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Methyl 3-(2-furylmethylidene)carbazate

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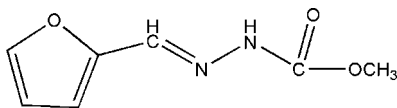
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å;
 R factor = 0.072; wR factor = 0.186; data-to-parameter ratio = 17.0.

The asymmetric unit of the title compound, $\text{C}_7\text{H}_8\text{N}_2\text{O}_3$, contains two approximately planar molecules (r.m.s. deviations = 0.058 and 0.070 Å). In the crystal, molecules are linked into [010] chains by way of alternating $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots(\text{N},\text{O})$ hydrogen-bond linkages.

Related literature

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



Experimental

Crystal data

$\text{C}_7\text{H}_8\text{N}_2\text{O}_3$
 $M_r = 168.15$
Monoclinic, $C2$
 $a = 14.668$ (5) Å

$b = 7.7356$ (15) Å
 $c = 14.720$ (3) Å
 $\beta = 104.11$ (4)°
 $V = 1619.8$ (7) Å³

$Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹

$T = 293$ K
 $0.25 \times 0.22 \times 0.18$ mm

Data collection

Bruker SMART CCD
diffractometer
7823 measured reflections

3695 independent reflections
2600 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.117$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.072$
 $wR(F^2) = 0.186$
 $S = 0.89$
3695 reflections
217 parameters

1 restraint
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O5}^i$	0.86	2.07	2.867 (4)	155
$\text{N3}-\text{H3A}\cdots\text{O2}$	0.86	2.30	3.085 (4)	152
$\text{N3}-\text{H3A}\cdots\text{N2}$	0.86	2.53	3.232 (3)	140

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; 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: HB5763).

References

- Bruker (1997). *SMART* and *SAINTE*. 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. (2011). E67, o64 [<https://doi.org/10.1107/S1600536810051019>]

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S1. Experimental

A mixture of methyl carbazate (0.1 mol), and furfural (0.1 mol) was stirred in refluxing ethanol (20 mL) for 4 h to afford the title compound (0.085 mol, yield 85%). Colourless blocks of the title compound were obtained by recrystallization from ethanol at room temperature.

S2. Refinement

The absolute structure was indeterminate in the present study. H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances = 0.93–0.97 Å; N—H = 0.86 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

