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N'-(3,4-Dimethylbenzylidene)furan-2-carbohydrazide

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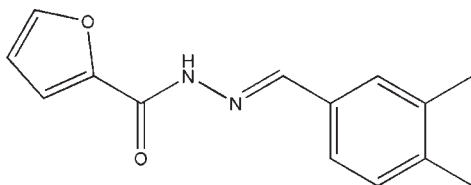
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.066; wR factor = 0.177; data-to-parameter ratio = 17.9.

The title compound, $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2$, was prepared by the reaction of 3,4-dimethylbenzaldehyde and furan-2-carbohydrazide. The dihedral angle between the aromatic rings is $35.48(14)^\circ$. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, generating $C(4)$ chains propagating in $[010]$.

Related literature

For a related structure, see: Li & Jian (2010).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2$
 $M_r = 242.27$

 Orthorhombic, *Pbca*

$a = 17.655(3)$ Å
 $b = 7.6304(15)$ Å
 $c = 19.020(4)$ Å
 $V = 2562.3(9)$ Å³

 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.09$ mm⁻¹
 $T = 293$ K

 $0.22 \times 0.21 \times 0.18$ mm

Data collection

Bruker SMART CCD
 diffractometer
 21744 measured reflections

2921 independent reflections
 1563 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.127$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.177$
 $S = 0.90$

2921 reflections

163 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.15$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O2}^i$	0.86	2.06	2.921 (3)	174

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5553).

References

- Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Li, Y.-F. & Jian, F.-F. (2010). *Acta Cryst.* **E66**, o1720.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2010). E66, o2061 [https://doi.org/10.1107/S1600536810027959]

N'-(3,4-Dimethylbenzylidene)furan-2-carbohydrazide

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S1. Experimental

A mixture of 3,4-dimethylbenzaldehyde (0.1 mol), and furan-2-carbohydrazide (0.1 mol) was stirred in refluxing ethanol (20 mL) for 2 h to afford the title compound (0.090 mol, yield 90%). colourless blocks of (I) were obtained by recrystallization from ethanol at room temperature.

S2. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances=0.97 Å, and with $U_{\text{iso}}=1.2-1.5U_{\text{eq}}$.

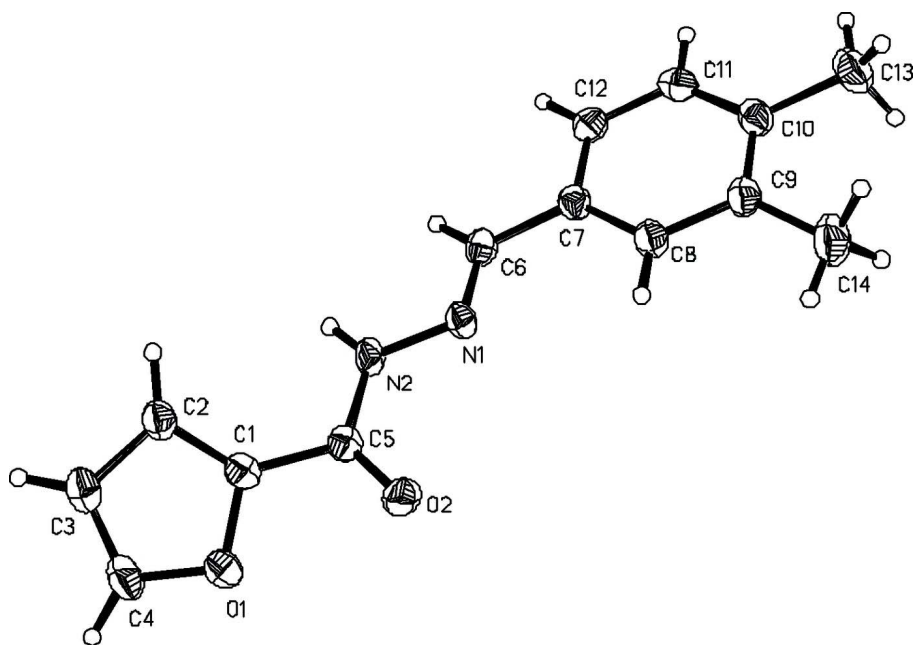


Figure 1

The molecular structure of (I) showing 30% probability displacement ellipsoids.

