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Crystal Structure of *trans*-3-(6-Methyl-2-pyridylthio)-propenic Acid

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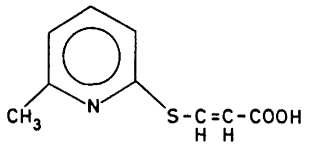
The crystals are monoclinic with space group $P2_1/c$ and cell dimensions $a = 9.17$ Å, $b = 5.86$ Å, $c = 18.34$ Å, $\beta = 90.0°$. There are four molecules in the unit cell. The structure was solved by the heavy atom method and refined by full-matrix least squares technique. The final R-value is 8.1 % for 896 reflections (film data). Dimers are formed by hydrogen bonds of length 2.62_0 Å. The two S–C bond distances are 1.78_6 Å and 1.72_7 Å, and the dihedral angle C=C–S–C is 174.9°.

Thiylation with 2-pyridthiones of α-bromo-α,β-unsaturated β-substituted carboxylic acids gives dihydrothiazolo[3,2-a]pyridinium salts. For one of these the 2,3-substituent has been shown to be *trans*.[1] 2-Pyridthiones will also undergo Michael type addition to activated triple bonds with formation of 2-pyridylthiovinyl ethers. The stereochemistry of the adduct is dependent on the *cis-trans* conversion rates of the intermediate anions as well as *cis-trans* stabilities of the final product. NMR evidence favours *trans* configuration for the major products. For structural assignments by NMR it is important to have the stereochemistry of one member of an analogous series determined by an absolute method. Therefore the crystal structure determination of *trans*-3-(6-methyl-2-pyridylthio)propenic acid has been carried out.



The crystals belong to the monoclinic system, the systematically absent reflections leading to the space group $P2_1/c$. Cell parameters, derived from

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Weissenberg and precession diagrams, are: $a = 9.17$ Å, $b = 5.86$ Å, $c = 18.34$ Å and $\beta = 90.0°$. The number of molecules in the unit cell is four ($\varrho_{calc} = 1.31$ g cm^{-3}, $\varrho_{obs} = 1.31$ g cm^{-3}). Intensity data were obtained (at room temperature) by photometric measurements of integrated equiinclination Weissenberg diagrams corresponding to $h0l, \cdots, h4l$ and integrated $0kl$ precession diagrams. 896 independent reflections were strong enough to be measured. No corrections have been made for absorption or secondary extinction effects.

The structure was solved by the heavy atom method and refined by full-matrix least squares techniques.[2]* Methyl and hydroxyl hydrogen atoms were localized in a difference Fourier map, while the positions of the other hydrogen atoms were calculated assuming C–H bond lengths of 1.03 Å. Anisotropic temperature factors were introduced for sulphur, oxygen, nitrogen, and carbon atoms. Only the positional parameters were refined for hydrogens. The weighting scheme in least squares refinement:

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

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.*[3] except for hydrogen.[4] The R-value arrived at was 8.1 % for 896 observed reflections. A final difference Fourier map, calculated with the phases determined by the parameters corresponding to $R = 8.1$ %, contained no density fluctuations larger than ± 0.3 eÅ^{-3}.

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

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

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 ones are given in Table 4.

Rigid-body analysis of translational, librational, and screw motion [5] 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 13 atoms, the value obtained was 0.0048 Å^2. This number does not support the assumption of regarding the molecules as an oscillating rigid body, and the coordinates were therefore not corrected for librational motion.

Bond distances and angles may be found in Fig. 1 and Fig. 2, respectively, where the molecule is viewed along [010], and clearly has the *trans* configuration. The standard deviations, given in parentheses, are estimated from the correlation matrix of the last least squares refinement cycle.

* All programs used are included in this reference.

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

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
S	38681	78932	05669	975	6121	327	−1096	616	−17
	22	39	10	33	107	7	83	22	36
O_1	12242	13221	−06125	2496	7114	282	−459	268	−737
	62	106	25	95	260	18	251	60	102
O_2	06188	20859	05378	1824	7120	357	−1146	325	−634
	56	99	27	74	261	18	225	58	104
N	25379	83708	18723	1441	4288	329	−240	203	58
	56	98	28	76	239	20	197	58	98
C_1	34593	93388	13994	1542	4671	279	−242	73	341
	72	126	33	96	325	22	263	68	113
C_2	41677	113906	15226	1938	4749	381	−997	119	577
	85	144	42	118	348	29	313	88	147
C_3	38500	125322	21481	2077	4081	465	−670	−314	463
	88	133	45	122	309	32	297	102	148
C_4	28607	116030	26368	1870	4385	370	321	−238	−390
	81	138	38	116	348	27	302	87	131
C_5	22464	95192	24847	1543	4939	351	−227	281	−83
	74	135	36	106	359	26	288	76	131
C_6	12173	83600	30171	3041	8354	381	−358	869	−82
	115	188	46	180	388	32	490	117	190
C_7	25686	57604	05430	1519	4943	278	164	244	−80
	73	125	34	93	296	22	250	65	117
C_8	24194	43607	−00290	2049	5790	282	−6	431	−110
	85	140	36	124	356	23	327	79	138
C_9	13466	25420	−00190	1590	5969	263	948	86	−51
	76	143	36	99	345	23	295	73	128

* 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 while H_0 is in the hydrogen bond).

