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3-Methyl-*N*′-(2-thienylcarboxyl)butyrohydrazide

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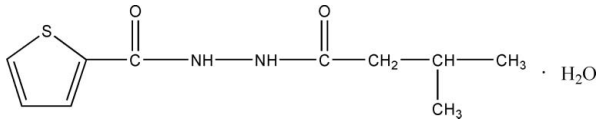
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.123; data-to-parameter ratio = 14.7.

In the crystal structure of the title compound, $C_{10}H_{14}N_2O_2S\cdot H_2O$, there are intermolecular O—H···O and N—H···O hydrogen bonds between the hydrazide O and N atoms and the water H atoms. These help to stabilize the structure and link the molecules to form a two-dimensional grid.

Related literature

For related literature, see: Wu *et al.* (2004); Xiao & Wang (2004).



Experimental

Crystal data

$C_{10}H_{14}N_2O_2S\cdot H_2O$
M_r = 244.31
Monoclinic, $P2_1/c$
a = 6.5648 (5) Å
b = 7.3451 (7) Å
c = 25.956 (2) Å
β = 96.387 (4)°
V = 1243.81 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 293 (2) K
0.60 × 0.30 × 0.10 mm

Data collection

Rigaku Mercury70 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
T_{min} = 0.801, T_{max} = 1.000
8853 measured reflections
2849 independent reflections
2423 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.123
S = 1.00
2849 reflections
194 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3^{i}	0.86	2.08	2.8173 (18)	143
N2—H2B···O3^{ii}	0.86	2.08	2.9147 (18)	162
O3—H3···O1	0.80 (3)	1.95 (3)	2.7416 (18)	171 (2)
O3—H4···O2^{iii}	0.90 (3)	1.83 (3)	2.7355 (17)	178 (2)

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

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); 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: WK2059).

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supplementary materials

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3-Methyl-*N*'-(2-thienylcarboxyl)butyrohydrazide

G.-X. Liu, W.-S. Wu, H.-P. Li and G.-D. Yang

Comment

A number of thiophene derivatives have been widely investigated in chemistry due to their wide range of medicinal applications. (Xiao *et al.*, 2004).

The molecular structure of the compound is illustrated in Figure 1 and selected bond distances and angles are given in Table 1. The bond distances are consistent with conjugation in the molecule.

The atoms C(1) C(2) C(3) C(4) S(1) C(5) O(1) lie in a plane with a r.m.s. deviation from planarity of 0.0340 Å. The atoms of N(1) N(2) C(6) O(2) are also in a plane, with 0.0124 Å) of r.m.s deviation and forms an angle of 78.95 (6)° to the previous plane.

N—H···O, O—H···O intermolecular hydrogen bonds in the compound lead to a two-dimensional grid structure (shown in Figure 2) and detailed in Table 2. Atom O3 is involved as a acceptor in four intermolecular hydrogen bonds (Figure 2).

Experimental

The compound was synthesized according to the method of Wu *et al.* (2004). The white powder of N,*N*'-thienylhydrazide (1 mmol) and the Isovaleric anhydride (1 mmol) were dissolved in ethanol, stirred in an ice bath for 15 min. Transparent colourless crystals of (I) grew from the mother liquor by slow evaporation at room temperature after one week.

Refinement

The C-bound H atoms were included in the riding model approximation with C—H = 0.93 Å. all these H atoms included in the final refinement. The positions of the water O-bound H atoms were located from a difference Fourier map and fixed.

Figures

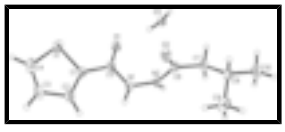

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme, with 30% displacement ellipsoids.

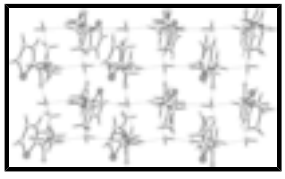

Fig. 2. Packing diagram of (I), showing hydrogen bonds as dashed lines.

