β_{11}	β_{22}	β_{33}	β_{23}	β_{13}	β_{12}
S(1)	0.0132	0.0295	0.0110	0.0213	0.0062	0.0034
S(2)	0.0119	0.0268	0.0155	0.0281	0.0120	0.0080
S(3)	0.0125	0.0253	0.0218	0.0276	0.0107	−0.0028
C(1)	0.0110	0.0211	0.0103	0.0185	0.0066	0.0113
C(2)	0.0098	0.0209	0.0108	0.0188	0.0050	0.0056
C(3)	0.0099	0.0198	0.0124	0.0207	0.0078	0.0085
C(4)	0.0090	0.0178	0.0137	0.0163	0.0046	0.0050
C(5)	0.0119	0.0209	0.0196	0.0216	0.0046	0.0025
C(6)	0.0091	0.0187	0.0127	0.0174	0.0014	0.0062
C(7)	0.0101	0.0214	0.0171	0.0202	0.0081	0.0065
C(8)	0.0109	0.0264	0.0202	0.0268	0.0077	0.0012
C(9)	0.0122	0.0245	0.0264	0.0318	0.0022	0.0027
C(10)	0.0204	0.0197	0.0189	0.0137	−0.0012	−0.0002
C(11)	0.0149	0.0203	0.0147	0.0130	0.0062	0.0070
C(12)	0.0091	0.0156	0.0094	0.0127	0.0006	0.0032
C(13)	0.0122	0.0213	0.0137	0.0203	0.0009	0.0064
C(14)	0.0191	0.0248	0.0158	0.0238	−0.0064	0.0061
C(15)	0.0197	0.0222	0.0143	0.0205	0.0085	0.0084
C(16)	0.0151	0.0314	0.0156	0.0250	0.0139	0.0162
C(17)	0.0121	0.0273	0.0125	0.0234	0.0062	0.0130

	B (Å²)		B (Å²)		B (Å²)
H(2)	3.4	H(9)	7.7	H(14)	2.7
H(5)	2.8	H(10)	4.6	H(15)	3.7
H(7)	5.5	H(11)	1.7	H(16)	4.9
H(8)	4.1	H(13)	1.4	H(17)	4.8

for secondary extinction according to Lipson's formula.[20] These reflections are marked with asterisks in Table 6. The final R factor is 0.070.

Final coordinates and temperature parameters are given in Tables 1 and 2, respectively.

DISCUSSION

Molecular shape and dimensions. Bond lengths and angles in the 2,4-diphenyl-thiothiophthene molecule, together with their standard deviations, are listed in Tables 3 and 4 and shown in Figs. 1a and 1b, respectively. The values correspond to the coordinates in Table 1.

The molecule is presented in Figs. 1a and 1b in a projection on to the least squares plane of the thiothiophthene system. The equation for this plane, with triple weight on sulphur, is

$$-0.74700\ X + 0.79237\ Y - 0.16987\ Z = -1.82479$$

with X, Y, and Z in Å units. Deviations from the plane for the atoms of the thiothiopthene system, the atoms C(6) and C(9) of phenyl group A and the atoms C(12) and C(15) of phenyl group D are given in Fig. 1a. It is seen that

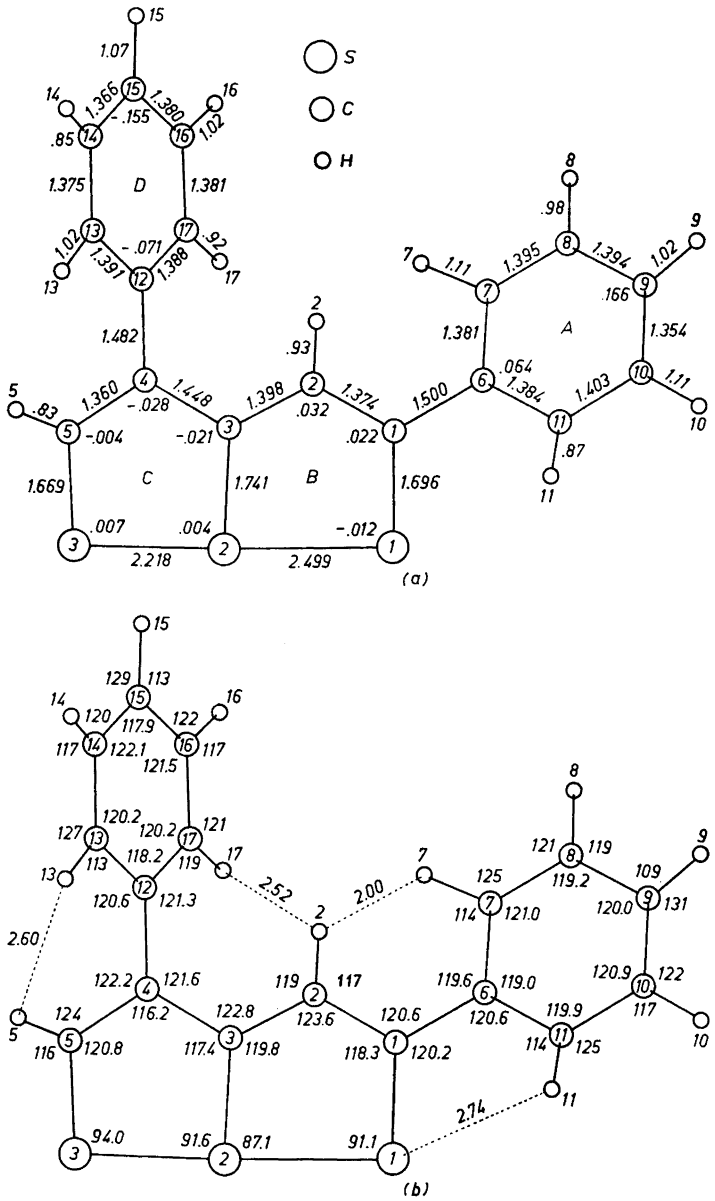


Fig. 1. (a) Bond lengths (Å) in the 2,4-diphenyl-thiothiophthene molecule, and atomic distances (Å) from the least squares plane of the thiothiophthene system. (b) Bond angles (°) and intramolecular non-bonding distances (Å).

Table 3. Bond lengths D(ij) in 2,4-diphenyl-thiothiopthene. The standard deviations given in parentheses refer to the last digits of respective values.

i	j	D(ij)	i	j	D(ij)
S(1)	S(2)	2.499(3) Å	C(13)	C(14)	1.375(9) Å
S(1)	C(1)	1.696(5)	C(14)	C(15)	1.366(9)
S(2)	S(3)	2.218(3)	C(15)	C(16)	1.380(9)
S(2)	C(3)	1.741(5)	C(16)	C(17)	1.381(9)
S(3)	C(5)	1.669(7)	C(17)	C(12)	1.388(7)
C(1)	C(2)	1.374(11)	C(2)	H(2)	0.93(5)
C(1)	C(6)	1.500(9)	C(5)	H(5)	0.83(8)
C(2)	C(3)	1.398(9)	C(7)	H(7)	1.11(6)
C(3)	C(4)	1.448(11)	C(8)	H(8)	0.98(5)
C(4)	C(5)	1.360(9)	C(9)	H(9)	1.02(4)
C(4)	C(12)	1.482(7)	C(10)	H(10)	1.11(6)
C(6)	C(7)	1.381(8)	C(11)	H(11)	0.87(6)
C(7)	C(8)	1.395(10)	C(13)	H(13)	1.02(5)
C(8)	C(9)	1.394(15)	C(14)	H(14)	0.85(5)
C(9)	C(10)	1.354(11)	C(15)	H(15)	1.07(5)
C(10)	C(11)	1.403(11)	C(16)	H(16)	1.02(4)
C(11)	C(6)	1.384(12)	C(17)	H(17)	0.92(4)
C(12)	C(13)	1.391(8)			

Table 4. Bond angles $\angle$(ijk) in 2,4-diphenyl-thiothiopthene. The standard deviations given in parentheses refer to the last digits of respective values. For bond angles including a hydrogen atom the standard deviation is estimated to be 4°.

i	j	k	$\angle$(ijk)	i	j	k	$\angle$(ijk)
C(1)	S(1)	S(2)	91.1(3)	C(13)	C(14)	C(15)	122.1(6)
S(1)	S(2)	C(3)	87.1(3)	C(14)	C(15)	C(16)	117.9(6)
S(1)	S(2)	S(3)	178.1(1)	C(15)	C(16)	C(17)	121.5(6)
C(3)	S(2)	S(3)	91.6(3)	C(16)	C(17)	C(12)	120.2(5)
S(2)	S(3)	C(5)	94.0(3)	H(2)	C(2)	C(1)	117
S(1)	C(1)	C(2)	118.3(5)	H(2)	C(2)	C(3)	119
S(1)	C(1)	C(6)	120.2(5)	H(5)	C(5)	S(3)	116
C(2)	C(1)	C(6)	120.6(5)	H(5)	C(5)	C(4)	124
C(1)	C(2)	C(3)	123.6(5)	H(7)	C(7)	C(6)	114
C(2)	C(3)	S(2)	119.8(6)	H(7)	C(7)	C(8)	125
C(2)	C(3)	C(4)	122.8(5)	H(8)	C(8)	C(7)	121
C(4)	C(3)	S(2)	117.4(4)	H(8)	C(8)	C(9)	119
C(3)	C(4)	C(5)	116.2(6)	H(9)	C(9)	C(8)	109
C(3)	C(4)	C(12)	121.6(5)	H(9)	C(9)	C(10)	131
C(5)	C(4)	C(12)	122.2(7)	H(10)	C(10)	C(9)	122
C(4)	C(5)	S(3)	120.8(7)	H(10)	C(10)	C(11)	117
C(1)	C(6)	C(7)	119.6(7)	H(11)	C(11)	C(10)	125
C(1)	C(6)	C(11)	120.6(5)	H(11)	C(11)	C(6)	114
C(7)	C(6)	C(11)	119.0(6)	H(13)	C(13)	C(12)	113
C(6)	C(7)	C(8)	121.0(8)	H(13)	C(13)	C(14)	127
C(7)	C(8)	C(9)	119.2(6)	H(14)	C(14)	C(13)	117
C(8)	C(9)	C(10)	120.0(8)	H(14)	C(14)	C(15)	120
C(9)	C(10)	C(11)	120.9(9)	H(15)	C(15)	C(14)	129
C(10)	C(11)	C(6)	119.9(6)	H(15)	C(15)	C(16)	113
C(4)	C(12)	C(13)	120.6(4)	H(16)	C(16)	C(15)	122
C(4)	C(12)	C(17)	121.3(5)	H(16)	C(16)	C(17)	117
C(13)	C(12)	C(17)	118.2(5)	H(17)	C(17)	C(16)	121
C(12)	C(13)	C(14)	120.2(5)	H(17)	C(17)	C(12)	119

the thiothiophthene system is almost planar and that the approximately linear sequences C(1)—C(6)—C(9) and C(4)—C(12)—C(15) point slightly up and down, respectively, from the plane.

The equation for the least squares plane through the carbon atoms of phenyl group *A* is

$$-0.69965\ X + 0.78433\ Y - 0.53757\ Z = -2.10106$$

and the deviations in Å units from this plane are, C(6) 0.012, C(7) −0.010, C(8) 0.005, C(9) −0.003, C(10) 0.005, and C(11) −0.010 Å.