Atom	x	y	z	B (Å^2)
H_2	0.473	1.191	0.117	5.0
	9	13	5	
H_3	0.425	1.430	0.223	5.0
	8	13	4	
H_4	0.284	1.232	0.304	5.0
	9	15	5	
H_7	0.192	0.555	0.106	5.0
	8	12	4	
H_8	0.308	0.481	−0.046	5.0
	11	17	6	
$H_{6,1}$	0.112	0.619	0.293	5.0
	13	24	6	
$H_{6,2}$	0.147	0.939	0.343	5.0
	11	17	6	
$H_{6,3}$	0.045	0.824	0.276	5.0
	13	20	6	
H_0	−0.047	0.071	0.057	5.0
	12	19	6	

Table 3. Observed and calculated structure factors on 10 times absolute scale.

h	k	l	Fo	Fc
0	0	2	337	339
0	0	4	717	781
0	0	6	190	-202
0	0	8	479	-527
0	0	10	457	-474
0	0	12	97	-93
0	0	14	210	215
0	0	16	345	334
0	0	18	191	171
0	0	20	188	178
0	0	22	36	35
0	1	1	153	170
0	1	2	222	263
0	1	3	288	-324
0	1	4	512	553
0	1	5	248	-260
0	1	6	253	254
0	1	8	106	107
0	1	9	326	-316
0	1	10	210	-183
0	1	11	143	-127
0	1	12	166	-155
0	1	14	147	-139
0	1	16	95	-79
0	2	1	185	221
0	2	2	148	-164
0	2	3	26	29
0	2	4	159	-184
0	2	5	78	78
0	2	6	79	83
0	2	7	141	136
0	2	8	362	392
0	2	9	155	-148
0	2	10	37	30
0	2	12	110	99
0	2	13	88	-85
0	2	15	87	-78
0	2	16	85	-81
0	2	17	55	68
0	3	2	86	84
0	3	3	78	-83
0	3	4	18	-16
0	3	5	50	54
0	3	6	46	41
0	3	7	109	116
0	3	9	255	273
0	3	10	76	71
0	3	11	20	22
0	3	12	21	-15
0	3	13	22	22
0	3	15	85	-83
0	3	17	74	-70
0	4	2	78	87
0	4	3	116	-124
0	4	4	89	85
0	4	5	77	-86
0	4	6	21	-25
0	4	7	147	-142
0	4	8	17	10
0	4	9	134	123
0	4	11	23	-17
0	4	12	17	-10
0	4	13	65	57
0	4	14	17	9
0	4	15	51	42
0	4	16	15	9
0	4	18	20	27
1	0	-18	46	-50
1	0	-14	216	-224
1	0	-12	105	-99
1	0	-10	82	83
1	0	-8	177	165
1	0	-6	182	170
1	0	-4	497	508
1	0	-2	817	924
1	0	0	95	-112
1	0	2	358	-352
1	0	4	136	119
1	0	6	165	-154
1	0	8	275	-285
1	0	10	261	267
1	0	12	203	194
1	0	14	159	147
1	0	16	114	102
1	1	-17	126	116
1	1	-14	312	-284
1	1	-13	76	63
1	1	-12	275	-256
1	1	-10	186	-178
1	1	-9	126	120
1	1	-8	263	-273
1	1	-7	275	-264
1	1	-6	369	344
1	1	-5	157	-144
1	1	-4	162	159
1	1	-3	117	-84
1	1	-1	497	490
1	1	0	54	41

