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Structure of an Annulated Derivative of Pyridopyrazine

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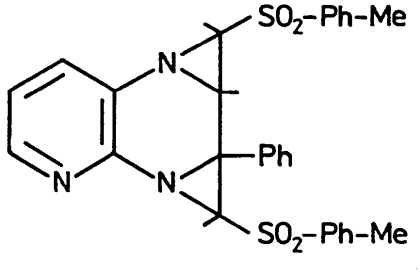
Abstract. 1b-Phenyl-1,2-bis(*p*-tolylsulfonyl)-1,1a,1b,-2-tetrahydrodiaziridino[1,2-*a*:2,1-*c*]pyrido[2,3-*b*]pyrazine, $C_{29}H_{25}N_3O_4S_2$, $M_r = 543{\cdot}67$, orthorhombic, *Pbcn*, $a = 19{\cdot}801\ (2)$, $b = 9{\cdot}686\ (2)$, $c = 28{\cdot}331\ (3)$ Å, $V = 5433{\cdot}7\ (6)$ Å^3, $Z = 8$, $D_x = 1{\cdot}329$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 19{\cdot}9$ cm^{-1}, $F(000) = 2272$, $T = 293$ K, $R = 0{\cdot}0707$ for 3626 reflections. All the bond lengths are within a range of standard values. The aziridine rings are bent in opposite directions with respect to the almost planar pyrazine plane [max. deviation 0·018 (5) Å]; the phenyl ring makes a dihedral angle of 71·6 (1)° with it. The pyridine ring, the phenyl ring and the C(21)–C(26) tolyl rings are each planar within their e.s.d.'s. The C(11)–C(16) tolyl ring is slightly deformed from planarity [max. deviation 0·014 (6) Å]. There are no intermolecular contacts less than the sum of the van der Waals radii.

Experimental. Colourless, very thin, elongated plates were grown by slow evaporation of a xylene solution. The space group and approximate cell parameters were defined from oscillation and Weissenberg photographs; systematic absences 0*kl*: *k* odd, *h*0*l*: *l* odd, *hk*0: $h + k$ odd, *h*00: *h* odd, 0*k*0: *k* odd, 00*l*: *l* odd.



Crystal 0·42 × 0·37 × 0·02 mm, cut from a large fragment, used for diffractometer measurement. Enraf–Nonius CAD-4 diffractometer; Cu $K\alpha$ radiation; graphite monochromator; unit-cell dimensions from refinement of 25 reflections in the range $8{\cdot}9 < \theta < 18{\cdot}3°$; ω–2θ scan mode, $\theta_{max} = 75°$; index ranges: *h* 0/23, *k* 0/11, *l* 0/33; three standard reflections monitored every hour, no significant changes in intensities; no absorption correction. 6233 reflections measured, 5490 unique reflections, 3629 of which considered observed with $F > 3\sigma(F)$. Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1986). Blocked full-matrix least-squares

Table 1. *Atomic fractional coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters*

$B_{eq} = 8\pi^2 D_u^{1/3}/(\sin\alpha^*\sin\beta^*\sin\gamma^*)^2$, where D_u is the determinant of the U_{ij} matrix.

	x	*y*	*z*	B_{eq}(Å^2)
S(1)	6242·3 (6)	−1973·9 (11)	8981·7 (4)	4·7
O(11)	6953 (2)	−2152 (4)	9059 (1)	6·4
O(12)	5783 (2)	−3068 (3)	9104 (1)	5·7
S(2)	6870·2 (5)	1732·4 (9)	6933·4 (4)	3·7
O(21)	6243 (1)	2412 (3)	6843 (1)	5·0
O(22)	7416 (1)	2493 (3)	7142 (1)	4·8
N(1)	6508 (2)	−2279 (3)	8027 (1)	3·5
N(2)	6023 (1)	−197 (3)	7350 (1)	3·0
N(3)	5606 (2)	−1967 (3)	6887 (1)	3·9
C(1)	6147 (2)	−2625 (3)	7609 (1)	3·4
C(2)	5924 (2)	−1662 (4)	7285 (1)	3·2
C(3)	5495 (2)	−3335 (4)	6801 (2)	4·4
C(4)	5705 (2)	−4356 (4)	7103 (2)	4·6
C(5)	6034 (2)	−4008 (4)	7509 (2)	4·2
C(6)	6104 (2)	−1524 (4)	8384 (1)	3·7
C(7)	6630 (2)	−799 (3)	8109 (1)	3·3
C(8)	6401 (2)	272 (3)	7765 (1)	3·0
C(9)	6729 (2)	254 (3)	7295 (1)	3·1
C(11)	5967 (3)	−445 (5)	9255 (2)	5·0
C(12)	5285 (3)	−233 (5)	9322 (2)	6·6
C(13)	5056 (4)	1000 (7)	9513 (2)	8·1
C(14)	5509 (5)	1995 (7)	9633 (2)	7·2
C(15)	6173 (4)	1801 (6)	9562 (2)	7·5
C(16)	6420 (3)	569 (6)	9366 (2)	6·6
C(10)	5253 (6)	3381 (7)	9832 (3)	10·5
C(21)	7169 (2)	952 (4)	6416 (1)	4·0
C(22)	7837 (3)	956 (7)	6319 (2)	7·0
C(23)	8080 (3)	246 (9)	5929 (2)	8·6
C(24)	7663 (3)	−456 (7)	5639 (2)	7·8
C(25)	6995 (4)	−451 (8)	5742 (2)	8·3
C(26)	6736 (3)	251 (7)	6126 (2)	6·7
C(20)	7944 (5)	−1275 (11)	5221 (3)	11·6
C(31)	6181 (2)	1565 (3)	8012 (1)	3·1
C(32)	5504 (2)	1802 (4)	8105 (1)	3·6
C(33)	5320 (2)	2909 (4)	8388 (2)	4·1
C(34)	5806 (2)	3773 (4)	8573 (2)	4·2
C(35)	6477 (2)	3535 (4)	8478 (2)	4·4
C(36)	6667 (2)	2434 (4)	8198 (2)	3·9

