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3-Benzoyl-1-[(4-methylphenyl)sulfonyl]-1a,2,3,7b-tetrahydro-1*H*-azireno[2,3-*c*]-quinoline-2-carbonitrile

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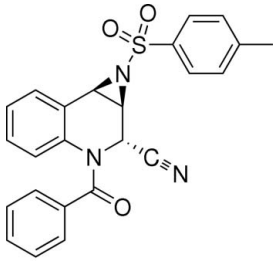
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.066; wR factor = 0.157; data-to-parameter ratio = 22.3.

The title compound, $C_{24}H_{19}N_3O_3S$, is the result of a diastereoselective aziridination reaction performed on the corresponding Reissert adduct (1-benzoyl-1,2-dihydroquinoline-2-carbonitrile). The aziridine ring is *trans* disposed to the nitrile group.

Related literature

For related literature, see: Evans *et al.* (1991, 1994); Gillespie (2001); Moon *et al.* (1993); Reissert (1905); Takamura *et al.* (2001).



Experimental

Crystal data

$C_{24}H_{19}N_3O_3S$
M_r = 429.5
Triclinic, $P\overline{1}$
a = 8.7215 (5) Å
b = 12.1042 (4) Å
c = 12.4678 (7) Å
α = 64.267 (3)°
β = 69.759 (5)°
γ = 87.895 (3)°
V = 1102.36 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 293 K
0.6 × 0.3 × 0.09 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: Gaussian (*JANA2006*; Petricek *et al.*, 2006)
T_{min} = 0.922, T_{max} = 0.977
34014 measured reflections
6242 independent reflections
4445 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.157
S = 2.06
6242 reflections
280 parameters
76 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *EVALCCD* (Duisenberg *et al.*, 2003); data reduction: *COLLECT*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *JANA2006* (Petricek *et al.*, 2006); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *JANA2006*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2055).

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supplementary materials

Acta Cryst. (2007). E**63**, o4023 [**doi:10.1107/S1600536807043644**]

3-Benzoyl-1-[(4-methylphenyl)sulfonyl]-1a,2,3,7b-tetrahydro-1*H*-azireno[2,3-*c*]quinoline-2-carbonitrile

M. Evain, L. Jean-Gérard, S. Collet and A. Guingant

Comment

Stereoselective additions to the double bond of 1-benzoyl-1,2-dihydroquinoline-2-carbonitrile (Reissert, 1905) have not been extensively studied in the past decades. Recently, an epoxidation reaction has been performed on an optically active Reissert-type compound (Takamura *et al.*, 2001) resulting in the formation of an epoxide ring *trans* disposed to the nitrile group.

In the course of an ongoing project directed towards the synthesis of sumanirole, a non-natural compound exhibiting anti-parkinsonian activities (Moon *et al.*, 1993), we were interested in introducing an aziridine ring at the C3—C4 positions of the racemic 1-benzoyl-1,2-dihydroquinoline-2-carbonitrile. Subsequent regioselective opening of this strained ring at its benzylic position would allow installation of the amine functionality found in sumanirole at carbon C3.

The aziridination reaction was performed using PhI=NTs (*N*-(*p*-tolylsulfonyl)imino)phenyliodinane (Gillespie, 2001) as a nitrene precursor and $Cu(acac)_2$ as a catalyst (Evans *et al.*, 1991, 1994). The reaction led to the formation of a single diastereoisomer in 70% isolated yield. To ascertain the stereochemical relationships between the two newly created stereogenic carbons (C3 and C4) and the carbon bearing the nitrile group (C2), compound (I) was subjected to X-ray crystal structure analysis.

The molecular structure of (I) reveals the *trans* relative stereochemistry between the aziridine ring and the nitrile group (Fig. 1).

Experimental

To a solution of 1-benzoyl-1,2-dihydroquinoline-2-carbonitrile (855 mg, 4.0 mmol) in dry CH_3CN (14 ml), $Cu(acac)_2$ (524 mg, 2.0 mmol) and PhI=NTs (10.4 g, 27.9 mmol) were added at room temperature. After 2 minutes of vigorous stirring, the temperature of the reaction mixture increased significantly and the initial blue suspension turned brown. The mixture was then allowed to cool to room temperature, filtered through a pad of silica and the filter cake was rinsed thoroughly with CH_2Cl_2. The filtrate was then concentrated *in vacuo* and the resulting solid was recrystallized in methylene chloride. The obtained crystals were filtered off. After being kept for 1 h at RT, the organic layer was filtered again. The resulting filtrate was concentrated *in vacuo* and the crude product was purified by silica gel chromatography (eluting with CH_2Cl_2) to afford (I) as a white solid in a 70% yield (1.2 g, 2.8 mmol). Single crystals of (I) suitable for X-ray analysis were obtained by slow crystallization from CH_2Cl_2/petroleum ether, at room temperature and without evaporation (m.p. 459 K).

