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The Crystal Structure of 1,3-Dithiolane-2-thione (Ethylene Trithiocarbonate)

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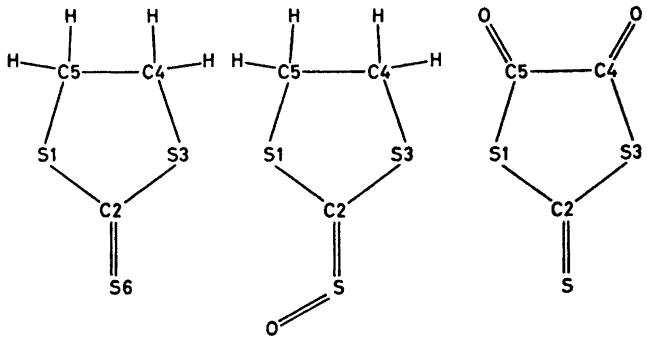
The crystals are monoclinic, space group $P2_1/c$, with cell dimensions $a = 8.63_9$ Å, $b = 7.18_0$ Å, $c = 9.23_2$ Å, and $\beta = 100.0_6°$. X-Ray intensity data were recorded on an automatic four-circle diffractometer. Full-matrix least-squares refinement gave $R_w = 5.5$ % ($R = 4.5$ %) for 1407 observed reflections and $R_w = 5.4$ % ($R = 4.8$ %) for 983 observed reflections with $\sin\theta/\lambda > 0.45$ Å^{-1}. The molecule is puckered with nearly C_2 symmetry, the torsional angle about the C–C bond being 44°. The bond lengths, corrected for thermal motion, are: 1.51_9 Å (C–C), 1.73_8 Å (C2–S1(3)) (average), 1.81_8 Å (C3(4)–S) (average), and 1.65_2 Å (C=S).

The conformations of several five-membered rings with hetero atoms have recently been studied by electron diffraction [1–4] and by spectroscopic methods [5] at this university. A more detailed IR and Raman investigation of 1,3-dithiolane-2-thione (I) is now being carried out.[6] The crystal structures of the related compounds 2-sulfinyl-1,3-dithiolane (II) [7] and 4,5-dioxo-2-thioxo-1,3-dithiolane (III) [8] have recently been determined, showing some



I II III

interesting differences in bond lengths. A structure determination of I was therefore of interest in connection with the spectroscopic study as well as for comparison to related compounds.

CRYSTAL DATA

Commercially obtained 1,3-dithiolane-2-thione was recrystallized from a mixture of methanol and butanol. Because of the relatively high vapour pressure (m.p. 36°C) the crystal was mounted in a capillary. The space group was found to be $P2_1/c$. The cell dimensions were determined on a manual four-circle diffractometer (CuK radiation) and found to be: $a = 8.6385(14)$ Å, $b = 7.1804(16)$ Å, $c = 9.2322(15)$ Å, and $\beta = 100.06(2)°$. The volume of the unit cell, which contains four molecules, is 563.9 Å^3 giving a calculated density of 1.60 g cm^{-3}.

1577 reflections with $2\theta < 60°$ were measured on an automatic four-circle diffractometer using Mo$K\alpha$ radiation (graphite monochromator) and $\omega/2\theta$ scan technique ($\mathbf{a}^* + \mathbf{c}^*$ parallel to the ϕ axis). 1408 reflections were recorded as observed, their intensities being greater than twice the standard deviations from counter statistics with a 2 % uncertainty in the measurement included.

The crystal dimensions were approximately $0.40 \times 0.35 \times 0.50$ mm, and the linear absorption coefficient is 1.11 mm^{-1}, giving a small variation in the applied transmission factor (0.724 to 0.755).

The program system for a CD 3300 computer described previously [9] was used in this investigation. The atomic form factors were those of Hanson *et al.*[10] (C and S) and of Stewart *et al.*[11] (H).

