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Crystal Structure of *cis*-2,3-(3′-Cyclohexanon-1′,2′-ylene)-5-methyl-8-ethoxydihydrothiazolo[3,2-a]-pyridinium Bromide

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The crystals are triclinic with space group $P\bar{1}$ and two molecules in the unit cell. The Dirichlet's reduced unit cell has the following parameters: $a = 6.89_5$ Å, $b = 10.19_6$ Å, $c = 11.25_9$ Å, $\alpha = 103{,}1_4°$, $\beta = 92.5_0°$, $\gamma = 107.5_2°$. The structure was solved by the heavy atom method, and the R-value arrived at was 4.5 % ($R_w = 6.7$ %) for 1785 reflections, which were measured on a four-circle diffractometer. The two S – C bond distances are 1.84_4 Å and 1.72_2 Å. The five-membered ring has the envelope conformation with C_9 0.6 Å out of the plane through the four other atoms. The cyclohexanone ring has the chair form and the angle C – (CO) – C is 115°. The bond distance $C-OC_2H_5$ of the ethoxy group is 1.36_0 Å.

When an α-bromo-α-β-unsaturated acid carries a β-substituent it has been shown [1,2] that the reaction with pyrid-2-thiones gives dihydrothiazolo [3,2-a]pyridinium-3-carboxylate derivatives where the configuration at the $C_2 - C_3$ carbons in the cyclic product is *trans* irrespective of the stereochemistry in the unsaturated acid.

Corresponding reactions with α-bromo-α,β-unsaturated cyclic ketones have recently been studied by Lie and Undheim.[3] In the case of a planar five-membered ring condensed at the 2,3 position a *cis* configuration of the product has been established.[3]

However, for a six-membered ring the *trans* configuration seems to be quite feasible and the NMR spectra cannot unambiguously be used to assign *cis* or *trans* fusion of the cyclohexanone ring. In order to clarify this problem, and to determine the conformation of the cyclohexanone ring, the crystal structure determination of 2,3-(3′-cyclohexanon-1′,2′-ylene)-5-methyl-8-ethoxydihydrothiazolo[3,2-a]pyridinium bromide has been carried out.

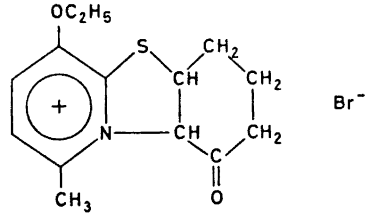

Table 1. Fractional atomic coordinates and anisotropic thermal vibration parameters with estimated standard deviations (multiplied by 10^5).[a] The temperature factor is given by $\exp[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)$.

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Br^-	12919	35322	18845	1883	836	965	663	280	416
	7	5	5	21	10	8	20	16	12
S	28149	06693	34161	1513	700	742	647	852	74
	19	14	12	37	19	14	42	33	25
O_1	01541	−22527	22296	1495	746	857	132	488	163
	50	37	30	93	48	36	108	91	66
O_2	76325	−02793	36330	2343	1168	796	1458	76	372
	57	42	32	111	60	37	137	98	74
N	51122	−01423	18403	1350	655	511	437	232	250
	55	41	33	106	54	36	126	96	71
C_1	−30694	−37531	25087	1688	1030	1285	15	1025	698
	86	66	58	150	87	71	187	164	124
C_2	−13662	−36415	17096	1827	584	995	11	204	241
	82	56	52	148	68	59	168	145	100
C_3	19421	−19432	17249	1048	743	649	371	197	384
	69	53	43	121	68	47	153	117	91
C_4	25207	−28019	07959	1975	771	598	748	−25	−119
	80	56	44	148	72	47	174	130	92
C_5	44396	−23068	04032	1876	809	554	721	357	−127
	79	57	45	147	75	45	176	129	91
C_6	57874	−09924	09342	1716	868	479	1124	474	300
	73	56	40	138	71	44	170	122	90
C_7	78718	−04312	05964	1512	1190	731	938	831	294
	79	63	46	135	83	52	179	132	105
C_8	32803	−05734	22458	1171	735	533	781	271	298
	68	52	40	123	67	43	154	112	85
C_9	50557	20496	31384	1732	616	820	700	676	376
	77	54	47	140	66	52	164	134	95
C_{10}	60127	32587	42969	2374	712	1011	373	415	46
	91	60	54	169	74	62	187	159	108
C_{11}	70863	28190	52796	2753	1207	740	1196	664	208
	94	66	51	181	91	54	217	155	111
C_{12}	87365	24186	46869	1991	1047	776	686	130	64
	84	63	49	155	83	54	188	145	108
C_{13}	76607	09105	36685	1434	843	615	575	478	243
	73	56	43	131	73	48	162	121	94
C_{14}	64756	12315	26533	1493	512	569	81	420	81
	72	49	41	129	62	45	148	117	83

