Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(Butan-2-ylidene)furan-2-carbohydrazide

Bu-wei Ma,[a] Zhen-xin Zhao[b]* and He-ping Li[c]

[a]Department of Architectural Environment, and Energy Engineering, Henan University of Urban Construction, Pingdingshan 467044, People's Republic of China, [b]Department of Chemistry and Chemical Engineering, Henan University of Urban Construction, Pingdingshan 467044, People's Republic of China, and [c]School of Chemistry and Biological Engineering, Guilin University of Technology, People's Republic of China
Correspondence e-mail: zhao_zhenxin@126.com

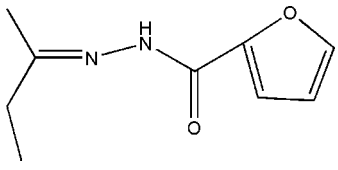
Received 19 September 2010; accepted 23 September 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.101; data-to-parameter ratio = 16.3.

The title Schiff base compound, $C_9H_{12}N_2O_2$, was obtained from a condensation reaction of butan-2-one and furan-2-carbohydrazide. The furan ring and the hydrazide fragment are roughly planar, the largest deviation from the mean plane being 0.069 (2)Å, but the butanylidene group is twisted slightly with respect to this plane by a dihedral angle of 5.2 (3)°. In the crystal, intermolecular N—H···O hydrogen bonds link pairs of inversion-related molecules, forming dimers of $R_2^2(8)$ graph-set motif.

Related literature

For general properties of Schiff bases, see: Kahwa *et al.* (1986); Santos *et al.* (2001). For related structures containing the furan-2-carbohydrazide fragment, see: Jing *et al.* (2007*a*,*b*); Yao & Jing (2007); Bakir & Gyles (2003); Tai *et al.* (2007*a*,*b*); Zhou *et al.* (2007); Butcher *et al.* (2007); Zhao *et al.* (2007). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

$C_9H_{12}N_2O_2$ M_r = 180.21
Monoclinic, $P2_1/c$
a = 8.2664 (15) Å
b = 16.6687 (13) Å
c = 7.5396 (11) Å
β = 113.171 (19)°
V = 955.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.21 × 0.19 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.978, T_{max} = 0.982
4182 measured reflections
1955 independent reflections
761 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.101
S = 0.74
1955 reflections
120 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.16	2.981 (2)	160

Symmetry code: (i) $-x+1, -y+1, -z$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2605).

References

Bakir, M. & Gyles, C. (2003). *J. Mol. Struct.* **649**, 133–135.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (1998). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Butcher, R. J., Jasinski, J. P., Kushawaha, S. K., Bharty, M. K. & Singh, N. K. (2007). *Acta Cryst.* E**63**, o4590–o4591.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Jing, Z.-L., Yu, M. & Chen, X. (2007*a*). *Acta Cryst.* E**63**, o3899.
Jing, Z.-L., Yu, M. & Chen, X. (2007*b*). *Acta Cryst.* E**63**, o3992.
Kahwa, I. A., Selbin, I., Hsieh, T. C. Y. & Laine, R. A. (1986). *Inorg. Chim. Acta*, **118**, 179–185.
Santos, M. L. P., Bagatin, I. A., Pereira, E. M. & Ferreira, A. M. D. C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 838–844.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tai, X.-S., Hao, M.-Y. & Feng, Y.-M. (2007*a*). *Acta Cryst.* E**63**, o2267–o2268.
Tai, X.-S., Yin, J., Hao, M.-Y. & Liang, Z.-P. (2007*b*). *Acta Cryst.* E**63**, o2144–o2145.
Yao, X.-L. & Jing, Z.-L. (2007). *Acta Cryst.* E**63**, o3900.
Zhao, Y.-L., Zhang, Q.-Z., Chen, X. & Yu, M. (2007). *Acta Cryst.* E**63**, o2952–o2953.
Zhou, Q.-L., Wang, C.-L. & Jing, Z.-L. (2007). *Acta Cryst.* E**63**, o898–o899.

supporting information

Acta Cryst. (2010). E**66**, o2657 [doi:10.1107/S1600536810038018]

N′-(Butan-2-ylidene)furan-2-carbohydrazide

Bu-wei Ma, Zhen-xin Zhao and He-ping Li

S1. Comment

The chemistry of Schiff base has attracted a great deal of interest in recent years. These compounds play an important role in the development of various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). In this paper, we synthesized the title compound and reported its crystal structure of the title compound.

