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N′-[4-(2-Furyl)but-3-en-2-ylidene]isonicotinohydrazide

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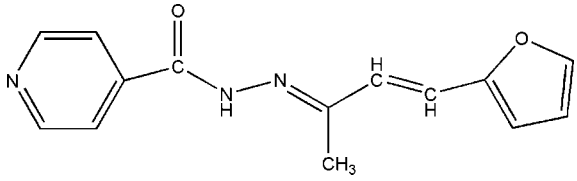
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.132; data-to-parameter ratio = 18.2.

The molecule of the title Schiff base compound, $C_{14}H_{13}N_3O_2$, is not perfectly planar; the furyl and pyridine rings are twisted with respect to each other along the $C_4N_2C_2$ organic chain, making a dihedral angle of 13.3 (1)°. The occurence of N—H···O hydrogen bonds builds up a chain developing parallel to the c axis.

Related literature

For background, see: Kahwa *et al.* (1986); Santos *et al.* (2001).



Experimental

Crystal data

$C_{14}H_{13}N_3O_2$
M_r = 255.27
Monoclinic, $P2_1/c$
a = 16.6325 (14) Å
b = 9.3572 (8) Å
c = 8.3554 (7) Å
β = 100.912 (1)°
V = 1276.87 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.25 × 0.23 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.965, T_{max} = 0.978
11728 measured reflections
3151 independent reflections
2124 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.132
S = 1.03
3151 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.26	2.9289 (16)	134

Symmetry code: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2389).

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supporting information

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S1. Comment

The chemistry of Schiff bases 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). As part of our interest in the study of the coordination chemistry of Schiff bases, we have synthesized the title compound (I) and reported its cyrstal structure.

The molecule of the title compound is not perfectly planar, the furyl and the pyridine rings are twisted to each other along the C5/C6/C7/C8/N1/N2/C9 organic chain making a dihedral angle of 13.3 (1)°(Fig. 1). The organic chain is nearly planar with the largest deviation from the mean plane being 0.039 (1)Å at C7. The occurence of N-H···O hydrogen bonds builts up a chain developing parallel to the c axis (Fig. 2, Table 1).

S2. Experimental

Pyridine-4-carboxylic acid hydrazide (1 mmol, 0.137 g) was dissolved in anhydrous methanol, H_2SO_4 (98% 0.5 ml) was added to this, the mixture was stirred for several minitutes at 351 K, furylideneacetone (1 mmol 0.136 g) in methanol (8 ml) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized in dichloromethane, brown single crystals of (I) was obtained after 5 d.

S3. Refinement

All H atoms were treated as riding on their parent atoms. Methyl H atoms were placed in calculated position with C—H=0.96Å and refined with U_{iso}(H)=1.5U_{eq}(C) Other H atoms were placed in calculated positions with C—H=0.93 Å, N—H=0.86Å and U_{iso}(H)=1.2U_{eq}(C,*N*).

