

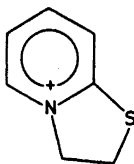
Crystal Structure of *trans* 2-Carboxy-5-methyl-dihydrothiazolo[3,2-a]pyridinium-3-carboxylate

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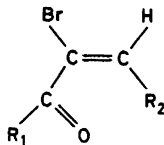
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The crystals are monoclinic with space group $P2_1/c$ and cell dimensions $a=10.70$, Å, $b=7.37$, Å, $c=16.68$, Å, $\beta=130.5^\circ$. There are four molecules in the unit cell. The structure was solved by the heavy atom method, and the weighted R -value arrived at was $R_w=3.5\%$ for 1089 reflections, which were measured on a four circle diffractometer. The two S-C bond distances are 1.83, Å and 1.73, Å. The *intermolecular* hydrogen bond is relatively short (2.47, Å) and forms infinite chains of zwitterions in the [010] direction. The hydrogen atom is at a distance of 1.05 Å from one of the oxygens, and the O-H...O angle is 177° .

The synthesis of the dihydrothiazolo[3,2-a]pyridinium ring system has been described by Bradsher and Lohr¹ and by Balsiger *et al.*² The properties of

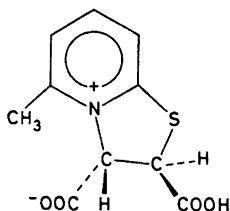


dihydrothiazolo[3,2-a]pyridinium derivatives are being studied extensively by Undheim *et al.*, who in a recent report³ have shown that such compounds may be obtained by addition of pyrid-2-thiones to α -bromo- α,β -unsaturated carbonyl compounds.



If the β -carbon in the unsaturated carbonyl compound carries a substituent, NMR evidence is in favour of a *trans* configuration in the bicyclic product. This appears to be true ⁴ irrespective of the stereochemistry about the double bond in the carbonyl compound.

In order to obtain more detailed information, the crystal structure determination of *trans* 2-carboxy-5-methyl-dihydrothiazolo[3,2-a]pyridinium-3-carboxylate has been carried out.



The crystals are monoclinic with space group $P2_1/c$. The cell dimensions, determined by a manual four circle diffractometer, with estimated standard deviations ⁵ * are:

$$a = 10.707(4) \text{ \AA}; b = 7.376(4) \text{ \AA}; c = 16.688(4) \text{ \AA}; \beta = 130.52(2)^\circ.$$

The unit cell contains four molecules ($\rho_{\text{calc}} = 1.58 \text{ g/cm}^3$, $\rho_{\text{obs}} = 1.58 \text{ g/cm}^3$).

About 1650 reflections were measured by an automatic four circle diffractometer ($2\theta_{\text{max}} = 50^\circ$), using $\text{MoK}\alpha$ -radiation and a highly orientated graphite crystal monochromator. With an observed-unobserved cutoff at $2.0 \cdot \sigma(I)$, 1089 reflections were recorded as observed. No corrections for absorption or secondary extinction effects have been carried out.

The structure was solved by the heavy atom method and refined by full-matrix least squares techniques. 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. The weights in least squares were calculated from the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I) = [C_T + (0.02 \times C_N)^2]^\dagger$$

where C_T is the total number of counts, and C_N the net count (peak minus background). The weighted R -value arrived at was 3.5 % (conventional value $R = 4.9$ %) for 1089 observed reflections. The atomic form factors were those of Hanson *et al.*⁶ except for hydrogen.⁷ A final difference Fourier map, calculated with the phases determined by the parameters corresponding to $R_w = 3.5$ %, contained no density fluctuations larger than $\pm 0.3 \text{ e. \AA}^{-3}$.

