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The Crystal Structure of 2-Dimethylsulfuranylidene-1,3-indanedione*

BY ARILD T. CHRISTENSEN AND ERLING THOM†

Syntex Research, Institute of Organic Chemistry, Palo Alto, California, U.S.A.

2-Dimethylsulfuranylidene-1,3-indandione, $C_{11}H_{10}SO_2$, crystallizes in space group $P2_12_12_1$, with $a = 18\cdot331$, $c = 9\cdot012$, $c = 5\cdot949$ Å, estimated standard deviations $0\cdot008$ Å. The structure was refined by full-matrix, least-squares procedures to a final R value of $0\cdot028$. The refinement was based on 1170 reflections collected with an automatic diffractometer. The exocyclic C–S ylid bond distance is $1\cdot71$ Å, and confirms the view that there is an appreciable amount of double-bond character in this type of bond.

Introduction

The recent interest in the chemistry of sulfonium ylids (Cook & Moffatt, 1968, and references cited therein) prompted the crystal-structure analysis of 2-dimethyl-sulfuranylidenemalononitrile (Christensen & Witmore, 1969), and the present work was carried out to continue the investigation of this class of compounds.

Experimental

Crystals of 2-dimethylsulfuranylidene-1,3-indandione were kindly supplied to us by Dr John Moffatt. Preliminary precession photographs indicated orthorhombic symmetry. The systematic extinctions, $h00$, $0k0$ and $00l$ with h, k and l odd, are consistent with space group $P2_12_12_1$.

The crystal used for the determination of cell parameters and collection of intensity data was ground into a sphere of diameter $0\cdot33$ mm by the method of Bond (1951). The cell parameters and orientation parameters were refined by least-squares; the refinement gave the following results: $a = 18\cdot331$, $b = 9\cdot012$ and $c = 5\cdot949$ Å, with estimated standard deviations of the order $0\cdot008$ Å. These values give a calculated density of $1\cdot39$ g.cm^{-3} for $Z = 4$. The calculated linear absorption coefficient for copper radiation is $25\cdot8$ cm^{-1}. Inten-

sities were collected, using Ni-filtered Cu $K\alpha$ radiation and an automated Picker diffractometer, of the 1200 reflections in the range $0 < 2\theta < 160$ that were measured using the $2\theta-\theta$ scan technique, 1170 had an intensity greater than three times the standard deviation and were recorded as 'observed'. No systematic change was detected in two standard reflections which were measured at regular intervals during the course of the data collection. The intensities were corrected for absorption using the spherical absorption factors published by Weber (1969).

Determination and refinement of the structure

The position of the sulfur atom was determined from a sharpened Patterson function, and phase angles computed from the sulfur atom parameters were refined by the tangent formula (Karle & Hauptman, 1956). An E map calculated from the refined phases revealed the positions of all atoms other than hydrogen. The parameters of these atoms, anisotropic temperature factors included, were refined by full-matrix least squares. A difference Fourier synthesis calculated when the R index was $0\cdot06$ revealed the positions of the ten hydrogen atoms, but showed no other significant maxima or minima. Inclusion of the hydrogen atoms in the structure factor calculation changed R to $0\cdot047$. At this point it was apparent that the strong low-order reflections suffered from secondary extinction effects. After application of the correction method of Zachariasen (1963), reflections with $\sin \theta/\lambda < 0\cdot40$ Å^{-1} were

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Table 1. *Final positional parameters and their standard deviations*

The e.s.d. (in parentheses) apply to the least significant digits.

C(1)	0·10008 (12) Å	−0·05523 (28) Å	1·18330 (43) Å
C(2)	0·07783 (12)	−0·12614 (26)	1·38522 (44)
C(3)	0·12478 (12)	−0·24872 (25)	1·43920 (49)
C(4)	0·18203 (12)	−0·24824 (27)	1·26019 (49)
C(5)	0·24261 (13)	−0·33758 (30)	1·23448 (58)
C(6)	0·28770 (14)	−0·31158 (32)	1·05134 (71)
C(7)	0·27250 (15)	−0·20004 (33)	0·89875 (60)
C(8)	0·21165 (14)	−0·10923 (29)	0·92521 (54)
C(9)	0·16740 (12)	−0·13548 (26)	1·10749 (46)
O(10)	0·07282 (9)	0·05253 (20)	1·08495 (34)
O(11)	0·12074 (9)	−0·33904 (19)	1·59239 (38)
S(12)	0·00963 (3)	−0·06841 (6)	1·55985 (11)
C(13)	−0·07276 (13)	−0·07369 (35)	1·40039 (67)
C(14)	0·02129 (15)	0·12778 (27)	1·58522 (56)
H(15)	0·19930 (212)	−0·03881 (481)	0·80885 (781)
H(16)	0·30718 (241)	−0·19207 (525)	0·75411 (927)
H(17)	0·33384 (208)	−0·37029 (451)	1·02650 (769)
H(18)	0·25517 (246)	−0·41118 (527)	1·35533 (861)
H(19)	0·02535 (214)	0·16566 (463)	1·42299 (865)
H(20)	0·07654 (237)	0·14432 (518)	1·68686 (835)
H(21)	−0·02535 (203)	0·16008 (451)	1·67677 (721)
H(22)	−0·08700 (240)	−0·16705 (541)	1·36148 (852)
H(23)	−0·11177 (269)	−0·03064 (609)	1·51986 (988)
H(24)	−0·06271 (218)	−0·03427 (509)	1·24319 (811)

included in a least-squares refinement of the hydrogen parameters. The refinement was concluded with three cycles of least squares in which the positional parameters and anisotropic temperature factors of all non-hydrogen atoms were included. The shifts in the last cycle were about 10% of the corresponding e.s.d.'s. The final R is 0·028, excluding the 'unobserved' reflections.

Analysis of the rigid-body motion of the molecule was done by the method of Schomaker & Trueblood (1968). The root-mean-square difference between calculated and observed U_{ij} values is 0·003 Å², so the rigid-body model does not seem to be quite satisfactory The translational motion was indicated to be fairly isotropic; the libration was more anisotropic with the largest amplitude ($\sim 5°$) about the longitudinal axis of the molecule.

The bond distances and angles were corrected for the effect of molecular libration (Cruickshank, 1961). However, none of the corrections were greater than the corresponding e.s.d.'s, and only uncorrected parameters are listed.

The positional and thermal parameters for the final structure are given in Tables 1 and 2 respectively. The rigid-body tensors are given in Table 3. Observed and calculated structure factors are given in Table 4.