Table 1. Fractional atomic coordinates and thermal parameters with e.s.d.'s (multiplied by 10^5). The temperature factor is given by $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)]$. One isotropic temperature factor (Å^2) is given for all the hydrogen atoms.

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
S1	8232	7426	21042	1116	2448	1372	260	541	941
	8	13	9	12	20	11	17	13	19
C2	26027	−2974	28308	1217	1369	1043	114	367	27
	30	32	26	28	40	25	54	43	48
S3	41665	4412	20599	1119	2050	1446	228	637	652
	8	12	9	12	19	12	15	13	17
C4	30412	16816	5293	1578	2068	1233	213	923	402
	40	47	35	41	57	33	76	57	66
C5	16262	25099	10427	1607	2186	1453	770	922	885
	43	53	39	44	60	37	79	65	79
S6	27686	−17934	41996	1613	1990	1577	222	684	1222
	10	12	10	14	19	13	19	17	19
H4	27321	7173	−3526						
	393	431	347						
H4'	36364	26455	2601	6.11					
	387	493	321	0.37					
H5	19982	36465	17414						
	387	458	338						
H5'	7974	2899	1888						
	391	450	359						

Table 2. Observed and calculated structure factors. The five columns list values of h, k, l, 10 F_o, and 10 F_o.

h	k	l	Fo	Fc
0	0	12	60	- 55
0	0	10	145	137
0	0	8	231	-213
0	0	6	290	-267
0	0	4	256	240
0	0	2	559	-541
0	1	12	18	- 11
0	1	11	21	- 19
0	1	9	93	85
0	1	8	188	-177
0	1	7	201	-187
0	1	6	200	188
0	1	5	511	474
0	1	4	221	190
0	1	3	460	-425
0	1	2	869	843
0	1	1	132	120
0	2	12	66	- 62
0	2	11	69	63
0	2	10	71	67
0	2	8	75	- 74
0	2	7	73	74
0	2	6	187	169
0	2	5	45	39
0	2	4	434	393
0	2	3	558	-513
0	2	2	906	-870
0	2	1	397	379
0	2	0	257	258
0	3	12	24	- 22
0	3	11	54	50
0	3	10	71	67
0	3	8	124	-117
0	3	7	154	-139
0	3	6	370	352
0	3	5	10	- 8
0	3	4	497	-468
0	3	3	208	-182
0	3	2	104	105
0	3	1	310	299
0	4	11	41	41
0	4	10	64	59
0	4	9	136	-130
0	4	7	16	- 10
0	4	6	47	47
0	4	5	118	99
0	4	4	59	- 63
0	4	3	86	- 75
0	4	2	127	-118
0	4	1	532	523
0	4	0	108	101
0	5	10	65	61
0	5	9	72	- 69
0	5	8	120	-114
0	5	7	109	106
0	5	6	128	125
0	5	5	30	25
0	5	4	368	-359
0	5	3	63	67
0	5	2	114	115
0	5	1	133	-136
0	6	10	29	- 29
0	6	9	32	- 31
0	6	8	64	- 56
0	6	7	87	89
0	6	6	48	- 39
0	6	4	69	- 69
0	6	3	132	-128
0	6	2	191	191
0	6	1	100	96
0	6	0	19	19
0	7	8	47	- 49
0	7	7	80	78
0	7	6	105	98
0	7	5	101	-103
0	7	3	56	54
0	7	2	143	146
0	7	1	24	- 20
0	8	7	19	17
0	8	6	41	41
0	8	5	53	- 52
0	8	4	18	- 16
0	8	2	38	38
0	8	0	140	-149
0	9	5	51	- 51
0	9	4	27	- 32
0	9	3	41	42
0	9	1	14	8
1	0	-12	16	10
1	0	-10	81	75
1	0	-8	60	63
1	0	-6	156	143
1	0	-4	418	-392
1	0	-2	37	- 40
1	0	0	58	- 58
1	0	2	91	-101
1	0	4	323	353
1	0	6	125	-138
1	0	8	30	33
1	0	10	50	- 51
1	0	12	21	- 16
1	1	-12	43	41
1	1	-11	25	29
1	1	-10	31	29
1	1	-9	97	- 89
1	1	-8	64	- 58
1	1	-7	26	24
1	1	-6	136	-131
1	1	-5	196	182
1	1	-4	251	-231
1	1	-3	161	-143
1	1	-2	217	200
1	1	-1	792	762
1	1	0	79	- 84
1	1	1	751	-776
1	1	2	142	147
1	1	3	164	172
1	1	4	85	- 55
1	1	5	203	-217
1	1	6	101	-107
1	1	7	41	- 46
1	1	8	14	- 6
1	1	9	98	105
1	1	10	18	- 15
1	1	11	35	- 38
1	1	12	25	29
1	2	-12	16	17
1	2	-11	17	16
1	2	-10	59	- 52
1	2	-9	34	- 33
1	2	-8	83	- 80
1	2	-7	243	227
1	2	-6	74	62
1	2	-5	9	8
1	2	-4	81	- 65
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1	2	-1	424	-393
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1	2	3	108	117
1	2	4	52	55
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1	2	6	85	- 88
1	2	7	219	241
1	2	8	68	71
1	3	-11	59	55
1	3	-10	29	- 33
1	3	-9	17	20
1	3	-7	119	109
1	3	-6	63	56
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1	3	-3	423	-384
1	3	-2	204	184
1	3	-1	16	10
1	3	0	233	-228
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1	3	4	35	- 31
1	3	5	85	95
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1	3	7	19	16
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1	3	9	40	- 40
1	3	10	42	- 45
1	3	11	55	- 55
1	4	-11	40	39
1	4	-10	60	- 55
1	4	-9	74	- 69
1	4	-8	27	27
1	4	-6	20	- 25
1	4	-5	334	-316
1	4	-4	25	- 21
1	4	-3	184	178
1	4	-2	245	234
1	4	-1	204	190
1	4	0	112	-111
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1	4	2	87	94