h	k	l	Fo	Fc
1	1	1	563	-574
1	1	2	278	-284
1	1	3	609	622
1	1	4	621	-689
1	1	5	77	75
1	1	6	323	-371
1	1	7	119	-125
1	1	8	423	-443
1	1	9	184	-177
1	1	11	30	31
1	1	13	92	-79
1	1	14	84	78
1	1	16	142	131
1	2	-17	33	27
1	2	-16	76	51
1	2	-14	70	70
1	2	-11	45	47
1	2	-10	27	-21
1	2	-9	156	161
1	2	-8	149	-161
1	2	-7	115	103
1	2	-6	369	-376
1	2	-5	296	318
1	2	-4	130	-128
1	2	-3	36	41
1	2	-2	198	-186
1	2	-1	123	-104
1	2	0	88	96
1	2	1	133	133
1	2	2	18	20
1	2	3	180	-200
1	2	4	129	131
1	2	5	109	99
1	2	6	68	70
1	2	7	84	69
1	2	8	84	-75
1	2	9	118	134
1	2	10	128	-131
1	2	11	46	44
1	2	12	71	-71
1	2	13	76	62
1	2	14	84	-72
1	2	15	33	29
1	2	16	33	-34
1	2	17	46	36
1	3	-16	44	43
1	3	-15	69	65
1	3	-14	78	64
1	3	-13	22	15
1	3	-12	85	72
1	3	-10	85	80
1	3	-9	35	43
1	3	-7	115	-115
1	3	-6	141	-135
1	3	-3	67	-84
1	3	-2	273	-260
1	3	-1	173	-155
1	3	0	120	116
1	3	1	217	236
1	3	2	59	-66
1	3	3	67	-78
1	3	4	216	251
1	3	5	75	84
1	3	6	94	108
1	3	7	133	135
1	3	8	146	165
1	3	9	72	-86
1	3	10	63	-81
1	3	12	80	-83
1	3	13	47	-44
1	3	14	82	-73
1	3	15	53	-49
1	3	16	54	-62
1	3	17	35	-12
1	4	-17	23	-25
1	4	-16	35	-36
1	4	-15	69	60
1	4	-14	16	-26
1	4	-13	103	-81
1	4	-12	22	-18
1	4	-11	84	80
1	4	-10	28	26
1	4	-9	93	85
1	4	-8	59	50
1	4	-7	78	73
1	4	-6	167	159
1	4	-5	139	-123
1	4	-4	56	35
1	4	-3	101	-94
1	4	-2	23	19
1	4	-1	35	-26
1	4	0	45	-51
1	4	1	55	-59
1	4	2	18	-18
1	4	3	69	73
1	4	4	88	-86
1	4	5	123	117
1	4	7	164	169

h	k	l	Fo	Fc
1	4	8	141	140
1	4	9	71	67
1	4	10	22	-15
1	4	12	92	101
1	4	13	17	-2
1	4	15	25	-24
1	4	16	15	4
2	0	-22	101	-118
2	0	-20	115	-117
2	0	-18	25	32
2	0	-16	160	-148
2	0	-14	288	272
2	0	-12	151	153
2	0	-8	295	-308
2	0	-6	521	-539
2	0	-4	538	-512
2	0	-2	652	-629
2	0	0	627	-611
2	0	2	1118	1175
2	0	4	323	-321
2	0	6	303	320
2	0	8	91	-100
2	0	10	152	-154
2	0	12	385	-389
2	0	14	109	-106
2	0	16	68	-54
2	0	20	38	38
2	1	-17	37	67
2	1	-16	266	-250
2	1	-15	37	36
2	1	-14	148	-130
2	1	-13	201	195
2	1	-11	76	80
2	1	-9	103	-100
2	1	-8	360	341
2	1	-7	59	-69
2	1	-6	459	453
2	1	-5	21	12
2	1	-4	216	-189
2	1	-3	43	-37
2	1	-2	753	-712
2	1	-1	200	190
2	1	0	485	-452
2	1	1	505	510
2	1	2	1390	-1442
2	1	3	647	642
2	1	4	51	48
2	1	6	61	-59
2	1	7	60	68
2	1	8	241	258
2	1	9	88	65
2	1	10	242	260
2	1	11	153	-154
2	1	12	47	46
2	1	16	161	-146
2	2	-17	55	-44
2	2	-16	73	-77
2	2	-15	132	-120
2	2	-14	115	-118
2	2	-13	59	50
2	2	-12	76	-66
2	2	-11	141	132
2	2	-10	69	72
2	2	-9	121	131
2	2	-8	98	-71
2	2	-7	328	369
2	2	-6	344	374
2	2	-5	168	-148
2	2	-4	46	21
2	2	-3	91	-87
2	2	-2	76	-66
2	2	-1	264	-238
2	2	0	296	-310
2	2	1	465	-471
2	2	2	374	-378
2	2	3	60	-67
2	2	5	203	213
2	2	6	148	-155
2	2	7	196	197
2	2	8	62	59
2	2	9	151	171
2	2	10	157	178
2	2	11	130	129
2	2	12	63	60
2	2	15	86	-70
2	3	-17	20	-14
2	3	-16	64	55
2	3	-15	22	-18
2	3	-14	22	-17
2	3	-13	116	-111
2	3	-12	22	-21
2	3	-11	34	-35
2	3	-10	72	-65
2	3	-9	27	-28
2	3	-8	29	-36
2	3	-7	45	83
2	3	-5	219	211
2	3	-4	126	122

