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4-Tosyl-1-oxa-4-azaspiro[4.5]deca-6,9-dien-8-one

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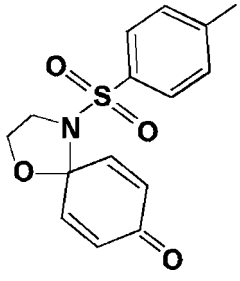
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.046; wR factor = 0.119; data-to-parameter ratio = 13.7.

In the molecule of the title compound, $C_{15}H_{15}NO_4S$, the two six-membered rings are almost parallel to each other [dihedral angle = 1.87 (9)°] and perpendicular to the mean plane through the five-membered ring [dihedral angles of 89.98 (10) and 89.04 (10)°]. The crystal structure is stabilized by intermolecular C—H···O hydrogen-bonding interactions.

Related literature

For general background to the catalytic oxidation of phenol derivatives using transition metal complexes, see: Bernini *et al.* (2006); Cheung *et al.* (2005). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{15}H_{15}NO_4S$ M_r = 305.34
Monoclinic, $P2_1/c$
a = 11.882 (3) Å
b = 14.973 (6) Å
c = 8.369 (5) Å
β = 107.00 (3)°
V = 1423.9 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 294 K
0.30 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
2996 measured reflections
2625 independent reflections
1526 reflections with $I > 2\sigma(I)$
R_{int} = 0.008
3 standard reflections
every 300 reflections
intensity decay: 1.6%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.119
S = 1.02
2625 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O4^{i}	0.93	2.48	3.211 (4)	134
C15—H15B···O2^{ii}	0.96	2.57	3.520 (5)	171

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x, y, z+1$.

Data collection: *DIFRAC* (Gabe & White, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2288).

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supporting information

Acta Cryst. (2009). E**65**, o649 [doi:10.1107/S1600536809001524]

4-Tosyl-1-oxa-4-azaspiro[4.5]deca-6,9-dien-8-one

Ping Yin, Wen-Wen Zhang, Hai-Bin Yin, Ling He and Wen-Cai Huang

S1. Comment

In the course of our studies aimed to prepare a substituted quinone from the corresponding aromatic ether by catalytic oxidation using transition metal complexes (Cheung *et al.*, 2005; Bernini *et al.*,2006), the title compound was unexpectedly obtained in about 70% yield. In the molecule of the title compound (Fig. 1) the C1–C6 and C9–C14 six-membered rings are almost parallel to each other(dihedral angle 1.87 (9)°) and perpendicular to the mean plane through the O1/N1/C1/C7/C8 ring, forming dihedral angles of 89.98 (10) and 89.04 (10)°, respectively. The five-membered ring adopts an envelope conformation, with puckering parameters $Q2$ = 0.269 (3) Å and $\varphi 2$ =104.4 (6)° ((Cremer & Pople, 1975). The crystal structure (Fig. 2) is enforced by intermolecular C—H···O hydrogen bonds (Table 1).

S2. Experimental

A solution of 2-phenoxyethyl-*p*-toluenesulfon amide (1 mmol) and indobenzene diacetate (1.5 mmol) in dichloromethane was charged in a reaction flask and 4 A molecular sieves was added. Then, the mixture was stirred at 303 K under a nitrogen atmosphere for 4 h. After cooling to room temperature, the resulting mixture was filtered and the solvent was removed under vacuo. The residue was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (5:1 *v*/*v*) as the eluent. Colorless crystals suitable for X-ray analysis were obtained by slow evaporation in a cyclohexane/ether solution (5:1 *v*/*v*) at room temperature.

S3. Refinement

H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined using a riding model, with U_{iso}(H) = 1.2–1.5U_{eq}(C).