1	4	3	214	220
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1	5	-6	33	27
1	5	-5	54	- 53
1	5	-4	87	88
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1	5	7	60	64
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1	6	-7	79	80
1	6	-6	21	- 25
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1	6	-4	63	- 52
1	6	-3	177	172
1	6	-2	86	- 87
1	6	-1	169	-168
1	6	0	137	-144
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1	6	3	36	40
1	6	4	18	16
1	6	5	12	- 10
1	6	6	58	61
1	6	7	77	83
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1	7	-7	51	48
1	7	-6	143	-138
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1	7	2	107	112
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1	8	-7	34	33
1	8	-6	65	- 66
1	8	-5	39	- 39
1	8	-4	70	70
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1	8	1	17	15
1	8	2	34	- 33
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1	9	-1	42	- 45
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1	9	1	41	44
1	9	2	14	- 16
1	9	3	16	18
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2	0	-8	58	- 57
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2	0	6	217	231
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2	1	-7	280	-273
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2	1	-5	212	206
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2	1	1	350	337
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2	1	4	381	402
2	1	5	204	215
2	1	6	42	42
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2	1	12	14	- 7
2	2	-12	19	- 15
2	2	-11	21	21
2	2	-10	64	60
2	2	-9	145	-137
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2	2	-7	39	- 42
2	2	-6	105	- 89
2	2	-5	41	42
2	2	-4	99	- 84
2	2	-3	59	62
2	2	-2	183	-174
2	2	-1	618	605
2	2	0	539	516
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2	2	3	76	78
2	2	4	73	- 73
2	2	5	176	-188
2	2	6	89	- 96
2	2	7	18	21
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2	2	10	32	34
2	3	-12	27	19
2	3	-11	65	- 60
2	3	-10	30	26
2	3	-9	39	36
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2	3	-4	193	-190
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2	4	3	282	297
2	4	4	93	-102
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2	4	6	107	111
2	4	7	30	- 35
2	4	8	73	- 77
2	5	-10	28	- 27
2	5	-9	42	40
2	5	-8	57	- 47
2	5	-7	34	- 35
2	5	-6	137	125
2	5	-5	150	-141
2	5	-4	57	58
2	5	-3	21	10
2	5	-2	101	98
2	5	-1	55	- 60
2	5	0	35	29
2	5	1	257	268
2	5	2	164	-174
2	5	3	50	- 54
2	5	4	21	22
2	5	5	35	- 40
2	5	6	108	-113
2	5	7	23	- 26
2	5	9	26	- 24
2	5	10	13	18
2	6	-10	18	- 20
2	6	-9	22	- 21
2	6	-8	84	77
2	6	-7	61	- 59
2	6	-6	50	45
2	6	-4	28	29
2	6	-3	88	- 84
2	6	-2	49	- 55
2	6	-1	199	201
2	6	0	216	-213
2	6	1	123	-125
2	6	2	39	43
2	6	3	30	- 34
2	6	4	94	- 95
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2	7	-9	19	- 18
2	7	-8	27	27
2	7	-6	24	- 16
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2	7	6	17	14
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2	7	8	23	- 28
2	8	-7	38	- 31
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2	8	-1	73	75
2	8	2	71	74
2	8	3	26	- 26
2	8	4	40	- 44
2	8	5	17	23
2	8	6	27	- 29
2	9	-5	21	19
2	9	-4	43	42
2	9	-2	20	- 18
2	9	0	42	44
2	9	1	53	- 51
2	9	2	49	- 54
3	0	-10	20	21
3	0	-8	402	-418
3	0	-6	384	388
3	0	-4	344	-329
3	0	-2	862	885
3	0	0	235	228
3	0	2	530	-497
3	0	4	345	360
3	0	6	390	-412
3	0	8	174	189
3	0	10	39	- 44
3	1	-12	19	- 16
3	1	-11	84	83
3	1	-10	98	- 97
3	1	-9	100	-100
3	1	-8	65	65
3	1	-7	127	120
3	1	-6	236	244
3	1	-5	28	27
3	1	-4	110	108
3	1	-3	500	-489
3	1	-2	213	208
3	1	-1	534	528
3	1	0	637	-597
3	1	1	546	-523
3	1	2	55	51
3	1	3	422	425
3	1	4	127	-135
3	1	5	31	23
3	1	6	60	61
3	1	7	48	- 49
3	1	8	49	58
3	1	9	57	58
3	1	10	21	- 24
3	1	11	59	- 61
3	2	-12	16	- 10
3	2	-11	39	39
3	2	-10	90	90
3	2	-9	82	- 81
3	2	-8	70	- 66
3	2	-7	109	113
3	2	-6	357	354
3	2	-5	391	-382
3	2	-4	534	-527
3	2	-3	261	262
3	2	-2	65	58

