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1-(1-Tosyl-1*H*-pyrrol-2-yl)ethanone

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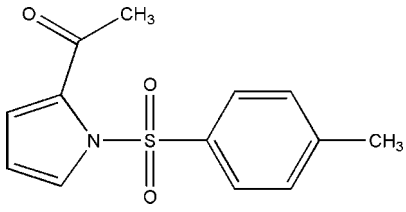
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.053; wR factor = 0.190; data-to-parameter ratio = 15.4.

In the molecule of the title compound, $C_{13}H_{13}NO_3S$, the dihedral angle between the planar benzene and pyrrole rings is 101.7 (2)°. In the crystal structure, intermolecular C—H···O hydrogen bonds link the molecules into chains.

Related literature

For general backgroud, see: Tonzola *et al.* (2003); Allen *et al.* (1987). For related literature, see: Zhu *et al.* (1999); Hou *et al.* (2004).



Experimental

Crystal data

$C_{13}H_{13}NO_3S$
M_r = 263.30
Monoclinic, $P2_1/n$
a = 8.0123 (7) Å
b = 13.3199 (15) Å
c = 12.5594 (12) Å
β = 105.15 (3)°
V = 1293.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 298 (2) K
0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.907, T_{max} = 0.952
2700 measured reflections
2518 independent reflections
1845 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.190
S = 1.06
2518 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O3^{i}	0.93	2.54	3.468 (4)	173

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2274).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2000). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Enraf–Nonius (1985). *CAD-4 Software* (Version 5.0). Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Hou, S. F., Huang, B. G., He, Y. C., Wang, J. & Tang, R. M. (2004). *Fine Chem.* **21**, 795–797.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tonzola, C. J., Alam, M. M., Kaminsky, W. & Jenekhe, S. A. (2003). *J. Am. Chem. Soc.* **125**, 13548–13558.
Zhu, H. Y., Liu, F. C. & He, H. P. (1999). *Appl. Chem.* **16**, 71–72.

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supplementary materials

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1-(1-Tosyl-1*H*-pyrrol-2-yl)ethanone

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Comment

The title compound, (I), is an intermediate of pyrrole derivatives, which widely exists in alkaloids and proteins in nature (Zhu *et al.*, 1999), and of which, acetylpyrroles have potential use for electronic transport compounds containing anthrazoline unit by a reaction with diamine compounds, according to the literature reported recently (Tonzola *et al.*, 2003). We here report its crystal structure, which is of interest to us in the field of electronic transport materials.

In the molecule of the title compound, (I), (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The rings A (C2—C7) and B (N/C8—C11) are, of course, and the dihedral angle between them is A/B = 101.7 (2)°.

In the crystal structure, intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into chains, in which they may be effective in the stabilization of the structure.

Experimental

The title compound, (I) was prepared by the literature method with a minor change (Hou *et al.*, 1968). The crystals were obtained by dissolving (I) (1.5 g, 5.7 mmole) in a mixed solvent (48 ml) (chloroform/hexane, 1:5, *v*/*v*) and evaporating the solvent slowly at room temperature for about 7 d (m.p. 383 K).

Refinement

H atoms were positioned geometrically, with C—H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.2$ for aromatic H and $x = 1.5$ for methy H atoms.

