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Ethyl 2-amino-4-isobutylthiophene-3-carboxylate

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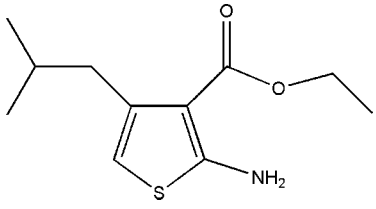
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.070; wR factor = 0.172; data-to-parameter ratio = 17.0.

The title compound, $C_{11}H_{17}NO_2S$, was synthesized by the Gewald reaction. Intramolecular N—H···O and C—H···O hydrogen bonds determine the conformation of the molecule. The packing of the molecules in the crystal structure is governed mainly by intermolecular N—H···O hydrogen-bonding interactions.

Related literature

A related synthetic method is described by Sabnis *et al.* (1999). For related literature, see: Ding *et al.* (2004); Liu & Hu (2006); Liu & Liao (2006).



Experimental

Crystal data

$C_{11}H_{17}NO_2S$
M_r = 227.32
Monoclinic, $P2_1/c$
a = 13.4321 (7) Å
b = 9.4977 (7) Å
c = 9.8629 (10) Å
β = 94.435 (1)°
V = 1254.48 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.931, T_{max} = 0.954
12038 measured reflections
2458 independent reflections
1979 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.172
S = 1.16
2458 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O1^{i}	0.85 (3)	2.11 (3)	2.929 (3)	162 (3)
N1—H1A···O1	0.86 (3)	2.07 (3)	2.728 (3)	132 (3)
C6—H6···O2	0.98	2.60	3.193 (4)	119
C5—H5B···O2	0.97	2.43	2.888 (3)	109

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2046).

References

Bruker (2001). *SMART* (Version 5.628) and *SAINT-Plus* (Version 6.45). Bruker AXS Inc., Madison, Wisconsin, USA.
Ding, M. W., Xu, S. Z. & Zhao, J. F. (2004). *J. Org. Chem.* **69**, 8366–8371.
Liu, M.-G. & Hu, Y.-G. (2006). *Acta Cryst.* E**62**, o3206–o3208.
Liu, M.-G. & Liao, Q.-B. (2006). *Acta Cryst.* E**62**, o2214–o2215.
Sabnis, R. W., Rangnekar, D. W. & Sonawane, N. D. (1999). *J. Heterocycl. Chem.* **36**, 333–345.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SHELXTL*. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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supplementary materials

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Ethyl 2-amino-4-isobutylthiophene-3-carboxylate

Q.-B. Liao, J. Huang, Z.-Z. Yang and M.-G. Liu

Comment

The chemistry of 2-aminothiophenes has received much attention because of the convenient availability through the most versatile synthetic methods developed by Gewald (Sabnis *et al.*, 1999). Recently we have been interested in the synthesis of derivatives of heterocycles using the aza-Wittig reaction. Some related X-ray crystal structure reports for pyrimidinone derivatives have been published (Ding *et al.*, 2004; Liu & Hu, 2006; Liu & Liao, 2006). Here, the structure of the title compound, which may be used as a new intermediate synthesized *via* the Gewald reaction, is reported (Fig. 1). The thiophene ring system is coplanar. Intramolecular N—H···O and C—H···O hydrogen bonds (Table 1) determine the conformation of the molecule. As can be seen from the packing diagram (Fig. 2), intramolecular and intermolecular N—H···O hydrogen bonds (Table 1) link the molecules.

Experimental

To a solution of ethyl cyanoacetate(1.13 g, 10 mmol), sulfur (0.32 g, 10 mmol) and 4-methylpentan-2-one (1.00 g, 10 mmol) in anhydrous ethanol (20 ml) was added in morpholine (0.8 ml). The mixture was stirred for 6 h at 313 − 323 K. The solution was concentrated under reduced pressure and the residue was recrystallized from dichloromethane and ethanol (1:2 *v*/*v*) to give the title compound. Crystals suitable for single-crystal X-ray diffraction were obtained by recrystallization from a mixed solvent of ethanol and dichloromethane (1:1 *v*/*v*) at room temperature.

Refinement

All H atoms were located in difference maps and treated as riding atoms, except those at N1, with the following distance restraints: C—H = 0.93 Å, U_{iso}=1.2U_{eq} (C) for Csp^2, C—H = 0.98 Å, U_{iso} = 1.2U_{eq} (C) for CH, C—H = 0.97 Å, U_{iso} = 1.2U_{eq} (C) for CH_2, N—H = 0.86 Å, U_{iso} = 1.2U_{eq} (N) for NH_2, C—H = 0.96 Å, U_{iso} = 1.5U_{eq} (C) for CH_3.

