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2-(Benzo[d]thiazol-2-ylsulfanyl)-N-(6-methyl-2-pyridyl)acetamide

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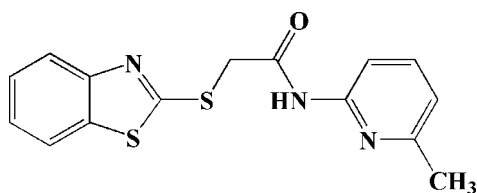
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Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.037; wR factor = 0.092; data-to-parameter ratio = 18.1.

In the title compound, $\text{C}_{15}\text{H}_{13}\text{N}_3\text{OS}_2$, the pyridine ring and the benzo[d]thiazole unit subtend a dihedral angle of $57.7(2)^\circ$. The length of the $\text{C}_{\text{sp}^2}-\text{S}$ bond [1.7462 (17) Å] is significantly shorter than that of the $\text{C}_{\text{sp}^3}-\text{S}$ bond [1.8133 (18) Å]. The crystal structure is stabilized by intramolecular $\text{N}-\text{H}\cdots\text{N}$ and intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{N}$ hydrogen-bond interactions. Furthermore, $\text{C}-\text{H}\cdots\pi$ interactions stabilize the crystal packing.

Related literature

For biologically active compounds containing the acylamide system, see: Bannasar *et al.* (2006); Ladziata *et al.* (2006). For bond-length data, see: Gao *et al.* (2007).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{13}\text{N}_3\text{OS}_2$
 $M_r = 315.40$

Triclinic, $P\bar{1}$
 $a = 8.1919(16)$ Å

$b = 9.0818(18)$ Å
 $c = 11.107(2)$ Å
 $\alpha = 74.78(3)^\circ$
 $\beta = 89.55(3)^\circ$
 $\gamma = 69.34(3)^\circ$
 $V = 742.8(3)$ Å³

$Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.36$ mm⁻¹
 $T = 113$ K
 $0.16 \times 0.14 \times 0.10$ mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\text{min}} = 0.945$, $T_{\text{max}} = 0.965$

9391 measured reflections
3526 independent reflections
2615 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.048$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.092$
 $S = 0.99$
3526 reflections
195 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2A}\cdots\text{N3}$	0.867 (18)	2.142 (18)	2.949 (2)	154.6 (16)
$\text{C2}-\text{H2}\cdots\text{O1}$	0.95	2.30	2.890 (2)	119
$\text{C8}-\text{H8A}\cdots\text{O1}^i$	0.99	2.31	3.239 (2)	156
$\text{C8}-\text{H8B}\cdots\text{N3}$	0.99	2.47	2.905 (2)	106
$\text{C12}-\text{H12}\cdots\text{N1}^{ii}$	0.95	2.57	3.498 (2)	166
$\text{C8}-\text{H8B}\cdots\text{Cg2}^{iii}$	0.99	2.68	3.494 (2)	140

Symmetry codes: (i) $-x, -y + 2, -z$; (ii) $-x + 1, -y + 2, -z + 1$; (iii) $-x + 1, -y + 2, -z$. Cg2 is the centroid of the $\text{N1/C1}-\text{C5}$ ring.

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: AT2753).

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supplementary materials

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2-(Benzo[*d*]thiazol-2-ylsulfanyl)-*N*-(6-methyl-2-pyridyl)acetamide

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Comment

The acylamide compound is an important class of medical intermediate. Recently, many biological compounds containing acylamide have been reported (Ladziata *et al.*, 2006; Bennasar *et al.*, 2006). Now, we have synthesized the title compound, (I), from the benzo[*d*]thiazole-2-thiol with 6-methylpyridine carbamic chloride. Here, we report its crystal structure.

The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. The molecule contains a pyridine ring and a benzo[*d*]thiazole ring. The dihedral angle between the benzene ring and benzo[*d*]thiazole ring is 57.7 (2)°. The methyl carbon attached to the pyridine ring is coplanar to the pyridine ring with an r.m.s deviation of 0.0064 (3) Å. The C1—N1—C7—C8 torsion angle of 178.66 (15)° indicates that the acylamide group are nearly coplanar with the pyridine ring plane. As a result of π - π conjugation, the C_{sp}²—S bond [S1—C9 = 1.7462 (17) Å] is significantly shorter than the C_{sp}³—S bond [S1—C8 = 1.8133 (18) Å]. These values compare with the values of 1.772 (3) and 1.801 (2) Å reported in the literature (Gao *et al.*, 2007). The crystal structure is stabilized by the intramolecular N—H···N and intermolecular C—H···O and C—H···N hydrogen bond interactions. Furthermore, C—H··· π interactions stabilize the crystal packing (Table 1).

