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1-[4-(2-Furyl)but-3-en-2-ylidene]-2-(2-nitrophenyl)hydrazine

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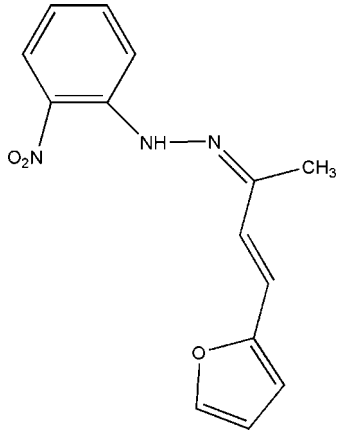
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.136; data-to-parameter ratio = 14.8.

In the title Schiff base compound, $C_{14}H_{13}N_3O_3$, the furan and benzene rings are oriented at a dihedral angle of 10.24 (13)°. Intramolecular N—H···O hydrogen bonding is observed between the imino and nitro groups.

Related literature

For applications of Schiff base compounds, see: Okabe *et al.* (1993).



Experimental

Crystal data

$C_{14}H_{13}N_3O_3$
M_r = 271.27
Triclinic, $P\overline{1}$
a = 8.2261 (2) Å
b = 9.0200 (2) Å
c = 9.1027 (2) Å
α = 89.166 (2)°
β = 77.549 (2)°
γ = 80.250 (2)°
V = 649.83 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.1 mm^{-1}
T = 296 K
0.12 × 0.10 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
9374 measured reflections
2687 independent reflections
1285 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.136
S = 0.90
2687 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2	0.86	2.00	2.611 (2)	127

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2582).

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supporting information

Acta Cryst. (2009). E**65**, o2162 [doi:10.1107/S1600536809031638]

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S1. Comment

4-Nitrophenylhydrazine has applications in organic synthesis, and some of its derivatives have been shown to be potentially DNA-damaging and mutagenic agents (Okabe *et al.*, 1993). As part of our work, the title compound (I) is synthesized in our group, and its structure is reported here (Fig. 1).

The molecular structure is non-planar, there is a dihedral angle of 9.19 (9)° between the benzene ring and the N2/N1/C7/C6/C5 plane, while the N2/N1/C7/C6/C5 planar and the furyl ring is nearly planar, making a dihedral angle of 4.26 (11)°. The furan and benzene rings are oriented at a dihedral angle of 10.24 (13)°. Intramolecular N—H···O hydrogen bonding is present between imino and nitro groups (Table 1).

S2. Experimental

2-Nitrophenylhydrazine (1 mmol, 0.153 g) was dissolved in anhydrous ethanol (15 ml). The mixture was stirred for several min at 351 K, then the furylideneacetone (1 mmol, 0.136 g) in ethanol (8 ml) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized from methanol, red single crystals of (I) were obtained after 3 d.

S3. Refinement

All H atoms were positioned geometrically and refined as riding with C—H = 0.93 (aromatic), 0.96 Å (methyl) and N—H = 0.86 Å, and refined in riding mode with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and U_{iso}(H) = 1.2U_{eq}(C,*N*) for the others.