Computing procedures

All calculations were performed on a CDC 6600 computer, for the most part using programs referenced in a previous paper (Christensen, 1970). The least-squares program minimizes the weighted sum of $(k \cdot |F_o| - G \cdot |F_c|)^2$; the weighting scheme used was that of Hughes (1941), with $F_o(\text{min}) = 1$. The anisotropic temperature factors are of the form: $\exp[-(h^2B_{11} + k^2B_{22}$

$+ l^2B_{33} + hkB_{12} + hlB_{13} + klB_{23})]$. The R index is defined by $R = \sum ||F_o| - |F_c||/\sum |F_o|$. The atomic form factors used where those given by Hanson, Herman, Lea &
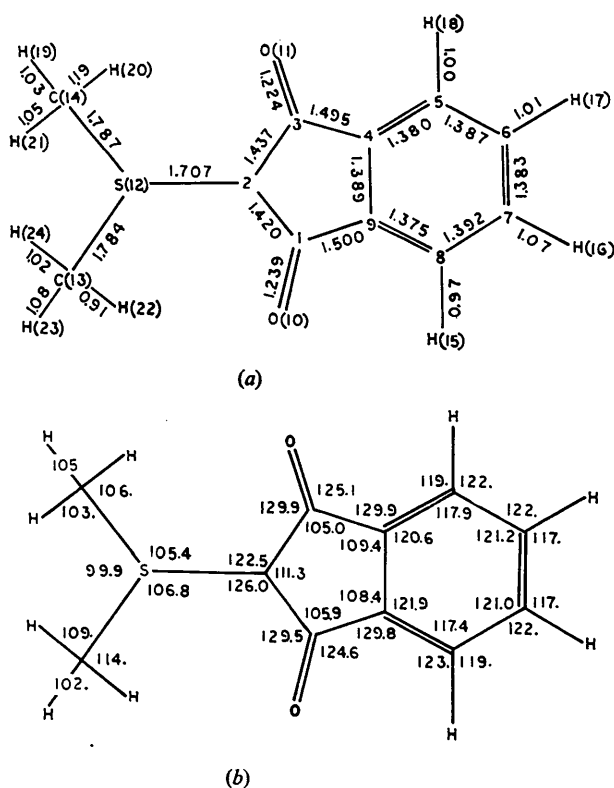

(a)

(b)

Fig. 1(a) Bond lengths, (b) bond angles. The e.s.d.'s are in the range 0·003–0·005 Å and 0·1–0·3° for distances and angles respectively involving the heavy atoms, and about 0·05 Å and 2·8° for those involving hydrogen atoms.

Skillman (1964). The secondary extinction program, written by A. T. Christensen, applies the correction in the form $F_{corr} = F_{obs}(1 + C\beta J_{obs})^{1/2}$ (Zachariasen, 1963)

where β equals $2(1 + \cos^4 2\theta)/(1 + \cos^2 2\theta)^2 \cdot \dfrac{dA^*/d\mu r}{dA_0^*/d\mu r}$.

Values of the derivative of the absorption factor with respect to the absorption coefficient as function of the Bragg angle were obtained from the spherical absorption factors published by Weber (1969). The value of the constant C, which was determined by minimizing $\sum(F_c - F_{corr})^2$, was found to be $2 \cdot 4 \times 10^{-5}$.

Analysis of the thermal motion and some of the molecular geometry calculations were done with a set of programs written by N. N. Trueblood.

The structure

Interatomic distances and angles calculated from the final positional parameters are shown in Fig. 1. Some least-squares planes are given in Table 5.
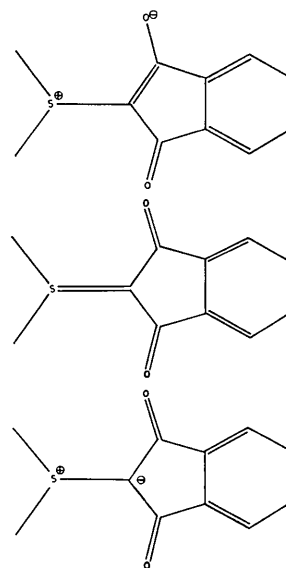
Fig. 2. Resonance forms.

Table 2. Final thermal parameters, with e.s.d.'s in parentheses

The anisotropic temperature factor has the form $\exp[-\frac{1}{4}(B_{11}h^2a^{*2}\ldots + 2B_{12}hka^*b^*\ldots)]$.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	3·10 (9)	2·98 (9)	2·90 (10)	−0·12 (9)	−0·17 (8)	−0·19 (9)
C(2)	2·75 (9)	2·93 (9)	2·96 (11)	0·23 (8)	0·30 (8)	0·03 (8)
C(3)	2·81 (8)	2·61 (8)	3·57 (11)	−0·07 (7)	−0·04 (10)	−0·15 (11)
C(4)	2·77 (9)	2·59 (9)	3·95 (12)	−0·17 (7)	0·19 (9)	−0·31 (9)
C(5)	3·04 (9)	3·27 (11)	5·56 (15)	0·18 (9)	0·44 (11)	−0·14 (11)
C(6)	3·30 (10)	3·93 (11)	6·98 (20)	−0·09 (10)	1·40 (14)	−1·24 (16)
C(7)	3·96 (11)	4·41 (13)	5·07 (16)	−0·42 (11)	1·72 (13)	−0·88 (14)
C(8)	3·80 (10)	4·00 (11)	3·62 (12)	−0·23 (9)	0·85 (11)	−0·33 (12)
C(9)	2·89 (9)	2·94 (9)	3·10 (11)	−0·44 (8)	0·22 (8)	−0·54 (9)
O(10)	4·60 (8)	3·99 (8)	3·41 (8)	0·94 (7)	0·24 (7)	0·87 (8)
O(11)	4·31 (8)	3·54 (7)	4·59 (9)	0·59 (6)	0·71 (8)	1·20 (8)
S(12)	2·99 (2)	2·57 (2)	2·97 (2)	0·29 (1)	0·62 (2)	0·39 (2)
C(13)	3·06 (10)	4·61 (13)	6·85 (19)	0·10 (10)	−0·57 (13)	−0·62 (17)
C(14)	5·28 (13)	2·76 (9)	4·02 (13)	−0·01 (9)	0·89 (12)	−0·70 (11)
H(15)	2·60 (123)					
H(16)	4·05 (139)					
H(17)	2·08 (116)					
H(18)	4·39 (141)					
H(19)	2·21 (116)					
H(20)	3·27 (134)					
H(21)	1·54 (120)					
H(22)	3·44 (134)					
H(23)	6·11 (162)					
H(24)	2·45 (120)					

