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2-(2-Pyridylsulfanyl)-*N*-*p*-tolylacetamide

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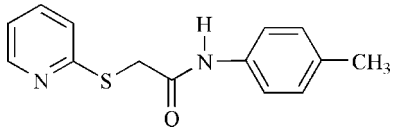
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.045; wR factor = 0.130; data-to-parameter ratio = 14.1.

The title compound, $C_{14}H_{14}N_2OS$, contains a pyridine and a benzene ring almost perpendicular to each other, subtending a dihedral angle of 88.2 (2)°. Intermolecular hydrogen bonds between adjacent NH groups and carbonyl O atoms link molecules together into chains parallel to [100], the shortest axis.

Related literature

For related literature, see: Koike *et al.* (1999).



Experimental

Crystal data

$C_{14}H_{14}N_2OS$
M_r = 258.33
Orthorhombic, *Pbca*
a = 9.348 (2) Å
b = 12.361 (3) Å
c = 23.184 (6) Å
V = 2678.9 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 294 (2) K
0.20 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.95, T_{max} = 0.97
12855 measured reflections
2366 independent reflections
1535 reflections with $I > 2\sigma(I)$
R_{int} = 0.066

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.130
S = 1.01
2366 reflections
168 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.89 (3)	2.09 (3)	2.904 (3)	152 (2)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2139).

References

Bruker (1997). *SMART* (Version 5.611), *SAINT* (Version 6.0), *SADABS* (Version 2.03) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Koike, K., Jia, Z., Nikaido, T., Liu, Y., Zhao, Y. & Guo, D. (1999). *Org. Lett.* **1**, 197–198.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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2-(2-Pyridylsulfanyl)-*N*-*p*-tolylacetamide

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Comment

2-Mercaptopyridine is an important class of medical intermediate. Recently, many biological copmpounds (Koike *et al.*,1999) have been prepared having 2-mercaptopyridine as raw material. We have synthesized the title compound, $C_{14}H_{14}N_2$ O S, (I), from 2-mercaptopyridine with *p*-tolylcarbamic chloride, and we are reporing herein its crystal structure.

The molecular structure of (I) contains a pyridinyl and a benzene rings, almost perpendicular to each other, subtending a dihedral angle of 91.8 (2)%. The methyl carbon is coplanar to the benzene ring (r.m.s deviation: 0.0075 (3) Å). Due to the π–π conjugation bwtween the S atom and pyridinyl ring, the S1—C5 bond distance [1.772 (3) Å] is slightly shorter than the S1—C6 one [1.801 (2) Å]. Intermolecular hydrogen bonds between adjacent N—H groups and carbonyl O atoms link molecules together into chains parallel to [100], the shortest axis direction.

Experimental

The title compound was synthesized by the reaction of pyridine-2-thiol with *p*-tolylcarbamic chloride in refluxing ethanol. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in chloroform/acetone.

Refinement

The H atom attached to N atom was located in a difference density map and the atomic coordinates allowed to refine freely. Other H atoms attached to carbon were positioned geometrically and refined as riding (C—H = 0.93–0.97%A). For the CH and CH_2 groups, U_{iso}(H) values were set equal to 1.2U_{eq}(C) and for the methyl groups they were set equal to 1.5U_{eq}(C).

Figures

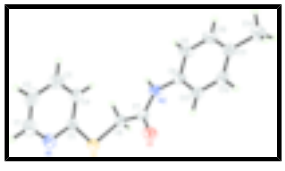

Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

2-(2-Pyridylsulfanyl)-*N*-*p*-tolylacetamide

Crystal data

$C_{14}H_{14}N_2OS$	D_x = 1.281 Mg m^{-3}
M_r = 258.33	Melting point: 403 K
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å

Hall symbol: -P 2ac 2ab	Cell parameters from 2537 reflections
a = 9.348 (2) Å	θ = 2.8–22.6º
b = 12.361 (3) Å	μ = 0.23 mm^{-1}
c = 23.184 (6) Å	T = 294 (2) K
V = 2678.9 (11) Å^3	Plate, colourless
Z = 8	0.20 × 0.18 × 0.14 mm
F_{000} = 1088	

