Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-tert-Butyl-5-(4-chlorophenyl)furan-2-carbohydrazide

Xi-Chen Li, Ying Li, Zi-Ning Cui, Xin-Ling Yang and Yun Ling*

Department of Applied Chemistry, College of Science, China Agricultural University, Key Laboratory of Pesticide Chemistry and Application Technology, Ministry of Agriculture, Beijing 100094, People's Republic of China
Correspondence e-mail: lyun@cau.edu.cn

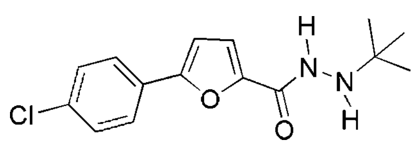
Received 26 December 2007; accepted 31 January 2008

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.027; wR factor = 0.057; data-to-parameter ratio = 18.2.

In the title molecule, $C_{15}H_{17}ClN_2O_2$, the furan and benzene rings form a dihedral angle of 15.35 (8)°. In the crystal structure, intermolecular N—H⋯O hydrogen bonds link the molecules into chains extended in the [010] direction.

Related literature

For general background, see: Wing (1988); Wing *et al.* (1988); Dhadialla *et al.* (1998); Heller *et al.* (1992); Mao *et al.* (2004). For details of some monoacylhydrazines and diacylhydrazines containing furan, see: Yang *et al.* (2002); Li *et al.* (2006).



Experimental

Crystal data

$C_{15}H_{17}ClN_2O_2$
M_r = 292.76
Orthorhombic, $P2_12_12_1$
a = 9.3770 (7) Å
b = 9.7861 (7) Å
c = 16.0119 (12) Å
V = 1469.32 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 113 (2) K
0.32 × 0.24 × 0.20 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.921, T_{max} = 0.949
13814 measured reflections
3496 independent reflections
2754 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.056
S = 0.96
3496 reflections
192 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 1490 Friedel pairs
Flack parameter: 0.00 (4)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯O2^{i}	0.882 (15)	2.026 (16)	2.8744 (15)	160.9 (14)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *CrystalStructure* (Rigaku/MSC, 2005).

This work was supported by the National Basic Research Programme of China (grant No. 2003CB114405), the National Natural Science Foundation of China (grant No. 20672138) and the National High Technology Research and Development Programme of China (grant No. 2006AA10A201). The authors also thank the State Key Laboratory and Institute of Elemento-Organic Chemistry, Nankai University, Tianjin, People's Republic of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2380).

References

Dhadialla, T. S., Carlson, G. R. & Le, D. P. (1998). *Annu. Rev. Entomol.* **43**, 545–569.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Heller, J. J., Mattioda, H., Klein, E. & Sagenmuller, A. (1992). *Brighton Crop. Prot. Conf. Pests. Dis.* **1**, 59–66.
Li, X. C., Yang, X. L., Kai, Z. P. & Ling, Y. (2006). *Huaxue Tongbao*, **69**, 668–673.
Mao, C. H., Wang, Q. M., Huang, R. Q., Bi, F. C., Chen, L., Liu, Y. X. & Shang, J. (2004). *J. Agric. Food Chem.* **52**, 6737–6741.
Rigaku/MSC (2005). *CrystalClear* (Version 1.36) and *CrystalStructure* (Version 3.7.0). Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wing, K. D. (1988). *Science*, **241**, 467–469.
Wing, K. D., Slawecki, R. A. & Carlson, G. R. (1988). *Science*, **241**, 470–472.
Yang, X. L., Qian, J. H., Chen, F. H. & Wang, D. Q. (2002). CN Patent No. 1370405.

supporting information

Acta Cryst. (2008). E**64**, o561 [doi:10.1107/S1600536808003486]