Refinement

C—H H atoms were positioned with idealized geometry and were refined isotropic (U_{iso}(H) = 1.2× U_{eq}(C)) using a riding model. The O—H H atoms were located in difference map syntheses and were refined isotropic (U_{iso}(H) = 1.2× U_{eq}(O)) with varying coordinates.

Figures

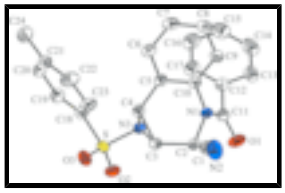


Fig. 1. Molecular structure showing 30% probability displacement ellipsoids. H atoms are omitted for clarity.

3-Benzoyl-1-[(4-methylphenyl)sulfonyl]-1a,2,3,7 b-tetrahydro-1*H*-azireno [2,3-*c*]quinoline-2-carbonitrile

Crystal data

$C_{24}H_{19}N_3O_3S$	V = 1102.36 (11) Å^3
M_r = 429.5	Z = 2
Triclinic, $P\bar{1}$	F_{000} = 448
Hall symbol: -P 1	D_x = 1.294 Mg m^{-3}
a = 8.7215 (5) Å	Mo $K\alpha$ radiation λ = 0.71069 Å
b = 12.1042 (4) Å	μ = 0.18 mm^{-1}
c = 12.4678 (7) Å	T = 293 K
α = 64.267 (3)º	Thick plate, colourless
β = 69.759 (5)º	0.6 × 0.3 × 0.09 mm
γ = 87.895 (3)º	

Data collection

Nonius KappaCCD diffractometer	6242 independent reflections
Radiation source: X-ray tube	4445 reflections with $I > 2\sigma(I)$
Monochromator: horizonally mounted graphite crystal	R_{int} = 0.055
Detector resolution: 9 pixels mm^{-1}	θ_{max} = 30.0º
T = 293 K	θ_{min} = 6.5º
CCD, φ and ω frames scans	h = −12→11
Absorption correction: Gaussian (JANA2006; Petricek *et al.*, 2006)	k = −17→16
T_{min} = 0.922, T_{max} = 0.977	l = −17→17
34014 measured reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.066$	Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.001936I^2)$
$wR(F^2) = 0.157$	$(\Delta/\sigma)_{max} = 0.001$
$S = 2.06$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
6242 reflections	$\Delta\rho_{min} = -0.36$ e Å^{-3}
280 parameters	Extinction correction: none
76 restraints	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.03291 (6)	0.08922 (4)	0.64456 (4)	0.0454 (2)
N1	0.38331 (17)	0.30804 (12)	0.24992 (13)	0.0384 (6)
N2	0.4895 (3)	0.0724 (2)	0.1684 (2)	0.0835 (12)
N3	0.18953 (17)	0.17068 (13)	0.50524 (13)	0.0390 (6)
O1	0.2135 (2)	0.41987 (14)	0.15447 (16)	0.0669 (9)
O2	−0.11266 (16)	0.12656 (15)	0.61889 (14)	0.0612 (8)
O3	0.04832 (19)	−0.04033 (13)	0.69441 (14)	0.0638 (7)
C1	0.4044 (3)	0.12536 (18)	0.21482 (18)	0.0519 (9)
C2	0.2965 (2)	0.18988 (15)	0.28240 (16)	0.0402 (7)
C3	0.2444 (2)	0.10530 (15)	0.42537 (16)	0.0403 (7)
C4	0.3496 (2)	0.11772 (15)	0.49134 (16)	0.0399 (7)
C5	0.5036 (2)	0.20893 (15)	0.41258 (16)	0.0383 (7)
C6	0.6338 (2)	0.20193 (17)	0.45378 (19)	0.0472 (9)
C7	0.7791 (2)	0.28230 (19)	0.3736 (2)	0.0548 (10)
C8	0.7955 (2)	0.36839 (19)	0.2523 (2)	0.0545 (10)
C9	0.6662 (2)	0.37820 (17)	0.20927 (18)	0.0456 (8)
C10	0.5196 (2)	0.29989 (15)	0.29066 (16)	0.0371 (7)
C11	0.3119 (2)	0.41407 (16)	0.20566 (17)	0.0426 (8)
C12	0.3561 (2)	0.51944 (17)	0.22680 (18)	0.0434 (8)
C13	0.3917 (2)	0.63836 (18)	0.1289 (2)	0.0573 (10)
C14	0.4231 (3)	0.7372 (2)	0.1500 (3)	0.0724 (12)
C15	0.4171 (3)	0.7182 (2)	0.2673 (3)	0.0734 (14)
C16	0.3781 (3)	0.6006 (2)	0.3665 (3)	0.0701 (14)
C17	0.3481 (3)	0.5005 (2)	0.3467 (2)	0.0570 (10)
C18	0.0664 (2)	0.14759 (17)	0.74182 (17)	0.0442 (8)
C19	0.1465 (3)	0.0827 (2)	0.82352 (19)	0.0556 (10)
C20	0.1657 (3)	0.1281 (2)	0.9038 (2)	0.0664 (11)
C21	0.1063 (3)	0.2348 (2)	0.90398 (19)	0.0602 (10)
C22	0.0286 (3)	0.2989 (2)	0.8193 (2)	0.0716 (14)
C23	0.0073 (3)	0.2564 (2)	0.7379 (2)	0.0623 (12)
C24	0.1240 (4)	0.2816 (3)	0.9939 (3)	0.0935 (16)
H1	0.203733	0.20999	0.254501	0.0483*
H2	0.199338	0.03397	0.424107	0.0484*