3-Methyl-*N*'-(2-thienylcarboxyl)butyrohydrazide

Crystal data

$C_{10}H_{14}N_2O_2S·H_2O$	F_{000} = 520
M_r = 244.31	D_x = 1.305 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2785 reflections
a = 6.5648 (5) Å	θ = 2.8–27.5º
b = 7.3451 (7) Å	μ = 0.26 mm^{-1}
c = 25.956 (2) Å	T = 293 (2) K
β = 96.387 (4)º	Prism, white
V = 1243.81 (18) Å^3	0.60 × 0.30 × 0.10 mm
Z = 4	

Data collection

Rigaku Mercury70 (2x2 bin mode) diffractometer	2849 independent reflections
Radiation source: Sealed Tube	2423 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 293(2) K	θ_{max} = 27.5º
CCD_Profile_fitting scans	θ_{min} = 2.9º
Absorption correction: multi-scan (CrystalClear; Rigaku, 2000)	h = −8→8
T_{min} = 0.801, T_{max} = 1.000	k = −9→9
8853 measured reflections	l = −33→14

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.044	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.123	$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2 + 0.5306P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.006
2849 reflections	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
194 parameters	$\Delta\rho_{min}$ = −0.29 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	0.81975 (8)	0.28733 (9)	0.210206 (18)	0.0589 (2)
N1	0.8807 (2)	0.15490 (19)	0.06560 (5)	0.0356 (3)
H1A	1.0116	0.1423	0.0682	0.043*
N2	0.7661 (2)	0.14048 (19)	0.01749 (5)	0.0358 (3)
H2B	0.7293	0.0353	0.0052	0.043*
O1	0.59982 (18)	0.1910 (2)	0.10851 (5)	0.0484 (3)
O2	0.7541 (2)	0.44417 (16)	0.00794 (5)	0.0480 (3)
C1	0.9259 (2)	0.2181 (2)	0.15619 (6)	0.0351 (3)
C2	1.1373 (3)	0.2035 (2)	0.16588 (6)	0.0409 (4)
H2A	1.2229	0.1678	0.1415	0.049*
C3	1.2057 (3)	0.2505 (3)	0.21810 (8)	0.0549 (5)
C4	1.0527 (4)	0.2977 (3)	0.24551 (8)	0.0613 (6)
C5	0.7868 (2)	0.1886 (2)	0.10829 (6)	0.0331 (3)
C6	0.7127 (2)	0.2931 (2)	−0.01001 (6)	0.0348 (3)
C7	0.5979 (3)	0.2597 (3)	−0.06275 (7)	0.0429 (4)
C8	0.6887 (3)	0.3566 (3)	−0.10687 (7)	0.0471 (4)
O3	0.27168 (19)	0.21322 (16)	0.03360 (5)	0.0376 (3)
C9	0.9125 (4)	0.3120 (4)	−0.10852 (10)	0.0646 (6)
H9A	0.960 (5)	0.370 (4)	−0.1390 (12)	0.095 (9)*
H9B	0.924 (6)	0.192 (5)	−0.1161 (14)	0.114*
H9C	1.004 (5)	0.348 (5)	−0.0758 (14)	0.114*
C10	0.5614 (5)	0.3099 (4)	−0.15790 (9)	0.0717 (7)
H3A	1.349 (4)	0.253 (3)	0.2300 (10)	0.064 (7)*
H4A	1.060 (4)	0.340 (4)	0.2795 (11)	0.082 (8)*
H7A	0.591 (3)	0.127 (4)	−0.0702 (9)	0.065 (7)*
H7B	0.467 (5)	0.303 (4)	−0.0612 (11)	0.085 (9)*
H8A	0.680 (3)	0.493 (3)	−0.1014 (9)	0.068 (7)*
H10A	0.566 (4)	0.174 (4)	−0.1630 (11)	0.081*
H10B	0.426 (5)	0.344 (4)	−0.1543 (12)	0.093 (10)*
H10C	0.615 (5)	0.375 (4)	−0.1879 (12)	0.096 (9)*
H3	0.375 (4)	0.210 (3)	0.0532 (10)	0.054 (7)*
H4	0.261 (4)	0.327 (3)	0.0205 (10)	0.064 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0568 (3)	0.0872 (4)	0.0332 (3)	0.0112 (3)	0.0069 (2)	−0.0078 (2)
N1	0.0335 (6)	0.0469 (7)	0.0256 (6)	0.0043 (6)	−0.0004 (5)	0.0002 (6)
N2	0.0428 (7)	0.0371 (7)	0.0262 (6)	−0.0009 (6)	−0.0025 (5)	−0.0018 (5)
O1	0.0340 (6)	0.0745 (9)	0.0364 (6)	0.0005 (6)	0.0022 (5)	−0.0027 (6)
O2	0.0644 (8)	0.0380 (6)	0.0392 (6)	−0.0027 (6)	−0.0043 (6)	0.0000 (5)
C1	0.0401 (8)	0.0401 (8)	0.0248 (7)	0.0004 (6)	0.0027 (6)	0.0027 (6)
C2	0.0392 (9)	0.0543 (10)	0.0282 (8)	−0.0023 (7)	−0.0002 (6)	0.0034 (7)
C3	0.0506 (11)	0.0732 (13)	0.0378 (10)	−0.0087 (10)	−0.0098 (8)	0.0061 (9)
C4	0.0738 (15)	0.0779 (15)	0.0300 (9)	−0.0049 (11)	−0.0045 (9)	−0.0052 (9)
C5	0.0347 (8)	0.0351 (7)	0.0289 (7)	0.0010 (6)	0.0017 (6)	0.0025 (6)
C6	0.0366 (8)	0.0415 (8)	0.0263 (7)	−0.0013 (6)	0.0031 (6)	0.0008 (6)
C7	0.0453 (10)	0.0540 (10)	0.0277 (8)	−0.0046 (8)	−0.0028 (7)	0.0016 (7)
C8	0.0697 (12)	0.0411 (9)	0.0301 (8)	0.0038 (8)	0.0039 (8)	0.0010 (7)
O3	0.0354 (6)	0.0389 (6)	0.0378 (6)	−0.0016 (5)	0.0009 (5)	−0.0010 (5)
C9	0.0687 (14)	0.0740 (15)	0.0538 (13)	−0.0077 (12)	0.0191 (11)	−0.0054 (12)
C10	0.093 (2)	0.0873 (18)	0.0314 (10)	0.0056 (15)	−0.0071 (11)	0.0036 (11)

