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

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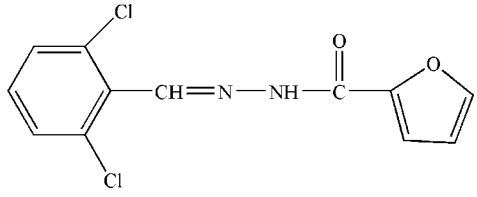
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.047; wR factor = 0.126; data-to-parameter ratio = 15.1.

In the title compound, $C_{12}H_8Cl_2N_2O_2$, the dihedral angle between the furan and benzene rings is 72.90 (16)°. In the crystal, molecules are linked by N—H···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

$C_{12}H_8Cl_2N_2O_2$
M_r = 283.10
Monoclinic, $P2_1/c$
a = 4.9046 (3) Å
b = 19.1113 (12) Å
c = 12.9469 (9) Å
β = 91.565 (5)°
V = 1213.10 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.53 mm^{-1}
T = 293 K
0.21 × 0.18 × 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_{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
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	2.07	2.890 (2)	159

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

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: HB6741).

References

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Ohba, S. (1996). *Acta Cryst.* C**52**, 2118–2119.
Okabe, N., Nakamura, T. & Fukuda, H. (1993). *Acta Cryst.* C**49**, 1678–1680.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2012). E**68**, o1455 [doi:10.1107/S160053681201639X]

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

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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)° 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···O hydrogen bonds, generating one-dimensional chains.

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, 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 dissoived in a mixture of ethanol and DMF.

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_{iso}(H)=1.2U_{eq}(CH, CH2 or NH).

Computing details

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

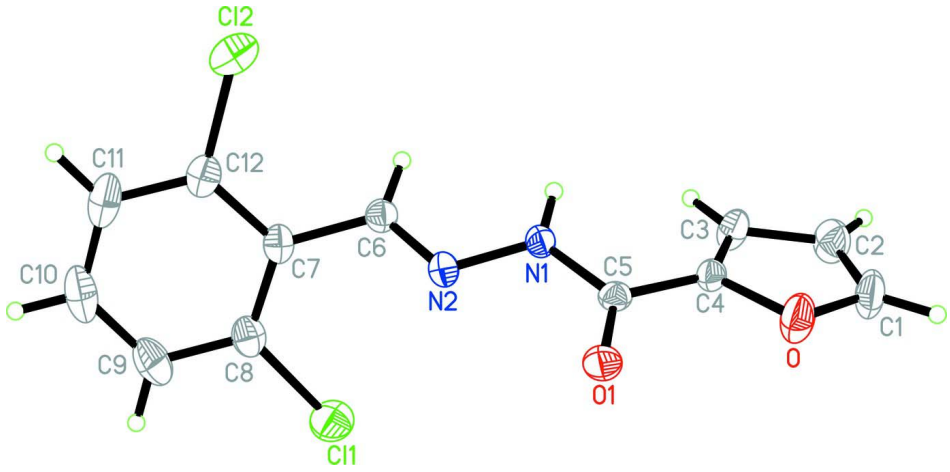

Figure 1

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

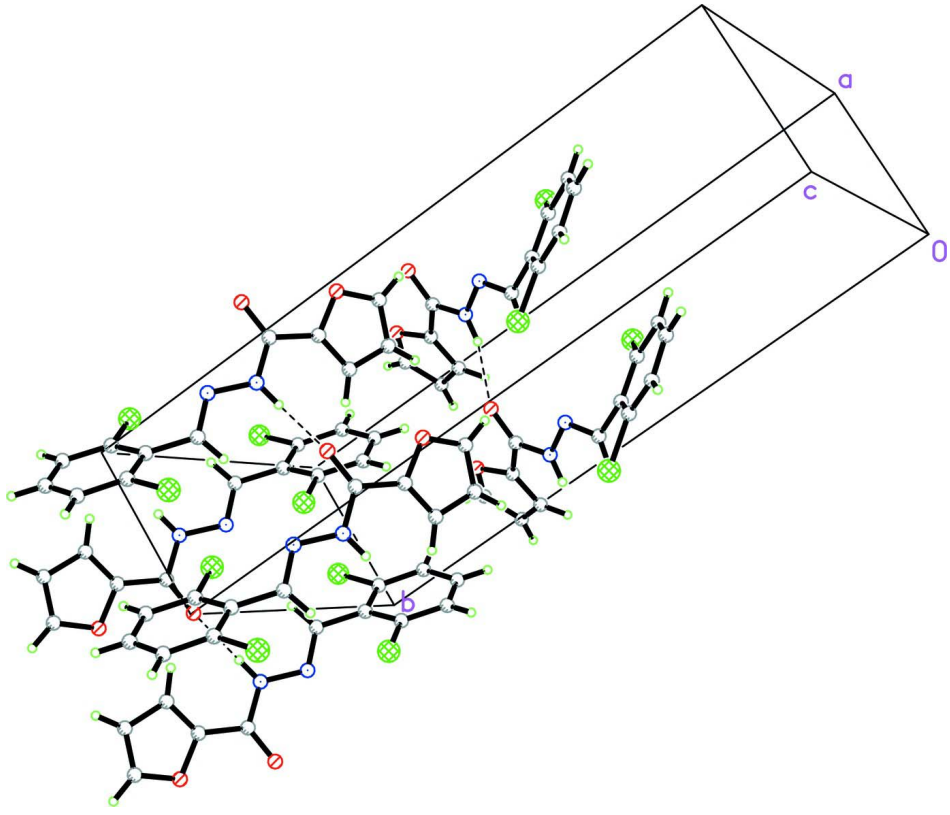

Figure 2

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) Å^3
$Z = 4$
$F(000) = 576$
$D_x = 1.550$ 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 × 0.18 × 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 $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.3°$
$h = -5 \rightarrow 6$
$k = -22 \rightarrow 23$
$l = -16 \rightarrow 15$

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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{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}
Cl1	0.0791 (6)	0.0484 (5)	0.0682 (6)	0.0052 (4)	0.0217 (4)	−0.0113 (4)
Cl2	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, º)

Cl1—C8	1.733 (3)	C12—C11	1.387 (4)
Cl2—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—Cl1	117.0 (2)
C1—O—C4	106.2 (2)	C7—C8—Cl1	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—Cl2	119.0 (2)	C11—C10—H10A	119.6
C7—C12—Cl2	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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.86	2.07	2.890 (2)	159

Symmetry code: (i) *x*−1, *y*, *z*.