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2-(4-Nitrophenyl)-5-phenylfuran

Zhi Guan, Yan-hong He* and Gangqiang Wang

School of Chemistry and Chemical Engineering, Southwest University, Chongqing 400715, People's Republic of China

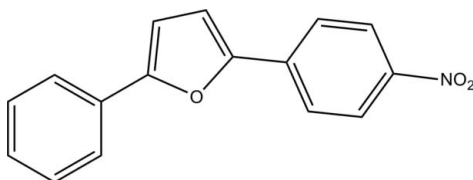
Correspondence e-mail: heyh@swu.edu.cn

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Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.063; wR factor = 0.174; data-to-parameter ratio = 16.1.

The molecular skeleton of the title molecule, $\text{C}_{16}\text{H}_{11}\text{NO}_3$, is nearly planar with the two aromatic rings forming a dihedral angle of $2.73(7)^\circ$. In the crystal, weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link molecules into ribbons extended along [101]. The crystal packing exhibits also $\pi-\pi$ interactions, as indicated by the short centroid-centroid distances between the aromatic rings [3.681 (3) Å] and between the aromatic and furan rings [3.811 (3) Å] of neighbouring molecules.

Related literature

For details of the synthesis, see: Wang *et al.* (2009).

Experimental

Crystal data

 $\text{C}_{16}\text{H}_{11}\text{NO}_3$
 $M_r = 265.26$
 Monoclinic, $P2_1/n$
 $a = 7.3213(15)$ Å
 $b = 16.290(3)$ Å
 $c = 10.904(2)$ Å

 $\beta = 100.81(3)^\circ$
 $V = 1277.3(4)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation

 $\mu = 0.10$ mm⁻¹
 $T = 113$ K
 $0.24 \times 0.22 \times 0.19$ mm

Data collection

 Rigaku Saturn CCD area-detector diffractometer
 Absorption correction: multi-scan (*CrystalClear*; Rigaku/MS, 2005)
 $T_{\min} = 0.977$, $T_{\max} = 0.982$

 10136 measured reflections
 2924 independent reflections
 1168 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.070$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.174$
 $S = 1.00$
 2924 reflections

 182 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C3}-\text{H3}\cdots\text{O3}^i$	0.95	2.51	3.373 (3)	152
$\text{C12}-\text{H12}\cdots\text{O2}^{ii}$	0.95	2.61	3.435 (3)	146

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

Data collection: *CrystalClear* (Rigaku/MS, 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: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2666).

References

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 Rigaku/MS (2005). *CrystalClear* and *CrystalStructure*. Rigaku/MS, The Woodlands, Texas, USA.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Wang, G., Guan, Z., Tang, R. & He, Y. (2009). *J. Heterocycl. Chem.* **46**, 540–543.

supporting information

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2-(4-Nitrophenyl)-5-phenylfuran

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S1. Comment

The title compound, (I), has been obtained as a by-product in our ongoing research of highly substituted furan derivatives (Wang *et al.*, 2009).

In (I) (Fig. 1), two aromatic rings form a dihedral angle of 2.73 (7)°. Weak intermolecular C—H···O hydrogen bonds (Table 1) and π - π stacking interactions with centroid-centroid separations of 3.681 (3) and 3.811 (3) Å consolidate the crystal packing.

S2. Experimental

A solution of ethyl 2-benzoyl-4-(4-nitrophenyl)-4-oxobutanoate (0.353 g, 0.7 mmol) in ionic liquid [bmim]HSO₄ (1.6 g, 6.7 mmol) was stirred at 150 °C for 4 h in oil bath. After cooling to r.t., the reaction mixture was extracted with diethyl ether thoroughly. The combined extracts were washed with water, brine, dried (Na₂SO₄), and filtered. The solvents were removed and the residue was purified by flash chromatography (petroleum ether / dichloromethane 2:1) to get Ethyl 5-(4-nitrophenyl)-2-phenylfuran-3-carboxylate (240 mg, 71%) and the title compound (50 mg, 14%) as yellow crystals.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model, with $d(\text{C—H}) = 0.95$ Å, $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$.

