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The Crystal and Molecular Structure of 2,3,4-Triphenyl-thiothiophthene

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An X-ray structure study of crystals of 2,3,4-triphenyl-thiothiophthene has been carried out. The crystals belong to the space group $P2_1$, with unit cell dimensions, $a = 6.296(6)$ Å, $b = 15.134(6)$ Å, $c = 9.862(4)$ Å, and $\beta = 91.52(5)°$. There are two molecules per unit cell; density, calc. 1.373, found 1.37 – 1.40 g/cm³.

The structure study is based on X-ray data collected on a Picker four-angle automatic diffractometer, using Cu$K\alpha$ radiation and omega scan. 1221 reflections were observed within $\sin\theta = 0.85$.

The structure was solved by three-dimensional Patterson synthesis, and refined by least squares methods. The final R factor is 0.056.

Unequal S – S distances occur in the linear three-sulphur sequence of the molecule: S(1) – S(2) = 2.270(4) Å, S(2) – S(3) = 2.375(4) Å, with the angle S(1) – S(2) – S(3) = 176.0(2)°. The other bond lengths in the thiothiophthene system are S(1) – C(1) = 1.680(8) Å, S(2) – C(3) = 1.759(8) Å, S(3) – C(5) = 1.669(10) Å, C(1) – C(2) = 1.379(12) Å, C(2) – C(3) = 1.433(11) Å, C(3) – C(4) = 1.414(12) Å, and C(4) – C(5) = 1.371(12) Å.

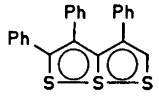
The C – C bonds connecting the phenyl groups to the thiothiophthene system are C(1) – C(6) = 1.498(11) Å, C(2) – C(12) = 1.498(9) Å, and C(4) – C(18) = 1.505(11) Å.

The S – S, S – C, and C – C bond lengths have been corrected for libration.

The phenyl groups bonded to C(1), C(2), and C(4) are twisted 53.6, 61.4, and 58.7° about the respective connection bonds.

The thiothiophthene system is not quite planar; the angle between the normals to the planes of the two five-membered rings is 9.1°.

The present structure investigation of 2,3,4-triphenyl-thiothiophthene (I) has been carried out in order to find to which degree the intramolecular strain, in this case due to the phenyl substituents, affects the bonding in the thiothiophthene system.



(I)

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STRUCTURE DETERMINATION

A brief account of the structure determination was reported at the *VIIIth IUC Meeting 1969*,[1] and a more detailed description is given here.

Crystals of 2,3,4-triphenyl-thiothiophthene (I) were generously supplied by Klingsberg.[2] The crystals are deep purple, and belong to the monoclinic space group $P2_1$.

The structure analysis is based on X-ray data collected on a Picker four-angle automatic diffractometer, using Cu$K\alpha$ radiation and omega scan. 1221 reflections were observed within $\sin\theta = 0.85$.

Approximate coordinates for the sulphur atoms and the carbon atoms of the thiothiophthene system were found from a three-dimensional Patterson map, and the carbon atoms of the phenyl groups revealed themselves during subsequent Fourier syntheses.

The structure refinement was carried out by Shiono's version of the Busing, Martin, and Levy full-matrix least-squares IBM 7090 program.[3,4] Anisotropic temperature factors were applied to sulphur and carbon, and isotropic to hydrogen. The final R factor is 0.056.

A rigid-body analysis of the 2,3,4-triphenyl-thiothiophthene molecule has been carried out according to the method of Schomaker and Trueblood,[5] and the S–S, S–C, and C–C bond lengths have been corrected for rigid-body libration according to Cruickshank's formula.[6]

For further details, with respect to the structure determination, see Experimental.

DISCUSSION

Molecular shape and dimensions. Bond lengths and angles in the 2,3,4-triphenyl-thiothiophthene molecule, together with their standard deviations, are listed in Tables 1 and 2, and shown in Figs. 1a and 1b.

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

$$-0.64457X + 0.64425Y + 0.43133Z = 1.81744$$

with X, Y, and Z in Å units. Deviations from the plane for the atoms of the thiothiophthene system, the atoms C(6) and C(9) of phenyl group A, the atoms C(12) and C(15) of phenyl group E, and the atoms C(18) and C(21) of phenyl group D, are given in Fig. 1a. It is seen that the deviations from the plane for the atoms of the thiothiophthene system range from 0.047 to 0.183 Å, and thus the thiothiophthene system deviates somewhat from planarity. One notes furthermore that the atomic sequences C(1)–C(6)–C(9), C(2)–C(12)–C(15), and C(4)–C(18)–C(21) point out of the plane. A view of the molecule along the edge of the least squares plane of the thiothiophthene system is given in Fig. 2.

The equation for the least squares plane through the atoms of ring B, with triple weight on sulphur, is

$$0.67620X - 0.65086Y - 0.36295Z = -1.76836$$

Table 1. Bond lengths (l) and standard deviations in bond lengths $\sigma(l)$ in 2,3,4-triphenyl-thiothiophthene. Bond lengths (l') include correction for rigid-body libration.

Bond	l' (Å)	l (Å)	$\sigma(l)$ (Å)
S(1) – S(2)	2.270	2.267	0.004
S(1) – C(1)	1.680	1.676	0.008
S(2) – S(3)	2.375	2.371	0.004
S(2) – C(3)	1.759	1.755	0.008
S(3) – C(5)	1.669	1.666	0.010
C(1) – C(2)	1.379	1.377	0.012
C(1) – C(6)	1.498	1.495	0.011
C(2) – C(3)	1.433	1.430	0.011
C(2) – C(12)	1.498	1.495	0.009
C(3) – C(4)	1.414	1.412	0.012
C(4) – C(5)	1.371	1.367	0.012
C(4) – C(18)	1.505	1.501	0.011
C(6) – C(7)	1.383	1.379	0.009
C(7) – C(8)	1.398	1.396	0.011
C(8) – C(9)	1.376	1.373	0.013
C(9) – C(10)	1.367	1.364	0.013
C(10) – C(11)	1.396	1.394	0.012
C(11) – C(6)	1.389	1.392	0.011
C(12) – C(13)	1.405	1.401	0.009
C(13) – C(14)	1.393	1.390	0.010
C(14) – C(15)	1.390	1.386	0.011
C(15) – C(16)	1.376	1.373	0.010
C(16) – C(17)	1.375	1.372	0.009
C(17) – C(12)	1.369	1.367	0.010
C(18) – C(19)	1.374	1.371	0.010
C(19) – C(20)	1.378	1.376	0.011
C(20) – C(21)	1.373	1.369	0.012
C(21) – C(22)	1.371	1.368	0.012
C(22) – C(23)	1.391	1.388	0.011
C(23) – C(18)	1.389	1.385	0.010

Table 2. Bond angles ∠(ijk) in 2,3,4-triphenyl-thiothiophthene. Standard deviations in parenthesis refer to the last digits of respective values.