Figures

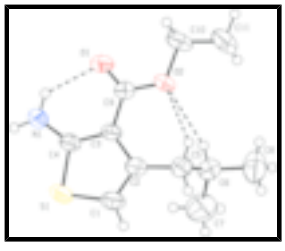

Fig. 1. A view of the title compound showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level, intramolecular hydrogen bonds are shown in dashed lines.

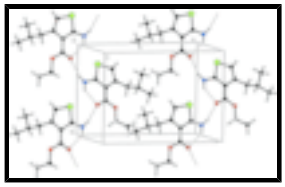

Fig. 2. The packing in the crystal structure, showing the N—H···O hydrogen bonds as dashed lines.

Ethyl 2-amino-4-isobutylthiophene-3-carboxylate

Crystal data

$C_{11}H_{17}NO_2S$	F_{000} = 488
M_r = 227.32	D_x = 1.204 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3288 reflections
a = 13.4321 (7) Å	θ = 2.5–27.0º
b = 9.4977 (7) Å	μ = 0.24 mm^{-1}
c = 9.8629 (10) Å	T = 293 (2) K
β = 94.4350 (10)º	Block, colorless
V = 1254.48 (17) Å^3	0.30 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	2458 independent reflections
Radiation source: fine-focus sealed tube	1979 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
T = 292(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −16→16
T_{min} = 0.931, T_{max} = 0.954	k = −11→11
12038 measured reflections	l = −11→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.070	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.172	$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.4453P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.16	$(\Delta/\sigma)_{max}$ = 0.001
2458 reflections	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
145 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1600 (2)	0.2199 (3)	0.5726 (3)	0.0552 (8)
H1	0.1868	0.1542	0.6351	0.066*
C2	0.1898 (2)	0.3551 (3)	0.5718 (3)	0.0440 (6)
C3	0.13509 (18)	0.4346 (2)	0.4638 (3)	0.0389 (6)
C4	0.0652 (2)	0.3515 (3)	0.3900 (3)	0.0423 (6)
C5	0.2705 (2)	0.4095 (3)	0.6717 (3)	0.0540 (7)
H5A	0.2773	0.3455	0.7485	0.065*
H5B	0.2498	0.5000	0.7053	0.065*
C6	0.3726 (2)	0.4269 (3)	0.6161 (4)	0.0634 (9)
H6	0.3654	0.4930	0.5398	0.076*
C7	0.4119 (3)	0.2894 (5)	0.5640 (5)	0.1067 (15)
H7A	0.4772	0.3041	0.5334	0.160*
H7B	0.3678	0.2559	0.4898	0.160*
H7C	0.4157	0.2210	0.6359	0.160*
C8	0.4457 (3)	0.4899 (5)	0.7246 (5)	0.1052 (15)
H8A	0.4205	0.5786	0.7538	0.158*
H8B	0.5091	0.5041	0.6878	0.158*
H8C	0.4538	0.4269	0.8008	0.158*
C9	0.1436 (2)	0.5818 (3)	0.4294 (3)	0.0456 (7)
C10	0.2205 (3)	0.8022 (3)	0.4833 (4)	0.0785 (11)
H10A	0.1593	0.8524	0.4976	0.094*
H10B	0.2351	0.8148	0.3893	0.094*
C11	0.3024 (3)	0.8574 (4)	0.5739 (5)	0.0965 (14)
H11A	0.2876	0.8437	0.6667	0.145*
H11B	0.3104	0.9561	0.5569	0.145*
H11C	0.3630	0.8088	0.5577	0.145*
N1	−0.0020 (2)	0.3892 (3)	0.2887 (3)	0.0533 (7)
H1B	−0.039 (2)	0.330 (3)	0.245 (3)	0.064*
H1A	0.003 (2)	0.476 (4)	0.264 (3)	0.064*
O1	0.09739 (17)	0.6374 (2)	0.3336 (2)	0.0669 (6)
O2	0.20850 (15)	0.65355 (18)	0.5122 (2)	0.0583 (6)
S1	0.06791 (6)	0.17970 (7)	0.44771 (8)	0.0563 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.071 (2)	0.0348 (14)	0.0594 (18)	0.0013 (13)	0.0021 (15)	0.0092 (13)
C2	0.0506 (15)	0.0360 (13)	0.0454 (15)	0.0012 (11)	0.0045 (12)	0.0013 (11)
C3	0.0460 (14)	0.0267 (12)	0.0439 (14)	0.0027 (10)	0.0022 (11)	−0.0006 (10)
C4	0.0532 (16)	0.0288 (12)	0.0448 (15)	0.0028 (10)	0.0036 (12)	−0.0028 (10)
C5	0.0629 (19)	0.0486 (16)	0.0484 (17)	0.0011 (13)	−0.0096 (14)	0.0034 (13)
C6	0.0577 (19)	0.0528 (18)	0.077 (2)	0.0024 (14)	−0.0096 (16)	0.0093 (16)
C7	0.076 (3)	0.088 (3)	0.157 (5)	0.021 (2)	0.011 (3)	−0.009 (3)