H3	0.398591	0.05981	0.548829	0.0479*
H4	0.623121	0.140591	0.539025	0.0567*
H5	0.869957	0.27826	0.402578	0.0657*
H6	0.899022	0.422553	0.196621	0.0654*
H7	0.678118	0.438968	0.123512	0.0548*
H8	0.394574	0.652419	0.045403	0.0688*
H9	0.449713	0.821142	0.081592	0.0869*
H10	0.44065	0.788185	0.280272	0.088*
H11	0.371761	0.588293	0.450215	0.0841*
H12	0.321681	0.417394	0.416576	0.0684*
H13	0.188175	0.006955	0.824521	0.0667*
H14	0.222353	0.083944	0.961486	0.0797*
H15	−0.011942	0.375303	0.816864	0.0859*
H16	−0.047788	0.301988	0.679431	0.0748*
H17	0.030292	0.328238	1.014529	0.1122*
H18	0.122915	0.209872	1.074466	0.1122*
H19	0.230573	0.337637	0.952164	0.1122*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0484 (3)	0.0468 (3)	0.0412 (2)	−0.00713 (18)	−0.01196 (18)	−0.0223 (2)
N1	0.0433 (7)	0.0354 (7)	0.0410 (7)	0.0015 (6)	−0.0209 (6)	−0.0167 (6)
N2	0.1091 (17)	0.0649 (13)	0.0662 (13)	0.0108 (12)	−0.0106 (12)	−0.0371 (11)
N3	0.0449 (8)	0.0370 (7)	0.0382 (7)	0.0010 (6)	−0.0151 (6)	−0.0194 (6)
O1	0.0785 (10)	0.0601 (9)	0.0908 (11)	0.0173 (7)	−0.0626 (9)	−0.0350 (9)
O2	0.0460 (7)	0.0875 (11)	0.0615 (9)	−0.0034 (7)	−0.0203 (6)	−0.0419 (8)
O3	0.0841 (10)	0.0413 (8)	0.0518 (8)	−0.0131 (6)	−0.0115 (7)	−0.0173 (6)
C1	0.0706 (13)	0.0428 (10)	0.0406 (10)	−0.0025 (9)	−0.0155 (9)	−0.0203 (8)
C2	0.0473 (9)	0.0389 (9)	0.0403 (9)	0.0002 (7)	−0.0194 (7)	−0.0198 (7)
C3	0.0511 (9)	0.0347 (8)	0.0404 (9)	−0.0012 (7)	−0.0163 (7)	−0.0213 (7)
C4	0.0498 (9)	0.0358 (8)	0.0397 (9)	0.0079 (7)	−0.0204 (7)	−0.0189 (7)
C5	0.0441 (9)	0.0365 (8)	0.0435 (9)	0.0081 (6)	−0.0200 (7)	−0.0231 (7)
C6	0.0565 (11)	0.0452 (10)	0.0551 (11)	0.0146 (8)	−0.0324 (9)	−0.0269 (9)
C7	0.0468 (10)	0.0600 (12)	0.0752 (14)	0.0122 (9)	−0.0351 (10)	−0.0362 (11)
C8	0.0417 (10)	0.0539 (12)	0.0700 (13)	0.0019 (8)	−0.0213 (9)	−0.0288 (11)
C9	0.0432 (9)	0.0424 (10)	0.0503 (10)	0.0009 (7)	−0.0171 (8)	−0.0197 (9)
C10	0.0388 (8)	0.0365 (8)	0.0438 (9)	0.0066 (6)	−0.0201 (7)	−0.0211 (7)
C11	0.0441 (9)	0.0425 (9)	0.0460 (9)	0.0047 (7)	−0.0220 (7)	−0.0198 (8)
C12	0.0411 (9)	0.0441 (10)	0.0513 (10)	0.0091 (7)	−0.0191 (7)	−0.0255 (8)
C13	0.0661 (13)	0.0449 (11)	0.0544 (12)	0.0037 (9)	−0.0157 (9)	−0.0213 (9)
C14	0.0865 (17)	0.0441 (12)	0.0748 (16)	−0.0027 (11)	−0.0144 (13)	−0.0272 (12)
C15	0.0778 (15)	0.0608 (15)	0.0934 (18)	0.0045 (11)	−0.0202 (13)	−0.0532 (14)
C16	0.0884 (16)	0.0706 (15)	0.0749 (16)	0.0185 (12)	−0.0350 (13)	−0.0498 (14)
C17	0.0729 (13)	0.0514 (12)	0.0575 (12)	0.0137 (10)	−0.0291 (10)	−0.0303 (10)
C18	0.0431 (9)	0.0515 (10)	0.0384 (9)	−0.0032 (7)	−0.0123 (7)	−0.0220 (8)
C19	0.0618 (12)	0.0534 (12)	0.0525 (11)	0.0030 (9)	−0.0254 (9)	−0.0208 (10)
C20	0.0790 (15)	0.0690 (15)	0.0489 (12)	−0.0123 (11)	−0.0330 (11)	−0.0155 (11)