Geometric parameters (Å, °)

S1—C4	1.695 (2)	C6—C7	1.507 (2)
S1—C1	1.7110 (16)	C7—C8	1.525 (3)
N1—C5	1.349 (2)	C7—H7A	0.99 (3)
N1—N2	1.3883 (17)	C7—H7B	0.92 (3)
N1—H1A	0.8600	C8—C9	1.511 (3)
N2—C6	1.354 (2)	C8—C10	1.524 (3)
N2—H2B	0.8600	C8—H8A	1.02 (3)
O1—C5	1.228 (2)	O3—H3	0.80 (3)
O2—C6	1.222 (2)	O3—H4	0.90 (3)
C1—C2	1.387 (2)	C9—H9A	0.98 (3)
C1—C5	1.474 (2)	C9—H9B	0.91 (3)
C2—C3	1.422 (2)	C9—H9C	1.02 (4)
C2—H2A	0.9300	C10—H10A	1.00 (3)
C3—C4	1.339 (3)	C10—H10B	0.94 (3)
C3—H3A	0.95 (3)	C10—H10C	1.01 (3)
C4—H4A	0.93 (3)		
C4—S1—C1	91.77 (10)	C6—C7—C8	114.11 (15)
C5—N1—N2	120.11 (13)	C6—C7—H7A	110.2 (13)
C5—N1—H1A	119.9	C8—C7—H7A	109.1 (14)
N2—N1—H1A	119.9	C6—C7—H7B	106.1 (18)
C6—N2—N1	119.57 (13)	C8—C7—H7B	108.3 (17)
C6—N2—H2B	120.2	H7A—C7—H7B	109 (2)
N1—N2—H2B	120.2	C9—C8—C10	111.6 (2)
C2—C1—C5	130.92 (15)	C9—C8—C7	112.47 (18)
C2—C1—S1	111.41 (12)	C10—C8—C7	108.98 (19)

