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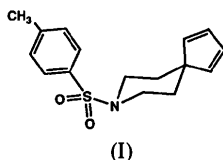# N-Tosyl-8-azaspiro[4.5]deca-1,3-diene

BY J. GABRIEL GARCIA, FRANK R. FRONCZEK AND MARK L. MCLAUGHLIN*

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803-1804, USA

Abstract. $C_{16}H_{19}NO_2S$, $M_r = 289 \cdot 4$, orthorhombic, $P2_12_12_1$, $a = 6 \cdot 444$ (3), $b = 7 \cdot 3726$ (11), $c = 32 \cdot 038$ (2) Å, $V = 1522 \cdot 2$ (8) Å^3, $Z = 4$, $D_x = 1 \cdot 266$ g cm^{-3}, $\lambda(Cu\ K\alpha) = 1 \cdot 54184$ Å, $\mu = 18 \cdot 5$ cm^{-1}, $F(000) = 616$, $T = 296$ K, $R = 0 \cdot 060$ for 1156 observations with $I > 3\sigma(I)$ (of 1851 unique data). The six atoms of the aromatic ring exhibit a maximum deviation of $0 \cdot 009$ (7) Å from coplanarity, with the sulfur atom and tolyl carbon atom lying $0 \cdot 108$ (2) and $0 \cdot 104$ (9) Å respectively to the same side of this plane. The five atoms of the cyclopentadienyl ring exhibit a maximum deviation of $0 \cdot 003$ (6) Å from coplanarity. The dihedral angle between the aromatic ring and the cyclopentadienyl ring is $105 \cdot 2$ (2)°. The N—S bond distance is $1 \cdot 631$ (5) Å and the S=O bond distances are $1 \cdot 446$ (4) and $1 \cdot 417$ (5) Å. The piperidine ring has a chair conformation.

Experimental. N-(p-Toluenesulfonyl)-8-azaspiro[4.5]deca-1,3-diene (I) was prepared by treating N-(p-toluenesulfonyl)bis[2-(p-toluenesulfonate)ethyl]amine, $TsN(CH_2CH_2OTs)_2$ (Searle & Geue, 1984) with sodium cyclopentadienide in hexamethylene phosphoramide (Halterman, Vollhardt, Welker, Bläser & Böse, 1987).

(I)

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* Author to whom correspondence should be addressed.

Crystals, m.p. 379–381 K, formed by slow cooling of a 2,2,4-trimethylpentane solution; a clear, colorless crystal with dimensions $0 \cdot 08 \times 0 \cdot 25 \times 0 \cdot 42$ mm was used for the data collection on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $25 < \theta < 30°$. The ω–2θ scans were designed for $I = 25\sigma(I)$, subject to max. scan time = 90 s, scan rates varied from $0 \cdot 57$–$3 \cdot 30°$ min^{-1}. One octant of data having $2 < \theta < 75°$, $0 \le h \le 8, 0 \le k \le 9, 0 \le l \le 40$ was measured and corrected for background, Lorentz, and polarization. Absorption corrections were based on ψ scans; the min. relative transmission coefficient was $0 \cdot 844$. Three standard reflections (200, 020, 006) showed only a random fluctuation in intensity so no correction for decay was applied. Systematic absences $h00$ with h odd, $0k0$ with k odd, and $00l$ with l odd indicated space group $P2_12_12_1$. The structure was solved by direct methods. An 11 atom fragment recognized in an E map from $RANTAN$ (Yao, 1981) was expanded using $DIRDIF$ (Beurskens, 1984) to the full structure and refined by full-matrix least squares based upon F, using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0 \cdot 02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). Non-H-atom coordinates were refined with anisotropic thermal parameters; many of the H-atom positions were visible in ΔF maps; however, H atoms were included as fixed contributions, $0 \cdot 95$ Å from the bonded C atom with isotropic $B = 1 \cdot 3$ B_{eq} for the C atom. Final $R = 0 \cdot 060$

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
S	0·4811 (2)	1·0023 (3)	0·36608 (5)	5·42 (3)
O1	0·2675 (6)	0·9446 (7)	0·3621 (2)	7·8 (1)
O2	0·5298 (7)	1·1792 (6)	0·3803 (1)	7·1 (1)
N	0·5923 (7)	0·8605 (7)	0·3981 (1)	4·7 (1)
C1	0·5434 (9)	0·6658 (9)	0·3941 (2)	5·3 (1)
C2	0·5960 (9)	0·5688 (8)	0·4339 (2)	5·1 (1)
C3	0·8288 (9)	0·5926 (8)	0·4461 (2)	4·5 (1)
C4	0·872 (1)	0·7961 (9)	0·4470 (2)	5·8 (2)
C5	0·8176 (9)	0·8941 (9)	0·4071 (2)	5·9 (2)
C6	0·9707 (9)	0·488 (1)	0·4178 (2)	6·4 (2)
C7	1·075 (1)	0·367 (1)	0·4399 (2)	7·3 (2)
C8	1·018 (1)	0·379 (1)	0·4825 (2)	7·7 (2)
C9	0·876 (1)	0·509 (1)	0·4869 (2)	6·2 (2)
C10	0·5924 (8)	0·9789 (8)	0·3169 (2)	4·4 (1)
C11	0·7922 (9)	1·0498 (8)	0·3092 (2)	5·3 (2)
C12	0·873 (1)	1·040 (1)	0·2697 (2)	6·3 (2)
C13	0·770 (1)	0·9643 (9)	0·2372 (2)	6·2 (2)
C14	0·575 (1)	0·8912 (9)	0·2450 (2)	6·9 (2)
C15	0·494 (1)	0·9018 (9)	0·2844 (2)	5·8 (2)
C16	0·857 (1)	0·967 (1)	0·1934 (2)	9·9 (2)