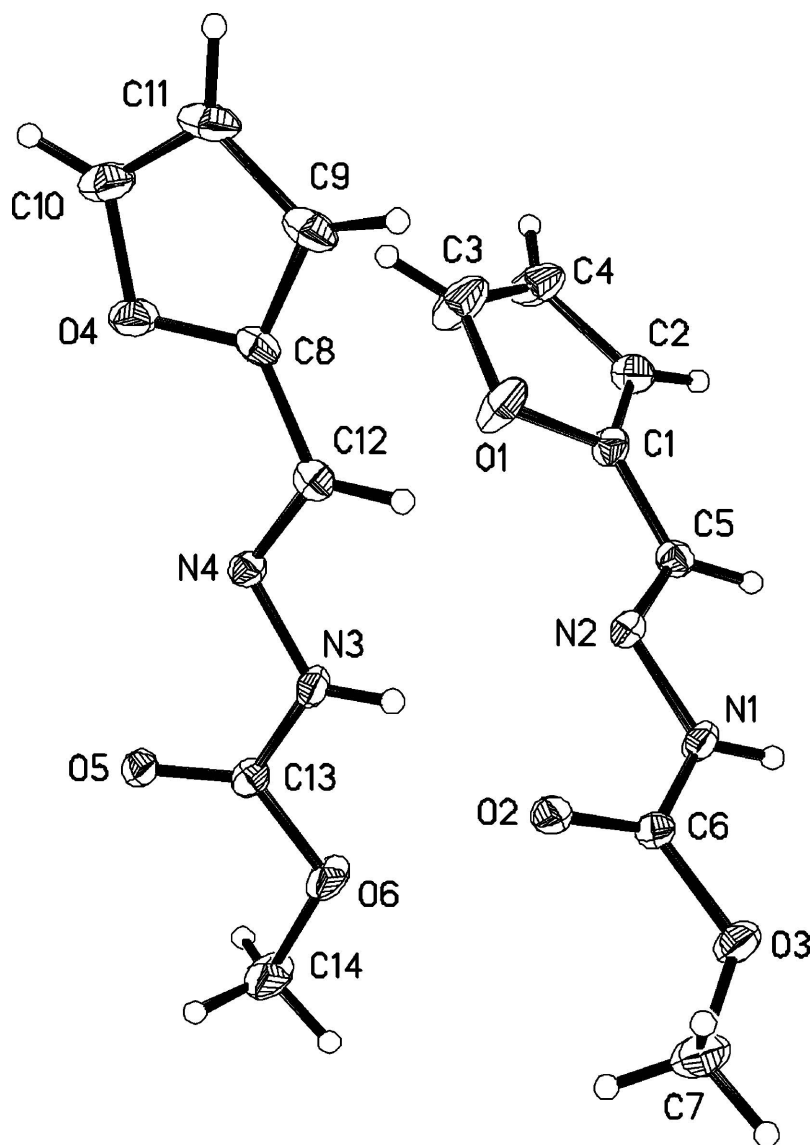


Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids.

Methyl 3-(2-furylmethylidene)carbazate

Crystal data

$C_7H_8N_2O_3$

$M_r = 168.15$

Monoclinic, $C2$

$a = 14.668 (5) \text{ \AA}$

$b = 7.7356 (15) \text{ \AA}$

$c = 14.720 (3) \text{ \AA}$

$\beta = 104.11 (4)^\circ$

$V = 1619.8 (7) \text{ \AA}^3$

$Z = 8$

$F(000) = 704$

$D_x = 1.379 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3695 reflections

$\theta = 3.2\text{--}27.5^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colorless

$0.25 \times 0.22 \times 0.18 \text{ mm}$

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

7823 measured reflections

3695 independent reflections

2600 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.117$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.2^\circ$

$h = -16 \rightarrow 18$

$k = -9 \rightarrow 10$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.072$

$wR(F^2) = 0.186$

$S = 0.89$

3695 reflections

217 parameters

1 restraint

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 $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.37 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.28 \text{ e } \text{\AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O5	0.18498 (16)	0.9708 (3)	0.08083 (14)	0.0286 (5)
N4	0.22925 (19)	0.8558 (3)	0.25908 (17)	0.0242 (5)
N3	0.1797 (2)	0.7585 (3)	0.18594 (17)	0.0281 (6)
H3A	0.1595	0.6572	0.1951	0.034*
O4	0.33569 (18)	1.0262 (3)	0.41175 (15)	0.0358 (6)
O6	0.1200 (2)	0.7115 (3)	0.03597 (15)	0.0397 (7)
C8	0.2972 (2)	0.8690 (4)	0.4211 (2)	0.0276 (7)
C12	0.2438 (2)	0.7836 (4)	0.3391 (2)	0.0260 (7)
H12A	0.2194	0.6741	0.3443	0.031*
C13	0.1635 (2)	0.8259 (4)	0.0994 (2)	0.0261 (7)
C9	0.3199 (3)	0.8194 (5)	0.5124 (2)	0.0376 (9)
H9A	0.3020	0.7180	0.5371	0.045*
C14	0.0973 (4)	0.7748 (5)	-0.0591 (2)	0.0489 (11)
H14A	0.0666	0.6853	-0.1005	0.073*
H14B	0.1539	0.8086	-0.0759	0.073*
H14C	0.0562	0.8728	-0.0640	0.073*
C11	0.3764 (3)	0.9529 (6)	0.5622 (2)	0.0419 (9)