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

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

* All programs are included in this reference.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> ₁₁	<i>B</i> ₂₂	<i>B</i> ₃₃	<i>B</i> ₁₂	<i>B</i> ₁₃	<i>B</i> ₂₃
S	35099	09085	19022	1346	1435	452	-487	1224	-380
	12	15	7	18	25	7	37	20	24
O ₁	06227	-17764	09435	1568	704	327	-331	804	-69
	29	35	17	50	54	17	87	50	53
O ₂	09051	-10284	23557	1485	1262	468	-560	1160	-95
	28	34	17	46	60	18	92	51	57
O ₃	05804	49823	13003	967	852	368	-42	397	228
	27	35	18	42	55	17	79	46	50
O ₄	31888	54124	27720	944	1005	484	-485	559	-114
	27	32	17	43	61	18	82	47	55
N	36438	20285	34268	788	780	313	53	650	76
	30	37	19	49	66	20	89	55	56
C ₁	45958	13596	32264	987	947	441	-74	938	2
	41	48	26	62	87	26	121	73	76
C ₂	62628	11210	40496	984	1097	713	201	1190	-2
	44	53	31	75	101	39	153	95	103
C ₃	69055	15262	50552	930	1360	522	89	620	-23
	46	55	31	76	109	36	152	90	101
C ₄	59021	21791	52347	1170	1247	301	-83	561	-35
	44	54	28	86	108	29	150	93	93
C ₅	42488	24569	44126	1030	779	260	-41	647	86
	40	47	24	72	88	27	130	79	79
C ₆	31185	31840	45603	1392	1491	671	42	1434	119
	54	66	36	90	118	39	171	104	112
C ₇	15607	11680	16269	976	805	310	90	665	46
	40	48	26	69	92	26	134	75	82
C ₈	09863	-06883	16799	664	907	265	171	334	199
	38	50	26	62	91	24	125	67	82
C ₉	19556	24905	24623	669	848	287	-89	481	5
	39	47	25	62	84	24	127	69	77
C ₁₀	19416	44937	21725	924	873	368	-28	780	-146
	44	47	27	68	96	27	135	78	86

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 motion⁸ gave relatively large r.m.s. discrepancy between atomic vibration tensor component calculated from the thermal parameters of Table 1, and those calculated from the rigid-body parameters. By including all 16 atoms, the value obtained was 0.0048 Å². When the methyl and carboxyl groups were excluded, the r.m.s.-value dropped to 0.0034 Å². These numbers do not strongly support the assumption of regarding neither the whole molecule, nor the bicyclic ring

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_B is in the hydrogen bond).

Atom	x	y	z	$B(\text{\AA}^2)$
H_2	.693	.068	.390	3.2
	4	4	2	6
H_3	.809	.136	.567	3.8
	4	4	2	7
H_4	.625	.243	.589	3.1
	4	4	2	7
H_7	.075	.174	.091	2.1
	4	4	2	6
H_9	.115	.232	.259	1.4
	4	4	2	6
$H_{6,1}$.227	.229	.434	2.5
	4	5	2	7
$H_{6,2}$.258	.429	.415	3.0
	4	5	2	7
$H_{6,3}$.366	.338	.521	8.3
	4	4	3	1.1
H_B	.056	.683	.106	7.7
	4	6	3	1.1

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

Some of the bond distances and angles with standard deviations, estimated from the correlation matrix of the last least squares refinement cycle, listed in Table 5, may also be found in Figs. 1 and 2, where the molecule is viewed along the directions [010] and [100], respectively. Dihedral angles of the five-membered ring are given in Table 6.