The crystals are triclinic with space group $P\bar{1}$. The dimensions of the Dirichlet's reduced unit cell,[4] determined by a manual four-circle diffractometer, with estimated standard deviations,[5]* are:

$$a = 6.895(4)\ \text{Å},\ b = 10.196(6)\text{Å},\ c = 11.259(4)\ \text{Å}$$
$$\alpha = 103.14(2)^\circ,\ \beta = 92.50(2)^\circ,\ \gamma = 107.52(2)^\circ$$

The unit cell contains two molecules ($\varrho_{calc} = 1.56$ g cm^{-1}, $\varrho_{obs} = 1.55$ g cm^{-1}).

* All programs used are included in this reference.

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

Table 2. Fractional atomic coordinates for the hydrogen atoms (H_m and $H_{m,n}$ are bonded to C_m). The B-value was taken as 4.0 Å^2.

Atom	x	y	z
$H_{2,1}$	−.066	−.443	.181
$H_{2,2}$	−.166	−.365	.089
H_4	.144	−.389	.035
H_5	.488	−.287	−.032
$H_{10,1}$	.703	.387	.389
$H_{10,2}$	.621	.292	.493
$H_{11,1}$	.202	.757	.529
$H_{11,2}$	.375	.786	.427
$H_{12,1}$	.967	.185	.526
$H_{12,2}$	.991	.280	.421
H_9	.468	.248	.245
H_{14}	.745	.183	.218

With 2θ-max. equal to 45° and Mo$K\alpha$-radiation, 1852 reflections were measured by an automatic four-circle diffractometer. With an observed-unobserved cutoff at 2.5 $\sigma(I)$, 1785 were recorded as observed. The intensities were corrected for absorption effects.

The structure was solved by the heavy atom method and refined by full-matrix least squares technique. Except for the methyl hydrogens, the H-positions were calculated assuming C−H bond lengths of 1.03 Å. None of the six methyl hydrogens could be localized in the difference Fourier map, and are not included in the calculations. Only the positional parameters were refined for H-atoms. Anisotropic temperature factors were introduced for all the heavy atoms, and the weights in least squares were calculated from the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I) = [C_T + (0.02C_N)^2]^{\frac{1}{2}}$$

were C_T is the total number of counts and C_N the net count (peak minus background). The conventional R-value arrived at was 4.5 % (weighted value $R_w = 6.7$ %) for 1785 observed reflections. The form factors used were those of Hanson *et al.*[6] except for hydrogen.[7] Final fractional coordinates with estimated standard deviations are given in Tables 1 and 2. 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.

A rigid-body analysis of translational, librational and screw motion [8] was carried out on the 13 ring atoms. The 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 was 0.0032. This

