Tabelle 4. *Geometrie der Wasserstoffbrücken*

	···O in Lage	O···O	∢ am H	H···O
O(1)–H(1)····O(3)	$x, \frac{1}{2}-y, \frac{1}{2}+z$	2,580 (5) Å	153 (6)°	1,91 (5) Å
O(4)–H(41)···O(2)	$-x, \frac{1}{2}+y, \frac{1}{2}-z$	2,592 (4)	162 (4)	1,68 (5)
O(4)–H(42)···O(2)	$\frac{1}{2}-x, \frac{1}{2}-y, z$	2,557 (4)	163 (4)	1,65 (4)
O(4)–H(43)···O(3)	$\frac{1}{2}+x, 1-y, \frac{1}{2}-z$	2,538 (4)	176 (7)	1,51 (7)

mit der längsten Bindung zum Phosphoratom ein Wasserstoffatom gebunden. Die drei anderen Wasserstoffatome der asymmetrischen Einheit bilden normale Bindungsabstände und Winkel mit dem zusätzlichen Sauerstoffatom O(4), wodurch diese Konfiguration sich als Oxoniumion H_3O^+ zu erkennen gibt.

(3) durch die Wasserstoffbrücken (Tabelle 4). Wie im Ammoniumsalz ist die Wasserstoffbrücke O(1)–H(1)···O(3) der einzige kurze intermolekulare O····O-Kontakt zwischen Phosphor-Sauerstoffatomen. Dies ist eine weitere Bestätigung dafür, dass das Anion $H_2P_2O_6^{2-}$ auch in der hier untersuchten Substanz vorliegt. Schliesslich ist auch die auffallende Kürze der drei von O(4) ausgehenden Wasserstoffbrücken O–H···O ein Hinweis auf entgegengesetzte Ladungen der Brückenpartner und damit auf die ionische Natur der Substanz.

Der grosse Unterschied zwischen den beiden O–P–OH-Bindungswinkeln (Tabelle 3: 113,4 und 106,6°), der auch beim Ammoniumsalz beobachtet wurde (113,3 und 106,2°), ist überraschend. Seine Ursache muss intermolekularer Natur sein, konnte aber nicht eindeutig erkannt werden. Die übrigen Bindungswinkel am Phosphoratom sind mit dem Anion $H_2P_2O_6^{2-}$ [H gebunden an O(1)] gut vereinbar.

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The Crystal Structure of 2,7-Dimethylthianthrene*

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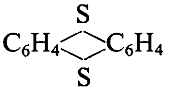
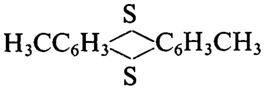
The crystal structure of 2,7-dimethylthianthrene has been determined from three-dimensional X-ray diffractometer data. The compound crystallizes with four molecules in an orthorhombic unit cell of symmetry $P2_12_12_1$ and parameters $a=8\cdot175$ (2), $b=11\cdot351$ (2), and $c=13\cdot197$ (2) Å. The structure was solved by the heavy-atom technique and was refined by the full-matrix least-squares method to $R(F)=3\cdot1\%$ based on 1282 independent intensity data. The molecule possesses an idealized C_2 symmetry, with all the nonhydrogen atoms approximately in two planes that intersect along the S–S line to form a folded configuration with a dihedral angle of 131·1°.

Introduction

The crystal and molecular structure of thianthrene  $C_6H_4\overset{S}{\underset{S}{<\!>}}C_6H_4$ has been determined previously by X-ray structure analyses (Lynton & Cox, 1956; Rowe & Post, 1958). Because of the tendency for the sulfur atoms to retain their tetrahedral valency angles, thianthrene possesses a configuration folded along the S–S line. Structural analysis of similar 2,7-dimethylthianthrene  $H_3CC_6H_3\overset{S}{\underset{S}{<\!>}}C_6H_3CH_3$ (known to be effective on parasitic skin diseases) by the single-crystal

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X-ray diffraction method was carried out to provide more accurate stereochemical information (including those of all the hydrogen atoms) on the nature of this heterocyclic system, and to provide a comparison of its molecular architecture, S–C_{sp2} distances in particular, with those of closely related compounds.

Experimental

Colorless crystals of 2,7-dimethylthianthrene were isolated from a scabies-treating medicine, SCABOL (product of Daiichi Seiyaku Co. Ltd. Tokyo, Japan), and were recrystallized from an n-hexane solution. The crystal system and approximate cell parameters were determined from Weissenberg and precession photographs. Eight strong reflections in the 2θ range of 85 to 100° were centered with an Oak Ridge computer-controlled diffractometer (Busing, Ellison, Levy, King & Roseberry, 1968) using Cu $K\alpha$ ($\lambda = 1{\cdot}54178$ Å) radiation. Cell parameters were refined by the least-squares method. Density was determined by flotation in a mixture of 1,2-dichloroethane and dichloromethane. The observed systematic absences of $h00$ for h odd, $0k0$ for k odd, and $00l$ for l odd are uniquely characteristic of the space group $P2_12_12_1$ (D_2^4, No. 19), which was later confirmed by the successful refinement of the structure. Crystallographic data are as follows:

$a = 8{\cdot}175\ (2)$ Å $b = 11{\cdot}351\ (2)$ $c = 13{\cdot}197\ (2)$
$V = 1224{\cdot}6$ Å^3 $F(000) = 512$ $D_{\text{obs}} = 1{\cdot}30\ (2)$ g.cm^{-3}
$D_{\text{calc}} = 1{\cdot}326$ g.cm^{-3} $Z = 4$.

