

Fig. 2. Stereoscopic view of the two independent molecules.

cant differences in the orientation of the phenyl groups and the ethyl groups with respect to the five-membered heterocycle and the distances of the nearest intermolecular neighbours to the five-membered heterocycle, C(3)···C 3.547 (6), C(4)···C 3.607 (6), N(1)···C 3.629 (6), C'(3)···C 3.447 (6), C'(4)···C

3.499 (6), N'(1)···C 3.480 (6) Å, indicate that the two independent molecules are deformed differently by packing forces.

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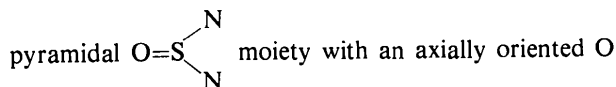
4-Phenoxy-10H-benzimidazo[2,1-c][1 λ^4 ,2,4,6]thiatriazine 2-Oxide

BY ALAJOS KÁLMÁN AND GYULA ARGAY

Central Research Institute of Chemistry, Hungarian Academy of Sciences, Budapest 114, POB 17, H-1525, Hungary

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Abstract. C₁₄H₁₀N₄O₂S, monoclinic, *P*2₁/*n*, *a* = 9.329 (2), *b* = 4.603 (5), *c* = 30.124 (7) Å, β = 92.57 (2)°, *V* = 1292 (2) Å³, *D*_c = 1.533 Mg m⁻³, *Z* = 4, *F*(000) = 616. The structure was solved by direct methods and refined by full-matrix least squares to a final *R* = 0.068 for 1089 intensities. A rare trigonal-



atom is embedded in the non-planar thiatriazine ring. However, the bonding and conformation of the thiatriazine *S*-oxide ring resemble those observed for 1,4-dihydro-3,5-dimethoxy-1-tosylimino-1 λ^4 ,2,4,6-thiatriazine [Kálmán, Argay, Fischer & Teller (1981). *Acta Cryst.* **B37**, 164–168]. The S=O distance of

1.452 (7) Å is considerably shorter than the 1.51 Å observed in various sulphoxides.

Introduction. As established by this X-ray analysis the reaction of 2-amino-1-phenoxyformimidoylbenzimidazole with *N*-sulphinyl-*p*-toluenesulphonamide and a consecutive but unexpected hydrolysis of the intermediate* resulted in the title compound. Intensities were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator using Mo *K* α radiation (λ = 0.71073 Å). Cell constants were determined by least-squares refinement from the setting angles of 25 reflexions; of the total of 1231 measure-

* A protected synthesis of the parent 1-tosyliminothiatriazine carried out by Professor E. Fischer and Dr M. Teller is in progress.

ments, 142 with $I < 2.5\sigma(I)$ were taken as unobserved. No absorption correction was applied. The phases for 217 reflexions having $E \geq 1.70$ were obtained by *MULTAN* (Germain, Main & Woolfson, 1971). The E map revealed the positions of all the non-hydrogen atoms ($R = 0.37$). No further heavy atoms could be located. Accordingly, taking into account the loss of the exocyclic *N*-tosyl group, the structure expected by Fischer & Teller (1980) had to be reconsidered. Full-matrix least-squares refinement of positional and vibrational parameters reduced R to 0.079. At this stage the coordinates of the H atoms were generated from assumed geometries. Further anisotropic refinement of non-hydrogen atoms with fixed H coordinates improved R to 0.068 ($R_w = 0.067$).^{*} In this procedure

^{*} Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36549 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates ($\times 10^4$) and mean temperature factors (\AA^2) for non-hydrogen atoms

E.s.d.'s are in parentheses. $B_{eq} = 4[B_{11}B_{22}B_{33}/(a^*{}^2b^*{}^2c^*{}^2)]^{1/3}$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
N(1)	937 (5)	5185 (16)	1443 (2)	3.5 (3)
S(2)	1961 (2)	4395 (6)	1004 (1)	3.8 (1)
N(3)	790 (5)	4788 (15)	573 (2)	3.3 (3)
C(4)	-191 (7)	6728 (19)	606 (2)	2.7 (4)
N(5)	-634 (5)	8064 (14)	1000 (2)	2.9 (3)
C(6)	-73 (6)	6952 (19)	1398 (2)	2.9 (3)
O(7)	-863 (4)	7911 (12)	1733 (1)	3.0 (3)
C(8)	-334 (6)	7229 (18)	2175 (2)	2.6 (4)
C(9)	-1094 (7)	5088 (19)	2396 (2)	3.5 (4)
C(10)	-632 (8)	4652 (21)	2842 (2)	4.4 (4)
C(11)	510 (8)	6090 (21)	3033 (2)	3.9 (5)
C(12)	1219 (7)	8153 (22)	2797 (2)	4.6 (4)
C(13)	772 (7)	8728 (21)	2353 (2)	4.0 (4)
N(14)	-1098 (5)	7810 (15)	281 (2)	2.9 (3)
C(15)	-2091 (6)	9711 (18)	442 (2)	2.8 (3)
C(16)	-1796 (6)	9937 (17)	905 (2)	2.6 (3)
C(17)	-2607 (7)	11713 (18)	1160 (2)	3.1 (4)
C(18)	-3709 (7)	13238 (19)	951 (2)	3.8 (4)
C(19)	-3999 (7)	12982 (19)	495 (3)	4.3 (4)
C(20)	-3172 (7)	11247 (20)	241 (2)	3.9 (4)
O(21)	2968 (5)	6793 (16)	987 (2)	6.0 (3)

