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2-Methyl-*N*′-(4-nitrobenzylidene)-benzohydrazide

Chun-Bao Tang

Department of Chemistry, Jiaying University, Meizhou 514015, People's Republic of China
Correspondence e-mail: tangchunbao@yahoo.com.cn

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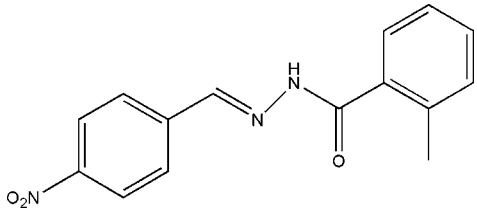
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.059; wR factor = 0.140; data-to-parameter ratio = 15.2.

The title hydrazone compound, $C_{15}H_{13}N_3O_3$, was prepared by the condensation of 4-nitrobenzaldehyde with 2-methylbenzohydrazide in methanol. The dihedral angle between the two benzene rings is 14.8 (2)°. In the crystal, molecules are linked through intermolecular N—H···O hydrogen bonds, forming chains along the *a* axis.

Related literature

For general background to hydrazones, see: Rasras *et al.* (2010); Pyta *et al.* (2010); Angelusiu *et al.* (2010); Fun *et al.* (2008); Singh & Singh (2010); Ahmad *et al.* (2010). For bond-length data, see: Allen *et al.* (1987). For a similar hydrazone compound reported recently by the author, see: Tang (2010).



Experimental

Crystal data

$C_{15}H_{13}N_3O_3$
M_r = 283.28
Monoclinic, $P2_1/n$
a = 7.416 (1) Å
b = 26.198 (3) Å
c = 7.860 (2) Å
β = 114.206 (1)°
V = 1392.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 K
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.981, T_{max} = 0.983
7336 measured reflections
2941 independent reflections
1696 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.140
S = 1.01
2941 reflections
194 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O3^{i}	0.90 (1)	1.99 (1)	2.870 (2)	167 (3)

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2493).

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supporting information

Acta Cryst. (2010). E**66**, o2715 [https://doi.org/10.1107/S1600536810038869]

2-Methyl-*N*′-(4-nitrobenzylidene)benzohydrazide

Chun-Bao Tang

S1. Comment

Hydrazone compounds have received much attention in biological chemistry and structural chemistry in the last few years (Rasras *et al.*, 2010; Pyta *et al.*, 2010; Angelusiu *et al.*, 2010; Fun *et al.*, 2008; Singh & Singh, 2010; Ahmad *et al.*, 2010). In the present paper, the author reports the crystal structure of the title new hydrazone compound (Fig. 1).

In the title molecule, the dihedral angle between the two benzene rings is 14.8 (2)°. The torsion angles C4—C7—N2—N3, C7—N2—N3—C8 and N2—N3—C8—C9 are 3.9 (2), 13.8 (2), and 1.5 (2)°, respectively. All the bond lengths are within normal values (Allen *et al.*, 1987) and comparable with those of a similar hydrazone compound the author reported recently (Tang, 2010).

In the crystal structure of the compound, molecules are linked through N–H···O intermolecular hydrogen bonds (Table 1), forming chains along the *a* axis (Fig. 2).

S2. Experimental

4-Nitrobenzaldehyde (0.1 mmol, 15.1 mg) and 3-methylbenzohydrazide (0.1 mmol, 15.0 mg) were dissolved in methanol (20 ml). The mixture was stirred at reflux for 10 min to give a clear yellow solution. Yellow block-shaped crystals of the title compound were formed by slow evaporation of the solvent over several days.

S3. Refinement

Atom H3 was located in a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90 (1) Å [U_{iso}(H) = 0.08 Å^2]. Other H atoms were constrained to ideal geometries, with C–H = 0.93–0.96 Å, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms.