Table 2. *Bond distances (Å), angles (°) and selected torsion angles (°)*

S—O1	1·446 (4)	C4—C5	1·511 (9)
S—O2	1·417 (5)	C6—C7	1·321 (10)
S—N	1·631 (5)	C7—C8	1·415 (11)
S—C10	1·740 (5)	C8—C9	1·336 (10)
N—C1	1·476 (8)	C10—C11	1·412 (8)
N—C5	1·501 (7)	C10—C15	1·345 (8)
C1—C2	1·501 (8)	C11—C12	1·371 (8)
C2—C3	1·560 (8)	C12—C13	1·358 (9)
C3—C4	1·527 (9)	C13—C14	1·389 (10)
C3—C6	1·503 (8)	C13—C16	1·511 (9)
C3—C9	1·476 (8)	C14—C15	1·367 (9)
O1—S—O2	120·7 (3)	C3—C4—C5	114·2 (5)
O1—S—N	106·6 (3)	N—C5—C4	108·0 (5)
O1—S—C10	106·4 (3)	C3—C6—C7	109·4 (5)
O2—S—N	106·8 (3)	C6—C7—C8	110·1 (6)
O2—S—C10	107·0 (3)	C7—C8—C9	108·9 (6)
N—S—C10	109·0 (3)	C3—C9—C8	110·3 (6)
S—N—C1	118·3 (4)	S—C10—C11	119·8 (4)
S—N—C5	116·1 (4)	S—C10—C15	123·2 (5)
C1—N—C5	112·6 (5)	C11—C10—C15	116·9 (5)
N—C1—C2	109·9 (5)	C10—C11—C12	119·3 (5)
C1—C2—C3	112·1 (5)	C11—C12—C13	122·9 (6)
C2—C3—C4	107·0 (5)	C12—C13—C14	117·7 (6)
C2—C3—C6	112·1 (5)	C12—C13—C16	121·7 (7)
C2—C3—C9	111·9 (5)	C14—C13—C16	120·6 (6)
C4—C3—C6	114·0 (5)	C13—C14—C15	119·3 (6)
C4—C3—C9	110·7 (5)	C10—C15—C14	123·9 (6)
C6—C3—C9	101·2 (5)		
O1—S—N—C1	41·4 (5)	C10—S—N—C1	−73·1 (5)
C10—S—N—C5	65·5 (5)	N—S—C10—C11	−74·8 (5)
S—N—C1—C2	−160·0 (4)	N—C1—C2—C3	−57·8 (6)
C1—C2—C3—C6	−71·6 (7)	C2—C3—C4—C5	−54·8 (6)
C9—C3—C6—C7	0·5 (7)	C6—C3—C9—C8	−0·4 (7)
C15—C10—C11—C12	1·2 (9)	C11—C12—C13—C16	175·9 (7)
C12—C13—C14—C15	1·4 (10)	O2—S—N—C1	41·4 (5)

for 1156 observed data (0·102 for all 1851 data), wR = 0·063, $S = 2·728$ for 182 variables. Max. shift 0·01σ in the final cycle, max. residual density 0·43, min. −0·32 e Å^{-3}, extinction coefficient $g = 2·5$ (3) × 10^{-6} where the factor $(1 + gI_c)^{-1}$ was applied to F_c. The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a perspective drawing

showing the atom numbering and Fig. 2 is a stereoview of the unit cell. Bond distances, angles and selected torsion angles are presented in Table 2.*

