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(*E*)-1-(3-Nitrophenyl)ethanone (2-methylphenyl)hydrazone

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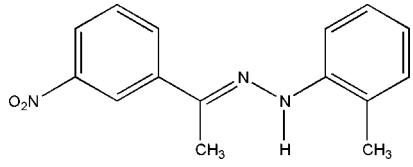
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.060; wR factor = 0.176; data-to-parameter ratio = 13.6.

In the title Schiff base compound, $C_{15}H_{15}N_3O_2$, the azomethine double bond adopts an E configuration. The dihedral angle between the two aromatic rings is 13.4 (12)°. In the crystal, molecules are arranged in wave-like layers parallel to (100) without any classical hydrogen bonding.

Related literature

For the biological activit of Schiff bases, see: Khan *et al.* (2009); Gerdemann *et al.* (2002); Mallikarjun & Sangamesh (1997); Solomon & Lowery (1993). For the role of Schiff bases and Amadori products in the process of glycation, see: Ahmad *et al.* (2007); Ahmed (2005). For the crystal structures of closely related compounds see: Fun *et al.* (2008); Tezcan *et al.* (2004).



Experimental

Crystal data

$C_{15}H_{15}N_3O_2$
M_r = 269.30
Monoclinic, $P2_1/n$
a = 7.4763 (18) Å
b = 25.742 (6) Å
c = 7.6564 (19) Å
β = 110.485 (5)°
V = 1380.3 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 273 K
0.51 × 0.46 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.956, T_{max} = 0.993
7958 measured reflections
2535 independent reflections
1746 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.176
S = 1.04
2535 reflections
187 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2637).

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supporting information

Acta Cryst. (2011). E**67**, o2801 [https://doi.org/10.1107/S1600536811037846]

(*E*)-1-(3-Nitrophenyl)ethanone (2-methylphenyl)hydrazone

A. Zeb and S. Yousuf

S1. Comment

Schiff bases represent an important class of organic compounds, with a wide range of biological properties including antifungal, antibacterial, herbicidal, antiproliferative, cytotoxic, anticonvulsant and anticancer activities (Khan *et al.*, 2009; Gerdemann *et al.*, 2002; Mallikarjun & Sangamesh, 1997; Solomon & Lowery, 1993). They are also known as important intermediates formed during the process of glycation (reaction of protein and glucose) undergoing rearrangement to form more stable Amadori products, which are considered as therapeutic targets to treat diabetes and its complications (Ahmad *et al.*, 2007; Ahmed, 2005). During our on going search for effective antiglycating agents, the title compound was prepared and crystallized.

The structure of title compound (Fig. 1) is not planar, the dihedral angle between the aromatic rings (C1—C6 and C8—C13) being 13.4 (12)°. The azomethine (C7═N2) double bond adopts an *E* configuration, with the N1–N2–C7—C6 torsion angle of 178.38 (16)°. The bond lengths and angle are similar to those observed in other structurally related compounds (Fun *et al.*, 2008; Tezcan *et al.*, 2004). In the crystal structure, the molecules are arranged in wave-like layers parallel to the (100) plane (Fig. 2) without any classical intermolecular hydrogen bonding.

S2. Experimental

The synthesis of title compound I was carried out by refluxing a mixture of 3-nitroacetophenone (165 mg, 1 mmol) and 1-(2-methylphenyl)hydrazine hydrochloride (159 mg, 1 mmol) with acetic acid (1 ml) in ethanol (10 ml) for 18 h. The progress of reaction was monitored by TLC. After cooling and filtration the crystalline product was collected, washed with hexane and dried to afford the title compound in 85% yield. Recrystallization from ethanol afforded yellowish crystals suitable for single-crystal X-ray diffraction studies. All chemicals were purchased from Sigma-Aldrich.

S3. Refinement

H atoms on methyl and methine groups were positioned geometrically with C—H = 0.96 and 0.93 Å respectively, and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms. The hydrazone H atom was located in a difference Fourier map and refined isotropically. A rotating group model was applied to the methyl groups.

