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Crystal Structure of 3-Methyl-benzoxazoline-2-thione

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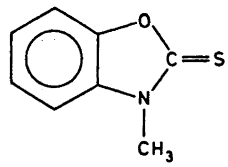
The crystals are monoclinic with space group $P2_1/c$ and cell dimensions $a = 9.24$ Å, $b = 6.90$ Å, $c = 12.86$ Å, $\beta = 99.0°$. There are four molecules in the unit cell. The structure was solved by direct methods and refined by full-matrix least squares technique. The final R-value is 7.7 % for 674 observed reflections (film data). The molecule is planar to within 0.02 Å with a C=S distance of 1.62_9 Å and a C(thionyl) – O distance of 1.39_3 Å.

The interactions of benzoxazolone, benzoxazolethione and their derivatives with fatty, aromatic and alkylaromatic amines have been studied by Simov *et al.*[1–4] The main features are: (1) the heteroatomic ring is opened and asymmetric di- or tri-substituted ureas or thioureas are obtained; (2) the products are derivatives of 2-aminobenzoxazole,[5] (3) no interactions take place; (4) substitution of H in NH by CH_3 reduces the interaction rate in the case of benzoxazolone, while it has the opposite effect for benzoxazolethione. In order to clarify these and some other characteristics, it is intended to carry out X-ray crystal structure analyses of some of these compounds. The structure determination of 3-methyl-benzoxazoline-2-thione is presented in this paper.



The crystals belong to the monoclinic system, the systematically absent reflections leading to the space group $P2_1/c$. Cell parameters, derived from Weissenberg and precession diagrams, are: $a = 9.24$ Å, $b = 6.90$ Å, $c = 12.86$ Å, and $\beta = 99.0°$. The number of molecules in the unit cell are four ($\varrho_{calc} = 1.35$ g cm^{-3}, $\varrho_{obs} = 1.34$ g cm^{-3}). Intensity data were obtained (at room temperature)

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by photometric measurements of equiinclination integrated Weissenberg diagrams corresponding to *h*0*l*,...,*h*4*l* and integrated 0*kl* precession diagrams. 674 independent reflections were strong enough to be measured. No corrections have been made for absorption or secondary extinction effects.

The structure was solved by direct methods [6]* and refined by full-matrix least squares techniques. The positions of the four benzene hydrogen atoms were calculated assuming C – H bond lengths of 1.03 Å, while methyl hydrogen atoms were localized in a difference Fourier map. Anisotropic temperature factors were introduced for sulphur, oxygen, nitrogen, and carbon atoms. Only the positional parameters were refined for the hydrogens. The weighting scheme in least squares refinement

$$\text{for } F_0 \leq \text{FB},\ W = \text{A1}(F_0)^{\text{B1}}$$
$$\text{for } F_0 > \text{FB},\ W = \text{A2}(F_0)^{\text{B2}}$$

was adapted by taking A1 = 10.0, A2 = 14.0, B1 = 0.0, B2 = −0.5 and FB = 2.0.

The atomic form factors used were those of Hanson *et al.*[7] except for hydrogen.[8] The *R*-value arrived at was 7.7 % for 674 observed reflections. A final difference Fourier map, calculated with the phases determined by the parameters corresponding to *R* = 7.7 %, contained no density fluctuations larger than ± 0.3 e.Å^{-3}.

Table 1. Fractional atomic coordinates and anisotropic thermal vibration parameters with estimated standard deviations (multiplied by 10^5).[a]

Atom	*x*	*y*	*z*	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
S	11530	40043	40299	01361	04240	00625	−00290	00731	−00078
	23	39	15	37	117	16	74	32	53
O	40122	39217	39546	01533	03199	00494	−00268	00212	−00043
	55	80	36	80	217	32	173	75	115
N	26381	38567	23825	01084	02475	00427	00051	00133	00066
	63	87	40	87	238	36	188	76	126
C_1	25757	39080	34273	01147	03192	00470	−00146	00137	−00271
	80	119	53	109	319	47	252	97	172
C_2	13572	38562	15454	00965	04908	00654	00289	00047	−00098
	91	184	66	114	425	57	311	114	234
C_3	49703	38843	31987	01136	03016	00670	00695	00506	00196
	83	116	59	119	329	54	262	112	189
C_4	64928	38828	33850	01304	02475	01045	00123	00104	00392
	94	127	78	132	327	78	264	148	227
C_5	71244	38289	24900	01469	03946	00883	00176	01096	00259
	106	138	75	147	401	73	316	158	243
C_6	62985	37717	15096	02231	01631	01158	00570	01874	00378
	109	127	82	185	349	89	309	199	228
C_7	47681	37517	13405	01300	02507	00721	00462	00728	00091
	88	118	64	126	316	57	275	122	189
C_8	41075	38210	22258	01277	02498	00496	−00315	00575	−00051
	78	111	51	120	298	45	246	104	166

* All programs used are included in this reference.