Intensity data were collected from a rectangular rod shaped crystal of dimensions $0{\cdot}18 \times 0{\cdot}20 \times 0{\cdot}58$ mm; the longest dimension, corresponding to the **a** direction, was nearly parallel to the φ axis of the diffractometer. The intensities of 1292 independent reflections with $2\theta \leq 135°$ (equivalent to a minimum crystal spacing of 0·850 Å) were measured with the θ–2θ scan technique. Of these, 1282 reflections had nonzero intensities and were subsequently utilized throughout the structural analysis. Absorption corrections calculated by the method of Busing & Levy (1957) were applied using a linear absorption coefficient of 35·2 cm^{-1}.

Solution and refinement of the structure

Trial positional parameters for the two independent sulfur atoms were obtained from inspection of a three-dimensional Patterson map (program *FORDAPER* of A. Zalkin modified by G. Brunton). The carbon atoms in an asymmetric unit were located in a three-dimensional Fourier synthesis phased on the atomic parameters of the sulfur atoms. This model was refined by the full-matrix least-squares program *ORFLS* (Busing, Martin & Levy, 1962) in which the minimized function was $\sum w(F_o^2 - F_c^2)^2$ with weights, w, equal to the reciprocals of the variances estimated from the empirical equation:

Table 1. *Positional and anisotropic thermal parameters* (10^5) *for nonhydrogen atoms*

The form of the anisotropic temperature factor is $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$.
Standard deviations of the last significant figures are given in parentheses here and in Table 2.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S(1)	0·41513 (11)	0·42264 (5)	0·00592 (5)	2256 (14)	674 (5)	547 (4)	42 (7)	69 (6)	−25 (4)
S(2)	0·20405 (9)	0·61009 (7)	0·13431 (5)	1336 (10)	1186 (7)	562 (4)	198 (7)	103 (5)	−3 (4)
C(1)	0·13879 (33)	0·43924 (22)	0·45427 (18)	1440 (40)	754 (19)	538 (13)	−170 (23)	61 (19)	−2 (14)
C(2)	0·09287 (36)	0·41288 (25)	0·35555 (19)	1600 (42)	878 (21)	529 (13)	−190 (28)	17 (22)	60 (15)
C(3)	0·14259 (36)	0·30898 (27)	0·30861 (20)	1747 (46)	1020 (26)	552 (15)	−277 (30)	221 (30)	−103 (16)
C(4)	0·09123 (61)	0·28170 (39)	0·20214 (25)	2670 (75)	1342 (36)	609 (19)	−537 (50)	274 (35)	−192 (22)
C(5)	0·23830 (44)	0·23123 (29)	0·36481 (25)	2293 (62)	969 (26)	765 (20)	90 (34)	345 (31)	−132 (19)
C(6)	0·28163 (44)	0·25518 (29)	0·46342 (24)	1816 (53)	1059 (28)	777 (20)	351 (35)	176 (27)	−3 (19)
C(7)	0·23271 (32)	0·35958 (23)	0·50916 (20)	1301 (40)	902 (21)	570 (15)	19 (23)	25 (21)	−15 (15)
C(8)	0·02969 (33)	0·53819 (23)	0·63066 (19)	1663 (43)	779 (19)	574 (14)	−76 (25)	−52 (23)	−93 (15)
C(9)	−0·10192 (40)	0·59410 (28)	0·67688 (24)	2075 (57)	952 (24)	767 (18)	231 (35)	−83 (28)	−199 (19)
C(10)	−0·14058 (42)	0·56995 (33)	0·77686 (25)	1816 (52)	1224 (32)	778 (20)	68 (35)	134 (37)	−326 (22)
C(11)	−0·05384 (40)	0·48642 (28)	0·83211 (21)	1793 (51)	1206 (29)	606 (16)	−320 (34)	32 (24)	−240 (19)
C(12)	−0·09639 (61)	0·45718 (52)	0·94055 (28)	2600 (83)	1940 (56)	−660 (20)	628 (61)	310 (35)	−211 (29)
C(13)	0·07539 (38)	0·42919 (26)	0·78449 (20)	1659 (45)	975 (23)	573 (14)	−196 (32)	−63 (23)	−28 (16)
C(14)	0·11926 (32)	0·45601 (23)	0·68549 (19)	1397 (40)	824 (19)	555 (14)	−98 (25)	−84 (20)	−67 (14)

$$\sigma^2(F^2)=As\{G+(t_G/t_B)^2B + 0{\cdot}0016[G-(t_G/t_B)B]^2\}/(\mathrm{Lp})^2,$$

where A = correction factor for absorption, s = scale factor, G = gross count, B = background count, t_G/t_B = ratio of the counting times of gross intensity and background, and Lp = Lorentz–polarization factor.

Two cycles of refinement with all nonhydrogen atoms treated isotropically resulted in unweighted discrepancy index, $R(F^2)=[\sum|F_o^2-F_c^2|/\sum F_o^2]\times 100=$

Table 2. *Positional and isotropic thermal parameters for hydrogen atoms*

	x	y	z	B
H(2)	0·036 (4)	0·464 (3)	0·321 (2)	4·4 (7) Å²
H(5)	0·273 (5)	0·155 (3)	0·344 (2)	6·5 (8)
H(6)	0·350 (6)	0·200 (3)	0·504 (3)	8·2 (11)
H(9)	−0·167 (4)	0·655 (3)	0·630 (2)	5·4 (7)
H(10)	−0·214 (5)	0·617 (4)	0·811 (3)	8·3 (11)
H(13)	0·140 (4)	0·361 (3)	0·815 (2)	6·8 (9)
H(4*a*)	−0·007 (6)	0·228 (4)	0·198 (3)	9·1 (13)
H(4*b*)	0·184 (7)	0·272 (4)	0·168 (4)	10·7 (15)
H(4*c*)	0·054 (7)	0·352 (5)	0·164 (4)	12·2 (17)
H(12*a*)	−0·228 (9)	0·456 (5)	0·960 (4)	12·4 (17)
H(12*b*)	−0·051 (8)	0·518 (6)	0·979 (4)	13·6 (19)
H(12*c*)	−0·109 (8)	0·378 (5)	0·956 (4)	12·2 (17)