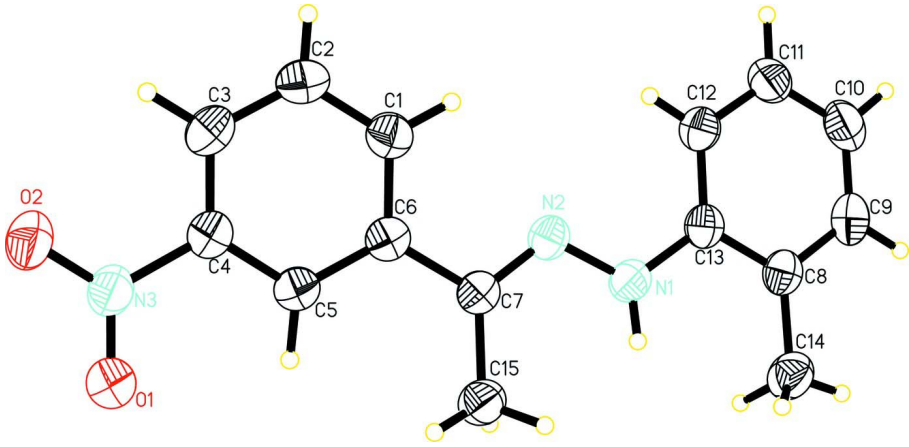

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at 30% probability level.

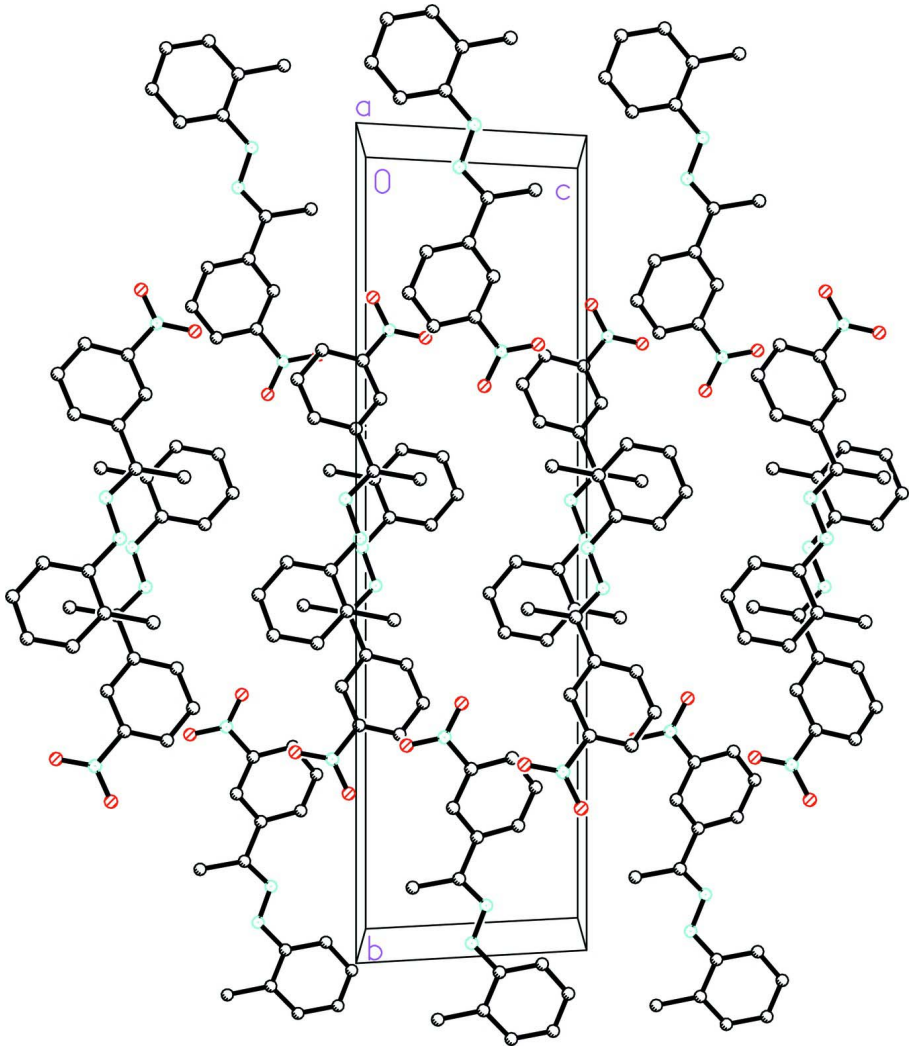

Figure 2

The crystal packing of the title compound viewed down the *a* axis. Hydrogen atoms are omitted.

