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4-[5-(Benzylsulfanyl)-1,3,4-oxadiazol-2-yl]pyridine from a single-pot reaction

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.047
wR factor = 0.125
Data-to-parameter ratio = 22.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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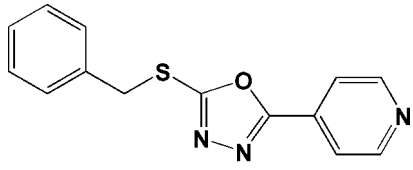
The asymmetric unit of the title compound, $C_{14}H_{11}N_3OS$, obtained from the one-pot reaction of isonicotinic acid hydrazide, CS_2 and benzyl chloride in the presence of triethylamine, contains two crystallographically independent molecules with similar geometry. The dihedral angles between the pyridine and 1,3,4-oxadiazole rings are 6.3 (1) and 7.0 (1)°, while those between the phenyl and oxadiazole rings are 69.61 (5) and 67.78 (6)°.

Comment

1,3,4-Oxadiazole derivatives are known to exhibit biological properties, which has given rise to a wide variety of applications, in particular as active compounds in both medicine and agriculture (Sharma & Tandon, 1984; Pachhamia & Parikh, 1988; Xu *et al.*, 2002). Our quest to obtain a suitable system for antitumour applications with novel coordination abilities has led us to 1,3,4-oxadiazole derivatives.



(I)

The title compound, (I), has been newly synthesized by the reaction of isonicotinic acid hydrazide, CS_2 and benzyl chloride, through isonicotinoyl-*S*-benzyldithiocarbazate. The base-catalysed cyclization of the intermediate isonicotinoyl-*S*-benzyldithiocarbazate by loss of H_2S leads to the formation of the 1,3,4-oxadiazole derivative in good yield.

The molecular structure of (I), together with the atom-labelling scheme, is shown in Fig. 1. The bond lengths and angles of the 1,3,4-oxadiazole rings (Table 1) are in good agreement with the values quoted in previous reports (Xu, Yu, Yin *et al.*, 2005; Xu, Yu, Xu & Li, 2005; Zhang *et al.* 2002). The asymmetric unit consists of two independent molecules of (I), labelled *A* and *B*. Both atom S1 and the pyridine ring lie almost in the plane of the 1,3,4-oxadiazole ring (C8/C9/N1/N2/O1). The dihedral angles between the pyridine and 1,3,4-oxadiazole rings are 6.3 (1) and 7.0 (1)°, while those between the phenyl and oxadiazole rings are 69.61 (5) and 67.78 (6)°, for molecules *A* and *B*, respectively.

Experimental

The title compound was synthesized by the reaction of CS_2 (1.5 ml, 0.02 mol) with a suspension of isonicotinic acid hydrazide (2.7 g, 0.019 mol) in methanol (20 ml) in the presence of triethylamine (2 ml,

0.014 mol). Benzyl chloride (1.5 ml, 0.01 mol) was added dropwise to the above clear solution, which was stirred continuously for 2 h at room temperature·The product obtained on evaporation of the solvent was filtered off, washed twice with portions of carbon tetrachloride and subsequently with water, and finally dried *in vacuo*. Colourless single crystals of (I) (m.p. 388 K) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution over a period of 8 d (yield 1.65 g, 68%). Spectroscopic anlysis: IR (KBr, ν, cm^{-1}): 3101 (–NH), 1639 (C═N), 853 (–C—S); ^{1}H NMR (DMSO-d_6, TMS, δ, p.p.m.): 7.55 (*m*, 5 H, aromatic), 8.23–9.25 (*m*, 4 H, pyridine), 4.12 (*s*, 2 H, methylene); ^{13}C NMR (DMSO-d_6, TMS, δ, p.p.m.): 139.24 (C1*A*), 129.16 (C2*A*, C6*A*), 130.29 (C3*A*, C5*A*), 121.69 (C4*A*), 40.38 (C7*A*), 177.87 (C8*A*), 164.20 (C9*A*), 158.97 (C10*A*), 121.69 (C11*A*), 150.41 (C12*A*), 150.04 (C13*A*), 119.89 (C14*A*).

Crystal data

$C_{14}H_{11}N_3OS$
M_r = 269.32
Monoclinic, $P2_1/c$
a = 11.2294 (6) Å
b = 7.5234 (4) Å
c = 31.1642 (15) Å
β = 92.824 (1)°
V = 2629.7 (2) Å^3
Z = 8
D_x = 1.361 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 298 (2) K
Needle, colourless
0.53 × 0.23 × 0.18 mm

Data collection

Bruker SMART APEX-II CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.883, T_{max} = 0.958
25340 measured reflections
7646 independent reflections
4375 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 30.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.125
S = 1.02
7646 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.2457P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1*A*—C8*A*	1.7259 (16)	N1*A*—C8*A*	1.286 (2)
S1*A*—C7*A*	1.8187 (17)	N1*A*—N2*A*	1.4082 (19)
O1*A*—C8*A*	1.3603 (19)	N2*A*—C9*A*	1.284 (2)
O1*A*—C9*A*	1.3688 (17)		
C8*A*—S1*A*—C7*A*	97.83 (8)	C8*A*—O1*A*—C9*A*	102.13 (12)

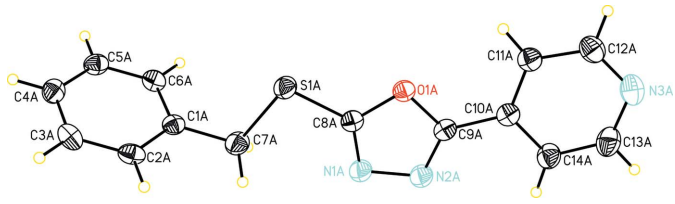


Figure 1
The molecular structure of (I), with the atom-numbering scheme (molecule *A* only). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

All H atoms were initially located in a difference Fourier map. They were then placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2006). *APEX2*. Version 2.0-2. Bruker AXS Inc., Madison, Wisconsin, USA.
Pachhamia, V. L. & Parikh, A. R. (1988). *J. Indian Chem. Soc.* **65**, 357–361.
Sharma, B. L. & Tandon, S. K. (1984). *Pharmazie*, **39**, 858–859.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Xu, L. Z., Jiao, K., Zhang, S. S. & Kuang, S. P. (2002). *Bull. Korean Chem. Soc.* **23**, 1699–1701.
Xu, L.-Z., Yu, G.-P., Xu, F.-L. & Li, W.-H. (2005). *Acta Cryst.* E**61**, o2061–o2062.
Xu, L.-Z., Yu, G.-P., Yin, S.-M., Zhou, K. & Yang, S.-H. (2005). *Acta Cryst.* E**61**, o3375–o3376.
Zhang, Y., Qiao, R. Z., Xu, P. F., Zhang, Z. Y., Wang, Q., Mao, L. M. & Yu, K. B. (2002). *J. Chin. Chem. Soc. (Taipei)*, **49**, 369–372.