The molecular structure of (I) adopts an E conformation with respect to the C=N double bond (Fig.1). The furan ring and the the C5/N1/N2/C6 group are roughly planar with the largest deviation from the mean plane being 0.069 (2)Å but the butan C6/C7/C8/C9 group is slightly twisted with respect to this plane by a dihedral angle of 5.2 (3)°. Distances and bond angles within the furan and the hydrazide moiety agree with related structures found in the literature (Jing *et al.,* 2007*a*,*b*; Yao & Jing, 2007; Bakir & Gyles, 2003; Tai *et al.*, 2007*a*,*b*; Zhou *et al.*, 2007; Butcher *et al.*, 2007; Zhao *et al.*, 2007.

Intermolecular N—H···O hydrogen bonds link the molecules two by two around inversion centers to form dimers with a $R_2^2(8)$ graph set motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995) (Table 1, Fig. 2).

S2. Experimental

Furan-2-carbohydrazine (1 mmol, 0.126 g) was dissolved in anhydrous ethanol (10 ml), The mixture was stirred for several minitutes at 351k, butan-2-one(1 mmol, 0.072 g) in ethanol (8 mm l) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized from methanol/dicholomethane(1:1), colorless single crystals of (I) was obtained after 3 d.

S3. Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.96 Å (methyl), 0.97 Å (methylene), 0.93Å (aromatic) and N—H = 0.86 Å with U_{iso}(H) = 1.2U_{eq}(C or N) or U_{iso}(H) = 1.5U_{eq}(Cmethyl).

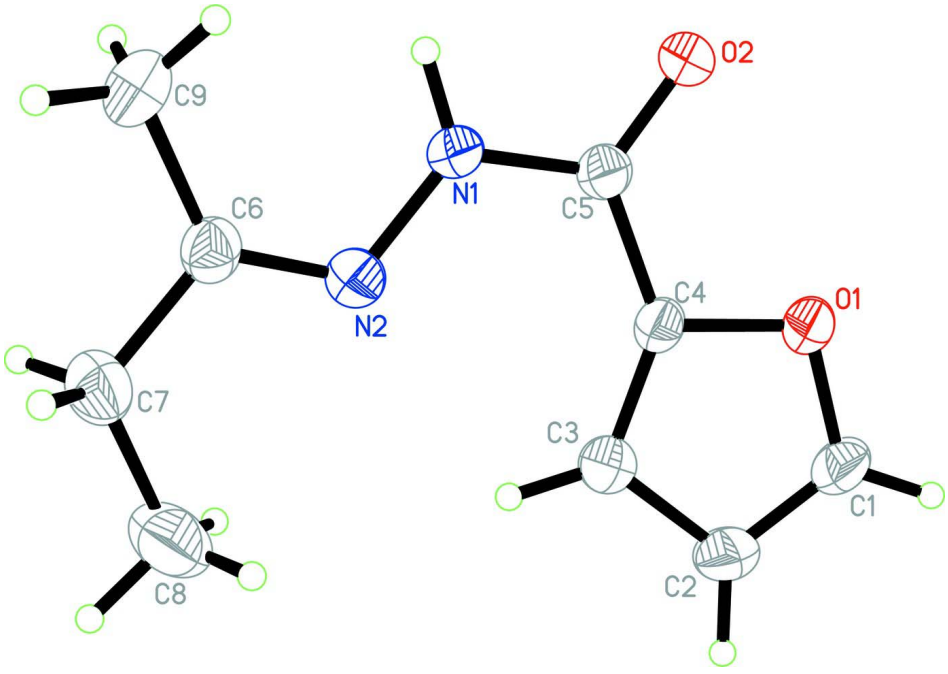


Figure 1

Molecular view of the title compound with the atom labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small sphere of arbitrary radii.