[a] For numbering of atoms, see Fig. 1.

Table 2. Fractional atomic coordinates for hydrogen atoms with estimated standard deviations. H_m and $H_{m,n}$ are bonded to C_m.

Atom	x	y	z	B (Å²)
H_4	.713	.399	.418	4.5
	8	10	6	
H_5	.825	.387	.255	4.5
	9	10	6	
H_6	.680	.371	.076	4.5
	8	10	6	
H_7	.394	.360	.071	4.5
	8	11	6	
$H_{8,1}$	.185	.356	.082	4.5
	8	10	6	
$H_{8,2}$	.071	.495	.171	4.5
	8	13	6	
$H_{8,3}$	.057	.323	.169	4.5
	8	12	6	

Final fractional coordinates and thermal parameters with estimated standard deviations are given in Tables 1 and 2. The expression for anisotropic vibration is:

$$\exp\left[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)\right]$$

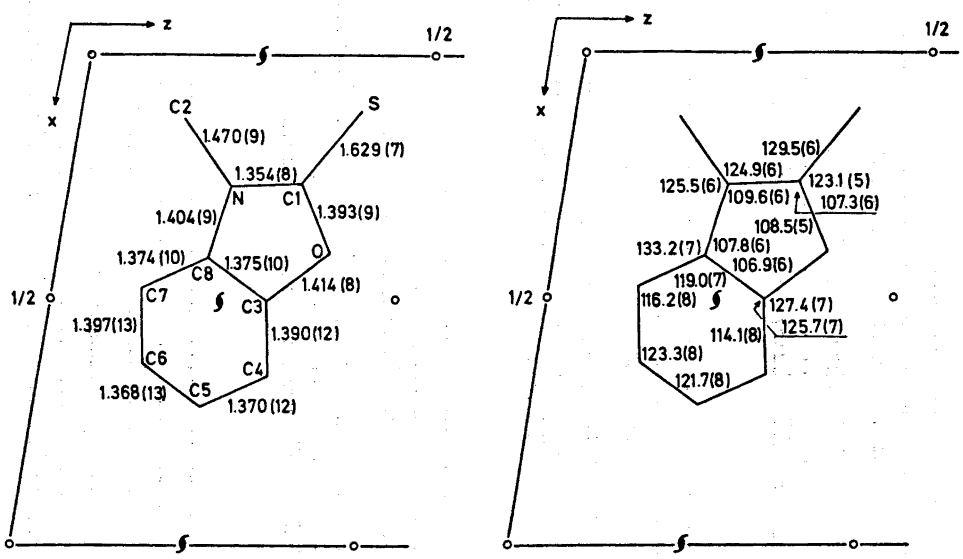

Fig. 1. Schematical drawing of the molecule viewed along [010] showing bond distances (a) and bond angles (b).

Table 3.