Similarly the plane equation for phenyl group *D* is

$$0.04349\ X + 0.81135\ Y + 0.04916\ Z = -1.45007$$

with deviations, C(12) 0.002, C(13) 0.001, C(14) −0.002, C(15) 0.001, C(16) 0.001, and C(17) −0.003 Å.

The twist angle about C(1)—C(6) of phenyl group *A* was taken as the angle between the normal to the plane through S(1), C(1), C(2), and C(6) and the normal to the plane through C(1), C(6), C(7), and C(11). This angle is 24.7°.

Similarly, the twist angle of phenyl group *D* about C(4)—C(12) was found to be 51.6°. It should be noted that the phenyl groups are twisted in opposite directions relative to the plane of the thiothiophthene system.

The bond lengths in the 2,4-diphenyl-thiothiophthene molecule as listed in Table 3 and shown in Fig. 1a, show that the C—C and C—S bonds in the rings *B* and *C* are shorter than corresponding single bonds. The C—C bonds are, C(1)—C(2)=1.374±0.011 Å, C(2)—C(3)=1.398±0.009 Å, C(3)—C(4) =1.448±0.011 Å, and C(4)—C(5)=1.360±0.009 Å, as compared with the accepted value 1.397 Å for the aromatic C—C bond in benzene; and the C—S bonds are, C(1)—S(1)=1.696±0.005 Å, C(3)—S(2)=1.741±0.005 Å, and C(5)—S(3)=1.669±0.007 Å, as compared with the values 1.82 and 1.61 Å, respectively for the length of a C—S single and double bond.[21,22] Thus the thiothiophthene system is stabilized through delocalized π-bonding.

The two sulphur-sulphur bonds are found to be different, *i.e.*, S(1)—S(2)=2.499±0.003 Å and S(2)—S(3)=2.218±0.003 Å, as compared with the value 2.10 Å for the length of a sulphur-sulphur single bond in a *cis*-planar disulphide group.[6]

The bonds between the phenyl groups and the thiothiophthene system are, C(1)—C(6)=1.500±0.009 Å and C(4)—C(12)=1.482±0.007 Å, as compared with the value 1.4943±0.002 Å found for the central bond in diphenyl through an X-ray crystallographic investigation of the compound.[23]

The average values for C—C bond length in the benzene rings *A* and *D* are found to be 1.385 and 1.381 Å, respectively, and thus slightly shorter than the aromatic C—C bond length, 1.397 Å. This result may, however, judging from the temperature parameters in Table 2, be due to the rigid body motion of the molecule.

From the intramolecular non-bonding atomic distances given in Fig. 1a, the *ortho*-hydrogens H(7) and H(11) of ring *A* are rather close to H(2) and S(1), respectively. The distances found are, H(7)···H(2)=2.00±0.10 Å and H(11)···S(1)=2.74±0.06 Å as compared with the corresponding van der

Waals distances of 2.40 Å for H···H and 3.05 Å for H···S.[10] The distances between the *ortho*-hydrogens in ring *D* and the hydrogens of the thiothiophthene system, H(17)···H(2)=2.52±0.10 Å and H(13)···H(5)=2.60±0.10 Å, are found to be longer than the corresponding van der Waals distance.

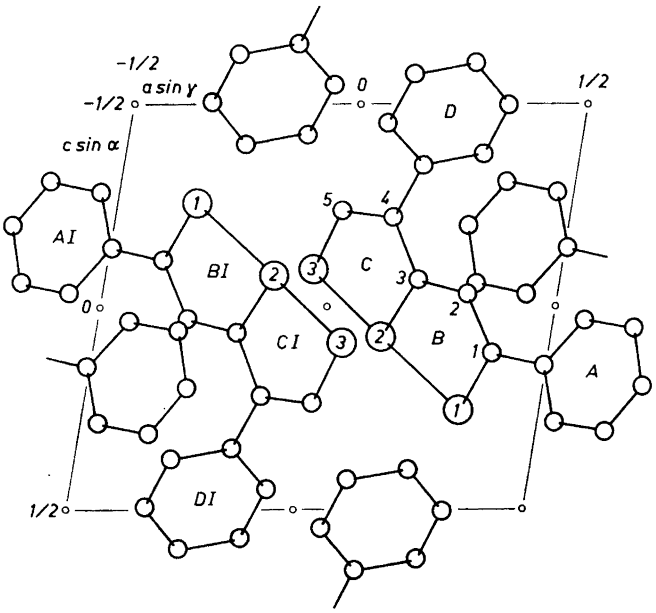


Fig. 2. The arrangement of 2,4-diphenyl-thiothiophthene molecules in the crystal as seen along the *b*-axis.

Molecular arrangement and intermolecular sulphur environment. The arrangement of molecules in the unit cell as viewed along the *b* axis is shown in Fig. 2. The reference molecule is marked *A—B—C—D* and the center of symmetry (origin) related molecule is marked *AI—BI—CI—DI*. The plane of the thiothiophthene system in the former is, due to the symmetry operation, parallel with the plane of the thiothiophthene system in the latter, and the distance between these planes is 3.65 Å. This distance is somewhat longer than two times the half-thickness of an aromatic molecule, 3.40 Å, which might be anticipated when atoms larger than carbon, in this case sulphur, are built into the aromatic system.

One may note the way in which the sulphur atoms S(2) and S(3) of ring *CI* approach the atoms of the rings *C* and *B*, respectively. The relevant intermolecular atomic distances are listed in Table 5 below. It is seen that $S(2)_{CI}$ and $S(3)_{CI}$ are located almost exactly above the centers of the respective rings.

As regards the intermolecular environment of S(1) there is no atom closer than corresponding van der Waals distance.

Table 5. Intermolecular atomic distances D(ij) which show the way in which S(2) and S(3) of the ring CI approach the atoms of the rings C and B, respectively.

i	j	D(ij)	i	j	D(ij)
$S(2)_{CI}$	$S(2)_C$	4.05 Å	$S(3)_{CI}$	$S(1)_B$	4.14 Å
$S(2)_{CI}$	$S(3)_C$	4.02	$S(3)_{CI}$	$S(2)_B$	4.02
$S(2)_{CI}$	$C(3)_C$	3.87	$S(3)_{CI}$	$C(1)_B$	3.90
$S(2)_{CI}$	$C(4)_C$	3.82	$S(3)_{CI}$	$C(2)_B$	3.75
$S(2)_{CI}$	$C(5)_C$	3.84	$S(3)_{IC}$	$C(3)_B$	3.81

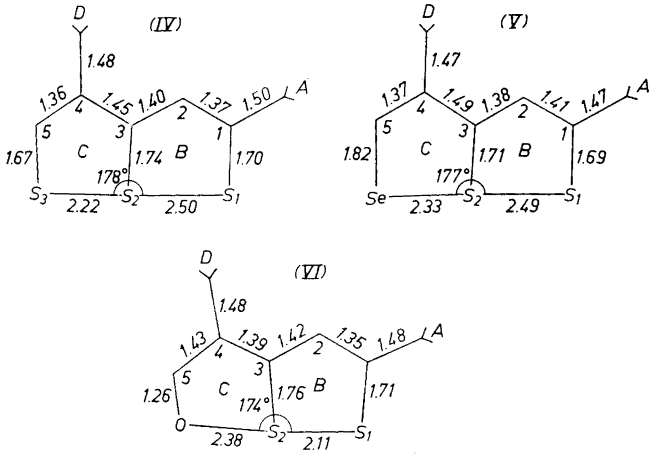


Fig. 3. Comparison of bond lengths in 2,4-diphenyl-thiothiophthene (IV) with bond lengths in the analogous compounds (V) and (VI). The bond lengths are given in Å units.

Comparison with related molecules. A comparison of the bond lengths in 2,4-diphenyl-thiothiophthene (IV) with the bond lengths in the selenium isologue (V) and the oxygen isologue (VI) is given in Fig. 3. Compound (V) has been studied by van der Hende and Klingsberg,[8] and compound (VI)[24] has been studied by the authors.* The standard deviations found for the bond lengths in (VI) are compatible with those found for the bond lengths in (IV); they are, ± 0.003 Å, ± 0.006, ± 0.007, ± 0.010 and ± 0.009-± 0.012 Å for S—S, S—O, S—C, C—O, and C—C, respectively. The standard deviations for the bond lengths in (V) are reported to be ± 0.003 Å for Se—S and S—S, ± 0.01 Å for Se—C and S—C and ± 0.02 Å for C—C.

From the present study, the atoms of the rings B and C in (IV) are almost in the same plane, and the same has been found for the atoms of B and C

* The structure study of (VI) by the authors is based on photographic data. Professor S. C. Nyholm, University of Toronto, is presently carrying out a refinement of the structure on diffractometer data. A joint paper, presenting the results from both studies, will eventually be submitted to *Acta Chem. Scand.*