Table 3. Results of rigid-body analysis of thermal parameters

	Eigenvalues	Eigenvectors Direction cosines of the eigenvectors to a, b and c		
Librational tensor, **L**	24·9 (deg)^2	0·7682	−0·3726	−0·5206
	11·3	0·3236	0·9277	−0·1863
	4·1	0·5524	−0·0253	0·8332
Translational tensor, **T**	0·044 Å^2	0·1692	−0·5633	0·8088
	0·036	0·7764	−0·4293	−0·4614
	0·027	0·6071	0·7060	0·3647
Symmetrized screw tensor, **S**	$\begin{pmatrix} 1133 & 734 & -754 \\ & 701 & 76 \\ & & 1835 \end{pmatrix} \times 10^6$ rad.. Å			

The unique origin in fractional coordinates along the crystal axes: $0 \cdot 1143$, $-0 \cdot 1173$, $1 \cdot 3328$

Inspection of Table 5 shows that the indane-1,3-dione moiety deviates significantly from a planar configuration, *e.g.* atoms C(2) and O(10) are displaced 0·07 Å from the plane through the benzene ring (plane 2). However, the sulfur atom and the 1,3-dione moiety are coplanar to within 0·04 Å (plane 3) and C(2) is displaced only 0·04 Å from the plane through C(1), S(12) and C(3). The deviation from a planar configuration is probably due to packing effects. The two carbon–carbon distances from C(2) in the sulfonium ylid aver-

Table 4. *Observed and calculated structure factors and phase angles*

The columns are 1, $10F_o$, $10F_c$ and the phase angle.