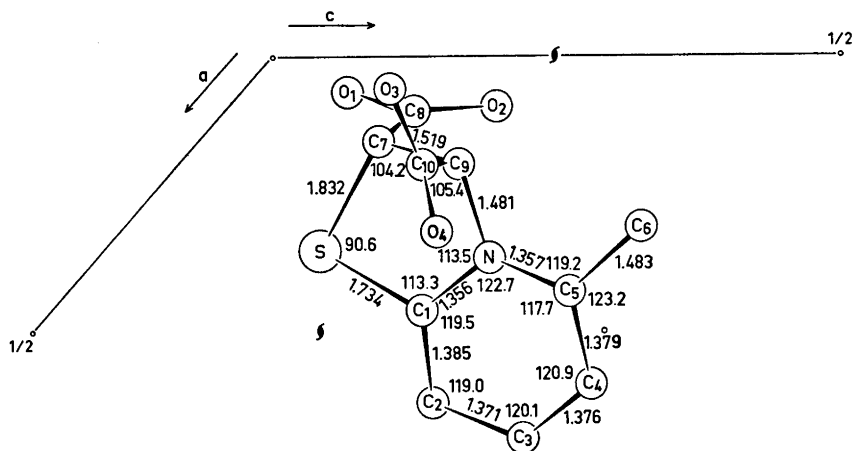


Fig. 1. Schematic drawing of the molecule viewed along [010].

Table 3. Continued.

Table with 4 columns of data (h, k, l, F0, Fc) repeated four times. Each column set contains numerical values for various indices and coordinates.

Table 3. Continued.

