0·011 (3) Å for C(9*A*) and −0·010 (3) Å for C(9). The remaining bond lengths and angles are within normal ranges for these types of heterocyclic systems. There are no noticeably short intermolecular contacts in the structure.

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Structure of 7-(*p*-Tolyl)-2,3-dihydro-5*H*-thiazolo[4,3-*b*]thiazole-3,5-dione

By Ivan Leban and Ljubo Golič

Department of Chemistry and Chemical Technology, E. Kardelj University, 61001 *Ljubljana, Yugoslavia*

Alenka Majcen Le-Maréchal

Faculty of Technical Sciences, University of Maribor, 62000 *Maribor, Yugoslavia*

and Albert Robert

Groupe de Recherches de Chimie Structurale, Université de Rennes, 35042 *Rennes, France*

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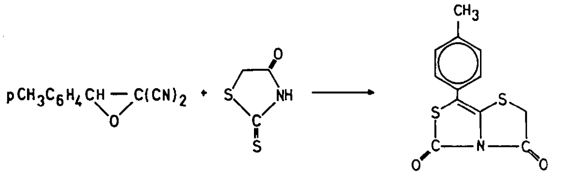
Abstract. $C_{12}H_9NO_2S_2$, $M_r = 263{\cdot}3$, monoclinic, $P2_1/c$, $a = 12{\cdot}607\ (2)$, $b = 7{\cdot}067\ (1)$, $c = 13{\cdot}776\ (3)$ Å, $\beta = 112{\cdot}40\ (1)°$, $V = 1134{\cdot}7\ (7)$ Å^3, $Z = 4$, $D_m = 1{\cdot}53\ (5)$, $D_x = 1{\cdot}540$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 4{\cdot}46$ cm^{-1}, $F(000) = 544$, $T = 293\ (1)$ K, final $R = 0{\cdot}040$ for 1552 observed reflexions. The two fused thiazole rings are coplanar [dihedral angle between ring planes: 0·7 (5)°]; the tolyl ring is twisted by 18·8 (6)° about the C(7)—C(8) bond.

Introduction. *gem*-Dicyanoepoxides normally react with *N*-monosubstituted thioamides to provide mesoionic thiazoles (Baudy & Robert, 1976, 1980). The reaction of the *gem*-dicyanoepoxide (I) and rhodanine (II) leads to the transposition product (III).



(I) (II) (III)

The epoxide and rhodanine were mixed in stoichiometric quantities and heated for 30 min at 423 K without solvent; compound (III) was isolated, purified and crystallized from benzene and acetone respectively.

Experimental. Density measured by flotation in chlorobenzene/CCl_4; colourless prisms, 0·38 × 0·28 × 0·41 mm; Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation (graphite monochromator), lattice parameters by least-squares fit to angular settings of 25 well centred reflexions ($6 < \theta < 11°$); $\omega/2\theta$ scans, variable scan rate (min. 1·10, max. 5·50° min^{-1}), max. scan time 60 s, scan width (2θ) $(0{\cdot}7 + 0{\cdot}3\tan\theta)°$, aperture $(2{\cdot}5 + 0{\cdot}9\tan\theta)$ mm, background measured for 0·25 of scan time at each scan limit, $[(\sin\theta)/\lambda]_{\max}$ in intensity measurements 0·66 Å^{-1}. Three reflexions monitored at intervals of 300 reflexions, no significant intensity decline (0·6%); data corrected for intensity variation and Lp effects, but absorption ignored; 6244 ($\pm h$, $-k$, $\pm l$) measured reflexions, 2580 unique [$R_{\text{int}}(I) = 0{\cdot}028$ for 6219 contributing reflexions], 1552 observed (h −16/15, k 0/8, l 0/16) with $I > 2\sigma(I)$, $\sigma(I)$ based on counting statistics. Structure solution by

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direct methods with *SHELXS*84 (Sheldrick, 1985), full-matrix least-squares refinement minimizing $\sum w(\Delta F)^2$ with *SHELX*76 (Sheldrick, 1976); final *R* and *wR* values: 0·040 and 0·063 with $w = 0{\cdot}147/[\sigma^2(F_o) + 0{\cdot}015F_o^2]$; H atoms found from a difference synthesis and included in the refinement at calculated positions [d(C—H) = 1·08 Å], a common isotropic temperature factor U for H atoms was fixed at 0·123 Å^2. $\Delta\rho$ within 0·34 and −0·28 e Å^{-3} in final difference map. At convergence max. and mean values of Δ/σ: 0·317 and 0·082. Scattering factors for S, O, N and C and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974), for H from Stewart, Davidson & Simpson (1965).

All calculations performed on the DEC-1091 computer at University Computer Centre, Ljubljana.

Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^4) (*Hamilton*, 1959)

	x	*y*	*z*	U_{eq}
S(1)	1550 (1)	0693 (1)	4475 (1)	474 (4)
C(2)	3080 (3)	0704 (6)	4816 (3)	558 (18)
C(3)	3326 (3)	1006 (5)	3837 (3)	465 (16)
O(3)	4251 (2)	1156 (5)	3805 (2)	676 (15)
N(4)	2287 (2)	1118 (4)	2961 (2)	393 (12)
C(5)	2129 (3)	1337 (5)	1905 (3)	474 (16)
O(5)	2883 (2)	1438 (5)	1572 (2)	710 (15)
S(6)	0648 (1)	1452 (1)	1174 (1)	473 (4)
C(7)	0304 (2)	1147 (4)	2292 (2)	345 (13)
C(7*A*)	1270 (2)	1006 (4)	3146 (2)	344 (13)
C(8)	−0899 (2)	1125 (4)	2178 (2)	373 (14)
C(9)	−1772 (3)	0790 (5)	1204 (3)	473 (16)
C(10)	−2915 (3)	0786 (5)	1102 (3)	573 (18)
C(11)	−3219 (3)	1118 (5)	1949 (3)	533 (17)
C(12)	−2350 (3)	1464 (5)	2914 (3)	531 (17)
C(13)	−1210 (3)	1468 (5)	3036 (3)	444 (15)
C(14)	−4468 (3)	1121 (7)	1827 (4)	809 (24)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

S(1)—C(2)	1·803 (4)	C(7)—C(7*A*)	1·336 (3)
S(1)—C(7*A*)	1·742 (3)	C(7)—C(8)	1·464 (4)
C(2)—C(3)	1·510 (6)	C(8)—C(9)	1·394 (4)
C(3)—O(3)	1·188 (5)	C(8)—C(13)	1·401 (6)
C(3)—N(4)	1·405 (4)	C(9)—C(10)	1·394 (6)
N(4)—C(5)	1·400 (5)	C(10)—C(11)	1·380 (7)
N(4)—C(7*A*)	1·402 (4)	C(11)—C(12)	1·385 (5)
C(5)—O(5)	1·205 (5)	C(11)—C(14)	1·519 (6)
C(5)—S(6)	1·753 (4)	C(12)—C(13)	1·382 (6)
S(6)—C(7)	1·765 (3)		

C(10)—C(11)—C(14)	121·1 (3)	O(5)—C(5)—S(6)	126·9 (3)
C(12)—C(11)—C(14)	120·9 (4)	C(5)—S(6)—C(7)	93·2 (2)
C(11)—C(12)—C(13)	121·5 (4)	S(6)—C(7)—C(7*A*)	109·4 (2)
C(8)—C(13)—C(12)	120·6 (3)	S(6)—C(7)—C(8)	119·8 (2)
C(2)—S(1)—C(7*A*)	92·4 (2)	C(7*A*)—C(7)—C(8)	130·8 (3)
S(1)—C(2)—C(3)	109·4 (2)	S(1)—C(7*A*)—N(4)	111·4 (2)
C(2)—C(3)—O(3)	125·8 (3)	S(1)—C(7*A*)—C(7)	133·4 (3)
C(2)—C(3)—N(4)	109·5 (3)	N(4)—C(7*A*)—C(7)	115·2 (3)
O(3)—C(3)—N(4)	124·8 (4)	C(7)—C(8)—C(9)	120·7 (3)
C(3)—N(4)—C(5)	128·0 (3)	C(7)—C(8)—C(13)	121·3 (2)
C(3)—N(4)—C(7*A*)	117·3 (3)	C(9)—C(8)—C(13)	118·0 (3)
C(5)—N(4)—C(7*A*)	114·7 (2)	C(8)—C(9)—C(10)	120·3 (4)
N(4)—C(5)—O(5)	125·6 (3)	C(9)—C(10)—C(11)	121·6 (3)
N(4)—C(5)—S(6)	107·5 (3)	C(10)—C(11)—C(12)	118·0 (4)

*XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) was used for data reduction and interpretation.

Discussion. The final atomic parameters are in Table 1.* Bond lengths and angles are presented in Table 2. Views of the molecule with the atomic numbering, and of the molecular packing are presented in Figs. 1 and 2. The two fused thiazole rings are coplanar, the dihedral angle being 0·7 (5)°. The tolyl ring is twisted with respect to the thiazole rings about C(7)—C(8) by 18·8 (6)°. The largest deviations from planarity in the individual rings are −0·013 (4) Å for C(3) in ring S(1), S(2), C(3), N(4), C(7*A*); −0·009 (4) Å for C(5) in ring N(4), C(5), S(6), C(7), C(7*A*); and −0·003 (3) Å for C(9) in the tolyl ring. From an inspection of Table 2 it can be seen that the bond C(2)—C(3) [1·510 (6) Å] is single, whereas bond C(7)—C(7*A*) [1·336 (3) Å] exhibits double-bond character. The typical 3- and 5-carbonyl distances C(3)—O(3) [1·188 (5) Å] and C(5)—O(5) [1·205 (5) Å] suggest only limited involvement of these groups in the pattern of delocalization within the molecule. The lengths of the bonds S(1)—C(2) [1·803 (4)], S(1)—C(7*A*) [1·741 (3)], S(6)—C(5) [1·753 (4)] and S(6)—C(7) [1·765 (3) Å] agree with the