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

h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc
0	0	1	167	108	0	5	-1	366	- 330	1	-8	3	91	56	1	-2	-10	65	- 59	1	2	9	219	- 230
0	0	2	774	- 717	0	5	0	119	109	1	-8	4	142	136	1	-2	-9	62	44	1	2	10	114	- 100
0	0	3	474	- 478	0	5	1	503	486	1	-8	5	50	- 47	1	-2	-8	71	64	1	3	-11	86	84
0	0	4	37	- 5	0	5	2	247	237	1	-8	6	124	- 122	1	-2	-7	231	216	1	3	-10	70	67
0	0	5	481	451	0	5	3	293	- 267	1	-8	7	100	- 94	1	-2	-6	110	- 105	1	3	-9	312	- 318
0	0	6	339	332	0	5	4	280	- 250	1	-8	8	21	23	1	-2	-5	669	- 651	1	3	-8	159	- 162
0	0	7	451	- 431	0	5	5	256	242	1	-8	9	167	161	1	-2	-4	408	- 388	1	3	-7	250	264
0	0	8	315	- 306	0	5	6	294	295	1	-7	-7	120	- 125	1	-2	-3	417	424	1	3	-6	121	115
0	0	11	172	166	0	5	7	129	121	1	-7	-6	210	- 202	1	-2	-2	763	717	1	3	-5	76	78
0	1	-10	182	- 176	0	5	8	36	- 26	1	-7	-4	87	84	1	-2	-1	62	- 65	1	3	-4	315	- 316
0	1	-9	31	- 28	0	6	-11	148	142	1	-7	-3	574	595	1	-2	0	690	- 713	1	3	-3	417	- 399
0	1	-8	155	147	0	6	-10	83	79	1	-7	-2	98	92	1	-2	1	526	- 541	1	3	-2	139	141
0	1	-7	53	43	0	6	-9	257	- 267	1	-7	-1	456	- 429	1	-2	2	1026	1066	1	3	-1	655	675
0	1	-6	44	45	0	6	-8	314	- 319	1	-7	0	151	- 144	1	-2	3	1427	1530	1	3	0	400	- 392
0	1	-5	498	- 504	0	6	-7	268	270	1	-7	1	239	225	1	-2	5	434	- 457	1	3	1	294	- 281
0	1	-4	515	- 534	0	6	-6	110	110	1	-7	2	305	292	1	-2	6	197	- 195	1	3	2	499	- 505
0	1	-3	466	459	0	6	-5	138	130	1	-7	3	149	159	1	-2	8	197	198	1	3	3	253	259
0	1	-2	319	- 365	0	6	-4	56	- 60	1	-7	4	381	- 347	1	-2	9	142	145	1	3	4	356	354
0	1	-1	91	67	0	6	-3	364	- 354	1	-7	5	443	- 429	1	-2	10	204	- 201	1	3	5	167	162
0	1	0	499	470	0	6	-2	148	- 153	1	-7	6	169	163	1	-2	11	139	- 138	1	3	6	194	- 175
0	1	1	479	- 536	0	6	-1	163	169	1	-7	7	178	177	1	-1	-11	168	- 169	1	3	7	24	- 10
0	1	2	371	342	0	6	0	75	88	1	-7	8	97	95	1	-1	-10	212	213	1	3	8	132	142
0	1	3	792	776	0	6	1	45	42	1	-7	9	56	- 51	1	-1	-9	153	152	1	3	9	94	85
0	1	4	63	65	0	6	2	179	- 169	1	-7	10	115	- 105	1	-1	-8	33	- 35	1	4	-11	130	- 118
0	1	5	53	48	0	6	3	359	- 365	1	-6	-8	68	- 72	1	-1	-7	149	- 162	1	4	-10	45	- 43
0	1	6	47	- 47	0	6	4	72	69	1	-6	-7	182	- 176	1	-1	-6	209	- 212	1	4	-9	96	95
0	1	7	388	- 375	0	6	5	327	319	1	-6	-6	124	123	1	-1	-5	30	34	1	4	-8	322	325
0	1	8	140	135	0	6	6	101	100	1	-6	-5	72	65	1	-1	-4	216	218	1	4	-7	30	23
0	1	9	198	153	0	6	7	66	- 62	1	-6	-4	127	- 122	1	-1	-3	45	- 32	1	4	-6	594	- 605
0	1	10	86	- 85	0	7	-10	26	19	1	-6	-3	94	- 92	1	-1	-2	603	- 625	1	4	-5	236	- 232