C8	0.077 (3)	0.115 (4)	0.118 (4)	−0.022 (2)	−0.028 (3)	0.004 (3)
C9	0.0459 (15)	0.0340 (13)	0.0560 (17)	0.0041 (11)	−0.0028 (13)	0.0018 (12)
C10	0.082 (2)	0.0294 (15)	0.122 (3)	−0.0067 (14)	−0.008 (2)	0.0089 (17)
C11	0.089 (3)	0.0433 (18)	0.154 (4)	−0.0178 (18)	−0.017 (3)	−0.006 (2)
N1	0.0638 (16)	0.0347 (12)	0.0587 (16)	−0.0010 (11)	−0.0130 (13)	−0.0068 (11)
O1	0.0819 (15)	0.0334 (10)	0.0808 (16)	0.0012 (10)	−0.0236 (12)	0.0112 (10)
O2	0.0646 (13)	0.0259 (9)	0.0815 (15)	−0.0064 (8)	−0.0132 (11)	0.0024 (9)
S1	0.0734 (6)	0.0293 (4)	0.0650 (5)	−0.0083 (3)	−0.0026 (4)	0.0018 (3)

Geometric parameters (Å, °)

C1—C2	1.345 (4)	C7—H7B	0.9600
C1—S1	1.720 (3)	C7—H7C	0.9600
C1—H1	0.9300	C8—H8A	0.9600
C2—C3	1.457 (3)	C8—H8B	0.9600
C2—C5	1.500 (4)	C8—H8C	0.9600
C3—C4	1.389 (4)	C9—O1	1.211 (3)
C3—C9	1.446 (3)	C9—O2	1.334 (3)
C4—N1	1.341 (4)	C10—O2	1.452 (3)
C4—S1	1.728 (3)	C10—C11	1.460 (5)
C5—C6	1.524 (4)	C10—H10A	0.9700
C5—H5A	0.9700	C10—H10B	0.9700
C5—H5B	0.9700	C11—H11A	0.9600
C6—C7	1.513 (5)	C11—H11B	0.9600
C6—C8	1.519 (5)	C11—H11C	0.9600
C6—H6	0.9800	N1—H1B	0.85 (3)
C7—H7A	0.9600	N1—H1A	0.86 (3)
C2—C1—S1	113.9 (2)	H7A—C7—H7C	109.5
C2—C1—H1	123.0	H7B—C7—H7C	109.5
S1—C1—H1	123.0	C6—C8—H8A	109.5
C1—C2—C3	111.4 (2)	C6—C8—H8B	109.5
C1—C2—C5	121.6 (3)	H8A—C8—H8B	109.5
C3—C2—C5	126.9 (2)	C6—C8—H8C	109.5
C4—C3—C9	119.2 (2)	H8A—C8—H8C	109.5
C4—C3—C2	111.8 (2)	H8B—C8—H8C	109.5
C9—C3—C2	128.9 (2)	O1—C9—O2	121.7 (2)
N1—C4—C3	128.8 (2)	O1—C9—C3	124.1 (3)
N1—C4—S1	119.6 (2)	O2—C9—C3	114.2 (2)
C3—C4—S1	111.54 (19)	O2—C10—C11	108.6 (3)
C2—C5—C6	115.2 (2)	O2—C10—H10A	110.0
C2—C5—H5A	108.5	C11—C10—H10A	110.0
C6—C5—H5A	108.5	O2—C10—H10B	110.0
C2—C5—H5B	108.5	C11—C10—H10B	110.0
C6—C5—H5B	108.5	H10A—C10—H10B	108.3
H5A—C5—H5B	107.5	C10—C11—H11A	109.5
C7—C6—C8	110.8 (3)	C10—C11—H11B	109.5
C7—C6—C5	112.1 (3)	H11A—C11—H11B	109.5
C8—C6—C5	109.9 (3)	C10—C11—H11C	109.5
C7—C6—H6	108.0	H11A—C11—H11C	109.5