Data collection

Bruker SMART CCD area-detector diffractometer	2366 independent reflections
Radiation source: fine-focus sealed tube	1535 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.066
T = 294(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −10→11
T_{min} = 0.95, T_{max} = 0.97	k = −14→14
12855 measured reflections	l = −27→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.130	$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.6597P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
2366 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
168 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
S1	0.15494 (7)	0.53279 (5)	0.32262 (3)	0.0510 (3)
O1	0.09884 (17)	0.36599 (16)	0.22875 (8)	0.0607 (6)
N1	0.1009 (3)	0.4497 (2)	0.42221 (10)	0.0619 (7)
N2	0.3332 (2)	0.33742 (17)	0.20810 (9)	0.0453 (5)
C1	0.1150 (4)	0.3798 (3)	0.46624 (14)	0.0786 (10)
H1	0.0559	0.3888	0.4981	0.094*
C2	0.2108 (4)	0.2963 (3)	0.46715 (14)	0.0789 (10)
H2	0.2156	0.2494	0.4984	0.095*
C3	0.3000 (4)	0.2838 (3)	0.42029 (13)	0.0691 (9)
H3	0.3672	0.2283	0.4198	0.083*
C4	0.2894 (3)	0.3536 (2)	0.37423 (12)	0.0538 (7)
H4	0.3489	0.3464	0.3423	0.065*
C5	0.1877 (3)	0.4347 (2)	0.37679 (11)	0.0439 (6)
C6	0.2722 (3)	0.4933 (2)	0.26470 (11)	0.0479 (7)
H6A	0.2788	0.5525	0.2374	0.057*
H6B	0.3671	0.4811	0.2803	0.057*
C7	0.2246 (2)	0.39226 (19)	0.23277 (10)	0.0407 (6)
C8	0.3274 (2)	0.24390 (19)	0.17239 (10)	0.0408 (6)
C9	0.4569 (3)	0.1969 (2)	0.15700 (12)	0.0554 (7)
H9	0.5421	0.2262	0.1705	0.067*
C10	0.4597 (3)	0.1067 (2)	0.12164 (13)	0.0625 (8)
H10	0.5475	0.0775	0.1110	0.075*
C11	0.3352 (3)	0.0589 (2)	0.10166 (12)	0.0517 (7)
C12	0.2078 (3)	0.1059 (2)	0.11785 (11)	0.0523 (7)
H12	0.1227	0.0754	0.1049	0.063*
C13	0.2016 (3)	0.1970 (2)	0.15273 (11)	0.0480 (6)
H13	0.1137	0.2265	0.1629	0.058*
C14	0.3383 (3)	−0.0406 (2)	0.06350 (15)	0.0742 (9)
H14A	0.2519	−0.0813	0.0689	0.111*
H14B	0.4191	−0.0846	0.0735	0.111*
H14C	0.3458	−0.0188	0.0239	0.111*
H2A	0.420 (3)	0.3602 (19)	0.2169 (10)	0.050 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0496 (4)	0.0530 (4)	0.0504 (4)	0.0121 (3)	0.0049 (3)	0.0018 (3)
O1	0.0300 (10)	0.0814 (14)	0.0706 (13)	−0.0041 (9)	0.0053 (9)	−0.0170 (10)
N1	0.0601 (14)	0.0777 (17)	0.0479 (14)	0.0104 (13)	0.0075 (12)	−0.0002 (12)
N2	0.0268 (11)	0.0523 (13)	0.0567 (14)	−0.0019 (10)	0.0002 (10)	−0.0017 (11)
C1	0.092 (2)	0.095 (3)	0.0492 (19)	0.004 (2)	0.0129 (17)	0.0102 (18)
C2	0.111 (3)	0.074 (2)	0.052 (2)	0.000 (2)	−0.005 (2)	0.0160 (17)
C3	0.086 (2)	0.0578 (18)	0.064 (2)	0.0111 (17)	−0.0137 (18)	−0.0023 (16)
C4	0.0583 (16)	0.0549 (16)	0.0481 (16)	0.0061 (14)	−0.0032 (13)	−0.0012 (13)

C5	0.0413 (14)	0.0496 (15)	0.0406 (14)	−0.0019 (12)	−0.0018 (11)	−0.0055 (12)
C6	0.0440 (15)	0.0504 (15)	0.0494 (16)	−0.0016 (12)	0.0052 (12)	0.0055 (12)
C7	0.0331 (13)	0.0514 (15)	0.0375 (14)	−0.0019 (11)	0.0016 (11)	0.0098 (11)
C8	0.0323 (13)	0.0427 (13)	0.0474 (15)	−0.0009 (11)	0.0024 (11)	0.0071 (12)
C9	0.0356 (14)	0.0513 (16)	0.079 (2)	−0.0003 (12)	−0.0004 (13)	−0.0028 (15)
C10	0.0449 (16)	0.0543 (17)	0.088 (2)	0.0103 (14)	0.0033 (15)	−0.0050 (16)
C11	0.0544 (17)	0.0455 (15)	0.0552 (17)	0.0018 (13)	−0.0038 (13)	0.0054 (13)
C12	0.0449 (15)	0.0626 (18)	0.0494 (16)	−0.0110 (13)	−0.0046 (12)	0.0009 (14)
C13	0.0339 (13)	0.0624 (17)	0.0477 (15)	0.0010 (12)	0.0011 (11)	0.0013 (14)
C14	0.081 (2)	0.0598 (19)	0.082 (2)	0.0022 (17)	−0.0014 (17)	−0.0118 (17)