h	k	l	Fo	Fc
0	0	2	835	- 854
0	0	4	717	- 724
0	0	6	251	- 258
0	0	8	251	225
0	0	12	146	162
0	1	2	261	259
0	1	3	114	- 93
0	1	4	221	224
0	1	5	361	- 409
0	1	6	150	- 150
0	1	7	66	- 56
0	1	8	114	- 110
0	1	9	47	51
0	1	10	131	- 123
0	1	11	233	242
0	1	12	163	162
0	1	13	66	- 68
0	2	2	55	- 53
0	2	3	283	323
0	2	4	130	- 120
0	2	5	166	- 168
0	2	6	58	- 57
0	2	7	294	- 307
0	2	9	37	31
0	2	11	61	- 58
0	2	13	113	130
0	3	2	332	348
0	3	4	176	185
0	3	5	111	87
0	3	6	171	- 159
0	3	8	108	- 112
0	3	10	121	- 117
0	3	11	110	- 109
0	4	2	266	250
0	4	3	119	93
0	4	4	219	213
0	4	5	22	- 21
0	4	6	83	86
0	4	7	88	- 78
0	4	8	111	- 104
0	4	12	69	- 70
1	0	-12	86	- 82
1	0	-10	204	172
1	0	-8	82	62
1	0	-6	132	127
1	0	+4	366	- 362
1	0	-2	83	68
1	0	0	624	- 609
1	0	2	723	789
1	0	4	318	312
1	0	6	416	- 427
1	0	8	169	- 138
1	0	12	191	183
1	1	-13	39	- 40
1	1	-11	54	- 51
1	1	-10	134	- 135
1	1	-9	187	200
1	1	-8	92	68
1	1	-7	119	101
1	1	-6	197	196
1	1	-5	192	- 190
1	1	-4	47	27
1	1	+3	205	- 205
1	1	-2	101	- 97
1	1	-1	268	- 269
1	1	0	438	- 451
1	1	1	490	508
1	1	2	83	77
1	1	3	301	335
1	1	4	275	301
1	1	5	165	- 176
1	1	6	110	101
1	1	7	263	- 273
1	1	8	162	- 167
1	1	9	98	84
1	1	12	70	- 55
1	1	13	76	81
1	2	-12	36	- 23
1	2	-11	132	- 142
1	2	-9	63	58
1	2	-8	47	47
1	2	-7	28	34
1	2	-5	220	216
1	2	-4	117	- 106
1	2	-3	113	- 115
1	2	-2	36	- 39
1	2	+1	170	172
1	2	0	21	24
1	2	1	658	- 679
1	2	2	134	134
1	2	3	182	186
1	2	4	21	25
1	2	5	419	440
1	2	6	81	- 85
1	2	7	105	- 92
1	2	9	137	- 112
1	2	11	126	- 122
1	2	13	39	35
1	3	-12	32	- 24
1	3	-11	45	49
1	3	-10	109	- 125
1	3	-9	76	- 66
1	3	-8	75	61
1	3	-7	58	- 58
1	3	-6	174	192
1	3	-5	82	72
1	3	-4	26	24
1	3	-3	65	72
1	3	-2	68	- 62
1	3	-1	54	53
1	3	0	291	- 342
1	3	1	141	- 149
1	3	2	114	70
1	3	3	44	- 45
1	3	4	238	236
1	3	5	23	18
1	3	6	115	114
1	3	7	88	80
1	3	8	179	- 168
1	3	9	55	- 42
1	3	10	33	- 6
1	3	11	39	35
1	4	-12	29	36
1	4	-11	55	- 53
1	4	-10	82	- 84
1	4	-7	51	56
1	4	-6	50	- 35
1	4	-5	67	60
1	4	+4	94	67
1	4	-3	53	- 42
1	4	-2	23	- 25
1	4	-1	57	58
1	4	0	211	250
1	4	1	91	- 93
1	4	2	251	- 272
1	4	3	88	80
1	4	4	149	- 127
1	4	5	105	98
1	4	6	204	184
1	4	8	40	- 34
1	4	8	28	36
1	4	11	28	- 29
1	4	12	56	- 77
2	0	-12	154	- 152
2	0	-10	195	168
2	0	-8	218	204
2	0	+4	527	- 575
2	0	-2	149	171
2	0	0	122	- 112
2	0	2	406	361
2	0	4	184	- 155
2	0	6	197	175
2	0	8	296	- 297
2	0	10	160	156
2	0	12	40	- 44
2	1	-13	107	- 109
2	1	-12	126	- 127
2	1	-11	112	109
2	1	-9	108	111
2	1	-8	118	125
2	1	-7	117	- 123
2	1	-6	148	- 146
2	1	-5	222	201
2	1	-4	271	232
2	1	+3	274	- 253
2	1	-2	38	29
2	1	-1	317	- 339
2	1	0	339	- 360
2	1	1	252	277
2	1	2	84	- 86
2	1	3	303	352
2	1	4	268	275
2	1	5	169	- 164
2	1	7	98	- 109
2	1	8	89	- 63
2	1	13	32	37
2	2	-13	34	19
2	2	-11	169	- 188
2	2	+10	39	34
2	2	-8	47	43
2	2	-7	149	152
2	2	-6	30	13
2	2	-5	263	277
2	2	-4	74	- 80
2	2	-3	317	- 312
2	2	-2	35	- 33
2	2	-1	23	25
2	2	0	21	17
2	2	1	221	- 217
2	2	2	138	101
2	2	3	229	222
2	2	5	110	- 100
2	2	7	211	201
2	2	8	62	- 54
2	2	9	200	- 197
2	2	11	62	70
2	3	-12	115	- 123
2	3	-11	66	- 70
2	3	-8	103	101
2	3	-7	96	101
2	3	-6	87	- 70
2	3	-5	126	- 115
2	3	-4	195	204
2	3	-3	59	46
2	3	-2	42	- 37
2	3	-1	94	93
2	3	0	214	- 263
