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N'-(2,6-Dichlorobenzylidene)furan-2-carbohydrazide

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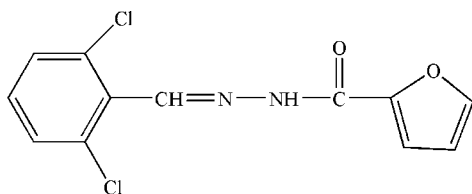
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.047; wR factor = 0.126; data-to-parameter ratio = 15.1.

In the title compound, $\text{C}_{12}\text{H}_8\text{Cl}_2\text{N}_2\text{O}_2$, the dihedral angle between the furan and benzene rings is $72.90(16)^\circ$. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, generating $C(4)$ chains propagating in $[100]$.

Related literature

 For related structures, see: Okabe *et al.* (1993); Ohba (1996); Bakir & Gyles (2003).


Experimental

Crystal data

 $\text{C}_{12}\text{H}_8\text{Cl}_2\text{N}_2\text{O}_2$
 $M_r = 283.10$

 Monoclinic, $P2_1/c$
 $a = 4.9046(3)$ Å

 $b = 19.1113(12)$ Å
 $c = 12.9469(9)$ Å
 $\beta = 91.565(5)^\circ$
 $V = 1213.10(14)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.53$ mm⁻¹
 $T = 293$ K

 $0.21 \times 0.18 \times 0.17$ mm

Data collection

 Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
 $T_{\min} = 0.814$, $T_{\max} = 0.847$

 4654 measured reflections
 2464 independent reflections
 1679 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.126$
 $S = 1.01$
 2464 reflections

 163 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}^i$	0.86	2.07	2.890 (2)	159

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; 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: HB6741).

References

- Bakir, M. & Gyles, C. (2003). *J. Mol. Struct.* **649**, 133–135.
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 Ohba, S. (1996). *Acta Cryst.* **C52**, 2118–2119.
 Okabe, N., Nakamura, T. & Fukuda, H. (1993). *Acta Cryst.* **C49**, 1678–1680.
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supporting information

Acta Cryst. (2012). E68, o1455 [doi:10.1107/S160053681201639X]

***N'*-(2,6-Dichlorobenzylidene)furan-2-carbohydrazide**

Jun Xu

S1. Comment

Several phenylhydrazone derivatives have been shown to be potentially DNA-damaging and are mutagenic agents (Okabe *et al.*, 1993). As part of our ongoing studies of these materials, we synthesized the title compound, (I), and the crystal structure is presented herein. In the molecular structure of the compound, the molecular is not planar, the furyl ring makes a dihedral angle of 72.90 (16) $^{\circ}$ with the benzene ring. Bond lengths and angles are in agreement with other hydrazone derivatives (Ohba, 1996; Bakir & Gyles, 2003).

In the crystal, molecules are linked by N—H \cdots O hydrogen bonds, generating one-dimensional chains.

S2. Experimental

Furan-2-carbohydrazine (1 mmol, 0.126 g) was dissolved in anhydrous ethanol (10 ml), The mixture was stirred for several minutes at 351k, 2,6-Dichlorobenzaldehyde (1 mmol, 0.175 g) in ethanol (20 mm l) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized from DMF. Colorless blocks were obtained by slow evaporation of the compound dissolved in a mixture of ethanol and DMF.

S3. Refinement

All H atoms were positioned geometrically and refined as riding with C—H=0.93 (aromatic), 0.97(methylene) and N—H=0.86 Å, with $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{CH, CH}_2 \text{ or NH})$.