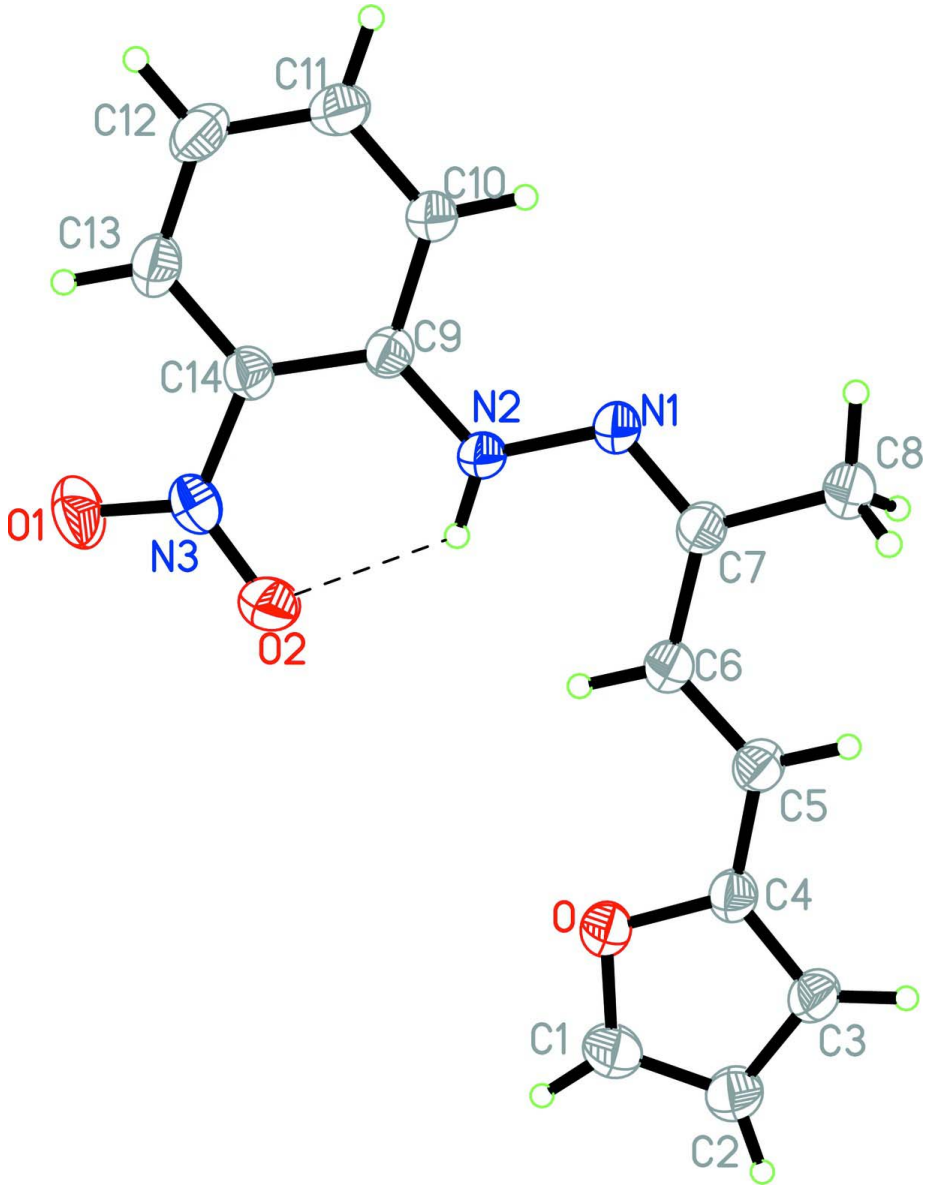

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bond is shown in dashed line.

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Crystal data

$C_{14}H_{13}N_3O_3$
$M_r = 271.27$
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Hall symbol: -P 1
$a = 8.2261$ (2) Å
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$c = 9.1027$ (2) Å
$\alpha = 89.166$ (2)°
$\beta = 77.549$ (2)°
$\gamma = 80.250$ (2)°
$V = 649.83$ (3) Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.386$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1117 reflections
$\theta = 2.3$–26.5°
$\mu = 0.1$ mm^{-1}

T = 296 K
Plate, red

0.12 × 0.10 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
9374 measured reflections
2687 independent reflections

1285 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.5°, θ_{min} = 2.3°
h = −10→10
k = −11→10
l = −11→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.136
S = 0.90
2687 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
N2	0.15950 (19)	0.59694 (17)	0.52496 (19)	0.0473 (5)
H2A	0.1972	0.5044	0.5403	0.057*
N1	0.2372 (2)	0.66928 (18)	0.40298 (19)	0.0470 (5)
O	0.58672 (18)	0.12781 (16)	0.35428 (17)	0.0599 (5)
C9	0.0217 (2)	0.6737 (2)	0.6216 (2)	0.0417 (5)
C14	−0.0559 (2)	0.6146 (2)	0.7579 (2)	0.0453 (6)
N3	−0.0014 (2)	0.4629 (2)	0.8002 (2)	0.0556 (5)
C7	0.3716 (2)	0.5962 (2)	0.3164 (2)	0.0461 (6)
O1	−0.0875 (2)	0.41053 (18)	0.9101 (2)	0.0751 (5)
C6	0.4454 (2)	0.4401 (2)	0.3302 (2)	0.0481 (6)
H6A	0.3927	0.3866	0.4093	0.058*
O2	0.1291 (2)	0.38750 (17)	0.72528 (19)	0.0727 (5)
C10	−0.0499 (3)	0.8183 (2)	0.5884 (3)	0.0519 (6)
H10A	−0.0067	0.8594	0.4969	0.062*
C3	0.7845 (3)	0.1262 (2)	0.1462 (3)	0.0581 (7)