H11A	0.4032	0.9564	0.6263	0.050*
C10	0.3839 (3)	1.0730 (6)	0.4994 (2)	0.0421 (9)
H10A	0.4176	1.1754	0.5137	0.051*
O2	0.04032 (17)	0.4665 (3)	0.19917 (14)	0.0296 (5)
N1	0.1456 (2)	0.2564 (4)	0.18947 (19)	0.0291 (6)
H1A	0.1539	0.1531	0.1715	0.035*
N2	0.22041 (19)	0.3544 (3)	0.23547 (17)	0.0265 (6)
C6	0.0590 (2)	0.3252 (4)	0.1731 (2)	0.0272 (7)
O3	-0.00370 (18)	0.2153 (3)	0.12255 (17)	0.0371 (6)
C5	0.3004 (2)	0.2814 (4)	0.2473 (2)	0.0264 (7)
H5A	0.3037	0.1687	0.2264	0.032*
O1	0.37821 (19)	0.5284 (3)	0.3290 (2)	0.0509 (8)
C1	0.3852 (2)	0.3697 (4)	0.2920 (2)	0.0282 (7)
C2	0.4759 (3)	0.3280 (5)	0.3040 (2)	0.0348 (8)
H2B	0.4995	0.2257	0.2857	0.042*
C7	-0.0995 (3)	0.2726 (6)	0.0996 (3)	0.0501 (10)
H7A	-0.1386	0.1857	0.0629	0.075*
H7B	-0.1046	0.3780	0.0644	0.075*
H7C	-0.1194	0.2924	0.1562	0.075*
C4	0.5291 (3)	0.4693 (6)	0.3499 (3)	0.0463 (10)
H4B	0.5943	0.4781	0.3669	0.056*
C3	0.4682 (3)	0.5862 (6)	0.3638 (3)	0.0587 (13)
H3B	0.4842	0.6924	0.3929	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O5	0.0362 (13)	0.0239 (12)	0.0235 (10)	-0.0029 (10)	0.0033 (9)	-0.0002 (8)
N4	0.0261 (13)	0.0244 (14)	0.0213 (12)	0.0009 (11)	0.0044 (9)	-0.0031 (10)
N3	0.0392 (16)	0.0191 (12)	0.0258 (13)	-0.0054 (11)	0.0074 (11)	-0.0036 (10)
O4	0.0394 (15)	0.0431 (15)	0.0218 (11)	-0.0052 (11)	0.0018 (10)	0.0004 (9)
O6	0.0650 (19)	0.0328 (14)	0.0197 (11)	-0.0137 (12)	0.0070 (11)	-0.0089 (9)
C8	0.0252 (17)	0.033 (2)	0.0244 (15)	0.0089 (13)	0.0056 (12)	0.0049 (12)
C12	0.0236 (15)	0.0287 (17)	0.0274 (15)	0.0047 (12)	0.0093 (12)	0.0010 (12)
C13	0.0276 (16)	0.0272 (17)	0.0249 (15)	-0.0004 (13)	0.0090 (12)	-0.0056 (12)
C9	0.040 (2)	0.049 (2)	0.0234 (16)	0.0136 (17)	0.0078 (14)	0.0077 (14)
C14	0.078 (3)	0.046 (2)	0.0189 (16)	-0.018 (2)	0.0044 (17)	-0.0067 (15)
C11	0.038 (2)	0.064 (3)	0.0198 (15)	0.0132 (18)	0.0010 (14)	-0.0020 (16)
C10	0.044 (2)	0.052 (2)	0.0260 (17)	-0.0013 (18)	0.0013 (15)	-0.0107 (16)
O2	0.0335 (12)	0.0265 (12)	0.0247 (11)	0.0025 (10)	-0.0006 (9)	-0.0045 (9)
N1	0.0292 (15)	0.0200 (13)	0.0360 (14)	-0.0038 (11)	0.0039 (11)	-0.0066 (10)
N2	0.0294 (15)	0.0209 (13)	0.0273 (13)	-0.0028 (11)	0.0035 (10)	-0.0039 (10)
C6	0.0319 (18)	0.0264 (16)	0.0206 (14)	-0.0028 (13)	0.0015 (12)	-0.0025 (12)
O3	0.0319 (14)	0.0315 (14)	0.0434 (14)	-0.0036 (10)	0.0006 (11)	-0.0118 (10)
C5	0.0294 (17)	0.0275 (16)	0.0223 (14)	0.0021 (12)	0.0062 (12)	-0.0001 (11)
O1	0.0242 (14)	0.0374 (15)	0.088 (2)	-0.0019 (11)	0.0086 (13)	-0.0304 (14)
C1	0.0311 (18)	0.0256 (17)	0.0284 (16)	0.0014 (13)	0.0082 (13)	-0.0049 (12)
C2	0.0346 (19)	0.0350 (18)	0.0327 (17)	0.0130 (15)	0.0043 (14)	-0.0072 (14)