i	j	k	∠(ijk)°	i	j	k	∠(ijk)°
C(1)	S(1)	S(2)	94.5 (3)	C(6)	C(7)	C(8)	118.9 (7)
S(1)	S(2)	S(3)	176.0 (2)	C(7)	C(8)	C(9)	121.4 (8)
S(1)	S(2)	C(3)	90.3 (3)	C(8)	C(9)	C(10)	119.6 (8)
C(3)	S(2)	S(3)	90.4 (3)	C(9)	C(10)	C(11)	120.1 (8)
S(2)	S(3)	C(5)	90.0 (3)	C(10)	C(11)	C(6)	120.3 (7)
S(1)	C(1)	C(2)	118.4 (6)	C(2)	C(12)	C(13)	119.6 (6)
S(1)	C(1)	C(6)	116.1 (6)	C(2)	C(12)	C(17)	121.9 (6)
C(2)	C(1)	C(6)	125.5 (7)	C(13)	C(12)	C(17)	118.3 (6)
C(1)	C(2)	C(3)	118.8 (7)	C(12)	C(13)	C(14)	120.3 (6)
C(1)	C(2)	C(12)	119.1 (7)	C(13)	C(14)	C(15)	119.4 (7)
C(3)	C(2)	C(12)	121.8 (7)	C(14)	C(15)	C(16)	120.5 (6)
C(2)	C(3)	S(2)	117.2 (6)	C(15)	C(16)	C(17)	119.2 (6)
C(2)	C(3)	C(4)	126.1 (7)	C(16)	C(17)	C(12)	122.3 (6)
S(2)	C(3)	C(4)	116.6 (6)	C(4)	C(18)	C(19)	122.4 (6)
C(3)	C(4)	C(5)	119.0 (8)	C(4)	C(18)	C(23)	119.5 (6)
C(3)	C(4)	C(18)	124.5 (7)	C(19)	C(18)	C(23)	117.6 (6)
C(5)	C(4)	C(18)	116.1 (8)	C(18)	C(19)	C(20)	121.6 (7)
C(4)	C(5)	S(3)	122.8 (7)	C(19)	C(20)	C(21)	120.5 (7)
C(1)	C(6)	C(7)	121.0 (7)	C(20)	C(21)	C(22)	119.1 (7)
C(1)	C(6)	C(11)	119.3 (6)	C(21)	C(22)	C(23)	120.3 (7)
C(7)	C(6)	C(11)	119.6 (7)	C(22)	C(23)	C(18)	120.9 (7)

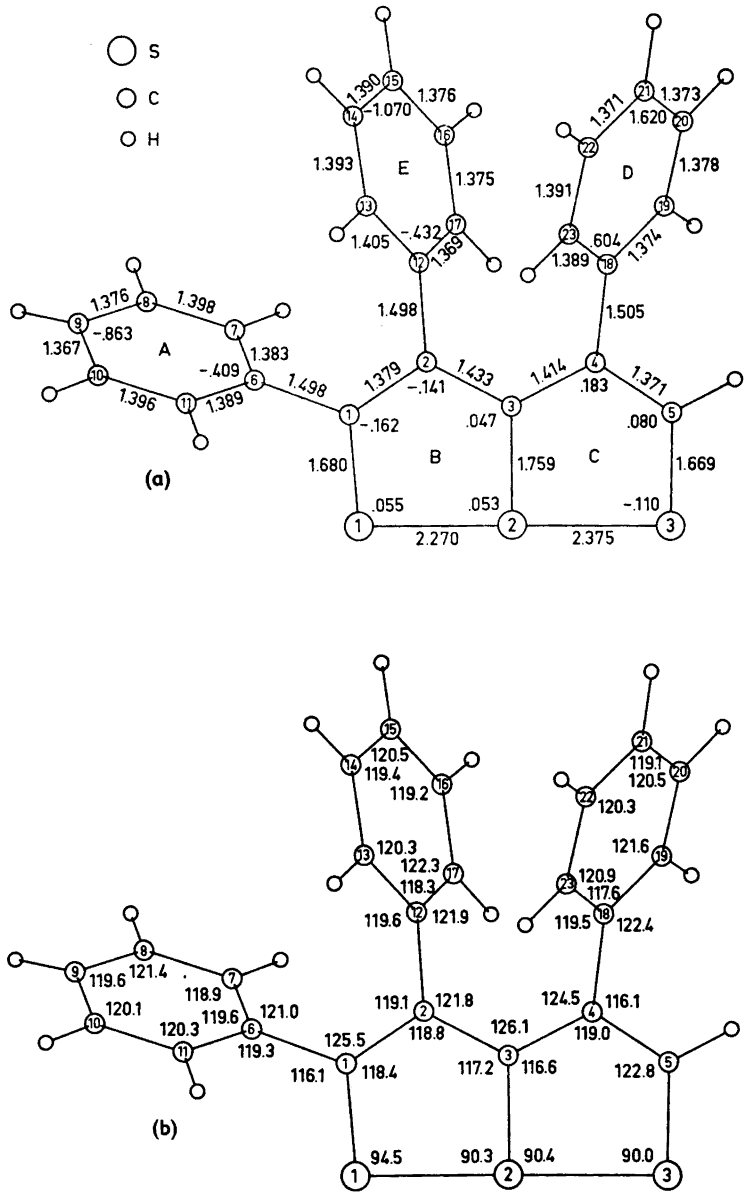

Fig. 1. (a) Bond lengths (Å) in the 2,3,4-triphenyl-thiothiophthene molecule, and atomic distances (Å) from the least squares plane of the thiothiophthene system. (b) Bond angles (°).

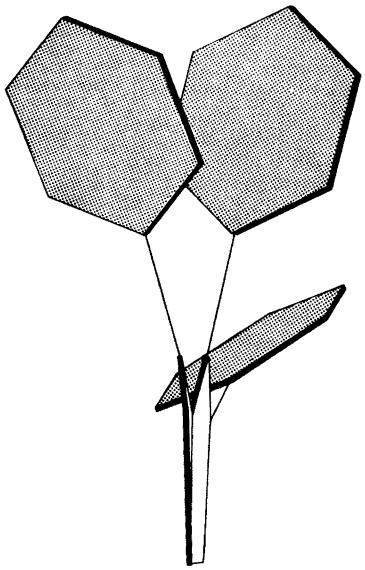

Fig. 2. The 2,3,4-triphenyl-thiothiophthene molecule as seen along the edge of the plane of the thiothiophthene system.

and the deviations in Å units from this plane are S(1) −0.029, S(2) 0.031, C(1) 0.064, C(2) 0.018, and C(3) −0.089.

Similarly, the equation for the plane of ring *C* is

$$0.59571X - 0.64218Y - 0.49804Z = -2.06945$$

with deviations S(2) −0.030, S(3) 0.027, C(3) 0.101, C(4) −0.045, and C(5) −0.046 Å.

The angle between the normal to the plane of ring *B* and the normal to the plane of ring *C* is 9.1°.

The equations for the least squares planes of the phenyl rings are

Ring A $0.36627X - 0.77236Y + 0.50907Z = -3.27650$

with deviations C(6) −0.003, C(7) 0.002, C(8) 0.005, C(9) −0.011, C(10) 0.010, and C(11) −0.003 Å.

Ring D $0.31293X + 0.69351Y + 0.64043Z = 5.36402$

with deviations C(18) 0.001, C(19) −0.002, C(20) 0.000, C(21) 0.003, C(22) −0.004, and C(23) 0.002 Å.

Ring E $0.21817X + 0.97090Y + 0.09300Z = 5.56418$

with deviations C(12) −0.002, C(13) 0.004, C(14) −0.003, C(15) 0.000, C(16) 0.002, and C(17) 0.001 Å.

The twist angle about C(1)−C(6) of phenyl group A is 53.6°. The twist angle 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).

Similarly, the twist angle of phenyl group *D* about C(4)–C(18), and that of phenyl group *E* about C(2)–C(12), are found to be 58.7 and 61.4°, respectively.