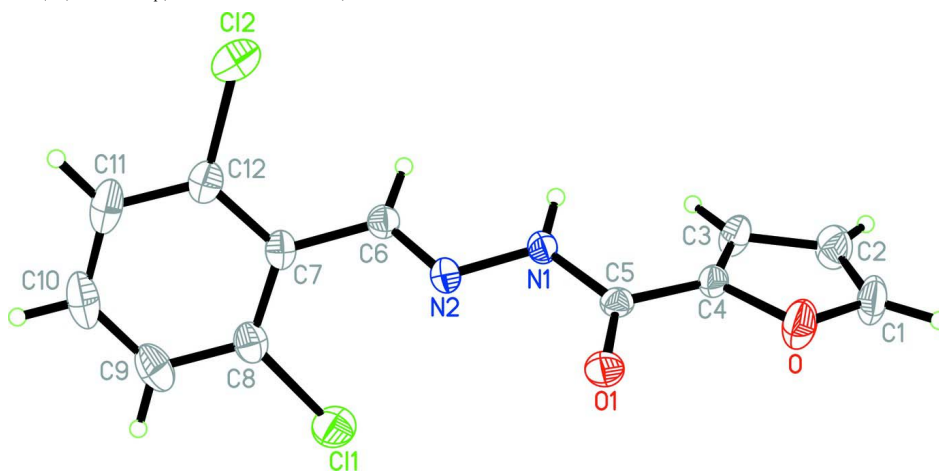
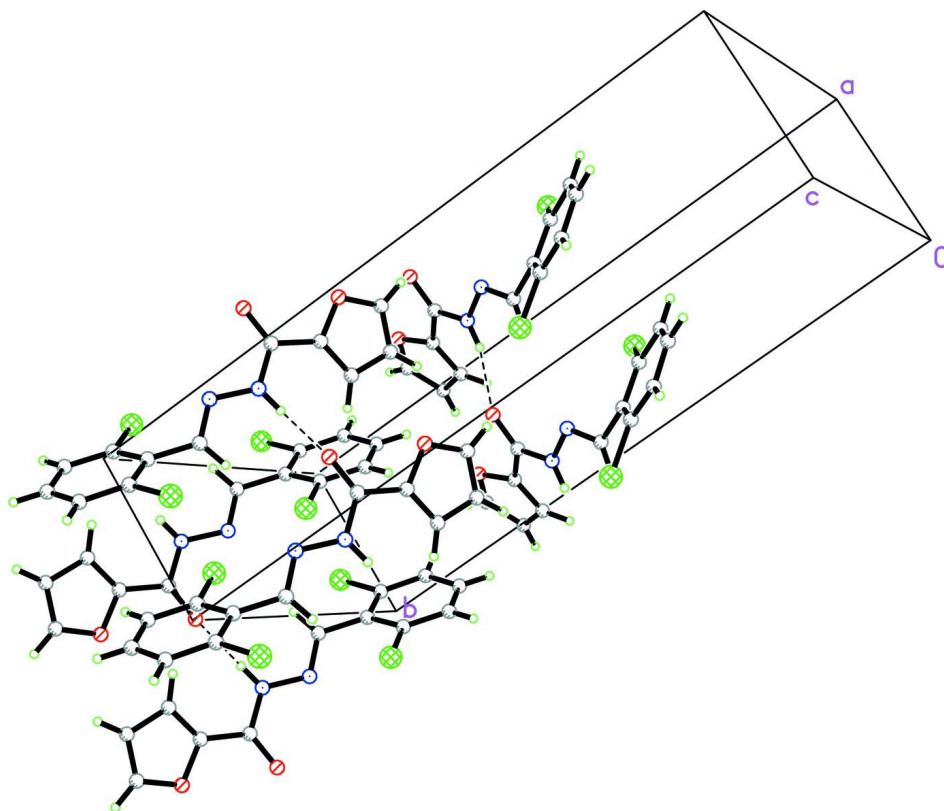


Figure 1

View of (I). Displacement ellipsoids are drawn at the 30% probability level, showing the intramolecular hydrogen bonds as dashed lines.