Table 2. Continued.

h	k	l	Fo	Fc
3	2	-1	131	125
3	2	0	21	-20
3	2	1	190	176
3	2	2	218	-212
3	2	3	301	312
3	2	4	383	398
3	2	5	249	-264
3	2	6	214	-229
3	2	7	37	39
3	2	8	98	103
3	2	9	96	-100
3	2	10	37	- 39
3	3	-12	65	64
3	3	-11	29	29
3	3	-10	57	- 56
3	3	-9	113	-110
3	3	-8	72	72
3	3	-7	19	22
3	3	-6	17	- 20
3	3	-5	40	36
3	3	-4	269	-261
3	3	-3	162	155
3	3	-2	553	531
3	3	-1	353	333
3	3	0	468	-449
3	3	1	221	-215
3	3	2	412	405
3	3	3	143	-144
3	3	4	212	-226
3	3	5	121	-124
3	3	7	70	- 70
3	3	8	61	63
3	3	9	77	81
3	3	10	48	- 51
3	3	11	15	12
3	4	-9	90	- 89
3	4	-7	233	221
3	4	-6	68	64
3	4	-5	128	-129
3	4	-4	77	- 69
3	4	-3	291	276
3	4	-2	10	- 12
3	4	-1	263	-260
3	4	0	75	- 78
3	4	1	300	-296
3	4	2	112	-117
3	4	3	126	134
3	4	4	113	118
3	4	5	118	-126
3	4	6	20	19
3	4	7	199	210
3	4	8	17	16
3	4	9	16	- 17
3	5	-11	22	- 19
3	5	-10	57	- 58
3	5	-9	24	24
3	5	-8	51	52
3	5	-7	90	87
3	5	-6	68	- 66
3	5	-5	82	- 76
3	5	-4	244	-233
3	5	-3	18	- 15
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8	4	5	28	- 24
8	4	6	30	- 32
8	5	-8	44	44
8	5	-6	13	- 1
8	5	-5	52	51
8	5	-4	64	61
8	5	-2	38	- 38
8	5	-1	71	74
8	5	0	57	- 59
8	5	1	93	- 93
8	5	2	23	19
8	5	4	25	- 27
8	6	-7	28	28
8	6	-6	16	12
8	6	-5	29	- 27
8	6	-4	44	- 47
8	6	-3	66	67
8	6	-2	64	- 59
8	6	-1	90	- 85
8	6	0	44	38
8	6	1	32	29
8	6	2	19	- 17
8	6	4	55	56
8	7	-4	26	- 26
8	7	-3	34	- 32
8	7	-2	45	44
8	7	-1	22	23
8	7	1	16	- 12
9	0	-10	55	- 60
9	0	-8	122	132
9	0	-6	133	-137
9	0	-4	214	-217
9	0	-2	171	-170
9	0	0	117	-114
9	0	2	93	86
9	0	4	98	- 97
9	0	6	134	130
9	1	-9	60	60
9	1	-8	17	10
9	1	-7	72	- 71
9	1	-5	56	- 60
9	1	-4	19	- 16
9	1	-3	117	122
9	1	-2	142	-141
9	1	-1	159	-155
9	1	0	102	98
9	1	1	191	186
9	1	2	18	8
9	1	3	86	- 83
9	1	4	71	69
9	2	-8	73	76
9	2	-7	100	-100
9	2	-6	155	-159
9	2	-5	130	137
9	2	-4	99	102
9	2	-3	30	- 28