2	3	1	42	- 48
2	3	2	53	- 57
2	3	3	101	- 110
2	3	4	205	223
2	3	5	64	55
2	3	6	27	11
2	3	8	63	- 59
2	3	12	36	- 24
2	4	-12	56	67
2	4	-11	66	- 52
2	4	-10	70	- 73
2	4	-8	85	- 76
2	4	-7	43	38
2	4	-5	68	57
2	4	-4	187	166
2	4	-3	26	- 16
2	4	-2	79	- 62
2	4	-1	37	- 35
2	4	0	35	39
2	4	1	89	- 90
2	4	2	149	- 123
2	4	3	60	61
2	4	4	96	85
2	4	5	39	35
2	4	6	85	- 81
2	4	8	106	117
3	0	-12	196	- 179
3	0	-10	154	- 130
3	0	-8	402	358
3	0	-6	248	215
3	0	-4	211	- 195
3	0	-2	402	- 410
3	0	0	175	- 151
3	0	2	104	99
3	0	4	594	613
3	0	6	177	- 143
3	0	8	259	- 262
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3	0	12	122	112
3	1	-14	47	57
3	1	-13	52	- 54
3	1	-12	59	54
3	1	-11	195	- 213
3	1	-10	197	- 195
3	1	-9	102	95
3	1	-8	151	- 156
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3	1	-6	308	319
3	1	-5	77	- 64
3	1	-4	210	211
3	1	-3	367	- 385
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3	1	-1	275	- 299
3	1	0	327	- 333
3	1	1	214	202
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3	1	4	110	102
3	1	5	68	66
3	1	6	113	106
3	1	7	85	- 71
3	1	8	59	65
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3	1	10	102	- 96
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3	1	12	38	- 24
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3	2	-12	36	- 44
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3	2	-6	69	60
3	2	-5	304	281
3	2	-4	60	- 58
3	2	-3	166	162
3	2	-2	105	- 100
3	2	-1	193	- 186
3	2	0	45	- 33
3	2	1	215	- 245
3	2	2	56	57
3	2	3	229	- 237
3	2	4	100	106
3	2	5	366	375
3	2	7	125	135
3	2	8	58	- 57
3	2	9	96	- 98
3	2	13	28	28
3	3	-12	52	59
3	3	-11	64	79
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3	3	-9	39	- 37
3	3	-8	140	- 155
3	3	-7	157	- 148
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3	3	-5	69	60
3	3	+4	200	209
3	3	-3	101	101
3	3	-2	69	- 78
3	3	-1	73	61
3	3	0	232	- 252
3	3	1	69	- 63
3	3	2	112	- 121
3	3	3	86	- 54
3	3	4	99	100
3	3	6	132	130
3	3	8	47	57
3	3	9	59	55
3	3	10	113	- 106
3	3	12	29	- 23
3	4	-12	80	65
3	4	-11	36	- 27
3	4	-10	56	53
3	4	+9	93	- 90
3	4	-8	184	- 139
3	4	-7	43	28
3	4	-6	82	- 81
3	4	-5	137	98
3	4	-4	62	50
3	4	+3	45	48
3	4	-2	168	153
3	4	-1	67	- 66
3	4	0	29	31
3	4	1	94	- 90
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3	4	3	44	- 69
3	4	4	199	- 190
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4	0	-2	370	- 367
4	0	0	317	- 336
4	0	2	248	- 225
4	0	4	357	370
4	0	6	163	149
4	0	10	148	- 133
4	0	12	46	- 28
4	1	-12	62	75
4	1	-11	88	- 67
4	1	-10	31	28
4	1	-9	194	- 197
4	1	-8	192	- 194
4	1	-7	105	105
4	1	-6	147	- 145
4	1	-5	346	375
4	1	+4	227	235
4	1	-3	77	- 82
4	1	-2	42	35
4	1	-1	26	- 23
4	1	0	208	207
4	1	1	329	- 329
4	1	2	153	- 158
4	1	3	69	- 62
4	1	4	220	- 226
4	1	5	275	293
4	1	6	80	76
4	1	7	114	106
4	1	8	167	167
4	1	9	158	- 170
4	1	10	45	- 38
4	1	11	52	- 47
4	1	12	33	- 51
4	2	-13	33	34
4	2	-11	123	129
4	2	-10	96	- 94
4	2	-9	189	- 187
4	2	-7	155	- 140
4	2	-6	75	65
4	2	-5	114	- 90
4	2	-4	92	88
4	2	+3	437	479
4	2	-2	61	- 54
4	2	-1	72	58
4	2	0	84	- 80
4	2	1	130	- 122
4	2	2	38	- 42
4	2	3	342	- 343
4	2	4	75	72
4	2	5	45	53
4	2	6	47	43
4	2	7	118	120
4	2	9	89	95
4	2	11	33	- 44
4	3	-12	62	78