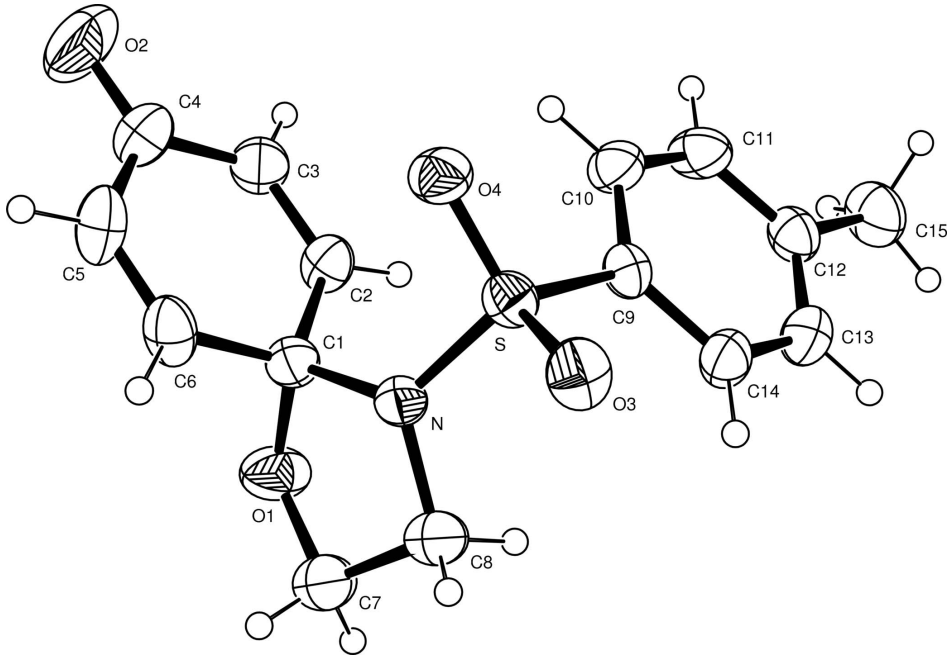

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

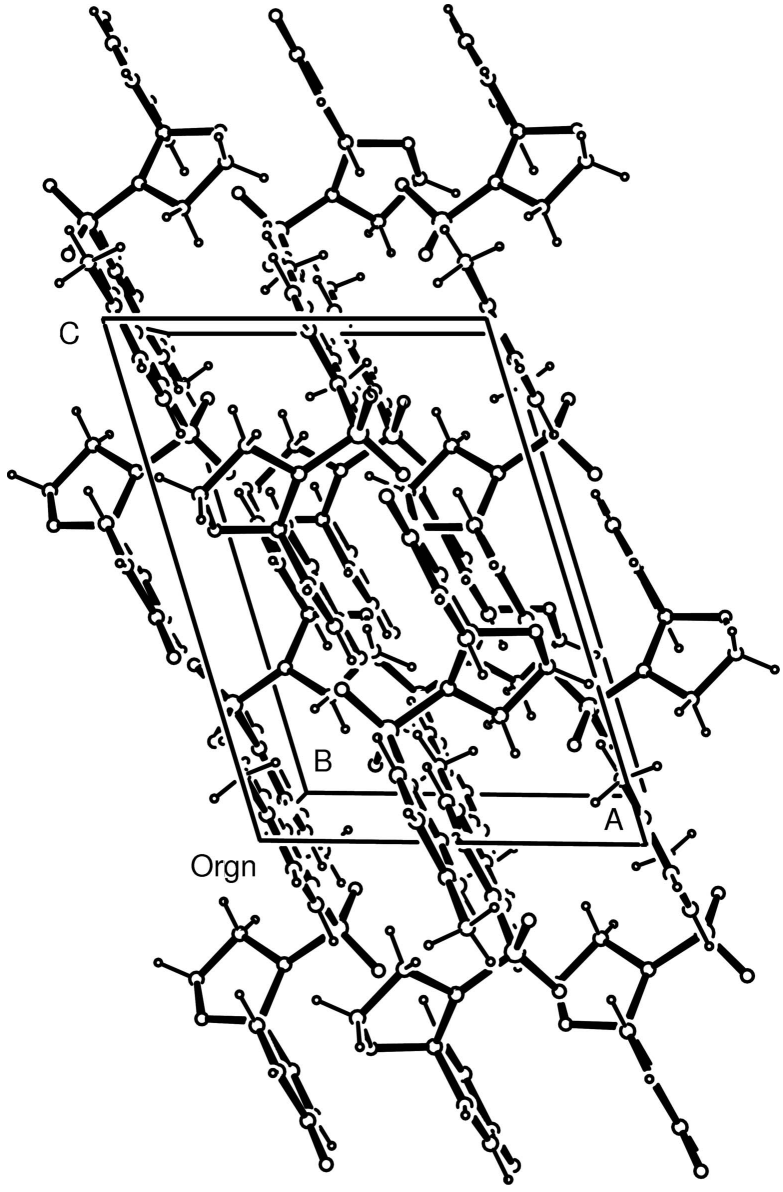

Figure 2

Crystal packing of the title compound approximately viewed along the *b* axis.

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Crystal data

$C_{15}H_{15}NO_4S$
$M_r = 305.34$
Monoclinic, $P2_1/c$
$a = 11.882$ (3) Å
$b = 14.973$ (6) Å
$c = 8.369$ (5) Å
$\beta = 107.00$ (3)°
$V = 1423.9$ (11) Å^3
$Z = 4$
$F(000) = 640$
$D_x = 1.424$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 22 reflections
$\theta = 4.5$–7.7°
$\mu = 0.24$ mm^{-1}
$T = 294$ K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2$–θ scans
2996 measured reflections
2625 independent reflections
1526 reflections with $I > 2\sigma(I)$
$R_{int} = 0.008$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.8°$
$h = -14 \rightarrow 3$
$k = -18 \rightarrow 0$
$l = -9 \rightarrow 10$
3 standard reflections every 300 reflections
