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(*E*)-1-(Pyridin-4-yl)propan-1-one *O*-tosyl oxime

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Keywords: crystal structure; pyridine; tosyl oxime; hydrogen bonding.

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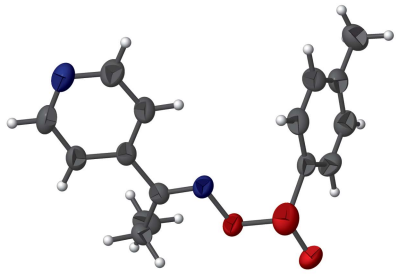
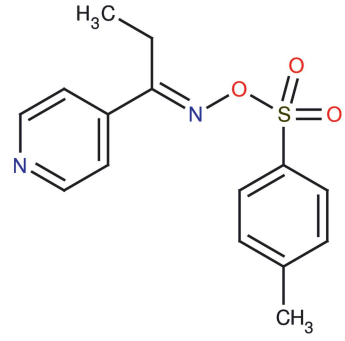
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{15}H_{16}N_2O_3S$, was obtained by the reaction of (*E*)-1-(pyridin-4-yl)propan-1-one oxime and *para*-toluenesulfonic acid. The pyridine ring makes a dihedral angle of 54.70 (10)° with the benzene ring. In the crystal, molecules are linked by C—H···O hydrogen bonds, forming a chain along the *c*-axis direction.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) was synthesized by the reaction of (*E*)-1-(pyridin-4-yl)propan-1-one oxime and *para*-toluenesulfonyl chloride. For the crystal structure of the starting material, see Eitel *et al.* (2016). The pyridine ring makes dihedral angles of 54.70 (10) and 14.06 (17)° with the benzene ring and the oxime plane, respectively. The dihedral angle between the benzene ring and the oxime plane is 68.38 (17)°. The orientation of the benzene ring is stabilized by an intramolecular C—H···O contact (Table 1).

In the crystal, molecules are linked by C—H···O hydrogen bonds, forming a chain along the *c*-axis direction (Table 1, Fig. 2).

Synthesis and crystallization

para-Toluenesulfonyl chloride (6.76 g, 35.48 mmol) was added to a solution of (*E*)-4-propionylpyridine oxime (4.44 g, 29.56 mmol) in anhydrous pyridine (20 ml). After reaction for 21.5 h at 298 K, the solution was diluted with ice–water (100 ml) and stirred for a further 3 h. The resulting white solid was filtered off, washed with cold water and dried under vacuum (yield: 78%, 7.05 g). Crystals of the title compound suitable for

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O13^{i}	0.94	2.53	3.138 (3)	122
C3—H3···O13^{i}	0.94	2.58	3.171 (3)	122
C20—H20···O14	0.94	2.56	2.929 (3)	104

Symmetry code: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{16}N_2O_3S$
M_r	304.36
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	213
a, b, c (Å)	14.9385 (12), 10.6630 (7), 9.7701 (7)
β (°)	100.044 (6)
V (Å^3)	1532.4 (2)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.98
Crystal size (mm)	0.16 × 0.09 × 0.04
Data collection	
Diffractometer	STOE IPDS 2T
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2006)
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10273, 2695, 1913
R_{int}	0.049
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.599
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.098, 0.98
No. of reflections	2695
No. of parameters	192
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.36

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2006), *SIR2004* (Burla *et al.*, 2005) and *SHELXL2013* (Sheldrick, 2015).

X-ray determination were obtained by slow evaporation of a solution of the solid in methanol at 298 K.