Comparison with related molecules. A comparison of the bond lengths in 2,3,4-triphenyl-thiothiophthene (I) with the bond lengths in 2,4-diphenyl-thiothiophthene (II) and the bond lengths in 3,4-diphenyl-thiothiophthene (III) is given in Fig. 3.[7,8] The bond lengths given for II include corrections for

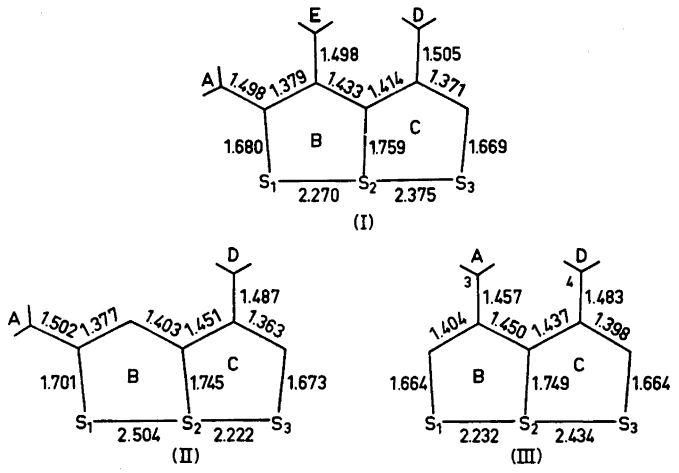

Fig. 3. Comparison of bond lengths in 2,3,4-triphenyl-thiothiophthene (I) with bond lengths in 2,4-diphenyl-thiothiophthene (II) and 3,4-diphenyl-thiothiophthene (III). The bond lengths are given in Å units.

rigid-body libration, and they are therefore somewhat different from those given in Ref. 7. The rigid-body analysis was carried out according to the method of Schomaker and Trueblood,[5] and the corrections were carried out according to Cruickshank's formula.[6] The standard deviations found for the bond lengths in II are compatible with those found for the bond lengths in I; they are ±0.003, ±0.005 to ±0.007, ±0.007 to ±0.011 Å, for S–S, S–C, and C–C, respectively. The standard deviations for the bond lengths in (III) are reported to be ±0.004 Å for S–S, ±0.013 Å for S–C, and ±0.020 Å for C–C.[8]

In (II), the atoms of rings *B* and *C* are almost in the same plane, and the phenyl groups *A* and *D* are twisted 24.7 and 51.6° about the respective connection bonds.

The thiothiophthene system in III is also effectively planar; maximum atomic deviation from the best plane is reported to be 0.02 Å. Furthermore:[8] "The two phenyl rings make angles of 70° and 74° with the best plane through the thiothiophthene ring." It is interesting to note that the carbon atoms C(3) and C(4) in III (*cf.* Fig. 3) are displaced by 0.06 Å only from the mentioned plane, in opposite directions. The deviations for the equivalent atoms in I

from the least squares plane of the thiothiophthene system are −0.432 and 0.604 Å, respectively (*cf.* Fig. 1a). Thus the spatial orientation of the 3- and 4-phenyl groups in III is different from that of the equivalent phenyl groups in I.

The sulphur−sulphur bond lengths in 2,3,4-triphenyl-thiothiophthene (I) are S(1)−S(2) = 2.270(4) Å, and S(2)−S(3) = 2.375 Å, and the lengths of the equivalent bonds in 2,4-diphenyl-thiothiophthene (II) are 2.504(3) and 2.222(3) Å, respectively (*cf.* Fig. 3). Thus, the introduction of a 3-phenyl group in (II) has caused a decrease of 0.234 Å in the S(1)−S(2) bond length, and an increase of 0.153 in the S(2)−S(3) bond length.

The difference between the S−S bond lengths in I, 0.105 Å, is less than the difference between the S−S bond lengths in III, 0.202 Å, but the sums of the S−S bond lengths in compounds I and III are nearly equal, 4.645 Å in the former, and 4.666 Å in the latter. One notes that these values are smaller than the sum of the S−S bond lengths in (II), 4.726 Å.

The average lengths of the central C−C bonds in the thiothiophthene systems of I, II, and III, are 1.424(11), 1.427(10), and 1.444(20) Å, respectively, and the average lengths of the terminal C−C bonds are 1.375(10), 1.370(10) and 1.401(20) Å, reckoned in the same order.

The length of the central C−S bond is 1.759(8) Å in I, 1.745(5) Å in II, and 1.749(13) Å in III, and the lengths of the terminal C−S bonds in the three compounds are 1.680(8) and 1.669(10) Å, 1.701(5) and 1.673(7) Å, and 1.664(13) and 1.664(13) Å, respectively.

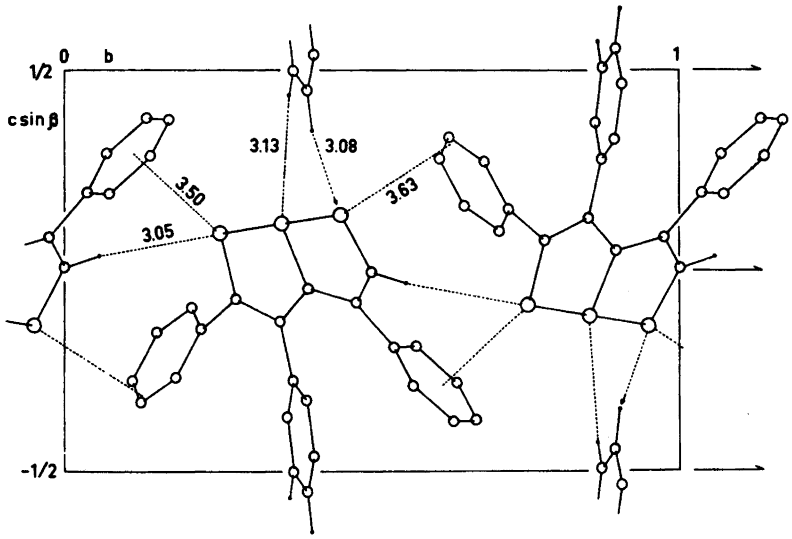

Fig. 4. The arrangement of 2,3,4-triphenyl-thiothiophthene molecules in the crystal, as seen along the *b*-axis. The small filled circles indicate hydrogen atoms, and the arrows that the atoms pointed at are situated one cell below. Atomic distances are given in Å units.

The crystal structure. The arrangement of 2,3,4-triphenyl-thiothiophthene molecules in the unit cell as seen along the *b*-axis is shown in Fig. 4. Broken lines show the way in which the closest neighbours are arranged around the sulphur atoms. One should remember that the sum of the van der Waals radii for sulphur and hydrogen is 3.05 Å, and that the sum of the van der Waals radius for sulphur and the half-thickness of an aromatic molecule is 3.55 Å.

Table 3. Atomic coordinates in fractions of corresponding cell edges. Standard deviations in parentheses, for sulphur and carbon, refer to the last digits of respective values.

Atom	*x*	*y*	*z*
S(1)	0.23923 (42)	0.25390 (24)	0.08795 (21)
S(2)	0.50602 (43)	0.35347 (24)	0.11445 (22)
S(3)	0.80171 (46)	0.44980 (24)	0.13799 (27)
C(1)	0.18622 (129)	0.28054 (56)	−0.07444 (81)
C(2)	0.29232 (121)	0.35102 (55)	−0.12933 (71)
C(3)	0.44475 (124)	0.39763 (57)	−0.04641 (76)
C(4)	0.55650 (124)	0.47446 (63)	−0.08343 (76)
C(5)	0.72736 (137)	0.50092 (62)	−0.00511 (92)
C(6)	0.03012 (114)	0.22257 (44)	−0.14884 (67)
C(7)	0.08266 (113)	0.18130 (51)	−0.26806 (71)
C(8)	−0.06509 (147)	0.12489 (55)	−0.33100 (82)
C(9)	−0.26116 (143)	0.11057 (55)	−0.27742 (97)
C(10)	−0.31140 (122)	0.15012 (65)	−0.15819 (91)
C(11)	−0.16728 (126)	0.20724 (56)	−0.09385 (73)
C(12)	0.25995 (100)	0.37160 (41)	−0.27662 (59)
C(13)	0.05645 (106)	0.39403 (47)	−0.32610 (66)
C(14)	0.01888 (124)	0.40569 (53)	−0.46443 (74)
C(15)	0.18512 (128)	0.39586 (55)	−0.55262 (66)
C(16)	0.38491 (109)	0.37431 (52)	−0.50429 (67)
C(17)	0.41838 (99)	0.36243 (48)	−0.36748 (61)
C(18)	0.48604 (108)	0.53680 (44)	−0.19417 (63)
C(19)	0.66217 (115)	0.56811 (55)	−0.28921 (74)
C(20)	0.56244 (135)	0.63361 (59)	−0.37910 (77)
C(21)	0.36304 (145)	0.66954 (55)	−0.37612 (76)
C(22)	0.22332 (119)	0.63908 (51)	−0.28301 (81)
C(23)	0.28443 (111)	0.57332 (51)	−0.19190 (69)
H(5)	0.8150	0.5565	−0.0350
H(7)	0.2270	0.1900	−0.3130
H(8)	−0.0225	0.0959	−0.4180
H(9)	−0.3500	0.0670	−0.3150
H(10)	−0.4500	0.1404	−0.1123
H(11)	−0.2050	0.2366	−0.0025
H(13)	−0.0700	0.4020	−0.2560
H(14)	−0.1350	0.4230	−0.4980
H(15)	0.1600	0.4039	−0.6550
H(16)	0.5100	0.3670	−0.5690
H(17)	0.5625	0.3462	−0.3310
H(19)	0.7750	0.5428	−0.2940
H(20)	0.6700	0.6530	−0.4530
H(21)	0.3150	0.7130	−0.4550
H(22)	0.0750	0.6718	−0.2690
H(23)	0.1850	0.5510	−0.1225