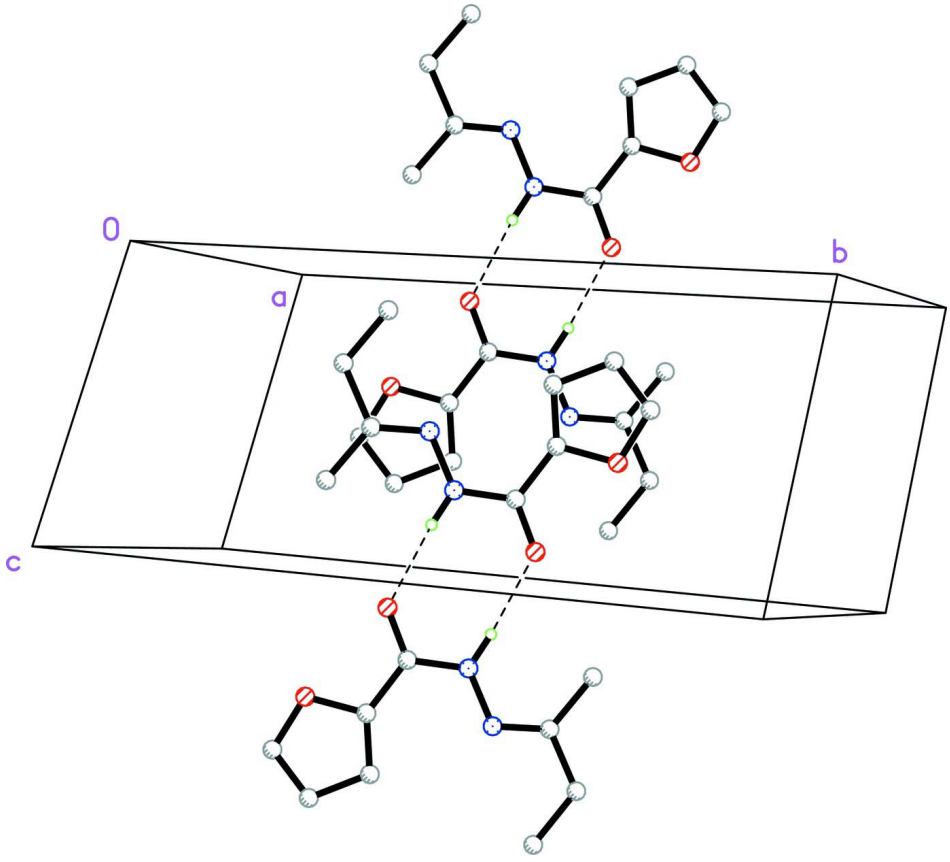


Figure 2

Partial packing view showing the formation of dimer through N-H···O hydogen bonds shown as dashed lines. H atoms not involved in hydrogen bondings have been omitted for clarity.

N′-(Butan-2-ylidene)furan-2-carbohydrazide

Crystal data

$C_9H_{12}N_2O_2$
$M_r = 180.21$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.2664\ (15)$ Å
$b = 16.6687\ (13)$ Å
$c = 7.5396\ (11)$ Å
$\beta = 113.171\ (19)°$
$V = 955.1\ (2)$ Å^3
$Z = 4$
$F(000) = 384$
$D_x = 1.253$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1520 reflections
$\theta = 3.1–28.8°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, colorless
0.21 × 0.19 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.978$, $T_{max} = 0.982$
4182 measured reflections
1955 independent reflections
761 reflections with $I > 2\sigma(I)$

R_{int} = 0.040
θ_{max} = 26.4°, θ_{min} = 3.2°
h = −11→9
k = −18→21
l = −6→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.101
S = 0.74
1955 reflections
120 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.4993 (2)	0.32297 (8)	0.4042 (2)	0.0598 (5)
O2	0.4694 (2)	0.41298 (8)	0.1056 (2)	0.0670 (6)
N1	0.6592 (3)	0.50672 (9)	0.2784 (3)	0.0511 (6)
H1	0.6497	0.5314	0.1746	0.061*
N2	0.7678 (3)	0.53804 (11)	0.4554 (3)	0.0505 (6)
C1	0.5264 (4)	0.29006 (14)	0.5768 (4)	0.0586 (8)
H1B	0.4800	0.2411	0.5932	0.070*
C2	0.6274 (4)	0.33618 (14)	0.7195 (4)	0.0638 (8)
H2B	0.6641	0.3262	0.8509	0.077*
C3	0.6686 (3)	0.40406 (13)	0.6316 (4)	0.0582 (7)
H3A	0.7383	0.4473	0.6956	0.070*
C4	0.5899 (3)	0.39476 (11)	0.4416 (3)	0.0437 (6)
C5	0.5674 (3)	0.43790 (12)	0.2653 (3)	0.0473 (7)
C6	0.8540 (3)	0.60130 (13)	0.4535 (3)	0.0502 (7)
C7	0.9668 (4)	0.63698 (13)	0.6436 (4)	0.0669 (8)
H7A	0.9302	0.6921	0.6457	0.080*
H7B	1.0872	0.6381	0.6529	0.080*
C8	0.9648 (4)	0.59519 (17)	0.8197 (4)	0.0933 (10)
H8A	1.0343	0.6251	0.9329	0.140*
H8B	1.0128	0.5422	0.8278	0.140*
H8C	0.8459	0.5916	0.8108	0.140*
C9	0.8539 (4)	0.64420 (13)	0.2796 (4)	0.0806 (10)