^{1}H NMR (400 MHz, DMSO-d_6) δ 0.99 (t, ^3J = 7.6 Hz, 3H), 2.41 (s, 3H), 2.81 (q, ^3J = 7.6 Hz, 2H), 7.50 (d, ^3J = 8.1 Hz, 2H), 7.56 (d, ^3J = 5.8 Hz, 2H), 7.91 (d, ^3J = 8.3 Hz, 2H), 8.67 (d, ^3J = 5.8 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 10.6, 20.5, 21.1, 121.1, 128.5, 130.1, 131.6, 139.5, 145.7, 150.5, 167.5.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

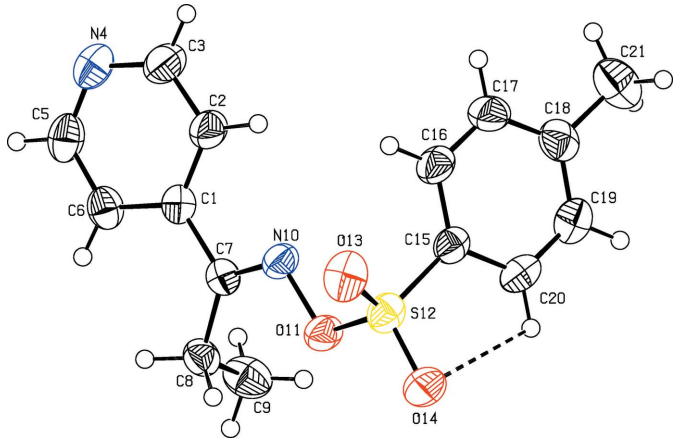


Figure 1
Molecular structure of the title compound with the atom labelling and displacement ellipsoids drawn at the 50% probability level. The intramolecular C—H···O contact is drawn with a dashed line.

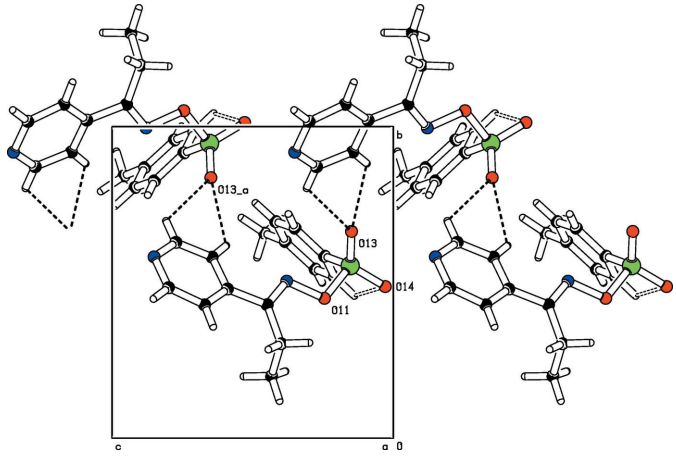


Figure 2
Partial packing diagram viewed along the *a* axis. Intramolecular C—H···O hydrogen bonds are shown with open dashed bonds. Intermolecular hydrogen bonds are indicated by dashed lines.

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full crystallographic data

IUCrData (2017). **2**, x171602 [https://doi.org/10.1107/S2414314617016029]

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Crystal data

$C_{15}H_{16}N_2O_3S$
M_r = 304.36
Monoclinic, $P2_1/c$
a = 14.9385 (12) Å
b = 10.6630 (7) Å
c = 9.7701 (7) Å
β = 100.044 (6)°
V = 1532.4 (2) Å^3
Z = 4
$F(000)$ = 640
D_x = 1.319 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 11862 reflections
θ = 3.0–67.8°
μ = 1.98 mm^{-1}
T = 213 K
Plate, colourless
0.16 × 0.09 × 0.04 mm