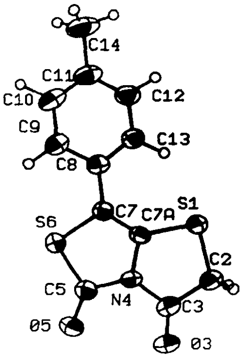

Fig. 1. *ORTEP* (Johnson, 1965) view of the molecule. The atoms are represented by thermal ellipsoids at 50% probability level.

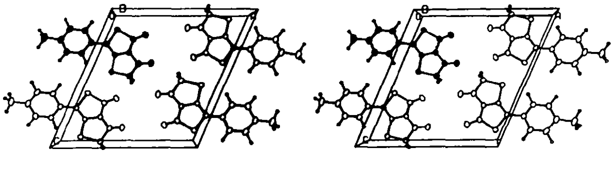

Fig. 2. View of the molecular packing.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, least-squares-planes calculations and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43984 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

observation of slightly elongated C—S single bonds in planar or near-planar systems (Andreetti, Bocelli & Sgarabotto, 1980; Ammon, Watts & Stewart, 1970). The above distances compare well to those found, for example, in 5,6-dihydrothiazolo[2,3-*c*][1,2,4]thiadiazol-3-one, 6*β*-(1-ethyl-1-hydroxypropyl)-5α-phenyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazole and 5,5-diphenyl-2,3-dihydroimidazo[2,1-*b*]thiazol-6(5*H*)-one (Cameron, Cameron & Duncanson, 1981). There are no intermolecular distances shorter than the van der Waals separations. The shortest intermolecular contact is C(14)···O(5) $(-1+x, y, z)$ of 3·230 (5) Å.

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Structure of Isoquinolinium Nitrate and a Redetermination of the Corresponding Chloride

BY JOHN J. DALY

ZFE, F. Hoffmann–La Roche AG, 4002 *Basel, Switzerland*

A. ALAN PINKERTON

Department of Chemistry, The University of Toledo, Toledo, OH 43606, *USA*

AND DIETER SCHWARZENBACH

Institut de Cristallographie, BSP, 1015 *Lausanne–Dorigny, Switzerland*

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Abstract. $C_9H_8N^+.NO_3^-$, $M_r = 192·17$, triclinic, $P\bar{1}$, $a = 5·114$ (1), $b = 9·304$ (2), $c = 9·973$ (3) Å, $\alpha = 100·85$ (2), $\beta = 104·39$ (2), $\gamma = 92·14$ (2)°, $V = 449·6$ (2) Å^3, $Z = 2$, $D_x = 1·42$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1·2$ cm^{-1}, $F(000) = 200$, $T = 295$ K, $R = 0·042$ for 809 unique observed reflections. $C_9H_8N^+.Cl^-$, $M_r = 165·6$, monoclinic, $P2_1/a$, $a = 9·192$ (5), $b = 17·486$ (6), $c = 5·161$ (3) Å, $\beta = 100·45$ (4)°, $V = 815·8$ (7) Å^3, $Z = 4$, $D_x = 1·35$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 4·0$ cm^{-1}, $F(000) = 344$, $T = 295$ K, $R = 0·044$ for 748 unique observed reflections. The isoquinolinium ion is planar and hydrogen-bonded to the counter ion. The variation in skeletal bond lengths is similar to that observed for isoelectronic naphthalene. The smaller size of N is manifested in shorter 1,2 and 2,3 bond lengths and an opening of the 4,10,5 angle (IUPAC atom numbering). Shorter C—C bonds in the heterocycle are attributed to π-electron transfer from the carbocycle due to the formal positive charge on N.

Introduction. As part of an NMR study we had need of accurate structural parameters for the isoquinolinium ion, $C_9H_8N^+$. A survey of the literature revealed the chloride (both anhydrous and the monohydrate) as the only simple salt to have been structurally characterized (Genet, 1965). The structures were determined from photographic data and refined to $R = 0·15$ and $R = 0·18$ respectively. In both cases no e.s.d.'s were quoted for the bond lengths and angles of the molecular skeleton, thus no discussion of the variation of these parameters is possible. The positions of the H atoms

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