H9A	0.8447	0.6059	0.1810	0.121*
H9B	0.9613	0.6740	0.3134	0.121*
H9C	0.7557	0.6803	0.2325	0.121*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0962 (15)	0.0377 (8)	0.0454 (11)	−0.0067 (9)	0.0276 (10)	0.0008 (7)
O2	0.1069 (16)	0.0467 (9)	0.0373 (11)	−0.0157 (9)	0.0174 (11)	−0.0023 (8)
N1	0.0748 (15)	0.0400 (10)	0.0386 (12)	−0.0059 (11)	0.0224 (11)	0.0015 (9)
N2	0.0599 (15)	0.0458 (11)	0.0440 (13)	−0.0015 (10)	0.0186 (11)	−0.0015 (9)
C1	0.085 (2)	0.0456 (13)	0.0495 (18)	−0.0007 (14)	0.0312 (16)	0.0107 (12)
C2	0.077 (2)	0.0659 (16)	0.0435 (17)	−0.0090 (15)	0.0182 (16)	0.0110 (13)
C3	0.067 (2)	0.0543 (14)	0.0472 (17)	−0.0152 (13)	0.0155 (15)	0.0028 (12)
C4	0.0572 (18)	0.0318 (12)	0.0431 (15)	0.0011 (11)	0.0208 (13)	0.0017 (10)
C5	0.0661 (19)	0.0369 (13)	0.0406 (16)	0.0038 (13)	0.0228 (15)	−0.0010 (11)
C6	0.0518 (18)	0.0415 (13)	0.0561 (17)	0.0021 (13)	0.0199 (14)	0.0008 (11)
C7	0.060 (2)	0.0655 (16)	0.069 (2)	−0.0095 (14)	0.0184 (17)	−0.0050 (14)
C8	0.092 (3)	0.123 (2)	0.059 (2)	−0.0321 (19)	0.0227 (18)	−0.0138 (18)
C9	0.101 (3)	0.0641 (17)	0.075 (2)	−0.0200 (16)	0.0328 (19)	0.0117 (14)

Geometric parameters (Å, º)

O1—C1	1.347 (2)	C4—C5	1.457 (3)
O1—C4	1.381 (2)	C6—C7	1.492 (3)
O2—C5	1.229 (2)	C6—C9	1.493 (3)
N1—C5	1.357 (3)	C7—C8	1.505 (3)
N1—N2	1.384 (2)	C7—H7A	0.9700
N1—H1	0.8600	C7—H7B	0.9700
N2—C6	1.276 (3)	C8—H8A	0.9600
C1—C2	1.319 (3)	C8—H8B	0.9600
C1—H1B	0.9300	C8—H8C	0.9600
C2—C3	1.419 (3)	C9—H9A	0.9600
C2—H2B	0.9300	C9—H9B	0.9600
C3—C4	1.329 (3)	C9—H9C	0.9600
C3—H3A	0.9300		
C1—O1—C4	106.56 (17)	N2—C6—C9	126.8 (2)
C5—N1—N2	121.40 (19)	C7—C6—C9	115.9 (2)
C5—N1—H1	119.3	C6—C7—C8	116.3 (2)
N2—N1—H1	119.3	C6—C7—H7A	108.2
C6—N2—N1	117.00 (19)	C8—C7—H7A	108.2
C2—C1—O1	111.2 (2)	C6—C7—H7B	108.2
C2—C1—H1B	124.4	C8—C7—H7B	108.2
O1—C1—H1B	124.4	H7A—C7—H7B	107.4
C1—C2—C3	106.0 (2)	C7—C8—H8A	109.5
C1—C2—H2B	127.0	C7—C8—H8B	109.5
C3—C2—H2B	127.0	H8A—C8—H8B	109.5

C4—C3—C2	107.7 (2)	C7—C8—H8C	109.5
C4—C3—H3A	126.1	H8A—C8—H8C	109.5
C2—C3—H3A	126.1	H8B—C8—H8C	109.5
C3—C4—O1	108.49 (19)	C6—C9—H9A	109.5
C3—C4—C5	139.3 (2)	C6—C9—H9B	109.5
O1—C4—C5	112.16 (19)	H9A—C9—H9B	109.5
O2—C5—N1	119.4 (2)	C6—C9—H9C	109.5
O2—C5—C4	121.6 (2)	H9A—C9—H9C	109.5
N1—C5—C4	118.9 (2)	H9B—C9—H9C	109.5
N2—C6—C7	117.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.16	2.981 (2)	160

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.