H= 0, K= 0			
2	604	606	0
4	242	237	180
6	290	290	180
H= 0, K= 1			
1	112	110	90
2	242	249	90
3	68	70	90
4	157	157	90
5	48	50	270
6	107	110	90
H= 0, K= 2			
0	30	29	180
1	82	79	0
2	197	189	0
3	16	23	180
4	75	72	180
5	215	219	180
6	5	4	180
H= 0, K= 3			
1	413	408	90
2	441	440	90
3	146	143	90
4	167	161	90
5	109	116	270
6	37	34	98
H= 0, K= 4			
0	419	409	180
1	28	27	180
2	168	160	180
3	375	375	180
4	160	163	0
5	153	151	180
6	49	50	0
7	3	4	180
H= 0, K= 5			
1	473	470	90
2	204	192	270
3	130	130	90
4	112	108	270
5	109	108	270
6	38	40	270
H= 0, K= 6			
0	236	234	180
1	162	162	180
2	140	140	180
3	313	317	180
4	41	41	180
5	84	82	180
6	53	53	0
H= 0, K= 7			
1	29	33	90
2	135	135	270
3	29	25	90
4	96	96	270
5	78	78	270
6	68	74	270
H= 0, K= 8			
0	139	134	180
1	35	37	0
2	84	86	180
3	104	106	0
4	5	3	0
5	12	13	0
H= 0, K= 9			
1	47	45	270
2	87	89	270
3	17	16	270
4	46	66	270
H= 0, K= 10			
0	169	176	180
1	78	78	0
2	5	5	0
3	104	102	0
H= -0, K=-11			
-0	4	0	90
H= 0, K= 11			
1	161	161	270
2	64	66	270
H= 1, K= 0			
1	276	279	90
2	195	188	180
3	195	199	270
4	10	3	180
5	176	177	270
6	11	4	0
7	53	60	270
H= 1, K= 1			
0	302	303	90
1	463	488	143
2	113	98	143
3	257	243	233
4	105	107	278
5	82	82	41
6	42	44	323
H= 1, K= 2			
0	412	417	270
1	320	310	7
2	185	187	220
3	110	117	216
4	32	29	195
5	153	153	241
6	27	24	190
H= 1, K= 3			
0	165	163	90
1	112	109	257
2	53	62	311
3	106	104	220
4	146	139	40
5	117	119	281
6	142	145	2
H= 1, K= 4			
0	539	539	270
1	143	134	108
2	207	192	307
3	96	92	83
4	30	30	358
5	132	138	53
6	44	48	81
7	27	30	91
H= 1, K= 5			
0	190	185	270
1	249	250	359
2	72	76	314
3	66	66	311
4	45	37	18
5	52	51	159
6	64	68	21
H= 1, K= 6			
0	121	125	270
1	116	111	215
2	103	105	202
3	98	87	138
4	65	64	217
5	73	76	60
6	25	21	142
H= 1, K= 7			
0	59	62	270
1	175	174	341
2	120	122	16
3	110	110	338
4	35	35	324
5	35	35	191
6	7	13	109
H= 1, K= 8			
0	127	120	90
1	49	47	90
2	112	115	48
3	107	105	93
4	27	26	22
5	82	82	91
H= 1, K= 9			
0	81	80	90
1	96	89	73
2	79	82	175
3	66	68	21
4	80	82	186
H= 1, K= 10			
0	99	102	90
1	32	33	305
2	86	95	57
3	31	30	67
H= -1, K=-11			
-0	60	61	90
H= 1, K= 11			
1	41	42	268
2	71	73	183
H= 2, K= 0			
0	268	283	0
1	158	160	270
2	457	460	0
3	135	134	270
4	116	115	180
5	45	45	90
6	60	59	180
H= 2, K= 1			
0	631	624	0
1	708	713	115
2	459	460	92
3	166	165	162
4	300	294	100
5	96	97	288
6	137	141	114
H= 2, K= 2			
0	979	968	0
1	658	657	57
2	193	192	324
3	96	87	215
4	97	94	278
5	85	83	207
6	101	103	164
H= 2, K= 3			
0	101	103	180
1	517	514	72
2	39	40	197
3	204	204	29
4	59	54	159
5	90	92	268
6	40	40	352
H= 2, K= 4			
0	130	128	180
1	258	249	181
2	73	73	113
3	284	278	186
4	46	47	274
5	182	185	183
6	17	18	55
7	22	28	146
H= 2, K= 5			
0	94	95	180
1	260	262	98
2	125	126	194
3	139	133	118
4	169	164	325
5	80	80	244
6	70	70	304
H= 2, K= 6			
0	305	303	180
1	109	110	224
2	152	156	153
3	161	162	162
4	15	14	299
5	92	90	160
6	44	43	18
H= 2, K= 7			
0	126	123	180
1	69	73	107
2	170	177	254
3	87	86	184
4	141	141	264
5	20	20	257
H= 2, K= 8			
0	164	161	180
1	64	64	296
2	87	81	203
3	51	50	141
4	41	44	241
5	29	32	343
H= 2, K= 9			
0	134	130	0
1	137	134	287
2	79	77	281
3	49	52	294
4	52	51	249
H= 2, K= 10			
0	75	79	180
1	31	33	29
2	81	84	193
3	110	109	17
H= -2, K=-11			
-0	60	62	180
H= 2, K= 11			
1	83	79	295
2	4	2	90
H= 3, K= 0			
1	463	483	270
2	579	589	180
3	180	181	270
4	129	134	180
5	185	187	270
6	13	10	180
H= 3, K= 1			
0	374	375	90
1	798	829	187
2	109	98	221
3	102	101	126
4	94	91	346
5	115	119	336
6	103	103	351
H= 3, K= 2			
0	36	29	90
1	121	123	108
2	273	275	198
3	201	195	263
4	106	103	219
5	102	95	336
6	55	55	157
H= 3, K= 3			
0	82	86	90
1	291	280	355
2	140	132	73
3	201	198	263
4	174	180	331
5	95	97	225
6	76	78	348
H= 3, K= 4			
0	256	253	270
1	212	204	197
2	248	237	280
3	111	108	143
4	103	98	333
5	73	75	332
6	59	63	61
7	34	37	3
H= 3, K= 5			
0	71	60	270
1	242	236	6
2	301	293	5
3	132	131	290
4	221	215	22
5	56	56	186
6	48	51	7
H= 3, K= 6			
0	225	227	270
1	155	154	100
2	135	131	297
3	154	148	108
4	61	61	99
5	140	145	73
6	72	71	78
H= 3, K= 7			
0	30	24	270
1	125	128	313
2	46	50	244
3	152	156	301
4	23	15	315
5	64	61	300
H= 3, K= 8			
0	49	45	90
1	48	51	332
2	137	141	15
3	74	69	108
4	23	26	14
5	42	43	100
H= 3, K= 9			
0	76	73	270
1	94	90	10
2	87	89	171
3	64	60	71
4	87	88	197
H= 3, K= 10			
0	158	166	90
1	74	75	117
2	85	90	75
3	37	40	57
H= 3, K= 11			
0	55	52	270
1	56	56	74
H= 4, K= 0			
0	337	348	0
1	42	37	90
2	177	107	0
3	5	4	90
4	122	116	0
5	76	78	90
6	83	81	180
H= 4, K= 1			
0	198	199	180
1	501	498	263
2	254	249	55
3	33	37	82
4	264	261	94
5	92	89	77
6	71	74	54
H= 4, K= 2			
0	184	181	0
1	545	545	176
2	319	317	325
3	321	316	190
4	56	61	267
5	134	135	188
6	101	101	137
H= 4, K= 3			
0	201	203	180
1	393	389	106
2	230	222	190
3	124	120	136
4	115	114	16
5	12	11	153
6	40	43	37
H= 4, K= 4			
0	187	177	180
1	293	286	87
2	164	160	132
3	118	113	209
4	81	82	152
5	130	127	219
6	28	29	46
H= 4, K= 5			
0	115	114	180
1	290	289	81
2	129	126	282
3	25	24	343
4	102	106	265
5	27	31	263
6	49	51	300
H= 4, K= 6			
0	93	94	180
1	88	83	183
2	188	183	159
3	101	102	144
4	43	48	320
5	43	41	188
6	43	46	321
H= 4, K= 7			
0	100	101	0
1	32	34	105
2	40	37	237
3	34	32	159
4	101	96	263
5	30	32	198
H= 4, K= 8			
0	187	186	180
1	137	138	253
2	141	140	201
3	60	62	49
4	47	50	249
5	67	65	62
H= 4, K= 9			
0	85	88	180
1	112	111	274
2	56	58	269
3	63	65	243
4	98	99	256
H= 4, K= 10			
0	48	49	0
1	64	66	8
2	64	63	284
3	74	73	12
H= 4, K= 11			
0	20	17	0
1	67	68	278
H= 5, K= 0			
1	369	376	270
2	36	35	180
3	548	548	270
4	93	94	180
5	173	173	270
6	90	92	180
H= 5, K= 1			
0	277	273	90
1	362	372	190
2	263	260	67
3	149	141	248
4	120	120	320
5	39	42	289
6	59	60	271
H= 5, K= 2			
0	355	362	270
1	496	492	247
2	417	415	241
3	158	154	284
4	90	86	188
5	106	107	302
6	89	94	72
H= 5, K= 3			
0	14	16	90
1	138	139	182
2	179	180	337
3	138	136	267
4	199	194	23
5	33	24	286
6	38	38	345
H= 5, K= 4			
0	210	202	270
1	107	115	122
2	255	254	233
3	46	45	150
4	66	68	193
5	52	53	42
6	94	95	84
H= 5, K= 5			
0	49	46	270
1	252	256	284
2	229	226	358
3	164	159	304
4	305	301	353
5	105	106	293
H= 5, K= 6			
0	108	112	270
1	153	154	105
2	189	190	324