Table 4. Temperature parameters β_{ij} for sulphur and carbon. The expression used is $\exp-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})$. Standard deviations in parentheses refer to the last digits of respective values. All values are multiplied by 10^4.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{23}	β_{13}
S(1)	507 (10)	41 (1)	66 (2)	−25 (3)	−8 (4)	13 (2)
S(2)	461 (9)	41 (1)	67 (2)	12 (3)	−53 (4)	−1 (1)
S(3)	458 (11)	56 (2)	157 (4)	−3 (4)	−148 (5)	−8 (2)
C(1)	283 (29)	27 (5)	107 (11)	−1 (9)	30 (14)	−1 (6)
C(2)	264 (26)	25 (4)	79 (9)	26 (9)	−1 (12)	−9 (5)
C(3)	255 (28)	31 (4)	93 (10)	5 (9)	26 (14)	−19 (6)
C(4)	206 (26)	45 (5)	93 (10)	4 (10)	−2 (13)	−24 (6)
C(5)	310 (32)	38 (5)	163 (14)	−9 (11)	−17 (17)	−25 (7)
C(6)	310 (26)	26 (4)	88 (9)	−6 (8)	16 (12)	18 (5)
C(7)	308 (25)	32 (4)	101 (10)	6 (8)	9 (12)	−2 (5)
C(8)	427 (33)	43 (5)	129 (12)	3 (11)	−26 (16)	−9 (6)
C(9)	329 (31)	48 (5)	177 (13)	−24 (10)	−63 (17)	14 (7)
C(10)	271 (27)	67 (6)	158 (13)	−14 (11)	22 (15)	31 (7)
C(11)	321 (28)	51 (5)	124 (11)	−17 (10)	11 (14)	14 (6)
C(12)	225 (21)	17 (3)	69 (7)	−2 (7)	−6 (10)	1 (4)
C(13)	267 (24)	32 (4)	78 (8)	9 (8)	17 (11)	12 (5)
C(14)	285 (24)	45 (4)	117 (10)	14 (9)	−41 (13)	10 (6)
C(15)	408 (29)	51 (5)	57 (8)	−11 (10)	−14 (13)	8 (5)
C(16)	278 (23)	47 (5)	87 (9)	1 (9)	5 (11)	−1 (5)
C(17)	230 (21)	36 (4)	70 (8)	1 (8)	4 (10)	−3 (5)
C(18)	233 (22)	26 (4)	74 (8)	−10 (7)	−20 (11)	−11 (4)
C(19)	285 (25)	41 (4)	109 (10)	−4 (9)	40 (13)	−5 (6)
C(20)	351 (30)	54 (5)	115 (11)	−32 (11)	33 (14)	4 (6)
C(21)	432 (32)	38 (5)	112 (11)	−20 (11)	−48 (15)	3 (6)
C(22)	275 (25)	38 (5)	134 (11)	4 (9)	−27 (14)	−6 (6)
C(23)	229 (23)	30 (4)	122 (10)	−4 (8)	−1 (12)	5 (5)

For the hydrogen atoms, a temperature factor $\exp[-3(\sin^2\theta/\lambda^2)]$ was used.

Table 5. Results from the rigid-body analysis of the 2,3,4-triphenyl-thiothiophthene molecule.

	Eigenvalues	Eigenvectors (Direction cosines × 10^4 relative to a, b, and c^*, respectively)		
Librational tensor, **L**	13.73 (°)²	3923	9116	−1227
	4.79	205	1244	9920
	2.31	9197	−3914	320
Translational tensor, **T**	0.0455 Å²	8724	4781	−1013
	0.0439	−3303	4221	−8452
	0.0318	−3604	7701	5249
Symmetrized screw tensor **S**		$\begin{pmatrix} 0 & 45 & 68 \\ & 2 & 10 \\ & & -3 \end{pmatrix}$ × 10^5 rad. Å		

Centre of gravity of the molecule is at $x=0.28472$, $y=0.37963$, $z=-0.16510$.
The origin which symmetrizes **S** is at $x=0.30333$, $y=0.38364$, $z=-0.22504$.

Table 6. Observed and calculated structure factors for 2,3,4-triphenyl-thiothiophthene. The values given are ten times the absolute values. The columns are: Index, $|F_{obs}|$, $|F_{calc}|$, A_{calc}, B_{calc}. Unobserved reflections marked with asterisks.