**Figure 2**

The molecular packing of the title compound,

N'-(2,6-Dichlorobenzylidene)furan-2-carbohydrazide

Crystal data

$C_{12}H_8Cl_2N_2O_2$

$M_r = 283.10$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 4.9046$ (3) Å

$b = 19.1113$ (12) Å

$c = 12.9469$ (9) Å

$\beta = 91.565$ (5)°

$V = 1213.10$ (14) Å³

$Z = 4$

$F(000) = 576$

$D_x = 1.550$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2790 reflections

$\theta = 3.2$ – 26.3 °

$\mu = 0.53$ mm⁻¹

$T = 293$ K

Block, colorless

$0.21 \times 0.18 \times 0.17$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 1998)

$T_{\min} = 0.814$, $T_{\max} = 0.847$

4654 measured reflections

2464 independent reflections

1679 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\text{max}} = 26.4$ °, $\theta_{\text{min}} = 3.3$ °

$h = -5 \rightarrow 6$

$k = -22 \rightarrow 23$

$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.126$
 $S = 1.01$
 2464 reflections
 163 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
 $w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.2769P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.43228 (17)	0.43978 (4)	0.09622 (7)	0.0649 (3)
Cl2	-0.28672 (16)	0.52357 (5)	0.38143 (7)	0.0679 (3)
O1	0.5843 (3)	0.68141 (9)	0.10167 (15)	0.0429 (5)
N1	0.1507 (4)	0.63974 (10)	0.09980 (16)	0.0345 (5)
H1A	-0.0175	0.6482	0.0838	0.041*
C5	0.3496 (5)	0.68267 (12)	0.06671 (19)	0.0322 (6)
N2	0.2201 (4)	0.58266 (10)	0.15913 (16)	0.0342 (5)
O	0.4344 (4)	0.78012 (11)	-0.04747 (17)	0.0591 (6)
C7	0.0788 (5)	0.47943 (13)	0.2441 (2)	0.0367 (6)
C4	0.2594 (5)	0.72916 (12)	-0.0172 (2)	0.0341 (6)
C12	-0.0570 (5)	0.46354 (15)	0.3342 (2)	0.0452 (7)
C6	0.0226 (5)	0.54460 (13)	0.1877 (2)	0.0357 (6)
H6A	-0.1564	0.5581	0.1729	0.043*
C3	0.0404 (5)	0.72915 (15)	-0.0800 (2)	0.0464 (7)
H3A	-0.1086	0.6993	-0.0761	0.056*
C11	-0.0117 (7)	0.40168 (19)	0.3879 (3)	0.0630 (9)
H11A	-0.1082	0.3920	0.4471	0.076*
C8	0.2643 (5)	0.42899 (14)	0.2112 (2)	0.0440 (7)
C9	0.3144 (6)	0.36796 (16)	0.2651 (3)	0.0596 (9)
H9A	0.4412	0.3358	0.2418	0.071*
C1	0.3139 (6)	0.81122 (17)	-0.1316 (3)	0.0603 (9)
H1B	0.3901	0.8477	-0.1686	0.072*
C10	0.1756 (7)	0.35512 (19)	0.3533 (3)	0.0698 (10)
H10A	0.2094	0.3141	0.3901	0.084*
C2	0.0749 (6)	0.78259 (17)	-0.1535 (2)	0.0578 (8)