H3A	0.8526	0.1572	0.0600	0.070*
C4	0.6560 (2)	0.2129 (2)	0.2398 (2)	0.0480 (6)
C11	−0.1819 (3)	0.9011 (2)	0.6869 (3)	0.0606 (7)
H11A	−0.2263	0.9974	0.6615	0.073*
C5	0.5839 (2)	0.3679 (2)	0.2372 (2)	0.0486 (6)
H5A	0.6390	0.4249	0.1628	0.058*
C8	0.4503 (3)	0.6843 (2)	0.1883 (3)	0.0645 (7)
H8A	0.3815	0.7816	0.1884	0.097*
H8B	0.4589	0.6319	0.0952	0.097*
H8C	0.5610	0.6961	0.1990	0.097*
C13	−0.1892 (3)	0.7005 (3)	0.8576 (3)	0.0588 (7)
H13A	−0.2368	0.6600	0.9479	0.071*
C12	−0.2507 (3)	0.8442 (3)	0.8237 (3)	0.0633 (7)
H12A	−0.3376	0.9029	0.8917	0.076*
C2	0.7968 (3)	−0.0203 (3)	0.2030 (3)	0.0648 (7)
H2B	0.8740	−0.1046	0.1615	0.078*
C1	0.6766 (3)	−0.0147 (3)	0.3274 (3)	0.0659 (7)
H1B	0.6563	−0.0965	0.3878	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0478 (10)	0.0362 (10)	0.0488 (12)	−0.0004 (8)	0.0038 (9)	0.0049 (9)
N1	0.0481 (10)	0.0431 (10)	0.0440 (11)	−0.0059 (8)	0.0013 (9)	0.0049 (9)
O	0.0627 (10)	0.0535 (10)	0.0539 (11)	−0.0022 (8)	0.0025 (8)	0.0052 (8)
C9	0.0403 (11)	0.0419 (12)	0.0415 (14)	−0.0082 (9)	−0.0047 (10)	−0.0008 (10)
C14	0.0456 (12)	0.0465 (13)	0.0440 (14)	−0.0111 (10)	−0.0078 (11)	0.0050 (11)
N3	0.0626 (13)	0.0520 (12)	0.0534 (13)	−0.0170 (10)	−0.0103 (11)	0.0130 (10)
C7	0.0441 (12)	0.0452 (13)	0.0458 (14)	−0.0076 (10)	−0.0023 (10)	−0.0006 (11)
O1	0.0893 (12)	0.0741 (12)	0.0618 (12)	−0.0307 (10)	−0.0041 (10)	0.0247 (9)
C6	0.0484 (12)	0.0458 (13)	0.0460 (14)	−0.0057 (10)	−0.0030 (11)	0.0015 (11)
O2	0.0690 (11)	0.0546 (10)	0.0825 (13)	0.0023 (8)	−0.0015 (10)	0.0187 (9)
C10	0.0535 (13)	0.0425 (13)	0.0514 (15)	−0.0020 (10)	0.0017 (11)	0.0051 (11)
C3	0.0548 (14)	0.0449 (14)	0.0628 (17)	−0.0019 (11)	0.0080 (12)	0.0013 (12)
C4	0.0465 (13)	0.0467 (13)	0.0480 (15)	−0.0096 (10)	−0.0028 (11)	0.0033 (12)
C11	0.0580 (14)	0.0459 (14)	0.0684 (18)	0.0011 (11)	−0.0007 (13)	−0.0017 (13)
C5	0.0470 (12)	0.0457 (13)	0.0499 (15)	−0.0082 (10)	−0.0034 (11)	−0.0021 (11)
C8	0.0672 (15)	0.0504 (14)	0.0618 (17)	−0.0025 (12)	0.0106 (13)	0.0065 (13)
C13	0.0563 (14)	0.0671 (16)	0.0476 (15)	−0.0122 (12)	0.0020 (12)	0.0023 (13)
C12	0.0538 (14)	0.0635 (16)	0.0607 (17)	−0.0011 (12)	0.0072 (12)	−0.0124 (14)
C2	0.0687 (16)	0.0495 (15)	0.0655 (18)	0.0015 (12)	0.0003 (14)	−0.0011 (13)
C1	0.0757 (17)	0.0452 (14)	0.0707 (18)	−0.0023 (12)	−0.0089 (15)	0.0070 (13)

Geometric parameters (Å, º)

N2—C9	1.364 (2)	C10—H10A	0.9300
N2—N1	1.372 (2)	C3—C4	1.344 (3)
N2—H2A	0.8600	C3—C2	1.407 (3)