h	k	l	Fo	Fc
2	3	-3	111	100
2	3	-2	318	281
2	3	-1	82	77
2	3	0	144	134
2	3	1	100	-100
2	3	2	108	99
2	3	3	149	-144
2	3	4	54	47
2	3	5	58	-50
2	3	6	43	-45
2	3	8	63	-68
2	3	9	19	-25
2	3	10	45	-50
2	3	11	175	199
2	3	12	65	77
2	3	15	54	54
2	3	16	46	42
2	4	-17	62	67
2	4	-16	29	32
2	4	-15	54	45
2	4	-12	24	-17
2	4	-11	66	-64
2	4	-10	16	-20
2	4	-9	17	-10
2	4	-8	24	-14
2	4	-7	264	-258
2	4	-6	75	-68
2	4	-5	22	19
2	4	-4	28	19
2	4	-2	35	30
2	4	-1	128	107
2	4	0	70	57
2	4	1	96	89
2	4	2	13	-10
2	4	3	173	167
2	4	4	35	32
2	4	5	15	-13
2	4	6	29	24
2	4	7	31	-38
2	4	8	77	-84
2	4	9	120	-127
2	4	12	24	-24
2	4	13	34	-33
2	4	16	15	6
2	4	17	37	34
3	0	-22	51	57
3	0	-20	67	61
3	0	-16	86	-79
3	0	-14	30	6
3	0	-12	429	-431
3	0	-10	192	-177
3	0	-8	19	-18
3	0	-6	82	-77
3	0	-4	230	207
3	0	-2	196	181
3	0	0	220	210
3	0	2	265	-223
3	0	4	323	-323
3	0	6	196	-205
3	0	8	198	-198
3	0	10	307	-324
3	0	12	83	88
3	0	18	30	27
3	0	20	38	-30
3	1	-16	59	55
3	1	-15	77	57
3	1	-14	87	85
3	1	-13	155	-138
3	1	-10	31	-31
3	1	-7	254	248
3	1	-6	182	-169
3	1	-5	113	-101
3	1	-4	124	84
3	1	-3	226	202
3	1	0	268	-225
3	1	1	160	169
3	1	2	176	159
3	1	3	404	-402
3	1	5	91	89
3	1	6	125	-128
3	1	8	96	79
3	1	12	34	-36
3	1	13	116	102
3	1	14	37	-42
3	1	15	37	18
3	2	-16	77	80
3	2	-15	41	-38
3	2	-14	158	164
3	2	-12	265	256
3	2	-11	113	-115
3	2	-10	161	164
3	2	-9	79	-81
3	2	-8	88	83
3	2	-7	42	-32
3	2	-6	81	-84
3	2	-5	76	-76
3	2	-4	445	-414
3	2	-3	89	99

Table 3. Continued.