N′-*tert*-Butyl-5-(4-chlorophenyl)furan-2-carbohydrazide

Xi-Chen Li, Ying Li, Zi-Ning Cui, Xin-Ling Yang and Yun Ling

S1. Comment

As one of molting hormone analogs, symmetrical *N′*-*tert*-butyl-*N,N′*-dibenzoylhydrazine (RH-5849) was first found to be a nonsteroidal ecdysone agonist in 1988 (Wing, 1988; Wing *et al.*, 1988). Afterward, several diacylhydrazine compounds were commercially developed as insect growth regulators (IGRs) which were widely used in agriculture (Dhadialla *et al.*, 1998; Heller *et al.*, 1992; Mao *et al.*, 2004). Recently, we synthesized a series of di- or mono- acylhydrazines containing furan for further study on the structure-activity relationship between monoacylhydrazines and diacylhydrazines. It was found that they both had good insecticidal activities (Yang *et al.*, 2002; Li *et al.*, 2006). In order to study the structural character and conformation of the monoacylhydrazine containing furan, the crystal structure of the title compound, (I), has been determined.

In (I) (Fig. 1), the benzene (C1—C6) and furan (O1/C7—C10) rings form a dihedral angle of 15.35 (8)°. The carbonyl group attached to the furan ring is almost coplanar with it. In the crystal, the intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into chains extended in direction [010].

S2. Experimental

The title compound, (I), was synthesized by the reaction of 5-(4-chlorophenyl)furan-2-carbonyl chloride (0.96 g, 4 mmol) with *tert*-butylhydrazine hydrochloride (1.99 g, 16 mmol) using sodium hydroxide (10%, 8.0 g, 20 mmol) as the acid-binding agent. The mixture was stirred at room temperature for 5 h and filtered to obtain a yellow solution. Then the organic phase was separated and dried with anhydrous magnesium sulfate overnight. After removal of the solvent, the residue was purified by vacuum column chromatography on silica gel with petroleum ether and ethyl acetate as the eluent ($V_{\text{petroleum ether}}$: $V_{\text{ethyl acetate}}$ = 3:1) and then recrystallized from hexane–ethyl acetate (V_{hexane}: $V_{\text{ethyl acetate}}$= 1:1) to give colourless crystals suitable for X-ray diffraction (Li *et al.*, 2006).

S3. Refinement

Atoms H1A and H2A were located on a difference map and isotropically refined. The rest H atoms were positioned geometrically (C—H = 0.95–0.98 Å), and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C).

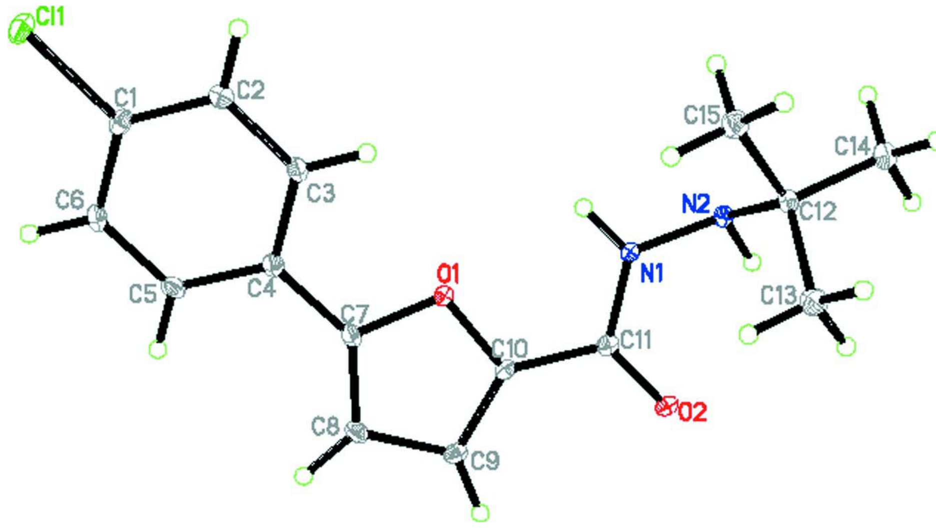


Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

***N′-tert*-Butyl-5-(4-chlorophenyl)furan-2-carbohydrazide**

Crystal data

$C_{15}H_{17}ClN_2O_2$
M_r = 292.76
Orthorhombic, $P2_12_12_1$
a = 9.3770 (7) Å
b = 9.7861 (7) Å
c = 16.0119 (12) Å
V = 1469.32 (19) Å^3
Z = 4
$F(000)$ = 616
D_x = 1.323 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71070 Å
Cell parameters from 3053 reflections
θ = 2.5–25.0°
μ = 0.26 mm^{-1}
T = 113 K
Prism, colourless
0.32 × 0.24 × 0.20 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.921, T_{max} = 0.949
13814 measured reflections
3496 independent reflections
2754 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.9°, θ_{min} = 2.4°
h = −12→12
k = −12→12
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.056
S = 0.96
3496 reflections
192 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0232P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}