	H= 0 K= 0				2	238	255	220-	129	9	41*	34	4-	34	-1	225	218	166-	141-	1	97	109	94	56
1	608	648	648	0	3	199	212	201-	67	10	54	52	2	52	0	417	402	349-	199-	2	72	74	16-	73-
2	69	69	69-	0	4	117	120	102-	65		H= 1 K= 2				1	129	123	119	30-	3	63	63	20-	59
3	185	192	192-	0	5	62	64	62-	14-	-10	38	31	7	30	2	232	233	178-	149-	4	70	76	72-	26
4	498	501	501-	0	6	88	93	86	36-	-9	141	131	110	72	3	221	227	220	54-	5	174	177	146-	101-
5	559	576	576-	0	7	104	102	102	12-	-8	217	224	153	164	4	413	424	415	89	6	75	71	71-	6
6	90	93	93-	0	8	97	90	86	28	-7	99	103	98	33-	5	178	187	177	59	7	56	55	23	50
7	49	47	47	0	9	80	70	55	43	-6	166	167	81	146-	6	88	89	82-	35	8	23*	13	3-	13
8	102	101	101	0		H= 0 K= 10				-5	311	315	85	304-	7	107	111	111-	5-		H= 1 K= 12			
9	39	42	42	0	1	213	227	226-	23-	-4	81	85	70-	48	8	151	151	137-	64-	-7	81	62	60-	17-
10	87	74	74	0	2	81	78	75	20-	-3	254	258	250-	65	9	160	144	135-	51-	-6	69	65	64-	12-
	H= 0 K= 1				3	175	184	182	27-	-2	484	486	374-	312	10	119	100	73-	68-	-5	80	78	48	61
1	200	203	94	180	4	276	298	298	1-	-1	792	789	398-	682		H= 1 K= 7				-4	167	176	131	117
2	398	401	154-	371-	5	160	168	139	95	0	310	295	204-	214	-9	71	66	18	64	-3	44	44	41	14
3	348	355	316-	161-	6	100	102	68	75	1	107	98	98-	4-	-8	91	78	44-	65-	-2	125	129	126	28-
4	161	153	72	135	7	43*	30	3-	30	2	609	604	588	139-	-7	68	64	38-	51-	-1	40	43	25	35-
5	176	176	7-	176	8	128	107	101-	36-	3	267	272	253	101-	-6	152	165	139-	88	0	61	70	1-	70-
6	254	264	54	259		H= 0 K= 11				4	271	270	97-	252-	-5	204	208	180-	103	1	134	139	129-	51
7	190	198	169	103	1	232	248	245	40	5	112	114	98-	59-	-4	161	159	157-	25	2	102	100	96-	27
8	126	136	136	6	2	232	250	143	205	6	39	36	22-	28	-3	61	56	56-	1	3	150	150	93-	117
9	111	111	88	68-	3	191	207	125	164	7	49	49	33-	37	-2	167	166	124	111-	4	62	64	12-	63
10	114	98	1-	98-	4	197	195	194	23-	8	44	53	9-	52	-1	158	147	146	4-	5	178*	14	10	9
	H= 0 K= 2				5	113	119	19	117-	9	18*	3	3-	1-	0	393	394	374	124-	6	90	84	51	66-
1	271	248	159-	191-	6	101	105	54-	90-	10	36	32	27-	17	1	281	279	155	232	7	46	31	9	30-
2	653	654	126-	641-	7	79	69	68-	14-		H= 1 K= 3				2	543	556	197	520		H= 1 K= 13			
3	345	349	47-	346	8	92	73	60-	41-	-10	79	63	26	57	3	191	195	170-	95	-6	50	40	26	30
4	312	312	239	200		H= 0 K= 12				-9	74	73	45	57-	4	220	232	231-	27	-5	63	59	59	1
5	364	366	233	282	1	111	126	50	116	-8	40	42	41	11-	5	64	66	54-	39	-4	118	120	80	90
6	239	243	242	17	2	78	90	88	18-	-7	80	84	80-	28	6	215	226	0	226-	-3	118	122	117	36
7	120	122	120	18-	3	74	81	31	75-	-6	130	131	41-	124-	7	191	196	78-	180-	-2	83	86	58	64-
8	109	115	31-	111-	4	121	132	6	132-	-5	162	160	88-	134-	8	63	61	28	54-	-1	131	142	77-	119-
9	131	131	11-	131-	5	89	86	1-	86-	-4	206	208	181-	103-	9	80	67	12	66	0	90	97	85-	48-
10	55	43	40-	15	6	81	85	63-	57-	-3	109	111	75-	82		H= 1 K= 8				1	105	110	109-	19-
	H= 0 K= 3				7	44*	36	28-	22	-2	515	511	132	494	-9	161	132	33-	128	2	140	146	109-	97
1	210	199	163-	113		H= 0 K= 13				-1	507	483	403	266	-8	175	171	165-	42	3	127	124	8-	124
2	618	562	295-	478	1	30*	20	18	8	0	949	925	370	848	-7	24*	34	34-	1-	4	106	106	10	105
3	59	56	50	25	2	81	82	5	82-	1	625	608	586	160	-6	130	138	48	130-	5	50	53	42	33
4	342	341	341	6-	3	73	77	59-	49-	2	591	581	357-	459-	-5	256	270	14	270-	6	44	42	42	6-
5	181	175	166	56	4	37*	33	31-	12-	3	329	319	299-	111-	-4	180	185	26	183-		H= 1 K= 14			
6	157	160	85	136-	5	36*	31	13-	28	4	275	268	250-	98-	-3	251	266	256	73	-5	41	41	34	21
7	33	37	3-	36-	6	70	63	63-	2	5	198	193	149-	121-	-2	179	191	105	160	-4	59	51	15	48-
8	138	143	111-	90-		H= 0 K= 14				6	219	228	126-	190	-1	62	68	64	24	-3	81	73	60-	41-
9	99	102	72-	72-	1	38	46	38	27-	7	133	139	28-	136	0	188	192	141-	131	-2	57	52	42-	30-
10	85	76	59-	48	2	41	38	24-	29-	8	162	165	89-	139	1	227	224	212-	72-	-1	35	36	36-	4-
	H= 0 K= 4				3	51	51	40-	32-	9	46	47	34-	33	2	280	285	275-	75-	0	80	79	79-	4
0	757	737	671-	306-	4	60	57	56-	11	10	100	92	65	64-	3	117	113	97-	58-	1	167	171	84	149
1	756	739	734	86	5	59	53	3	53		H= 1 K= 4				4	125	131	87	98	2	83	83	82	10
2	323	315	288	127-	6	94	75	42	62	-10	36	34	32-	11-	5	95	102	71	73-	3	53	46	45	12-
3	125	123	110-	54		H= 0 K= 15				-9	90	87	86-	9-	6	44	41	41	6	4	98	90	81	39-