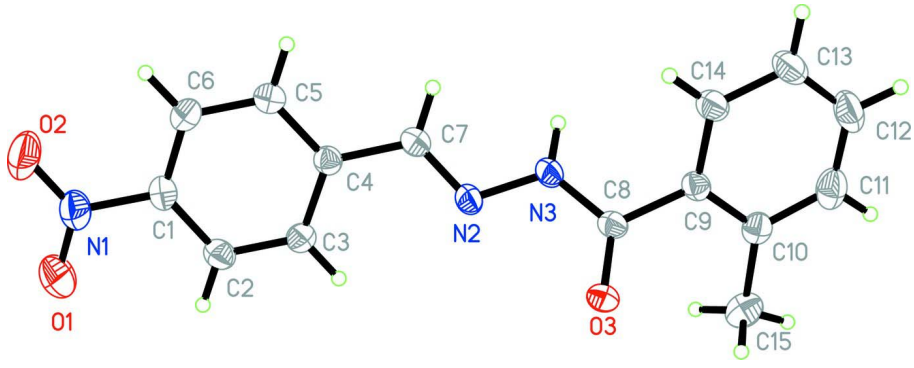

Figure 1

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

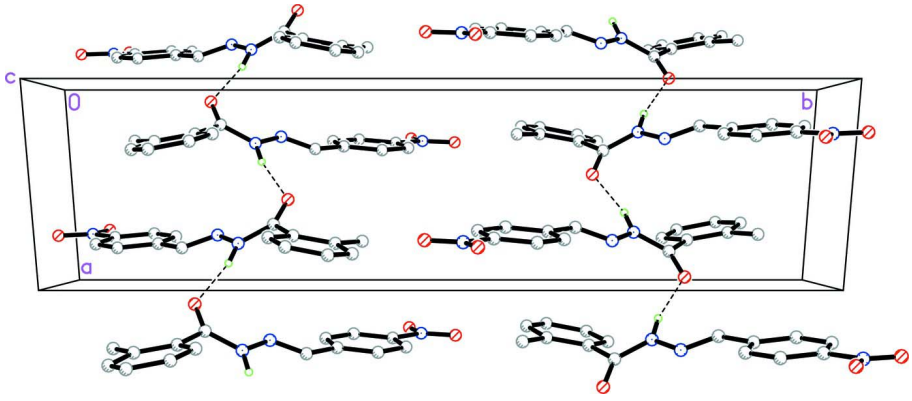

Figure 2

Molecular packing of the title compound viewed along the *c* axis, with hydrogen bonds shown as dashed lines.