Table 3. Continued.

h	k	l	Fo	Fc
4	3	-10	33	29
4	3	-9	74	72
4	3	-8	196	- 202
4	3	-6	129	- 120
4	3	-5	111	- 107
4	3	-4	230	226
4	3	-3	40	31
4	3	-2	72	74
4	3	-1	75	- 73
4	3	0	143	135
4	3	1	139	136
4	3	2	173	- 178
4	3	3	49	43
4	3	4	210	- 212
4	3	5	114	- 91
4	3	6	95	101
4	3	7	37	- 45
4	3	8	155	163
4	3	9	72	58
4	3	10	31	- 41
4	4	-10	125	116
4	4	-9	52	- 45
4	4	-8	33	- 17
4	4	-7	87	- 67
4	4	-6	64	- 48
4	4	-4	220	- 188
4	4	-3	115	120
4	4	-2	149	132
4	4	-1	32	29
4	4	0	96	102
4	4	1	40	- 38
4	4	2	89	76
4	4	3	85	- 87
4	4	4	105	- 122
5	0	-12	94	- 89
5	0	-10	41	47
5	0	-8	242	- 234
5	0	-6	93	76
5	0	-4	279	256
5	0	-2	135	112
5	0	0	326	- 324
5	0	8	168	158
5	0	10	76	- 53
5	0	12	70	- 71
5	1	-12	43	43
5	1	-11	32	- 33
5	1	-10	43	45
5	1	-9	143	- 139
5	1	-8	103	- 106
5	1	-7	29	32
5	1	-6	64	- 56
5	1	-5	105	108
5	1	-4	25	- 15
5	1	-3	190	197
5	1	-2	228	239
5	1	-1	240	- 255
5	1	0	146	- 133
5	1	1	58	42
5	1	2	48	52
5	1	3	58	- 68
5	1	4	51	- 24
5	1	5	29	- 26
5	1	6	87	- 86
5	1	7	95	102
5	1	9	48	48
5	1	10	60	73
5	1	11	67	- 75
5	2	-9	105	115
5	2	-8	35	- 41
5	2	-7	169	- 182
5	2	-6	32	33
5	2	-5	126	- 114
5	2	-4	70	70
5	2	-3	111	109
5	2	-1	232	230
5	2	0	58	- 75
5	2	1	136	- 134
5	2	5	53	- 50
5	2	7	75	- 78
5	2	8	36	29
5	2	9	120	113
5	2	11	30	26
5	3	-12	41	43
5	3	-10	52	48
5	3	-9	46	49
5	3	-8	103	- 106
5	3	-7	32	- 21
5	3	-6	72	- 67
5	3	-5	30	- 19
5	3	-3	67	- 68
5	3	-2	218	226
5	3	-1	114	119
5	3	0	111	- 89
5	3	1	68	- 72
5	3	3	30	22
5	3	5	40	44
5	3	6	85	- 78
5	3	9	35	- 34
5	3	11	48	26
5	4	-12	41	51
5	4	-10	39	- 45
5	4	-8	105	98
5	4	-7	81	- 66
5	4	-5	28	- 27
5	4	-4	101	- 88
5	4	-3	72	64
5	4	-2	53	- 42
5	4	-1	45	57
5	4	0	99	102
5	4	11	18	11
6	0	-12	100	89
6	0	-10	129	- 126
6	0	-8	295	- 295
6	0	-6	280	259
6	0	-4	112	108
6	0	-2	124	104
6	0	0	205	- 167
6	0	2	30	- 24
6	0	4	128	- 124
6	0	6	185	153
6	0	10	33	15
6	1	-13	63	64
6	1	-12	72	64
6	1	-11	46	- 46
6	1	-9	57	- 48
6	1	-7	111	- 121
6	1	-6	117	- 112
6	1	-5	90	75
6	1	-4	77	- 80
6	1	-3	190	191
6	1	-2	87	92
6	1	-1	65	55
6	1	0	191	190
6	1	1	239	- 244
6	1	2	72	- 80
6	1	3	116	- 109
6	1	4	189	- 189