Figures

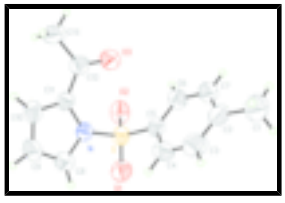

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

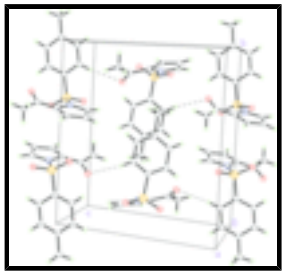

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines

1-(1-Tosyl-1*H*-pyrrol-2-yl)ethanone

Crystal data

$C_{13}H_{13}NO_3S$	F_{000} = 552
M_r = 263.30	D_x = 1.352 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 383 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.0123 (7) Å	Cell parameters from 25 reflections
b = 13.3199 (15) Å	θ = 10–13º
c = 12.5594 (12) Å	μ = 0.25 mm^{-1}
β = 105.15 (3)º	T = 298 (2) K
V = 1293.8 (3) Å^3	Plate, colorless
Z = 4	0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.068
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 2.3º
T = 298(2) K	h = 0→9
ω/2θ scans	k = 0→16
Absorption correction: ψ scan (North *et al.*, 1968)	l = −15→14
T_{min} = 0.907, T_{max} = 0.952	3 standard reflections
2700 measured reflections	every 120 min
2518 independent reflections	intensity decay: none
1845 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.053	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.8P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.190	$(\Delta/\sigma)_{max} < 0.001$
S = 1.06	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
2518 reflections	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
164 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.041 (6)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.77713 (10)	0.18073 (7)	0.47420 (7)	0.0514 (3)
O1	0.9324 (3)	0.1618 (2)	0.5581 (2)	0.0711 (8)
O2	0.7475 (4)	0.13029 (19)	0.3715 (2)	0.0671 (8)
O3	0.4118 (4)	0.2119 (2)	0.3186 (2)	0.0798 (9)
N	0.6188 (3)	0.1493 (2)	0.5354 (2)	0.0461 (7)
C1	0.7224 (7)	0.6309 (3)	0.4173 (4)	0.0895 (15)
H1B	0.6951	0.6480	0.3404	0.134*
H1C	0.8315	0.6601	0.4546	0.134*
H1D	0.6338	0.6563	0.4488	0.134*
C2	0.7328 (5)	0.5177 (3)	0.4299 (3)	0.0617 (9)
C3	0.7721 (6)	0.4739 (3)	0.5338 (3)	0.0764 (12)
H3A	0.7889	0.5148	0.5957	0.092*
C4	0.7867 (5)	0.3715 (3)	0.5478 (3)	0.0651 (10)
H4A	0.8173	0.3435	0.6180	0.078*
C5	0.7548 (4)	0.3109 (2)	0.4547 (3)	0.0474 (8)
C6	0.7114 (5)	0.3523 (3)	0.3507 (3)	0.0542 (8)
H6A	0.6892	0.3110	0.2889	0.065*
C7	0.7010 (5)	0.4558 (3)	0.3384 (3)	0.0602 (9)
H7A	0.6725	0.4837	0.2681	0.072*
C8	0.6564 (5)	0.1250 (3)	0.6460 (3)	0.0594 (9)
H8A	0.7646	0.1291	0.6961	0.071*
C9	0.5095 (5)	0.0943 (3)	0.6688 (3)	0.0669 (10)
H9A	0.4994	0.0726	0.7372	0.080*
C10	0.3754 (5)	0.1005 (3)	0.5734 (3)	0.0559 (9)
H10A	0.2602	0.0847	0.5671	0.067*
C11	0.4420 (4)	0.1338 (2)	0.4903 (3)	0.0439 (7)
C12	0.3477 (4)	0.1621 (3)	0.3791 (3)	0.0535 (8)
C13	0.1630 (5)	0.1285 (4)	0.3391 (4)	0.0811 (13)
H13A	0.1156	0.1519	0.2650	0.122*
H13B	0.0972	0.1556	0.3862	0.122*
H13C	0.1581	0.0565	0.3404	0.122*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0462 (5)	0.0525 (5)	0.0606 (6)	0.0007 (4)	0.0232 (4)	0.0017 (4)
O1	0.0451 (14)	0.0790 (18)	0.090 (2)	0.0079 (12)	0.0182 (13)	0.0135 (15)
O2	0.0844 (19)	0.0576 (15)	0.0723 (17)	−0.0025 (13)	0.0434 (15)	−0.0103 (13)
O3	0.0692 (18)	0.106 (2)	0.0597 (16)	−0.0163 (16)	0.0082 (13)	0.0214 (16)
N	0.0429 (15)	0.0503 (15)	0.0468 (15)	−0.0030 (11)	0.0146 (12)	0.0036 (12)
C1	0.094 (3)	0.051 (2)	0.112 (4)	0.000 (2)	0.008 (3)	−0.003 (2)
C2	0.053 (2)	0.057 (2)	0.071 (2)	−0.0067 (16)	0.0079 (17)	−0.0059 (18)
C3	0.093 (3)	0.064 (2)	0.063 (2)	−0.007 (2)	0.004 (2)	−0.016 (2)
C4	0.079 (3)	0.065 (2)	0.0446 (19)	−0.0059 (19)	0.0052 (18)	0.0025 (17)
C5	0.0417 (16)	0.0511 (18)	0.0515 (18)	−0.0057 (14)	0.0161 (14)	0.0011 (15)
C6	0.060 (2)	0.059 (2)	0.0482 (18)	−0.0036 (16)	0.0225 (16)	−0.0015 (15)
C7	0.062 (2)	0.062 (2)	0.057 (2)	−0.0026 (17)	0.0168 (17)	0.0093 (17)
C8	0.057 (2)	0.069 (2)	0.0496 (19)	−0.0028 (17)	0.0098 (16)	0.0070 (17)
C9	0.073 (2)	0.082 (3)	0.050 (2)	−0.004 (2)	0.0243 (18)	0.0155 (19)
C10	0.0510 (19)	0.059 (2)	0.065 (2)	0.0000 (16)	0.0270 (17)	0.0059 (17)
C11	0.0408 (16)	0.0443 (16)	0.0480 (17)	0.0006 (13)	0.0141 (14)	−0.0007 (13)
C12	0.0494 (19)	0.059 (2)	0.0523 (19)	−0.0003 (15)	0.0132 (15)	−0.0039 (16)
C13	0.050 (2)	0.115 (4)	0.071 (3)	−0.002 (2)	0.0007 (19)	0.009 (3)