9	2	-2	38	- 36
9	2	-1	76	75
9	2	0	40	37
9	2	2	106	101
9	2	3	89	- 86
9	2	4	128	-120
9	2	5	44	42
9	2	6	46	42
9	3	-9	36	43
9	3	-8	49	- 49
9	3	-6	27	- 31
9	3	-5	62	63
9	3	-4	104	104
9	3	-3	42	41
9	3	-2	123	-121
9	3	-1	113	-111
9	3	0	162	158
9	3	1	13	2
9	3	2	145	-138
9	3	3	25	- 27
9	3	4	39	41
9	4	-7	53	- 59
9	4	-6	13	- 13
9	4	-5	82	83
9	4	-4	40	40
9	4	-3	152	-153
9	4	0	20	- 19
9	4	1	48	45
9	4	2	33	34
9	4	3	33	- 31
9	4	4	21	- 18
9	5	-7	36	- 37
9	5	-6	13	18
9	5	-4	76	78
9	5	-3	35	- 34
9	5	-2	105	-100
9	5	-1	68	66
9	5	0	105	99
9	5	1	15	- 9
9	5	2	86	- 86
9	5	3	23	23
9	6	-5	53	51
9	6	-4	40	- 39
9	6	-3	23	- 26
9	6	-1	22	21
9	6	0	24	- 30
10	0	-8	57	- 56
10	0	-6	25	24
10	0	-4	73	73
10	0	0	77	74
10	0	2	74	- 70
10	1	-7	47	- 47
10	1	-6	65	66
10	1	-5	58	54
10	1	-3	74	- 70
10	1	-2	20	- 20
10	1	-1	15	7
10	1	0	33	- 29
10	1	1	43	39
10	1	2	35	- 38
10	1	3	40	- 37
10	1	4	22	18
10	2	-8	16	- 12
10	2	-7	20	- 25
10	2	-6	26	21
10	2	-5	19	16
10	2	-4	39	- 38
10	2	-3	46	- 45
10	2	-2	50	- 45
10	2	-1	89	87
10	2	0	43	39
10	2	1	20	- 17
10	2	2	21	- 22
10	2	3	22	21
10	2	4	25	27
10	3	-7	26	- 26
10	3	-6	23	25
10	3	-5	50	53
10	3	-4	33	- 30
10	3	-3	12	- 10
10	3	-2	29	26
10	3	-1	40	42
10	3	0	13	11
10	3	1	25	- 23
10	3	2	27	- 29
10	3	3	46	- 41
10	4	-6	45	44
10	4	-5	34	37
10	4	-4	42	- 48
10	4	-3	44	- 46
10	4	-2	12	4
10	4	-1	30	28
10	4	0	15	- 17
10	4	1	71	- 69
10	5	-4	52	- 54
10	5	-3	40	- 37
10	5	-2	30	32
10	5	-1	17	16
11	0	-6	64	- 72
11	0	-4	31	37
11	0	-2	33	- 36
11	0	0	38	37
11	0	2	37	35
11	1	-6	15	- 7
11	1	-5	14	- 17
11	1	-3	70	73
11	1	-1	86	- 88
11	1	0	32	32
11	1	1	40	41
11	1	2	38	- 36
11	2	-6	26	- 28
11	2	-5	33	31
11	2	-4	47	50
11	2	-3	73	- 75
11	2	0	25	- 27
11	3	-5	65	- 66
11	3	-4	14	16
11	3	-3	32	34
11	3	-2	44	- 45
11	3	-1	15	- 15
11	3	0	32	34
11	3	1	53	49
12	0	-2	128	-128