$\Delta\rho_{min} = -0.21$ e Å^{-3}

Absolute structure: Flack (1983), 1490 Friedel pairs

Absolute structure parameter: 0.00 (4)

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	1.32887 (4)	0.17773 (4)	1.09957 (2)	0.02303 (9)
O1	1.13067 (10)	0.70996 (9)	0.86903 (6)	0.0151 (2)
O2	0.98638 (10)	0.97666 (9)	0.74249 (6)	0.0174 (2)
N1	0.91782 (13)	0.75551 (12)	0.76336 (7)	0.0151 (3)
N2	0.80263 (13)	0.76074 (12)	0.70613 (7)	0.0146 (3)
C1	1.30520 (15)	0.33360 (15)	1.04901 (8)	0.0159 (3)
C2	1.17767 (16)	0.35872 (14)	1.00795 (8)	0.0168 (3)
H2	1.1040	0.2921	1.0078	0.020*
C3	1.15907 (16)	0.48241 (13)	0.96720 (8)	0.0159 (3)
H3	1.0720	0.5002	0.9389	0.019*
C4	1.26701 (15)	0.58168 (15)	0.96708 (9)	0.0149 (3)
C5	1.39448 (15)	0.55382 (14)	1.00941 (8)	0.0176 (3)
H5	1.4685	0.6201	1.0100	0.021*
C6	1.41381 (15)	0.43007 (14)	1.05056 (9)	0.0175 (3)
H6	1.5003	0.4117	1.0794	0.021*
C7	1.24400 (15)	0.71056 (14)	0.92346 (8)	0.0153 (3)
C8	1.30641 (15)	0.83596 (15)	0.92271 (8)	0.0175 (3)
H8	1.3868	0.8635	0.9547	0.021*
C9	1.22900 (14)	0.91848 (15)	0.86505 (9)	0.0165 (3)
H9	1.2472	1.0113	0.8515	0.020*
C10	1.12421 (14)	0.83819 (14)	0.83345 (8)	0.0137 (3)
C11	1.00485 (15)	0.86322 (14)	0.77523 (8)	0.0138 (3)
C12	0.66234 (15)	0.78352 (14)	0.74892 (9)	0.0163 (3)
C13	0.65461 (17)	0.92221 (15)	0.79274 (9)	0.0240 (4)
H13A	0.7283	0.9270	0.8360	0.036*
H13B	0.5604	0.9333	0.8184	0.036*
H13C	0.6701	0.9952	0.7518	0.036*
C14	0.55159 (16)	0.77717 (16)	0.67895 (10)	0.0241 (4)
H14A	0.5713	0.8491	0.6380	0.036*
H14B	0.4561	0.7906	0.7025	0.036*
H14C	0.5563	0.6877	0.6516	0.036*
C15	0.63850 (16)	0.66767 (15)	0.81165 (9)	0.0230 (3)