C8—C6—H6	108.0	H11B—C11—H11C	109.5
C5—C6—H6	108.0	C4—N1—H1B	122 (2)
C6—C7—H7A	109.5	C4—N1—H1A	114 (2)
C6—C7—H7B	109.5	H1B—N1—H1A	123 (3)
H7A—C7—H7B	109.5	C9—O2—C10	117.0 (2)
C6—C7—H7C	109.5	C1—S1—C4	91.27 (13)
S1—C1—C2—C3	0.3 (3)	C2—C5—C6—C7	−59.4 (4)
S1—C1—C2—C5	−179.1 (2)	C2—C5—C6—C8	176.9 (3)
C1—C2—C3—C4	0.7 (3)	C4—C3—C9—O1	−4.7 (4)
C5—C2—C3—C4	−179.9 (2)	C2—C3—C9—O1	176.8 (3)
C1—C2—C3—C9	179.3 (3)	C4—C3—C9—O2	176.0 (2)
C5—C2—C3—C9	−1.4 (4)	C2—C3—C9—O2	−2.5 (4)
C9—C3—C4—N1	−2.6 (4)	O1—C9—O2—C10	0.8 (4)
C2—C3—C4—N1	176.1 (3)	C3—C9—O2—C10	−179.9 (3)
C9—C3—C4—S1	179.87 (19)	C11—C10—O2—C9	−173.3 (3)
C2—C3—C4—S1	−1.4 (3)	C2—C1—S1—C4	−1.0 (2)
C1—C2—C5—C6	101.9 (3)	N1—C4—S1—C1	−176.5 (2)
C3—C2—C5—C6	−77.4 (4)	C3—C4—S1—C1	1.4 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1B···O1^{i}	0.85 (3)	2.11 (3)	2.929 (3)	162 (3)
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Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1/2.

Fig. 1

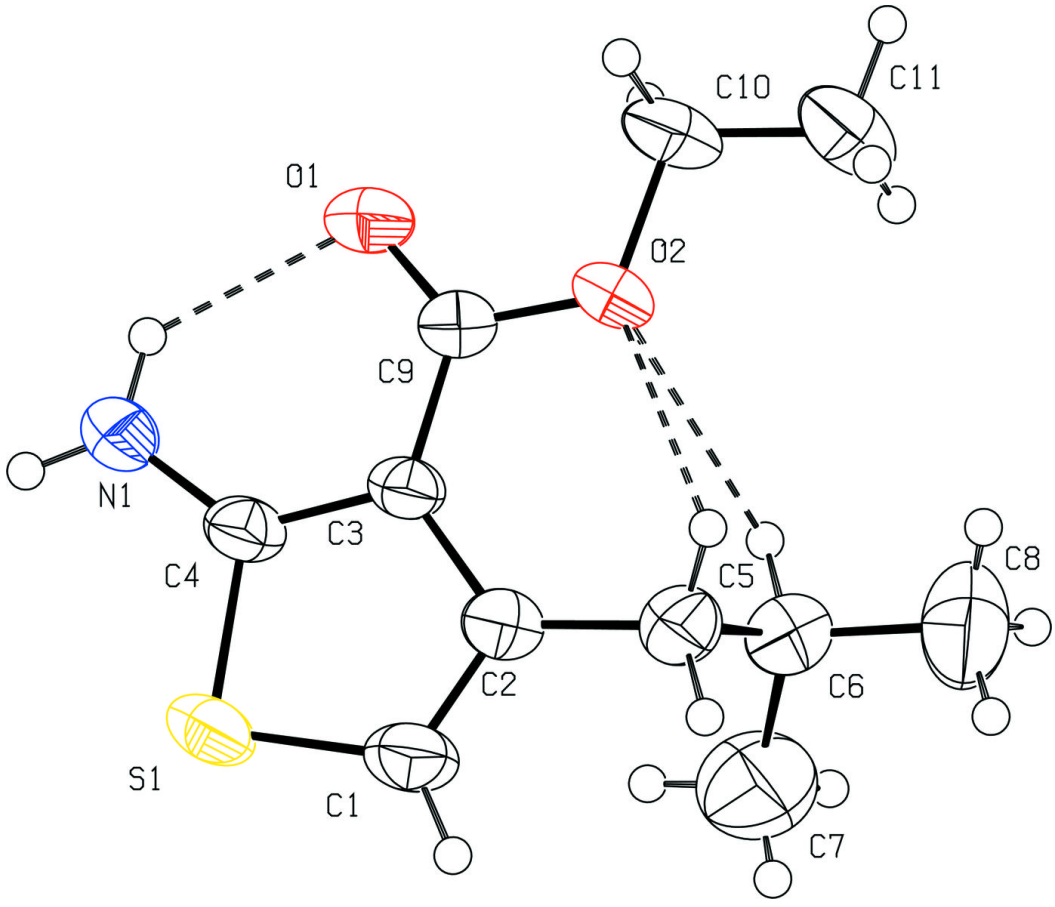

Fig. 2