6	1	5	207	212
6	1	6	108	116
6	2	-13	28	19
6	2	-11	141	143
6	2	-9	52	59
6	2	-8	57	- 50
6	2	-7	297	- 312
6	2	-5	55	49
6	2	-4	40	41
6	2	-3	32	40
6	2	-2	32	31
6	2	-1	166	152
6	2	0	42	- 38
6	2	1	40	- 37
6	2	2	33	- 31
6	2	5	141	- 152
6	2	7	69	57
6	3	-9	33	- 20
6	3	-7	37	47
6	3	-6	133	- 130
6	3	-4	65	- 67
6	3	-3	76	- 60
6	3	-2	112	103
6	3	-1	43	- 48
6	3	0	173	176
6	3	1	99	102
6	3	2	76	- 75
6	3	3	49	42
6	3	4	178	- 175
6	3	5	100	- 109
6	3	6	87	89
6	3	7	32	36
6	4	-11	33	34
6	4	-10	58	53
6	4	-9	28	15
6	4	-8	107	133
6	4	-7	48	- 63
6	4	-6	151	- 138
6	4	-4	29	- 14
6	4	-2	28	- 30
6	4	-1	28	54
6	4	0	28	57
7	0	-12	140	143
7	0	-10	33	- 24
7	0	-8	48	27
7	0	-6	424	- 435
7	0	-4	214	200
7	0	-2	266	247
7	0	0	143	113
7	0	2	283	- 266
7	0	4	68	- 49
7	0	6	34	22
7	0	8	91	77
7	1	-13	66	77
7	1	-12	26	27
7	1	-11	35	49
7	1	-10	62	66
7	1	-9	45	- 48
7	1	-8	65	47
7	1	-7	137	- 134
7	1	-6	40	- 49
7	1	-5	78	- 78
7	1	-4	135	- 134
7	1	-3	147	144
7	1	-2	44	- 32
7	1	-1	200	206
7	1	0	177	181
7	1	1	141	- 147
7	1	2	31	- 23
7	1	3	31	- 23
7	1	4	32	45
7	1	5	103	- 105
7	1	6	97	- 103
7	1	7	70	71
7	1	9	26	38
7	2	-11	84	96
7	2	-9	50	33
7	2	-8	36	- 21
7	2	-7	186	205
7	2	-6	51	- 59
7	2	-5	352	- 355
7	2	-4	35	32
7	2	-3	75	- 79
7	2	-2	57	61
7	2	-1	105	107
7	2	1	231	229
7	2	2	51	- 48
7	2	3	95	- 88
7	2	5	51	- 65
7	2	7	34	- 44
7	3	-10	40	71
7	3	-8	52	55
7	3	-7	33	24
7	3	-6	72	- 54
7	3	-4	149	- 151
7	3	-1	71	- 65
7	3	0	196	158
7	3	1	33	42
7	3	3	33	- 24
7	3	5	63	68
7	3	6	96	- 99
7	4	-9	71	- 78
7	4	-4	74	- 97
7	4	-2	90	- 99
7	4	0	29	- 42
8	0	-12	87	74
8	0	-10	38	40
8	0	-8	85	- 70
8	0	-4	190	- 170
8	0	-2	42	- 36
8	0	0	288	278
8	0	2	35	- 26
8	0	4	130	- 118
8	0	6	35	- 39
8	1	-12	50	- 60
8	1	-11	99	113
8	1	-10	37	51
8	1	-9	19	24
8	1	-8	69	87
8	1	-7	94	- 105
8	1	-6	32	- 20
8	1	-5	54	- 56
8	1	-4	45	- 51
8	1	-3	25	5
8	1	-2	45	- 38
8	1	-1	48	47
8	1	0	32	- 33
8	1	1	115	117
8	1	2	90	95
8	1	3	60	- 67
8	2	-10	31	22
8	2	-9	55	62
8	2	-5	111	110
8	2	-4	36	- 21
8	2	-3	94	- 93
8	2	-1	172	- 179
8	2	0	55	52
8	2	1	141	150
8	2	3	76	82
8	3	1	55	- 41
8	3	2	106	109