N'-(3,4-Dimethylbenzylidene)furan-2-carbohydrazide

Crystal data

$\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2$

$M_r = 242.27$

Orthorhombic, *Pbca*

Hall symbol: $-P\ 2ac\ 2ab$

$a = 17.655\ (3)\ \text{\AA}$

$b = 7.6304\ (15)\ \text{\AA}$

$c = 19.020\ (4)\ \text{\AA}$

$V = 2562.3\ (9)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1024$

$D_x = 1.256 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 1563 reflections
 $\theta = 3.1\text{--}27.5^\circ$

$\mu = 0.09 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Block, colorless
 $0.22 \times 0.21 \times 0.18 \text{ mm}$

Data collection

Bruker SMART CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 phi and ω scans
 21744 measured reflections
 2921 independent reflections

1563 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.127$
 $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.1^\circ$
 $h = -22 \rightarrow 22$
 $k = -9 \rightarrow 9$
 $l = -24 \rightarrow 23$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.177$
 $S = 0.90$
 2921 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
 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$
O2	0.34251 (10)	0.2908 (2)	0.40087 (10)	0.0493 (5)
N2	0.23842 (12)	0.1209 (2)	0.38067 (10)	0.0404 (5)
H2A	0.2147	0.0251	0.3897	0.048*
N1	0.20989 (11)	0.2401 (3)	0.33290 (10)	0.0391 (5)
C1	0.32632 (14)	0.0301 (3)	0.46664 (13)	0.0393 (6)
C6	0.15127 (14)	0.1915 (3)	0.29934 (12)	0.0412 (6)
H6A	0.1308	0.0813	0.3079	0.049*
C5	0.30454 (14)	0.1586 (3)	0.41282 (12)	0.0384 (6)
C7	0.11563 (14)	0.3062 (3)	0.24746 (12)	0.0397 (6)
C9	0.11430 (15)	0.5784 (3)	0.18082 (13)	0.0456 (7)
C4	0.40945 (19)	-0.1050 (4)	0.53228 (14)	0.0580 (8)
H4A	0.4554	-0.1425	0.5508	0.070*
C8	0.14661 (15)	0.4693 (3)	0.23064 (13)	0.0439 (7)

H8A	0.1903	0.5058	0.2536	0.053*
C2	0.28720 (16)	-0.0713 (3)	0.51175 (13)	0.0469 (7)
H2B	0.2348	-0.0819	0.5147	0.056*
C12	0.04994 (14)	0.2541 (3)	0.21381 (12)	0.0441 (7)
H12A	0.0277	0.1472	0.2249	0.053*
C11	0.01738 (15)	0.3619 (4)	0.16343 (13)	0.0475 (7)
H11A	-0.0265	0.3252	0.1407	0.057*
C3	0.34193 (18)	-0.1595 (4)	0.55411 (14)	0.0532 (7)
H3A	0.3323	-0.2394	0.5899	0.064*
C14	0.1495 (2)	0.7546 (4)	0.16590 (19)	0.0728 (10)
H14A	0.1940	0.7691	0.1944	0.109*
H14B	0.1138	0.8455	0.1767	0.109*
H14C	0.1632	0.7614	0.1171	0.109*
C13	0.01177 (19)	0.6353 (4)	0.09024 (15)	0.0709 (9)
H13A	-0.0325	0.5771	0.0724	0.106*
H13B	0.0470	0.6543	0.0525	0.106*
H13C	-0.0025	0.7459	0.1103	0.106*
O1	0.40208 (10)	0.0140 (3)	0.47873 (9)	0.0542 (6)
C10	0.04847 (15)	0.5226 (4)	0.14611 (12)	0.0459 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0437 (11)	0.0356 (10)	0.0687 (12)	-0.0029 (9)	0.0024 (8)	0.0115 (9)
N2	0.0499 (14)	0.0268 (11)	0.0444 (12)	-0.0013 (10)	-0.0043 (10)	0.0097 (9)
N1	0.0449 (13)	0.0310 (12)	0.0414 (12)	0.0036 (10)	-0.0041 (10)	0.0055 (9)
C1	0.0420 (15)	0.0323 (14)	0.0437 (14)	0.0024 (12)	-0.0047 (11)	-0.0028 (10)
C6	0.0483 (16)	0.0307 (14)	0.0446 (15)	-0.0021 (12)	0.0013 (12)	0.0039 (11)
C5	0.0420 (15)	0.0307 (14)	0.0424 (14)	0.0062 (12)	0.0032 (11)	-0.0005 (11)
C7	0.0424 (15)	0.0371 (15)	0.0395 (13)	0.0048 (11)	0.0014 (11)	0.0012 (11)
C9	0.0524 (17)	0.0404 (16)	0.0439 (14)	0.0035 (13)	0.0026 (12)	0.0060 (11)
C4	0.064 (2)	0.059 (2)	0.0503 (16)	0.0179 (17)	-0.0090 (14)	0.0095 (14)
C8	0.0466 (16)	0.0403 (16)	0.0448 (15)	0.0000 (13)	-0.0052 (12)	0.0042 (11)
C2	0.0533 (17)	0.0425 (16)	0.0450 (15)	-0.0056 (13)	-0.0088 (12)	0.0069 (12)
C12	0.0444 (16)	0.0435 (16)	0.0444 (15)	-0.0036 (13)	0.0012 (12)	-0.0010 (11)
C11	0.0379 (15)	0.0606 (19)	0.0440 (14)	0.0055 (14)	-0.0016 (11)	-0.0060 (13)
C3	0.068 (2)	0.0452 (17)	0.0461 (16)	0.0005 (15)	-0.0108 (14)	0.0060 (13)
C14	0.084 (2)	0.050 (2)	0.085 (2)	-0.0049 (18)	-0.0098 (18)	0.0256 (16)
C13	0.071 (2)	0.085 (2)	0.0564 (17)	0.0207 (19)	-0.0098 (16)	0.0175 (16)
O1	0.0446 (11)	0.0575 (13)	0.0604 (12)	0.0110 (10)	-0.0020 (9)	0.0143 (9)
C10	0.0486 (16)	0.0547 (18)	0.0345 (13)	0.0138 (14)	0.0026 (11)	0.0035 (12)