Table 3. *Observed and calculated structure amplitudes* (× 10) *for* 2,7-*dimethylthianthrene*

K	L	10FO	10FC
**** H = 0 ****			
0	2	35	20
0	4	62	60
0	6	1046	1010
0	8	250	258
0	10	48	52
0	12	79	85
0	14	177	173
1	1	493	486
1	2	195	194
1	3	274	270
1	4	167	166
1	5	234	227
1	6	536	511
1	7	237	224
1	8	168	165
1	9	64	66
1	10	146	149
1	11	195	193
1	12	103	113
1	13	51	55
1	14	24	21
1	15	16	20
2	0	920	913
2	1	43	43
2	2	135	133
2	3	244	240
2	4	56	55
2	5	189	188
2	6	188	186
2	7	213	203
2	8	88	86
2	9	108	109
2	10	15	14
2	11	24	14
2	12	71	71
2	13	122	119
2	14	62	59
2	15	8	3
3	1	396	388
3	2	368	372
3	3	671	675
3	4	54	53
3	5	356	342
3	6	27	27
3	7	97	94
3	8	99	98
3	9	105	106
3	10	65	63
3	11	234	233
3	12	74	74
3	13	78	75
3	14	12	7
3	15	9	1
4	0	229	225
4	1	134	136
4	2	135	131
4	3	198	190
4	4	30	33
4	5	65	61
4	6	175	171
4	7	29	28
4	8	327	322
4	9	205	208
4	11	14	13
4	12	28	22
4	13	57	56
4	14	99	95
4	15	43	42
5	1	267	263
5	2	236	232
5	3	149	141
5	4	43	42
5	5	169	165
5	6	90	88
5	7	195	195
5	8	173	168
5	9	212	206
5	10	21	9
5	11	88	85
5	12	61	61
5	13	96	94
5	14	6	4
6	0	763	761
6	1	176	169
6	2	202	188
6	3	127	117
6	4	170	169
6	5	64	66
6	6	200	202
6	7	32	31
6	8	233	229
6	9	130	130
6	10	24	20
6	11	99	99
6	12	27	25
6	13	75	73
6	14	99	99
7	1	177	165
7	2	28	20
7	3	163	156
7	4	58	55
7	5	288	281
7	6	42	39
7	7	207	203
7	8	35	29
7	9	18	7
7	10	49	49
7	11	199	194
7	12	87	87
7	13	30	26
8	0	167	158
8	1	53	49
8	2	49	50
8	3	87	86
8	4	273	265
8	5	103	96
8	6	38	38
8	7	174	177
8	8	41	37
8	9	74	67
8	10	124	122
8	12	23	22
9	1	181	180
9	2	146	146
9	3	242	239
9	4	16	18
9	5	226	223
9	6	23	25
9	7	97	96
9	8	7	1
9	9	107	106
9	10	75	75
9	11	115	116
10	0	25	17
10	1	101	97
10	2	76	72
10	3	77	74
10	4	88	87
10	5	39	39
10	6	53	53
10	7	49	45
10	8	37	32
10	9	75	74
10	10	7	4
11	1	79	78
11	2	60	55
11	3	32	37
11	4	77	78
11	5	58	58
11	6	32	33
11	7	89	88
11	8	18	18
11	9	87	89
12	0	67	72
12	1	11	3
12	2	86	83
12	3	37	40
12	4	115	113
12	5	20	15
12	6	60	60
12	7	12	4
13	1	13	15
13	2	49	48
13	3	13	19
13	4	28	28
**** H = 1 ****			
0	1	379	381
0	2	259	247
0	3	37	37
0	4	100	100
0	5	155	155
0	6	185	182
0	7	138	132
0	8	18	1
0	9	12	5
0	10	133	137
0	11	16	21
0	12	37	32
0	13	89	93
0	14	15	2
0	15	26	25
1	0	839	872
1	1	471	489
1	2	448	440
1	3	256	278
1	4	63	64
1	5	284	287
1	6	369	361
1	7	416	411
1	8	322	305
1	9	110	113
1	10	54	54
1	11	145	146
1	12	51	53
1	13	39	35
1	14	83	86
1	15	59	60
2	0	87	89
2	1	890	933
2	2	563	567
2	3	453	440
2	4	112	117
2	5	215	206
2	6	176	180
2	7	284	281
2	8	338	330
2	9	163	153
2	10	100	103
2	11	151	150
2	12	89	88
2	13	56	57
2	14	45	50
2	15	49	53
3	0	370	374
3	1	751	762
3	2	609	605
3	3	79	78
3	4	21	22
3	5	306	300
3	6	110	105
3	7	94	90
3	8	162	165
3	9	98	101
3	10	130	129
3	11	78	77
3	12	24	33
3	13	85	90
3	14	48	42
3	15	35	33
4	0	275	281
4	1	122	129
4	2	309	317
4	3	355	368
4	4	244	253
4	5	317	305
4	6	307	304
4	7	37	31
4	8	267	268
4	9	208	207
4	10	284	283
4	11	136	128
4	12	84	83
4	13	75	77
4	14	56	57
4	15	16	17
5	0	404	401
5	1	280	287
5	2	304	305
5	3	406	390
5	4	227	224
5	5	214	211
5	6	159	151
5	7	165	164
5	8	304	304
5	9	195	192
5	10	83	84
5	11	124	124
5	12	48	43
5	13	69	68
5	14	100	97
6	0	201	198
6	1	180	174
6	2	152	143
6	3	175	169
6	4	78	74
6	5	163	160
6	6	70	66
6	7	232	226
6	8	168	169
6	9	151	151
6	10	51	48
6	11	112	111
6	12	18	25
6	13	64	64
6	14	31	27
7	0	128	128
7	1	297	295
7	2	73	69
7	3	160	163
7	4	116	117
7	5	180	176
7	6	81	81
7	7	224	220
7	8	49	44
7	9	60	54
7	10	39	39
7	11	104	103
7	12	33	28
7	13	63	62
8	0	156	158
8	1	72	74
8	2	149	154
8	3	162	157
8	4	121	126
8	5	133	134
8	6	128	128
8	7	54	51
8	8	28	31
8	9	67	64
8	10	47	47
8	11	82	84
8	12	77	77
9	0	23	14
9	1	119	119
9	2	62	59
9	4	97	99