H2B -0.0466 0.7951 -0.2068 0.069*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0791 (6)	0.0484 (5)	0.0682 (6)	0.0052 (4)	0.0217 (4)	-0.0113 (4)
C12	0.0658 (5)	0.0828 (7)	0.0559 (5)	-0.0035 (4)	0.0182 (4)	-0.0036 (5)
O1	0.0292 (9)	0.0457 (11)	0.0537 (12)	-0.0007 (7)	-0.0008 (8)	0.0043 (10)
N1	0.0292 (10)	0.0329 (11)	0.0413 (13)	0.0004 (8)	0.0004 (9)	0.0082 (11)
C5	0.0318 (12)	0.0296 (12)	0.0355 (14)	0.0012 (9)	0.0062 (10)	-0.0048 (12)
N2	0.0384 (11)	0.0289 (10)	0.0352 (12)	-0.0006 (9)	-0.0006 (9)	0.0026 (10)
O	0.0528 (11)	0.0557 (13)	0.0682 (15)	-0.0172 (9)	-0.0062 (10)	0.0223 (12)
C7	0.0410 (13)	0.0336 (13)	0.0352 (14)	-0.0083 (10)	-0.0076 (11)	0.0036 (12)
C4	0.0382 (12)	0.0274 (12)	0.0371 (14)	-0.0015 (10)	0.0076 (11)	0.0011 (12)
C12	0.0477 (15)	0.0479 (17)	0.0399 (16)	-0.0100 (12)	-0.0039 (12)	0.0042 (14)
C6	0.0349 (13)	0.0362 (13)	0.0359 (15)	-0.0011 (10)	-0.0002 (11)	0.0022 (12)
C3	0.0424 (14)	0.0544 (17)	0.0424 (16)	-0.0126 (12)	-0.0027 (12)	0.0150 (15)
C11	0.074 (2)	0.067 (2)	0.0473 (19)	-0.0242 (18)	-0.0090 (16)	0.0222 (18)
C8	0.0497 (15)	0.0348 (14)	0.0470 (17)	-0.0055 (12)	-0.0073 (13)	0.0009 (14)
C9	0.0660 (19)	0.0359 (16)	0.076 (2)	0.0018 (13)	-0.0177 (17)	0.0025 (17)
C1	0.070 (2)	0.0499 (18)	0.061 (2)	-0.0085 (15)	0.0033 (17)	0.0271 (18)
C10	0.085 (2)	0.051 (2)	0.072 (3)	-0.0080 (17)	-0.025 (2)	0.025 (2)
C2	0.0581 (18)	0.064 (2)	0.0505 (19)	0.0017 (15)	-0.0056 (14)	0.0179 (18)

Geometric parameters (Å, °)

C11—C8	1.733 (3)	C12—C11	1.387 (4)
C12—C12	1.731 (3)	C6—H6A	0.9300
O1—C5	1.226 (3)	C3—C2	1.410 (4)
N1—C5	1.353 (3)	C3—H3A	0.9300
N1—N2	1.372 (3)	C11—C10	1.363 (5)
N1—H1A	0.8600	C11—H11A	0.9300
C5—C4	1.463 (3)	C8—C9	1.378 (4)
N2—C6	1.275 (3)	C9—C10	1.367 (4)
O—C1	1.362 (4)	C9—H9A	0.9300
O—C4	1.363 (3)	C1—C2	1.318 (4)
C7—C12	1.392 (4)	C1—H1B	0.9300
C7—C8	1.400 (4)	C10—H10A	0.9300
C7—C6	1.466 (3)	C2—H2B	0.9300
C4—C3	1.329 (4)		
C5—N1—N2	119.29 (19)	C4—C3—H3A	126.2
C5—N1—H1A	120.4	C2—C3—H3A	126.2
N2—N1—H1A	120.4	C10—C11—C12	119.4 (3)
O1—C5—N1	123.3 (2)	C10—C11—H11A	120.3
O1—C5—C4	123.3 (2)	C12—C11—H11A	120.3
N1—C5—C4	113.4 (2)	C9—C8—C7	122.5 (3)
C6—N2—N1	115.94 (19)	C9—C8—C11	117.0 (2)

C1—O—C4	106.2 (2)	C7—C8—C11	120.5 (2)
C12—C7—C8	115.8 (2)	C10—C9—C8	119.3 (3)
C12—C7—C6	121.0 (2)	C10—C9—H9A	120.4
C8—C7—C6	123.3 (2)	C8—C9—H9A	120.4
C3—C4—O	109.2 (2)	C2—C1—O	110.8 (3)
C3—C4—C5	132.6 (2)	C2—C1—H1B	124.6
O—C4—C5	117.9 (2)	O—C1—H1B	124.6
C11—C12—C7	122.2 (3)	C11—C10—C9	120.8 (3)
C11—C12—C12	119.0 (2)	C11—C10—H10A	119.6
C7—C12—C12	118.8 (2)	C9—C10—H10A	119.6
N2—C6—C7	119.7 (2)	C1—C2—C3	106.1 (3)
N2—C6—H6A	120.2	C1—C2—H2B	126.9
C7—C6—H6A	120.2	C3—C2—H2B	126.9
C4—C3—C2	107.7 (2)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1A...O1 ⁱ	0.86	2.07	2.890 (2)	159

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