intensity decay: 1.6%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.119$
$S = 1.02$
2625 reflections
192 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.2087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.28106 (6)	0.58803 (5)	0.06825 (9)	0.0407 (2)
O1	0.09259 (19)	0.57997 (15)	−0.3866 (3)	0.0638 (7)
O2	−0.1639 (2)	0.7616 (2)	−0.1273 (4)	0.1088 (11)
O3	0.20120 (17)	0.62375 (14)	0.1501 (3)	0.0500 (5)
O4	0.35194 (17)	0.51210 (13)	0.1369 (3)	0.0531 (6)
N1	0.20196 (18)	0.55681 (15)	−0.1165 (3)	0.0408 (6)
C1	0.0935 (2)	0.60359 (18)	−0.2213 (4)	0.0420 (7)
C2	0.0995 (3)	0.70272 (19)	−0.2139 (4)	0.0448 (7)
H2	0.1650	0.7308	−0.2304	0.054*
C3	0.0165 (3)	0.7524 (2)	−0.1852 (4)	0.0532 (8)
H3	0.0239	0.8142	−0.1869	0.064*
C4	−0.0868 (3)	0.7143 (3)	−0.1509 (5)	0.0648 (10)
C5	−0.0943 (3)	0.6167 (2)	−0.1518 (4)	0.0612 (9)
H5	−0.1587	0.5897	−0.1301	0.073*
C6	−0.0124 (3)	0.5663 (2)	−0.1826 (4)	0.0523 (8)
H6	−0.0207	0.5046	−0.1802	0.063*