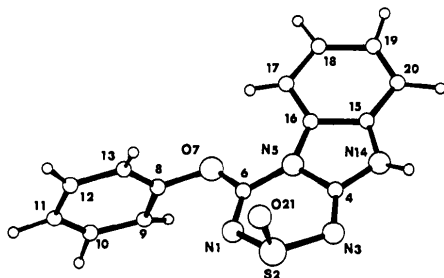


Fig. 1. A perspective view of the molecule with the atomic numbering. The numbers are for carbon atoms, unless indicated otherwise.

Table 2. Bond lengths (\AA) and angles ($^\circ$) for the non-hydrogen atoms

E.s.d.'s are in parentheses.

N(1)—S(2)	1.705 (6)	C(8)—C(13)	1.334 (10)
N(1)—C(6)	1.247 (9)	C(9)—C(10)	1.407 (8)
S(2)—N(3)	1.669 (6)	C(10)—C(11)	1.360 (11)
S(2)—O(21)	1.452 (7)	C(11)—C(12)	1.374 (12)
N(3)—C(4)	1.286 (10)	C(12)—C(13)	1.408 (8)
C(4)—N(5)	1.415 (9)	N(14)—C(15)	1.378 (9)
C(4)—N(14)	1.359 (9)	C(15)—C(16)	1.413 (8)
N(5)—C(6)	1.385 (8)	C(16)—C(20)	1.352 (10)
N(5)—C(16)	1.404 (8)	C(15)—C(17)	1.372 (10)
C(6)—O(7)	1.350 (7)	C(17)—C(18)	1.374 (10)
O(7)—C(8)	1.434 (7)	C(18)—C(19)	1.393 (10)
C(8)—C(9)	1.400 (10)	C(19)—C(20)	1.368 (11)
S(2)—N(1)—C(6)	120.1 (9)	O(9)—C(8)—C(13)	124.9 (11)
N(1)—S(2)—N(3)	102.1 (5)	C(8)—C(9)—C(10)	114.5 (11)
N(1)—S(2)—O(21)	104.5 (6)	C(9)—C(10)—C(11)	122.4 (12)
N(3)—S(2)—O(21)	107.0 (6)	C(10)—C(11)—C(12)	120.3 (13)
S(2)—N(3)—C(4)	117.3 (9)	C(11)—C(12)—C(13)	119.5 (12)
N(3)—C(4)—N(5)	127.0 (11)	C(8)—C(13)—C(12)	118.4 (12)
N(3)—C(4)—N(14)	128.4 (11)	C(4)—N(14)—C(15)	112.7 (10)
N(5)—C(4)—N(14)	104.4 (10)	N(14)—C(15)—C(16)	106.8 (10)
C(4)—N(5)—C(6)	116.9 (10)	N(14)—C(15)—C(20)	132.4 (11)
C(4)—N(5)—C(16)	110.3 (9)	C(16)—C(15)—C(20)	120.8 (11)
C(6)—N(5)—C(16)	131.4 (10)	N(5)—C(16)—C(15)	105.7 (10)
N(1)—C(6)—N(5)	126.1 (11)	N(5)—C(16)—C(17)	133.8 (11)
N(1)—C(6)—O(7)	124.5 (11)	C(15)—C(16)—C(17)	120.6 (11)
N(5)—C(6)—O(7)	109.2 (10)	C(16)—C(17)—C(18)	117.9 (11)
C(6)—O(7)—C(8)	116.5 (9)	C(17)—C(18)—C(19)	121.1 (12)
O(7)—C(8)—C(9)	115.8 (10)	C(18)—C(19)—C(20)	120.8 (12)
O(7)—C(8)—C(13)	119.2 (10)	C(15)—C(20)—C(19)	118.9 (12)


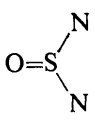
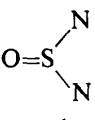
H atoms were assigned a mean temperature factor of 4.0\AA^2 . Scattering factors were taken from *International Tables for X-ray Crystallography* (1962). All calculations were performed on a PDP 11/34 mini-computer with the Enraf-Nonius SDP-34 system. The atomic coordinates for the non-hydrogen atoms are given in Table 1, bond lengths and angles in Table 2. The molecular geometry is shown in Fig. 1.