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Table 2. *Selected bond distances* (Å), *angles* (°) *and torsion angles* (°)

O(11)—S(1)	1·435 (4)	C(9)—N(2)	1·473 (4)
O(12)—S(1)	1·438 (4)	C(2)—N(3)	1·325 (4)
C(6)—S(1)	1·771 (3)	C(3)—N(3)	1·365 (5)
C(11)—S(1)	1·757 (5)	C(2)—C(1)	1·381 (5)
O(21)—S(2)	1·429 (2)	C(5)—C(1)	1·387 (5)
O(22)—S(2)	1·436 (2)	C(4)—C(3)	1·372 (7)
C(9)—S(2)	1·783 (3)	C(5)—C(4)	1·364 (7)
C(21)—S(2)	1·751 (3)	C(7)—C(6)	1·478 (5)
C(1)—N(1)	1·423 (4)	C(8)—C(7)	1·494 (4)
C(6)—N(1)	1·482 (4)	C(9)—C(8)	1·482 (4)
C(7)—N(1)	1·472 (4)	C(31)—C(8)	1·499 (4)
C(2)—N(2)	1·444 (5)	C(10)—C(14)	1·542 (10)
C(8)—N(2)	1·466 (4)	C(20)—C(24)	1·530 (11)

O(11)—S(1)—O(12)	119·7 (2)	C(6)—N(1)—C(7)	60·0 (2)
O(11)—S(1)—C(6)	109·1 (2)	N(1)—C(7)—C(6)	60·3 (2)
O(11)—S(1)—C(11)	109·8 (3)	N(1)—C(7)—C(8)	121·6 (3)
O(12)—S(1)—C(6)	108·3 (2)	N(2)—C(2)—N(3)	113·1 (3)
O(12)—S(1)—C(11)	108·6 (2)	N(2)—C(2)—C(1)	122·4 (3)
S(1)—C(6)—N(1)	116·6 (3)	C(2)—N(2)—C(8)	118·4 (3)
S(1)—C(6)—C(7)	120·8 (3)	C(2)—N(2)—C(9)	114·0 (3)
C(6)—S(1)—C(11)	99·5 (2)	N(2)—C(8)—C(7)	117·6 (3)
S(1)—C(11)—C(12)	119·3 (4)	N(2)—C(8)—C(9)	60·0 (2)
S(1)—C(1)—C(16)	120·2 (5)	N(2)—C(8)—C(31)	119·0 (3)
O(21)—S(2)—O(22)	119·5 (2)	C(8)—N(2)—C(9)	60·5 (2)
O(21)—S(2)—C(9)	109·7 (2)	N(2)—C(9)—C(8)	59·5 (2)
O(21)—S(2)—C(21)	110·0 (2)	N(3)—C(2)—C(1)	124·5 (3)
O(22)—S(2)—C(9)	107·0 (2)	C(2)—N(3)—C(3)	116·4 (3)
O(22)—S(2)—C(21)	108·2 (2)	N(3)—C(3)—C(4)	122·7 (4)
S(2)—C(9)—N(2)	116·6 (2)	C(2)—C(1)—C(5)	117·7 (3)
S(2)—C(9)—C(8)	125·2 (2)	C(1)—C(5)—C(4)	119·2 (4)
C(9)—S(2)—C(21)	100·8 (2)	C(3)—C(4)—C(5)	119·5 (4)
S(2)—C(21)—C(22)	120·3 (3)	C(6)—C(7)—C(8)	117·4 (3)
S(2)—C(21)—C(26)	120·3 (4)	C(7)—C(8)—C(9)	116·4 (3)
N(1)—C(1)—C(2)	123·7 (3)	C(7)—C(8)—C(31)	111·3 (2)
N(1)—C(1)—C(5)	118·6 (3)	C(9)—C(8)—C(31)	123·8 (3)
C(1)—N(1)—C(6)	114·4 (2)	C(8)—C(31)—C(32)	120·6 (3)
C(1)—N(1)—C(7)	116·3 (3)	C(8)—C(31)—C(36)	119·0 (4)
N(1)—C(6)—C(7)	59·6 (2)		