h	k	l	F _o	F _c	h	k	l	F _o	F _c	h	k	l	F _o	F _c	h	k	l	F _o	F _c
6	6	-7	73	-60	7	4	-12	97	-50	8	3	-8	120	-125	10	0	-18	122	-126
6	6	-6	198	-141	7	4	-11	202	-267	8	3	-5	154	-139	10	0	-16	162	-99
6	6	-4	159	167	7	4	-10	201	207	8	3	-4	154	-142	10	0	-14	91	55
6	6	-3	96	-57	7	4	-9	369	368	8	3	-3	174	-171	10	0	-10	250	-282
6	6	-2	129	128	7	4	-8	198	-194	8	3	-1	129	131	10	0	-6	325	-250
6	7	-10	79	68	7	4	-7	96	-89	8	4	-13	211	-202	10	0	-6	202	-201
6	7	-9	71	-29	7	4	-5	76	54	8	4	-12	114	100	10	0	-4	154	140
6	7	-8	87	-95	7	4	-4	193	96	8	4	-11	176	192	10	1	-17	94	96
6	7	-6	86	-105	7	4	-2	76	98	8	4	-9	129	-152	10	1	-15	150	-100
6	7	-3	74	-85	7	5	-15	107	112	8	4	-8	129	-138	10	1	-14	142	144
6	7	-2	105	-76	7	5	-13	106	-104	8	4	-6	152	167	10	1	-13	96	105
7	0	-18	163	151	7	5	-11	65	-64	8	4	-5	70	63	10	1	-11	109	-108
7	0	-16	74	77	7	5	-7	95	-98	8	4	-4	92	-73	10	1	-6	77	89
7	0	-15	240	-244	7	5	-5	208	-209	8	4	-3	93	-70	10	1	-5	119	-119
7	0	-8	62	63	7	5	-3	234	-240	8	4	-2	92	68	10	1	-3	63	68
7	0	-6	91	-91	7	5	-2	113	-117	8	4	0	82	-104	10	1	-2	69	-56
7	0	-4	175	169	7	5	1	98	84	8	5	-15	63	-65	10	2	-18	159	-150
7	0	-2	336	344	7	6	-8	119	-105	8	5	-8	102	111	10	2	-17	105	-100
7	0	0	116	122	7	6	-7	126	-132	8	5	-7	151	130	10	2	-16	130	-169
7	0	2	222	222	7	6	-5	152	146	8	5	-4	108	97	10	2	-15	152	-141
7	0	4	72	49	7	6	-4	110	120	8	5	-2	103	83	10	2	-12	159	-109
7	1	-15	130	-130	7	6	-2	107	-103	8	6	-10	196	-175	10	2	-12	95	85
7	1	-14	507	-509	7	7	-8	76	49	9	0	-16	270	-276	10	2	-9	194	-106
7	1	-13	311	309	7	7	-6	185	-207	9	0	-14	322	-322	10	2	-7	257	-257
7	1	-12	94	-96	8	0	-18	175	-207	9	0	-12	216	-210	10	2	-4	91	75
7	1	-11	247	-255	8	0	-16	77	67	9	0	-10	71	-43	10	2	-3	122	51
7	1	-10	155	164	8	0	-14	66	-73	9	0	-8	162	-168	10	3	-16	77	64
7	1	-9	103	-179	8	0	-12	94	-83	9	0	-6	225	-258	10	3	-12	166	-189
7	1	-8	165	117	8	0	-10	105	-119	9	0	-4	226	-246	10	3	-10	123	112
7	1	-7	271	-271	8	0	-8	91	-69	9	1	-19	92	-107	10	3	-8	134	-144
7	1	-6	533	-535	8	0	0	294	-208	9	1	-17	86	71	10	3	-7	61	99
7	1	-5	351	-357	8	0	2	78	44	9	1	-16	89	88	10	3	-6	67	-46
7	1	-3	170	167	8	1	-18	152	-227	9	1	-13	101	-161	10	3	-5	63	-71
7	1	-2	195	171	8	1	-17	136	-110	9	1	-11	71	74	10	3	-4	147	138
7	1	-1	163	-131	8	1	-15	346	346	9	1	-10	127	-128	10	4	-12	95	89
7	1	0	157	-152	8	1	-14	240	-247	9	1	-9	69	-70	10	4	-9	157	126
7	1	1	125	110	8	1	-13	173	-195	9	1	-8	76	84	10	4	-8	137	-138
7	1	3	78	-60	8	1	-12	69	70	9	1	-7	132	130	10	4	-5	71	56
7	2	-17	242	-235	8	1	-10	65	66	9	1	-6	288	-298	10	5	-12	151	-137
7	2	-16	73	-98	8	1	-9	130	132	9	1	-3	196	-197	10	5	-10	197	-142
7	2	-15	145	135	8	1	-8	93	-89	9	1	-1	166	168	11	0	-18	98	-91
7	2	-14	105	-190	8	1	-6	216	-216	9	1	0	190	-87	11	0	-16	133	99
7	2	-12	251	-248	8	1	-5	193	-179	9	2	-19	76	-87	11	0	-14	102	-161
7	2	-11	164	-130	8	1	-4	142	130	9	2	-17	191	102	11	0	-6	77	46
7	2	-10	67	-61	8	1	-1	119	-116	9	2	-15	103	-78	11	1	-16	93	62
7	2	-9	286	288	8	1	0	162	79	9	2	-12	165	114	11	1	-15	117	104
7	2	-8	190	184	8	1	1	151	145	9	2	-11	69	49	11	1	-14	166	-149
7	2	-7	345	-350	8	1	3	95	-65	9	2	-9	214	-221	11	1	-13	102	-100
7	2	-6	146	-142	8	2	-17	74	-72	9	2	-8	162	-162	11	1	-9	84	69
7	2	-5	62	-55	8	2	-16	165	124	9	2	-7	242	-241	11	1	-8	156	-144
7	2	-1	78	-85	8	2	-15	226	-257	9	2	-4	63	-73	11	1	-6	116	110
7	2	0	158	-127	8	2	-14	147	-134	9	2	-3	143	125	11	2	-17	162	110
7	3	-16	134	-129	8	2	-13	212	-214	9	3	-16	105	-171	11	2	-11	152	116
7	3	-14	293	195	8	2	-11	218	-217	9	3	-15	83	79	11	2	-9	176	-170
7	3	-13	94	-76	8	2	-10	137	137	9	3	-11	114	-114	11	2	-8	142	-113
7	3	-12	345	-351	8	2	-8	97	-109	9	3	-7	106	-109	11	2	-7	91	80
7	3	-10	58	40	8	2	-7	106	-102	9	3	-6	63	64	11	3	-15	90	-62
7	3	-9	248	-250	8	2	-6	85	-89	9	3	-5	79	78	11	3	-14	243	-213
7	3	-8	190	137	8	2	-4	222	-228	9	3	-4	64	-90	11	3	-12	119	108
7	3	-6	247	-217	8	2	-3	117	114	9	3	-2	144	-143	11	3	-9	111	-121
7	3	-5	61	89	8	2	-2	64	62	9	4	-16	103	-151	11	4	-11	108	102
7	3	-4	333	336	8	2	1	119	-97	9	4	-14	91	-65	11	4	-10	124	-108
7	3	-1	76	-83	8	3	-16	229	-224	9	4	-13	69	-74	12	1	-15	194	-172
7	3	0	99	-96	8	3	-15	62	-98	9	4	-7	95	89	12	1	-14	75	47
7	3	1	166	174	8	3	-14	69	-56	9	4	-5	175	-179	12	1	-11	174	52
7	3	2	90	94	8	3	-12	74	-78	9	4	-3	69	64	12	1	-10	96	-99
7	4	-17	160	-149	8	3	-10	174	-181	9	5	-12	78	39	12	2	-13	70	64
7	4	-15	161	140	8	3	-9	65	-81	9	5	-11	229	-229	12	2	-11	70	-64
										9	5	-9	166	111	12	2	-10	101	-88