2-Methyl-*N*′-(4-nitrobenzylidene)benzohydrazide

Crystal data

$C_{15}H_{13}N_3O_3$
$M_r = 283.28$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.416$ (1) Å
$b = 26.198$ (3) Å
$c = 7.860$ (2) Å
$\beta = 114.206$ (1)°
$V = 1392.8$ (4) Å^3
$Z = 4$
$F(000) = 592$
$D_x = 1.351$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 996 reflections
$\theta = 2.7$–24.5°
$\mu = 0.10$ mm^{-1}
$T = 298$ K
Block, yellow
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.981$, $T_{max} = 0.983$
7336 measured reflections
2941 independent reflections
1696 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 27.0°$, $\theta_{min} = 3.0°$
$h = -6 \rightarrow 9$
$k = -28 \rightarrow 33$
$l = -10 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.140$
$S = 1.01$
2941 reflections
194 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.3174P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ 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}
N1	0.2497 (4)	0.47404 (10)	−0.4463 (3)	0.0669 (7)
N2	0.2408 (3)	0.28740 (7)	0.0842 (2)	0.0433 (5)
N3	0.2780 (3)	0.25697 (8)	0.2388 (3)	0.0448 (5)
O1	0.2312 (4)	0.45771 (9)	−0.5969 (3)	0.0985 (8)
O2	0.2575 (5)	0.51917 (9)	−0.4093 (3)	0.1195 (10)
O3	0.0631 (2)	0.19571 (6)	0.0688 (2)	0.0510 (5)
C1	0.2623 (3)	0.43743 (9)	−0.3004 (3)	0.0456 (6)
C2	0.2438 (3)	0.38615 (9)	−0.3415 (3)	0.0470 (6)
H2	0.2237	0.3747	−0.4599	0.056*
C3	0.2557 (3)	0.35214 (9)	−0.2037 (3)	0.0440 (6)
H3A	0.2420	0.3174	−0.2301	0.053*
C4	0.2877 (3)	0.36894 (8)	−0.0257 (3)	0.0380 (5)
C5	0.3081 (4)	0.42089 (9)	0.0113 (3)	0.0504 (6)
H5	0.3316	0.4326	0.1302	0.060*
C6	0.2938 (4)	0.45538 (9)	−0.1266 (3)	0.0527 (7)
H6	0.3055	0.4902	−0.1019	0.063*
C7	0.3044 (3)	0.33305 (9)	0.1220 (3)	0.0431 (6)
H7	0.3625	0.3436	0.2458	0.052*
C8	0.1838 (3)	0.21176 (9)	0.2194 (3)	0.0383 (5)
C9	0.2356 (3)	0.18386 (9)	0.3991 (3)	0.0382 (5)
C10	0.2726 (3)	0.13129 (9)	0.4118 (3)	0.0440 (6)
C11	0.3170 (4)	0.10849 (11)	0.5841 (4)	0.0620 (8)
H11	0.3466	0.0738	0.5981	0.074*
C12	0.3184 (4)	0.13555 (13)	0.7343 (4)	0.0689 (9)
H12	0.3460	0.1188	0.8466	0.083*
C13	0.2796 (4)	0.18702 (12)	0.7209 (3)	0.0620 (8)
H13	0.2801	0.2052	0.8226	0.074*
C14	0.2399 (4)	0.21091 (10)	0.5539 (3)	0.0489 (6)
H14	0.2155	0.2458	0.5437	0.059*
C15	0.2671 (4)	0.09958 (10)	0.2502 (4)	0.0589 (7)
H15A	0.1333	0.0970	0.1585	0.088*
H15B	0.3172	0.0660	0.2935	0.088*
H15C	0.3474	0.1154	0.1954	0.088*
H3	0.373 (3)	0.2669 (10)	0.349 (2)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0865 (18)	0.0589 (17)	0.0603 (16)	0.0004 (13)	0.0353 (14)	0.0137 (13)
N2	0.0457 (12)	0.0447 (12)	0.0331 (10)	−0.0031 (9)	0.0098 (9)	0.0067 (9)
N3	0.0462 (13)	0.0476 (12)	0.0294 (10)	−0.0062 (10)	0.0041 (9)	0.0065 (9)
O1	0.158 (2)	0.0905 (17)	0.0585 (14)	−0.0072 (15)	0.0554 (15)	0.0135 (12)
O2	0.222 (3)	0.0534 (15)	0.1011 (19)	0.0010 (17)	0.084 (2)	0.0192 (14)
O3	0.0561 (11)	0.0503 (10)	0.0322 (9)	−0.0084 (8)	0.0035 (8)	−0.0006 (8)
C1	0.0468 (15)	0.0457 (15)	0.0461 (14)	0.0031 (12)	0.0209 (12)	0.0088 (12)
C2	0.0510 (16)	0.0525 (16)	0.0385 (13)	0.0020 (12)	0.0193 (12)	−0.0007 (12)
C3	0.0493 (15)	0.0384 (13)	0.0427 (14)	0.0021 (11)	0.0172 (12)	−0.0015 (11)
C4	0.0350 (13)	0.0400 (14)	0.0365 (13)	0.0002 (10)	0.0121 (11)	0.0020 (10)
C5	0.0614 (17)	0.0467 (16)	0.0421 (14)	−0.0016 (12)	0.0204 (13)	−0.0029 (12)
C6	0.0657 (18)	0.0376 (14)	0.0555 (16)	−0.0021 (12)	0.0255 (14)	−0.0009 (12)
C7	0.0437 (14)	0.0463 (15)	0.0343 (13)	−0.0003 (11)	0.0109 (11)	0.0007 (11)
C8	0.0382 (13)	0.0418 (14)	0.0309 (12)	0.0019 (11)	0.0101 (11)	−0.0012 (10)
C9	0.0310 (12)	0.0467 (15)	0.0322 (12)	−0.0024 (10)	0.0081 (10)	0.0031 (10)
C10	0.0331 (13)	0.0458 (15)	0.0479 (14)	−0.0018 (11)	0.0113 (11)	0.0043 (11)
C11	0.0518 (17)	0.0580 (17)	0.0681 (19)	−0.0012 (14)	0.0162 (15)	0.0195 (15)
C12	0.0616 (19)	0.090 (2)	0.0449 (16)	−0.0146 (17)	0.0116 (14)	0.0229 (16)
C13	0.0640 (19)	0.085 (2)	0.0356 (14)	−0.0184 (16)	0.0189 (13)	−0.0034 (14)
C14	0.0499 (15)	0.0561 (16)	0.0381 (13)	−0.0060 (12)	0.0156 (12)	−0.0016 (12)
C15	0.0514 (17)	0.0499 (16)	0.0742 (19)	0.0054 (13)	0.0246 (15)	−0.0065 (14)