0	1	11	37	- 36	0	7	-9	93	- 94	1	-6	-2	105	- 102	1	-1	-1	142	- 132	1	4	-4	255	244
0	2	-11	83	- 79	0	7	-8	159	162	1	-6	-1	89	83	1	-1	0	497	- 544	1	4	-3	57	65
0	2	-9	322	321	0	7	-7	206	193	1	-6	0	609	580	1	-1	1	154	142	1	4	-2	462	447
0	2	-8	97	89	0	7	-6	279	- 280	1	-6	1	175	178	1	-1	2	401	390	1	4	-1	59	- 64
0	2	-7	304	- 301	0	7	-5	207	- 210	1	-6	2	386	- 355	1	-1	3	432	432	1	4	0	593	- 610
0	2	-6	237	- 263	0	7	-4	17	15	1	-6	3	76	- 70	1	-1	4	354	- 395	1	4	1	337	- 336
0	2	-5	11	- 8	0	7	-3	177	176	1	-6	4	76	- 56	1	-1	5	50	52	1	4	2	207	192
0	2	-4	294	287	0	7	-2	382	381	1	-6	5	189	184	1	-1	6	297	290	1	4	3	273	272
0	2	-3	456	500	0	7	-1	44	- 37	1	-6	6	218	219	1	-1	7	181	183	1	4	4	104	100
0	2	-2	825	- 884	0	7	0	526	- 533	1	-6	7	174	- 165	1	-1	8	66	57	1	4	5	338	- 330
0	2	-1	299	- 308	0	7	1	73	- 74	1	-6	8	183	- 175	1	-1	9	87	- 94	1	4	6	318	- 321
0	2	0	281	- 328	0	7	2	242	238	1	-6	10	54	59	1	-1	10	251	- 239	1	4	7	200	206
0	2	1	223	223	0	7	3	96	98	1	-6	11	102	95	1	-1	11	98	91	1	4	8	235	222
0	2	2	530	474	0	7	4	42	38	1	-5	-9	29	- 22	1	0	-11	60	58	1	5	-11	78	76
0	2	3	395	365	0	7	5	205	- 206	1	-5	-8	162	158	1	0	-10	85	88	1	5	-10	150	151
0	2	4	939	- 874	0	7	6	153	- 161	1	-5	-7	30	27	1	0	-9	214	- 205	1	5	-9	30	26
0	2	5	22	- 16	0	8	-9	20	- 14	1	-5	-6	46	54	1	0	-8	230	- 237	1	5	-8	67	61
0	2	6	293	310	0	8	-8	29	21	1	-5	-5	288	- 250	1	0	-7	239	235	1	5	-7	204	- 208
0	2	7	97	86	0	8	-7	158	- 168	1	-5	-4	373	- 346	1	0	-6	375	371	1	5	-6	162	- 157
0	2	8	134	130	0	8	-6	148	- 154	1	-5	-3	156	153	1	0	-5	129	130	1	5	-5	362	345
0	2	9	204	- 195	0	8	-5	215	223	1	-5	-2	403	414	1	0	-4	290	- 261	1	5	-4	268	277
0	2	10	119	- 110	0	8	-4	259	267	1	-5	-1	40	- 32	1	0	-3	781	- 785	1	5	-3	439	- 441
0	3	-11	107	102	0	8	-3	186	- 186	1	-5	0	93	- 73	1	0	-2	100	97	1	5	-2	218	- 205
0	3	-10	51	52	0	8	-2	122	- 124	1	-5	1	481	- 452	1	0	-1	506	476	1	5	-1	69	- 88
0	3	-9	47	- 47	0	8	-1	127	- 125	1	-5	2	75	- 57	1	0	0	89	87	1	5	0	301	304
0	3	-8	304	- 313	0	8	0	39	45	1	-5	3	643	641	1	0	1	165	- 226	1	5	1	414	411
0	3	-7	44	- 56	0	8	1	361	372	1	-5	4	128	134	1	0	2	573	- 634	1	5	2	90	87
0	3	-6	412	431	0	8	2	48	- 51	1	-5	5	131	- 131	1	0	3	320	- 312	1	5	3	534	- 540
0	3	-5	305	283	0	8	3	165	- 168	1	-5	6	307	- 296	1	0	4	269	258	1	5	4	76	65
0	3	-4	191	- 181	0	8	4	36	- 45	1	-5	7	83	- 98	1	0	5	506	523	1	5	5	80	78
0	3	-3	447	- 471	0	9	-8	39	- 40	1	-5	8	133	131	1	0	6	15	- 5	1	5	6	92	80
0	3	-2	399	- 408	0	9	-7	101	97	1	-5	9	258	255	1	0	7	428	- 421	1	5	7	45	52