C5—C1—S1	117.65 (12)	C9—C8—H8A	106.7 (13)
C1—C2—C3	110.95 (16)	C10—C8—H8A	108.1 (13)
C1—C2—H2A	124.5	C7—C8—H8A	108.9 (13)
C3—C2—H2A	124.5	H3—O3—H4	107 (2)
C4—C3—C2	113.15 (18)	C8—C9—H9A	109.1 (18)
C4—C3—H3A	126.2 (15)	C8—C9—H9B	109 (2)
C2—C3—H3A	120.5 (15)	H9A—C9—H9B	102 (3)
C3—C4—S1	112.71 (16)	C8—C9—H9C	114 (2)
C3—C4—H4A	128.9 (17)	H9A—C9—H9C	110 (3)
S1—C4—H4A	118.2 (17)	H9B—C9—H9C	112 (3)
O1—C5—N1	123.64 (15)	C8—C10—H10A	108.5 (16)
O1—C5—C1	121.31 (15)	C8—C10—H10B	106.6 (19)
N1—C5—C1	115.02 (13)	H10A—C10—H10B	109 (2)
O2—C6—N2	121.14 (14)	C8—C10—H10C	111.1 (17)
O2—C6—C7	124.16 (15)	H10A—C10—H10C	110 (2)
N2—C6—C7	114.69 (14)	H10B—C10—H10C	111 (3)
C5—N1—N2—C6	83.78 (19)	C2—C1—C5—O1	172.76 (17)
C4—S1—C1—C2	0.11 (15)	S1—C1—C5—O1	−9.0 (2)
C4—S1—C1—C5	−178.45 (14)	C2—C1—C5—N1	−5.6 (3)
C5—C1—C2—C3	178.30 (17)	S1—C1—C5—N1	172.61 (12)
S1—C1—C2—C3	0.0 (2)	N1—N2—C6—O2	−4.0 (2)
C1—C2—C3—C4	−0.1 (3)	N1—N2—C6—C7	177.00 (14)
C2—C3—C4—S1	0.2 (3)	O2—C6—C7—C8	52.8 (2)
C1—S1—C4—C3	−0.2 (2)	N2—C6—C7—C8	−128.14 (17)
N2—N1—C5—O1	7.2 (2)	C6—C7—C8—C9	55.0 (2)
N2—N1—C5—C1	−174.48 (13)	C6—C7—C8—C10	179.19 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O3^{i}	0.86	2.08	2.8173 (18)	143
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O3—H3···O1	0.80 (3)	1.95 (3)	2.7416 (18)	171 (2)
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Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*, −*z*; (iii) −*x*+1, −*y*+1, −*z*.

Fig. 1

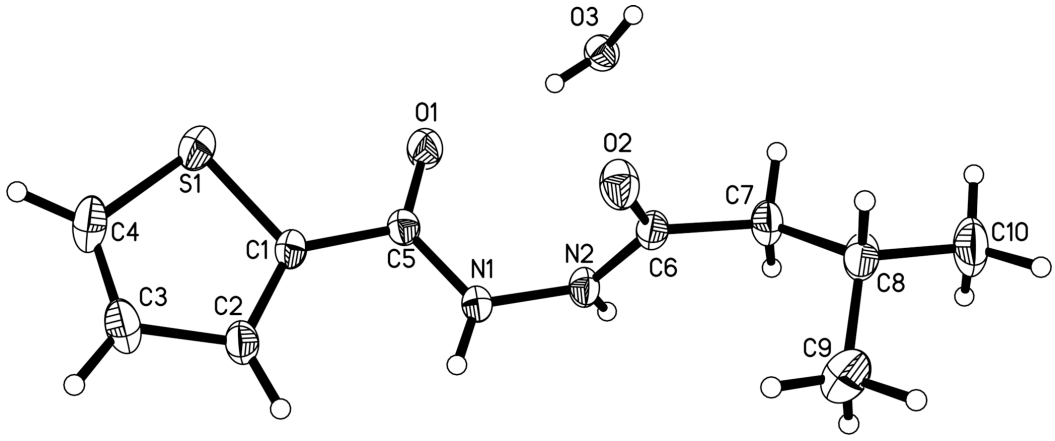

Fig. 2