Bond distances in the six-membered ring agree within error limits with the micro wave results for pyridine⁹ (C-N: 1.340 ± 0.001 Å, and C-C: 1.394 ± 0.001 Å), while the C-N-C angle of 122.7° is significantly larger than in pyridine (116.8°). The six-membered ring is planar, the largest deviation from the least squares plane being only 0.009 Å.

In the five-membered ring, four of the atoms lie nearly in a plane (to within 0.05 Å), while the fifth, C₇, is out of the plane by 0.57 Å.

The ring geometry may be compared with those of thiazole and tetrahydrothiophene, which are known from microwave¹⁰ and electron diffraction¹¹ investigations (Table 7).

Taking into account the extent to which these distances and angles are comparable, the agreement is satisfactory. The dihedral angle S-C₇-C₉-N

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	$(\bar{u}^2)^{\dagger}$ Å	B (Å ²)
S	.096	-.079	.053	.227	4.08
	.076	.103	.020	.187	2.76
	-.007	.040	.055	.151	1.81
O ₁	.103	-.016	.010	.254	5.10
	.065	-.017	.077	.162	2.06
	.021	.134	.011	.136	1.47
O ₂	.108	-.058	.033	.234	4.31
	.049	.057	.069	.187	2.78
	.032	.109	-.019	.171	2.30
O ₃	-.054	.025	.030	.247	4.81
	.099	.069	.059	.164	2.13
	.048	-.114	.043	.143	1.61
O ₄	-.035	.019	.042	.248	4.84
	.088	-.085	.055	.186	2.74
	.078	.104	.037	.148	1.72
N	.121	.023	.052	.163	2.10
	-.011	.046	.052	.161	2.05
	.018	-.126	.928	.144	1.64
C ₁	.096	-.004	.077	.190	2.86
	-.066	.063	.015	.170	2.29
	.037	.120	-.005	.159	2.00
C ₂	.051	-.005	.076	.246	4.79
	.064	.112	.015	.181	.259
	.091	-.076	.016	.156	1.93
C ₃	.022	.012	-.050	.247	4.83
	.015	.134	.013	.913	2.96
	.120	-.019	.060	.174	2.38
C ₄	-.089	.006	.005	.230	4.18
	.003	-.135	.007	.185	2.72
	.085	.012	.078	.156	1.93
C ₅	.108	-.016	.017	.196	3.04
	.046	.104	.050	.153	1.84
	.036	-.085	.058	.134	1.43
C ₆	.089	.024	.077	.236	4.39
	.044	-.125	.010	.203	3.26
	.073	.047	-.014	.188	2.80
C ₇	.102	.010	.010	.190	2.86
	.063	.039	.075	.159	2.00
	.026	-.130	.023	.148	1.72
C ₈	-.049	.026	.034	.195	3.01
	.060	.119	.027	.163	2.10
	.096	-.061	.066	.129	1.31
C ₉	-.044	.034	.036	.173	2.38
	.036	-.120	.037	.153	1.84
	.109	.054	.606	.145	1.67
C ₁₀	.079	-.054	.072	.178	2.50
	.094	.042	.006	.175	2.42
	.003	.117	.031	.147	1.71