0	3	-1	451	461	0	9	-6	64	62	1	-5	10	85	- 84	1	0	8	17	8	1	5	8	172	- 176
0	3	0	849	817	0	9	-5	115	118	1	-5	11	74	- 73	1	0	9	98	104	1	6	-10	102	- 102
0	3	1	168	- 152	0	9	-4	163	- 168	1	-4	-9	240	234	1	0	10	54	49	1	6	-9	121	- 117
0	3	2	201	- 202	0	9	-3	241	- 240	1	-4	-8	106	110	1	0	11	56	56	1	6	-8	107	102
0	3	3	329	- 307	0	9	-2	156	155	1	-4	-7	269	- 265	1	1	-11	143	- 141	1	6	-7	187	182
0	3	4	101	- 94	0	9	-1	107	109	1	-4	-6	205	- 202	1	1	-10	64	- 63	1	6	-6	143	141
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0	3	6	37	38	0	9	1	21	- 8	1	-4	-4	233	238	1	1	-8	202	210	1	6	-4	334	- 353
0	3	7	309	- 288	0	9	2	215	- 224	1	-4	-3	484	483	1	1	-7	292	286	1	6	-3	202	- 212
0	3	8	154	- 143	0	9	3	32	- 33	1	-4	-2	338	- 329	1	1	-6	366	- 366	1	6	-2	382	386
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0	3	10	69	67	0	10	-5	54	- 52	1	-4	0	172	153	1	1	-4	94	95	1	6	0	200	- 198
0	4	-11	173	- 170	0	10	-4	34	- 35	1	-4	1	268	269	1	1	-3	253	309	1	6	1	39	- 41
0	4	-10	81	- 80	0	10	-3	105	111	1	-4	2	294	271	1	1	-2	879	912	1	6	2	204	- 208
0	4	-9	15	16	0	10	-2	150	156	1	-4	3	17	- 8	1	1	-1	540	- 582	1	6	3	62	56
0	4	-8	131	123	0	10	-1	116	- 118	1	-4	4	527	- 521	1	1	0	541	- 553	1	6	4	301	309
0	4	-7	170	169	0	10	0	135	- 138	1	-4	5	444	- 425	1	1	1	415	- 419	1	6	6	97	- 89
0	4	-6	90	92	1	-10	-1	121	- 124	1	-4	6	284	275	1	1	2	683	705	1	6	7	31	- 40
0	4	-5	465	- 480	1	-10	0	222	- 220	1	-4	7	202	204	1	1	3	360	341	1	7	-10	71	- 65
0	4	-4	205	- 186	1	-10	1	71	74	1	-4	8	20	13	1	1	4	189	202	1	7	-9	165	159
0	4	-3	171	178	1	-10	2	211	203	1	-4	9	138	- 132	1	1	5	307	- 306	1	7	-8	146	145
0	4	-2	371	385	1	-10	3	72	79	1	-4	10	166	- 158	1	1	6	298	- 303	1	7	-7	20	- 15
0	4	-1	177	154	1	-10	4	66	- 56	1	-4	11	89	88	1	1	7	31	37	1	7	-6	273	- 277
0	4	0	295	- 235	1	-10	5	184	- 177	1	-3	-10	37	- 27	1	1	8	291	277	1	7	-5	74	- 70
0	4	1	432	- 436	1	-10	6	29	- 35	1	-3	-9	250	- 253	1	1	10	158	- 160	1	7	-4	51	35
0	4	2	417	407	1	-9	-3	214	- 217	1	-3	-7	39	- 25	1	2	-11	21	- 22	1	7	-3	234	241
0	4	3	344	338	1	-9	-2	92	- 89	1	-3	-6	291	280	1	2	-10	237	241	1	7	-2	177	174
0	4	4	243	221	1	-9	-1	123	- 121	1	-3	-5	51	55	1	2	-9	27	- 23	1	7	-1	278	- 282
0	4	5	21	22	1	-9	0	229	228	1	-3	-4	194	- 186	1	2	-8	113	- 117	1	7	0	285	- 288
0	4	6	244	- 240	1	-9	1	247	236	1	-3	-3	103	- 109	1	2	-7	90	- 82	1	7	1	147	143
0	4	7	164	- 152	1	-9	2	229	- 221	1	-3	-2	189	- 169	1	2	-6	253	- 251	1	7	2	119	127
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Table 3. Continued.

