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(*E*)-*N*′-[(5-Methylfuran-2-yl)methylene]-furan-2-carbohydrazide

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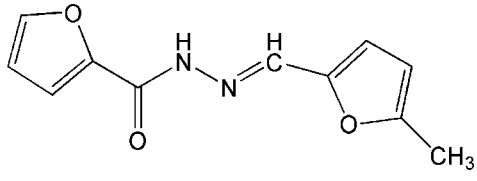
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.126; data-to-parameter ratio = 13.2.

In the title compound, $C_{11}H_{10}N_2O_3$, the dihedral angle between the two furan rings is 4.2 (2)°. Intermolecular N—H···O and C—H···O hydrogen bonds link the molecules into a two-dimensional network. Strong π–π stacking interactions are present between inversion-related furan rings, with a centroid-to-centroid distance of 3.4866 (14) Å.

Related literature

For general background, see: Belloni *et al.* (2005); Kahwa *et al.* (1986); Parashar *et al.* (1988); Santos *et al.* (2001); Tynan *et al.* (2005).



Experimental

Crystal data

$C_{11}H_{10}N_2O_3$
M_r = 218.21
Orthorhombic, *Pbca*
a = 11.402 (2) Å
b = 7.9941 (16) Å
c = 24.039 (5) Å
V = 2191.1 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.14 × 0.10 × 0.06 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.986, T_{max} = 0.994
12318 measured reflections
1931 independent reflections
1673 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.126
S = 1.12
1931 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.86	2.09	2.902 (2)	157
C9—H9···O2^{ii}	0.93	2.58	3.473 (3)	161

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); 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: *CrystalStructure* (Rigaku/MSC, 2005); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2448).

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supplementary materials

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(*E*)-*N*'-[(5-Methylfuran-2-yl)methylene]furan-2-carbohydrazide

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Comment

In order to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable (Belloni *et al.*, 2005; Tynan *et al.*, 2005; Parashar *et al.*, 1988), the synthesis of new and designed crystal structures has become a major strand of modern chemistry. Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*,2001). As part of an investigation of the coordination properties of Schiff bases functioning as ligands, we report the synthesis and crystal structure of the title compound, (I).

In the molecular structure of the compound (I) (Fig. 1), the geometric parameters are normal. The O1/C2—C5 furan ring is planar, with an r.m.s. deviation for the fitted atoms of 0.004 Å, as is the furan ring O3/C8—C11, with an r.m.s. deviation of 0.003 Å. The dihedral angle between these two planes is 4.2 (2)°. The O2/C7/N2/N1/C6 plane (r.m.s. deviation 0.035 Å) forms dihedral angles of 3.1 (1)° and 5.1 (1)°, respectively, with the O1/C2—C5 and O3/C8—C11 furan rings.

Intermolecular N—H···O hydrogen bonds link the molecules into chains running along the *b* axis. These chains are cross-linked *via* C—H···O hydrogen bonds (Table 1) forming a two-dimensional network structure, as illustrated in Fig. 2. In addition, π-π stacking interactions involving the inversion-related O3/C8—C11 furan rings, with a centroid-to-centroid distance of 3.4866 (14) Å is observed.

Experimental

An anhydrous ethanol solution (50 ml) of 5-methylfuran-2-carbaldehyde (1.10 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of furan-2-carbohydrazide (1.26 g, 10 mmol), and the mixture was stirred at 350 K for 6 h under N_2, yielding a colourless precipitate. The product was isolated, recrystallized from anhydrous ethanol and then dried *in vacuo* to give pure compound (I) in 92% yield. Colourless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Refinement

H atoms were included in calculated positions [N—H = 0.86 Å, C—H = 0.93 (aromatic) or 0.96 Å (methyl)] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C, N) or 1.5U_{eq}(C) for methyl H atoms.

Figures

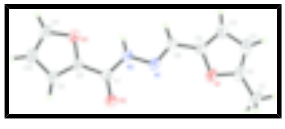

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

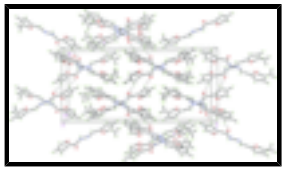

Fig. 2. The crystal packing of (I), viwed down the *b* axis. Hydrogen bonds are indicated by dashed lines.

(*E*)-*N*'-[(5-Methylfuran-2-yl)methylene]furan-2-carbohydrazide

Crystal data

$C_{11}H_{10}N_2O_3$	F_{000} = 912
M_r = 218.21	D_x = 1.323 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 8064 reflections
a = 11.402 (2) Å	θ = 1.8–27.4º
b = 7.9941 (16) Å	μ = 0.10 mm^{-1}
c = 24.039 (5) Å	*T* = 293 (2) K
V = 2191.1 (7) Å^3	Block, colourless
Z = 8	0.14 × 0.10 × 0.06 mm

Data collection

Rigaku Saturn diffractometer	1931 independent reflections
Radiation source: rotating anode	1673 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.044
T = 293(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −13→13
T_{min} = 0.986, T_{max} = 0.994	*k* = −9→8
12318 measured reflections	*l* = −28→26