3	264	266	102
4	82	82	5
5	81	84	96
6	54	50	98
H= 5, K= 7			
0	53	57	270
1	187	180	14
2	14	17	169
3	38	40	286
4	28	21	6
5	43	47	128
H= 5, K= 8			
0	21	18	90
1	116	115	126
2	61	65	7
3	48	49	61
4	57	58	46
5	75	78	67
H8 525K=		94	270
2	60	59	229
3	30	27	95
4	45	48	161
H= 5, K= 10			
0	120	124	90
1	25	26	27
3	49	48	73
H= 5, K= 11			
0	22	21	270
1	25	24	44
H= 6, K= 0			
0	165	158	180
1	31	33	90
2	206	219	0
3	5	4	90
4	110	110	0
5	47	43	270
6	63	61	180
H= 6, K= 1			
0	428	435	180
1	255	250	339
2	399	392	129
3	152	151	24
4	253	254	69
5	104	108	60
6	40	42	58
H= 6, K= 2			
0	61	53	180
1	50	52	258
2	202	199	24
3	121	121	179
4	84	84	80
5	57	56	179
6	25	23	165
H= 6, K= 3			
0	166	169	0
1	59	48	252
2	342	340	312
3	147	142	113
4	18	15	211
5	89	91	100
6	61	60	141
7	45	46	293
H= 6, K= 4			
0	7	12	0
1	240	239	180
2	229	225	224
3	120	121	170
4	42	42	357
5	104	103	185
6	15	18	97
H= 6, K= 5			
0	64	64	0
1	248	245	111
2	137	139	286
3	136	131	178
4	129	127	266
5	67	70	209
6	50	51	214
H= 6, K= 6			
0	196	188	180
1	63	59	184
2	103	104	109
3	59	51	222
4	126	125	136
5	43	44	170
6	44	47	359
H= 6, K= 7			
0	107	106	180
1	52	56	90
2	16	10	327
3	45	42	282
4	99	100	279
H= 6, K= 8			
0	19	18	0
1	109	107	67
2	111	112	173
3	53	55	53
4	19	17	138
5	39	40	356
H= 6, K= 9			
0	28	25	180
1	134	141	256
2	50	56	181
3	32	30	256
4	45	44	280
H= 6, K= 10			
0	30	32	180
1	40	44	316
2	75	72	250
3	84	87	348
H= 6, K= 11			
0	6	6	180
H= 7, K= 0			
1	458	462	270
2	147	140	180
3	339	353	270
4	217	217	180
5	109	105	270
6	15	19	0
H= 7, K= 1			
0	317	314	90
1	368	375	167
2	312	317	30
3	174	179	155
4	140	137	347
5	34	33	321
6	88	91	272
H= 7, K= 2			
0	69	66	90
1	167	170	237
2	46	45	293
3	258	249	265
4	87	88	242
5	120	124	259
6	22	24	116
H= 7, K= 3			
0	286	280	270
1	271	260	223
2	463	460	8
3	115	105	40
4	304	306	3
5	81	75	337
6	54	54	23
H= 7, K= 4			
0	304	301	270
1	84	83	142
2	273	270	277
3	64	66	88
4	30	25	205
5	38	36	160
6	69	70	108
H= 7, K= 5			
0	58	63	270
1	137	136	328
2	101	103	315
3	248	253	278
4	117	113	19
5	24	27	205
6	65	62	24
H= 7, K= 6			
0	231	230	270
1	151	152	160
2	146	147	297
3	115	115	61
4	107	107	35
5	76	81	52
6	80	81	73
H= 7, K= 7			
0	88	86	90
1	202	208	338
2	56	55	311
3	90	86	64
4	63	63	331
5	40	38	210
H= 7, K= 8			
0	21	26	90
1	89	89	95
2	53	49	150
3	137	139	86
4	20	20	15
H= 7, K= 9			
0	51	52	270
1	93	93	26
2	52	53	213
4	65	66	181
H= 7, K= 10			
0	86	90	90
2	55	54	87
H= 8, K= 0			
0	231	244	0
1	171	171	270
2	236	237	0
3	6	0	90
4	56	61	0
5	10	10	270
6	27	29	180
H= 8, K= 1			
0	148	146	0
1	273	274	57
2	165	162	63
3	180	181	39
4	113	114	95
5	53	60	311
6	74	72	108
H= 8, K= 2			
0	67	62	180
1	370	357	249
2	163	154	108
3	36	36	157
4	146	147	51
5	74	76	141
6	10	4	296
7	36	39	109
H= 8, K= 3			
0	79	69	180
1	55	63	40
2	138	136	58
3	102	98	119
4	19	16	138
5	22	21	83
6	34	33	168
H= 8, K= 4			
0	66	67	180
1	38	43	132
2	139	141	55
3	203	206	164
4	199	197	119
5	104	102	176
H= 8, K= 5			
0	179	175	0
1	75	71	90
2	206	206	334
3	101	104	71
4	93	91	266
5	7	12	52
6	76	80	197
H= 8, K= 6			
0	22	15	180
1	170	173	86
2	196	192	203
3	44	40	187
4	28	28	109
5	70	69	219
H= 8, K= 7			
0	84	87	0
1	79	79	191
2	146	139	309
3	63	64	202
4	101	102	247
5	34	35	203
H= 8, K= 8			
0	139	139	180
1	60	62	79
2	9	8	82
3	75	83	337
H= 8, K= 9			
0	88	87	180
1	53	55	229
2	46	55	183
3	49	52	208
H= 8, K= 10			
1	45	44	61
2	44	47	179
H= 9, K= 0			
1	272	273	270
2	65	62	180
3	389	391	270
4	81	80	180
5	165	171	270
6	5	6	180
H= 9, K= 1			
0	71	71	270
1	200	198	195
2	176	176	50
3	117	116	187
4	28	20	63
5	63	59	19
6	27	28	298
H= 9, K= 2			
0	33	36	270
1	76	68	320
2	127	122	267
3	229	226	262
4	132	130	227
5	111	115	237
6	23	19	184
H= 9, K= 3			
0	97	98	90
1	140	138	158
2	190	191	343
3	95	98	209
4	129	127	1
5	38	34	323
6	87	88	344
H= 9, K= 4			
0	416	418	270
1	196	204	156
2	207	202	323
3	220	224	91
4	105	103	24
5	48	46	38
6	64	64	95
H= 9, K= 5			
0	36	35	270
1	94	94	329
2	160	154	356
3	122	118	57
4	88	87	342
5	55	54	273
6	47	45	18
H= 9, K= 6			
0	198	201	270
1	86	84	145
2	72	77	260
3	27	35	38
4	33	31	39
5	71	74	74
H= 9, K= 7			
0	117	117	270
1	126	124	2
2	118	119	338
3	117	119	307
4	53	53	11
5	34	35	178
H= 9, K= 8			
0	72	76	90
1	49	51	344
2	61	59	139
3	92	97	77
4	30	30	9
H= 9, K= 9			
0	7	1	270
1	41	44	355
2	78	84	214
3	64	63	52
H= -0, K=-10			
-0	92	91	270
H= 9, K= 10			
1	63	63	23
2	57	58	92
H= 10, K= 0			
0	417	426	0
1	584	609	270
2	221	220	0
3	61	59	270
4	24	17	0
5	169	175	90
6	44	45	180
H= 10, K= 1			
0	44	48	0
1	150	147	24
2	136	139	42
3	192	190	3
4	48	40	79
5	80	80	351
6	79	80	99
H= 10, K= 2			
0	158	157	0
1	20	13	18
2	40	40	345
3	190	191	175
4	30	26	102
5	87	85	176
6	14	17	106
H= 10, K= 3			
0	278	279	0
1	153	153	85
2	131	125	63
3	158	157	56
4	67	69	67
5	53	52	313
H= 10, K= 4			
0	31	19	180
1	85	65	196
2	160	155	133
3	121	114	191
4	88	91	75
5	102	104	185
6	27	26	61
H= 10, K= 5			
0	232	233	0
1	138	136	144
2	107	194	348
4	120	124	210
5	41	41	221
6	66	73	219
H= 10, K= 6			
0	196	201	180
1	66	68	85
2	134	139	100
3	63	59	144
4	122	124	107
5	60	57	191
H= 10, K= 7			
0	83	85	0
1	23	21	340
2	119	127	245
3	20	19	143
4	75	75	251
5	49	50	201
H= 10, K= 8			
0	110	109	180
1	113	115	64
2	77	78	201
3	22	26	344
4	10	11	283
H= 10, K= 9			
0	62	64	180
1	37	39	276
3	31	29	267
H= 10, K= 10			
0	23	27	180
1	72	48	84
H= 11, K= 0			
1	105	101	90
2	68	63	180
3	110	108	270
4	44	42	180
5	107	105	270
6	23	20	180
H= 11, K= 1			
0	149	145	90
1	223	217	208
2	15	13	142
3	123	128	192
4	76	79	30
5	47	45	341
6	60	58	335
H= 11, K= 2			
0	326	330	270
1	53	48	326
2	55	54	277
3	47	51	175
4	45	48	97
5	71	70	259
H= 11, K= 3			
0	126	124	90
1	46	42	278
2	120	115	28
3	126	126	268
4	92	95	305
5	81	81	281
6	66	65	0
H= 11, K= 4			
0	111	112	270
1	72	74	179
2	123	120	302
3	146	141	254
4	79	79	314
5	39	42	35
6	7	10	346
H= 11, K= 5			
0	145	141	270
1	172	171	50
2	145	146	320
3	36	35	39
4	72	69	10
5	34	37	229
H= 11, K= 6			
0	76	73	270
1	104	100	191
2	37	42	213
3	150	148	102
4	19	25	203
5	62	63	52
H= 11, K= 7			
0	136	135	270
1	77	75	358
2	62	62	258
3	64	67	16
H= 11, K= 8			
0	5	2	270
1	69	72	46
2	56	61	35
4	43	49	19
H= 11, K= 9			
0	38	37	270
1	37	41	346