Experimental

The title compound was synthesized by the reaction of from the benzo[*d*]thiazole-2-thiol with 6-methylpyridine carbamic chloride in the 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 different density map and the atomic coordinates allowed to refine freely. Other H atoms were positioned geometrically and refined as riding (C—H = 0.95–0.99 Å) and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$ or $1.5U_{\text{eq}}(\text{parent})$.

Figures

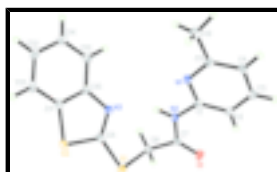


Fig. 1. View of the molecule of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 35% probability level.

2-(Benzo[d]thiazol-2-ylsulfanyl)-N-(6-methyl-2-pyridyl)acetamide

Crystal data

$C_{15}H_{13}N_3OS_2$	$Z = 2$
$M_r = 315.40$	$F_{000} = 328$
Triclinic, $P\bar{1}$	$D_x = 1.410 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 8.1919 (16) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 9.0818 (18) \text{ \AA}$	Cell parameters from 2582 reflections
$c = 11.107 (2) \text{ \AA}$	$\theta = 1.9\text{--}27.9^\circ$
$\alpha = 74.78 (3)^\circ$	$\mu = 0.36 \text{ mm}^{-1}$
$\beta = 89.55 (3)^\circ$	$T = 113 \text{ K}$
$\gamma = 69.34 (3)^\circ$	Prism, colourless
$V = 742.8 (3) \text{ \AA}^3$	$0.16 \times 0.14 \times 0.10 \text{ mm}$

Data collection

Rigaku Saturn diffractometer	3526 independent reflections
Radiation source: rotating anode	2615 reflections with $I > 2\sigma(I)$
Monochromator: confocal	$R_{\text{int}} = 0.048$
$T = 113 \text{ K}$	$\theta_{\text{max}} = 27.9^\circ$
ω scans	$\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.945$, $T_{\text{max}} = 0.965$	$k = -11 \rightarrow 11$
9391 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.092$	$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2]$
$S = 0.99$	where $P = (F_o^2 + 2F_c^2)/3$
3526 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
195 parameters	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.38 \text{ e \AA}^{-3}$
	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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.08893 (5)	1.05001 (5)	0.25028 (4)	0.02336 (13)
S2	0.20597 (5)	1.15671 (5)	0.45732 (4)	0.02242 (13)
N1	0.74042 (17)	0.74849 (16)	0.19582 (13)	0.0200 (3)
N2	0.45173 (18)	0.88209 (17)	0.12486 (14)	0.0210 (3)
N3	0.37052 (17)	1.13818 (15)	0.25715 (13)	0.0192 (3)
O1	0.24938 (15)	0.82797 (14)	0.02096 (13)	0.0320 (3)
C1	0.6056 (2)	0.74785 (18)	0.12785 (15)	0.0189 (4)
C2	0.6186 (2)	0.62959 (19)	0.06672 (16)	0.0217 (4)
H2	0.5203	0.6332	0.0196	0.026*
C3	0.7803 (2)	0.50655 (19)	0.07723 (16)	0.0233 (4)
H3	0.7949	0.4229	0.0371	0.028*
C4	0.9210 (2)	0.50511 (19)	0.14621 (16)	0.0217 (4)
H4	1.0329	0.4214	0.1533	0.026*
C5	0.8961 (2)	0.62787 (19)	0.20491 (16)	0.0204 (4)
C6	1.0426 (2)	0.6313 (2)	0.28327 (18)	0.0300 (4)
H6A	1.0281	0.7452	0.2761	0.045*
H6B	1.1550	0.5765	0.2538	0.045*
H6C	1.0403	0.5745	0.3711	0.045*
C7	0.2879 (2)	0.9140 (2)	0.07486 (16)	0.0217 (4)
C8	0.1511 (2)	1.06990 (19)	0.09143 (16)	0.0222 (4)
H8A	0.0453	1.1009	0.0339	0.027*
H8B	0.1975	1.1594	0.0676	0.027*
C9	0.2379 (2)	1.11504 (18)	0.31235 (16)	0.0193 (4)
C10	0.4590 (2)	1.19559 (18)	0.32980 (15)	0.0184 (4)
C11	0.3900 (2)	1.21228 (18)	0.44347 (16)	0.0194 (4)
C12	0.4660 (2)	1.26537 (19)	0.52672 (16)	0.0239 (4)
H12	0.4194	1.2750	0.6041	0.029*
C13	0.6122 (2)	1.3037 (2)	0.49261 (17)	0.0257 (4)
H13	0.6667	1.3405	0.5476	0.031*
C14	0.6811 (2)	1.2894 (2)	0.37940 (17)	0.0255 (4)
H14	0.7806	1.3181	0.3583	0.031*
C15	0.6076 (2)	1.23436 (19)	0.29716 (16)	0.0225 (4)
H15	0.6563	1.2231	0.2207	0.027*