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.051	H-atom parameters constrained
$wR(F^2)$ = 0.126	$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.3175P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.12	$(\Delta/\sigma)_{max}$ = 0.001
1931 reflections	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
146 parameters	$\Delta\rho_{min}$ = −0.19 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}
O1	0.82709 (12)	0.11965 (16)	0.75358 (5)	0.0600 (4)
O2	0.61482 (11)	0.07779 (15)	0.57388 (5)	0.0553 (4)
O3	0.67924 (12)	0.45165 (16)	0.49989 (5)	0.0633 (4)
N1	0.75995 (13)	0.22824 (19)	0.64816 (5)	0.0504 (4)
N2	0.73032 (13)	0.29844 (18)	0.59727 (5)	0.0506 (4)
H2	0.7579	0.3945	0.5878	0.061*
C1	0.8569 (2)	−0.0544 (4)	0.83485 (10)	0.1005 (9)
H1A	0.9007	−0.0553	0.8690	0.151*
H1B	0.8811	−0.1468	0.8120	0.151*
H1C	0.7748	−0.0642	0.8429	0.151*
C2	0.87878 (18)	0.1036 (3)	0.80507 (8)	0.0669 (6)
C3	0.9411 (2)	0.2403 (3)	0.81651 (9)	0.0780 (7)
H3	0.9849	0.2594	0.8485	0.094*
C4	0.9284 (2)	0.3510 (3)	0.77109 (9)	0.0749 (6)
H4	0.9612	0.4570	0.7677	0.090*
C5	0.85954 (16)	0.2732 (2)	0.73358 (7)	0.0550 (5)
C6	0.81970 (16)	0.3244 (2)	0.67981 (7)	0.0532 (5)
H6	0.8379	0.4315	0.6674	0.064*
C7	0.65803 (15)	0.2147 (2)	0.56285 (7)	0.0450 (4)
C8	0.63024 (15)	0.2980 (2)	0.51031 (7)	0.0480 (4)
C9	0.55807 (19)	0.2560 (3)	0.46885 (7)	0.0618 (5)
H9	0.5140	0.1585	0.4662	0.074*
C10	0.5615 (2)	0.3884 (3)	0.42979 (8)	0.0738 (6)
H10	0.5208	0.3943	0.3963	0.089*
C11	0.6341 (2)	0.5024 (3)	0.45028 (8)	0.0730 (6)
H11	0.6519	0.6034	0.4330	0.088*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0632 (8)	0.0663 (9)	0.0505 (8)	0.0005 (6)	−0.0081 (6)	0.0187 (6)
O2	0.0660 (8)	0.0460 (7)	0.0538 (8)	−0.0036 (6)	0.0003 (6)	0.0027 (5)

O3	0.0770 (9)	0.0610 (9)	0.0520 (8)	−0.0064 (7)	−0.0096 (6)	0.0144 (6)
N1	0.0594 (9)	0.0488 (9)	0.0431 (8)	0.0021 (7)	−0.0040 (7)	0.0120 (7)
N2	0.0634 (9)	0.0434 (8)	0.0449 (8)	−0.0035 (7)	−0.0054 (7)	0.0119 (6)
C1	0.0980 (18)	0.125 (2)	0.0788 (16)	0.0043 (16)	−0.0065 (14)	0.0576 (16)
C2	0.0590 (12)	0.0928 (16)	0.0490 (11)	0.0119 (11)	−0.0041 (9)	0.0228 (11)
C3	0.0727 (14)	0.112 (2)	0.0490 (12)	0.0048 (14)	−0.0152 (10)	0.0068 (12)
C4	0.0829 (15)	0.0836 (16)	0.0583 (13)	−0.0145 (12)	−0.0127 (11)	0.0051 (11)
C5	0.0606 (11)	0.0566 (11)	0.0479 (10)	−0.0004 (9)	−0.0040 (9)	0.0099 (8)
C6	0.0633 (11)	0.0492 (11)	0.0472 (10)	−0.0014 (9)	−0.0032 (9)	0.0069 (8)
C7	0.0503 (9)	0.0429 (10)	0.0420 (9)	0.0062 (8)	0.0050 (7)	0.0011 (7)
C8	0.0575 (10)	0.0445 (10)	0.0422 (9)	0.0057 (8)	0.0025 (8)	0.0008 (7)
C9	0.0807 (13)	0.0561 (12)	0.0487 (11)	0.0034 (10)	−0.0073 (10)	−0.0061 (8)
C10	0.1039 (17)	0.0730 (14)	0.0445 (11)	0.0125 (13)	−0.0174 (11)	−0.0015 (10)
C11	0.1045 (18)	0.0669 (14)	0.0477 (11)	0.0048 (13)	−0.0100 (11)	0.0181 (10)

Geometric parameters (Å, °)