Geometric parameters (Å, °)

O2—C5	1.232 (3)	C4—H4A	0.9300
N2—C5	1.349 (3)	C8—H8A	0.9300
N2—N1	1.381 (3)	C2—C3	1.427 (4)
N2—H2A	0.8600	C2—H2B	0.9300

N1—C6	1.271 (3)	C12—C11	1.388 (4)
C1—C2	1.346 (4)	C12—H12A	0.9300
C1—O1	1.363 (3)	C11—C10	1.383 (4)
C1—C5	1.469 (3)	C11—H11A	0.9300
C6—C7	1.462 (3)	C3—H3A	0.9300
C6—H6A	0.9300	C14—H14A	0.9600
C7—C12	1.383 (3)	C14—H14B	0.9600
C7—C8	1.396 (4)	C14—H14C	0.9600
C9—C8	1.384 (3)	C13—C10	1.513 (4)
C9—C10	1.403 (4)	C13—H13A	0.9600
C9—C14	1.508 (4)	C13—H13B	0.9600
C4—C3	1.329 (4)	C13—H13C	0.9600
C4—O1	1.371 (3)		
C5—N2—N1	118.3 (2)	C3—C2—H2B	126.8
C5—N2—H2A	120.9	C7—C12—C11	119.8 (2)
N1—N2—H2A	120.9	C7—C12—H12A	120.1
C6—N1—N2	115.8 (2)	C11—C12—H12A	120.1
C2—C1—O1	110.1 (2)	C10—C11—C12	121.7 (3)
C2—C1—C5	133.9 (2)	C10—C11—H11A	119.1
O1—C1—C5	115.8 (2)	C12—C11—H11A	119.1
N1—C6—C7	121.0 (2)	C4—C3—C2	106.5 (3)
N1—C6—H6A	119.5	C4—C3—H3A	126.8
C7—C6—H6A	119.5	C2—C3—H3A	126.8
O2—C5—N2	124.2 (2)	C9—C14—H14A	109.5
O2—C5—C1	122.2 (2)	C9—C14—H14B	109.5
N2—C5—C1	113.6 (2)	H14A—C14—H14B	109.5
C12—C7—C8	118.6 (2)	C9—C14—H14C	109.5
C12—C7—C6	120.1 (2)	H14A—C14—H14C	109.5
C8—C7—C6	121.3 (2)	H14B—C14—H14C	109.5
C8—C9—C10	118.7 (2)	C10—C13—H13A	109.5
C8—C9—C14	119.7 (3)	C10—C13—H13B	109.5
C10—C9—C14	121.6 (2)	H13A—C13—H13B	109.5
C3—C4—O1	110.8 (3)	C10—C13—H13C	109.5
C3—C4—H4A	124.6	H13A—C13—H13C	109.5
O1—C4—H4A	124.6	H13B—C13—H13C	109.5
C9—C8—C7	122.1 (2)	C1—O1—C4	106.2 (2)
C9—C8—H8A	118.9	C11—C10—C9	119.1 (2)
C7—C8—H8A	118.9	C11—C10—C13	120.1 (3)
C1—C2—C3	106.5 (3)	C9—C10—C13	120.9 (3)
C1—C2—H2B	126.8		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A \cdots O2 ⁱ	0.86	2.06	2.921 (3)	174

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