C7	0.1620 (3)	0.5036 (2)	−0.3849 (4)	0.0662 (10)
H7A	0.1950	0.5048	−0.4780	0.079*
H7B	0.1151	0.4498	−0.3926	0.079*
C8	0.2563 (3)	0.5062 (2)	−0.2247 (4)	0.0613 (9)
H8A	0.2779	0.4465	−0.1815	0.074*
H8B	0.3257	0.5364	−0.2365	0.074*
C9	0.3779 (2)	0.67304 (18)	0.0454 (3)	0.0383 (7)
C10	0.3497 (3)	0.7621 (2)	0.0559 (4)	0.0493 (8)
H10	0.2795	0.7780	0.0765	0.059*
C11	0.4264 (3)	0.8267 (2)	0.0357 (4)	0.0536 (9)
H11	0.4072	0.8865	0.0431	0.064*
C12	0.5312 (3)	0.8055 (2)	0.0047 (4)	0.0472 (8)
C13	0.5568 (3)	0.7168 (2)	−0.0071 (4)	0.0535 (8)
H13	0.6266	0.7012	−0.0288	0.064*
C14	0.4821 (2)	0.6503 (2)	0.0123 (4)	0.0487 (8)
H14	0.5012	0.5907	0.0034	0.058*
C15	0.6133 (3)	0.8785 (2)	−0.0157 (4)	0.0669 (10)
H15A	0.5717	0.9187	−0.1021	0.100*
H15B	0.6783	0.8526	−0.0456	0.100*
H15C	0.6421	0.9105	0.0875	0.100*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0436 (4)	0.0356 (4)	0.0419 (4)	−0.0024 (4)	0.0108 (3)	0.0011 (4)
O1	0.0715 (15)	0.0670 (15)	0.0453 (13)	0.0231 (13)	0.0053 (11)	−0.0080 (12)
O2	0.0677 (17)	0.102 (2)	0.170 (3)	0.0206 (17)	0.0560 (19)	−0.020 (2)
O3	0.0541 (12)	0.0512 (12)	0.0523 (13)	−0.0066 (10)	0.0274 (11)	−0.0068 (10)
O4	0.0538 (13)	0.0428 (12)	0.0567 (13)	0.0034 (10)	0.0068 (10)	0.0121 (10)
N1	0.0385 (13)	0.0350 (13)	0.0467 (14)	0.0030 (10)	0.0090 (11)	−0.0056 (11)
C1	0.0415 (16)	0.0367 (17)	0.0463 (17)	0.0037 (13)	0.0106 (13)	−0.0020 (13)
C2	0.0433 (17)	0.0389 (17)	0.055 (2)	−0.0021 (14)	0.0191 (15)	0.0070 (15)
C3	0.0524 (19)	0.0370 (17)	0.069 (2)	0.0035 (15)	0.0153 (17)	−0.0028 (16)
C4	0.046 (2)	0.073 (3)	0.077 (3)	0.0122 (19)	0.0189 (18)	−0.006 (2)
C5	0.0384 (18)	0.068 (2)	0.076 (2)	−0.0077 (17)	0.0154 (17)	0.014 (2)
C6	0.0427 (17)	0.0369 (17)	0.070 (2)	−0.0053 (15)	0.0056 (16)	0.0093 (16)
C7	0.056 (2)	0.074 (3)	0.062 (2)	0.0135 (19)	0.0072 (18)	−0.0205 (19)
C8	0.0557 (19)	0.063 (2)	0.062 (2)	0.0097 (18)	0.0110 (17)	−0.0179 (18)
C9	0.0354 (16)	0.0394 (16)	0.0388 (16)	−0.0060 (13)	0.0088 (13)	0.0004 (13)
C10	0.0471 (18)	0.0431 (18)	0.065 (2)	−0.0050 (14)	0.0273 (16)	−0.0088 (16)
C11	0.064 (2)	0.0366 (17)	0.067 (2)	−0.0055 (16)	0.0306 (18)	−0.0081 (16)
C12	0.0453 (18)	0.050 (2)	0.0463 (19)	−0.0113 (15)	0.0135 (15)	−0.0031 (15)
C13	0.0389 (17)	0.060 (2)	0.064 (2)	−0.0015 (16)	0.0178 (16)	−0.0038 (18)
C14	0.0379 (16)	0.0429 (18)	0.063 (2)	0.0027 (14)	0.0107 (15)	−0.0012 (15)
C15	0.062 (2)	0.070 (2)	0.073 (2)	−0.0234 (19)	0.0262 (19)	−0.007 (2)

Geometric parameters (Å, º)