Data collection

STOE IPDS 2T diffractometer
Radiation source: Incoatec microSource Cu
X-ray mirror monochromator
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration (X-RED32; Stoe & Cie, 2006)
10273 measured reflections
2695 independent reflections
1913 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 67.5°, θ_{min} = 3.0°
h = −16→17
k = −12→11
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.098
S = 0.98
2695 reflections
192 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.89270 (13)	0.47865 (18)	0.5899 (2)	0.0378 (4)
C2	0.85364 (15)	0.5811 (2)	0.6446 (2)	0.0508 (6)
H2	0.8009	0.6182	0.5945	0.061*
C3	0.89270 (16)	0.6283 (2)	0.7731 (2)	0.0564 (6)
H3	0.8646	0.6973	0.8080	0.068*
N4	0.96781 (13)	0.58192 (18)	0.85057 (19)	0.0532 (5)
C5	1.00375 (15)	0.4829 (2)	0.7992 (2)	0.0523 (6)
H5	1.0559	0.4470	0.8527	0.063*
C6	0.96963 (14)	0.4280 (2)	0.6714 (2)	0.0446 (5)
H6	0.9984	0.3576	0.6407	0.054*
C7	0.85434 (12)	0.42844 (18)	0.4487 (2)	0.0370 (4)
C8	0.88195 (13)	0.30317 (18)	0.3995 (2)	0.0427 (5)
H8A	0.9445	0.2846	0.4442	0.051*
H8B	0.8802	0.3062	0.2989	0.051*
C9	0.81924 (17)	0.1994 (2)	0.4332 (3)	0.0588 (6)
H9A	0.8192	0.1982	0.5325	0.088*
H9B	0.8406	0.1192	0.4048	0.088*
H9C	0.7580	0.2146	0.3839	0.088*
N10	0.79590 (11)	0.50387 (16)	0.37916 (16)	0.0414 (4)
O11	0.75806 (9)	0.44884 (12)	0.24519 (13)	0.0446 (4)
S12	0.69674 (4)	0.55354 (5)	0.15352 (5)	0.04410 (17)
O13	0.74961 (11)	0.66367 (15)	0.14888 (17)	0.0594 (4)
O14	0.66291 (11)	0.48672 (16)	0.02890 (14)	0.0575 (4)
C15	0.60796 (14)	0.58428 (18)	0.2447 (2)	0.0397 (5)
C16	0.61762 (15)	0.67382 (19)	0.3494 (2)	0.0487 (6)
H16	0.6721	0.7191	0.3730	0.058*
C17	0.54569 (16)	0.6953 (2)	0.4183 (2)	0.0533 (6)
H17	0.5523	0.7549	0.4902	0.064*
C18	0.46367 (15)	0.6310 (2)	0.3839 (2)	0.0499 (5)
C19	0.45625 (16)	0.5429 (2)	0.2783 (2)	0.0534 (6)
H19	0.4013	0.4988	0.2531	0.064*
C20	0.52712 (15)	0.5181 (2)	0.2093 (2)	0.0491 (5)
H20	0.5209	0.4570	0.1390	0.059*
C21	0.38537 (18)	0.6580 (3)	0.4576 (3)	0.0722 (8)
H21A	0.4066	0.7077	0.5400	0.108*
H21B	0.3602	0.5797	0.4843	0.108*
H21C	0.3388	0.7041	0.3960	0.108*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0354 (10)	0.0375 (11)	0.0398 (10)	−0.0010 (8)	0.0050 (8)	0.0063 (8)
C2	0.0493 (12)	0.0450 (13)	0.0520 (13)	0.0080 (9)	−0.0081 (10)	−0.0068 (10)
C3	0.0612 (14)	0.0496 (14)	0.0524 (13)	0.0021 (11)	−0.0070 (11)	−0.0078 (10)
N4	0.0531 (11)	0.0562 (12)	0.0454 (10)	−0.0067 (9)	−0.0049 (9)	0.0039 (9)