Geometric parameters (Å, °)

S—O2	1.418 (3)	C4—H4A	0.9300
S—O1	1.427 (3)	C5—C6	1.376 (5)
S—N	1.699 (3)	C6—C7	1.387 (5)
S—C5	1.754 (3)	C6—H6A	0.9300
O3—C12	1.220 (4)	C7—H7A	0.9300
N—C8	1.381 (4)	C8—C9	1.346 (5)
N—C11	1.396 (4)	C8—H8A	0.9300
C1—C2	1.515 (6)	C9—C10	1.387 (5)
C1—H1B	0.9600	C9—H9A	0.9300
C1—H1C	0.9600	C10—C11	1.363 (5)
C1—H1D	0.9600	C10—H10A	0.9300
C2—C7	1.384 (5)	C11—C12	1.453 (5)
C2—C3	1.389 (6)	C12—C13	1.501 (5)
C3—C4	1.376 (6)	C13—H13A	0.9600
C3—H3A	0.9300	C13—H13B	0.9600
C4—C5	1.387 (5)	C13—H13C	0.9600
O2—S—O1	119.63 (18)	C5—C6—C7	119.8 (3)
O2—S—N	109.10 (15)	C5—C6—H6A	120.1
O1—S—N	103.43 (15)	C7—C6—H6A	120.1
O2—S—C5	110.79 (16)	C2—C7—C6	120.5 (3)
O1—S—C5	108.38 (17)	C2—C7—H7A	119.8
N—S—C5	104.23 (14)	C6—C7—H7A	119.8
C8—N—C11	107.7 (3)	C9—C8—N	108.2 (3)