C7	0.033 (2)	0.051 (2)	0.058 (2)	-0.0012 (18)	-0.0055 (17)	-0.016 (2)
C4	0.0242 (19)	0.055 (2)	0.055 (2)	0.0022 (17)	0.0021 (16)	-0.023 (2)
C3	0.025 (2)	0.054 (3)	0.093 (3)	-0.0031 (18)	0.006 (2)	-0.035 (2)

Geometric parameters (Å, °)

O5—C13	1.214 (4)	O2—C6	1.211 (4)
N4—C12	1.273 (4)	N1—C6	1.344 (4)
N4—N3	1.368 (3)	N1—N2	1.369 (4)
N3—C13	1.343 (4)	N1—H1A	0.8600
N3—H3A	0.8600	N2—C5	1.275 (4)
O4—C10	1.360 (4)	C6—O3	1.338 (4)
O4—C8	1.361 (4)	O3—C7	1.432 (5)
O6—C13	1.331 (4)	C5—C1	1.431 (4)
O6—C14	1.442 (4)	C5—H5A	0.9300
C8—C9	1.359 (4)	O1—C1	1.357 (4)
C8—C12	1.430 (4)	O1—C3	1.370 (5)
C12—H12A	0.9300	C1—C2	1.339 (5)
C9—C11	1.412 (6)	C2—C4	1.414 (6)
C9—H9A	0.9300	C2—H2B	0.9300
C14—H14A	0.9600	C7—H7A	0.9600
C14—H14B	0.9600	C7—H7B	0.9600
C14—H14C	0.9600	C7—H7C	0.9600
C11—C10	1.334 (6)	C4—C3	1.321 (6)
C11—H11A	0.9300	C4—H4B	0.9300
C10—H10A	0.9300	C3—H3B	0.9300
C12—N4—N3	115.1 (3)	C6—N1—N2	118.5 (3)
C13—N3—N4	118.1 (3)	C6—N1—H1A	120.7
C13—N3—H3A	121.0	N2—N1—H1A	120.7
N4—N3—H3A	121.0	C5—N2—N1	115.0 (3)
C10—O4—C8	105.9 (3)	O2—C6—O3	124.8 (3)
C13—O6—C14	114.4 (3)	O2—C6—N1	125.3 (3)
C9—C8—O4	110.1 (3)	O3—C6—N1	109.9 (3)
C9—C8—C12	131.0 (3)	C6—O3—C7	115.9 (3)
O4—C8—C12	118.9 (3)	N2—C5—C1	121.2 (3)
N4—C12—C8	120.8 (3)	N2—C5—H5A	119.4
N4—C12—H12A	119.6	C1—C5—H5A	119.4
C8—C12—H12A	119.6	C1—O1—C3	106.6 (3)
O5—C13—O6	124.2 (3)	C2—C1—O1	109.6 (3)
O5—C13—N3	125.2 (3)	C2—C1—C5	132.1 (3)
O6—C13—N3	110.6 (3)	O1—C1—C5	118.4 (3)
C8—C9—C11	106.2 (3)	C1—C2—C4	106.9 (3)
C8—C9—H9A	126.9	C1—C2—H2B	126.5
C11—C9—H9A	126.9	C4—C2—H2B	126.5
O6—C14—H14A	109.5	O3—C7—H7A	109.5
O6—C14—H14B	109.5	O3—C7—H7B	109.5
H14A—C14—H14B	109.5	H7A—C7—H7B	109.5

O6—C14—H14C	109.5	O3—C7—H7C	109.5
H14A—C14—H14C	109.5	H7A—C7—H7C	109.5
H14B—C14—H14C	109.5	H7B—C7—H7C	109.5
C10—C11—C9	106.6 (3)	C3—C4—C2	106.8 (3)
C10—C11—H11A	126.7	C3—C4—H4B	126.6
C9—C11—H11A	126.7	C2—C4—H4B	126.6
C11—C10—O4	111.1 (4)	C4—C3—O1	110.2 (4)
C11—C10—H10A	124.4	C4—C3—H3B	124.9
O4—C10—H10A	124.4	O1—C3—H3B	124.9

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots O5 ⁱ	0.86	2.07	2.867 (4)	155
N3—H3A \cdots O2	0.86	2.30	3.085 (4)	152
N3—H3A \cdots N2	0.86	2.53	3.232 (3)	140

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