Related literature. The structures of N-(1-(4-p-fluorophenyl-4-oxobutyl)-4-phenyl-4-piperidinyl-methyl)acetamide (Van Opdenbosch, Evrard, Dorval, Durant & Koch, 1977), 1′,1″-dimethyldispiro{1,6,20,25-tetraoxa[6.1.6.1]paracyclophane-13,4′:32,4″-bis(piperidine)}-p-xylene clathrate (Krieger & Diederich, 1985), and 4-ethoxycarbonyl-
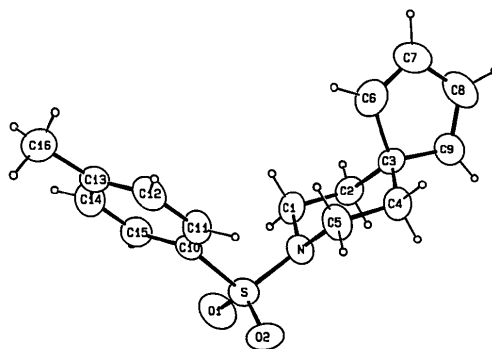
Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius.
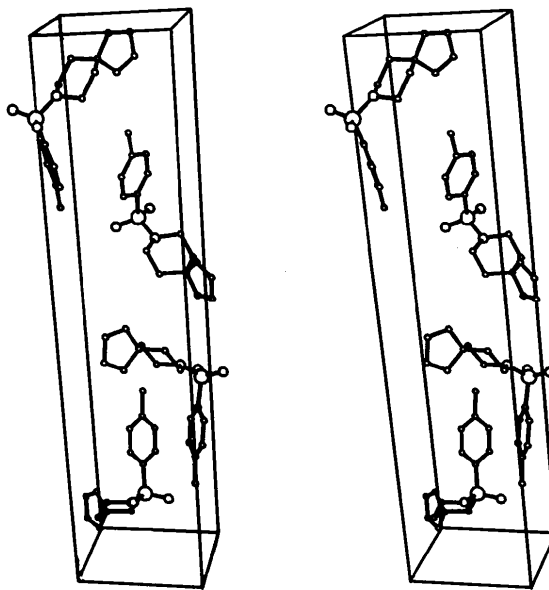

Fig. 2. Stereoview of the unit cell. c is vertical, b is horizontal and a is into the plane of the paper.

1-methyl-4-phenylpiperidine hydrochloride (Tillack, Seccombe, Kennard & Oh, 1974) exhibit the normal chair conformation for the piperidine ring, as does the title compound. The bond distances C6—C7 and C8—C9 in the title compound [1·321 (10) and 1·336 (10) Å] are similar to those in 9-(2,4-cyclopentadienylidene)bicyclo[3.3.1]nonane (Fronczek, Garcia & McLaughlin, 1990) at 1·340 (1) and 1·339 (2) Å, respectively.

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Structure of 3-Ethyl-4-oxa-1,5,6-trihydrophthalimide

BY KEVIN A. ANDERSEN AND OREN P. ANDERSON*

Department of Chemistry, Colorado State University, Fort Collins, Colorado 80523, USA

Abstract. 4-Ethyl-1,3,6,7-tetrahydro-5H-pyrano-[3,4-c]pyrrole-1,3-dione, $C_9H_{11}NO_3$, $M_r = 181·19$, monoclinic, $P2_1/c$, $a = 12·262$ (3), $b = 8·027$ (2), $c = 8·596$ (1) Å, $\beta = 92·36$ (2)°, $V = 845·4$ (3) Å^3, $Z = 4$, $D_x = 1·42$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0·7107$ Å, $\mu = 1·2$ cm^{-1}, $F(000) = 384$, $T = 165$ K, $R = 0·050$ ($wR = 0·075$) for 1365 unique, observed reflections. The compound is a derivative of phthalimide substituted in the six-membered ring by an ether oxygen and an ethyl group.

Experimental. Crystals (colorless prisms) of $C_9H_{11}NO_3$ [hereafter (1)] obtained from an ethyl acetate/hexane solution by Sean Esslinger and Professor Robert M. Williams (Colorado State University). Crystal dimensions $0·47 \times 0·60 \times 0·44$ mm. Nicolet $R3m$ diffractometer, unit-cell constants from least-squares fit of setting angles for 25 reflections ($2\theta_{av} = 31·40°$). Data collected ($\theta/2\theta$ scans) to $(\sin\theta)/\lambda = 0·5947$ Å^{-1}, $-11 \leq h \leq 11, 0 \leq k \leq 10$, $0 \leq l \leq 15$. Three standard reflections (400, 020, 005) measured every 97 reflections, no trend in intensity observed; Lorentz–polarization corrections;

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no absorption correction applied due to low absorption coefficient; 1623 unique reflections, 1365 reflections with $F_o > 4·0\sigma(F_o)$ observed.

(1)

Structure solved by direct methods (*SOLV*; Sheldrick, 1983); block-diagonal (max. 103 parameters/block, 125 parameters total, data/parameters = 10·9) weighted $\{w = [\sigma^2(F) + gF^2]^{-1}, \quad g = 1·37 \times 10^{-3}\}$ least-squares refinement on F. H atoms in idealized positions [C—H = 0·96 Å, $U(H) = 1·2 \times U_{iso}(C)$] with the exception of the H atom on N(1) (located in difference map and refined with isotropic thermal parameters). Non-H atoms refined with anisotropic thermal parameters. At convergence $[(\Delta/\sigma)_{max} =$