9	5	42	41
9	6	74	75
9	7	74	74
9	8	58	60
9	9	22	20
9	10	41	38
9	11	30	30
10	0	88	88
10	1	113	113
10	2	118	116
10	3	75	72
10	4	149	152
10	5	81	82
10	6	112	107
10	7	79	74
10	8	57	58
10	9	67	64
10	10	93	93
11	0	22	19
11	1	58	59
11	2	31	27
11	3	101	99
11	4	41	39
11	5	75	70
11	6	55	54
11	7	36	34
11	8	47	45
11	9	74	71
12	0	6	7
12	1	37	35
12	2	66	61
12	3	86	84
12	4	38	41
12	5	40	41
12	6	35	34
12	7	48	45
13	0	10	4
13	1	60	60
13	2	76	76
13	3	90	93
13	4	86	89
**** H = 2 ****			
0	0	162	160
0	1	724	732
0	2	194	201
0	3	457	451
0	4	268	268
0	5	383	392
0	6	38	35
0	7	103	105
0	8	162	162
0	9	14	2
0	10	147	143
0	11	151	151
0	12	113	116
0	13	79	80
0	15	30	30
1	0	328	311
1	1	896	928
1	2	712	746
1	3	307	310
1	4	137	137
1	5	194	196
1	6	118	127
1	7	236	243
1	8	275	273
1	9	122	118
1	10	49	46
1	11	119	125
1	12	93	93
1	13	99	99
1	14	42	41
1	15	73	73
2	0	730	739
2	1	692	721
2	2	363	362
2	3	343	350
2	4	60	55
2	5	422	423
2	6	230	225
2	7	62	61
2	8	93	89
2	9	234	234
2	10	214	217
2	11	57	59
2	12	65	62
2	13	55	55
2	14	18	17
2	15	39	41
3	0	61	63
3	1	433	433
3	2	474	488
3	3	121	119
3	4	87	86
3	5	222	217
3	6	269	266
3	7	226	222
3	8	273	275
3	9	147	149
3	10	182	186
3	11	65	69
3	12	72	69
3	13	61	64
3	14	97	95
3	15	67	68
4	0	259	265
4	1	54	55
4	2	184	176
4	3	400	409
4	4	243	225
4	5	189	192
4	6	96	92
4	7	31	32
4	8	112	110
4	9	146	141
4	10	234	227
4	11	52	50
4	12	69	71
4	13	33	37
4	14	51	50
5	0	66	67
5	1	198	193
5	2	409	410
5	3	155	148
5	4	103	108
5	5	166	170
5	6	167	163
5	7	99	96
5	8	140	139
5	9	143	142
5	10	100	102
5	11	70	72
5	12	117	118
5	13	60	62
5	14	66	64
6	0	20	19
6	1	199	201
6	2	139	140
6	3	330	336
6	4	239	234
6	5	102	102
6	6	99	96
6	7	151	150
6	8	39	38
6	9	109	111
6	10	132	125
6	11	44	42
6	12	70	71
6	13	84	79
7	0	142	143
7	1	148	160
7	2	49	47
7	3	24	27
7	4	37	37
7	5	13	9
7	6	87	88
7	7	95	97
7	8	51	57
7	9	35	35
7	10	55	54
7	11	55	50
7	12	64	62
7	13	43	42
8	0	270	267
8	1	250	256
8	2	138	140
8	3	76	74
8	5	137	142
8	6	95	97
8	7	79	80
8	8	139	140
8	9	66	65
8	10	41	42
8	11	18	6
8	12	31	29
9	0	9	2
9	1	113	106
9	2	225	225
9	3	55	56
9	4	123	123
9	5	130	135
9	6	147	144
9	7	25	32
9	8	117	114
9	9	40	38
9	10	95	94
9	11	48	48
10	0	14	1
10	1	80	78
10	2	67	70
10	3	52	51
10	4	85	87
10	5	42	46
10	6	98	90
10	7	45	42
10	8	36	35
10	9	34	32
10	10	31	32
11	0	9	3
11	1	78	77
11	2	85	84
11	3	121	122
11	4	87	85
11	5	106	107
11	6	76	76
11	7	24	24
11	8	25	24
12	0	46	43
12	1	65	65
12	2	29	32
12	3	70	73
12	4	40	37
12	5	40	38
12	6	52	55
13	0	32	36
13	1	4	4
13	2	15	14
13	3	20	21
**** H = 3 ****			
0	1	278	259
0	2	28	28
0	3	296	299
0	4	149	153
0	5	315	310
0	6	218	220
0	7	57	53
0	9	177	176
0	10	52	50
0	11	22	23
0	12	99	98
0	13	106	109
0	14	22	26
0	15	14	10
1	0	230	229
1	1	111	110
1	2	260	275
1	3	187	191
1	4	326	329
1	5	256	240
1	6	192	196
1	7	109	115
1	8	114	116
1	9	136	137
1	10	171	171
1	11	148	144
1	12	45	45
1	13	36	36
1	14	69	67
1	15	59	59
2	0	142	138
2	1	371	365
2	2	158	166
2	3	377	375
2	4	191	201
2	5	204	215
2	6	25	26
2	7	265	263
2	8	165	168
2	9	331	344
2	10	92	96
2	11	16	18
2	12	72	68
2	13	90	93
2	14	57	58
3	0	114	112
3	1	54	53
3	2	225	222
3	3	538	541
3	4	172	171
3	5	113	110
3	6	124	129
3	7	46	47
3	8	189	194
3	9	333	339
3	10	72	71
3	11	47	42
3	12	45	46
3	13	36	33
3	14	49	50
4	0	93	95
4	1	147	147
4	2	530	536
4	3	225	236
4	4	179	180
4	5	147	143
4	6	89	89
4	7	167	169
4	8	131	131
4	9	264	270
4	10	73	74
4	11	46	43
4	12	51	51
4	13	89	92
4	14	49	47
5	0	13	11
5	1	141	142
5	2	283	293
5	3	211	213
5	4	300	297
5	5	39	44
5	6	78	77
5	7	65	65
5	8	59	53
5	9	31	33
5	10	74	77
5	11	57	52
5	12	101	103
5	13	16	17
6	0	227	234
6	1	123	122
6	2	117	118
6	3	131	136
6	4	210	209
6	5	89	88
6	6	109	112
6	7	78	79
6	8	31	31