h	k	l	Fo	Fc
3	2	-2	214	- 184
3	2	-1	113	+ 92
3	2	0	127	+ 132
3	2	1	205	+ 186
3	2	2	240	261
3	2	3	263	247
3	2	4	167	189
3	2	5	312	+ 310
3	2	6	392	422
3	2	8	131	133
3	2	10	89	89
3	2	11	30	+ 29
3	2	12	105	+ 119
3	2	13	62	+ 62
3	3	-16	85	+ 85
3	3	-13	115	107
3	3	-12	22	35
3	3	-11	61	61
3	3	-10	131	135
3	3	-8	148	142
3	3	-6	221	216
3	3	-5	108	+ 94
3	3	-4	164	158
3	3	-3	168	+ 166
3	3	-2	56	+ 58
3	3	-1	72	+ 60
3	3	0	85	+ 79
3	3	1	41	+ 33
3	3	2	15	+ 14
3	3	3	74	67
3	3	4	41	+ 38
3	3	5	41	44
3	3	6	94	88
3	3	7	62	64
3	3	8	108	122
3	3	9	93	103
3	3	10	188	219
3	3	11	42	+ 51
3	3	14	57	60
3	3	15	22	+ 20
3	4	-17	27	+ 34
3	4	-15	49	+ 48
3	4	-14	69	+ 73
3	4	-13	27	+ 33
3	4	-12	58	+ 56
3	4	-10	46	+ 69
3	4	-9	39	+ 30
3	4	-8	80	+ 91
3	4	-7	17	+ 5
3	4	-6	107	111
3	4	-5	109	104
3	4	-4	57	52
3	4	-3	82	+ 71
3	4	-2	95	91
3	4	-1	24	+ 10
3	4	0	41	36
3	4	1	42	25
3	4	2	62	+ 59
3	4	3	120	+ 118
3	4	4	66	+ 70
3	4	5	59	+ 46
3	4	6	160	+ 160
3	4	7	61	+ 58
3	4	8	50	+ 55
3	4	12	24	+ 25
3	4	13	23	+ 13
3	4	14	61	70
4	0	-18	116	+ 103
4	0	-16	54	+ 37
4	0	-14	253	+ 240
4	0	-12	192	195
4	0	-10	183	162
4	0	-8	362	370
4	0	-6	35	41
4	0	-4	282	242
4	0	-2	353	+ 334
4	0	0	355	+ 356
4	0	2	261	+ 245
4	0	6	64	64
4	0	8	101	93
4	0	10	125	132
4	0	12	81	74
4	0	14	92	+ 110
4	0	18	51	+ 52
4	1	-14	81	+ 84
4	1	-13	110	+ 124
4	1	-12	171	+ 173
4	1	-11	156	166
4	1	-10	67	53
4	1	-9	81	85
4	1	-8	276	282
4	1	-6	254	242
4	1	-5	61	62
4	1	-4	471	460
4	1	-3	185	+ 169
4	1	-1	341	+ 301
4	1	0	150	+ 142
4	1	1	373	+ 336
4	1	2	176	+ 162
4	1	3	231	+ 201
4	1	4	592	+ 624
4	1	5	71	52
4	1	6	206	194
4	1	7	29	9
4	1	9	107	85
4	1	10	164	177
4	1	11	85	80
4	1	12	138	146
4	1	13	37	- 33
4	1	14	112	105
4	1	15	53	+ 56
4	2	-15	80	+ 73
4	2	-14	87	83
4	2	-13	141	+ 139
4	2	-12	46	+ 39
4	2	-11	102	+ 104
4	2	-10	68	+ 74
4	2	-8	113	+ 97
4	2	-7	157	169
4	2	-6	127	+ 122
4	2	-5	259	243
4	2	-4	196	182
4	2	-3	101	98
4	2	-2	250	256
4	2	-1	60	53
4	2	0	360	372
4	2	1	91	+ 80
4	2	2	174	178
4	2	3	346	+ 367
4	2	4	266	266
4	2	5	213	+ 215
4	2	6	49	+ 41
4	2	8	52	+ 48
4	2	10	43	+ 41
4	2	11	39	42
4	2	12	56	39
4	2	13	143	147
4	2	14	59	52
4	3	-15	33	36
4	3	-14	83	84
4	3	-13	35	38
4	3	-11	112	+ 115
4	3	-10	31	+ 31
4	3	-9	107	+ 110
4	3	-8	110	+ 122
4	3	-7	50	+ 58
4	3	-6	90	+ 89
4	3	-5	217	+ 208
4	3	-4	207	+ 186
4	3	-3	183	165
4	3	-2	53	34
4	3	-1	60	60
4	3	0	130	122
4	3	1	127	118
4	3	2	118	113
4	3	3	43	40
4	3	4	93	88
4	3	6	95	83
4	3	7	90	+ 104
4	3	9	67	+ 66
4	3	10	101	+ 100
4	3	11	87	+ 98
4	3	12	42	+ 54
4	3	13	49	55
4	3	15	30	+ 30
4	4	-17	9	4
4	4	-16	13	+ 16
4	4	-15	59	54
4	4	-13	16	19
4	4	-12	16	3
4	4	-9	23	+ 35
4	4	-8	32	35
4	4	-7	22	28
4	4	-6	27	+ 24
4	4	-5	167	+ 148
4	4	-4	102	+ 91
4	4	-3	15	18
4	4	-1	32	32
4	4	0	79	+ 75
4	4	1	64	55
4	4	2	18	+ 18
4	4	3	17	14
4	4	4	27	+ 26
4	4	5	74	67
4	4	7	31	+ 33
4	4	9	22	+ 26
4	4	10	36	+ 35
4	4	11	43	+ 47
4	4	13	17	+ 7
4	4	14	22	+ 27
5	0	-18	61	65
5	0	-14	54	+ 53
5	0	-12	36	31
5	0	-10	249	+ 280
5	0	-8	30	+ 19
5	0	-6	144	110
5	0	-4	24	13
5	0	-2	181	177
5	0	0	134	101
5	0	2	108	+ 111
5	0	4	77	+ 68
5	0	6	201	+ 194
5	0	8	79	+ 78
5	0	10	29	36
5	0	12	41	+ 51
5	0	14	74	73
5	1	-14	128	127
5	1	-13	76	85
5	1	-12	142	135
5	1	-11	36	+ 43