N(1)—C(6)—S(1)—O(11)	41·7 (3)	C(8)—C(9)—S(2)—O(22)	74·0 (4)
C(7)—C(6)—S(1)—O(11)	−27·3 (4)	N(2)—C(9)—S(2)—C(21)	−103·0 (2)
N(1)—C(6)—S(1)—O(12)	−90·1 (3)	C(8)—C(9)—S(2)—C(21)	−173·0 (3)
C(7)—C(6)—S(1)—O(12)	−159·0 (3)	C(32)—C(31)—C(8)—N(2)	44·6 (4)
N(1)—C(6)—S(1)—C(11)	156·6 (3)	C(36)—C(31)—C(8)—N(2)	−143·5 (4)
C(7)—C(6)—S(1)—C(11)	87·7 (4)	C(32)—C(31)—C(8)—C(7)	−97·2 (4)
N(2)—C(9)—S(2)—O(21)	13·0 (3)	C(36)—C(31)—C(8)—C(7)	74·8 (4)
C(8)—C(9)—S(2)—O(21)	−57·0 (4)	C(32)—C(31)—C(8)—C(9)	116·0 (4)
N(2)—C(9)—S(2)—O(22)	144·0 (2)	C(36)—C(31)—C(80)—C(9)	−71·0 (5)

refinement on F, using *SHELX*76 (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms, H atoms were added in calculated positions and refined with isotropic thermal parameters. Scattering factors as supplied with the program. Final $R = 0{\cdot}0707$, $wR = 0{\cdot}0646$, $w = 4{\cdot}3522/[\sigma^2(F) + 0{\cdot}00053F^2]$, three reflections omitted in the last cycles; 443 parameters refined, $(\Delta/\sigma)_{max}$ in final cycle 0·026 for non-H atoms, 0·033 for H atoms, extreme values in final difference map +0·30, −0·38 e Å^{-3}. The final atomic parameters are given in Table 1,* selected bond lengths, angles and torsion angles are given in Table 2. A view of the molecule with the atom-numbering scheme is shown in Fig. 1. The compound was prepared by the method described by Goliński, Mąkosza & Rykowski (1983).

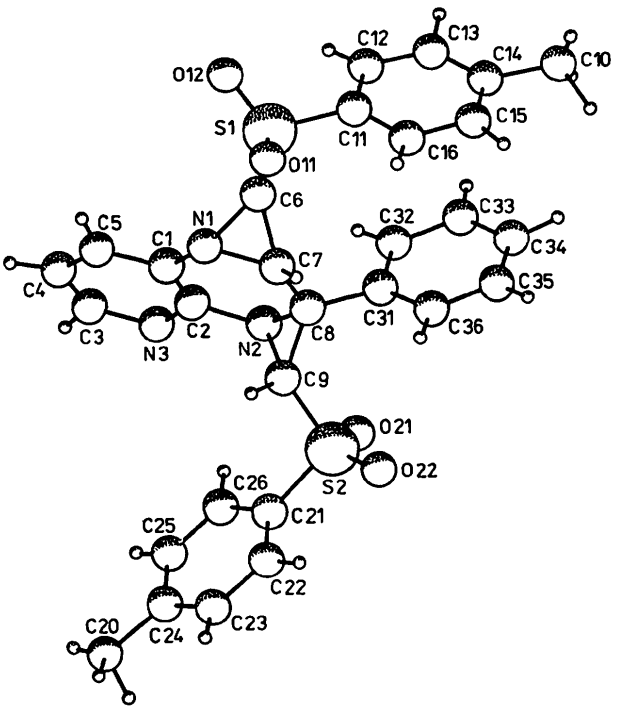

Fig. 1. View of the molecule with atom numbering.

Related literature. Related structures of derivatives of quinoxaline (Pniewska & Anulewicz, 1986), 1,8-naphthyridine (Pniewska & Anulewicz, 1987) and 1,5-napthyridine (Pniewska, Rykowski & Anulewicz, 1990) have been published.

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* Lists of structure factors, anisotropic thermal parameters, a full list of bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53609 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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