Table 6. Observed and calculated $hk0-hk8$, $h0l$ and $h1l$ structure factors for 2,4-diphenylthiothiophthene. The values given are fifty times the absolute values. Structure factors marked with asterisks have been corrected for secondary extinction.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	0	0	939	983	-5	4	0	942	899	-2	0	1	974	1012	6	6	-1	250	229
2	0	0	527	-482	-5	6	0	171	-116	-3	0	1	300	314	7	2	-1	222	-174
3	0	0	1676	1836	-5	8	0	286	-290	-1	1	1	2287*	2308	7	3	-1	523	-472
4	0	0	710	-665	-6	1	0	1365	1390	-1	2	1	1755	1942	7	4	-1	759	-748
5	0	0	1622	-1811	-6	3	0	321	-291	-1	3	1	912	-977	7	5	-1	271	286
6	0	0	936	954	-6	4	0	393	-320	-1	4	1	1681	-1792	1	-2	1	201	-188
7	0	0	434	349	-6	5	0	485	-435	-1	5	1	745	-658	1	-3	1	500	414
8	0	0	281	-260	-6	8	0	253	-270	-1	6	1	278	247	1	-4	1	617	-570
9	0	0	174	144	-7	1	0	1077	1107	-1	7	1	346	333	1	-5	1	765	-735
11	0	0	341	-364	-7	2	0	462	455	-1	8	1	356	311	1	-6	1	575	518
0	1	0	1083	1088	-7	3	0	157	157	-2	1	1	2716*	-2753	1	-8	1	190	184
0	2	0	2723*	-2725	-7	5	0	705	-725	-2	2	1	606	-605	2	-1	1	1477	1534
0	3	0	1549	-1693	-7	6	0	334	-269	-2	3	1	803	892	2	-2	1	7606*	-7395
0	4	0	1623	-1608	-8	1	0	205	191	-2	4	1	1431	-1467	2	-3	1	1124	-1090
0	5	0	192	141	-8	2	0	477	-406	-2	5	1	416	-352	2	-4	1	609	579
0	6	0	1002	1032	-8	3	0	168	124	-2	6	1	195	175	2	-6	1	1056	1069
0	7	0	656	-683	-9	1	0	343	319	-2	7	1	126	98	2	-7	1	493	499
0	8	0	269	-264	-9	2	0	443	-434	-2	8	1	337	332	2	-8	1	365	-384
1	1	0	2224*	2214	-9	3	0	619	-621	-2	9	1	154	169	2	-9	1	234	207
1	2	0	1033	1080	-9	5	0	170	-201	-3	1	1	2200*	-2249	3	-1	1	78	-59
1	3	0	225	208	-9	7	0	209	261	-3	2	1	1041	-1140	3	-2	1	827	872
1	4	0	1172	-1198	-12	1	0	162	-144	-3	3	1	399	376	3	-3	1	737	-657
1	5	0	417	405						-3	5	1	275	252	3	-4	1	154	163
1	6	0	218	-168	1	0	1	385	368	-3	6	1	479	479	3	-5	1	337	-331
1	8	0	219	-217	2	0	1	768	781	-4	1	1	804	785	3	-6	1	427	346
2	1	0	497	488	3	0	1	823	-778	-4	2	1	454	439	3	-7	1	450	422
2	2	0	2883*	2952	4	0	1	1148	-1199	-4	4	1	467	-443	4	-1	1	402	393
2	3	0	2030	2198	5	0	1	245	206	-4	6	1	812	908	4	-2	1	617	-599
2	4	0	741	-725	6	0	1	244	-210	-4	7	1	203	191	4	-3	1	2215	2199
2	5	0	741	-710	8	0	1	161	156	-4	9	1	165	-208	4	-4	1	1991	1978
2	6	0	525	502	9	0	1	216	-178	-5	1	1	538	482	4	-5	1	156	-115
2	7	0	176	-160	10	0	1	281	-279	-5	2	1	595	-555	4	-7	1	129	-118
3	1	0	203	-182	11	0	1	223	-196	-5	3	1	784	788	4	-8	1	309	-318
3	2	0	293	273	0	1	1	3015*	-2907	-5	5	1	221	199	5	-1	1	911	915
3	3	0	443	430	0	2	1	880	914	-5	6	1	216	163	5	-2	1	838	-790
3	4	0	477	-431	0	3	1	2093	2263	-5	8	1	231	-226	5	-3	1	253	209
3	5	0	453	448	0	4	1	348	-277	-6	2	1	355	-360	5	-4	1	1222	1077
3	6	0	381	369	0	5	1	530	-536	-6	4	1	584	553	5	-6	1	225	134
4	1	0	2897*	-2958	0	6	1	406	-381	-6	5	1	127	136	5	-7	1	142	-153
4	2	0	465	463	0	7	1	551	-589	-6	7	1	336	-378	5	-8	1	253	-286
4	3	0	519	529	1	1	1	2752*	-2712	-6	8	1	172	-174	5	-9	1	138	-108
4	4	0	508	-497	1	2	1	1311	1413	-7	3	1	396	415	6	-1	1	1953	2052
4	5	0	521	527	1	3	1	2028*	2118	-7	5	1	282	-290	6	-2	1	761	697
4	7	0	186	-184	1	4	1	333	265	-8	5	1	200	-199	6	-4	1	499	-447
5	1	0	754	-791	1	6	1	793	-848	0	2	-1	2155*	2201	6	-5	1	1240	-1207
5	2	0	510	516	1	7	1	550	-597	0	3	-1	1377	1348	6	-6	1	561	-541
5	3	0	403	376	1	8	1	171	177	0	4	-1	1879	-1856	6	-7	1	398	403
5	4	0	295	-258	2	1	1	472	-424	0	5	-1	1887	-1985	7	-1	1	485	484
6	1	0	824	-805	2	2	1	972	1025	0	7	-1	296	287	7	-2	1	475	475
6	3	0	290	-280	2	4	1	493	-474	0	9	-1	139	138	7	-3	1	450	-385
6	4	0	275	-259	2	5	1	1112	1137	1	1	-1	380	360	7	-4	1	162	165
6	5	0	211	225	2	6	1	198	193	1	2	-1	1666	-1876	7	-5	1	610	-608
7	1	0	543	-563	2	7	1	225	-269	1	3	-1	622	595	7	-6	1	267	-230
7	3	0	293	-275	3	1	1	1773	-1870	1	4	-1	133	95	8	-1	1	477	469
7	5	0	230	260	3	2	1	180	-125	1	5	-1	481	-423	8	-2	1	960	-930
8	2	0	330	328	3	3	1	165	-142	1	6	-1	194	-160	8	-3	1	945	-905
8	4	0	213	-203	3	4	1	932	939	1	9	-1	178	144	8	-5	1	403	398
9	1	0	172	198	3	5	1	611	664	2	1	-1	965	-995	8	-6	1	275	297
9	2	0	429	476	3	6	1	309	307	2	2	-1	636	-631	8	-7	1	150	179
9	3	0	244	-269	3	7	1	230	-241	2	3	-1	3008*	2979	9	-1	1	129	117
-1	1	0	211	135	4	1	1	1893	-2025	2	4	-1	472	392	9	-2	1	275	-233
-1	2	0	2765*	-2762	4	2	1	497	485	2	5	-1	471	-440	9	-3	1	526	-482
-1	3	0	779	786	4	3	1	334	325	2	6	-1	440	385	9	-7	1	256	258
-1	4	0	250	-236	4	4	1	769	-759	2	7	-1	582	-607	9	-8	1	123	124
-1	5	0	546	-537	4	5	1	598	558	2	9	-1	141	190	10	-1	1	194	-178
-1	8	0	154	131	4	7	1	124	-107	3	1	-1	87	-83	10	-4	1	337	321
-1	9	0	322	307	5	2	1	108	84	3	2	-1	1722	1830	10	-5	1	119	85
-2	1	0	5578*	-5210	5	3	1	1120	-1199	3	3	-1	1194	1277	11	-3	1	175	-173
-2	2	0	4330*	-3961	5	4	1	524	-528	3	4	-1	349	-325	11	-4	1	274	274
-2	3	0	951	914	5	5	1	268	254	3	5	-1	154	-139	11	-5	1	347	318
-2	4	0	545	561	5	7	1	209	187	3	6	-1	271	-214	12	-1	1	111	-100
-2	5	0	226	-158	6	1	1	460	-465	3	7	-1	204	-199	12	-3	1	140	132
-2	6	0	371	333	6	2	1	256	231	4	1	-1	2137	-2072					
-2	8	0	157	102	6	3	1	429	-451	4	2	-1	1213	-1146	1	0	2	650	629
-2	9	0	303	300	6	5	1	248	263	4	3	-1	560	517	2	0	2	564	-521
-3	1	0	1283	-1301	6	6	1	92	-80	4	5	-1	374	342	3	0	2	2054	-2120
-3	2	0	2075	-2246	7	2	1	561	560	4	6	-1	198	221	4	0	2	798	-803
-3	3	0	332	-324	7	5	1	142	-122	4	7	-1	255	-246	5	0	2	281	265
-3	4	0	411	-398	8	1	1	458	502	5	1	-1	1403	-1423	0	0	2	695	646
-3	5	0	622	614	8	2	1	236	208	5	2	-1	1059	-1068	0	1	2	47	99
-3	6	0	766	749	8	3	1	325	-323	5	3	-1	462	438	0	2	2	1948	1935
-3	7	0	212	187	8	4	1	201	-189	5	4	-1	328	305	0	3	2	480	441
-4	1	0	289	-259	8	5	1	172	124	5	5	-1	733	709	0	4	2	332	-318
-4	3	0	2316	2359	9	2	1	211	200	5	6	-1	235	200	0	6	2	426	-426
-4	4	0	1261	1176	9	3	1	174	-197	5	7	-1	266	-248	0	7	2	258	-280
-4	5	0	759	-689	9	4	1	242	196	6	1	-1	401	-378	1	1	2	1777	1873
-4	6	0	409	360	10	1	1	112	110	6	2	-1	565	542	1	2	2	1847	1843
-5	1	0	316	-296	10	2	1	208	206	6	3	-1	257	-193	1	3	2	182	141
-5	2	0	356	-354	11	1	1	180	194	6	4	-1	341	-305	1	4	2	699	-671
-5	3	0	1652	1803	-1	0	1	400	308	6	5	-1	212	190	1	5	2	396	-380

Table 6. Continued.