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2	-10	2	71	- 70
2	-10	3	115	105
2	-10	4	126	- 120
2	-10	5	214	- 201
2	-10	6	74	71
2	-10	7	112	115
2	-9	-4	146	- 147
2	-9	-3	162	- 171
2	-9	-2	39	- 41
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2	-8	-1	20	- 26
2	-8	0	53	- 50
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3	0	1	428	- 443
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2	7	-1	142	- 138
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Table 3. Continued.

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3	1	-4	413	422
3	1	-3	580	576
3	1	-2	309	- 288
3	1	-1	683	- 639
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3	1	1	308	311
3	1	2	160	161
3	1	3	117	- 114
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3	1	5	57	- 48
3	1	6	93	99
3	1	7	173	174
3	1	8	87	- 83
3	1	9	41	- 39
3	2	-11	29	29
3	2	-10	169	- 170
3	2	-9	47	- 42
3	2	-8	177	- 183
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3	2	-3	71	- 65
3	2	-2	166	- 163
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3	2	2	309	- 326
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3	2	4	106	99
3	2	5	124	125
3	2	6	184	183
3	2	7	146	- 147
3	2	8	126	- 128
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4	10	2	187	198
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Table 3. Continued.

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5	2	-7	162	163	6	-7	4	58	62	6	-2	0	115	102	6	3	0	143	137	2	0	-8	14	- 1
5	2	-6	53	- 63	6	-6	-5	105	- 111	6	-2	1	155	164	7	-6	-1	180	- 176	2	1	-10	16	- 11
5	2	-5	264	- 283	6	-6	-4	112	114	6	-2	2	147	- 148	7	-6	0	129	- 121	2	1	9	18	13
5	2	-4	176	- 176	6	-6	-3	145	150	6	-2	3	210	- 216	7	-6	1	182	182	2	2	7	16	2
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5	4	2	180	- 187	6	-4	6	149	- 154	6	1	-4	96	- 90	7	-1	1	118	- 119	5	-5	-3	17	- 2
5	5	-5	101	- 104	6	-3	-7	36	- 47	6	1	-3	214	- 216	7	0	-2	142	- 130	5	-3	-8	18	2
5	5	-4	137	- 131	6	-3	-6	180	- 180	6	1	-2	47	- 48	7	0	-1	30	- 29	5	-3	-7	17	- 7
5	5	-3	97	92	6	-3	-5	65	74	6	1	-1	84	68	8	0	0	25	- 24	5	-1	5	16	12
5	5	-2	246	244	6	-3	-4	260	259	6	1	0	177	175	0	0	10	23	6	5	1	4	17	12
5	5	-1	30	- 31	6	-3	-3	76	- 80	6	1	1	65	64	0	1	-11	17	12	5	4	-7	19	28
5	5	0	168	- 171	6	-3	-2	104	- 102	6	1	2	365	- 354	0	2	-10	15	- 1	5	4	-2	22	- 14
6	-8	-2	160	- 172	6	-3	-1	93	- 76	6	1	3	146	- 144	0	8	5	21	12	6	-8	-1	24	22
6	-8	0	240	222	6	-3	0	236	- 204	6	2	-6	142	- 137	1	-9	7	19	1	6	-8	1	24	23
6	-8	2	90	- 84	6	-3	1	247	273	6	2	-5	54	- 38	1	-7	-5	19	8	6	-7	5	18	11
6	-8	3	107	- 110	6	-3	2	164	172	6	2	-4	66	- 58	1	-6	9	16	- 4	6	-4	5	17	16
6	-8	4	95	- 103	6	-3	3	110	- 114	6	2	-3	162	155	1	-3	-9	17	- 7	6	-3	5	17	- 14
6	-7	-3	79	73	6	-3	4	134	- 146	6	2	-2	212	190	1	-2	7	13	- 33	6	-3	6	20	5
6	-7	-2	121	131	6	-2	-7	71	65	6	2	-1	127	- 122	1	1	9	17	4	6	-1	-1	19	- 10
6	-7	-1	157	154	6	-2	-6	53	43	6	2	0	159	- 157	1	2	8	16	- 16	6	-2	1	21	3
6	-7	0	64	- 53	6	-2	-5	165	169	6	2	2	38	45	1	6	5	19	- 16	7	-5	0	26	- 8
6	-7	1	224	- 217	6	-2	-4	39	- 36	6	3	-4	67	72	1	8	-9	18	4	7	-4	1	17	- 12
					6	-2	-3	343	- 347	6	3	-3	42	- 41	1	9	-3	19	3					

number indicates that the ring system may be regarded as an oscillating rigid body, and the corresponding coordinates were corrected for librational motion. However, the largest angular oscillation amplitude was only 2.50°, and none of the corrections were greater than one e.s.d. The final coordinates as well as the bond distances and angles are therefore given with their uncorrected values (Tables 1, 2, and 5). Some of the distances and angles are also shown in Fig. 1 where the molecule is viewed along [100]. It clearly shows that the configuration is *cis*.