4	76	71	22-	68	1	39*	35	19	29	-8	93	94	89	31	7	23*	18	13	12	5	128	118	111	39-
5	85	80	76	24	2	115	139	105	29-	-7	143	147	99-	108-	8	50	45	25-	37		H= 1 K= 15			
6	138	141	136-	37-	3	137	125	116	46-	-6	129	128	51-	118-	9	75	58	43	39	-4	51	44	43	11-
7	127	135	120-	62-	4	21*	19	18-	5-	-5	67	62	2	62		H= 1 K= 9				-3	53	45	44-	9-
8	119	121	119-	21-	5	23*	15	14-	5	-4	106	103	4	103	-9	34	23	8-	22	-2	85	78	73-	27-
9	57	52	3-	52		H= 0 K= 16				-3	76	79	22-	75	-8	34	31	7	30	-1	15*	23	18-	15
10	130	110	47	99	1	40*	32	31-	9	-2	315	309	292	101	-7	120	118	110	44-	0	30	24	19	15
	H= 0 K= 5				2	37*	33	31-	12	-1	424	404	382	132	-6	84	84	74	39	1	30	30	18	24
1	458	447	408-	182-	3	81	69	69	1	0	564	545	321	440-	-5	162	167	106	129	2	162	162	99	128
2	249	242	238-	42-		H= 1 K= 0				1	204	198	77	182-	-4	96	97	96	12	3	109	101	90	46
3	140	132	105	81	-10	146	130	130-	0	2	299	288	25-	287	-3	123	127	27-	125	4	47	41	25	32-
4	70	69	17-	67-	-9	120	119	119-	0	3	351	342	235-	248	-2	182	192	148-	122		H= 1 K= 16			
5	70	66	65	13	-8	248	252	252-	0	4	489	496	495-	30-	-1	83	86	75-	41-	-3	86	75	74	16
6	36	30	9	29	-7	62	68	68	0	5	228	237	138-	193	0	236	246	115-	218-	-2	36	30	28	12-
7	65	65	65-	2-	-6	63	57	57	0	6	75	77	50-	59	1	71	68	68-	3	-1	74	58	55	17
8	49	49	17-	45-	-5	142	149	149	0	7	133	147	130	69-	2	68	71	14	69-	0	84	75	22	72
9	53	48	40	27-	-4	340	344	344	0	8	250	260	228	125-	3	94	102	89	50-	1	57	47	43	20
10	72	62	61	12	-3	240	242	242	0	9	140	127	69	107-	4	39	48	48	1	2	73	61	60-	11
	H= 0 K= 6				-2	196	193	193-	0	10	103	90	84	32-	5	160	172	6-	171		H= 2 K= 0			
1	92	92	9-	92-	-1	453	458	458-	0		H= 1 K= 5				6	86	90	61	67	-10	21*	4	4-	0
2	98	98	92-	36	0	370	346	346-	0	-10	43*	33	7-	32-	7	56	54	53	10-	-9	51	46	46	0
3	100	97	59	77-	1	27*	15	15	0	-9	24*	19	10	16-	8	41*	34	33-	8	-8	24*	17	17-	0
4	126	126	88-	90	2	54	56	56-	0	-8	54	55	22	51-	9	19*	8	2	8-	-7	54	59	59-	0
5	87	96	89	35-	3	138	135	135-	0	-7	57	60	31-	51		H= 1 K= 10				-6	206	218	218	0
6	57	58	51	27	4	50	45	45	0	-6	52	53	49	20-	-8	131	103	92	47	-5	82	86	86	0
7	85	89	89	1-	5	253	257	257-	0	-5	54	57	57	1-	-7	18*	12	6	11	-4	139	140	140-	0
8	184	187	184	29	6	46	48	48	0	-4	174	174	100	143	-6	115	115	72-	89	-3	164	168	168	0
9	44*	36	20	30	7	101	106	106	0	-3	55	56	29	48	-5	139	145	138-	47-	-2	74	73	73-	0
10	37*	23	9	21	8	55	53	53	0	-2	49	45	44	11	-4	183	192	190-	26-	-1	485	466	466-	0
	H= 0 K= 7				9	132	135	135	0	-1	41*	29	19	21	-3	86	98	92-	35-	0	230	226	226-	0
1	219	212	198-	75	10	61	53	53-	0	0	281	271	29	270	-2	42	48	47-	10	1	149	144	144-	0
2	131	135	86	104-		H= 1 K= 1				1	278	271	155-	222-	-1	95	100	41	91	2	188	193	193	0
3	122	119	118	12	-10	93	79	55-	57-	2	225	226	145-	172-	0	242	257	229	115	3	87	87	87-	0
4	57	59	59	1-	-9	22	18	18-	3-	3	454	449	445-	60	1	135	141	99-	101-	4	33	35	35-	0
5	161	168	149	78-	-8	120	119	67	98	4	270	266	155-	217	2	70	74	24-	70	5	45	52	52	0
6	24*	20	11-	16	-7	189	184	163	86	5	150	159	156	34	3	22*	17	16-	3	6	71	72	72-	0
7	111	114	107-	39-	-6	327	335	289	169	6	303	317	277	154	4	75	76	1-	76-	7	93	89	89-	0
8	133	126	103-	72	-5	268	274	272	29	7	263	276	262	86	5	61	64	12	63-	8	108	116	116	0
9	130	110	96-	54	-4	41	43	3-	43-	8	157	161	160	21	6	75	76	72	24-	9	87	86	86	0
	H= 0 K= 8				-3	168	168	165-	31	9	41*	28	28-	4-	7	11*	4	0	4	10	32*	9	9	0
1	236	240	37	237-	-2	320	324	306-	109	10	150	116	115-	14-	8	44*	32	31-	11		H= 2 K= 1			
2	178	181	174	50	-1	746	786	513-	595		H= 1 K= 6					H= 1 K= 11				-10	91	70	69	10
3	196	201	136	148	0	391	366	276-	241-	-10	54	40	30	27-	-8	29*	18	2	18	-9	66	56	31	46-
4	209	216	45-	211	1	328	318	317	23	-9	53	45	29-	35-	-7	121	110	94-	56-	-8	120	126	96	81
5	190	200	157-	124	2	283	280	197-	199	-8	51	48	0	48-	-6	139	140	120-	72-	-7	118	132	91-	95-
6	75	78	77-	12	3	109	108	99	41-	-7	109	110	104	35	-5	70	71	70-	11	-6	15*	3	3-	0
7	122	118	76-	90-	4	123	125	56	112-	-6	93	93	62	69-	-4	120	125	18-	124	-5	104	108	98-	56
8	136	130	25	128-	5	118	112	107	34	-5	74	76	18-	74	-3	104	113	79	81	-4	49	51	40-	31
9	80	62	41	46-	6	108	114	30	110-	-4	67	69	58	36	-2	91	97	78	56	-3	295	297	42-	294-
	H= 0 K= 9				7	51	42	24	35	-3	169	173	36	170	-1	90	94	94	8-	-2	128	124	123	17
1	122	126	125-	15	8	28*	19	15-	12	-2	140	142	49-	133	0	60	68	23	64	-1	127	117	117-	2