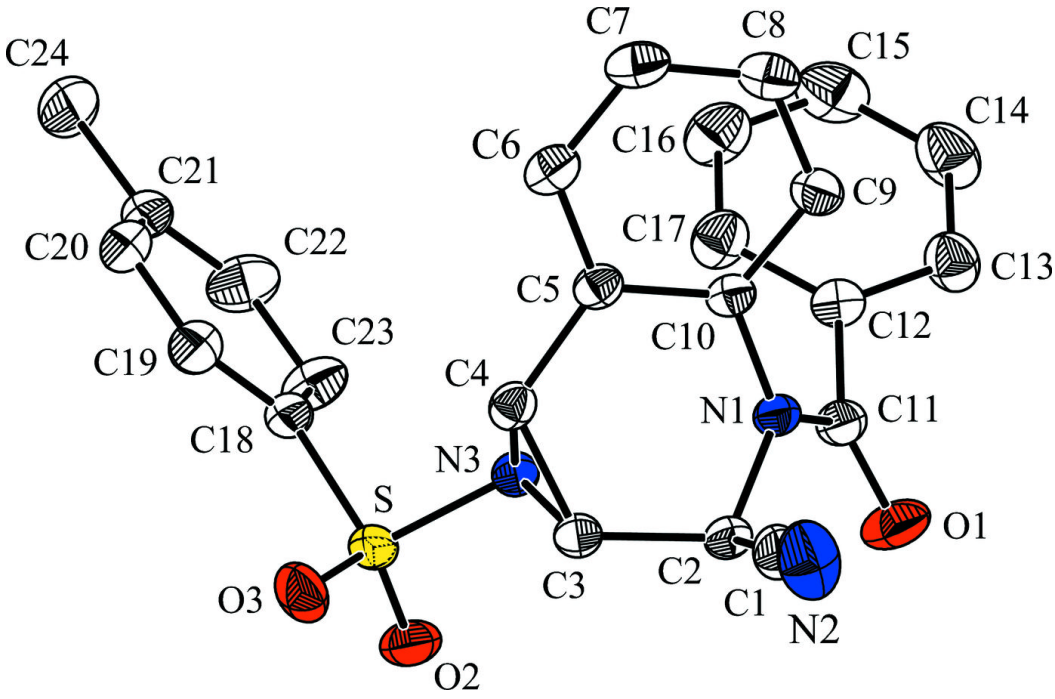
C21	0.0654 (13)	0.0712 (14)	0.0427 (10)	−0.0187 (10)	−0.0136 (9)	−0.0273 (10)
C22	0.0804 (16)	0.0755 (16)	0.0899 (17)	0.0158 (12)	−0.0390 (14)	−0.0583 (14)
C23	0.0741 (14)	0.0674 (14)	0.0749 (15)	0.0198 (11)	−0.0444 (12)	−0.0452 (12)
C24	0.131 (2)	0.096 (2)	0.0582 (15)	−0.0318 (17)	−0.0286 (16)	−0.0393 (15)