O1—C5	1.369 (2)	C3—C4	1.413 (3)
O1—C2	1.377 (2)	C3—H3	0.93
O2—C7	1.229 (2)	C4—C5	1.347 (3)
O3—C11	1.361 (2)	C4—H4	0.93
O3—C8	1.372 (2)	C5—C6	1.430 (2)
N1—C6	1.278 (2)	C6—H6	0.93
N1—N2	1.3876 (18)	C7—C8	1.463 (2)
N2—C7	1.346 (2)	C8—C9	1.335 (2)
N2—H2	0.86	C9—C10	1.415 (3)
C1—C2	1.473 (3)	C9—H9	0.93
C1—H1A	0.96	C10—C11	1.326 (3)
C1—H1B	0.96	C10—H10	0.93
C1—H1C	0.96	C11—H11	0.93
C2—C3	1.332 (3)		
C5—O1—C2	106.47 (16)	C4—C5—O1	109.64 (17)
C11—O3—C8	105.83 (15)	C4—C5—C6	131.16 (19)
C6—N1—N2	114.29 (15)	O1—C5—C6	119.21 (16)
C7—N2—N1	119.33 (15)	N1—C6—C5	122.38 (17)
C7—N2—H2	120.3	N1—C6—H6	118.8
N1—N2—H2	120.3	C5—C6—H6	118.8
C2—C1—H1A	109.5	O2—C7—N2	123.75 (15)
C2—C1—H1B	109.5	O2—C7—C8	120.33 (16)
H1A—C1—H1B	109.5	N2—C7—C8	115.91 (16)
C2—C1—H1C	109.5	C9—C8—O3	109.85 (16)
H1A—C1—H1C	109.5	C9—C8—C7	131.57 (18)
H1B—C1—H1C	109.5	O3—C8—C7	118.48 (15)
C3—C2—O1	109.71 (18)	C8—C9—C10	106.88 (19)
C3—C2—C1	133.9 (2)	C8—C9—H9	126.6
O1—C2—C1	116.4 (2)	C10—C9—H9	126.6
C2—C3—C4	107.44 (19)	C11—C10—C9	106.52 (18)
C2—C3—H3	126.3	C11—C10—H10	126.7
C4—C3—H3	126.3	C9—C10—H10	126.7

C5—C4—C3	106.7 (2)	C10—C11—O3	110.90 (19)
C5—C4—H4	126.6	C10—C11—H11	124.5
C3—C4—H4	126.6	O3—C11—H11	124.5
C6—N1—N2—C7	−173.65 (15)	N1—N2—C7—O2	0.6 (3)
C5—O1—C2—C3	−0.3 (2)	N1—N2—C7—C8	179.41 (14)
C5—O1—C2—C1	−179.49 (19)	C11—O3—C8—C9	0.0 (2)
O1—C2—C3—C4	0.8 (3)	C11—O3—C8—C7	−176.96 (16)
C1—C2—C3—C4	179.8 (3)	O2—C7—C8—C9	3.0 (3)
C2—C3—C4—C5	−1.0 (3)	N2—C7—C8—C9	−175.80 (18)
C3—C4—C5—O1	0.8 (3)	O2—C7—C8—O3	179.23 (15)
C3—C4—C5—C6	−178.7 (2)	N2—C7—C8—O3	0.4 (2)
C2—O1—C5—C4	−0.3 (2)	O3—C8—C9—C10	0.4 (2)
C2—O1—C5—C6	179.21 (17)	C7—C8—C9—C10	176.85 (19)
N2—N1—C6—C5	−179.21 (16)	C8—C9—C10—C11	−0.7 (3)
C4—C5—C6—N1	175.4 (2)	C9—C10—C11—O3	0.7 (3)
O1—C5—C6—N1	−4.0 (3)	C8—O3—C11—C10	−0.5 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.86	2.09	2.902 (2)	157
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Fig. 1

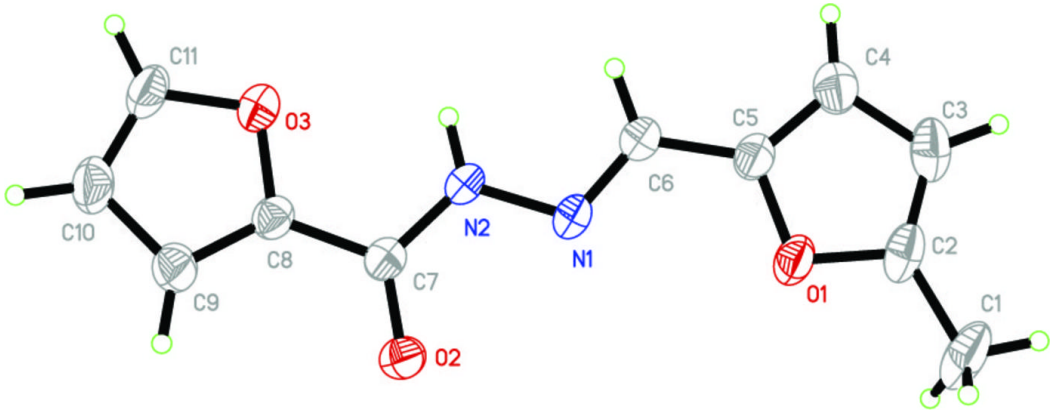

Fig. 2