Table 5. Bond distances and angles with estimated standard deviations.^a

Distance	(Å)	Angle	(°)
O ₁ -C ₆	1.298 (4)	C ₁₀ -C ₉ -N	108.4 (3)
O ₂ -C ₈	1.212 (4)	C ₁₀ -C ₉ -C ₇	112.6 (3)
O ₃ -C ₁₀	1.271 (4)	C ₇ -S-C ₁	90.6 (2)
O ₄ -C ₁₀	1.228 (4)	S-C ₁ -N	113.3 (2)
C ₃ -C ₇	1.527 (5)	C ₁ -N-C ₉	113.5 (3)
C ₁₀ -C ₉	1.552 (5)	N-C ₉ -C ₇	105.4 (3)
S-C ₁	1.832 (3)	C ₉ -C ₇ -S	104.2 (2)
S-C ₁	1.734 (3)	S-C ₁ -C ₈	127.2 (3)
C ₁ -N	1.356 (4)	C ₉ -N-C ₅	123.2 (3)
N-C ₉	1.481 (4)	C ₁ -C ₂ -C ₃	119.0 (4)
C ₉ -C ₇	1.519 (4)	C ₂ -C ₃ -C ₄	120.1 (4)
C ₁ -C ₂	1.385 (4)	C ₃ -C ₄ -C ₅	120.9 (4)
C ₂ -C ₃	1.371 (5)	C ₄ -C ₅ -N	117.7 (3)
C ₃ -C ₄	1.376 (5)	C ₅ -N-C ₁	122.7 (3)
C ₄ -C ₅	1.379 (4)	N-C ₁ -C ₂	119.5 (3)
C ₅ -N	1.357 (4)	C ₄ -C ₅ -C ₆	123.2 (3)
C ₆ -C ₅	1.483 (5)	N-C ₅ -C ₆	119.2 (3)
O ₃ ...O ₁ '	2.471 (3)	C ₁ -C ₂ -H ₄	119 (2)
C ₂ -H ₂	0.95 (3)	H ₂ -C ₂ -C ₃	122 (2)
C ₃ -H ₃	0.99 (3)	C ₂ -C ₃ -H ₃	121 (2)
C ₄ -H ₄	0.92 (3)	H ₃ -C ₃ -C ₄	118 (2)
C ₇ -H ₇	1.00 (3)	C ₃ -C ₄ -H ₄	124 (2)
C ₉ -H ₉	1.02 (3)	H ₄ -C ₄ -C ₅	115 (2)
C ₆ -H _{6,1}	0.98 (3)	C ₅ -C ₆ -H ₇	112 (2)
C ₆ -H _{6,3}	0.98 (3)	S-C ₇ -H ₇	108 (2)
C ₆ -H _{6,3}	0.86 (3)	C ₉ -C ₇ -H ₇	109 (2)
O ₃ ...H _B	1.42 (5)	C ₇ -C ₉ -H ₉	110 (2)
H _B -O ₁ '	1.05 (5)	C ₁₀ -C ₉ -H ₉	110 (2)
		N-C ₉ -H ₉	110 (2)
		C ₆ -C ₅ -H _{6,1}	111 (2)
		C ₆ -C ₅ -H _{6,3}	111 (2)
		C ₅ -C ₆ -H _{6,3}	109 (2)
		H _{6,1} -C ₆ -H _{6,3}	109 (2)
		H _{6,1} -C ₆ -H _{6,3}	106 (3)
		H _{6,3} -C ₆ -H _{6,3}	111 (3)
		C ₁₀ -O ₃ ...O ₁ '	111.3 (2)
		O ₃ ...O ₁ '-C ₈	114.3 (2)
		C ₁₀ -O ₃ -H _B	113 (1)
		C ₈ '-O ₁ '-H _B	116 (2)
		O ₃ ...H _B -O ₁ '	177 (4)
Angle	(°)		
O ₁ -C ₆ -O ₂	126.5 (3)		
O ₁ -C ₆ -C ₇	112.6 (3)		
O ₂ -C ₆ -C ₇	121.0 (3)		
O ₃ -C ₁₀ -O ₄	127.4 (3)		
O ₃ -C ₁₀ -C ₉	113.5 (3)		
O ₄ -C ₁₀ -C ₉	119.1 (3)		
C ₉ -C ₇ -S	109.1 (2)		
C ₈ -C ₇ -C ₉	113.9 (3)		

^a For numbering of atoms, see Figs. 1 and 2.