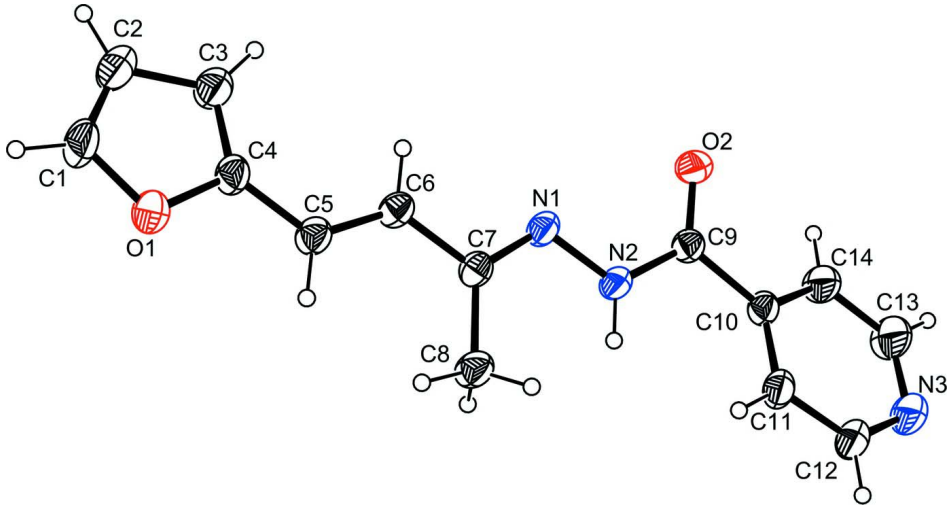

Figure 1

Molecular view of (I) with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

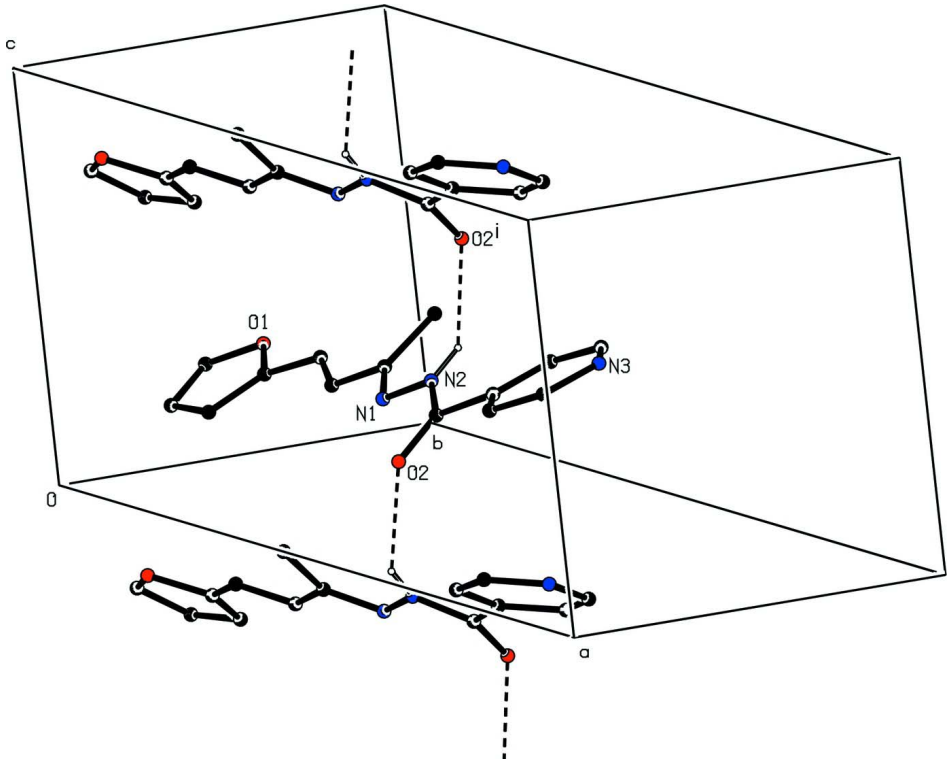

Figure 2

Partial packing view showing the N-H···O hydrogen bondings resulting in the formation of a chain parallel to the c axis. H atoms not involved in hydrogen bondings have been omitted for clarity. H bonds are shown as dashed lines. [Symmetry code: (i) x, -y+3/2, z+1/2]

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Crystal data

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Hall symbol: -P 2ybc
$a = 16.6325\ (14)$ Å
$b = 9.3572\ (8)$ Å
$c = 8.3554\ (7)$ Å
$\beta = 100.912\ (1)°$
$V = 1276.87\ (19)$ Å^3
$Z = 4$
$F(000) = 536$
$D_x = 1.328$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2552 reflections
$\theta = 2.2–24.8°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, brown
0.25 × 0.23 × 0.16 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)
$T_{min} = 0.965$, $T_{max} = 0.978$
11728 measured reflections
3151 independent reflections
2124 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.5°$
$h = -22\rightarrow20$
$k = -12\rightarrow12$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.132$
$S = 1.03$
3151 reflections
173 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
$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.1613P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	−0.34240 (10)	0.8729 (3)	0.0347 (2)	0.0765 (6)
H1	−0.3937	0.9084	0.0428	0.092*
C2	−0.33013 (10)	0.7542 (2)	−0.0421 (2)	0.0679 (5)
H2	−0.3700	0.6929	−0.0970	0.081*