A comparison between observed and calculated structure factors is presented in Table 3. The principal axes of the thermal vibration ellipsoids were calculated from the thermal parameters of Table 1. Root mean square amplitudes and the corresponding B-values for the atomic anisotropic thermal vibration along the principal axes together with the components of these axes along the crystal axes are given in Table 4.

Rigid-body analysis of translational, librational, and screw motion [9] gave relatively large r.m.s. discrepancy between atomic vibration tensor components calculated from the thermal parameters of Table 1, and those calculated from the rigid-body parameters. By including all 11 atoms, the value obtained was 0.0080 Å^2. This number does not support the assumption

Table 4. The principal axes of the thermal vibration ellipsoids given by the components of a unit vector in fractional coordinates e_x, e_y, e_z; the corresponding r.m.s. amplitudes, and the B-values.

Atom	e_x	e_y	e_z	$(\overline{u^2})^{\frac{1}{2}}$ Å	B (Å^2)
	.012	−.143	.005	.321	8.12
S	.090	.017	.054	.256	5.16
	.061	.004	−0.58	.196	3.03
	.033	−.138	.002	.280	6.20
O	.103	.044	−.001	.253	5.05
	.018	.004	.079	.201	3.18
	.005	.145	.005	.245	4.72
N	−.105	.004	.011	.216	3.69
	.031	−.009	.078	.186	2.73
	.007	−.143	.012	.279	6.15
C_1	.103	.002	−.014	.223	3.92
	.036	.022	.077	.193	2.93
	.007	.145	−.003	.345	9.38
C_2	−.031	.010	.071	.238	4.48
	.105	−.007	.034	.197	3.08
	.042	.131	.021	.279	6.16
C_3	.040	−.051	.071	.234	4.32
	−.093	.037	.026	.201	3.20
	−.015	.037	.073	.302	7.19
C_4	.059	.120	−.004	.244	4.70
	.091	−.072	.030	.228	4.12
	.031	.130	.030	.313	7.75
C_5	.068	−.065	.058	.289	6.59
	.080	.004	−.044	.202	3.21
	.083	.018	.060	.361	10.32
C_6	.071	.009	−.051	.216	3.69
	.015	−.144	.004	.195	2.99
	.073	.073	.051	.267	5.62
C_7	.021	−.120	.043	.241	4.60
	−.079	.035	.042	.202	3.22
	.062	−.116	.022	.253	5.04
C_8	.079	.087	.037	.232	4.26
	.044	.009	−.066	.184	2.66

of regarding the molecule as an oscillating rigid body, and the coordinates were therefore not corrected for librational motion.

Bond distances and angles are presented in Fig. 1. Standard deviations, estimated from the correlation matrix of the last least squares refinement cycle, are given in parentheses. The molecule is planar (to within 0.02 Å). The C=S distance of 1.62_9 Å agrees within error limits with values obtained for 4-methyl-1,2-dithiacyclopent-4-ene-3-thione (1.62_7 Å),[10] 2-mercaptobenzothiazole (1.60_0 Å),[11] 4,5-dioxo-2-thioxo-1,3-dithiolane (1.60_8 Å),[12] and 1,3-dithiolane-2-thione (1.65_2 Å),[13] but is significantly shorter than that of ethylenethiourea (1.70_8 Å).[14]

The opening of the heteroatomic ring occurs at the C_1-O bond. Possible variations of this bond length on substitution of S with O, respectively CH_3

with H, may elucidate the difference in interaction rates mentioned above. In the present compound $C_1 - O$ is 1.39_3 Å which is somewhat longer than in 1-oxa-azulen-2-one (1.35_0 Å).[15]

A further discussion of the molecular geometry is postponed until the analysis of some related compounds are presented.

No short *inter* molecular contacts are observed.

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