C5	0.0438 (11)	0.0648 (16)	0.0445 (12)	−0.0008 (11)	−0.0029 (10)	0.0139 (11)
C6	0.0405 (11)	0.0498 (13)	0.0432 (11)	0.0056 (9)	0.0064 (9)	0.0107 (9)
C7	0.0348 (10)	0.0368 (11)	0.0396 (10)	0.0015 (8)	0.0073 (8)	0.0029 (8)
C8	0.0414 (11)	0.0405 (12)	0.0460 (12)	0.0071 (8)	0.0072 (9)	0.0013 (9)
C9	0.0687 (15)	0.0399 (13)	0.0725 (16)	0.0007 (11)	0.0256 (13)	0.0015 (11)
N10	0.0430 (9)	0.0414 (10)	0.0366 (9)	0.0012 (7)	−0.0021 (7)	−0.0042 (7)
O11	0.0497 (8)	0.0421 (8)	0.0380 (7)	0.0065 (6)	−0.0030 (6)	−0.0032 (6)
S12	0.0470 (3)	0.0438 (3)	0.0385 (3)	0.0016 (2)	−0.0009 (2)	0.0049 (2)
O13	0.0589 (10)	0.0530 (10)	0.0644 (10)	−0.0093 (7)	0.0053 (8)	0.0152 (8)
O14	0.0606 (9)	0.0725 (11)	0.0357 (8)	0.0066 (8)	−0.0019 (7)	−0.0085 (7)
C15	0.0439 (10)	0.0331 (11)	0.0382 (10)	0.0030 (8)	−0.0039 (8)	0.0026 (8)
C16	0.0495 (12)	0.0333 (11)	0.0575 (13)	0.0011 (9)	−0.0071 (10)	−0.0068 (10)
C17	0.0588 (13)	0.0425 (13)	0.0554 (14)	0.0104 (10)	0.0013 (11)	−0.0113 (10)
C18	0.0519 (12)	0.0478 (13)	0.0483 (12)	0.0140 (10)	0.0040 (10)	0.0084 (10)
C19	0.0456 (12)	0.0599 (15)	0.0522 (13)	−0.0089 (10)	0.0017 (10)	0.0020 (11)
C20	0.0541 (13)	0.0480 (13)	0.0419 (11)	−0.0076 (10)	−0.0012 (10)	−0.0076 (9)
C21	0.0684 (17)	0.0771 (19)	0.0743 (18)	0.0219 (14)	0.0213 (14)	0.0099 (14)

Geometric parameters (Å, º)

C1—C6	1.388 (3)	N10—O11	1.4557 (19)
C1—C2	1.388 (3)	O11—S12	1.6129 (13)
C1—C7	1.497 (3)	S12—O13	1.4204 (16)
C2—C3	1.384 (3)	S12—O14	1.4250 (15)
C2—H2	0.9400	S12—C15	1.752 (2)
C3—N4	1.334 (3)	C15—C16	1.388 (3)
C3—H3	0.9400	C15—C20	1.389 (3)
N4—C5	1.323 (3)	C16—C17	1.383 (3)
C5—C6	1.392 (3)	C16—H16	0.9400
C5—H5	0.9400	C17—C18	1.393 (3)
C6—H6	0.9400	C17—H17	0.9400
C7—N10	1.290 (2)	C18—C19	1.386 (3)
C7—C8	1.501 (3)	C18—C21	1.504 (3)
C8—C9	1.522 (3)	C19—C20	1.376 (3)
C8—H8A	0.9800	C19—H19	0.9400
C8—H8B	0.9800	C20—H20	0.9400
C9—H9A	0.9700	C21—H21A	0.9700
C9—H9B	0.9700	C21—H21B	0.9700
C9—H9C	0.9700	C21—H21C	0.9700
C6—C1—C2	116.52 (18)	N10—O11—S12	108.31 (11)
C6—C1—C7	122.35 (19)	O13—S12—O14	120.16 (10)
C2—C1—C7	121.12 (16)	O13—S12—O11	108.96 (9)
C3—C2—C1	119.8 (2)	O14—S12—O11	102.17 (9)
C3—C2—H2	120.1	O13—S12—C15	109.62 (10)
C1—C2—H2	120.1	O14—S12—C15	109.92 (9)
N4—C3—C2	124.0 (2)	O11—S12—C15	104.72 (8)
N4—C3—H3	118.0	C16—C15—C20	120.4 (2)