C3	−0.24415 (10)	0.73836 (19)	−0.0245 (2)	0.0606 (4)
H3	−0.2168	0.6641	−0.0654	0.073*
C4	−0.20947 (9)	0.85094 (17)	0.06234 (19)	0.0510 (4)
C5	−0.12679 (9)	0.89496 (16)	0.12619 (19)	0.0496 (4)
H5	−0.1190	0.9739	0.1949	0.060*
C6	−0.06062 (8)	0.82982 (14)	0.09315 (18)	0.0454 (3)
H6	−0.0694	0.7556	0.0178	0.055*
C7	0.02404 (8)	0.86343 (14)	0.16352 (17)	0.0424 (3)
C8	0.04425 (9)	0.98435 (16)	0.2800 (2)	0.0547 (4)
H8A	0.0616	0.9475	0.3883	0.082*
H8B	−0.0033	1.0432	0.2765	0.082*
H8C	0.0875	1.0403	0.2503	0.082*
C9	0.21798 (8)	0.73017 (15)	0.13795 (17)	0.0439 (3)
C10	0.30344 (8)	0.77406 (15)	0.21280 (17)	0.0450 (3)
C11	0.32441 (9)	0.90777 (17)	0.2786 (2)	0.0540 (4)
H11	0.2842	0.9759	0.2829	0.065*
C12	0.40575 (10)	0.93870 (19)	0.3378 (2)	0.0653 (5)
H12	0.4186	1.0289	0.3821	0.078*
C13	0.44536 (11)	0.7198 (2)	0.2728 (3)	0.0838 (6)
H13	0.4868	0.6536	0.2703	0.101*
C14	0.36583 (10)	0.67795 (19)	0.2110 (2)	0.0651 (5)
H14	0.3547	0.5865	0.1689	0.078*
N1	0.07718 (7)	0.77927 (12)	0.11825 (15)	0.0466 (3)
N2	0.15805 (7)	0.80590 (12)	0.18733 (15)	0.0475 (3)
H2A	0.1701	0.8704	0.2613	0.057*
N3	0.46678 (9)	0.84786 (19)	0.3355 (2)	0.0772 (5)
O1	−0.26987 (7)	0.93681 (14)	0.10076 (15)	0.0705 (4)
O2	0.20525 (6)	0.63417 (12)	0.03656 (13)	0.0573 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0337 (9)	0.1172 (16)	0.0746 (13)	0.0063 (9)	0.0004 (8)	−0.0027 (12)
C2	0.0450 (10)	0.0840 (12)	0.0703 (12)	−0.0101 (9)	−0.0003 (8)	0.0038 (10)
C3	0.0477 (9)	0.0634 (9)	0.0681 (11)	−0.0004 (7)	0.0043 (8)	−0.0046 (8)
C4	0.0366 (8)	0.0627 (9)	0.0519 (9)	0.0068 (6)	0.0039 (6)	0.0025 (7)
C5	0.0401 (8)	0.0524 (8)	0.0533 (9)	0.0031 (6)	0.0012 (6)	−0.0001 (7)
C6	0.0390 (8)	0.0452 (7)	0.0489 (8)	0.0002 (6)	0.0002 (6)	0.0010 (6)
C7	0.0375 (7)	0.0415 (7)	0.0463 (8)	0.0010 (5)	0.0033 (6)	0.0039 (6)
C8	0.0425 (8)	0.0518 (8)	0.0649 (10)	0.0036 (6)	−0.0024 (7)	−0.0087 (7)
C9	0.0383 (7)	0.0502 (7)	0.0421 (8)	0.0003 (6)	0.0045 (6)	0.0005 (6)
C10	0.0357 (7)	0.0553 (8)	0.0434 (8)	−0.0004 (6)	0.0059 (6)	0.0005 (6)
C11	0.0382 (8)	0.0560 (9)	0.0693 (10)	−0.0045 (6)	0.0138 (7)	−0.0041 (8)
C12	0.0436 (9)	0.0708 (10)	0.0827 (12)	−0.0121 (8)	0.0147 (8)	−0.0144 (9)
C13	0.0405 (10)	0.0959 (14)	0.1109 (17)	0.0142 (9)	0.0039 (10)	−0.0275 (13)
C14	0.0449 (9)	0.0705 (10)	0.0769 (12)	0.0066 (8)	0.0039 (8)	−0.0174 (9)
N1	0.0329 (6)	0.0516 (6)	0.0526 (7)	−0.0027 (5)	0.0010 (5)	−0.0038 (5)
N2	0.0332 (6)	0.0528 (7)	0.0540 (7)	−0.0018 (5)	0.0021 (5)	−0.0111 (6)