supplementary materials

H2A 0.463 (2) 0.951 (2) 0.1624 (17) 0.027 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0181 (2)	0.0224 (2)	0.0319 (3)	-0.00785 (18)	0.00497 (18)	-0.0107 (2)
S2	0.0215 (3)	0.0227 (2)	0.0215 (2)	-0.00602 (18)	0.00643 (18)	-0.00652 (18)
N1	0.0180 (7)	0.0194 (7)	0.0224 (8)	-0.0055 (6)	0.0019 (6)	-0.0072 (6)
N2	0.0166 (8)	0.0207 (7)	0.0274 (8)	-0.0035 (6)	-0.0001 (6)	-0.0139 (7)
N3	0.0175 (8)	0.0178 (7)	0.0215 (7)	-0.0049 (6)	0.0015 (6)	-0.0062 (6)
O1	0.0229 (7)	0.0330 (7)	0.0440 (8)	-0.0057 (5)	-0.0046 (6)	-0.0229 (7)
C1	0.0186 (9)	0.0183 (8)	0.0186 (8)	-0.0054 (7)	0.0036 (7)	-0.0052 (7)
C2	0.0220 (9)	0.0220 (8)	0.0237 (9)	-0.0086 (7)	0.0046 (7)	-0.0095 (7)
C3	0.0281 (10)	0.0191 (8)	0.0246 (9)	-0.0088 (7)	0.0078 (8)	-0.0094 (7)
C4	0.0206 (9)	0.0156 (8)	0.0250 (9)	-0.0032 (7)	0.0070 (7)	-0.0042 (7)
C5	0.0187 (9)	0.0191 (8)	0.0207 (9)	-0.0056 (7)	0.0046 (7)	-0.0030 (7)
C6	0.0215 (10)	0.0271 (9)	0.0374 (11)	-0.0034 (8)	-0.0007 (8)	-0.0097 (9)
C7	0.0178 (9)	0.0236 (8)	0.0230 (9)	-0.0056 (7)	0.0016 (7)	-0.0082 (8)
C8	0.0179 (9)	0.0229 (9)	0.0252 (9)	-0.0046 (7)	-0.0016 (7)	-0.0096 (8)
C9	0.0185 (9)	0.0137 (7)	0.0211 (9)	-0.0005 (6)	0.0015 (7)	-0.0047 (7)
C10	0.0198 (9)	0.0142 (7)	0.0188 (8)	-0.0036 (6)	-0.0017 (7)	-0.0041 (7)
C11	0.0194 (9)	0.0147 (8)	0.0199 (9)	-0.0023 (6)	0.0010 (7)	-0.0033 (7)
C12	0.0284 (10)	0.0212 (8)	0.0178 (9)	-0.0029 (7)	-0.0002 (7)	-0.0068 (7)
C13	0.0289 (10)	0.0201 (8)	0.0270 (10)	-0.0068 (7)	-0.0042 (8)	-0.0076 (8)
C14	0.0255 (10)	0.0233 (9)	0.0284 (10)	-0.0115 (8)	-0.0009 (8)	-0.0046 (8)
C15	0.0216 (9)	0.0241 (9)	0.0208 (9)	-0.0081 (7)	0.0043 (7)	-0.0049 (7)

Geometric parameters (\AA , $^\circ$)

S1—C9	1.7462 (17)	C4—H4	0.9500
S1—C8	1.8133 (18)	C5—C6	1.502 (2)
S2—C11	1.7444 (17)	C6—H6A	0.9800
S2—C9	1.7459 (17)	C6—H6B	0.9800
N1—C5	1.340 (2)	C6—H6C	0.9800
N1—C1	1.345 (2)	C7—C8	1.519 (2)
N2—C7	1.359 (2)	C8—H8A	0.9900
N2—C1	1.404 (2)	C8—H8B	0.9900
N2—H2A	0.863 (17)	C10—C11	1.402 (2)
N3—C9	1.297 (2)	C10—C15	1.402 (2)
N3—C10	1.395 (2)	C11—C12	1.389 (2)
O1—C7	1.2212 (19)	C12—C13	1.385 (2)
C1—C2	1.388 (2)	C12—H12	0.9500
C2—C3	1.381 (2)	C13—C14	1.393 (3)
C2—H2	0.9500	C13—H13	0.9500
C3—C4	1.383 (2)	C14—C15	1.381 (2)
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.389 (2)	C15—H15	0.9500
C9—S1—C8	100.19 (8)	O1—C7—C8	121.40 (15)