2	17	17	108
3	35	37	25
H= 11, K= 10			
0	56	60	90
1	65	70	339
H= 12, K= 0			
0	258	259	0
1	218	220	270
2	40	33	180
3	86	89	270
4	16	6	180
5	82	79	90
H= 12, K= 1			
0	202	200	0
1	72	71	77
2	202	197	83
3	192	186	14
4	163	159	89
5	78	76	354
H= 12, K= 2			
0	274	278	0
1	173	170	268
2	214	218	17
3	63	59	286
4	37	32	150
5	107	110	131
6	56	56	137
H= 12, K= 3			
0	127	132	0
1	269	271	89
2	98	106	331
3	62	59	87
4	59	57	228
5	78	80	304
6	12	19	135
H= 12, K= 4			
0	118	119	180
1	62	61	90
2	166	163	106
3	133	130	171
4	101	103	78
5	84	82	189
H= 12, K= 5			
0	291	292	0
1	138	134	91
2	20	23	299
3	31	29	138
4	38	40	261
5	45	42	237
H= 12, K= 6			
0	125	124	180
1	113	113	120
2	102	106	142
3	108	110	168
4	42	39	81
5	41	39	241
H= 12, K= 7			
0	76	73	0
1	26	28	144
2	81	80	319
3	73	79	149
4	71	76	227
H= 12, K= 8			
0	77	78	180
1	55	49	116
2	63	69	164
3	27	27	77
H= 12, K= 9			
1	113	107	278
2	78	76	227
H= 13, K= 0			
1	109	111	90
2	194	198	180
3	93	96	90
4	121	117	180
5	106	103	270
6	50	51	180
H= 13, K= 1			
0	219	225	90
1	210	217	144
2	82	79	80
3	54	51	262
4	59	56	302
5	20	21	317
6	49	48	323
H= 13, K= 2			
0	208	211	270
1	59	61	177
2	161	162	244
3	73	71	223
4	41	39	116
5	31	25	346
6	41	42	130
H= 13, K= 3			
0	13	11	270
1	77	75	24
2	94	95	224
3	42	48	151
4	60	56	56
5	61	65	233
H= 13, K= 4			
0	100	104	270
1	180	174	219
2	87	85	313
3	86	84	206
4	45	41	320
H= 13, K= 5			
0	50	57	270
1	72	70	329
2	62	57	350
3	150	149	286
4	83	81	7
5	66	63	278
H= 13, K= 6			
0	69	69	90
1	97	99	114
2	92	92	0
3	56	54	101
H= 13, K= 7			
0	89	90	270
1	76	74	354
2	21	27	222
3	44	44	317
4	30	35	239
H= 13, K= 8			
0	21	23	90
1	44	45	357
3	39	41	87
H= 13, K= 9			
0	71	69	270
1	61	62	49
2	46	48	238
H= 14, K= 0			
0	205	210	0
1	68	66	270
2	279	287	0
3	59	59	270
4	60	56	180
5	50	49	90
6	49	50	180
H= 14, K= 1			
0	47	49	180
1	50	49	59
2	55	47	211
3	103	103	337
4	88	83	79
5	91	90	348
6	46	53	81
H= 14, K= 2			
0	168	174	0
1	146	142	249
2	134	130	81
4	76	80	77
5	82	84	155
H= 14, K= 3			
0	226	229	0
1	225	221	95
2	123	118	51
3	48	52	1
4	88	83	98
H= 14, K= 4			
0	60	63	180
1	76	79	151
2	49	47	82
3	37	38	145
4	54	52	101
5	51	50	180
H= 14, K= 5			
0	60	61	0
1	123	122	84
2	68	64	13
3	51	50	126
4	37	36	248
5	26	30	194
H= 14, K= 6			
0	111	110	180
1	86	79	127
2	108	111	140
3	37	37	174
4	34	36	62
H= 14, K= 7			
0	53	51	0
1	5	9	151
2	46	40	271
3	66	65	192
H= 14, K= 8			
0	99	100	180
2	63	58	171
H= 14, K= 9			
0	38	37	180
1	31	37	201
H= 15, K= 0			
1	114	111	270
2	89	81	180
3	44	38	270
4	109	102	180
5	36	39	270
H= 15, K= 1			
0	259	259	90
1	131	126	222
2	154	146	135
3	15	17	124
4	36	36	262
5	71	72	255
H= 15, K= 2			
0	104	110	270
1	125	125	174
2	105	100	217
3	7	12	196
4	64	63	194
H= 15, K= 3			
0	57	62	270
1	31	35	187
2	38	33	127
3	118	115	278
4	86	82	16
5	39	42	255
H= 15, K= 4			
0	36	29	270
1	74	77	157
2	103	104	237
3	48	51	117
4	22	21	225
5	42	42	54
H= 15, K= 5			
0	106	108	270
1	151	152	314
2	67	72	269
3	148	147	271
H= 15, K= 6			
0	12	10	270
1	73	70	196
2	106	107	339
3	38	33	178
4	64	65	0
H= 15, K= 7			
0	28	26	270
1	47	46	15
2	41	43	268
3	29	27	344
H=-15, K= -8			
-0	62	58	270
H= 15, K= 8			
1	45	41	114
2	73	72	34
H= 16, K= 0			
0	156	153	0
1	122	121	270
2	30	24	0
3	5	5	270
4	49	45	0
5	16	19	90
H= 16, K= 1			
0	46	43	180
1	224	221	70
2	45	50	14
3	127	123	12
4	97	95	106
5	62	64	326
H= 16, K= 2			
0	13	6	180
1	60	60	230
2	102	103	344
3	21	17	334
4	48	45	107
5	27	24	172
H= 16, K= 3			
0	68	68	0
1	94	95	77
2	66	62	31
3	31	26	172
4	34	33	35
5	16	14	320
H= 16, K= 4			
0	51	58	0
1	75	74	133
2	55	56	82
3	100	100	194
H= 16, K= 5			
0	67	67	0
1	75	73	116
2	72	65	356
3	61	56	164
4	20	19	275
H= 16, K= 6			
0	78	79	180
1	33	35	109
2	93	95	130
3	29	28	102
H= 16, K= 7			
0	18	18	180
1	45	48	184
2	50	49	267
3	30	36	197
H= 16, K= 8			
0	13	17	180
1	29	26	125
H= 17, K= 0			
1	65	69	90
2	97	88	180
3	31	32	270
4	143	134	180
5	7	7	270
H= 17, K= 1			
0	80	78	90
1	56	56	164
2	155	151	46
3	61	57	236
4	51	50	354
5	24	23	160
H= 17, K= 2			
0	31	29	90
1	146	146	211
2	93	90	204
3	32	26	300
4	102	102	191
5	57	64	310
H= 17, K= 3			
0	61	59	90
1	120	119	255
2	81	83	10
3	106	107	273
H= 17, K= 4			
0	117	113	270
1	122	125	170
2	60	55	267
3	32	30	159
4	16	16	328
H= 17, K= 5			
0	51	49	270
1	72	74	285
2	58	62	194
4	13	14	72
H= 17, K= 6			
0	7	5	270
1	40	35	172
3	21	22	115
H= 17, K= 7			
0	56	57	270
1	103	131	333
2	47	49	281
H= 18, K= 0			
0	77	71	0
1	165	161	270
2	98	98	0
3	6	2	270
H= 18, K= 1			
0	135	137	0
1	66	66	330
2	51	50	68
3	49	43	32
H= 18, K= 2			
0	11	15	0
1	23	28	279
2	23	19	127
3	64	59	194
4	63	64	65
H= 18, K= 3			
0	49	45	180
1	40	39	37
2	51	54	3
3	66	67	44
4	24	23	117
H= 18, K= 4			
0	5	4	180
1	33	33	124
2	84	80	104
4	84	97	96
H= 18, K= 5			
0	29	30	0
1	42	42	119
2	68	71	323
3	36	38	150
H= 18, K= 6			
1	74	75	89
2	27	26	109
H= 18, K= 7			
0	10	9	183
1	38	39	172
H= 19, K= 0			
1	104	99	270
2	109	106	180
3	76	78	270
4	98	100	183
H= 19, K= 1			
0	111	112	90
1	45	42	307
2	83	84	63
3	67	66	209
4	21	21	340
H= 19, K= 2			
0	116	121	270
1	44	51	114
2	86	82	202
3	57	53	236
4	85	81	190
H= 19, K= 3			
0	11	8	270
1	92	93	222
2	86	84	32
3	76	74	269
H= 19, K= 4			
0	20	16	270
1	114	115	141
2	62	59	335
3	20	22	105
H= 19, K= 5			
0	32	35	270
2	60	58	319
H= 19, K= 6			
0	34	34	270
1	70	70	160
H= 20, K= 0			
0	105	107	180
1	82	77	270
2	40	40	180
3	63	59	270
H= 20, K= 1			
0	12	12	0
1	33	36	320
2	66	66	38
3	81	77	18
H= 20, K= 2			
0	79	79	0
1	58	61	211
2	60	62	76
3	58	57	184
H= 20, K= 3			
0	119	125	0
1	35	37	13
3	65	58	70
H= 20, K= 4			
0	7	1	180
2	45	50	86
H= 20, K= 5			
0	43	41	0
1	40	37	218
2	34	42	351
H= 21, K= 0			
1	20	19	90
3	64	63	270
H= 21, K= 1			
0	75	77	90
1	49	48	206
2	68	68	52
3	41	43	207
H= 21, K= 2			
0	28	33	90
1	53	55	236
2	26	22	219
3	22	30	202
H= 21, K= 3			
1	37	34	345
2	47	44	8
H= 21, K= 4			
0	57	56	270
1	45	44	172
2	6	6	240
H= 21, K= 5			
0	43	52	270
H= 22, K= 0			
0	32	30	180
1	49	47	270
2	17	17	180
H= 22, K= 1			
0	5	5	0
1	39	43	6
2	17	13	106
H= 22, K= 2			
0	23	24	180
1	93	89	265
2	50	60	73
H= 22, K= 3			
0	69	66	3
1	44	42	160
H= 23, K= 0			
1	9	10	270
H= 23, K= 1			
0	76	75	90
1	10	14	289
H= 23, K= 2			
0	45	57	270