Table 6. Continued.

0	75	75	8	75	2	223	216	46	211-	2	70	79	79-	2-	-4	85	85	82	22-	-8	87	67	50-	45
1	194	190	36	187	3	346	375	126-	353-	3	63	64	55-	33-	-3	202	202	130-	155	-7	51	41	38	16-
2	30	24	21-	12-	4	215	217	193-	101-	4	55	56	52	20-	-2	174	176	70-	161	-6	79	75	40-	63-
3	127	130	87	97-	5	78	74	47-	56-	5	71	71	26-	66	-1	234	233	226-	57-	-5	69	66	41-	52-
4	144	155	38	150-	6	138	147	141-	42	6	28*	20	20-	4-	0	340	338	307-	143-	-4	48	49	28-	40-
5	38	37	28	24	7	120	120	56-	106			H=	2 K=	13	1	200	195	82	177-	-3	62	61	58-	20-
6	130	138	96	100	8	64	61	4	61	-6	32*	21	21	0	2	57	56	34	44	-2	154	159	148-	60
7	129	135	83	106	9	36	27	24	8	-5	47	46	43-	8-	3	162	159	141	74-	-1	94	92	31-	87
8	90	90	3	90		H=	2 K=	7		-4	85	84	56-	63-	4	43	43	28	56-	0	129	130	130	0
9	62	61	54-	28-	-9	20*	7	4	6-	-3	142	135	127-	46	5	45	48	44	19	1	271	285	277	65-
10	106	76	59-	48-	-8	138	128	126-	18-	-2	112	107	41-	99	6	106	117	99-	63	2	195	194	136	139-
	H=	2 K=	2		-7	136	130	73	107-	-1	76	82	5-	81	7	73	69	37	58-	3	132	134	50	124-
-10	71	57	57	8-	-6	196	202	8	202-	0	124	121	120-	19-	8	51	54	19-	51	4	92	89	16	87-
-9	89	80	16	78-	-5	59	57	15-	55-	1	152	161	103	124-	9	85	66	58	32	5	174	178	165-	67
-8	105	103	74-	72-	-4	89	96	24-	93	2	152	151	80	128-			H=	3 K=	4	6	132	129	129-	0
-7	141	148	130-	69	-3	105	113	82	77	3	120	114	98	58-	-9	88	75	74	9-	7	139	119	88-	81
-6	132	132	129-	30	-2	170	169	71	153	4	136	131	129	21-	-8	63	55	52	18		H=		3 K=	10
-5	86	95	88-	37	-1	147	150	111	101	5	152	138	119	69	-7	133	129	117	53	-7	57	51	44-	24-
-4	98	101	100-	11	0	71	71	36-	61	6	75	60	33-	50	-6	29*	33	27	20-	-6	52	46	43	16-
-3	77	75	71-	25	1	157	163	2-	163-			H=	2 K=	14	-5	19*	17	11	12	-5	82	78	26-	73-
-2	81	82	82	3	2	113	111	54-	97-	-5	114	88	85-	21	-4	67	68	61-	29	-4	40	42	36-	22-
-1	195	192	7-	192-	3	248	249	238-	73-	-4	107	97	97-	4-	-3	47	46	32	34-	-3	39	36	26	25
0	266	253	85	238-	4	114	119	88-	80-	-3	42	45	39-	18-	-2	76	70	33-	62-	-2	34	33	26	20-
1	170	162	158	34	5	80	85	81-	26-	-2	68	58	46	35-	-1	109	103	58-	85	-1	54	55	4-	55
2	233	227	197	112-	6	165	182	17-	182	-1	34	29	29	3-	0	92	88	88-	12	0	106	104	104	7-
3	198	203	192	65-	7	58	65	29	58	0	68	56	10	55-	1	95	87	85	16-	1	116	123	108	61
4	46	52	52	2	8	126	112	108	28	1	27*	15	13	8	2	90	88	84	28	2	123	131	38-	125
5	256	272	191-	194	9	173	135	125	50-	2	70	79	28	74	3	59	57	30	48	3	127	128	123-	38-
6	240	246	246-	12		H=	2 K=	8		3	39	38	26-	27	4	34	30	28	13-	4	108	99	86-	48-
7	245	254	253-	17	-9	70	55	44	33-	4	126	113	112-	6-	5	81	86	27	81-	5	81	68	68-	1
8	149	144	142-	25	-8	74	73	66	3C-	5	56	51	50-	7-	6	73	74	74-	3-	6	73	68	38-	56-
9	43*	38	4	38-	-7	19*	12	9	8-			H=	2 K=	15	7	44	40	23	33	7	110	87	79	36
10	137	109	75	78-	-6	86	88	53	7C	-3	168	146	142	31-	8	28*	29	29	1-		H=		3 K=	11
	H=	2 K=	3		-5	113	119	62	102	-2	101	89	79	42	9	20*	9	0	9-	-6	34*	24	10-	22-
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-9	136	126	114-	53	-3	84	94	85-	40	0	36*	20	20-	3-	-9	25*	20	19-	4	-4	89	92	84	38
-8	64	61	29	54-	-2	53	52	13	50-	1	141	123	82	92-	-8	33*	26	17-	19-	-3	84	85	84	11
-7	168	172	146-	91	-1	104	98	34	92-	2	140	128	127-	15-	-7	171	167	167-	15-	-2	87	85	59	61
-6	140	148	40-	143	0	25*	16	10-	12-	3	97	83	80-	23-	-6	154	154	149-	37-	-1	56	57	17-	54-
-5	88	88	84-	26	1	138	140	126-	42-			H=	2 K=	16	-5	67	75	36-	66-	0	58	65	43-	49-
-4	91	99	37	91-	2	76	77	35-	68-	-1	134	106	69-	81-	-4	129	133	69	114-	1	66	68	45-	51-
-3	71	75	14	74-	3	90	91	90-	15-	0	159	132	106-	78-	-3	98	105	105	3-	2	220	223	179-	133-
-2	351	355	146	323-	4	228	242	206-	127	1	84	62	57-	24-	-2	161	166	141	88	3	137	144	105-	99-
-1	248	239	44	234-	5	94	100	55	84			H=	3 K=	0	-1	105	106	75	74	4	49	51	20-	48-
0	269	259	240	96-	6	198	207	192	79	-10	33*	22	22-	0	0	86	82	72	39	5	99	91	57	71
1	357	347	29	346	7	31	22	21	7-	-9	60	53	53-	0	1	84	80	59-	54-	6	139	129	74	105
2	378	372	152-	339	8	77	67	30	60-	-8	70	63	63	0	2	83	75	58	48		H=		3 K=	12
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5	148	153	99-	117-	-8	122	103	69	53	-5	45	47	47	0	5	108	107	79-	72-	-4	109	103	96-	38
6	202	212	42	207-	-7	41	42	25	34	-4	98	99	99	0	6	80	78	45-	63	-3	115	114	66-	93
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9	89	77	23	74	-4	141	151	142-	53	-1	277	287	287-	0	9	29*	13	6	12	0	133	136	86	107-
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	H=	2 K=	4		-2	48	52	6-	52	1	341	350	350-	0	-9	111	88	62-	63-	2	26*	24	21	11-
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-7	137	144	139	36	2	52	56	36-	43-	5	225	234	234	0	-5	88	88	88	4	6	46*	34	33-	7-
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-2	396	399	379-	124	7	50	43	10-	42-			H=	3 K=	1	0	71	68	22-	65	-2	91	84	70	47-
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1	171	167	160-	49-	-8	48	39	22-	32-	-7	81	88	25-	84	3	132	136	91-	101	1	65	68	23-	64
2	350	344	298-	171-	-7	23*	19	3	19	-6	28*	24	10	22	4	102	104	104-	11	2	30*	33	16-	29
3	110	112	42	105-	-6	42*	40	34-	21-	-5	25*	21	7	19-	5	117	119	8	119-	3	73	69	55-	42-
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-4	88	82	61	12-		H=	2 K=	11			H=	3 K=	2		2	171	171	169	25		H=	4 K=		0
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-2	617	623	568-	256-	-6	55	51	42	28	-8	74	65	50-	42-	4	28*	19	15	12	-8	34*	26	26	0
-1	320	311	257-	174-	-5	89	91	80	44-	-7	143	145	106-	99	5	68	69	5-	69-	-7	68	59	59	0
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2	423	422	421	36-	-2	62	60	57	16-	-4	140	142	110	90-	8	48*	37	31	21-	-4	60	61	61-	0
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6	205	221	219-	32	2	22*	13	0	13-	0	245	241	32-	239	-6	48*	40	38	11	0	299	293	293	0
7	64	74	74-	2-	3	103	108	41	100	1	234	225	170-	147	-5	72	73	14	72-	1	214	224	224	0
8	42*	36	15-	33-	4	88	97	14-	96	2	199	204	121-	164	-4	80	82	17-	81-	2	101	103	103	0
9	44*	38	22	30-	5	55	51	24-	45	3	155	166	7	166-	-3	157	165	151-	68-	3	101	111	111-	0
	H=	2 K=	6		6	165	166	34-	162-	4	235	241	26	240-	-2	125	129	123-	41-	4	82	84	84-	0
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-8	26*	25	1	25		H=	2 K=	12		6	124	116	27-	113-	0	44	39	11	37	6	42	46	46-	0
-7	97	99	98-	16-	-7	80	64	63	11-	7	21*	19	8	11-	1	119	114	66	93	7	70	63	63	0
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-4	163	173	132-	112-	-4	48	49	34	35			H=	3 K=	3	4	95	96	17	95-	-9	91	65	65-	3-
-3	83	84	75-	40	-3	35	37	35	11-	-9	17*	9	8	2-	5	95	95	30-	90-	-8	113	93	93-	4
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0	351	354	200	291	0	59	64	53-	37-	-6	96	100	98	20-	8	99	78	23-	74	-5	56	62	62	2-
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Table 6. Continued.