H	K	L	FO	FC
1	6	2	127	- 82
1	7	2	82	62
2	1	2	1740	-1858
2	2	2	364	320
2	3	2	186	200
2	5	2	1043	1059
2	7	2	133	- 129
3	1	2	1123	-1137
3	2	2	723	716
3	4	2	958	920
3	5	2	298	317
3	6	2	268	- 223
4	1	2	241	236
4	2	2	538	504
4	3	2	959	- 931
4	4	2	535	- 552
4	5	2	148	187
5	1	2	345	- 315
5	2	2	850	- 855
5	3	2	984	- 816
5	4	2	237	- 220
5	5	2	315	326
6	1	2	234	189
6	2	2	931	935
6	3	2	428	- 464
- 1	0	2	1652	1867
- 2	0	2	1124	1081
- 3	0	2	1280	1255
- 4	0	2	164	163
- 5	0	2	106	- 58
- 6	0	2	559	- 501
- 7	0	2	272	221
- 8	0	2	277	264
- 9	0	2	319	315
-11	0	2	146	- 119
-12	0	2	328	- 309
- 1	1	2	1152	-1096
- 1	2	2	354	- 280
- 1	3	2	915	- 825
- 1	4	2	348	- 346
- 1	5	2	366	330
- 1	6	2	174	191
- 1	7	2	152	- 170
- 1	8	2	80	58
- 2	1	2	315	260
- 2	2	2	278	- 282
- 2	3	2	272	- 239
- 2	4	2	1594	-1684
- 2	5	2	157	- 119
- 2	6	2	130	131
- 2	7	2	231	264
- 2	8	2	137	146
- 3	1	2	1121	-1228
- 3	3	2	311	- 281
- 3	6	2	231	241
- 3	7	2	195	- 223
- 4	1	2	996	-1017
- 4	2	2	1583	-1609
- 4	3	2	212	166
- 4	4	2	434	420
- 4	5	2	738	736
- 4	6	2	320	300
- 4	7	2	119	- 108
- 4	8	2	78	- 104
- 5	1	2	812	- 818
- 5	2	2	557	- 495
- 5	3	2	478	417
- 5	6	2	141	142
- 6	1	2	160	- 167
- 6	2	2	566	493
- 6	3	2	571	584
- 6	4	2	124	120
- 6	6	2	171	- 205
- 6	7	2	103	- 95
- 7	1	2	256	185
- 7	2	2	401	- 322
- 7	3	2	728	722
- 7	4	2	446	431
- 7	5	2	123	- 122
- 7	6	2	175	179
- 7	7	2	109	- 123
- 7	8	2	101	- 117
- 8	1	2	712	733
- 8	3	2	91	- 89
- 8	4	2	92	71
- 8	6	2	104	- 116
- 9	1	2	437	397
- 9	3	2	175	200
- 9	5	2	370	- 436
- 9	6	2	199	- 183
-10	5	2	167	- 196
-10	6	2	128	- 127
-11	1	2	325	351
-11	2	2	355	- 380
-11	3	2	293	- 284
-12	1	2	141	- 122
-12	3	2	162	- 189
0	1	- 2	2378*	2573
0	2	- 2	1846*	-1853
0	3	- 2	85	- 91
0	4	- 2	296	313
0	5	- 2	1328	-1287
0	6	- 2	219	- 183
0	7	- 2	422	447
0	8	- 2	315	- 334
0	9	- 2	174	229
1	1	- 2	2833*	2696
1	2	- 2	409	- 387
1	3	- 2	425	- 404
1	4	- 2	1164	-1234
1	5	- 2	1000	-1060
1	6	- 2	328	341
1	7	- 2	116	77
1	8	- 2	282	244
1	9	- 2	187	192
2	1	- 2	371	362
2	2	- 2	1254	-1123
2	3	- 2	1342	1230
2	5	- 2	272	- 250
2	6	- 2	293	307
2	7	- 2	174	- 166
2	8	- 2	90	- 113
2	9	- 2	112	115
3	1	- 2	272	- 232
3	2	- 2	936	892
3	3	- 2	755	781
3	4	- 2	657	579
3	5	- 2	319	- 335
3	6	- 2	222	- 242
3	7	- 2	535	- 524
3	8	- 2	160	- 157
4	1	- 2	979	946
4	3	- 2	1106	1094
4	4	- 2	812	- 772
4	5	- 2	242	- 222
4	6	- 2	130	99
4	7	- 2	133	- 143
4	8	- 2	103	- 88
5	1	- 2	859	- 822
5	3	- 2	467	413
5	5	- 2	475	454
5	6	- 2	568	589
5	7	- 2	179	- 206
6	1	- 2	1427	-1433
6	3	- 2	662	653
6	4	- 2	299	293
6	5	- 2	540	531
6	6	- 2	92	104
6	7	- 2	237	- 244
7	1	- 2	748	- 690
7	3	- 2	141	- 102
7	4	- 2	765	- 773
7	5	- 2	165	194
7	6	- 2	83	75
8	2	- 2	315	- 319
8	3	- 2	846	- 869
8	4	- 2	419	- 462
8	5	- 2	236	244
8	6	- 2	203	185
9	1	- 2	292	- 279
9	2	- 2	458	443
9	4	- 2	285	- 317
10	1	- 2	375	385
10	2	- 2	752	772
10	4	- 2	271	- 280
11	2	- 2	253	284
12	1	- 2	173	- 196
1	- 1	2	2064*	-2416
1	- 2	2	618	- 594
1	- 3	2	1648	-1601
1	- 4	2	503	465
1	- 5	2	340	354
1	- 6	2	622	610
1	- 7	2	141	112
1	- 8	2	90	80
1	- 9	2	189	- 207
2	- 1	2	2692*	2511
2	- 2	2	4462*	-4052
2	- 3	2	1112	-1010
2	- 4	2	214	- 154
2	- 6	2	894	972
2	- 7	2	727	740
2	- 8	2	270	- 294
3	- 1	2	412	- 452
3	- 2	2	429	- 364
3	- 3	2	128	- 116
3	- 4	2	1625	1623
3	- 5	2	211	160
3	- 6	2	149	- 120
3	- 8	2	267	- 280
4	- 1	2	121	111
4	- 2	2	288	- 272
4	- 3	2	61	- 72
4	- 4	2	2528	2707
4	- 5	2	715	651
4	- 6	2	429	- 439
4	- 7	2	116	- 138
4	- 8	2	404	- 468
4	- 9	2	202	- 185
4	-10	2	129	139
5	- 1	2	1417	1468
5	- 2	2	642	627
5	- 3	2	225	216
5	- 4	2	93	- 82
5	- 6	2	147	- 109
5	- 7	2	116	- 98
5	- 8	2	97	- 117
5	- 9	2	84	- 85
6	- 2	2	335	288
6	- 4	2	712	698
6	- 5	2	806	- 798
6	- 6	2	1102	-1191
6	- 7	2	175	- 145
6	- 9	2	199	231
7	- 3	2	321	- 308
7	- 4	2	339	318
7	- 5	2	364	- 352
7	- 6	2	708	- 697
7	- 8	2	210	200
7	- 9	2	122	147
8	- 3	2	649	- 666
8	- 4	2	568	- 631
8	- 5	2	405	- 401
8	- 6	2	163	142
8	- 7	2	574	706
8	- 8	2	345	387
8	- 9	2	84	- 122
9	- 4	2	291	- 289
9	- 5	2	122	152
9	- 7	2	333	367
9	- 8	2	151	143
10	- 5	2	336	347
10	- 6	2	172	215
10	- 8	2	128	- 172
1	0	3	503	- 439
2	0	3	2786*	-2836
3	0	3	1487	-1529
4	0	3	197	116
5	0	3	413	- 383
0	0	3	756	735
0	1	3	470	418
0	2	3	2395*	2449
0	4	3	329	- 285
0	5	3	274	- 219
0	6	3	253	- 213
0	7	3	119	- 120
1	1	3	937	851
1	2	3	274	235
1	3	3	290	- 255
1	5	3	207	244
1	6	3	137	- 102
1	7	3	62	48
2	1	3	1472	-1575
2	2	3	627	545
2	3	3	497	486
2	4	3	637	598
2	5	3	350	310
2	6	3	135	- 102
2	7	3	95	- 70
3	1	3	703	- 673
3	2	3	435	417
3	3	3	359	337
3	4	3	279	200
3	5	3	77	50
3	6	3	135	- 117
4	1	3	413	- 385
4	2	3	538	- 467
4	3	3	1320	-1325
4	4	3	328	261
4	5	3	258	231
5	1	3	75	- 137
5	2	3	1021	-1033
5	4	3	161	167
6	2	3	565	508
- 1	0	3	351	- 386
- 2	0	3	1577	1739
- 3	0	3	1871	1901
- 4	0	3	570	- 530
- 5	0	3	801	722
- 6	0	3	296	261
- 7	0	3	466	- 410
- 9	0	3	1000	1033
-11	0	3	259	257
- 1	1	3	118	- 79
- 1	2	3	167	161
- 1	3	3	475	459
- 1	4	3	134	- 126
- 1	5	3	179	162
- 1	6	3	173	- 171
- 1	7	3	71	- 114
- 2	1	3	673	- 645
- 2	2	3	470	- 473
- 2	3	3	664	- 583
- 2	4	3	518	- 487
- 2	5	3	145	127
- 3	1	3	197	147
- 3	2	3	519	477
- 3	4	3	652	- 658
- 3	5	3	301	- 270
- 3	6	3	79	57
- 3	7	3	62	- 61
- 3	8	3	105	129
- 4	1	3	355	- 347
- 4	2	3	391	- 310
- 4	3	3	258	206
- 4	6	3	89	64
- 4	7	3	138	114
- 5	1	3	1691	-1772
- 5	2	3	1106	-1181
- 5	4	3	233	- 206
- 5	5	3	334	340
- 5	6	3	334	330
- 6	1	3	62	65
- 6	2	3	451	- 389
- 6	3	3	122	122
- 7	1	3	245	- 245
- 7	2	3	226	230
- 7	3	3	1053	1083
- 7	4	3	461	444
- 7	5	3	194	- 173
- 7	6	3	136	111
- 7	7	3	114	- 112
- 8	1	3	141	107
- 8	3	3	425	415
- 8	4	3	267	234
- 8	6	3	208	- 176
- 9	1	3	800	797
- 9	2	3	216	210
- 9	4	3	324	- 366
- 9	5	3	475	- 479
- 9	6	3	102	114
-10	1	3	566	606
-10	4	3	163	- 179
-10	5	3	230	- 211
-10	6	3	52	- 77
-11	2	3	605	- 637
-11	3	3	245	- 256
-12	2	3	200	- 211
-12	3	3	311	- 271
-12	4	3	78	- 94
0	2	- 3	2558*	-2664
0	3	- 3	756	- 811
0	4	- 3	1015	1029
0	5	- 3	452	- 436
0	6	- 3	496	480
0	7	- 3	309	257
0	8	- 3	355	- 360
1	1	- 3	357	346
1	2	- 3	1186	-1260
1	3	- 3	344	333
1	4	- 3	849	- 879
1	5	- 3	327	- 334
1	8	- 3	244	240
1	9	- 3	290	274
2	1	- 3	1477	1687
2	2	- 3	436	394
2	3	- 3	278	- 311
2	4	- 3	607	- 650
2	5	- 3	1272	-1372
2	6	- 3	334	323
2	7	- 3	391	372
2	8	- 3	197	144
2	9	- 3	184	169
3	1	- 3	325	333
3	2	- 3	425	- 353
3	3	- 3	593	579
3	4	- 3	88	- 53
3	5	- 3	560	- 517
3	7	- 3	453	- 450
3	8	- 3	288	- 277
3	9	- 3	187	200
4	1	- 3	152	- 101
4	2	- 3	201	127
4	3	- 3	1662	1830
4	4	- 3	250	192
4	5	- 3	312	- 316
4	6	- 3	603	- 352
4	7	- 3	631	- 635
4	8	- 3	215	- 177
5	1	- 3	248	234
5	2	- 3	490	402
5	3	- 3	938	972
5	4	- 3	134	- 100

Table 6. Continued.