Geometric parameters (Å, °)

S—N3	1.6809 (12)	C16—C17	1.387 (4)
S—O2	1.4255 (16)	C18—C19	1.378 (3)
S—O3	1.4350 (15)	C18—C23	1.384 (3)
S—C18	1.749 (3)	C19—C20	1.390 (4)
N1—C2	1.465 (3)	C20—C21	1.375 (4)
N1—C10	1.431 (3)	C21—C22	1.386 (4)
N1—C11	1.380 (2)	C21—C24	1.509 (5)
N2—C1	1.134 (4)	C22—C23	1.384 (5)
N3—C3	1.474 (3)	C2—H1	0.970
N3—C4	1.501 (2)	C3—H2	0.970
O1—C11	1.218 (3)	C4—H3	0.970
C1—C2	1.474 (3)	C6—H4	0.970
C2—C3	1.523 (2)	C7—H5	0.970
C3—C4	1.477 (3)	C8—H6	0.970
C4—C5	1.497 (2)	C9—H7	0.970
C5—C6	1.385 (3)	C13—H8	0.970
C5—C10	1.398 (2)	C14—H9	0.970
C6—C7	1.386 (2)	C15—H10	0.970
C7—C8	1.374 (3)	C16—H11	0.970
C8—C9	1.388 (3)	C17—H12	0.970
C9—C10	1.385 (2)	C19—H13	0.970
C11—C12	1.498 (3)	C20—H14	0.970
C12—C13	1.380 (2)	C22—H15	0.970
C12—C17	1.387 (4)	C23—H16	0.970
C13—C14	1.388 (4)	C24—H17	1.000
C14—C15	1.360 (5)	C24—H18	1.000
C15—C16	1.373 (3)	C24—H19	1.000
N3—S—O2	105.04 (8)	C8—C9—C10	119.04 (17)
N3—S—O3	110.74 (8)	N1—C10—C5	118.70 (13)
N3—S—C18	101.40 (9)	N1—C10—C9	120.56 (15)
O2—S—O3	117.70 (11)	C5—C10—C9	120.71 (19)
O2—S—C18	110.99 (11)	N1—C11—O1	120.9 (2)
O3—S—C18	109.65 (10)	N1—C11—C12	117.40 (19)
C2—N1—C10	115.81 (14)	O1—C11—C12	121.66 (18)
C2—N1—C11	118.48 (16)	C11—C12—C13	119.7 (2)
C10—N1—C11	124.53 (17)	C11—C12—C17	120.77 (16)
S—N3—C3	113.62 (11)	C13—C12—C17	119.3 (2)
S—N3—C4	115.41 (9)	C12—C13—C14	120.0 (2)
C3—N3—C4	59.51 (13)	C13—C14—C15	120.5 (2)
N2—C1—C2	176.4 (3)	C14—C15—C16	120.1 (3)
N1—C2—C1	110.98 (14)	C15—C16—C17	120.1 (3)
N1—C2—C3	110.03 (18)	C12—C17—C16	119.87 (19)

C1—C2—C3	107.33 (14)	S—C18—C19	119.53 (18)
N3—C3—C2	113.77 (14)	S—C18—C23	119.17 (19)
N3—C3—C4	61.15 (13)	C19—C18—C23	121.3 (3)
C2—C3—C4	117.69 (14)	C18—C19—C20	118.5 (2)
N3—C4—C3	59.33 (13)	C19—C20—C21	121.8 (2)
N3—C4—C5	116.36 (13)	C20—C21—C22	118.2 (3)
C3—C4—C5	117.95 (15)	C20—C21—C24	121.3 (2)
C4—C5—C6	122.16 (15)	C22—C21—C24	120.5 (3)
C4—C5—C10	118.65 (18)	C21—C22—C23	121.6 (3)
C6—C5—C10	119.09 (14)	C18—C23—C22	118.6 (2)
C5—C6—C7	120.20 (18)	H17—C24—H18	109.5
C6—C7—C8	120.2 (2)	H17—C24—H19	109.5
C7—C8—C9	120.70 (17)	H18—C24—H19	109.5

Fig. 1


C24
C21
C20
C22
C19
C23
C18
S
O3
O2
N3
C4
C5
C6
C7
C8
C15
C14
C16
C9
C17
C13
C10
C12
N1
C11
O1
C3
C2
C1
N2