N3	0.0382 (8)	0.0978 (12)	0.0931 (12)	−0.0041 (7)	0.0058 (7)	−0.0236 (9)
O1	0.0407 (6)	0.0913 (9)	0.0753 (8)	0.0138 (6)	0.0007 (5)	−0.0177 (7)
O2	0.0467 (6)	0.0676 (7)	0.0548 (7)	0.0016 (5)	0.0025 (5)	−0.0162 (5)

Geometric parameters (Å, º)

C1—C2	1.317 (3)	C8—H8C	0.9600
C1—O1	1.366 (2)	C9—O2	1.2253 (17)
C1—H1	0.9300	C9—N2	1.3496 (18)
C2—C3	1.417 (2)	C9—C10	1.4980 (19)
C2—H2	0.9300	C10—C14	1.375 (2)
C3—C4	1.346 (2)	C10—C11	1.385 (2)
C3—H3	0.9300	C11—C12	1.380 (2)
C4—O1	1.3711 (18)	C11—H11	0.9300
C4—C5	1.438 (2)	C12—N3	1.327 (2)
C5—C6	1.332 (2)	C12—H12	0.9300
C5—H5	0.9300	C13—N3	1.330 (2)
C6—C7	1.4540 (19)	C13—C14	1.382 (2)
C6—H6	0.9300	C13—H13	0.9300
C7—N1	1.2926 (18)	C14—H14	0.9300
C7—C8	1.489 (2)	N1—N2	1.3822 (15)
C8—H8A	0.9600	N2—H2A	0.8600
C8—H8B	0.9600		

C2—C1—O1	111.08 (16)	H8B—C8—H8C	109.5
C2—C1—H1	124.5	O2—C9—N2	123.73 (13)
O1—C1—H1	124.5	O2—C9—C10	121.05 (13)
C1—C2—C3	106.49 (16)	N2—C9—C10	115.20 (12)
C1—C2—H2	126.8	C14—C10—C11	117.48 (14)
C3—C2—H2	126.8	C14—C10—C9	118.32 (14)
C4—C3—C2	107.16 (16)	C11—C10—C9	124.15 (13)
C4—C3—H3	126.4	C12—C11—C10	119.20 (15)
C2—C3—H3	126.4	C12—C11—H11	120.4
C3—C4—O1	109.08 (13)	C10—C11—H11	120.4
C3—C4—C5	134.95 (15)	N3—C12—C11	124.13 (16)
O1—C4—C5	115.91 (13)	N3—C12—H12	117.9
C6—C5—C4	124.18 (14)	C11—C12—H12	117.9
C6—C5—H5	117.9	N3—C13—C14	124.65 (17)
C4—C5—H5	117.9	N3—C13—H13	117.7
C5—C6—C7	126.31 (14)	C14—C13—H13	117.7
C5—C6—H6	116.8	C10—C14—C13	118.77 (16)
C7—C6—H6	116.8	C10—C14—H14	120.6
N1—C7—C6	114.42 (12)	C13—C14—H14	120.6
N1—C7—C8	124.89 (12)	C7—N1—N2	115.67 (11)
C6—C7—C8	120.68 (12)	C9—N2—N1	119.63 (12)
C7—C8—H8A	109.5	C9—N2—H2A	120.2
C7—C8—H8B	109.5	N1—N2—H2A	120.2
H8A—C8—H8B	109.5	C12—N3—C13	115.76 (15)

C7—C8—H8C	109.5	C1—O1—C4	106.18 (14)
H8A—C8—H8C	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2^{i}	0.86	2.26	2.9289 (16)	134

Symmetry code: (i) $x, -y+3/2, z+1/2$.