5	1	-10	212	211
5	1	-9	42	+ 48
5	1	-7	60	+ 60
5	1	-6	56	+ 57
5	1	-5	77	67
5	1	-4	183	+ 174
5	1	-3	181	+ 167
5	1	-2	149	+ 137
5	1	0	65	+ 56
5	1	1	49	+ 47
5	1	2	48	+ 36
5	1	3	64	77
5	1	4	280	271
5	1	6	140	119
5	1	8	78	75
5	1	9	35	11
5	1	10	50	53
5	1	11	36	+ 27
5	1	12	37	39
5	1	13	46	+ 52
5	1	14	89	+ 78
5	2	-12	41	38
5	2	-11	160	176
5	2	-10	92	103
5	2	-9	45	49
5	2	-8	120	135
5	2	-7	30	30
5	2	-6	94	82
5	2	-5	38	+ 29
5	2	-4	110	94
5	2	-3	27	+ 14
5	2	-2	130	+ 136
5	2	-1	64	+ 75
5	2	0	68	+ 71
5	2	1	54	+ 35
5	2	2	38	+ 31
5	2	3	60	+ 61
5	2	5	225	234
5	2	7	69	+ 62
5	2	8	151	161
5	2	9	103	91
5	2	10	32	41
5	2	12	88	99
5	2	14	40	+ 33
5	3	-14	126	+ 130
5	3	-12	127	+ 141
5	3	-11	42	46
5	3	-10	138	+ 147
5	3	-9	60	50
5	3	-8	93	+ 82
5	3	-6	79	+ 75
5	3	-5	25	19
5	3	-4	161	161
5	3	-3	28	31
5	3	-2	114	105
5	3	-1	116	+ 113
5	3	2	43	+ 33
5	3	3	33	30
5	3	4	108	+ 107
5	3	5	51	+ 47
5	3	6	133	+ 145
5	3	7	45	38
5	3	8	170	+ 177
5	3	10	43	+ 41
5	3	11	22	17
5	3	12	22	17
5	3	14	21	+ 22
5	4	-15	18	+ 22
5	4	-14	14	17
5	4	-13	21	+ 22
5	4	-12	22	+ 26
5	4	-11	23	+ 28
5	4	-8	70	+ 65
5	4	-6	96	+ 91
5	4	-5	69	54
5	4	-4	64	62
5	4	-3	143	140
5	4	-2	45	+ 40
5	4	-1	26	33
5	4	0	83	79
5	4	1	16	14
5	4	2	25	21
5	4	3	41	30
5	4	4	18	22
5	4	5	51	+ 55
5	4	6	40	+ 44
5	4	7	17	+ 17
5	4	8	37	+ 41
5	4	10	31	+ 33
5	4	11	16	14
5	4	12	22	+ 26
5	4	13	29	36
6	0	-18	52	+ 56
6	0	-16	140	+ 144
6	0	-14	140	+ 146
6	0	-12	182	+ 176
6	0	-10	51	51
6	0	-8	201	200
6	0	-6	314	320
6	0	-4	77	58
6	0	-2	211	207
6	0	0	356	+ 342
6	0	2	271	+ 259
6	0	4	254	+ 252
6	0	6	181	+ 171
6	0	8	152	142
6	0	10	50	53
6	0	12	106	121
6	0	14	47	57
6	0	16	22	+ 18
6	0	18	43	+ 42
6	1	-11	105	+ 105
6	1	-10	188	+ 191
6	1	-9	79	78
6	1	-8	36	+ 39
6	1	-7	87	52
6	1	-5	101	103
6	1	-4	87	90
6	1	-3	168	139
6	1	-2	165	171
6	1	0	164	165
6	1	2	123	109
6	1	4	41	26
6	1	5	71	+ 78
6	1	6	136	+ 131
6	2	-13	32	34
6	2	-9	141	+ 161
6	2	-8	128	+ 123
6	2	-6	190	+ 191
6	2	-4	189	+ 181
6	2	-3	43	+ 42
6	2	-2	112	+ 98
6	2	-1	112	108
6	2	0	64	81
6	2	1	109	112
6	2	2	89	94
6	2	3	94	110
6	2	4	30	29
6	2	5	44	+ 39
6	2	6	120	125
6	2	8	72	+ 80
6	3	-13	70	77
6	3	-12	21	20
6	3	-11	59	56
6	3	-9	29	+ 33
6	3	-7	66	+ 74
6	3	-5	31	+ 28
6	3	-3	179	+ 168
6	3	-2	142	+ 129
6	3	-1	124	109
6	3	1	50	53
6	3	2	56	+ 52
6	3	3	99	93
6	3	4	28	+ 27
6	3	5	68	74
6	3	6	67	+ 67
6	3	7	58	55
6	3	8	22	21
6	3	9	59	+ 63
6	4	-13	28	39
6	4	-10	16	19
6	4	-9	30	44
6	4	-8	35	21
6	4	-6	70	70
6	4	-3	74	+ 57
6	4	-1	53	+ 38
6	4	2	46	+ 38
6	4	4	22	+ 20
6	4	7	57	53
6	4	9	17	14
7	0	-16	53	51
7	0	-14	106	107
7	0	-12	103	117
7	0	-10	152	151
7	0	-6	82	+ 79
7	0	-4	56	+ 48
7	0	-2	141	+ 128
7	0	2	100	72
7	0	4	59	49
7	0	6	162	167
7	0	10	25	12
7	0	12	25	11
7	0	14	23	+ 25
7	0	16	31	+ 22
7	1	-10	92	95
7	1	-8	195	202
7	1	-7	59	16
7	1	-4	50	+ 49
7	1	-2	62	+ 62
7	1	0	111	+ 112
7	1	2	62	+ 53
7	1	3	51	+ 37
7	1	4	44	32
7	1	6	114	104
7	2	-9	33	59
7	2	-8	85	+ 77
7	2	-7	14	+ 14
7	2	-2	113	116
7	2	0	45	+ 39
7	2	1	74	+ 67
7	2	2	32	+ 16
7	2	4	33	+ 21
7	2	7	33	37
7	2	8	47	+ 67
7	3	-10	21	+ 28
7	3	-9	30	+ 34
7	3	-8	31	+ 12
7	3	-6	27	+ 28
7	3	-5	22	7
7	3	-4	30	+ 33
7	3	-3	62	60
7	3	-2	94	103
7	3	-1	65	72
7	3	0	32	36
7	3	1	31	+ 21
7	3	2	31	35
7	3	6	22	14