6	9	82	87
6	10	69	68
6	11	44	44
6	12	92	89
6	13	45	43
7	0	407	420
7	1	29	30
7	2	231	233
7	3	22	14
7	4	176	174
7	5	83	80
7	6	265	269
7	7	48	47
7	8	151	151
7	9	59	55
7	10	107	109
7	11	24	23
7	12	55	54
8	0	144	148
8	1	114	114
8	2	24	16
8	3	81	82
8	4	65	62
8	5	144	146
8	6	59	63
8	7	44	52
8	8	68	66
8	9	54	54
8	10	39	38
8	11	35	35
9	0	153	156
9	1	81	81
9	2	84	88
9	3	51	53
9	4	37	41
9	5	18	11
9	6	77	78
9	7	52	53
9	8	46	51
9	9	34	35
9	10	8	4
10	0	14	20
10	1	94	94
10	2	89	92
10	3	116	116
10	4	55	53
10	5	113	116
10	6	41	44
10	7	44	43
10	8	26	24
10	9	62	62
11	0	26	26
11	1	63	64
11	2	108	105
11	3	20	20
11	4	84	84
11	5	36	38
11	6	42	41
11	7	16	18
12	0	30	31
12	1	39	37
12	2	29	25
12	3	40	40
12	4	51	49
12	5	79	81
**** H = 4 ****			
0	0	330	322
0	1	543	536
0	2	152	141
0	3	11	6
0	4	315	317
0	5	101	94
0	6	35	40
0	7	437	445
0	8	206	212
0	9	62	61
0	10	122	123
0	11	62	62
0	12	40	42
0	13	51	52
0	14	16	9
1	0	136	131
1	1	310	296
1	2	265	269
1	3	95	92
1	4	231	236
1	5	44	49
1	6	60	54
1	7	184	187
1	8	20	21
1	9	151	151
1	10	131	139
1	11	113	113
1	12	154	150
1	13	77	74
1	14	37	34
2	0	328	327
2	1	345	344
2	2	291	292
2	3	137	137
2	4	144	140
2	5	93	101
2	6	145	145
2	7	184	191
2	8	32	39
2	9	113	112
2	10	22	22
2	11	53	55
2	12	56	58
2	13	34	34
2	14	78	76
3	0	12	14
3	1	90	84
3	2	396	398
3	3	153	157
3	4	438	452
3	5	145	145
3	6	54	53
3	7	121	123
3	8	42	40
3	9	74	78
3	10	120	121
3	11	114	116
3	12	108	106
3	13	66	61
3	14	20	16
4	0	22	32
4	1	98	103
4	2	22	20
4	3	102	104
4	4	78	74
4	5	103	105
4	6	71	65
4	7	144	145
4	8	42	40
4	9	121	121
4	10	30	32
4	11	80	74
4	12	49	49
4	13	17	16
5	0	143	139
5	1	169	177
5	2	123	122
5	3	125	124
5	4	28	32
5	5	43	49
5	6	82	83
5	7	24	23
5	8	98	99
5	9	106	104
5	10	51	50
5	11	60	57
5	12	63	65
5	13	37	39
6	0	167	174
6	1	215	219
6	2	130	135
6	3	171	167
6	4	143	146
6	5	98	90
6	6	167	171
6	7	196	202
6	8	60	59
6	9	99	102
6	10	99	102
6	11	23	23
6	12	56	57
7	0	201	207
7	1	161	165
7	2	37	44
7	3	114	118
7	4	175	179
7	5	104	104
7	6	31	42
7	7	61	64
7	8	55	56
7	9	13	5
7	10	72	74
7	11	34	31
8	0	262	265
8	1	129	133
8	2	80	80
8	3	161	162
8	4	72	74
8	5	87	87
8	6	107	112
8	7	59	59
8	8	29	24
8	9	46	44
8	10	18	23
8	11	68	66
9	0	27	26
9	1	17	10
9	2	119	122
9	3	59	57
9	4	125	130
9	5	67	64
9	6	78	81
9	7	54	59
9	8	38	37
9	9	46	43
10	0	37	37
10	1	37	37
10	2	52	51
10	3	19	22
10	4	34	32
10	5	31	34
10	6	37	36
10	7	56	58
10	8	38	35
11	0	80	81
11	1	23	25
11	2	64	65
11	3	38	38
11	4	48	49
11	5	51	52
11	6	51	50
12	0	57	55
12	1	51	49
12	2	52	52
12	3	58	58
**** H = 5 ****			
0	1	256	245
0	2	96	96
0	3	59	60
0	4	36	34
0	5	151	150
0	6	18	16
0	7	16	9
0	8	126	132
0	9	60	62
0	10	10	12
0	11	13	6
0	12	50	48
0	13	30	29
1	0	120	118
1	1	287	273
1	2	107	100
1	3	103	101
1	4	128	126
1	5	77	77
1	6	159	165
1	7	262	266
1	8	161	168
1	9	67	60
1	10	67	71
1	11	6	14
1	12	43	45
1	13	24	20
2	0	200	208
2	1	172	162
2	2	229	224
2	3	31	29
2	4	382	385
2	5	95	91
2	6	97	93
2	7	98	99
2	8	127	120
2	9	41	41
2	10	152	153
2	11	63	60
2	12	156	154
2	13	45	42
3	0	40	38
3	1	75	75
3	2	116	107
3	3	147	144
3	4	18	27
3	5	112	115
3	6	177	184
3	7	51	53
3	8	16	19
3	9	30	28
3	10	62	63
3	11	82	82
3	12	39	42
3	13	35	36
4	0	32	29
4	1	192	187
4	2	114	114
4	3	168	169
4	4	219	217
4	5	88	86
4	6	88	88
4	7	63	64
4	8	47	43
4	9	109	109
4	10	125	124
4	11	49	50
4	12	75	76
5	0	172	172
5	1	267	273
5	2	57	52
5	3	102	109
5	4	110	109
5	5	78	81
5	6	94	94
5	7	203	210
5	8	52	50
5	9	63	67
5	10	50	53
5	11	32	30
5	12	37	40
6	0	18	11
6	1	162	160
6	2	86	84
6	3	26	31
6	4	134	139
6	5	65	67
6	6	20	21
6	7	35	42
6	8	112	116
6	9	35	31
6	10	28	27
6	11	37	35
7	0	24	16
7	1	147	148
7	2	74	69
7	3	111	114
7	4	66	61
7	5	109	111
7	6	79	83
7	7	124	125
7	8	22	20