Bond distances and angles and the degree of planarity of the aromatic six-membered ring agree within probable limits of error with those of *trans* 2-carboxy-5-methyl-dihydro-thiazolo[3,2-a]pyridinium-3-carboxylate.[2] The five-membered ring has the envelope conformation with one of the carbon atoms, C_9, 0.60 Å out of the plane through the four other atoms (which are coplanar to within 0.05 Å). The corresponding value for the carboxylate [2] is 0.57 Å.

The distance $C_3 - O_1$ of the ethoxy group is 1.360 Å, which is somewhat shorter than the methoxy distance (1.394 Å) of 3-carbomethoxy-5-anisyl-1,2,4-trioxacyclopentane,[9] but agrees closely with the anisole value of 1.363 Å [10] obtained by an electron diffraction investigation of the free molecule in the gas phase, and with the two distances found in the crystals of *p*-azoxyanisole [11]

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	$(\bar{u}^2)^{\frac{1}{2}}$Å	B(Å^2)	Atom	e_x	e_y	e_z	$(\bar{u}^2)^{\frac{1}{2}}$Å	B(Å^2)
	.005	.007	.090	.242	4.63	C_5	.033	−.060	.052	.230	4.18
Br^-	−.123	.033	.003	.207	3.38	C_6	.150	.048	.000	.200	3.15
	.092	.101	.017	.192	2.90		.000	.074	.076	.154	1.88
	.063	−.021	.072	.240	4.54		.117	.091	.021	.205	3.32
S	.106	.096	.020	.178	2.50	C_7	.076	.041	.052	.184	2.68
	−.092	.040	.053	.150	1.77		−.065	.036	.072	1.58	1.97
	.052	−.030	.070	.245	4.76		.016	−0.76	.039	.244	4.71
O_1	−0.61	.062	.058	.199	3.11	C_8	.092	.075	.071	.216	3.70
	.131	.081	.012	.162	2.07		−1.22	.003	.044	.155	1.90
	.107	.072	−.030	.245	4.75		.060	.097	−.014	.183	2.64
O_2	.021	.069	.082	.217	3.71	C_9	.046	.040	.090	.179	2.54
	.108	−.037	.030	.203	3.25		−.133	.018	0.12	.151	1.80
	.077	−.053	.037	.185	2.69		.072	.014	.083	.230	4.18
N	−.032	.042	.083	.173	2.37	C_9	.133	.012	−0.35	.182	2.63
	.129	.083	.012	.165	2.15		.025	.105	.016	.165	2.14
	.059	.002	.083	.285	6.43		.056	−.022	.073	.266	5.60
C_1	−.046	.083	.037	.245	4.72	C_{10}	−.126	.004	.040	.231	4.21
	.134	.067	−.011	.158	.198		.067	.104	.038	.174	2.38
	.020	−.004	.088	.250	4.92		.092	−.038	.043	.254	5.09
C_2	−.118	.036	.018	.223	3.92	C_{11}	.119	.085	−.001	.240	4.55
	.096	.100	.022	.152	1.82		−.028	.051	.081	.197	3.05
	.005	.040	.090	.198	3.09		.005	−.066	.052	.247	4.82
C_3	.023	−.087	.016	.188	2.78	C_{12}	−.124	.015	.037	.214	3.62
	.151	.046	.006	.149	1.76		.091	.082	.066	.194	2.97
	.054	.061	−.057	.227	4.08		.046	−.056	.051	.213	3.57
C_4	.137	−.013	.013	.207	3.38	C_{13}	.069	.090	.065	.187	2.77
	.043	.086	.071	.160	2.02		.129	.012	−.040	.167	2.20
							.091	−.031	.052	.216	3.69
						C_{14}	−.080	.018	.071	.172	2.34
							.094	.100	.028	.143	1.62

(1.352 Å and 1.359 Å). The C−O−C angles are about 118° for all these compounds.

As in the case of the free molecule,[12] the cyclohexanone ring has the chair conformation. The bond distance $C_{11}-C_{12}$ is very long (1.592(8) Å), but, according to error limits, not significantly longer than corresponding bonds in cyclohexanone [12] (1.54(1) Å), cyclohexanone-1,4-dione [13] (1.552(7) Å and 1.557(10) Å) and 2,2,6,6-tetramethyl-4-piperidone-*N*-oxyl [14] (1.57(2) Å) (the last value is also for the free molecule).