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Crystal data

$C_{15}H_{15}N_3O_2$
$M_r = 269.30$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.4763\ (18)$ Å
$b = 25.742\ (6)$ Å
$c = 7.6564\ (19)$ Å
$\beta = 110.485\ (5)°$
$V = 1380.3\ (6)$ Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.296$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2070 reflections
$\theta = 3.0–24.4°$
$\mu = 0.09$ mm^{-1}
$T = 273$ K
Plate, yellow
0.51 × 0.46 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.956$, $T_{max} = 0.993$
7958 measured reflections
2535 independent reflections
1746 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.6°$
$h = -9\rightarrow9$
$k = -31\rightarrow29$
$l = -9\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.176$
$S = 1.04$
2535 reflections
187 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
$w = 1/[\sigma^2(F_o^2) + (0.0991P)^2 + 0.0824P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.0105 (4)	0.25761 (8)	0.7851 (3)	0.1120 (8)

O2	−0.0998 (3)	0.30826 (7)	0.5495 (3)	0.0983 (6)
N1	0.2485 (3)	0.00558 (7)	0.5057 (3)	0.0652 (5)
N2	0.1857 (2)	0.05385 (7)	0.4420 (2)	0.0600 (5)
N3	−0.0373 (3)	0.26679 (8)	0.6232 (3)	0.0753 (6)
C1	0.0995 (4)	0.15320 (9)	0.3011 (3)	0.0716 (6)
H1B	0.1320	0.1284	0.2290	0.086*
C2	0.0380 (4)	0.20121 (10)	0.2255 (3)	0.0780 (7)
H2B	0.0275	0.2082	0.1032	0.094*
C3	−0.0082 (3)	0.23888 (9)	0.3293 (3)	0.0705 (6)
H3A	−0.0495	0.2716	0.2797	0.085*
C4	0.0086 (3)	0.22673 (8)	0.5084 (3)	0.0603 (5)
C5	0.0684 (3)	0.17883 (8)	0.5874 (3)	0.0581 (5)
H5A	0.0778	0.1722	0.7097	0.070*
C6	0.1147 (3)	0.14059 (8)	0.4826 (3)	0.0557 (5)
C7	0.1796 (3)	0.08832 (8)	0.5615 (3)	0.0548 (5)
C8	0.3256 (3)	−0.08197 (8)	0.4538 (3)	0.0619 (6)
C9	0.3307 (3)	−0.12021 (9)	0.3286 (4)	0.0759 (7)
H9A	0.3731	−0.1533	0.3731	0.091*
C10	0.2748 (4)	−0.11080 (11)	0.1401 (4)	0.0856 (8)
H10A	0.2791	−0.1372	0.0588	0.103*
C11	0.2130 (4)	−0.06233 (11)	0.0734 (3)	0.0821 (7)
H11A	0.1747	−0.0558	−0.0539	0.099*
C12	0.2068 (3)	−0.02331 (9)	0.1923 (3)	0.0691 (6)
H12A	0.1665	0.0097	0.1456	0.083*
C13	0.2604 (3)	−0.03265 (8)	0.3823 (3)	0.0576 (5)
C14	0.3872 (4)	−0.09290 (10)	0.6573 (3)	0.0836 (7)
H14A	0.4376	−0.1275	0.6814	0.125*
H14B	0.2796	−0.0898	0.6976	0.125*
H14C	0.4840	−0.0685	0.7242	0.125*
C15	0.2345 (4)	0.07845 (9)	0.7658 (3)	0.0728 (6)
H15A	0.3563	0.0614	0.8110	0.109*
H15B	0.1401	0.0567	0.7877	0.109*
H15C	0.2422	0.1109	0.8299	0.109*
H1N1	0.285 (4)	−0.0027 (9)	0.622 (4)	0.079 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.164 (2)	0.0973 (14)	0.0794 (12)	0.0453 (13)	0.0483 (12)	0.0098 (10)
O2	0.1248 (17)	0.0622 (11)	0.0986 (13)	0.0146 (10)	0.0274 (11)	0.0023 (9)
N1	0.0681 (13)	0.0615 (11)	0.0611 (11)	0.0066 (9)	0.0163 (9)	0.0031 (9)
N2	0.0516 (11)	0.0601 (11)	0.0647 (10)	0.0009 (8)	0.0160 (8)	0.0015 (8)
N3	0.0795 (14)	0.0627 (12)	0.0776 (13)	0.0076 (10)	0.0199 (10)	0.0030 (10)
C1	0.0782 (16)	0.0720 (15)	0.0702 (14)	0.0032 (11)	0.0332 (12)	0.0025 (11)
C2	0.0897 (19)	0.0802 (17)	0.0677 (14)	0.0014 (13)	0.0320 (13)	0.0129 (12)
C3	0.0662 (15)	0.0631 (14)	0.0774 (15)	−0.0015 (11)	0.0192 (11)	0.0118 (11)
C4	0.0520 (13)	0.0586 (12)	0.0665 (12)	−0.0038 (9)	0.0161 (10)	0.0011 (10)
C5	0.0509 (12)	0.0613 (13)	0.0574 (11)	−0.0066 (9)	0.0130 (9)	0.0002 (9)