H	K	L	FO	FC
5	6	3	654	705
5	7	3	287	- 273
6	1	3	1097	-1079
6	2	3	431	- 375
6	3	3	314	253
6	5	3	851	916
6	6	3	489	542
6	7	3	242	- 226
7	1	3	1349	-1336
7	2	3	477	- 417
7	3	3	528	512
7	4	3	311	- 285
7	5	3	100	115
7	6	3	241	262
7	7	3	143	- 136
8	1	3	112	- 98
8	3	3	336	- 309
8	4	3	816	- 852
8	6	3	295	282
9	1	3	668	- 707
9	4	3	473	- 463
9	5	3	183	138
10	2	3	360	322
10	3	3	162	165
10	4	3	129	- 144
11	1	3	104	113
11	2	3	389	396
12	1	3	132	- 98
12	2	3	51	110
1	1	3	272	192
1	2	3	299	- 301
1	3	3	657	- 674
1	4	3	576	520
1	5	3	111	106
1	6	3	238	210
1	7	3	457	436
1	10	3	137	- 142
2	1	3	578	- 600
2	2	3	761	810
2	3	3	613	611
2	4	3	915	938
2	5	3	509	- 483
2	6	3	362	- 332
2	7	3	498	505
2	8	3	253	238
2	9	3	158	- 185
3	1	3	148	- 124
3	2	3	136	- 122
3	3	3	1114	1107
3	4	3	445	- 353
3	5	3	226	141
3	6	3	147	139
3	7	3	166	125
3	8	3	285	- 274
3	10	3	186	- 179
4	1	3	1969	2204
4	2	3	1221	1259
4	3	3	1802	-1915
4	4	3	1043	1094
4	5	3	115	48
4	7	3	272	226
4	8	3	341	- 359
4	9	3	408	- 401
5	1	3	1237	898
5	2	3	1288	1378
5	3	3	519	524
5	4	3	454	- 444
5	5	3	100	96
5	6	3	477	- 480
6	3	3	762	- 745
6	4	3	156	- 118
6	6	3	516	- 513
6	7	3	131	- 133
7	2	3	487	- 448
7	3	3	1297	-1351
7	4	3	565	- 562
7	5	3	276	220
7	6	3	252	225
7	8	3	133	- 147
7	10	3	101	89
8	3	3	451	- 446
8	4	3	290	255
8	5	3	351	307
8	6	3	242	- 218
8	8	3	293	311
8	9	3	81	90
9	4	3	327	329
9	5	3	395	386
9	6	3	142	- 165
9	8	3	201	217
9	9	3	95	101
10	5	3	210	157
10	6	3	135	131
1	0	4	721	- 741
3	0	4	397	364
2	0	4	1495	-1458
0	0	4	207	187
0	1	4	219	- 200
0	2	4	787	698
0	3	4	119	- 102
0	4	4	198	181
1	1	4	872	- 916
2	1	4	366	292
3	1	4	589	475
2	2	4	191	190
3	2	4	102	- 89
2	3	4	282	- 253
3	3	4	404	- 421
1	4	4	347	312
2	4	4	189	124
3	4	4	165	- 155
1	5	4	277	283
2	5	4	158	152
1	6	4	118	- 121
- 1	0	4	860	- 855
- 2	0	4	926	920
- 3	0	4	2025	2283
- 4	0	4	868	814
- 5	0	4	591	- 271
- 6	0	4	427	428
- 7	0	4	162	- 152
- 8	0	4	315	- 300
- 9	0	4	1104	1181
-10	0	4	377	402
-11	0	4	155	155
-12	0	4	168	171
- 1	1	4	738	723
- 2	1	4	156	- 156
- 4	1	4	225	- 223
- 5	1	4	1880	-1896
- 6	1	4	886	- 854
- 7	1	4	292	- 278
- 8	1	4	176	- 144
- 9	1	4	332	295
-10	1	4	507	534
-11	1	4	173	- 186
-12	1	4	105	- 109
- 1	2	4	1214	1200
- 2	2	4	645	576
- 3	2	4	233	- 202
- 4	2	4	218	179
- 5	2	4	603	- 549
- 6	2	4	821	- 805
- 7	2	4	503	480
- 8	2	4	323	327
- 9	2	4	242	- 216
-10	2	4	82	122
-11	2	4	287	- 293
-12	2	4	362	- 387
- 1	3	4	397	365
- 3	3	4	505	- 481
- 4	3	4	234	- 222
- 5	3	4	438	449
- 6	3	4	283	- 251
- 7	3	4	809	800
- 8	3	4	735	679
-12	3	4	198	- 197
- 1	4	4	412	- 406
- 2	4	4	247	- 191
- 3	4	4	451	- 409
- 4	4	4	379	- 353
- 5	4	4	477	- 428
- 6	4	4	350	323
- 7	4	4	177	- 157
- 8	4	4	181	186
- 9	4	4	109	- 98
-10	4	4	163	- 175
- 3	5	4	230	211
- 4	5	4	102	- 106
- 5	5	4	176	160
- 6	5	4	128	157
- 7	5	4	87	95
-10	5	4	217	- 216
- 1	6	4	174	- 179
- 6	6	4	196	208
- 7	6	4	199	167
- 8	6	4	71	- 52
- 2	7	4	66	- 76
- 3	7	4	96	- 99
- 4	7	4	68	48
0	2	- 4	878	-1021
0	3	- 4	935	- 988
0	4	- 4	138	- 107
0	5	- 4	399	- 366
0	6	- 4	531	529
0	7	- 4	878	853
0	9	- 4	143	- 144
0	10	- 4	148	- 141
1	1	- 4	885	- 937
2	1	- 4	443	- 330
3	1	- 4	3024	3092
4	1	- 4	372	352
5	1	- 4	68	97
6	1	- 4	139	108
7	1	- 4	1053	-1005
8	1	- 4	995	-1061
11	1	- 4	106	- 113
12	1	- 4	68	93
1	2	- 4	3296*	-3240
2	2	- 4	599	- 530
3	2	- 4	862	- 726
4	2	- 4	650	- 583
5	2	- 4	501	502
6	2	- 4	309	275
7	2	- 4	1561	-1477
9	2	- 4	244	- 213
10	2	- 4	111	- 123
11	2	- 4	321	327
12	2	- 4	245	289
1	3	- 4	75	- 111
2	3	- 4	171	106
3	3	- 4	797	- 744
4	3	- 4	932	890
5	3	- 4	2089	2158
6	3	- 4	231	171
7	3	- 4	735	710
8	3	- 4	425	432
9	3	- 4	111	- 117
11	3	- 4	330	324
1	4	- 4	679	663
2	4	- 4	615	634
3	4	- 4	1196	-1198
4	4	- 4	272	213
5	4	- 4	603	548
6	4	- 4	283	- 212
7	4	- 4	94	- 57
8	4	- 4	376	- 345
9	4	- 4	587	- 603
10	4	- 4	292	- 285
1	5	- 4	185	184
2	5	- 4	12334	-1305
3	5	- 4	954	- 933
4	5	- 4	407	- 382
5	5	- 4	556	- 554
6	5	- 4	200	198
8	5	- 4	197	- 181
1	6	- 4	206	153
2	6	- 4	156	- 111
3	6	- 4	284	248
4	6	- 4	301	250
5	6	- 4	247	224
6	6	- 4	724	765
7	6	- 4	613	641
8	6	- 4	186	178
9	6	- 4	210	232
1	7	- 4	333	289
3	7	- 4	157	136
4	7	- 4	558	- 579
5	7	- 4	312	- 260
6	7	- 4	90	80
3	8	- 4	503	- 506
4	8	- 4	464	- 486
5	8	- 4	236	- 235
6	8	- 4	199	- 211
1	9	- 4	316	337
2	9	- 4	427	456
3	9	- 4	220	193
4	9	- 4	62	89
1	10	- 4	122	140
1	1	4	540	529
2	1	4	216	- 209
3	1	4	713	- 590
4	1	4	784	728
1	2	4	459	393
2	2	4	805	- 772
3	2	4	557	- 486
4	2	4	1410	1467
5	2	4	796	727
7	2	4	369	370
8	2	4	157	125
1	3	4	697	717
2	3	4	695	- 666
3	3	4	326	286
4	3	4	645	581
5	3	4	325	302
6	3	4	882	- 854
7	3	4	127	- 124
8	3	4	198	- 188
9	3	4	346	- 353
1	4	4	1013	981
2	4	4	1872	1938
3	4	4	79	34
4	4	4	585	563
5	4	4	306	282
6	4	4	735	- 753
7	4	4	640	- 626
1	5	4	363	- 338
2	5	4	948	973
3	5	4	350	319
4	5	4	737	- 712
5	5	4	411	- 363
6	5	4	439	422
7	5	4	587	- 602
8	5	4	315	305
9	5	4	472	514
10	5	4	82	139
1	6	4	349	324
2	6	4	252	- 238
3	6	4	308	- 287
4	6	4	909	- 885
5	6	4	175	- 168
6	6	4	456	- 441
7	6	4	126	129
8	6	4	118	99
9	6	4	336	363
1	7	4	219	175
3	7	4	287	- 297
4	7	4	86	- 82
6	7	4	94	124
8	7	4	105	154
10	7	4	79	- 51
11	7	4	137	- 175
1	8	4	221	- 249
2	8	4	441	- 452
3	8	4	173	- 144
4	8	4	103	120
6	8	4	241	234
7	8	4	189	175
8	8	4	117	144
2	9	4	334	- 336
1	10	4	159	- 174
2	10	4	210	244
4	10	4	76	51
5	10	4	81	74
6	10	4	68	54
1	0	5	700	- 703
2	0	5	710	- 690
3	0	5	219	- 200
0	0	5	949	- 978
0	1	5	315	- 301
0	4	5	309	301
0	5	5	311	317
1	1	5	215	- 156
2	1	5	290	225
3	1	5	449	- 399
4	1	5	360	332
5	1	5	358	320
1	2	5	576	579
2	2	5	290	- 272
3	2	5	392	- 311
4	2	5	118	- 83
1	3	5	216	- 197
2	3	5	319	- 281
3	3	5	191	- 195
4	3	5	117	- 89
2	4	5	116	- 122
3	4	5	331	307
1	5	5	103	- 92
2	5	5	94	98
- 1	0	5	587	576
- 2	0	5	357	- 321
- 3	0	5	212	- 156
- 4	0	5	1255	1334
- 5	0	5	668	- 625
- 6	0	5	897	- 866
- 7	0	5	459	393
- 8	0	5	148	127
- 9	0	5	415	359
-10	0	5	623	625
-11	0	5	259	241
- 2	1	5	200	192
- 3	1	5	785	- 811
- 4	1	5	134	129
- 5	1	5	923	- 964
- 6	1	5	1132	-1220
- 7	1	5	102	- 74
- 9	1	5	329	- 326
-10	1	5	350	300
-12	1	5	194	- 208
- 1	2	5	125	86
- 2	2	5	809	767
- 3	2	5	979	1001
- 4	2	5	439	- 356
- 5	2	5	398	321
- 7	2	5	89	126
- 8	2	5	273	300
- 9	2	5	155	75
-11	2	5	90	- 70
-12	2	5	127	- 130
- 1	3	5	410	- 394
- 2	3	5	443	428
- 3	3	5	100	82

Table 6. Continued.