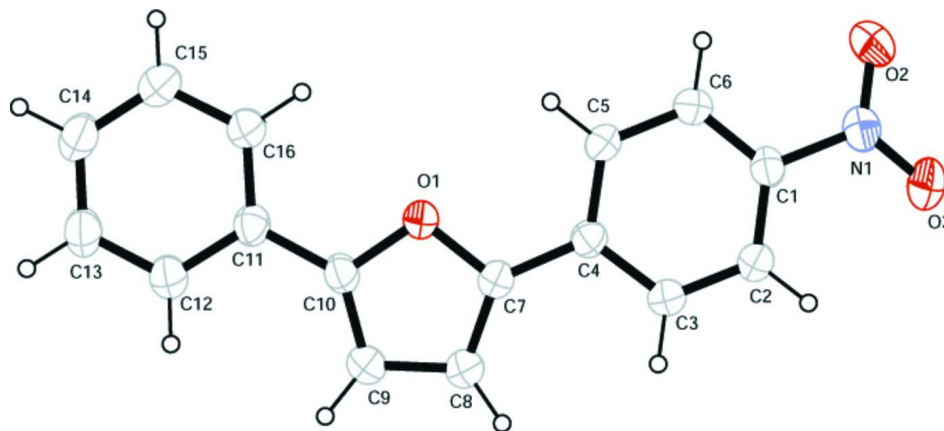


Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

2-(4-Nitrophenyl)-5-phenylfuran

Crystal data

C₁₆H₁₁NO₃ $M_r = 265.26$ Monoclinic, $P2_1/n$ $a = 7.3213$ (15) Å $b = 16.290$ (3) Å $c = 10.904$ (2) Å $\beta = 100.81$ (3)° $V = 1277.3$ (4) Å³ $Z = 4$ $F(000) = 552$ $D_x = 1.379$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3226 reflections

 $\theta = 2.3$ – 27.7° $\mu = 0.10$ mm⁻¹ $T = 113$ K

Block, orange

 $0.24 \times 0.22 \times 0.19$ mm

Data collection

Rigaku Saturn CCD area-detector
diffractometer

Radiation source: rotating anode

Confocal monochromator

Detector resolution: 7.31 pixels mm⁻¹ ω and φ scansAbsorption correction: multi-scan
(*CrystalClear*; Rigaku/MSO, 2005) $T_{\min} = 0.977$, $T_{\max} = 0.982$

10136 measured reflections

2924 independent reflections

1168 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.070$ $\theta_{\max} = 27.6^\circ$, $\theta_{\min} = 3.1^\circ$ $h = -9 \rightarrow 9$ $k = -21 \rightarrow 21$ $l = -12 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.063$ $wR(F^2) = 0.174$ $S = 1.00$

2924 reflections

182 parameters

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0654P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.22$ e Å⁻³ $\Delta\rho_{\min} = -0.19$ e Å⁻³Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.086 (10)

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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.83658 (18)	0.47058 (7)	0.09998 (13)	0.0540 (5)
O2	0.3323 (3)	0.56325 (12)	-0.48419 (15)	0.0968 (7)
O3	0.3467 (3)	0.69148 (10)	-0.43913 (17)	0.0971 (7)
N1	0.3792 (3)	0.61960 (13)	-0.41169 (19)	0.0685 (6)