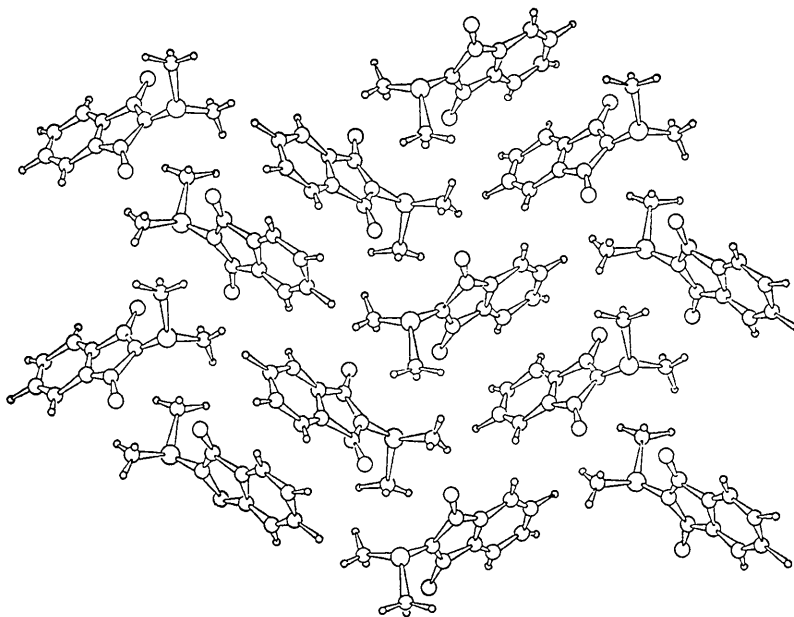
Fig. 3. Projection of the structure along [001]. The a-axis runs horizontally.

