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3-Benzoyl-1-tosylpyrrole

By Mark S. Erickson, Frank R. Fronczek and Mark L. McLaughlin*

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

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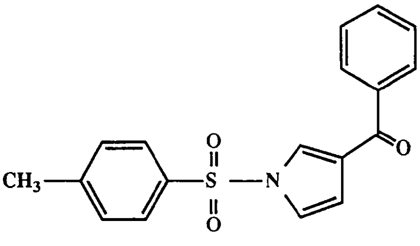
Abstract. $C_{18}H_{15}NO_3S$, $M_r = 325{\cdot}4$, monoclinic, $P2_1/c$, $a = 13{\cdot}5365$ (10), $b = 10{\cdot}1835$ (9), $c = 12{\cdot}5382$ (8) Å, $\beta = 111{\cdot}609$ (6)°, $V = 1606{\cdot}9$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}345$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 18{\cdot}7$ cm^{-1}, $F(000) = 680$, $T = 296$ K, $R = 0.036$ for 3022 observed data (of 3298 unique data). The pyrrole nitrogen is trigonal, lying only 0·014 (1) Å from the plane defined by the three atoms bonded to it. The pyrrole ring atoms have an average deviation of 0·0039 (12) Å and a maximum deviation of 0·0061 (11) Å from the plane of the ring. The pyrrole plane forms a dihedral angle of 51·63 (6)° with the benzoyl phenyl ring. The S—N distance is 1·6866 (9) Å, while the S—O distances are 1·419 (1) and 1·426 (1) Å.

Experimental. The title compound (1) was prepared by Friedel–Crafts acylation of 1-*p*-tolylsulfonylpyrrole with benzoyl chloride and anhydrous $AlCl_3$ in 1,2-dichloroethane at 298 K and recrystallized by slow cooling from ethyl acetate to yield colorless prisms, m.p. 385·5–386·5 K (Kakushima, Hamel, Frenette & Rokach, 1983).



(1)

Crystal 0·30 × 0·42 × 0·50 mm, mounted in a random orientation on an Enraf–Nonius CAD-4 diffractometer. Cell dimensions were determined at 296 K by least-squares fit to setting angles of 25 reflections having $32 > \theta > 25°$. A hemisphere of data having $2 < \theta < 75°$, $-16 \leq h \leq 16$, $0 \leq k \leq 12$, $-15 \leq l \leq 15$ was measured using graphite-monochromated Cu $K\alpha$ radiation; ω–2θ scans at speeds ranging from 0·47 to 3·30° min^{-1} to measure all significant data with approximately equal precision. Three standard reflections (200, 040, 008) exhibited only a random fluctuation in intensity, so no correction for decay was applied; data reduction included background, Lorentz and polarization corrections; absorption corrections were also applied, based on ψ scans, with minimum and maximum relative transmission coefficients of 89·72 and 94.91%. The two equivalent quadrants of data were averaged, $R_{\rm int} = 0{\cdot}018$ for 5055 data, 3298 unique.

Space group determined by systematic absences $h0l$ with l odd and $0k0$ with k odd. Structure solved by direct methods using *RANTAN* (Yao Jia-xing, 1981) and 3022 observed data $[I > 3\sigma(I)]$ refined by full-matrix least squares based upon F, $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz, 1980), scattering factors of Cromer & Waber (1974), anomalous-dispersion coefficients of Cromer (1974). Heavy-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF synthesis and refined with isotropic thermal parameters except methyl H atoms, which were included as fixed contributors with a C—H distance of 0·95 Å and $B = 1{\cdot}3\,B_{\rm eq}$ for bonded C atom. Final $R = 0{\cdot}036$ ($R = 0{\cdot}039$ for all 3298 data), $wR = 0{\cdot}058$, $S = 3{\cdot}165$ for 257 variables. Largest shift was $0{\cdot}06\sigma$ in the final cycle, maximum and minimum residual density 0·24 and −0·42 e Å^{-3}, extinction coefficient $g = 5{\cdot}07\,(12) \times 10^{-6}$, where the correction 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 unit-cell diagram. Selected distances, angles and torsion angles are presented in Table 2.†

Related literature. The crystal structure of 1-phenylsulfonylpyrrole was determined along with

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* To whom correspondence should be sent.

† Lists of H-atom coordinates and isotropic thermal parameters, anisotropic thermal parameters, bond angles, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54452 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Coordinates and equivalent isotropic thermal parameters*