C1	0.4782 (3)	0.60055 (13)	-0.2862 (2)	0.0528 (6)
C2	0.5230 (3)	0.66262 (13)	-0.2022 (2)	0.0632 (7)
H2	0.4856	0.7174	-0.2240	0.076*
C3	0.6219 (3)	0.64521 (13)	-0.0866 (2)	0.0612 (7)
H3	0.6524	0.6882	-0.0275	0.073*
C4	0.6791 (3)	0.56563 (12)	-0.05378 (19)	0.0504 (6)
C5	0.6301 (3)	0.50391 (12)	-0.1400 (2)	0.0569 (6)
H5	0.6657	0.4489	-0.1185	0.068*
C6	0.5298 (3)	0.52119 (12)	-0.2572 (2)	0.0595 (6)
H6	0.4971	0.4787	-0.3169	0.071*
C7	0.7901 (3)	0.55033 (12)	0.0684 (2)	0.0554 (6)
C9	0.9599 (3)	0.54956 (13)	0.2595 (2)	0.0696 (8)
H9	1.0268	0.5672	0.3382	0.084*
C8	0.8639 (3)	0.59996 (14)	0.1642 (2)	0.0700 (8)
H8	0.8530	0.6580	0.1666	0.084*
C10	0.9392 (3)	0.47133 (13)	0.2184 (2)	0.0547 (6)
C11	1.0059 (3)	0.39286 (13)	0.2725 (2)	0.0552 (6)
C12	1.0985 (3)	0.38948 (14)	0.3963 (2)	0.0673 (7)
H12	1.1158	0.4382	0.4450	0.081*
C13	1.1648 (3)	0.31612 (15)	0.4481 (2)	0.0787 (8)
H13	1.2278	0.3145	0.5326	0.094*
C14	1.1415 (4)	0.24587 (16)	0.3802 (3)	0.0848 (9)
H14	1.1872	0.1953	0.4173	0.102*
C15	1.0523 (4)	0.24782 (14)	0.2581 (3)	0.0847 (9)
H15	1.0371	0.1986	0.2104	0.102*
C16	0.9841 (3)	0.32104 (13)	0.2038 (2)	0.0683 (7)
H16	0.9221	0.3220	0.1191	0.082*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0608 (9)	0.0479 (9)	0.0501 (10)	0.0013 (7)	0.0021 (8)	0.0039 (7)
O2	0.1211 (16)	0.0947 (14)	0.0616 (12)	0.0146 (11)	-0.0160 (11)	-0.0143 (10)
O3	0.1200 (16)	0.0727 (13)	0.0855 (14)	0.0136 (10)	-0.0141 (12)	0.0234 (10)
N1	0.0684 (13)	0.0728 (14)	0.0594 (14)	0.0080 (10)	-0.0007 (11)	0.0052 (11)
C1	0.0501 (12)	0.0567 (13)	0.0489 (13)	0.0029 (10)	0.0023 (10)	0.0059 (10)
C2	0.0694 (15)	0.0474 (13)	0.0655 (16)	0.0056 (11)	-0.0063 (12)	0.0013 (11)
C3	0.0701 (15)	0.0471 (13)	0.0605 (15)	0.0021 (10)	-0.0031 (12)	-0.0038 (11)
C4	0.0537 (13)	0.0457 (12)	0.0507 (13)	-0.0042 (9)	0.0072 (10)	0.0037 (10)
C5	0.0726 (15)	0.0420 (12)	0.0540 (14)	0.0031 (10)	0.0063 (12)	0.0040 (10)
C6	0.0739 (16)	0.0502 (13)	0.0516 (14)	-0.0001 (10)	0.0048 (12)	-0.0039 (10)
C7	0.0614 (13)	0.0468 (12)	0.0547 (14)	0.0027 (10)	0.0029 (11)	0.0032 (10)
C9	0.0815 (17)	0.0579 (15)	0.0600 (15)	-0.0001 (12)	-0.0111 (13)	-0.0044 (12)
C8	0.0825 (17)	0.0520 (14)	0.0660 (16)	0.0046 (12)	-0.0102 (13)	-0.0033 (12)
C10	0.0535 (13)	0.0579 (14)	0.0484 (14)	-0.0008 (10)	-0.0015 (11)	0.0047 (10)
C11	0.0484 (12)	0.0554 (13)	0.0588 (15)	-0.0014 (10)	0.0023 (11)	0.0082 (11)
C12	0.0727 (16)	0.0644 (15)	0.0594 (16)	0.0006 (12)	-0.0015 (13)	0.0066 (12)
C13	0.0857 (18)	0.0737 (18)	0.0680 (18)	0.0021 (14)	-0.0076 (15)	0.0174 (14)

C14	0.100 (2)	0.0590 (16)	0.087 (2)	0.0009 (14)	-0.0049 (17)	0.0195 (14)
C15	0.106 (2)	0.0523 (15)	0.087 (2)	-0.0018 (13)	-0.0051 (17)	0.0046 (14)
C16	0.0772 (16)	0.0555 (14)	0.0641 (16)	-0.0037 (12)	-0.0074 (13)	0.0029 (12)

Geometric parameters (Å, °)