7	9	20	24
7	10	56	57
8	0	5	2
8	1	63	62
8	2	62	64
8	3	63	65
8	4	118	123
8	5	107	108
8	6	39	39
8	7	52	53
8	8	38	40
8	9	43	44
9	0	34	30
9	1	55	50
9	2	52	51
9	3	81	83
9	4	56	54
9	5	77	77
9	6	15	14
9	7	60	60
9	8	42	39
10	0	110	112
10	1	63	64
10	2	65	64
10	3	65	61
10	4	55	60
10	5	58	61
10	6	73	69
10	7	18	20
11	0	8	3
11	1	58	57
11	2	66	63
11	3	10	8
11	4	12	15
**** H = 6 ****			
0	0	321	311
0	1	175	180
0	2	127	127
0	3	111	116
0	4	152	152
0	5	26	14
0	6	177	181
0	7	22	11
0	8	60	59
0	10	70	69
0	11	99	97
0	12	59	59
1	0	249	236
1	1	71	72
1	2	86	84
1	3	31	30
1	4	98	99
1	5	203	205
1	6	105	96
1	7	46	45
1	8	31	27
1	9	48	47
1	10	42	40
1	11	41	42
1	12	90	91
2	0	74	69
2	1	23	16
2	2	181	175
2	3	37	37
2	4	104	103
2	5	81	84
2	6	175	180
2	7	30	31
2	8	45	42
2	9	35	33
2	10	64	66
2	11	24	21
2	12	64	63
3	0	244	239
3	1	188	183
3	2	109	113
3	3	145	148
3	4	196	203
3	5	91	98
3	6	68	68
3	7	99	91
3	8	69	72
3	9	102	105
3	10	79	80
3	11	7	5
3	12	48	48
4	0	35	35
4	1	70	67
4	2	124	125
4	3	201	205
4	4	114	112
4	5	121	123
4	6	78	81
4	7	123	123
4	8	41	40
4	9	57	54
4	10	37	32
4	11	79	82
5	0	105	108
5	1	131	126
5	2	59	63
5	3	140	141
5	4	83	81
5	5	144	150
5	6	52	47
5	7	61	54
5	8	49	49
5	9	86	87
5	10	23	17
5	11	36	38
6	0	133	135
6	1	120	118
6	2	101	100
6	3	23	26
6	4	74	71
6	5	13	15
6	6	101	101
6	7	63	57
6	8	60	59
6	9	18	14
6	10	57	56
7	0	53	52
7	1	75	75
7	2	14	18
7	3	17	7
7	4	59	58
7	5	8	10
7	6	49	44
7	7	28	30
7	8	62	65
7	9	35	33
8	0	13	14
8	1	46	46
8	2	38	41
8	3	33	30
8	4	48	49
8	5	83	87
8	6	24	27
8	7	68	70
8	8	42	40
9	0	9	2
9	1	122	123
9	2	11	12
9	3	62	63
9	4	18	22
9	5	96	97
9	6	10	8
10	0	8	1
10	1	11	16
10	2	44	43
10	5	46	44
10	4	8	10
**** H = 7 ****			
0	1	84	86
0	2	141	138
0	3	27	28
0	4	41	38
0	5	201	202
0	6	143	141
0	7	127	128
0	8	45	42
0	9	61	61
0	10	51	49
0	11	35	34
1	0	28	23
1	1	62	59
1	2	89	84
1	3	190	187
1	4	128	126
1	5	78	75
1	6	133	129
1	7	30	36
1	8	29	20
1	9	75	75
1	10	63	62
1	11	80	81
2	0	167	157
2	1	32	30
2	2	78	75
2	4	93	91
2	5	35	34
2	6	102	100
2	7	36	41
2	8	65	67
2	9	52	51
2	10	27	27
3	0	23	11
3	1	32	31
3	2	167	163
3	3	88	89
3	4	87	88
3	5	31	27
3	6	27	20
3	7	42	43
3	8	33	31
3	9	54	55
3	10	71	67
4	0	56	52
4	1	81	85
4	2	145	142
4	3	105	102
4	4	54	52
4	5	80	77
4	6	71	70
4	7	47	49
4	8	50	49
4	9	81	83
4	10	13	15
5	0	15	3
5	1	40	34
5	2	68	69
5	3	73	66
5	4	90	85
5	5	118	120
5	6	38	42
5	7	21	11
5	8	45	42
5	9	24	21
6	0	37	38
6	1	53	54
6	2	88	87
6	3	41	41
6	4	91	94
6	5	104	105
6	6	45	46
6	7	28	32
6	8	49	48
7	1	62	67
7	2	88	84
7	3	35	37
7	4	49	44
7	5	73	70
7	6	57	56
7	7	66	65
8	0	71	68
8	1	81	79
8	2	56	56
8	3	36	33
8	4	15	11
8	5	17	19
8	6	49	47
9	0	38	41
9	1	39	40
9	2	5	8
9	3	35	34
**** H = 8 ****			
0	0	27	31
0	1	13	20
0	2	20	7
0	3	174	172
0	4	64	65
0	5	46	44
0	6	27	24
0	7	26	27
0	8	80	79
0	9	26	29
1	0	63	66
1	1	55	52
1	2	59	53
1	3	76	76
1	4	58	54
1	5	123	125
1	6	44	45
1	7	47	46
1	8	28	24
1	9	46	45
2	0	43	39
2	1	87	84
2	2	60	60
2	3	64	62
2	4	28	27
2	5	60	60
2	6	90	92
2	7	36	33
2	8	55	52
3	0	66	65
3	1	36	33
3	2	74	75
3	3	85	80
3	4	69	68
3	5	102	101
3	6	25	25
3	7	55	52
3	8	56	58
4	0	60	56
4	1	41	43
4	2	18	21
4	3	90	91
4	4	70	70
4	5	59	55
4	6	10	13
4	7	18	19
5	0	9	8
5	1	72	73
5	2	116	117
5	3	39	39
5	4	108	109
5	5	96	96
5	6	47	52
5	7	14	11
6	0	39	38
6	1	31	29
6	2	93	92
6	3	30	35
6	4	20	21
6	5	48	48
7	0	51	50
7	1	43	43
7	2	24	18
7	3	58	57
7	4	32	32
**** H = 9 ****			
0	1	38	38
0	2	56	58
0	4	29	30
0	5	57	59
0	6	28	30
1	0	73	74
1	1	50	47
1	2	24	28
1	3	28	28
1	4	24	23
1	5	69	70
1	6	58	56
2	0	29	30
2	1	5	5
2	2	33	33
2	3	108	106
2	4	78	73
2	5	89	92
3	0	26	30
3	1	37	41
3	2	40	36
3	3	69	71
3	4	49	49
3	5	35	34
4	0	11	2
4	1	23	26
4	2	43	45
4	3	90	87
4	4	4	12
5	0	98	96
5	1	8	8
5	2	29	35