S1—O3	1.427 (2)	C7—C8	1.478 (4)
S1—O4	1.431 (2)	C7—H7A	0.9700
S1—N1	1.626 (2)	C7—H7B	0.9700
S1—C9	1.763 (3)	C8—H8A	0.9700
O1—C7	1.409 (4)	C8—H8B	0.9700
O1—C1	1.424 (4)	C9—C10	1.384 (4)
O2—C4	1.218 (4)	C9—C14	1.388 (4)
N1—C8	1.469 (4)	C10—C11	1.372 (4)
N1—C1	1.503 (3)	C10—H10	0.9300
C1—C2	1.487 (4)	C11—C12	1.381 (4)
C1—C6	1.496 (4)	C11—H11	0.9300
C2—C3	1.313 (4)	C12—C13	1.373 (4)
C2—H2	0.9300	C12—C15	1.508 (4)
C3—C4	1.455 (4)	C13—C14	1.374 (4)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.463 (5)	C14—H14	0.9300
C5—C6	1.314 (4)	C15—H15A	0.9600
C5—H5	0.9300	C15—H15B	0.9600
C6—H6	0.9300	C15—H15C	0.9600
O3—S1—O4	120.05 (13)	O1—C7—H7B	110.5
O3—S1—N1	106.48 (12)	C8—C7—H7B	110.5
O4—S1—N1	105.19 (12)	H7A—C7—H7B	108.7
O3—S1—C9	109.16 (13)	N1—C8—C7	102.7 (2)
O4—S1—C9	106.92 (13)	N1—C8—H8A	111.2
N1—S1—C9	108.57 (13)	C7—C8—H8A	111.2
C7—O1—C1	110.7 (2)	N1—C8—H8B	111.2
C8—N1—C1	109.7 (2)	C7—C8—H8B	111.2
C8—N1—S1	119.94 (18)	H8A—C8—H8B	109.1
C1—N1—S1	125.44 (18)	C10—C9—C14	119.7 (3)
O1—C1—C2	106.0 (2)	C10—C9—S1	120.7 (2)
O1—C1—C6	110.4 (2)	C14—C9—S1	119.5 (2)
C2—C1—C6	113.4 (2)	C11—C10—C9	119.3 (3)
O1—C1—N1	102.3 (2)	C11—C10—H10	120.4
C2—C1—N1	114.7 (2)	C9—C10—H10	120.4
C6—C1—N1	109.3 (2)	C10—C11—C12	121.9 (3)
C3—C2—C1	123.0 (3)	C10—C11—H11	119.0
C3—C2—H2	118.5	C12—C11—H11	119.0
C1—C2—H2	118.5	C13—C12—C11	117.8 (3)
C2—C3—C4	122.4 (3)	C13—C12—C15	121.9 (3)
C2—C3—H3	118.8	C11—C12—C15	120.2 (3)
C4—C3—H3	118.8	C12—C13—C14	121.9 (3)
O2—C4—C3	121.4 (4)	C12—C13—H13	119.1
O2—C4—C5	122.2 (3)	C14—C13—H13	119.1
C3—C4—C5	116.3 (3)	C13—C14—C9	119.4 (3)
C6—C5—C4	121.8 (3)	C13—C14—H14	120.3

C6—C5—H5	119.1	C9—C14—H14	120.3
C4—C5—H5	119.1	C12—C15—H15A	109.5
C5—C6—C1	123.0 (3)	C12—C15—H15B	109.5
C5—C6—H6	118.5	H15A—C15—H15B	109.5
C1—C6—H6	118.5	C12—C15—H15C	109.5
O1—C7—C8	105.9 (3)	H15A—C15—H15C	109.5
O1—C7—H7A	110.5	H15B—C15—H15C	109.5
C8—C7—H7A	110.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O4^{i}	0.93	2.48	3.211 (4)	134
C15—H15*B*···O2^{ii}	0.96	2.57	3.520 (5)	171

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) *x*, *y*, *z*+1.