O1—C10	1.367 (2)	C9—C10	1.350 (3)
O1—C7	1.371 (2)	C9—C8	1.405 (3)
O2—N1	1.218 (2)	C9—H9	0.9500
O3—N1	1.221 (2)	C8—H8	0.9500
N1—C1	1.457 (3)	C10—C11	1.453 (3)
C1—C2	1.363 (3)	C11—C16	1.382 (3)
C1—C6	1.367 (3)	C11—C12	1.394 (3)
C2—C3	1.360 (3)	C12—C13	1.371 (3)
C2—H2	0.9500	C12—H12	0.9500
C3—C4	1.388 (3)	C13—C14	1.357 (3)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.377 (3)	C14—C15	1.369 (3)
C4—C7	1.446 (3)	C14—H14	0.9500
C5—C6	1.379 (3)	C15—C16	1.382 (3)
C5—H5	0.9500	C15—H15	0.9500
C6—H6	0.9500	C16—H16	0.9500
C7—C8	1.351 (3)		
C10—O1—C7	107.20 (15)	C10—C9—H9	126.4
O2—N1—O3	123.0 (2)	C8—C9—H9	126.4
O2—N1—C1	118.64 (19)	C7—C8—C9	107.1 (2)
O3—N1—C1	118.31 (19)	C7—C8—H8	126.4
C2—C1—C6	121.7 (2)	C9—C8—H8	126.4
C2—C1—N1	119.23 (19)	C9—C10—O1	109.24 (17)
C6—C1—N1	119.06 (19)	C9—C10—C11	133.4 (2)
C3—C2—C1	119.2 (2)	O1—C10—C11	117.31 (18)
C3—C2—H2	120.4	C16—C11—C12	118.48 (19)
C1—C2—H2	120.4	C16—C11—C10	122.0 (2)
C2—C3—C4	121.1 (2)	C12—C11—C10	119.55 (19)
C2—C3—H3	119.5	C13—C12—C11	120.3 (2)
C4—C3—H3	119.5	C13—C12—H12	119.9
C5—C4—C3	118.56 (19)	C11—C12—H12	119.9
C5—C4—C7	122.16 (18)	C14—C13—C12	120.8 (2)
C3—C4—C7	119.27 (18)	C14—C13—H13	119.6
C4—C5—C6	120.60 (19)	C12—C13—H13	119.6
C4—C5—H5	119.7	C13—C14—C15	120.0 (2)
C6—C5—H5	119.7	C13—C14—H14	120.0
C1—C6—C5	118.91 (19)	C15—C14—H14	120.0
C1—C6—H6	120.5	C14—C15—C16	120.3 (2)
C5—C6—H6	120.5	C14—C15—H15	119.9
C8—C7—O1	109.16 (18)	C16—C15—H15	119.9
C8—C7—C4	133.11 (19)	C11—C16—C15	120.2 (2)

O1—C7—C4	117.73 (17)	C11—C16—H16	119.9
C10—C9—C8	107.3 (2)	C15—C16—H16	119.9
O2—N1—C1—C2	-176.6 (2)	O1—C7—C8—C9	-0.1 (3)
O3—N1—C1—C2	3.1 (3)	C4—C7—C8—C9	-179.7 (2)
O2—N1—C1—C6	5.8 (3)	C10—C9—C8—C7	-0.6 (3)
O3—N1—C1—C6	-174.4 (2)	C8—C9—C10—O1	1.0 (3)
C6—C1—C2—C3	0.2 (4)	C8—C9—C10—C11	179.3 (2)
N1—C1—C2—C3	-177.37 (19)	C7—O1—C10—C9	-1.1 (2)
C1—C2—C3—C4	0.6 (4)	C7—O1—C10—C11	-179.68 (19)
C2—C3—C4—C5	-1.4 (3)	C9—C10—C11—C16	-171.6 (2)
C2—C3—C4—C7	177.7 (2)	O1—C10—C11—C16	6.5 (3)
C3—C4—C5—C6	1.4 (3)	C9—C10—C11—C12	6.9 (4)
C7—C4—C5—C6	-177.6 (2)	O1—C10—C11—C12	-174.98 (19)
C2—C1—C6—C5	-0.2 (4)	C16—C11—C12—C13	-0.4 (4)
N1—C1—C6—C5	177.36 (19)	C10—C11—C12—C13	-179.0 (2)
C4—C5—C6—C1	-0.6 (4)	C11—C12—C13—C14	0.0 (4)
C10—O1—C7—C8	0.8 (2)	C12—C13—C14—C15	0.5 (4)
C10—O1—C7—C4	-179.61 (18)	C13—C14—C15—C16	-0.6 (4)
C5—C4—C7—C8	175.4 (2)	C12—C11—C16—C15	0.4 (4)
C3—C4—C7—C8	-3.6 (4)	C10—C11—C16—C15	178.9 (2)
C5—C4—C7—O1	-4.1 (3)	C14—C15—C16—C11	0.1 (4)
C3—C4—C7—O1	176.93 (19)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3 \cdots O3 ⁱ	0.95	2.51	3.373 (3)	152
C12—H12 \cdots O2 ⁱⁱ	0.95	2.61	3.435 (3)	146

Symmetry codes: (i) $x+1/2, -y+3/2, z+1/2$; (ii) $x+1, y, z+1$.