13·9%. The refinement was continued with all nonhydrogen atoms assigned anisotropic temperature factors. After two cycles, $R(F^2)$ became 9·7%. From a three-dimensional difference synthesis calculated at this stage, all 12 hydrogen atoms were located. The full-matrix least-squares refinement was carried further with nonhydrogen atoms assigned anisotropic thermal parameters and hydrogen atoms assigned isotropic thermal parameters. After six cycles the values of $R(F^2)$ and $R_w(F^2)$ (defined as $[\sum w|F_o^2-F_c^2|^2/\sum wF_o^4]^{1/2}\times 100$) remained at 4·7 and 7·3%, respectively. The unweighted discrepancy index based on F was 3·1%. Parameter shifts in the last cycle for the nonhydrogen atoms were all less than 5% of the corresponding standard deviation, whereas the maximum shift in the hydrogen parameters was 83% of the corresponding standard deviation. The standard deviation of an observation of unit weight, defined as

$$[\sum w|\Delta F^2|^2/(n-p)]^{1/2}$$

where n is the number of observations and p the number of parameters fitted to the data set, was 1·386. Scattering factors that were used were those of Dawson (1960) for sulfur, those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for carbon, and those of Stewart, Davidson & Simpson (1965) for hydrogen.

Final values of the atomic parameters and their estimated standard deviations are given in Table 1 for the nonhydrogen atoms and in Table 2 for the hydrogen atoms. Observed and calculated structure amplitudes are listed in Table 3. The Busing, Martin & Levy (1964) function and error program *ORFEE* was utilized to calculate the molecular parameters with their standard deviations.