STRUCTURE DETERMINATION

The positions of the sulphur atoms were found from a three-dimensional sharpened Patterson map, and the first Fourier synthesis revealed the carbon atoms. The structure was refined by full-matrix least-squares technique including anisotropic thermal parameters for the carbon and sulphur atoms and one common isotropic B value for the hydrogens, to $R_w = 6.5$ % (conventional $R = 4.9$ %). The corresponding C – H distances varied from 0.85

to 1.32 Å, and the C–C distance seemed unreasonably small (1.474 Å). Inspection showed that one reflection (400) caused about 14 % of the weighted sum of squares of residuals, mainly because of the large weight, ΔF being 3.6 and $F_o = 5.4$. (The observed value may be due to systematic multiple reflection.) This reflection was discarded and a new least-squares refinement gave $R_w = 5.5$ % ($R = 4.5$ %). The C–H distances became normal and the C–C distance increased to 1.493 Å.

A third refinement keeping the hydrogen parameters fixed and using only the 983 reflections with sin $\theta/\lambda > 0.45$ Å^{-1} gave $R_w = 5.4$ % ($R = 4.8$ %). The corresponding atomic parameters are given in Table 1, and the observed and calculated structure factors in Table 2.

Table 3. Bond distances (Å) and bond angles (degrees) with e.s.d.'s in 1,3-dithiolane-2-thione and the related compounds (II) and (III). *a:* inner data included; *b:* including only reflections with sin $\theta/\lambda > 0.45$ Å^{-1}; *c:* the results in *b* corrected for thermal motion.