Table 5. *Least-squares planes*

The normal equations are given. Deviations are in Å. Planes are defined by atoms listed before slash (/)

(1) $10{\cdot}42267X + 5{\cdot}57447Y + 3{\cdot}22619Z = 4{\cdot}59399.$

    Deviations: 10, $-0{\cdot}042$; 1, $-0{\cdot}0411$; 9, $-0{\cdot}032$; 8, $-0{\cdot}012$; 7, $0{\cdot}031$; 6, $0{\cdot}059$; 5, $0{\cdot}035$; 4, $-0{\cdot}015$; 3, $-0{\cdot}037$; 11, $-0{\cdot}0882$; 2, $-0{\cdot}017$; 12, $0{\cdot}157/13$, $-1{\cdot}245$; 14, $1{\cdot}454$.

(2) $9{\cdot}93455X + 5{\cdot}75149Y + 3{\cdot}25287Z = 4{\cdot}48345.$

    Deviations: 4, $-0{\cdot}004$; 5, $0{\cdot}001$; 6, $0{\cdot}002$; 7, $-0{\cdot}003$; 8, $0{\cdot}001$; 9, $0{\cdot}003/10$, $0{\cdot}071$; 1, $0{\cdot}042$; 2, $0{\cdot}070$; 3, $0{\cdot}007$; 11, $-0{\cdot}054$; 12, $0{\cdot}293$.

(3) $11{\cdot}65258X + 5{\cdot}30473Y + 2{\cdot}97112Z = 4{\cdot}37126.$

    Deviations: 1, $0{\cdot}0178$; 2, $-0{\cdot}018$; 3, $0{\cdot}039$; 10, $-0{\cdot}020$; 11, $-0{\cdot}032$; 12, $0{\cdot}013/.$

(4) $11{\cdot}53119X + 5{\cdot}38456Y + 2{\cdot}95839Z = 4{\cdot}35742$

    Deviations: 1, $0{\cdot}000$; 3, $0{\cdot}000$; 12, $0{\cdot}000/2$, $-0{\cdot}041$; 10, $-0{\cdot}025$; 11, $-0{\cdot}079$; 9, $0{\cdot}119$; 4, $0{\cdot}133$.

age 1·43 Å and are significantly shorter than the corresponding distances of 1·53 Å found in triketoindane (Bolton, 1965). The two > C=O distances in the ylid average 1·23 Å and appear to be somewhat longer than the usual > C=O distance of 1·215 Å. Comparison of the C–S distances of 1·71 Å in the present work and 1·72 Å in 2-dimethylsulfuranylidenemalononitrile (Christensen & Witmore, 1969) with the > C=S bond lengths found in thiourea (Truter & Kunchur, 1958) and thioacetamide (Truter, 1960) of 1·71 and 1·713 Å respectively, leads to the conclusion that there is an appreciable amount of double-bond character in the

C–S bond of the two sulfoniumylids investigated. It is also interesting to note the similarity in the two C–S ylid bond distances.

The above results indicate that the unshared electrons on the 'ylid carbanion' (using the terminology of Johnson, 1966) are delocalized into the $3d$ orbitals of the sulfur atom (Johnson, 1966, and references cited therein) as well as into the 1,3-dione system. The structure seems to be best described by the resonance forms illustrated in Fig. 2. The arrangement around the sulfur atom is pyramidal with the sulfur atom sitting 0·73 Å from the plane through atoms C(11), C(13) and C(14). The two S–CH$_3$ distances average 1·785 Å and appear to be shorter than the accepted C–S single-bond distance of 1·81 Å.

The two angles 4–5–6 and 7–8–9 average 117·6° and are significantly smaller than the other angles in the benzene ring; similar observations can be made in the structures of triketoindane (Bolton, 1965) and phthal-imidocyclohexane (Groth, 1969). This angular distortion can be explained by the strain due to fusion of the five-membered ring.

All packing contacts are normal; the packing is illustrated in Fig. 3.

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Crystal and Molecular Structure of S,S-Diphenyl-N-p-tolylsulphonyl Sulphilimine*

BY A. KÁLMÁN†

Department of Chemistry, University of Manchester Institute of Science and Technology, Manchester M60 1QD, England

B. DUFFIN

Unilever Research Laboratory, Port Sunlight, Cheshire, England

AND Á. KUCSMAN

Institute of Organic Chemistry, L. Eötvös University, Budapest, Hungary

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The crystal structure of S,S-diphenyl-N-p-tolylsulphonyl sulphilimine has been solved by the symbolic addition method and refined by least-squares analysis of three-dimensional data collected by the multiple-film technique to the final residual, $R = 0.095$. The S–N(sp^2) bond lengths (1·598 and 1·628 Å) indicate a π-bond system delocalized to the S–N–S moiety. The S–N–S bond angle of 113·4° is considerably less than the theoretical 120°. The S(VI)–C(11) distance of 1·756 Å is in a good agreement with the calculated 1·75 Å for the S(VI)–C(sp^2) single bond. Due to the different S(IV)–C(sp^2) π-bonding one of the S(IV)–C(sp^2) bonds is significantly shorter (1·769 Å) than the other (1·799 Å) and than the theoretical 1·80 Å value. The mean S(VI)–O distance is 1·432 Å. The conformation of the molecule, which is fairly similar to that of the S,S-dimethyl-N-methylsulphonyl sulphilimine, is discussed in detail.

Introduction

N-Sulphoacyl sulphilimines (RR'SNSO₂Q) containing different SN bonds are compounds of theoretical interest. In the course of the structure investigation of *N*-acyl sulphilimines, started by Kucsman, Ruff & Kapovits (1966),‡ the crystal structure of S,S-dimethyl-*N*-methylsulphonyl sulphilimine (DMMSS; Me₂SNSO₂Me) has been determined (Kálmán, 1967). On this basis the electronic structure and conformation of *N*-sulphoacyl sulphilimines were discussed in detail (Kucsman, Kálmán & Kapovits, 1967).§ In order to get more information about the fine-structure of sul-

philimines, the crystal and molecular structure of the S,S-diphenyl-*N*-p-tolylsulphonyl sulphilimine (DPTSS; Ph₂SNSO₂C₆H₄Me) of the S-aryl-*N*-arylsulphonyl type has been determined.

Experimental

DPTSS was prepared by the method of Kucsman, Kapovits & Balla (1962) and crystallized from ethanol. Infrared and melting point data showed that the product was of analytical purity. The crystals were colourless transparent needles the main axis of which was parallel to the monoclinic unique axis, *b*. The lattice parameters were determined from precession photographs and the density was measured by flotation.

Crystal data

$C_{19}H_{17}NO_2S_2$, $M = 355.48$. Monoclinic;

$a = 10.616 \pm 0.011$, $b = 10.540 \pm 0.006$,

$c = 15.931 \pm 0.013$ Å,

$\beta = 100.90 \pm 0.09°$;

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* This paper is to be regarded as part XI of the series: *The Bond System of the N-Acyl Sulphilimines*, published by Kucsman *et al.* (Part X: Mezey, 1970*a*).

† Permanent address: Central Research Institute for Chemistry Hungarian Academy of Sciences, Budapest, Pf. 114/17, Hungary.

‡ Part I of the series.

§ Part IV of the series.