Discussion

The crystal structure comprises molecules with an idealized configuration (depicted in Fig. 1 with some selected bond distances and angles) of C_2 symmetry. All carbon atoms of each methylphenylene fragment are essentially coplanar as shown in Table 4(*a*) and (*c*), whereas the two other best molecular planes, each defined by the nonhydrogen atoms of the $H_3CC_6H_3S_2$ moiety [Table 4(*b*) and (*d*)], are not as good as the ones defined by the corresponding methylphenylene fragments. Still, the molecule can be considered to be formed by the fusion of two nearly planar $H_3CC_6H_3S_2$ moieties along the common S–S line. Such folded configurations have also been found in the similar structures of 1,4-dithiadiene (Howell, Curtis & Lipscomb, 1954), thianthrene, *β*-thianthrene dioxide (Hosoya, 1965; Ammon, Watts & Stewart, 1970), thioxanthen-9-ol 10-oxide (Ternay, Chasar & Sax, 1967), and *cis*-9-methylthioxanthene 10-oxide (Jackobs & Sundaralingam, 1969). For this structure, the dihedral angle between the planes of the $H_3CC_6H_3S_2$ moieties, defined by parameters (*b*) and (*d*) of Table 4, is 131·1°. This value compares roughly with the corresponding values of 128° given by Lynton & Cox and 128·1° reported by Rowe & Post, both for thianthrene. The two chemically equivalent C–S–C valence angles of 101·6 (1)° at S(1) and S(2) do not differ significantly from the average values of 100·2 (5)° (Lynton & Cox) and 100·4 (3)° (Rowe & Post) in thianthrene or from the angle of 100 (2)° (for model A) found in 1,4-dithiadiene. The four equivalent S–C_{sp2} bonding distances in this structure are equal within their standard deviations. The average value of 1·764 (2) Å is approximately 0·05 Å shorter than that of 1·812 Å, the sum of the covalent radii given by Pauling (1960); it does not differ significantly from those of 1·759 (15) Å (Lynton & Cox) and 1·773 (5) Å (Rowe & Post), both for thianthrene, and that of 1·78 (5) Å in 1,4-dithiadiene. However, this value is slightly shorter than those of 1·782 (2) Å in *cis*-9-methylthioxanthene 10-oxide, 1·775 (8) Å in thioxanthen-9-ol 10-oxide, and 1·785 (5) Å in the re-examined structure of *β*-thianthrene (Ammon, Watts & Stewart, 1970). Thus, it seems safe to conclude that the S–C_{sp2} distance is lengthened slightly by the order of 0·01 to 0·02 Å as

Table 4. *Equations of best molecular planes and perpendicular distances* (Å) *of constituent atoms from these planes*

Calculations were performed with the program written by Smith (1962).
x, y and z represent atomic fractional coordinates in terms of the respective crystal axial systems. Last term in each equation is the perpendicular distance (Å) from the origin to the plane.

(*a*) $6·769x+5·113y-4·408z=1·176$
(*b*) $6·839x+4·888y-4·469z=1·098$
(*c*) $5·052x+8·165y+4·187z=7·183$
(*d*) $4·878x+8·310y+4·337z=7·385$

	(*a*)	(*b*)		(*c*)	(*d*)
C(1)	0·007	−0·031	C(8)	0·001	−0·033
C(2)	−0·004	−0·033	C(9)	−0·014	−0·010
C(3)	0·009	0·009	C(10)	0·013	0·035
C(4)	−0·009	−0·000	C(11)	0·000	0·003
C(5)	0·011	0·032	C(12)	0·001	0·023
C(6)	−0·008	0·005	C(13)	−0·014	−0·048
C(7)	−0·007	−0·024	C(14)	0·013	−0·041
S(1)	– –	0·044	S(1)	– –	0·021
S(2)	– –	−0·002	S(2)	– –	0·050

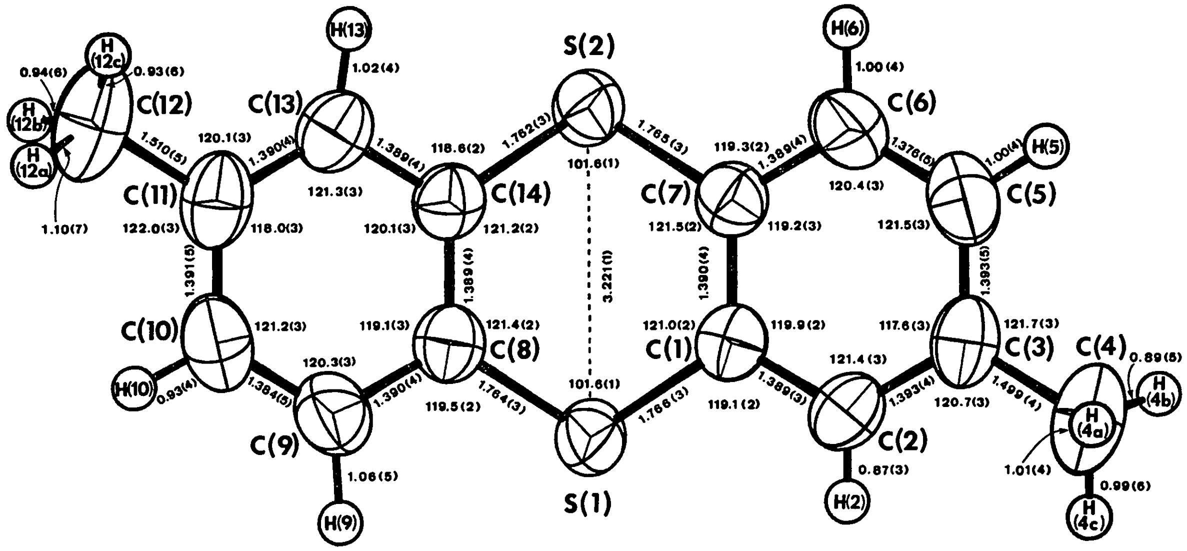

Fig. 1. Molecular configuration of 2,7-dimethylthianthrene viewed in a direction parallel to the idealized molecular twofold axis. All nonhydrogen atoms are represented by 50% probability ellipsoids of thermal displacement (Johnson, 1965).

the coordination number of the sulfur atom is increased from two to three in this group of heterocyclic compounds, in agreement with an observation made by Jackobs & Sundaralingam (1969).

The mean C–C bond distance of 1·389 (1) Å for the benzene rings is compatible with the expected value of 1·394 (5) Å (*Tables of Interatomic Distances and Configuration in Molecules and Ions*, 1965). The C_{sp2}–C_{sp3} distances of 1·499 (4) Å and 1·510 (5) Å, the C–H distances [ranging from 0·87 (3) Å for C(2)–H(2) to 1·10 (7) Å for C(12)–H(12*a*)], and all other molecular parameters are not unlike those obtained in structural determinations of other common organic compounds by the X-ray diffraction method. The closest intermolecular contact in this structure is 2·89 Å between C(1) and H(6) of adjacent molecules, thus indicating that only molecular forces are responsible for the crystal packing.

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