	a	*b*	*c*	II[7]	III[8]
S1C2	1.732 (2)	1.735 (3)	1.748	1.72 (1)	1.733 (5)
S3C2	1.722 (2)	1.717 (3)	1.727	1.73 (1)	1.738 (5)
S1C5	1.812 (3)	1.814 (4)	1.821	1.80 (2)	1.764 (5)
S3C4	1.808 (3)	1.804 (3)	1.814	1.81 (2)	1.757 (5)
C2S6	1.642 (2)	1.646 (2)	1.652	1.64 (1)	1.603 (5)
C4C5	1.493 (4)	1.508 (4)	1.519	1.50 (2)	1.503 (7)
S1C2S3	114.4 (1)	114.5 (1)	114.7	116.8 (7)	116.3 (3)
S1C5C4	107.8 (2)	107.5 (2)	107.5	110.0 (10)	113.4 (4)
S3C4C5	108.1 (2)	107.7 (2)	107.8	106.9 (10)	114.8 (4)
C2S1C5	96.6 (1)	96.5 (1)	96.4	94.7 (7)	97.9 (3)
C2S3C4	96.7 (1)	97.0 (1)	96.8	94.7 (7)	97.2 (3)
S1C2S6	122.4 (1)	122.0 (2)	122.1	121.7 (7)	121.4 (3)
S3C2S6	123.2 (1)	123.4 (2)	123.3	121.5 (8)	122.3 (3)

Table 3 gives structure parameters corresponding to the two last refinements. Parameters involving hydrogen atoms are given in Table 4 which also shows the torsional angles and the deviations of the atoms from a plane through S1, C2, and S3.

The results of an analysis of the atomic vibration are presented in Table 5. The direction cosines show that the principal axes of the thermal vibration ellipsoids for the S atoms are nearly parallel to the principal axes of inertia for the molecule. The root-mean-square discrepancy between "observed" atomic vibration tensor components and those calculated from a rigid-body model[12] is 0.0011 Å^2 indicating that the molecule may, to a close approximation, be treated as a rgid system. The r.m.s. translational amplitudes are nearly equal, the reduced values being 0.206 Å, 0.203 Å, and 0.184 Å The r.m.s. vibrational amplitudes are 7.6°, 4.8°, and 3.1°, the two first roughly along the principal axes I_1 and I_2 respectively, the last closely parallel to the I_3 axis (*cf.* Table 5).

Table 4. Additional structural parameters for 1,3-dithiolane-2-thione.

Some parameters involving hydrogen

C4H4	1.07 (3) Å
C4H4′	0.92 (3) »
C5H5	1.05 (3) »
C5H5′	1.01 (3) »
H4C4H4′	110.3 (2.4)°
H5C5H5′	110.7 (2.6)°

Atomic deviations (Å) from a plane through S1, C2, and S3

C4	−0.351
C5	0.317
S6	0.066
H4	−1.41
H4′	0.02
H5	1.36
H5′	−0.05

Torsional angles (degrees)

S1C2S3C4	−11.3 (2)
C2S3C4C5	33.9 (3)
S3C4C5S1	−43.8 (3)
C4C5S1C2	33.0 (3)
C5S1C2S3	−10.1 (2)
S6C2S3C4	171.4 (2)
S6C2S1C5	167.2 (2)

Table 5. The principal axes of the thermal vibration ellipsoids given by the components of a unit vector (e_x, e_y, e_z) in fractional coordinates, the corresponding root-mean-square amplitudes (in Å) and B values (in Å²). The direction cosines relating the ellipsoid axes to the principal axes of inertia ($I_1 = 156$ a.m.u. Å², $I_2 = 291$ a.m.u. Å², and $I_3 = 434$ a.m.u. Å²) are also given.