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

	x	y	z	B_{eq} (Å^2)
S	0·87826 (2)	0·24443 (3)	0·91662 (3)	3·896 (6)
O1	0·44390 (7)	0·0046 (1)	0·62490 (8)	4·59 (2)
O2	0·86647 (7)	0·2204 (1)	1·02335 (8)	4·63 (2)
O3	0·96210 (6)	0·1877 (1)	0·88998 (9)	5·41 (2)
N	0·76575 (7)	0·1854 (1)	0·81671 (9)	3·84 (2)
C1	0·67269 (9)	0·1613 (1)	0·8334 (1)	3·63 (2)
C2	0·59974 (9)	0·1160 (1)	0·7319 (1)	3·55 (2)
C3	0·6509 (1)	0·1136 (1)	0·6503 (1)	4·10 (3)
C4	0·75162 (9)	0·1574 (1)	0·7036 (1)	4·21 (2)
C5	0·49256 (9)	0·0671 (1)	0·71195 (9)	3·47 (2)
C6	0·44499 (8)	0·0891 (1)	0·8005 (1)	3·44 (2)
C7	0·38845 (9)	−0·0135 (1)	0·8245 (1)	3·94 (2)
C8	0·3417 (1)	0·0023 (2)	0·9040 (1)	5·00 (3)
C9	0·3503 (1)	0·1210 (2)	0·9606 (1)	5·61 (3)
C10	0·4043 (1)	0·2240 (2)	0·9362 (1)	5·27 (3)
C11	0·45217 (9)	0·2086 (1)	0·8561 (1)	4·33 (3)
C12	0·87343 (8)	0·4134 (1)	0·8900 (1)	3·91 (2)
C13	0·8262 (1)	0·4957 (2)	0·9446 (1)	4·83 (3)
C14	0·8210 (1)	0·6285 (2)	0·9206 (2)	5·91 (4)
C15	0·8617 (1)	0·6798 (2)	0·8434 (2)	5·74 (4)
C16	0·9093 (1)	0·5963 (2)	0·7922 (2)	6·31 (4)
C17	0·9157 (1)	0·4627 (2)	0·8127 (1)	5·38 (3)
C18	0·8534 (1)	0·8255 (2)	0·8189 (2)	8·40 (6)

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

S—O2	1·426 (1)	C6—C11	1·387 (2)
S—O3	1·419 (1)	C7—C8	1·373 (2)
S—N	1·6866 (9)	C8—C9	1·385 (2)
S—C12	1·749 (1)	C9—C10	1·375 (3)
O1—C5	1·225 (1)	C10—C11	1·390 (3)
N—C1	1·373 (2)	C12—C13	1·379 (2)
N—C4	1·389 (2)	C12—C17	1·388 (2)
C1—C2	1·372 (1)	C13—C14	1·382 (2)
C2—C3	1·432 (2)	C14—C15	1·382 (3)
C2—C5	1·465 (2)	C15—C16	1·361 (3)
C3—C4	1·354 (2)	C15—C18	1·510 (3)
C5—C6	1·492 (2)	C16—C17	1·381 (3)
C6—C7	1·392 (2)		
O2—S—O3	121·78 (6)	C5—C6—C7	118·0 (1)
O2—S—N	104·67 (6)	C5—C6—C11	122·4 (1)
O2—S—C12	109·78 (7)	O3—S—N	105·18 (6)
O3—S—C12	109·44 (7)	N—S—C12	104·40 (5)
S—N—C1	125·36 (9)	S—N—C4	125·35 (9)
S—C12—C13	119·7 (1)	S—C12—C17	119·5 (1)
C1—C2—C5	127·0 (1)	C3—C2—C5	125·5 (1)
C1—N—C4	109·25 (9)		
S—N—C1—C2	179·2 (1)	C1—C2—C5—C6	12·7 (2)
S—N—C4—C3	−179·3 (1)	C3—C2—C5—C6	−173·4 (1)
N—C1—C2—C5	174·4 (1)	C2—C5—C6—C7	−138·4 (1)
C5—C2—C3—C4	−175·2 (1)	N—S—C12—C13	90·4 (1)

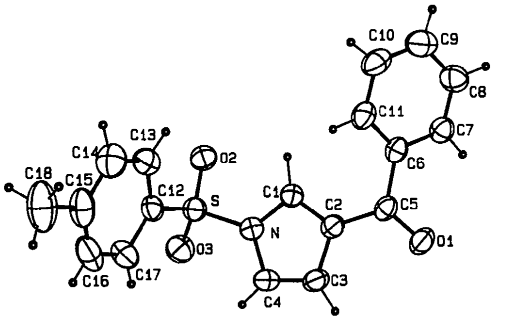

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the title molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as spheres of arbitrary radius.

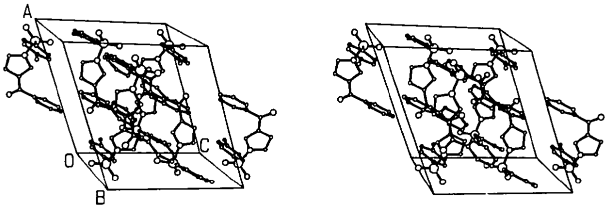

Fig. 2. *PLUTO* drawing (Motherwell & Clegg, 1978) illustrating the molecular packing, viewed slightly oblique to the *b* axis.

other substituted pyrroles and indoles (Beddoes, Dalton, Joule, Mills, Street & Watt, 1986). The structure of the compound (1) is similar to that of 1-phenylsulfonylpyrrole, with both compounds having nearly planar coordination about the pyrrole N atom. 1-Phenylsulfonylpyrrole has an N bond angle sum of 359·3°. The endocyclic N bond angle for 1-phenylsulfonylpyrrole and 3-benzoyl-1-tosylpyrrole is 112·6 and 109·25 (9)°, respectively.

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