-3	170	179	147	104	-5	51	57	31-	48	-1	76	63	10-	63	3	160	152	107-	108	-3	108	115	115-	0
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1	99	109	94-	55-	-1	52	62	28	56-	3	73	57	6-	57-	7	105	78	49	61-	1	49	51	51	0
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7	36*	23	3	23	5	48	42	10-	41-	0	28*	19	8	17	-3	103	99	76	63	-5	25*	18	16	8
8	74	60	38	46-	6	128	126	28	122-	1	63	50	43	25-	-2	39	33	27-	19-	-4	29*	20	1-	20
	H=	4	K=	2	7	54	37	27	26	2	40*	25	13-	21-	-1	68	65	42-	49	-3	21*	2	0	2
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-8	57	49	19	45-	-7	66	54	53	11-	-7	101	90	90-	0	1	45	33	23-	24	-1	81	81	18	79
-7	33*	23	23-	4-	-6	78	75	28	70	-6	19*	16	16	0	2	96	82	80-	17-	0	42	32	30-	13
-6	35*	41	25	32-	-5	44	43	21	37	-5	40	38	38-	0	3	79	65	51-	40	1	37	32	30-	10
-5	95	100	71	70	-4	47	54	32-	43	-4	30*	22	22	0	4	25	18	17-	8	2	85	80	24	76-
-4	76	78	46	63	-3	93	103	50-	90	-3	96	103	103	0	5	124	120	118	20-	3	117	109	55	94-
-3	49	53	10	52-	-2	49	53	49-	20	-2	81	92	92	0	6	101	86	86	6	4	63	60	17-	57-
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-5	96	104	14-	103	-2	126	132	58-	119	-2	126	134	49-	125	-6	46*	33	33-	2	-4	30*	19	13	14
-4	65	74	72-	15	-1	89	91	86	31-	-1	41	35	16-	31	-5	83	74	33-	67	-3	54	51	50	9-
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	H=	4	K=	4	-3	143	140	117	76-	-4	27	20	18-	8-	-4	56	43	34	26-	-2	108	97	96-	16
-8	31*	20	16-	12-	-2	93	96	44	85	-3	71	82	28-	77	-3	29*	23	22	7	-1	112	103	45-	92
-7	81	74	65-	36-	-1	90	101	1-	101	-2	90	99	6-	99-	-2	74	59	45	52	0	29*	23	23	2
-6	19*	20	2	20-	0	104	110	19-	109	-1	93	98	36-	91	-1	31*	26	26-	4	1	80	72	37-	61-
-5	62	65	30	58	1	176	179	136-	115	0	42	48	1	48-	0	31*	26	7-	26	2	59	53	11-	52-
-4	58	63	63-	8	2	78	77	65-	42	1	20	9	9-	1	1	72	72	71-	11	3	87	78	78	1
-3	30	31	26-	23-	3	62	62	45-	43-	2	67	65	24-	61	2	45	38	28-	26	4	53	48	29	38
-2	95	101	41	93-	4	61	53	27-	45-	3	31	31	25-	18	3	24*	19	6-	18-		H=	6	K=	5
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1	67	70	42	57		H=	4	K=	10	6	68	63	31	54-		H=	5	K=	9	-2	30*	22	16	16
2	154	153	127	85	-6	107	88	87	11	7	99	83	81	15	-5	32*	15	8-	13	-1	99	90	42-	80-
3	33	33	13	30-	-5	112	105	103	20		H=	5	K=	3	-4	26*	5	4	3	0	55	46	44-	13-
4	106	106	55	90-	-4	113	110	107	23	-7	15*	3	1	3-	-3	51	49	10-	47-	1	49	41	31	28-
5	109	112	74	84-	-3	82	82	62	53-	-6	75	73	72-	10	-2	35	28	27	8	2	46	30	23	19-
6	99	98	61	77-	-2	20*	18	17	8-	-5	65	66	48-	45-	-1	31*	25	7	24	3	41	32	0	32-
7	107	110	106-	29-	-1	151	150	149-	9	-4	29*	29	8	28	0	22*	11	1-	11-	4	43*	30	30	2-
8	106	83	40-	73	0	97	99	90-	41	-3	81	81	15-	67	1	46	37	2-	37		H=	6	K=	6
	H=	4	K=	5	1	83	84	34-	76-	-2	33	29	11-	27	2	62	53	4	53	-4	65	41	37-	17-
-8	57	45	45-	4-	2	23*	18	17	6-	-1	129	137	83	110	3	50	42	6-	42-	-3	31*	19	14-	13-
-7	33	34	23	25	3	94	85	75	41	0	72	68	65	19	4	41	38	22	32-	-2	59	41	4-	41
-6	18*	16	3	16	4	67	55	55	1	1	84	85	8	85-		H=	5	K=	10	-1	70	68	10	67
-5	30	34	18-	29-	5	32*	23	22	3-	2	64	64	32-	56-	-4	67	54	53-	12	0	31*	20	11	17
-4	75	76	60	47-	6	50	35	6-	35	3	61	55	48-	26	-3	137	119	106-	55-	1	80	67	51	43-
-3	42	39	30	25		H=	4	K=	11	4	50	54	36-	39	-2	79	75	1	75-	2	61	43	10-	41-
-2	69	72	25	67-	-5	35*	30	7-	29	5	39	29	1-	29	-1	65	34	9	32	3	64	44	16-	41-
-1	46	38	30	23	-4	28*	15	9-	11	6	53	42	30	30	0	34*	23	18	15-		H=	6	K=	7
0	123	127	86-	93	-3	87	75	28-	69-	7	58	48	9-	47	1	36*	24	15	18-	-3	24*	11	5	10
1	39	40	28-	29	-2	62	61	25-	55-		H=	5	K=	4	2	47	37	33	15	-2	55	43	42	12
2	52	59	40-	43-	-1	70	60	58-	14	-7	28	25	23	9	3	39*	31	8	30	-1	43	37	4-	37
3	47	47	46	12-	0	38	31	9-	29	-6	77	65	51-	41-		H=	5	K=	11	0	67	51	25	45-
4	119	120	119	16	1	61	53	29	44	-5	18*	9	9-	0	-2	74	57	0	57-	1	61	47	34-	33-
5	57	53	48-	22-	2	102	91	71	58	-4	67	59	7-	59	-1	27*	20	5	19-	2	58	41	38-	15
6	135	133	129-	34	3	73	64	37	52	-3	94	92	4	91	0	34*	18	18	4	3	81	60	58-	14-
7	71	61	59-	14-	4	63	50	41	29-	-2	25*	16	15	6	1	39*	27	27	1-		H=	6	K=	8
8	49	37	36-	7	5	52	41	14	38-	-1	177	183	179	40-	2	65	45	45	4	-2	41*	26	11	23
	H=	4	K=	6		H=	4	K=	12	0	99	97	50	82-		H=	6	K=	0	-1	56	38	16-	35-
-8	26*	21	8	20-	-4	46	39	23-	32-	1	84	88	68	55-	-5	36	40	40	0	0	79	57	57-	9
-7	105	95	95	8-	-3	35*	20	13-	15	2	77	79	78-	7-	-4	46	43	43-	0	1	31*	12	4	11-
-6	89	92	21	90	-2	75	69	66	20-															

EXPERIMENTAL

The unit cell dimensions for crystals of 2,3,4-triphenyl-thiothiophthene were determined by measuring the 2θ settings for 21 reflections on the diffractometer, with Cu$K\beta$ radiation ($\lambda = 1.39217$ Å). From a least squares treatment, the cell dimensions are $a = 6.296(6)$ Å, $b = 15.134(6)$ Å, $c = 9.862(4)$ Å, and $\beta = 91.52(5)°$. Two molecules per unit cell give a calculated density of 1.373 g/cm³, as compared with the density 1.37 – 1.40 g/cm³ found by flotation.

The intensities were reduced to the structure amplitudes by means of an IBM 7090 program.[9] A small crystal, with dimensions $0.15 \times 0.10 \times 0.07$ mm in the three axial directions, was used for the data collection. Absorption corrections were not applied.

The scattering factors used for sulphur and carbon in the structure factor calculations were those given by Cromer and Waber.[10] For hydrogen, the scattering factor curve given by Stewart *et al.*[11] was used.

All the hydrogen atoms in the molecule are bonded to sp^2-hybridized carbon, and their positions could therefore be estimated by assuming a C–H distance of 1.05 Å, and C–C–H angles of 120°. The hydrogen positions were not refined, and constant isotropic temperature factors $\exp-[3(\sin^2\theta/\lambda^2)]$ were applied to them.

The final atomic coordinates are listed in Table 3, and the temperature parameters in Table 4. The final list of structure factors is given in Table 6.

An analysis of the thermal parameters of the S and C atoms, assuming the whole molecule a rigid body, was carried out according to the method of Schomaker and Trueblood.[5] The rigid-body tensors arrived at are given in Table 5. The origin which symmetrizes S lies about 0.60 Å from the centre of gravity of the molecule, displaced along the three crystal axes by 0.12, 0.06, and −0.59 Å, respectively. The r.m.s. difference between observed and calculated U_{ij}'s is 0.0106 Å^2.

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