H	K	L	FO	FC
-4	3	5	480	-470
-5	3	5	88	-66
-6	3	5	128	108
-8	3	5	294	267
-9	3	5	416	396
-11	3	5	117	113
-1	4	5	122	99
-4	4	5	534	-506
-5	4	5	551	-524
-6	4	5	147	162
-7	4	5	341	-336
-8	4	5	119	108
-9	4	5	84	77
-10	4	5	152	-156
-11	4	5	113	-151
-1	5	5	379	387
-2	5	5	224	-211
-7	5	5	445	453
-2	6	5	213	-236
-3	6	5	263	-285
-7	6	5	329	311
0	1	-5	516	551
0	3	-5	181	-181
0	4	-5	531	492
0	5	-5	206	189
0	6	-5	266	-242
0	7	-5	817	836
0	9	-5	82	-94
1	1	-5	850	952
2	1	-5	402	358
3	1	-5	2393*	2411
4	1	-5	1252	1367
5	1	-5	348	300
7	1	-5	452	-412
8	1	-5	999	-973
9	1	-5	300	259
10	1	-5	138	126
11	1	-5	147	-113
1	2	-5	2034*	-2188
2	2	-5	578	-603
3	2	-5	647	638
4	2	-5	404	-411
5	2	-5	193	108
6	2	-5	590	563
7	2	-5	1738	-1886
8	2	-5	843	-848
9	2	-5	262	-262
10	2	-5	272	-270
11	2	-5	76	-62
1	3	-5	3279*	-3104
2	3	-5	885	-858
3	3	-5	392	329
4	3	-5	89	45
5	3	-5	1417	1361
6	3	-5	1589	1643
7	3	-5	372	355
8	3	-5	182	-145
9	3	-5	229	197
10	3	-5	124	-145
11	3	-5	218	165
1	4	-5	200	-135
2	4	-5	224	200
3	4	-5	586	-559
4	4	-5	495	-447
5	4	-5	1563	1571
6	4	-5	612	591
7	4	-5	442	397
8	4	-5	336	330
9	4	-5	437	-388
10	4	-5	493	-496
1	5	-5	459	395
2	5	-5	781	-730
3	5	-5	2240*	-2408
4	5	-5	799	-741
5	5	-5	640	-636
6	5	-5	699	-698
7	5	-5	146	150
8	5	-5	319	355
9	5	-5	365	-294
10	5	-5	67	-77
1	6	-5	1287	1357
2	6	-5	124	117
3	6	-5	709	-699
4	6	-5	170	132
5	6	-5	182	-177
7	6	-5	546	547
8	6	-5	459	466
9	6	-5	334	323
1	7	-5	1046	1079
2	7	-5	555	539
3	7	-5	601	607
5	7	-5	128	-107
6	7	-5	77	-61
7	7	-5	95	-91
8	7	-5	183	-231
1	8	-5	135	-124
2	8	-5	139	-136
4	8	-5	276	-283
5	8	-5	438	-423
6	8	-5	152	-147
1	9	-5	179	-202
2	9	-5	156	177
3	9	-5	272	259
1	10	-5	152	142
2	10	-5	206	203
1	-1	5	762	-836
2	-1	5	743	726
3	-1	5	717	703
3	-2	5	1141	1197
4	-2	5	662	652
5	-2	5	394	-339
6	-2	5	385	322
1	-3	5	342	307
2	-3	5	330	-318
4	-3	5	181	-120
5	-3	5	456	-424
6	-3	5	593	-534
1	-4	5	2209	2526
2	-4	5	886	789
3	-4	5	358	-270
4	-4	5	87	-58
5	-4	5	131	84
6	-4	5	677	-653
7	-4	5	103	-77
1	-5	5	700	675
2	-5	5	534	500
3	-5	5	859	-874
4	-5	5	294	300
5	-5	5	278	-240
6	-5	5	142	156
8	-5	5	368	375
10	-5	5	183	169
2	-6	5	269	-237
3	-6	5	821	-827
4	-6	5	803	-742
5	-6	5	372	403
6	-6	5	332	-341
8	-6	5	301	290
9	-6	5	191	214
1	-7	5	461	-465
2	-7	5	272	278
3	-7	5	117	96
4	-7	5	342	-309
5	-7	5	209	179
6	-7	5	319	291
9	-7	5	177	175
10	-7	5	223	-200
1	-8	5	516	-500
2	-8	5	344	-344
6	-8	5	347	321
9	-8	5	100	-103
10	-8	5	67	-66
1	-9	5	222	-227
2	-9	5	347	-381
8	-9	5	86	-84
3	-10	5	178	183
4	-10	5	134	135
1	0	6	221	-172
2	0	6	282	-259
3	0	6	475	-462
0	0	6	1437	-1530
0	1	6	427	-389
0	2	6	300	326
0	3	6	493	448
0	4	6	278	263
1	1	6	315	291
2	1	6	372	-333
3	1	6	321	279
1	2	6	135	-86
2	2	6	679	-608
1	3	6	568	-561
2	3	6	306	-287
-1	0	6	1453	-1576
-2	0	6	290	285
-3	0	6	268	-230
-4	0	6	192	167
-5	0	6	517	491
-7	0	6	272	-220
-8	0	6	235	-248
-9	0	6	85	-46
-10	0	6	129	127
-11	0	6	204	197
-12	0	6	149	173
-1	1	6	406	-383
-2	1	6	1224	1178
-3	1	6	574	516
-5	1	6	282	266
-6	1	6	433	-392
-7	1	6	1087	-1170
-8	1	6	197	-181
-9	1	6	160	-168
-11	1	6	71	67
-12	1	6	47	62
-1	2	6	146	112
-2	2	6	852	851
-3	2	6	1222	1278
-4	2	6	446	-426
-5	2	6	350	311
-6	2	6	328	-273
-7	2	6	416	-428
-8	2	6	200	187
-9	2	6	416	461
-11	2	6	128	111
-1	3	6	88	92
-2	3	6	294	-230
-4	3	6	156	-153
-5	3	6	1097	-1136
-6	3	6	186	-144
-7	3	6	128	95
-8	3	6	162	170
-9	3	6	269	269
-10	3	6	220	171
-1	4	6	462	448
-2	4	6	282	-282
-3	4	6	129	-105
-4	4	6	140	-144
-5	4	6	532	-573
-7	4	6	372	426
-1	5	6	153	137
-2	5	6	129	-89
-3	5	6	141	-142
-5	5	6	81	82
-6	5	6	218	208
-7	5	6	451	409
-3	6	6	202	-209
-5	6	6	120	159
0	1	-6	809	-840
0	3	-6	485	489
0	4	-6	1137	1167
0	5	-6	868	847
0	6	-6	532	-540
0	7	-6	179	152
0	8	-6	145	-140
0	9	-6	183	-185
0	10	-6	140	123
1	1	-6	264	234
2	1	-6	312	-277
3	1	-6	474	439
4	1	-6	1332	1454
5	1	-6	773	699
6	1	-6	354	309
7	1	-6	897	903
8	1	-6	299	-224
9	1	-6	203	-205
10	1	-6	158	152
11	1	-6	123	-77
1	2	-6	440	544
2	2	-6	1457	-1706
3	2	-6	385	-348
4	2	-6	765	670
5	2	-6	735	-636
6	2	-6	453	-389
8	2	-6	453	-404
9	2	-6	481	-489
10	2	-6	145	-139
11	2	-6	150	-134
1	3	-6	1390	-1529
2	3	-6	1359	-1455
3	3	-6	496	-440
4	3	-6	199	-144
5	3	-6	443	-385
6	3	-6	856	764
7	3	-6	211	-150
9	3	-6	351	340
2	4	-6	620	579
3	4	-6	637	524
4	4	-6	591	-514
5	4	-6	1640	1683
6	4	-6	706	682
8	4	-6	193	159
9	4	-6	222	203
10	4	-6	452	-411
1	5	-6	262	-201
2	5	-6	212	158
3	5	-6	895	-829
4	5	-6	1098	-1063
5	5	-6	275	-222
6	5	-6	197	170
7	5	-6	400	-388
9	5	-6	226	-191
10	5	-6	194	-235
1	6	-6	268	240
2	6	-6	256	222
3	6	-6	1103	-1120
4	6	-6	597	-549
5	6	-6	88	-87
6	6	-7	134	-117
7	6	-6	404	397
8	6	-6	520	542
9	6	-6	256	247
1	7	-6	1240	1348
2	7	-6	697	642
3	7	-6	166	168
4	7	-6	356	375
6	7	-6	331	-314
7	7	-6	346	317
8	7	-6	211	201
1	8	-6	488	484
2	8	-6	117	-93
3	8	-6	89	60
5	8	-6	540	-547
6	8	-6	556	-538
1	9	-6	303	-303
3	9	-6	131	162
4	9	-6	119	125
1	-1	6	176	131
2	-1	6	573	806
3	-1	6	164	92
4	-1	6	284	-208
1	-2	6	237	171
2	-2	6	1524	1617
3	-2	6	406	350
4	-2	6	322	299
5	-2	6	275	233
7	-2	6	273	-260
1	-3	6	484	-447
3	-3	6	327	307
4	-3	6	906	-820
5	-3	6	193	-114
7	-3	6	306	-282
1	-4	6	1449	1450
3	-4	6	218	152
4	-4	6	386	-315
5	-4	6	600	-588
6	-4	6	189	-197
8	-4	6	100	-105
9	-4	6	141	143
1	-5	6	1282	1346
2	-5	6	311	-290
3	-5	6	293	-240
4	-5	6	352	297
5	-5	6	368	-350
6	-5	6	182	131
7	-5	6	380	346
8	-5	6	228	196
1	-6	6	105	-115
2	-6	6	584	-569
3	-6	6	1321	-1283
4	-6	6	470	-430
7	-6	6	111	81
8	-6	6	188	186
1	-7	6	227	-211
3	-7	6	410	-366
4	-7	6	252	-207
5	-7	6	561	512
7	-7	6	191	200
8	-7	6	194	-215
1	-8	6	590	-573
2	-8	6	244	252
4	-8	6	276	270
5	-8	6	534	550
6	-8	6	290	303
7	-8	6	222	-235
8	-8	6	99	140
1	-9	6	503	-553
2	-9	6	288	-309
3	-9	6	229	251
5	-9	6	100	86
7	-9	6	233	-223
3	-10	6	329	356
4	-10	6	101	121
5	-10	6	103	-116
6	-10	6	79	-60
1	0	7	248	174
2	0	7	147	-149
0	0	7	605	-585
0	1	7	250	208
0	2	7	106	134
0	3	7	67	27
1	1	7	240	-222
2	1	7	211	-204
1	2	7	760	-756
2	2	7	244	-244
1	3	7	272	-301
-1	0	7	1606	-1767
-2	0	7	597	557
-3	0	7	128	-76
-4	0	7	178	-168
-5	0	7	319	301
-6	0	7	203	-210
-7	0	7	771	-817
-8	0	7	69	-93

Table 6. Continued.