The dihedral angles $N-C_{14}-C_9-C_{10}$ and $S-C_9-C_{14}-C_{13}$ are 161.1° and 79.5°, respectively.

No short *inter*-molecular contacts are observed. It should, however, be pointed out that the six methyl hydrogens are not considered in the analysis.

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

Distance	(Å)	Angle	(°)
C_1-C_2	1.503(8)	$C_1-C_2-O_1$	1.063(4)
C_2-O_1	1.457(8)	$C_2-O_1-C_3$	117.6(4)
O_1-C_3	1.360(6)	$O_1-C_3-C_4$	127.4(4)
C_3-C_4	1.360(7)	$O_1-C_3-C_8$	113.9(4)
C_4-C_5	1.397(7)	$C_3-C_4-C_8$	119.5(5)
C_5-C_4	1.362(7)	$C_4-C_5-C_5$	122.3(5)
C_6-C_7	1.480(7)	$C_5-C_6-C_6$	124.9(5)
C_6-N	1.375(6)	C_7-C_6-N	119.0(5)
$N-C_8$	1.343(6)	C_5-C_6-N	116.2(4)
C_8-C_3	1.397(7)	C_6-N-C_7	123.5(4)
C_8-S	1.722(5)	$N-C_8-C_2$	119.8(4)
$S-C_9$	1.844(5)	$N-C_8-S$	114.7(4)
C_9-C_{14}	1.511(7)	$C_8-C_3-C_4$	118.7(4)
$C_{14}-N$	1.494(6)	C_3-C_8-S	125.5(3)
$C_{13}-C_{14}$	1.523(7)	C_8-N-C_{14}	112.0(4)
$C_{13}-O_2$	1.199(6)	C_6-N-C_{14}	123.5(4)
$C_{12}-C_{13}$	1.470(8)	$N-C_{14}-C_9$	105.2(4)
$C_{11}-C_{12}$	1.592(8)	$C_{14}-C_9-S$	103.5(3)
$C_{10}-C_{11}$	1.525(9)	C_9-S-C_8	90.0(2)
C_9-C_{10}	1.536(8)	$N-C_{14}-C_{13}$	108.6(4)
		$C_9-C_{14}-C_{13}$	112.9(4)
		$C_{14}-C_{13}-O_2$	121.2(4)
		$C_{14}-C_{13}-C_{12}$	114.9(5)
		$O_2-C_{13}-C_{12}$	123.9(5)
		$C_{13}-C_{12}-C_{11}$	108.1(4)
		$C_{12}-C_{11}-C_{10}$	108.9(5)
		$C_{11}-C_{10}-C_9$	113.7(5)
		$C_{13}-C_9-C_{14}$	113.7(4)
		$C_{10}-C_9-S$	112.5(4)

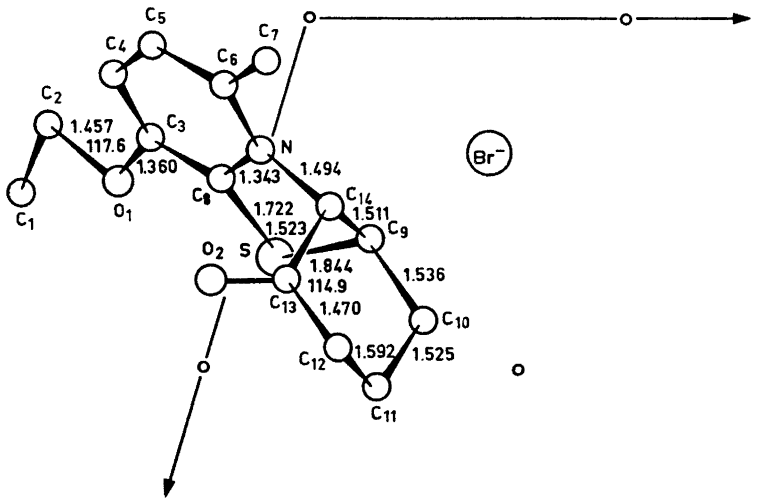

Fig. 1. Schematical drawing of the molecule viewed along [100].

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