Table 3. Continued.

h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc
7	3	7	64	- 77	8	0	-12	40	- 41	8	2	-6	33	- 29					
7	3	11	30	- 9	8	0	-10	45	- 57	8	2	-4	33	- 38	8	4	8	18	25
7	4	-13	10	- 23	8	0	-8	68	- 70	8	2	0	47	- 47	9	0	-12	47	51
7	4	-12	17	29	8	0	-6	91	78	8	2	1	47	53	9	0	-10	66	58
7	4	-11	18	- 39	8	0	-4	131	132	8	2	2	41	43	9	0	-8	104	109
7	4	-10	20	29	8	0	-2	115	94	8	2	3	41	39	9	0	-6	66	72
7	4	-9	57	19	8	0	0	197	186	8	3	-7	20	32	9	0	-4	39	- 24
7	4	-7	48	- 54	8	0	2	25	26	8	3	-6	30	33	9	0	-2	40	- 32
7	4	-5	23	- 25	8	0	4	25	23	8	3	-5	41	- 23	9	0	0	56	- 58
7	4	-4	26	- 22	8	0	6	47	- 37	8	3	-4	34	45	9	0	2	67	- 57
7	4	-1	47	54	8	0	8	60	- 60	8	3	-2	16	13	9	0	8	70	64
7	4	0	17	- 23	8	0	10	52	51	8	3	0	35	- 39	9	0	12	23	24
7	4	2	36	34	8	0	14	47	44	8	3	1	22	11	10	0	-12	14	17
7	4	4	16	16	8	0	16	35	40	8	3	2	25	19	10	0	-6	97	- 107
7	4	6	38	39	8	1	-8	138	- 149	8	3	3	22	- 18	10	0	-4	38	- 55
7	4	7	16	- 33	8	1	-6	125	- 135	8	3	4	21	- 8	10	0	-2	32	- 42
7	4	8	15	- 10	8	1	-4	125	- 129	8	4	3	21	- 27	10	0	0	28	- 26
7	4	9	23	- 39	8	1	2	152	149	8	4	5	21	- 32	10	0	2	22	14
7	4	10	20	29	8	1	4	72	74	8	4	7	19	- 29	10	0	4	36	43
8	0	-16	70	88	8	2	-7	89	- 90										