C2—C3—H3	118.0	C16—C15—S12	120.83 (16)
C5—N4—C3	115.96 (19)	C20—C15—S12	118.73 (16)
N4—C5—C6	124.5 (2)	C17—C16—C15	118.9 (2)
N4—C5—H5	117.8	C17—C16—H16	120.6
C6—C5—H5	117.8	C15—C16—H16	120.6
C1—C6—C5	119.2 (2)	C16—C17—C18	121.8 (2)
C1—C6—H6	120.4	C16—C17—H17	119.1
C5—C6—H6	120.4	C18—C17—H17	119.1
N10—C7—C1	112.18 (17)	C19—C18—C17	117.8 (2)
N10—C7—C8	125.77 (18)	C19—C18—C21	121.2 (2)
C1—C7—C8	122.04 (16)	C17—C18—C21	121.0 (2)
C7—C8—C9	111.33 (18)	C20—C19—C18	121.8 (2)
C7—C8—H8A	109.4	C20—C19—H19	119.1
C9—C8—H8A	109.4	C18—C19—H19	119.1
C7—C8—H8B	109.4	C19—C20—C15	119.4 (2)
C9—C8—H8B	109.4	C19—C20—H20	120.3
H8A—C8—H8B	108.0	C15—C20—H20	120.3
C8—C9—H9A	109.5	C18—C21—H21A	109.5
C8—C9—H9B	109.5	C18—C21—H21B	109.5
H9A—C9—H9B	109.5	H21A—C21—H21B	109.5
C8—C9—H9C	109.5	C18—C21—H21C	109.5
H9A—C9—H9C	109.5	H21A—C21—H21C	109.5
H9B—C9—H9C	109.5	H21B—C21—H21C	109.5
C7—N10—O11	110.08 (16)		

C6—C1—C2—C3	1.0 (3)	N10—O11—S12—O14	−178.64 (12)
C7—C1—C2—C3	−177.5 (2)	N10—O11—S12—C15	−64.00 (13)
C1—C2—C3—N4	0.5 (4)	O13—S12—C15—C16	−29.48 (19)
C2—C3—N4—C5	−1.7 (4)	O14—S12—C15—C16	−163.63 (16)
C3—N4—C5—C6	1.5 (3)	O11—S12—C15—C16	87.28 (17)
C2—C1—C6—C5	−1.3 (3)	O13—S12—C15—C20	149.92 (16)
C7—C1—C6—C5	177.30 (19)	O14—S12—C15—C20	15.77 (19)
N4—C5—C6—C1	0.0 (3)	O11—S12—C15—C20	−93.32 (16)
C6—C1—C7—N10	−166.03 (19)	C20—C15—C16—C17	0.4 (3)
C2—C1—C7—N10	12.5 (3)	S12—C15—C16—C17	179.80 (16)
C6—C1—C7—C8	14.8 (3)	C15—C16—C17—C18	−1.0 (3)
C2—C1—C7—C8	−166.7 (2)	C16—C17—C18—C19	0.6 (3)
N10—C7—C8—C9	−87.7 (3)	C16—C17—C18—C21	−178.4 (2)
C1—C7—C8—C9	91.4 (2)	C17—C18—C19—C20	0.4 (3)
C1—C7—N10—O11	−177.79 (15)	C21—C18—C19—C20	179.4 (2)
C8—C7—N10—O11	1.3 (3)	C18—C19—C20—C15	−1.0 (3)
C7—N10—O11—S12	−171.97 (13)	C16—C15—C20—C19	0.6 (3)
N10—O11—S12—O13	53.22 (14)	S12—C15—C20—C19	−178.83 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O13^{i}	0.94	2.53	3.138 (3)	122

C3—H3···O13^{i}	0.94	2.58	3.171 (3)	122
C20—H20···O14	0.94	2.56	2.929 (3)	104

Symmetry code: (i) x, $-y+3/2$, $z+1/2$.