Atom	e_x	e_y	e_z	$(\overline{u^2})^{\frac{1}{2}}$	B	Direction cosines I_1	I_2	I_3
	0.017	0.108	0.069	0.277	6.05	−0.052	−0.077	0.996
S1	0.006	−0.087	0.085	0.212	3.54	0.998	−0.047	0.048
	0.116	−0.012	0.005	0.200	3.17	0.042	0.996	0.080
	0.106	0.023	−0.026	0.213	3.57	−0.324	0.944	0.070
C2	0.046	0.015	0.107	0.209	3.44	0.642	0.165	0.749
	0.022	−0.137	0.007	0.188	2.80	0.695	0.287	−0.659
	0.027	0.082	0.089	0.262	5.42	0.203	−0.014	0.979
S3	0.008	−0.112	0.065	0.214	3.61	0.979	0.031	−0.203
	−0.114	0.011	0.006	0.199	3.12	0.028	−0.999	−0.020
	0.085	0.066	0.069	0.256	5.19	0.125	0.536	0.835
C4	0.068	−0.113	0.003	0.228	4.12	0.537	0.671	−0.511
	0.044	0.049	−0.086	0.206	3.35	−0.835	0.512	−0.204
	0.066	0.085	0.072	0.282	6.30	0.057	0.352	0.934
C5	−0.088	0.011	0.057	0.225	4.00	0.367	−0.877	0.309
	0.042	−0.110	0.061	0.209	3.44	0.928	0.325	−0.179
	0.024	0.080	0.090	0.284	6.36	0.217	−0.034	0.976
S6	−0.115	0.012	0.002	0.241	4.59	0.002	−0.999	−0.036
	0.005	−0.113	0.064	0.194	2.96	0.976	0.009	−0.217

DISCUSSION

Table 3 includes bond distances and angles corrected for thermal motion (column *c*). The corrections were based on the coordinates obtained by excluding the low angle data to reduce possible effects of the valence electrons. Results (uncorrected) for the related compounds (II) and (III) are given for comparison.

The five-membered ring in 1,3-dithiolane-2-thione is puckered, and the molecule has nearly C_2 symmetry. The experimental torsional angles are given in Table 4, while a calculation by the Westheimer-Hendrickson method [13] gave $-11.5°$, $35.8°$, and $-47.3°$ for these parameters.*

The exocyclic C–S bond is somewhat longer than found in thioformaldehyde (1.611 Å).[14] Using the value from Table 3, column *c*, the amount of double bond character is about 86 % according to the relationship proposed by Abrahams.[15] The C–S bond in the planar ethylene thiourea is considerably longer (1.708 (7) Å) [16] which corresponds to a double bond character of only 65 % . The length of the exocyclic C–S bond in (III),[8] which is also nearly planar, corresponds to a pure double bond (Table 3). It is interesting to note that the exocyclic C–S bonds, as well as the ring bonds, are very similar in 1,3-dithiolane-2-thione and the corresponding oxide (II).[7]

The C2–S1 and C2–S3 bonds have some double bond character and are considerably shorter than the C(sp^3)–S bonds. This is in agreement with the force constants obtained by Henriksen *et al.*,[6] their values for the stretching force constants for the C2–S and C=S bonds being quite similar. The difference between the average lengths of the two C–S bond types in the ring is slightly larger than in methyl vinyl sulphide where 1.806(6) Å and 1.748(6) Å have been reported.[17]

The molecular packing is shown in Fig. 1. The shortest intermolecular S···H contact is about 2.84 Å and the shortest S···S contact 3.58 Å (S1···S6).

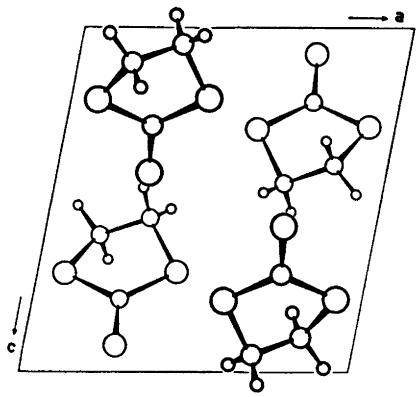

Fig. 1. The structure viewed along the *b* axis.

* Most of the necessary potential constants were the same as used for tetrahydrothiophene. (Table 1, column c of Ref. 3). The natural angle for S1C2S3 was taken as 118°, and the potential about the C2–S bonds neglected, since it is unknown and has only a minor effect on the results.

The other intermolecular distances are well above the sum of the van der Waals radii. It seems likely that the molecular structure in the gaseous phase is very similar to the structure found in the crystal.

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