H	K	L	FO	FC
-10	0	7	156	- 166
-11	0	7	207	214
- 3	1	7	1353	1319
- 4	1	7	89	76
- 5	1	7	431	- 416
- 7	1	7	337	- 306
- 8	1	7	309	- 296
-10	1	7	84	106
- 1	2	7	156	141
- 3	2	7	634	619
- 5	2	7	440	- 425
- 6	2	7	115	131
- 7	2	7	237	253
- 8	2	7	144	- 136
- 9	2	7	246	257
-10	2	7	230	240
- 1	3	7	216	192
- 2	3	7	131	- 133
- 3	3	7	131	- 121
- 5	3	7	478	- 526
- 6	3	7	77	40
- 7	3	7	78	95
- 8	3	7	178	- 109
- 9	3	7	66	67
-10	3	7	131	163
- 1	4	7	346	337
- 2	4	7	141	144
- 3	4	7	90	- 119
- 4	4	7	118	90
- 5	4	7	75	42
- 6	4	7	77	- 98
- 3	5	7	122	- 108
- 5	5	7	131	143
- 6	5	7	125	- 151
0	1	7	947	- 931
0	3	7	140	- 155
0	4	7	682	606
0	5	7	1034	996
0	6	7	113	86
0	7	7	237	170
0	8	7	82	- 76
0	9	7	475	- 563
0	10	7	131	- 121
1	1	7	736	- 769
2	1	7	350	337
3	1	7	52	- 42
4	1	7	372	363
5	1	7	1353	1532
6	1	7	338	238
7	1	7	141	- 105
8	1	7	115	88
9	1	7	84	- 109
10	1	7	267	- 281
11	1	7	83	- 51
1	2	7	301	321
2	2	7	123	- 101
3	2	7	524	- 538
4	2	7	240	246
5	2	7	762	690
6	2	7	253	218
8	2	7	141	- 130
9	2	7	422	- 450
10	2	7	442	- 482
11	2	7	188	- 194
2	3	7	1159	-1236
3	3	7	1262	-1238
4	3	7	221	- 174
5	3	7	658	- 616
6	3	7	379	385
7	3	7	484	471
8	3	7	178	157
10	3	7	102	112
11	3	7	131	- 141
1	4	7	706	698
2	4	7	618	- 583
4	4	7	312	270
6	4	7	247	204
7	4	7	844	930
8	4	7	354	367
9	4	7	246	258
10	4	7	86	83
1	5	7	421	370
3	5	7	456	435
4	5	7	788	- 757
5	5	7	506	- 496
7	5	7	337	- 346
1	6	7	551	- 492
2	6	7	460	422
3	6	7	812	- 818
4	6	7	594	- 573
5	6	7	235	- 222
6	6	7	262	- 274
7	6	7	327	- 368
8	6	7	344	277
9	6	7	298	326
1	7	7	611	557
2	7	7	664	649
3	7	7	419	374
4	7	7	133	122
5	7	7	329	360
1	8	7	619	644
2	8	7	374	334
3	8	7	111	115
4	8	7	77	109
5	8	7	94	- 90
6	8	7	331	- 334
1	9	7	63	- 101
2	9	7	110	- 136
3	9	7	97	- 120
4	9	7	199	204
1	10	7	62	- 98
2	10	7	69	- 81
1	1	7	1035	1057
1	2	7	1389	1364
2	2	7	1112	1033
3	2	7	395	- 363
4	2	7	348	297
6	2	7	512	- 499
1	3	7	472	- 451
2	3	7	488	428
3	3	7	429	- 366
4	3	7	787	- 716
5	3	7	66	51
6	3	7	133	98
8	3	7	359	343
1	4	7	812	- 798
2	4	7	503	447
3	4	7	197	- 153
4	4	7	1116	-1071
5	4	7	66	54
6	4	7	115	76
7	4	7	94	- 90
9	4	7	140	82
1	5	7	944	865
2	5	7	79	112
3	5	7	558	524
4	5	7	214	- 181
5	5	7	213	151
6	5	7	603	574
7	5	7	88	- 106
9	5	7	141	167
1	6	7	330	297
2	6	7	604	- 566
3	6	7	695	- 666
5	6	7	217	- 177
6	6	7	289	296
9	6	7	64	42
1	7	7	263	216
2	7	7	774	- 830
3	7	7	650	- 664
4	7	7	69	76
6	7	7	241	- 240
7	7	7	219	268
8	7	7	180	- 183
9	7	7	100	- 96
1	8	7	375	- 371
3	8	7	150	- 149
4	8	7	327	333
5	8	7	442	460
6	8	7	166	195
7	8	7	84	67
9	8	7	59	- 65
1	9	7	700	- 823
2	9	7	119	- 128
3	9	7	118	175
4	9	7	69	72
5	9	7	245	241
6	9	7	176	149
7	9	7	189	- 170
8	9	7	50	- 47
1	10	7	262	- 249
2	10	7	165	192
3	10	7	331	359
5	10	7	121	107
6	10	7	78	- 121
0	0	8	159	90
1	0	8	469	- 496
0	1	8	272	258
0	2	8	217	- 228
1	1	8	209	- 160
- 1	0	8	515	- 542
- 2	0	8	220	- 214
- 3	0	8	283	268
- 4	0	8	176	124
- 5	0	8	288	- 282
- 6	0	8	362	372
- 7	0	8	190	- 184
- 8	0	8	356	- 352
- 9	0	8	99	- 81
- 1	1	8	302	279
- 2	1	8	198	- 174
- 3	1	8	768	779
- 4	1	8	232	189
- 6	1	8	120	116
- 8	1	8	382	- 395
- 9	1	8	123	- 130
- 3	2	8	84	- 71
- 4	2	8	258	270
- 6	2	8	103	- 95
- 7	2	8	70	- 38
- 9	2	8	79	- 81
- 2	3	8	240	192
- 3	3	8	197	- 183
- 4	3	8	95	62
- 6	3	8	253	- 296
- 7	3	8	69	- 39
- 8	3	8	54	- 50
- 3	4	8	90	89
- 4	4	8	73	- 52
0	1	8	205	- 149
0	2	8	450	- 513
0	3	8	173	- 198
0	4	8	486	498
0	5	8	764	793
0	6	8	111	- 61
0	7	8	300	- 297
0	8	8	225	- 188
1	1	8	1045	-1165
2	1	8	386	- 372
3	1	8	387	369
4	1	8	142	- 78
5	1	8	469	431
6	1	8	622	660
7	1	8	292	291
8	1	8	71	- 76
9	1	8	152	171
10	1	8	196	- 142
11	1	8	154	- 193
1	2	8	383	- 375
2	2	8	287	295
3	2	8	349	- 365
4	2	8	437	- 410
5	2	8	727	730
6	2	8	336	284
7	2	8	140	- 114
8	2	8	276	282
9	2	8	219	- 166
10	2	8	569	- 528
3	3	8	1199	-1428
4	3	8	724	- 744
5	3	8	66	- 40
6	3	8	267	- 229
8	3	8	498	500
9	3	8	160	117
10	3	8	206	- 253
1	4	8	736	714
2	4	8	173	98
3	4	8	600	- 543
5	4	8	69	- 80
6	4	8	216	- 186
7	4	8	508	477
8	4	8	666	693
9	4	8	178	175
10	4	8	168	170
1	5	8	1259	1343
2	5	8	267	224
3	5	8	547	580
5	5	8	375	- 321
6	5	8	522	- 508
8	5	8	123	- 118
1	6	8	257	247
2	6	8	176	- 149
3	6	8	189	- 143
4	6	8	356	317
5	6	8	634	- 683
6	6	8	373	- 390
7	6	8	221	- 199
8	6	8	76	- 65
1	7	8	402	- 424
2	7	8	238	189
3	7	8	490	483
4	7	8	221	179
5	7	8	242	256
6	7	8	202	184
2	8	8	160	186
3	8	8	178	166
5	8	8	188	198
6	8	8	134	- 119
1	9	8	214	- 213
2	9	8	132	- 137
3	9	8	219	- 192
1	1	8	470	- 467
1	2	8	1519	1622
2	2	8	720	686
3	2	8	331	296
4	2	8	332	273
5	2	8	271	- 225
6	2	8	378	- 399
1	3	8	1293	1456
2	3	8	1052	985
3	3	8	881	- 779
5	3	8	138	- 145
6	3	8	297	- 306
7	3	8	212	190
1	4	8	172	175
3	4	8	1328	-1356
4	4	8	909	- 930
5	4	8	102	90
6	4	8	372	- 372
7	4	8	134	129
8	4	8	338	337
1	5	8	356	- 333
2	5	8	443	- 425
3	5	8	291	- 277
4	5	8	533	- 504
5	5	8	384	335
6	5	8	503	510
7	5	8	75	61
8	5	8	125	82
1	6	8	1192	-1221
2	6	8	503	- 479
3	6	8	447	399
4	6	8	143	- 125
5	6	8	428	407
6	6	8	773	804
1	7	8	514	- 510
2	7	8	337	- 315
3	7	8	137	114
4	7	8	207	189
6	7	8	202	195
7	7	8	181	- 194
8	7	8	356	- 337
1	8	8	449	486
2	8	8	254	246
3	8	8	210	194
4	8	8	331	318
5	8	8	185	150
6	8	8	90	- 111
3	9	8	130	- 120
4	9	8	237	278
6	9	8	116	- 116
1	10	8	210	- 223
2	10	8	141	- 139
4	10	8	76	- 110
6	10	8	119	- 165
12	0	0	106	- 111
0	0	9	198	- 155
0	0	10	205	- 193
6	0	2	691	- 735
7	0	2	83	- 87
8	0	2	438	- 476
9	0	2	529	- 614
11	0	2	105	- 100
12	0	2	39	- 36
6	0	3	81	89
7	0	3	838	977
8	0	3	400	- 405
9	0	3	387	- 469
10	0	3	103	- 122
11	0	3	180	- 172
4	0	4	652	- 767
5	0	4	412	- 476
6	0	4	249	221
7	0	4	312	315
8	0	4	424	- 475
9	0	4	197	213
5	0	5	429	436
6	0	5	144	- 98
7	0	5	152	99
9	0	5	111	- 114
4	0	6	461	435
6	0	6	251	- 232
8	0	6	134	162
9	0	6	178	- 261
3	0	7	303	284
4	0	7	617	594
5	0	7	151	- 174
2	0	8	284	- 288
3	0	8	599	554
4	0	8	117	119
5	0	8	95	70
6	0	8	226	194
1	0	9	303	258
2	0	9	583	608
3	0	9	198	147
4	0	9	214	- 181
5	0	9	194	155
1	0	10	187	170
2	0	10	110	124
4	0	10	112	118
1	0	11	244	238
4	0	- 1	870	757

Table 6. Continued.

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
5	0	-1	257	-281	5	1	7	83	80	1	1	-12	44	-91	4	-1	9	261	-269
6	0	-1	825	713	6	1	3	668	659	2	1	-9	238	-217	4	-1	10	120	-108
7	0	-1	612	593	6	1	4	827	854	2	1	-10	69	-97	5	-1	5	130	150
9	0	-1	81	81	6	1	5	368	388	2	1	-11	185	221	5	-1	6	253	268
10	0	-1	320	387	6	1	7	215	208	3	1	-10	633	-691	5	-1	7	392	-393
11	0	-1	319	-292	7	1	2	1026	1133	3	1	-11	235	-203	5	-1	8	420	-472
12	0	-1	341	-371	7	1	3	759	837	4	1	-10	95	-78	5	-1	9	253	-215
1	0	-9	203	152	7	1	4	244	-217	4	1	-11	622	-575	6	-1	2	842	793
2	0	-9	334	-302	7	1	6	218	212	5	1	-9	219	-211	6	-1	3	272	244
3	0	-9	311	-302	7	1	7	49	-23	5	1	-10	83	104	6	-1	4	69	96
7	0	-9	131	121	8	1	3	442	514	5	1	-11	92	91	6	-1	5	134	99
8	0	-9	170	-144	8	1	4	180	177	5	1	-12	187	-160	6	-1	6	373	-381
9	0	-9	309	-355	8	1	5	206	189	6	1	-9	450	466	6	-1	7	597	-650
1	0	-10	78	-78	9	1	2	191	-182	7	1	-1	209	-209	6	-1	9	47	24
3	0	-10	244	-242	9	1	3	261	316	7	1	-9	504	476	7	-1	2	358	-333
4	0	-10	164	-139	9	1	4	97	68	7	1	-10	325	294	7	-1	3	590	575
5	0	-10	261	283	9	1	5	166	-190	8	1	-1	364	-392	7	-1	4	95	111
8	0	-10	117	130	10	1	2	137	139	8	1	-2	66	-40	7	-1	5	533	-538
1	0	-11	110	-91	10	1	4	76	-33	8	1	-5	988	-973	7	-1	6	101	-100
2	0	-11	114	95	-11	1	0	88	-76	8	1	-10	335	312	7	-1	7	59	45
4	0	-11	279	-332	-6	1	1	466	-461	9	1	-1	192	184	8	-1	2	447	445
6	0	-11	246	310	-7	1	1	848	806	10	1	-1	441	504	8	-1	3	522	-487
					-8	1	1	616	590	1	-1	9	84	-96	8	-1	4	315	-288
8	1	0	112	97	-8	1	3	131	107	1	-1	11	194	205	8	-1	5	377	-394
10	1	0	131	151	-9	1	1	287	280	2	-1	8	212	-207	8	-1	6	228	-211
11	1	0	56	-38	-11	1	1	151	149	2	-1	10	414	408	8	-1	7	107	-85
2	1	8	134	-76	-12	1	1	349	-359	3	-1	7	268	-250	9	-1	3	984	-1007
3	1	7	716	799	0	1	-3	396	-397	3	-1	8	435	478	9	-1	4	297	-293
3	1	8	107	114	0	1	-4	485	526	3	-1	9	63	-82	9	-1	5	121	77
4	1	4	274	-255	0	1	-9	516	544	3	-1	10	52	-84	10	-1	2	120	-138
4	1	6	601	602	0	1	-10	171	-198	3	-1	11	86	-102	10	-1	3	84	-100
4	1	7	219	220	1	1	-9	515	-504	4	-1	5	99	95	10	-1	4	118	-114
4	1	8	76	-86	1	1	-10	89	30	4	-1	7	254	268	10	-1	5	223	-193
5	1	1	68	-109	1	1	-11	144	-135	4	-1	8	177	209	11	-1	2	221	183
5	1	4	150	118															

in (VI). With regard to (V) there seems to be a small angle, 6.6°, between the plane of *B* and the plane of *C*.