C8—N—S	121.4 (2)	C9—C8—H8A	125.9
C11—N—S	130.7 (2)	N—C8—H8A	125.9
C2—C1—H1B	109.5	C8—C9—C10	108.7 (3)
C2—C1—H1C	109.5	C8—C9—H9A	125.6
H1B—C1—H1C	109.5	C10—C9—H9A	125.6
C2—C1—H1D	109.5	C11—C10—C9	108.1 (3)
H1B—C1—H1D	109.5	C11—C10—H10A	125.9
H1C—C1—H1D	109.5	C9—C10—H10A	125.9
C7—C2—C3	118.5 (4)	C10—C11—N	107.2 (3)
C7—C2—C1	120.8 (4)	C10—C11—C12	127.6 (3)
C3—C2—C1	120.7 (4)	N—C11—C12	124.6 (3)
C4—C3—C2	121.8 (4)	O3—C12—C11	122.8 (3)
C4—C3—H3A	119.1	O3—C12—C13	119.5 (3)
C2—C3—H3A	119.1	C11—C12—C13	117.6 (3)
C3—C4—C5	118.6 (3)	C12—C13—H13A	109.5
C3—C4—H4A	120.7	C12—C13—H13B	109.5
C5—C4—H4A	120.7	H13A—C13—H13B	109.5
C6—C5—C4	120.8 (3)	C12—C13—H13C	109.5
C6—C5—S	121.3 (3)	H13A—C13—H13C	109.5
C4—C5—S	117.9 (3)	H13B—C13—H13C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4A···O3^{i}	0.93	2.54	3.468 (4)	173

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

Fig. 1

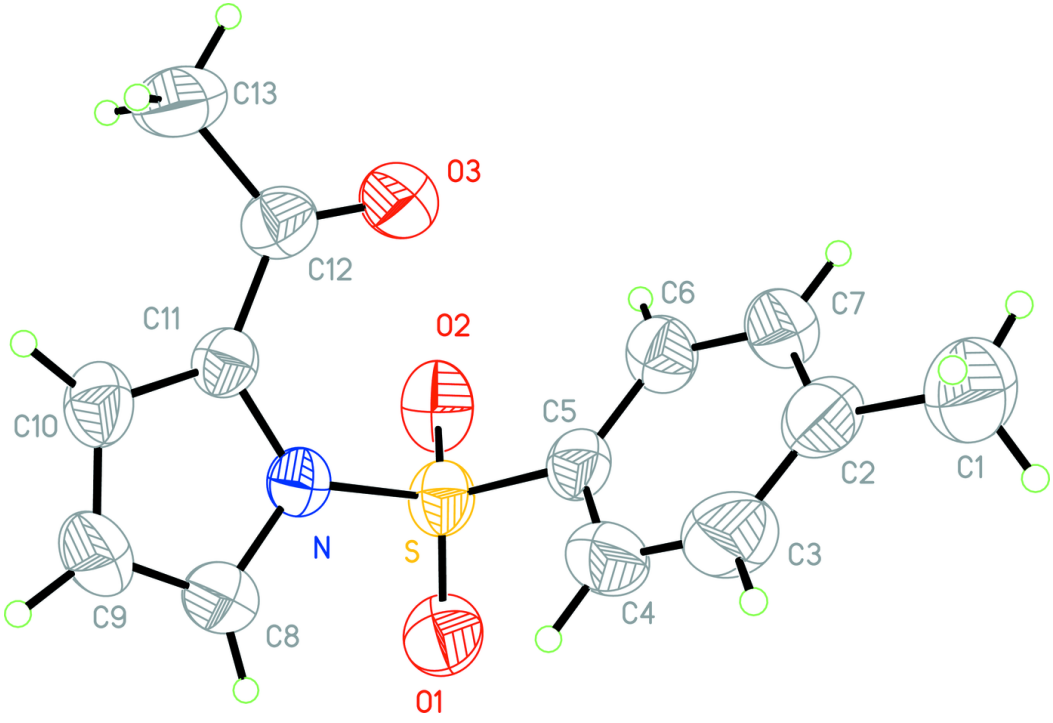

Fig. 2