H15A	0.6501	0.5795	0.7834	0.035*
H15B	0.5419	0.6742	0.8347	0.035*
H15C	0.7083	0.6751	0.8570	0.035*
H2A	0.8186 (15)	0.8364 (13)	0.6731 (8)	0.012 (4)*
H1A	0.9484 (16)	0.6722 (16)	0.7743 (9)	0.029 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02355 (19)	0.02007 (19)	0.0255 (2)	0.00437 (17)	−0.00042 (17)	0.00723 (17)
O1	0.0162 (5)	0.0122 (5)	0.0169 (5)	0.0000 (4)	−0.0040 (4)	0.0010 (4)
O2	0.0205 (5)	0.0104 (5)	0.0214 (5)	−0.0001 (4)	−0.0019 (5)	0.0019 (4)
N1	0.0154 (6)	0.0107 (7)	0.0192 (7)	0.0012 (5)	−0.0047 (5)	0.0004 (5)
N2	0.0145 (7)	0.0146 (7)	0.0146 (6)	0.0003 (5)	−0.0032 (5)	0.0022 (5)
C1	0.0218 (8)	0.0154 (7)	0.0105 (7)	0.0048 (7)	0.0022 (6)	0.0016 (6)
C2	0.0168 (7)	0.0180 (8)	0.0157 (7)	−0.0019 (6)	0.0012 (7)	−0.0011 (6)
C3	0.0146 (7)	0.0187 (8)	0.0144 (7)	0.0016 (6)	−0.0031 (6)	−0.0010 (6)
C4	0.0157 (7)	0.0162 (8)	0.0128 (7)	0.0021 (6)	−0.0004 (6)	−0.0034 (6)
C5	0.0180 (8)	0.0172 (8)	0.0177 (8)	−0.0014 (6)	−0.0022 (6)	−0.0043 (6)
C6	0.0157 (8)	0.0225 (8)	0.0144 (8)	0.0045 (7)	−0.0029 (6)	−0.0021 (6)
C7	0.0125 (7)	0.0185 (8)	0.0150 (8)	0.0015 (6)	−0.0024 (6)	−0.0025 (6)
C8	0.0137 (7)	0.0185 (7)	0.0202 (8)	−0.0026 (6)	−0.0021 (6)	−0.0018 (6)
C9	0.0169 (7)	0.0129 (7)	0.0196 (8)	−0.0008 (6)	0.0022 (6)	0.0011 (6)
C10	0.0157 (7)	0.0106 (7)	0.0149 (7)	0.0023 (6)	0.0015 (6)	0.0004 (6)
C11	0.0139 (7)	0.0139 (8)	0.0137 (7)	0.0019 (6)	0.0041 (6)	−0.0023 (6)
C12	0.0134 (7)	0.0168 (7)	0.0187 (8)	0.0007 (6)	−0.0017 (7)	0.0000 (6)
C13	0.0207 (9)	0.0222 (8)	0.0292 (9)	0.0024 (7)	−0.0007 (7)	−0.0052 (7)
C14	0.0209 (8)	0.0245 (9)	0.0270 (9)	0.0008 (7)	−0.0075 (7)	0.0012 (7)
C15	0.0224 (8)	0.0230 (8)	0.0237 (8)	−0.0003 (8)	−0.0005 (7)	0.0029 (7)

Geometric parameters (Å, º)

Cl1—C1	1.7411 (14)	C6—H6	0.9500
O1—C7	1.3744 (15)	C7—C8	1.360 (2)
O1—C10	1.3795 (16)	C8—C9	1.4252 (19)
O2—C11	1.2398 (15)	C8—H8	0.9500
N1—C11	1.3465 (17)	C9—C10	1.3561 (18)
N1—N2	1.4174 (16)	C9—H9	0.9500
N1—H1A	0.882 (15)	C10—C11	1.4771 (19)
N2—C12	1.4999 (18)	C12—C14	1.5289 (19)
N2—H2A	0.922 (13)	C12—C13	1.5296 (18)
C1—C2	1.3866 (19)	C12—C15	1.5311 (18)
C1—C6	1.3889 (19)	C13—H13A	0.9800
C2—C3	1.3861 (17)	C13—H13B	0.9800
C2—H2	0.9500	C13—H13C	0.9800
C3—C4	1.4029 (19)	C14—H14A	0.9800
C3—H3	0.9500	C14—H14B	0.9800
C4—C5	1.4009 (18)	C14—H14C	0.9800