The spatial orientation of phenyl groups *A* and *B* relative to the central ring system is rather similar in the three compounds. Thus, reckoned in the order IV—VI, the twist angles are 25, 24 and 36° for *A* and 52, 60 and 59° for *B*.

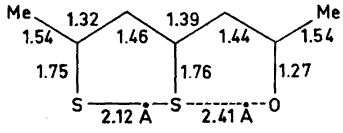
If one subtracts the difference in covalent radii of selenium and sulphur, 0.12 Å, from the S(2)—Se distance in (V), one arrives at the value 2.21 Å which is close to that found for the S(2)—S(3) distance in (IV), 2.22 Å. This, and the close fit between the S(1)—S(2) distance in the two compounds, 2.50 Å in (IV) and 2.49 Å in (V), show that the bonding in the linear three-atom sequence of (IV) remains almost unchanged if S(3) is replaced by selenium. Substitution of S(3) with oxygen, on the other hand, causes a pronounced shortening of the S(1)—S(2) bond, *cf.* compound (VI), as discussed below.

The S(1)—S(2) distance in (VI), 2.11 Å, is close to the value 2.10 Å for a sulphur-sulphur single bond in a *cis*-planar disulphide group, and 0.39 Å shorter than the S(1)—S(2) distance in (IV). The S(2)—O distance, 2.38 Å, is 0.87 Å shorter than the corresponding van der Waals distance and 0.66 Å longer than the sum of the covalent radii for oxygen and sulphur, and there is thus partial bonding between S(2) and O. The linearity of the sulphur-sulphur-oxygen sequence in (VI), S(1)—S(2)—O= 174°, is almost the same as that of the tri-sulphur sequence in (IV), S(1)—S(2)—S(3)=178°, although the C—O bond in (VI) is 0.41 Å shorter than the corresponding C—S(3) bond in (IV). Due to better overlap, one may expect the three-center four-electron bonding in the S—S—O sequence to be most efficient when the three atoms are exactly on a line. It is therefore interesting to note that molecule (VI) seems to have adjusted itself to attain a linear S—S—O sequence.

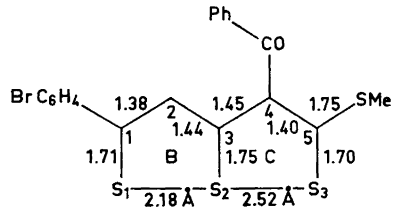
The difference between the bonding in the S—S—S arrangement of (IV) and the S—S—O arrangement of (VI) may be explained by reference to the bonding in the linear trihalide ions. It has been found for the latter, through structure investigations and by theoretical calculations,[12,13,25,26] that the less electronegative of the terminal halogen atoms forms the strongest bond with the central halogen atom. Hence, with regard to (VI), S(1)—S(2) should be a stronger bond than S(2)—O, which in fact has been found.

In accordance with this, one may also understand why S(1)—S(2) in (IV) is a weaker bond than S(2)—S(3). As phenyl group *A* there is closer to S(1) than phenyl group *D* is to S(3), S(1) becomes more electronegative than S(3) and thus S(1)—S(2) longer than S(2)—S(3).

From the values given in Fig. 3, there is good agreement between corresponding C—S and C—C bond lengths in (IV) and (V). It should be noted that in both compounds, C(3)—C(4) is found to be the longest and C(4)—C(5) the shortest C—C bond, *i.e.*, C(3)—C(4)=1.45 Å in (IV) and 1.49 Å in (V), and C(4)—C(5)=1.36 Å in (IV) and 1.37 Å in (V). Compound (IV) and (V) differ in this respect from (VI). There, C(3)—C(4)=1.39 Å, is found to be shorter than C(4)—C(5), 1.43 Å. Thus there are alternating short and long C—C bonds in (VI) from C(1) to C(5). This, in fact, has also been found for the related compound 2,5-dimethyl-dithiofurophthene (VII).[27]



(VII)



(VIII)

The structure of the unsymmetrical thiothiophthene derivative (VIII) has been studied by Paul *et al.*[7] The bond lengths given for this compound are averages of respective distances from two independent molecules. According to the authors "there is good agreement between the two independent molecules with the exception of the "long" S—S distance", which is 2.47 Å in one and 2.57 Å in the other. Since the standard deviation, σ, in S—S distance is reported to be ± 0.007 Å, it is assumed that this difference of 14 σ is significant. This in fact supports the assumption that intermolecular as well as intramolecular environment may influence the bonding in the linear trisulphur sequence.

There is good agreement between the C—S and C—C bond lengths in (VIII) and the corresponding C—S and C—C bond lengths in (IV). It should be noted that the average value of the C(2)—C(3) and C(3)—C(4) bond lengths in (VIII), 1.45 Å, is larger than the average values of the C(1)—C(2) and C(4)—C(5) bond lengths there, 1.39 Å. Corresponding average values for

(IV) are 1.43 and 1.37 Å, respectively. As regards this structural feature, the thiothiophthene system resembles naphthalene.

In compound (VIII) there are two substituent groups to ring *C* and one to ring *B*. Furthermore, S(1)—S(2) has been found to be shorter than S(2)—S(3). If one assumes that the benzoyl and the methylmercapto groups together are able to withdraw more electrons from S(3) than the *para*-bromophenyl group withdraws from S(1), thus making S(3) more electronegative than S(1), one should expect S(1)—S(2) to be a shorter bond than S(2)—S(3), in agreement with the experimental results.

Charge distribution and bond orders. The π-electron distribution and the π-bond orders in the thiothiophthene system of 2,4-diphenyl-thiothiophthene are given in Fig. 4. The values have been calculated according to a self-

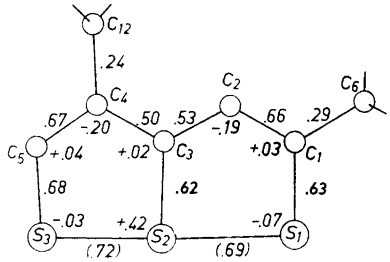


Fig. 4. π-Electron distribution, π-bond orders and σ-bond orders (in parentheses) in the thiothiophthene system of 2,4-diphenyl-thiothiophthene.

consistent parameter MO-method devised by Bergson.[28] The same method has been used by two of us (A.H. and E.S.) [22] for the electronic structures of the 3- and 4-phenyl-1,2-dithiolium ions, where it gave a good description of the structures.

The values given in parentheses in Fig. 4 are σ-bond orders for the sulphur-sulphur bonds. The model used for the tri-sulphur sequence is that of the three-center four-electron bond.[12,13] The values, 0.69 and 0.72 for the σ-bond orders, were obtained by assuming S(1) to be slightly more electronegative than S(3) and adjusting the coulomb integral of S(1) accordingly. The adjustment was made by the ω-formula,[29] taking the electron-density difference between S(1) and S(3) equal to that of the π-densities, 0.04 electrons.

The π-bond orders found for the S(1)—S(2) and S(2)—S(3) bonds, 0.24, are not given in Fig. 4 because one may doubt how they should be interpreted. They appear because the Slater *p*-orbitals for sulphur, also at sulphur-sulphur distances as in the present compound, overlap to some degree.

There is good qualitative agreement between the bond orders in Fig. 4 and the bond lengths in Fig. 1a.

Acknowledgements. The authors are indebted to Dr. E. Klingsberg, American Cyanamid Co., for providing a sample of 2,4-diphenyl-thiothiophthene, and to Professor Olav Foss, this Institute, for valuable criticism.

Furthermore, one of us (A.H.) who stayed at the Crystallography Laboratory, University of Pittsburgh, while writing this paper, wishes to thank Professor George A. Jeffrey and his staff members for great hospitality. He is grateful to Mrs. Joan Klinger who typed the manuscript.

A grant from the *Norwegian Council for Scientific and Industrial Research* is gratefully acknowledged.

REFERENCES

1. Giacometti, G. and Rigatti, G. *J. Chem. Phys.* **30** (1959) 1633.
2. Gleiter, R. and Hoffmann, R. *Tetrahedron* **24** (1968) 5899.
3. Bezzi, S., Mammi, M. and Garbuglio, C. *Nature* **182** (1958) 247.
4. Bezzi, S., Garbuglio, C., Mammi, M. and Traverso, G. *Gazz. Chim. Ital.* **88** (1958) 1226.
5. Mammi, M., Bardi, R., Garbuglio, C. and Bezzi, S. *Acta Cryst.* **13** (1960) 1048.
6. Hordvik, A. *Acta Chem. Scand.* **20** (1966) 1885.
7. Johnson, S. M., Newton, M. G., Paul, I. C., Beer, R. J. S. and Cartwright, D. *Chem. Commun.* **1967** 1170.
8. van der Hende, J. and Klingsberg, E. *J. Am. Chem. Soc.* **88** (1966) 5045.
9. Hordvik, A. *Acta Chem. Scand.* **22** (1968) 2397.
10. Pauling, L. *The Nature of the Chemical Bond*, 3rd Ed., Cornell University Press, Ithaca, New York 1960.
11. Hassel, O. and Strømme, K. O. *Acta Chem. Scand.* **13** (1959) 1781.
12. Havinga, E. E. and Wiebenga, E. H. *Rec. Trav. Chim.* **78** (1959) 724.
13. Wiebenga, E. H., Havinga, E. E. and Boswijk, K. H. *Advan. Inorg. Chem. Radiochem.* **3** (1961) 133.
14. Hordvik, A., Sletten, E. and Sletten, J. *Acta Chem. Scand.* **20** (1966) 1172.
15. Wilson, A. J. C. *Acta Cryst.* **2** (1949) 318.
16. Howells, E. R., Phillips, D. C. and Rogers, D. *Acta Cryst.* **3** (1950) 210.
17. *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham 1962, Vol. III, p. 204.
18. Hordvik, A., Sletten, E. and Sletten, J. *Acta Chem. Scand.* **20** (1966) 2001.
19. Mair, G. A. *Structure Factor and Least Squares Programs for the IBM 1620*, Pure Chemistry Division, National Research Council, Ottawa, Canada 1963.
20. Pimock, P. R., Taylor, C. A. and Lipson, H. *Acta Cryst.* **9** (1956) 173.
21. Abrahams, S. C. *Quart. Rev. (London)* **10** (1956) 407.
22. Hordvik, A. and Sletten, E. *Acta Chem. Scand.* **20** (1966) 1938.
23. Robertson, G. B. *Nature* **191** (1961) 593.
24. Hordvik, A., Sletten, E. and Sletten, J. *American Crystallographic Association*, Summer Meeting, Buffalo 1968. Abstr. 32, and *Acta Chem. Scand. To be published.*
25. Migchelsen, T. and Vos, A. *Acta Cryst.* **22** (1967) 812.
26. Snyder, R. L. *A Study of the Triatomic Polyhalide Anions.* Doctoral Thesis, Fordham University, New York, N. Y. 1968.
27. Mammi, M., Bardi, R., Traverso, G. and Bezzi, S. *Nature* **192** (1961) 1282.
28. Bergson, G. *Arkiv Kemi* **19** (1962) 181.
29. Streitwieser, A., Jr. *Molecular Orbital Theory for Organic Chemists*, Wiley, New York 1961, p. 109.

Received October 9, 1968.