N1—C7	1.293 (2)	C3—H3A	0.9300
O—C4	1.367 (2)	C4—C5	1.425 (3)
O—C1	1.367 (2)	C11—C12	1.382 (3)
C9—C10	1.394 (3)	C11—H11A	0.9300
C9—C14	1.409 (3)	C5—H5A	0.9300
C14—C13	1.389 (3)	C8—H8A	0.9600
C14—N3	1.440 (3)	C8—H8B	0.9600
N3—O1	1.232 (2)	C8—H8C	0.9600
N3—O2	1.238 (2)	C13—C12	1.366 (3)
C7—C6	1.451 (3)	C13—H13A	0.9300
C7—C8	1.494 (3)	C12—H12A	0.9300
C6—C5	1.337 (2)	C2—C1	1.328 (3)
C6—H6A	0.9300	C2—H2B	0.9300
C10—C11	1.366 (3)	C1—H1B	0.9300

C9—N2—N1	119.11 (16)	C3—C4—C5	132.1 (2)
C9—N2—H2A	120.4	O—C4—C5	118.40 (18)
N1—N2—H2A	120.4	C10—C11—C12	121.2 (2)
C7—N1—N2	118.00 (17)	C10—C11—H11A	119.4
C4—O—C1	106.09 (16)	C12—C11—H11A	119.4
N2—C9—C10	120.34 (19)	C6—C5—C4	127.0 (2)
N2—C9—C14	123.30 (18)	C6—C5—H5A	116.5
C10—C9—C14	116.36 (18)	C4—C5—H5A	116.5
C13—C14—C9	121.3 (2)	C7—C8—H8A	109.5
C13—C14—N3	117.0 (2)	C7—C8—H8B	109.5
C9—C14—N3	121.75 (18)	H8A—C8—H8B	109.5
O1—N3—O2	121.45 (19)	C7—C8—H8C	109.5
O1—N3—C14	118.91 (19)	H8A—C8—H8C	109.5
O2—N3—C14	119.64 (18)	H8B—C8—H8C	109.5
N1—C7—C6	126.3 (2)	C12—C13—C14	120.4 (2)
N1—C7—C8	114.50 (19)	C12—C13—H13A	119.8
C6—C7—C8	119.16 (18)	C14—C13—H13A	119.8
C5—C6—C7	124.5 (2)	C13—C12—C11	119.0 (2)
C5—C6—H6A	117.7	C13—C12—H12A	120.5
C7—C6—H6A	117.7	C11—C12—H12A	120.5
C11—C10—C9	121.6 (2)	C1—C2—C3	106.61 (19)
C11—C10—H10A	119.2	C1—C2—H2B	126.7
C9—C10—H10A	119.2	C3—C2—H2B	126.7
C4—C3—C2	107.2 (2)	C2—C1—O	110.6 (2)
C4—C3—H3A	126.4	C2—C1—H1B	124.7
C2—C3—H3A	126.4	O—C1—H1B	124.7
C3—C4—O	109.46 (18)		

C9—N2—N1—C7	177.25 (18)	C14—C9—C10—C11	3.7 (3)
N1—N2—C9—C10	6.7 (3)	C2—C3—C4—O	0.4 (3)
N1—N2—C9—C14	−173.15 (18)	C2—C3—C4—C5	−178.4 (2)
N2—C9—C14—C13	175.70 (19)	C1—O—C4—C3	−0.3 (3)
C10—C9—C14—C13	−4.2 (3)	C1—O—C4—C5	178.63 (19)

N2—C9—C14—N3	−4.5 (3)	C9—C10—C11—C12	−0.4 (4)
C10—C9—C14—N3	175.66 (18)	C7—C6—C5—C4	−175.9 (2)
C13—C14—N3—O1	9.4 (3)	C3—C4—C5—C6	173.7 (2)
C9—C14—N3—O1	−170.41 (19)	O—C4—C5—C6	−5.0 (3)
C13—C14—N3—O2	−171.21 (19)	C9—C14—C13—C12	1.5 (3)
C9—C14—N3—O2	8.9 (3)	N3—C14—C13—C12	−178.4 (2)
N2—N1—C7—C6	3.5 (3)	C14—C13—C12—C11	2.0 (4)
N2—N1—C7—C8	−178.71 (18)	C10—C11—C12—C13	−2.5 (4)
N1—C7—C6—C5	179.3 (2)	C4—C3—C2—C1	−0.2 (3)
C8—C7—C6—C5	1.6 (3)	C3—C2—C1—O	0.0 (3)
N2—C9—C10—C11	−176.2 (2)	C4—O—C1—C2	0.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2	0.86	2.00	2.611 (2)	127