C4—C7	1.4578 (19)	C15—H15A	0.9800
C5—C6	1.3906 (19)	C15—H15B	0.9800
C5—H5	0.9500	C15—H15C	0.9800

C7—O1—C10	106.97 (10)	C10—C9—H9	126.8
C11—N1—N2	121.66 (12)	C8—C9—H9	126.8
C11—N1—H1A	119.9 (10)	C9—C10—O1	109.95 (12)
N2—N1—H1A	114.2 (10)	C9—C10—C11	133.51 (13)
N1—N2—C12	112.24 (10)	O1—C10—C11	116.41 (12)
N1—N2—H2A	106.0 (9)	O2—C11—N1	123.82 (13)
C12—N2—H2A	106.6 (9)	O2—C11—C10	121.41 (13)
C2—C1—C6	121.36 (13)	N1—C11—C10	114.74 (12)
C2—C1—Cl1	119.06 (11)	N2—C12—C14	104.77 (11)
C6—C1—Cl1	119.58 (11)	N2—C12—C13	112.52 (12)
C3—C2—C1	119.11 (13)	C14—C12—C13	109.88 (12)
C3—C2—H2	120.4	N2—C12—C15	108.52 (11)
C1—C2—H2	120.4	C14—C12—C15	110.58 (12)
C2—C3—C4	120.97 (14)	C13—C12—C15	110.44 (12)
C2—C3—H3	119.5	C12—C13—H13A	109.5
C4—C3—H3	119.5	C12—C13—H13B	109.5
C5—C4—C3	118.69 (13)	H13A—C13—H13B	109.5
C5—C4—C7	121.77 (13)	C12—C13—H13C	109.5
C3—C4—C7	119.54 (13)	H13A—C13—H13C	109.5
C6—C5—C4	120.66 (13)	H13B—C13—H13C	109.5
C6—C5—H5	119.7	C12—C14—H14A	109.5
C4—C5—H5	119.7	C12—C14—H14B	109.5
C1—C6—C5	119.20 (13)	H14A—C14—H14B	109.5
C1—C6—H6	120.4	C12—C14—H14C	109.5
C5—C6—H6	120.4	H14A—C14—H14C	109.5
C8—C7—O1	109.33 (12)	H14B—C14—H14C	109.5
C8—C7—C4	136.18 (13)	C12—C15—H15A	109.5
O1—C7—C4	114.49 (12)	C12—C15—H15B	109.5
C7—C8—C9	107.33 (13)	H15A—C15—H15B	109.5
C7—C8—H8	126.3	C12—C15—H15C	109.5
C9—C8—H8	126.3	H15A—C15—H15C	109.5
C10—C9—C8	106.41 (13)	H15B—C15—H15C	109.5

C11—N1—N2—C12	−101.13 (14)	O1—C7—C8—C9	−0.05 (16)
C6—C1—C2—C3	0.5 (2)	C4—C7—C8—C9	−179.46 (15)
Cl1—C1—C2—C3	−179.57 (10)	C7—C8—C9—C10	−0.42 (16)
C1—C2—C3—C4	0.0 (2)	C8—C9—C10—O1	0.73 (16)
C2—C3—C4—C5	−0.3 (2)	C8—C9—C10—C11	176.25 (14)
C2—C3—C4—C7	179.88 (13)	C7—O1—C10—C9	−0.77 (15)
C3—C4—C5—C6	0.2 (2)	C7—O1—C10—C11	−177.14 (11)
C7—C4—C5—C6	180.00 (13)	N2—N1—C11—O2	5.2 (2)
C2—C1—C6—C5	−0.6 (2)	N2—N1—C11—C10	−176.72 (11)
Cl1—C1—C6—C5	179.45 (10)	C9—C10—C11—O2	1.9 (2)
C4—C5—C6—C1	0.3 (2)	O1—C10—C11—O2	177.18 (12)

C10—O1—C7—C8	0.49 (14)	C9—C10—C11—N1	−176.21 (15)
C10—O1—C7—C4	−179.95 (11)	O1—C10—C11—N1	−0.92 (17)
C5—C4—C7—C8	−15.6 (3)	N1—N2—C12—C14	−176.55 (11)
C3—C4—C7—C8	164.26 (16)	N1—N2—C12—C13	64.11 (15)
C5—C4—C7—O1	165.04 (12)	N1—N2—C12—C15	−58.41 (14)
C3—C4—C7—O1	−15.13 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.882 (15)	2.026 (16)	2.8744 (15)	160.9 (14)

Symmetry code: (i) −*x*+2, *y*−1/2, −*z*+3/2.