Geometric parameters (Å, °)

S1—C5	1.772 (3)	C6—H6A	0.9700
S1—C6	1.801 (2)	C6—H6B	0.9700
O1—C7	1.224 (3)	C8—C13	1.388 (3)
N1—C5	1.342 (3)	C8—C9	1.389 (3)
N1—C1	1.344 (4)	C9—C10	1.384 (4)
N2—C7	1.347 (3)	C9—H9	0.9300
N2—C8	1.423 (3)	C10—C11	1.385 (4)
N2—H2A	0.89 (3)	C10—H10	0.9300
C1—C2	1.366 (5)	C11—C12	1.377 (4)
C1—H1	0.9300	C11—C14	1.515 (4)
C2—C3	1.378 (4)	C12—C13	1.388 (4)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.376 (4)	C13—H13	0.9300
C3—H3	0.9300	C14—H14A	0.9600
C4—C5	1.384 (3)	C14—H14B	0.9600
C4—H4	0.9300	C14—H14C	0.9600
C6—C7	1.518 (3)		
C5—S1—C6	103.77 (12)	O1—C7—C6	122.4 (2)
C5—N1—C1	116.6 (3)	N2—C7—C6	113.6 (2)
C7—N2—C8	128.8 (2)	C13—C8—C9	118.6 (2)
C7—N2—H2A	115.8 (16)	C13—C8—N2	124.2 (2)
C8—N2—H2A	115.2 (16)	C9—C8—N2	117.2 (2)
N1—C1—C2	124.2 (3)	C10—C9—C8	120.4 (2)
N1—C1—H1	117.9	C10—C9—H9	119.8
C2—C1—H1	117.9	C8—C9—H9	119.8
C1—C2—C3	118.0 (3)	C9—C10—C11	121.7 (2)
C1—C2—H2	121.0	C9—C10—H10	119.1
C3—C2—H2	121.0	C11—C10—H10	119.1
C4—C3—C2	119.8 (3)	C12—C11—C10	117.1 (2)
C4—C3—H3	120.1	C12—C11—C14	121.2 (2)
C2—C3—H3	120.1	C10—C11—C14	121.7 (2)
C3—C4—C5	118.1 (3)	C11—C12—C13	122.5 (2)
C3—C4—H4	121.0	C11—C12—H12	118.8
C5—C4—H4	121.0	C13—C12—H12	118.8
N1—C5—C4	123.3 (3)	C12—C13—C8	119.7 (2)
N1—C5—S1	110.94 (19)	C12—C13—H13	120.2

C4—C5—S1	125.7 (2)	C8—C13—H13	120.2
C7—C6—S1	114.11 (17)	C11—C14—H14A	109.5
C7—C6—H6A	108.7	C11—C14—H14B	109.5
S1—C6—H6A	108.7	H14A—C14—H14B	109.5
C7—C6—H6B	108.7	C11—C14—H14C	109.5
S1—C6—H6B	108.7	H14A—C14—H14C	109.5
H6A—C6—H6B	107.6	H14B—C14—H14C	109.5
O1—C7—N2	123.9 (2)		
C5—N1—C1—C2	0.0 (5)	S1—C6—C7—N2	−153.34 (18)
N1—C1—C2—C3	0.9 (6)	C7—N2—C8—C13	5.5 (4)
C1—C2—C3—C4	−0.8 (5)	C7—N2—C8—C9	−173.9 (2)
C2—C3—C4—C5	−0.1 (4)	C13—C8—C9—C10	1.6 (4)
C1—N1—C5—C4	−1.0 (4)	N2—C8—C9—C10	−179.0 (2)
C1—N1—C5—S1	179.7 (2)	C8—C9—C10—C11	−1.6 (4)
C3—C4—C5—N1	1.1 (4)	C9—C10—C11—C12	0.9 (4)
C3—C4—C5—S1	−179.8 (2)	C9—C10—C11—C14	−179.3 (3)
C6—S1—C5—N1	−176.38 (19)	C10—C11—C12—C13	−0.2 (4)
C6—S1—C5—C4	4.4 (3)	C14—C11—C12—C13	−180.0 (3)
C5—S1—C6—C7	72.68 (19)	C11—C12—C13—C8	0.1 (4)
C8—N2—C7—O1	2.0 (4)	C9—C8—C13—C12	−0.8 (4)
C8—N2—C7—C6	−175.9 (2)	N2—C8—C13—C12	179.8 (2)
S1—C6—C7—O1	28.7 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1^{i}	0.89 (3)	2.09 (3)	2.904 (3)	152 (2)

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

Fig. 1