C6	0.0431 (12)	0.0613 (12)	0.0592 (11)	−0.0076 (9)	0.0134 (9)	−0.0007 (9)
C7	0.0432 (11)	0.0590 (12)	0.0593 (11)	−0.0054 (9)	0.0144 (9)	−0.0009 (9)
C8	0.0457 (12)	0.0651 (13)	0.0742 (13)	−0.0017 (9)	0.0200 (10)	0.0006 (10)
C9	0.0613 (15)	0.0673 (15)	0.1014 (18)	0.0070 (11)	0.0315 (13)	−0.0033 (12)
C10	0.0767 (18)	0.095 (2)	0.0891 (18)	0.0090 (14)	0.0334 (14)	−0.0222 (14)
C11	0.0724 (17)	0.106 (2)	0.0678 (14)	0.0161 (14)	0.0244 (12)	−0.0065 (14)
C12	0.0586 (14)	0.0786 (15)	0.0685 (13)	0.0098 (11)	0.0202 (11)	0.0034 (11)
C13	0.0413 (11)	0.0634 (13)	0.0673 (13)	−0.0017 (9)	0.0180 (9)	−0.0010 (10)
C14	0.0877 (19)	0.0762 (16)	0.0867 (17)	0.0121 (13)	0.0302 (14)	0.0189 (13)
C15	0.0769 (17)	0.0654 (14)	0.0697 (14)	0.0015 (11)	0.0180 (12)	0.0036 (10)

Geometric parameters (Å, º)