Table 6. Dihedral angles.

Angle	(°)
S-C ₁ -N-C ₉	8.9 (4)
C ₁ -N-C ₉ -C ₇	30.8 (4)
N-C ₉ -C ₇ -S	36.8 (3)
C ₉ -C ₇ -S-C ₁	28.6 (2)
C ₇ -S-C ₁ -N	12.4 (3)

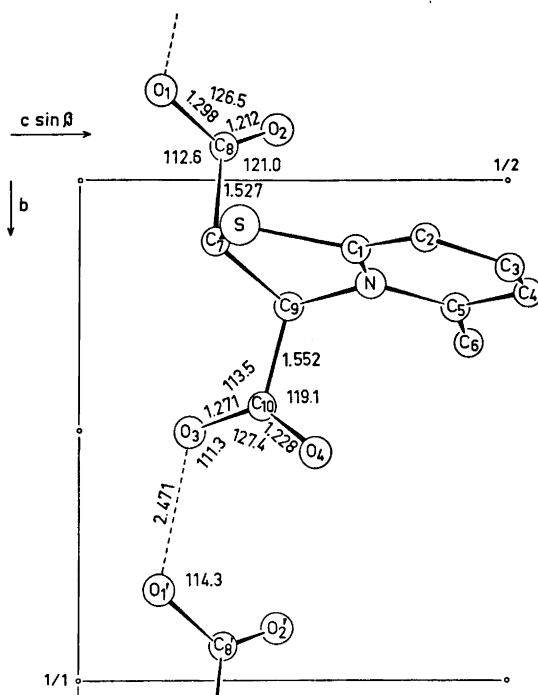


Fig. 2. Schematical drawing of the molecule viewed along [100].

(36.8°) agrees also fairly well with the tetrahydrothiophene S-C-C-C value of 40.4° (1.2°).

Although the *intermolecular* hydrogen bond between O₃ and O₁' is as short as 2.471 Å, it is far from symmetric (O₃···H_B = 1.42 Å, and H_B-O₁' = 1.05 Å), and no evidence for disorder has been observed. The hydrogen atom was localized in the difference Fourier map about 1.0 Å from O₁', and no

Table 7. Ring geometry of the title compound compared to those of tetrahydrothiophene and thiazole.

Distance	Tetrahydrothiophene	Thiazole	Present compound
S-C	1.839 (2)		1.832 (3) (S-C ₇)
S-C		1.726 (1)	1.734 (3) (S-C ₁)
C-C	1.536 (2)		1.519 (4) (C ₇ -C ₉)
Angle			
C-S-C	93.4 (5)	98.33 (3)	90.6 (2) (C ₁ -S-C ₇)
S-C-C	106.1 (4)		104.1 (2) (S-C ₇ -C ₉)
S-C-N		115.18 (1)	113.3 (2) (S-C ₁ -N)
C-C-C	105.0 (5)		105.4 (3) (C ₇ -C ₉ -N)
C-N-C		110.12 (2)	113.5 (3) (C ₁ -N-C ₉)

abnormal shifts occurred when refining the coordinates and isotropic temperature factors. Distances and angles of the carboxyl groups are normal.

Fig. 2 clearly shows that the configuration of the molecule is *trans*, in agreement with NMR evidence.⁴ The dihedral angle C₈-C₇-C₉-C₁₀ is 160.1°, and the angle between the planes C₈, O₁, O₂ and C₁₀, O₃, O₄ is 114.3°.

There are some relatively short *intermolecular* contacts between oxygens and H₃, H₄, H₇ (2.35 Å, 2.42 Å, 2.47 Å).

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