C11—S2—C9	88.51 (8)	N2—C7—C8	113.96 (14)
C5—N1—C1	117.98 (13)	C7—C8—S1	113.25 (12)
C7—N2—C1	128.33 (14)	C7—C8—H8A	108.9
C7—N2—H2A	116.3 (12)	S1—C8—H8A	108.9
C1—N2—H2A	115.3 (12)	C7—C8—H8B	108.9
C9—N3—C10	110.02 (14)	S1—C8—H8B	108.9
N1—C1—C2	123.80 (15)	H8A—C8—H8B	107.7
N1—C1—N2	111.89 (13)	N3—C9—S2	116.86 (12)
C2—C1—N2	124.31 (15)	N3—C9—S1	124.62 (13)
C3—C2—C1	117.24 (15)	S2—C9—S1	118.51 (10)
C3—C2—H2	121.4	N3—C10—C11	115.18 (15)
C1—C2—H2	121.4	N3—C10—C15	124.86 (16)
C2—C3—C4	119.97 (15)	C11—C10—C15	119.96 (15)
C2—C3—H3	120.0	C12—C11—C10	121.74 (16)
C4—C3—H3	120.0	C12—C11—S2	128.85 (14)
C3—C4—C5	119.00 (16)	C10—C11—S2	109.42 (12)
C3—C4—H4	120.5	C13—C12—C11	117.45 (17)
C5—C4—H4	120.5	C13—C12—H12	121.3
N1—C5—C4	122.01 (15)	C11—C12—H12	121.3
N1—C5—C6	116.40 (14)	C12—C13—C14	121.46 (16)
C4—C5—C6	121.59 (15)	C12—C13—H13	119.3
C5—C6—H6A	109.5	C14—C13—H13	119.3
C5—C6—H6B	109.5	C15—C14—C13	121.31 (17)
H6A—C6—H6B	109.5	C15—C14—H14	119.3
C5—C6—H6C	109.5	C13—C14—H14	119.3
H6A—C6—H6C	109.5	C14—C15—C10	118.07 (16)
H6B—C6—H6C	109.5	C14—C15—H15	121.0
O1—C7—N2	124.63 (16)	C10—C15—H15	121.0
C5—N1—C1—C2	0.0 (3)	C11—S2—C9—N3	0.20 (13)
C5—N1—C1—N2	-179.13 (14)	C11—S2—C9—S1	-178.48 (10)
C7—N2—C1—N1	-173.97 (16)	C8—S1—C9—N3	-10.77 (15)
C7—N2—C1—C2	6.9 (3)	C8—S1—C9—S2	167.80 (9)
N1—C1—C2—C3	-0.1 (3)	C9—N3—C10—C11	1.10 (19)
N2—C1—C2—C3	178.95 (16)	C9—N3—C10—C15	-179.48 (14)
C1—C2—C3—C4	-0.2 (2)	N3—C10—C11—C12	178.84 (14)
C2—C3—C4—C5	0.5 (2)	C15—C10—C11—C12	-0.6 (2)
C1—N1—C5—C4	0.3 (2)	N3—C10—C11—S2	-0.95 (17)
C1—N1—C5—C6	-179.05 (15)	C15—C10—C11—S2	179.60 (12)
C3—C4—C5—N1	-0.6 (3)	C9—S2—C11—C12	-179.36 (15)
C3—C4—C5—C6	178.74 (16)	C9—S2—C11—C10	0.42 (12)
C1—N2—C7—O1	-1.3 (3)	C10—C11—C12—C13	0.8 (2)
C1—N2—C7—C8	178.66 (15)	S2—C11—C12—C13	-179.40 (12)
O1—C7—C8—S1	105.85 (18)	C11—C12—C13—C14	-0.1 (2)
N2—C7—C8—S1	-74.11 (17)	C12—C13—C14—C15	-0.8 (3)
C9—S1—C8—C7	91.58 (12)	C13—C14—C15—C10	1.1 (2)
C10—N3—C9—S2	-0.76 (17)	N3—C10—C15—C14	-179.76 (14)
C10—N3—C9—S1	177.84 (11)	C11—C10—C15—C14	-0.4 (2)

supplementary materials

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A \cdots N3	0.867 (18)	2.142 (18)	2.949 (2)	154.6 (16)
C2—H2 \cdots O1	0.95	2.30	2.890 (2)	119
C8—H8A \cdots O1 ⁱ	0.99	2.31	3.239 (2)	156
C8—H8B \cdots N3	0.99	2.47	2.905 (2)	106
C12—H12 \cdots N1 ⁱⁱ	0.95	2.57	3.498 (2)	166
C8—H8B \cdots Cg2 ⁱⁱⁱ	0.99	2.68	3.494 (2)	140

Symmetry codes: (i) $-x, -y+2, -z$; (ii) $-x+1, -y+2, -z+1$; (iii) $-x+1, -y+2, -z$.

Fig. 1