O1—N3	1.207 (2)	C7—C15	1.493 (3)
O2—N3	1.222 (2)	C8—C9	1.384 (3)
N1—N2	1.357 (2)	C8—C13	1.401 (3)
N1—C13	1.389 (3)	C8—C14	1.489 (3)
N1—H1N1	0.86 (2)	C9—C10	1.376 (3)
N2—C7	1.286 (2)	C9—H9A	0.9300
N3—C4	1.471 (3)	C10—C11	1.366 (4)
C1—C2	1.374 (3)	C10—H10A	0.9300
C1—C6	1.392 (3)	C11—C12	1.367 (3)
C1—H1B	0.9300	C11—H11A	0.9300
C2—C3	1.372 (3)	C12—C13	1.388 (3)
C2—H2B	0.9300	C12—H12A	0.9300
C3—C4	1.369 (3)	C14—H14A	0.9600
C3—H3A	0.9300	C14—H14B	0.9600
C4—C5	1.377 (3)	C14—H14C	0.9600
C5—C6	1.388 (3)	C15—H15A	0.9600
C5—H5A	0.9300	C15—H15B	0.9600
C6—C7	1.485 (3)	C15—H15C	0.9600
N2—N1—C13	120.06 (18)	C9—C8—C14	121.1 (2)
N2—N1—H1N1	122.9 (16)	C13—C8—C14	121.22 (19)
C13—N1—H1N1	117.0 (16)	C10—C9—C8	122.0 (2)
C7—N2—N1	118.04 (17)	C10—C9—H9A	119.0
O1—N3—O2	123.0 (2)	C8—C9—H9A	119.0
O1—N3—C4	119.10 (19)	C11—C10—C9	119.4 (2)
O2—N3—C4	117.9 (2)	C11—C10—H10A	120.3
C2—C1—C6	121.9 (2)	C9—C10—H10A	120.3
C2—C1—H1B	119.1	C10—C11—C12	120.6 (2)
C6—C1—H1B	119.1	C10—C11—H11A	119.7
C3—C2—C1	120.5 (2)	C12—C11—H11A	119.7
C3—C2—H2B	119.8	C11—C12—C13	120.4 (2)
C1—C2—H2B	119.8	C11—C12—H12A	119.8
C4—C3—C2	117.7 (2)	C13—C12—H12A	119.8
C4—C3—H3A	121.1	C12—C13—N1	121.79 (19)
C2—C3—H3A	121.1	C12—C13—C8	120.01 (19)

C3—C4—C5	123.11 (19)	N1—C13—C8	118.19 (19)
C3—C4—N3	118.66 (19)	C8—C14—H14A	109.5
C5—C4—N3	118.24 (19)	C8—C14—H14B	109.5
C4—C5—C6	119.32 (19)	H14A—C14—H14B	109.5
C4—C5—H5A	120.3	C8—C14—H14C	109.5
C6—C5—H5A	120.3	H14A—C14—H14C	109.5
C5—C6—C1	117.51 (19)	H14B—C14—H14C	109.5
C5—C6—C7	121.31 (18)	C7—C15—H15A	109.5
C1—C6—C7	121.17 (18)	C7—C15—H15B	109.5
N2—C7—C6	115.08 (17)	H15A—C15—H15B	109.5
N2—C7—C15	124.21 (18)	C7—C15—H15C	109.5
C6—C7—C15	120.72 (17)	H15A—C15—H15C	109.5
C9—C8—C13	117.6 (2)	H15B—C15—H15C	109.5

C13—N1—N2—C7	−178.96 (16)	C5—C6—C7—N2	168.33 (17)
C6—C1—C2—C3	1.0 (4)	C1—C6—C7—N2	−12.2 (3)
C1—C2—C3—C4	−0.3 (4)	C5—C6—C7—C15	−12.3 (3)
C2—C3—C4—C5	−0.2 (3)	C1—C6—C7—C15	167.1 (2)
C2—C3—C4—N3	179.1 (2)	C13—C8—C9—C10	0.2 (3)
O1—N3—C4—C3	−175.0 (2)	C14—C8—C9—C10	−179.8 (2)
O2—N3—C4—C3	4.4 (3)	C8—C9—C10—C11	0.2 (4)
O1—N3—C4—C5	4.4 (3)	C9—C10—C11—C12	0.2 (4)
O2—N3—C4—C5	−176.2 (2)	C10—C11—C12—C13	−1.1 (4)
C3—C4—C5—C6	0.1 (3)	C11—C12—C13—N1	−177.4 (2)
N3—C4—C5—C6	−179.27 (18)	C11—C12—C13—C8	1.6 (3)
C4—C5—C6—C1	0.6 (3)	N2—N1—C13—C12	−1.0 (3)
C4—C5—C6—C7	−179.92 (18)	N2—N1—C13—C8	−179.92 (17)
C2—C1—C6—C5	−1.2 (3)	C9—C8—C13—C12	−1.1 (3)
C2—C1—C6—C7	179.4 (2)	C14—C8—C13—C12	179.0 (2)
N1—N2—C7—C6	178.38 (16)	C9—C8—C13—N1	177.85 (18)
N1—N2—C7—C15	−1.0 (3)	C14—C8—C13—N1	−2.1 (3)