Table 4. The principal axes of the thermal vibration ellipsoids given by 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)
	.061	−.136	.013	.342	9.24
S	.070	.102	.026	.300	7.11
	.058	.019	−.046	.198	3.11
	.037	−.154	.014	.363	10.41
O_1	.101	.062	.003	.323	8.26
	−.016	.038	.053	.204	3.28
	.035	−.155	.015	.367	10.62
O_2	.093	.068	.018	.275	5.99
	−.045	.022	.049	.228	4.12
	.027	−.165	.002	.275	5.95
N	.083	.036	.033	.260	5.32
	.065	.022	−.043	.222	3.88
	−.015	.164	.013	.290	6.63
C_1	.106	.015	.011	.257	5.21
	.019	.044	−.052	.211	3.50
	−.063	.132	.015	.314	7.76
C_2	.080	.058	.032	.283	6.34
	.039	.092	−.042	.227	4.06
	−.077	.070	.031	−.323	8.26
C_3	.077	.076	.030	.269	5.71
	.003	−.136	.033	.249	4.88
	.074	.097	−.026	.301	7.13
C_4	.074	−.125	.005	.271	5.81
	.032	−065	.048	.235	4.35
	.026	−.163	.010	.295	6.88
C_5	.082	.052	.032	.272	5.83
	.067	.001	−.043	.225	3.99
	.071	−.120	.016	.447	15.75
C_6	.070	.121	.016	.304	7.29
	−.045	.001	.050	.219	3.79
	.010	.170	−.002	.294	0.81
C_7	.098	−.011	.023	.264	5.49
	−.046	.011	.049	.206	3.34
	.016	−.168	.006	.318	7.98
C_8	.100	.030	.019	.307	7.45
	−.040	.008	.051	.202	3.21
	.033	.163	.000	.329	8.53
C_9	.102	−.051	.011	.254	5.08
	−.020	.011	.054	.210	3.48

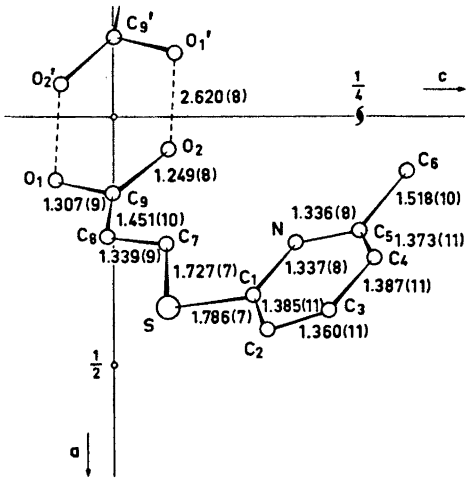

Fig. 1. Schematical drawing of the molecule (viewed along [010]) showing bond distances.

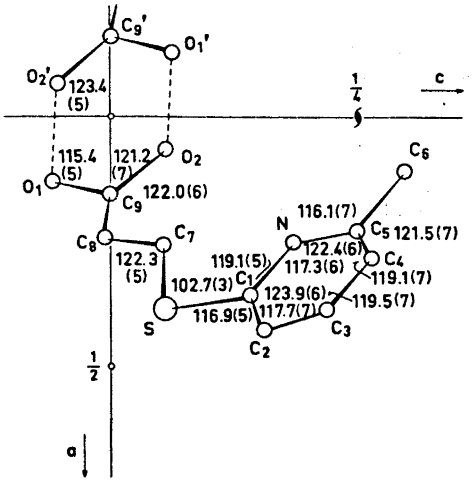

Fig. 2. Schematical drawing of the molecule (viewed along [010]) showing bond angles.

Bond distances and angles of the six-membered ring agree within error limits with the micro wave results for pyridine [6] (C–N: 1.340 Å, C–C: 1.394 Å, C–C–C: 118.5°, C–C–N: 123.9°, C–N–C: 116.8°).

The geometry of the ($C_1-S-C_7-C_8$) part may be compared to that of methyl vinyl sulphide which recently has been studied in the gas phase by electron diffraction technique.[7] As to be expected, the distance $S-C_1$ (1.786 Å$_7$ is somewhat shorter than the normal $S-C(sp^3)$ bond (1.806(6) Å). The S–C distance of 1.727 Å appears also to be shorter than the corresponding distance (1.748(6) Å) in methyl vinyl sulphide. However, the uncertainties are large in both cases, and the differences cannot be regarded as significant. The C–S–C angle (102.7°) agrees within probable limits of error with the value obtained for the free methyl vinyl sulphide molecule (104.5(7)°), while S–C=C (122.3°) is significantly smaller (125.9(5)°).

Dimers are formed by moderately strong hydrogen bonds of length 2.620 Å between centro-symmetry equivalent molecules. Distances and angles of the carboxyl group are normal.

The largest deviations from least squares planes through O_1, O_2, C_9, C_8, C_7, S and S, C_1, C_2, C_3, C_4, C_5, C_6, N are 0.06 Å and 0.03 Å, respectively, and the angle between these planes is 168.2°. The dihedral angle $C_8=C_7-S-C_1$ of 174.9° is widely different from that of methyl vinyl sulphide (106.6°).

Except for the hydrogen bonds no short *inter*-molecular contacts are observed.

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