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

Zhi-gang Yin,* Heng-yu Qian, Chun-xia Zhang and Zhi-qiang Yao

Key Laboratory of Surface and Interface Science of Henan, School of Materials & Chemical Engineering, Zhengzhou University of Light Industry, Zhengzhou 450002, People's Republic of China

Correspondence e-mail: yinck@263.net

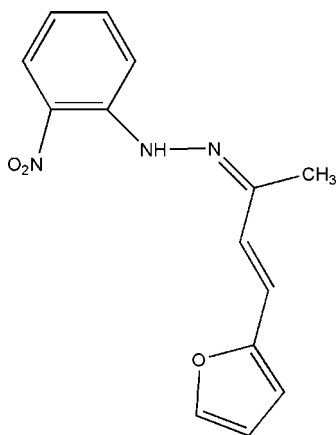
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.050; wR factor = 0.136; data-to-parameter ratio = 14.8.

In the title Schiff base compound, $\text{C}_{14}\text{H}_{13}\text{N}_3\text{O}_3$, the furan and benzene rings are oriented at a dihedral angle of 10.24 (13)°. Intramolecular $\text{N}-\text{H}\cdots\text{O}$ 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

| | |
|--|-----------------------------------|
| $\text{C}_{14}\text{H}_{13}\text{N}_3\text{O}_3$ | $\gamma = 80.250$ (2)° |
| $M_r = 271.27$ | $V = 649.83$ (3) Å ³ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 8.2261$ (2) Å | Mo $K\alpha$ radiation |
| $b = 9.0200$ (2) Å | $\mu = 0.1$ mm ⁻¹ |
| $c = 9.1027$ (2) Å | $T = 296$ K |
| $\alpha = 89.166$ (2)° | $0.12 \times 0.10 \times 0.07$ mm |
| $\beta = 77.549$ (2)° | |

Data collection

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

Refinement

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

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|-------|-------------|-------------|---------------|
| $\text{N2}-\text{H2A}\cdots\text{O2}$ | 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).

References

- Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Okabe, N., Nakamura, T. & Fukuda, H. (1993). *Acta Cryst.* **C49**, 1678–1680.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2009). E65, o2162 [doi:10.1107/S1600536809031638]

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

Z. Yin, H. Qian, C. Zhang and Z. Yao

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).

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.

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 $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ for the others.

Figures

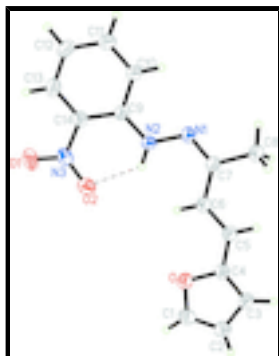


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bond is shown in dashed line.

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

Crystal data

| | |
|--------------------------------|---|
| $C_{14}H_{13}N_3O_3$ | $Z = 2$ |
| $M_r = 271.27$ | $F_{000} = 284$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.386 \text{ Mg m}^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $a = 8.2261 (2) \text{ \AA}$ | Cell parameters from 1117 reflections |
| $b = 9.0200 (2) \text{ \AA}$ | $\theta = 2.3\text{--}26.5^\circ$ |
| $c = 9.1027 (2) \text{ \AA}$ | $\mu = 0.1 \text{ mm}^{-1}$ |
| $\alpha = 89.166 (2)^\circ$ | $T = 296 \text{ K}$ |
| $\beta = 77.549 (2)^\circ$ | Plate, red |
| $\gamma = 80.250 (2)^\circ$ | $0.12 \times 0.10 \times 0.07 \text{ mm}$ |
| $V = 649.83 (3) \text{ \AA}^3$ | |

Data collection

| | |
|---|--|
| Bruker SMART CCD area-detector diffractometer | 1285 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{\text{int}} = 0.039$ |
| Monochromator: graphite | $\theta_{\text{max}} = 26.5^\circ$ |
| $T = 296 \text{ K}$ | $\theta_{\text{min}} = 2.3^\circ$ |
| ω scans | $h = -10 \rightarrow 10$ |
| Absorption correction: none | $k = -11 \rightarrow 10$ |
| 9374 measured reflections | $l = -11 \rightarrow 10$ |
| 2687 independent reflections | |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.050$ | H-atom parameters constrained |
| $wR(F^2) = 0.136$ | $w = 1/[\sigma^2(F_o^2) + (0.0668P)^2]$ |
| $S = 0.90$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2687 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 182 parameters | $\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$ |
| | Extinction correction: none |

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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| 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 (\AA^2)

U^{11} U^{22} U^{33} U^{12} U^{13} U^{23}

supplementary materials

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| 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 (\AA , $^\circ$)

| | | | |
|------------|-------------|--------------|-------------|
| 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 (\AA , $^\circ$)

| <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> |
|-------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N2—H2A \cdots O2 | 0.86 | 2.00 | 2.611 (2) | 127 |

Fig. 1