Geometric parameters (Å, º)

N1—O1	1.213 (3)	C6—H6	0.9300
N1—O2	1.213 (3)	C7—H7	0.9300
N1—C1	1.468 (3)	C8—C9	1.494 (3)
N2—C7	1.275 (3)	C9—C14	1.397 (3)
N2—N3	1.383 (2)	C9—C10	1.400 (3)
N3—C8	1.351 (3)	C10—C11	1.391 (3)
N3—H3	0.900 (10)	C10—C15	1.504 (3)
O3—C8	1.229 (2)	C11—C12	1.373 (4)
C1—C6	1.371 (3)	C11—H11	0.9300
C1—C2	1.375 (3)	C12—C13	1.374 (4)
C2—C3	1.377 (3)	C12—H12	0.9300
C2—H2	0.9300	C13—C14	1.373 (3)
C3—C4	1.391 (3)	C13—H13	0.9300
C3—H3A	0.9300	C14—H14	0.9300
C4—C5	1.387 (3)	C15—H15A	0.9600
C4—C7	1.459 (3)	C15—H15B	0.9600
C5—C6	1.382 (3)	C15—H15C	0.9600
C5—H5	0.9300		
O1—N1—O2	123.6 (3)	O3—C8—N3	123.2 (2)
O1—N1—C1	118.5 (3)	O3—C8—C9	123.1 (2)

O2—N1—C1	117.9 (2)	N3—C8—C9	113.66 (19)
C7—N2—N3	114.48 (19)	C14—C9—C10	120.2 (2)
C8—N3—N2	119.86 (18)	C14—C9—C8	118.7 (2)
C8—N3—H3	121.8 (18)	C10—C9—C8	121.0 (2)
N2—N3—H3	118.2 (18)	C11—C10—C9	116.9 (2)
C6—C1—C2	121.9 (2)	C11—C10—C15	119.9 (2)
C6—C1—N1	119.0 (2)	C9—C10—C15	123.1 (2)
C2—C1—N1	119.1 (2)	C12—C11—C10	122.1 (3)
C1—C2—C3	118.6 (2)	C12—C11—H11	119.0
C1—C2—H2	120.7	C10—C11—H11	119.0
C3—C2—H2	120.7	C11—C12—C13	120.9 (3)
C2—C3—C4	121.0 (2)	C11—C12—H12	119.5
C2—C3—H3A	119.5	C13—C12—H12	119.5
C4—C3—H3A	119.5	C14—C13—C12	118.5 (3)
C5—C4—C3	118.7 (2)	C14—C13—H13	120.8
C5—C4—C7	119.9 (2)	C12—C13—H13	120.8
C3—C4—C7	121.3 (2)	C13—C14—C9	121.4 (2)
C6—C5—C4	120.7 (2)	C13—C14—H14	119.3
C6—C5—H5	119.7	C9—C14—H14	119.3
C4—C5—H5	119.7	C10—C15—H15A	109.5
C1—C6—C5	119.0 (2)	C10—C15—H15B	109.5
C1—C6—H6	120.5	H15A—C15—H15B	109.5
C5—C6—H6	120.5	C10—C15—H15C	109.5
N2—C7—C4	121.1 (2)	H15A—C15—H15C	109.5
N2—C7—H7	119.5	H15B—C15—H15C	109.5
C4—C7—H7	119.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O3^{i}	0.90 (1)	1.99 (1)	2.870 (2)	167 (3)

Symmetry code: (i) $x+1/2, -y+1/2, z+1/2$.