Discussion. The trigonal-pyramidal $\text{O}=\text{S}(\text{N})_2$ moiety is

embedded in the non-planar six-membered thiaziazine ring (Kálmán, Argay, Fischer & Teller, 1981, and references therein). This rare group could only be found in three structures (BOTSTZ, CATZQO and PHTDZO) by a search of the Cambridge Crystallographic Data File containing 27 998 entries to September 1980. In each of these compounds the $\text{S}=\text{O}$ length is shorter (Table 3) than the usual $\text{S}=\text{O}$ distance (1.51 \AA) observed in several sulphoxides, while the $\text{S}-\text{N}$ distances are in the neighbourhood of the $\text{S}-\text{N}$ single bond (1.75 \AA). It is worth noting that the

analogous $\text{O}=\text{S}(\text{O})_2$ group confined to the five-

membered ring of *o*-phenylene sulphite (a gas electron diffraction study of Schultz, Serke & Kapovits, 1979)

Table 3. Relevant parameters of the  groups and  and  groups assumes an axial or a pseudoaxial orientation. The thiaziazine ring possesses an envelope conformation [$Q = 0.36 \text{ \AA}$, $\varphi = 245.4^\circ$, $\theta = 113.8^\circ$ (Cremer & Pople, 1975)], to which a coplanar benzimidazole ring system is fused. The 4-phenoxy group is bound equatorially. The C(8)—O(7)—C(6)—N(1) torsion angle is 11.3° (synperiplanar). The dihedral angle between the best planes of the phenyl rings is 85.2° . Dimeric associates related by a centre of symmetry are formed *via* hydrogen-bond pairs: N(14)—H...N(3): 2.864 (1), 2.02 (3) Å, 153 (2)°.

	S=O	S—N	OSN	NSN	OSNC
Title compound	1.452 Å	1.705 Å	104.5°	102.1°	80.5°
BOTSTZ*	1.430	1.717	107.0	90.6	-76.7
CATZQO†	1.439	1.741	111.9	82.9	91.8
PHTDZO‡	1.478	1.716	108.5	97.2	-96.5
		1.692	110.5		128.2
		1.692	110.5		-128.2
		S—O	OSO	OSO	OSOC
<i>o</i> -Phenylene sulphite§	1.427	1.670	107.7	93.0	-90.3
		1.670	107.7		90.3

* L'Abbé, van Asch, Declercq, Germain & Van Meerssche (1978).

† Reimlinger, Vandewalle, King, Lingier & Merényi (1970).

‡ Arora (1974).

§ Schultz, Serke & Kapovits (1979).

exhibits a similarly short S=O distance [1.433 (5) Å] accompanied by two S—O single bonds [1.669 (4) Å]. Accordingly, as claimed by Bennett, Cotton, Weaver, Williams & Watson (1967), it must be assumed that S is using at least one *d* orbital from back-accepting π electrons from O. However, the bond order 2.5 for the S=O distance of 1.476 Å calculated by Bennett and co-workers has to be taken with care. It seems to be overestimated. At any rate these S=O distances approach those of the sulphone groups [*cf.*, for example, 1.431–1.439 (1) Å of Kálmán *et al.* (1981)]. As shown by the corresponding torsional angles in Table 3, except in PHTDZO, the *exo* O atom of both

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Structure of Ethyl 1,2,2-Tricyano-3-(4-nitrophenyl)cyclopropane-1-carboxylate

BY N. RAMASUBBU AND K. VENKATESAN

Department of Organic Chemistry, Indian Institute of Science, Bangalore 560012, India

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Abstract. C₁₅H₁₀N₄O₄, monoclinic, $P2_1/c$, $a = 10.694$ (8), $b = 11.743$ (8), $c = 12.658$ (8) Å, $\beta = 113.10$ (7)°, $V = 1462.1$ Å³, $Z = 4$, $D_m = 1.38$, $D_c = 1.408$ g cm⁻³, $\mu(\text{Mo } K\alpha, \lambda = 0.7107 \text{ \AA}) = 0.99$ cm⁻¹, $F(000) = 640$. The structure was solved by direct

methods and refined to an *R* value of 0.054 using 1398 intensity measurements. The relative magnitudes of interaction of the substituents and the extent to which a ring can accommodate interactions with substituents are discussed.