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N′-(2-Methoxybenzylidene)-2-nitrobenzohydrazide

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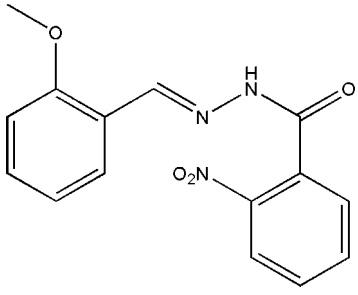
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.132; data-to-parameter ratio = 15.5.

The title compound, $C_{15}H_{13}N_3O_4$, was synthesized by the reaction of equimolar quantities of 2-methoxybenzaldehyde and 2-nitrobenzohydrazide in methanol. The dihedral angle between the two substituted benzene rings is 68.3 (2)°. In the crystal structure, inversion dimers linked by pairs of N—H···O hydrogen bonds occur.

Related literature

For the pharmacological properties of hydrazone compounds, see: Beraldo & Gambino (2004). For related structures, see: Galić *et al.* (2001); Richardson & Bernhardt (1999); Ali *et al.* (2004). For bond length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{15}H_{13}N_3O_4$
M_r = 299.28
Triclinic, $P\overline{1}$
a = 7.491 (2) Å
b = 9.427 (3) Å
c = 10.977 (3) Å
α = 91.748 (4)°
β = 106.218 (4)°
γ = 92.221 (4)°
V = 743.1 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 K
0.23 × 0.23 × 0.22 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.978, T_{max} = 0.979
6232 measured reflections
3140 independent reflections
2018 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.132
S = 1.03
3140 reflections
203 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.910 (9)	1.943 (10)	2.844 (2)	170.3 (18)

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: SJ2574).

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supplementary materials

Acta Cryst. (2009). E**65**, o577 [**doi:10.1107/S1600536809005753**]

N'-(2-Methoxybenzylidene)-2-nitrobenzohydrazide

G.-J. Xiao and C. Wei

Comment

Hydrazone compounds have received considerable attention due to their pharmacological properties (Beraldo & Gambino, 2004). In the last few years, the crystal structures and properties of a series of hydrazone compounds have been reported (Galić *et al.*, 2001; Richardson & Bernhardt, 1999; Ali *et al.*, 2004). As a continuation of work on these compounds, we report here the structure of the title compound, (I) Fig. 1.

In (I), the dihedral angle between the C1—C6 and C9—C14 benzene rings is 111.7 (2)° while that between the O2—N3—O3 nitro plane and the plane of the C1—C6 benzene ring is 26.7 (2)°. Bond lengths in the compound are found to have normal values (Allen *et al.*, 1987). The methoxy group is coplanar with the C9—C14 benzene ring, with a C15—O4—C10—C11 torsion angle of -3.2 (2)°.

In the crystal packing, adjacent molecules are linked through intermolecular N1–H1···O1 hydrogen bonds (Table 1), forming dimers (Fig. 2).

Experimental

The title compound was synthesized by the reaction of equimolar quantities (1.0 mmol each) of 2-methoxybenzaldehyde and 2-nitrobenzohydrazide in methanol (100 ml) for 3 h at room temperature. The solution was kept in air for a few days, forming colorless block-like crystals of the compound.

Refinement

The N-bound H atom was located in a difference Fourier map and was refined with an N–H distance restraint of 0.90 (1) Å. C-bound H atoms were placed in calculated positions (C–H = 0.93–0.96 Å) and refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(C15). Crystals were small and weakly diffracting which explains the relatively low data fraction.

Figures

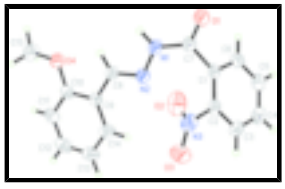

Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

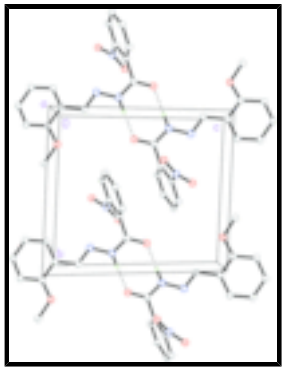

Fig. 2. The crystal packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

N'-(2-Methoxybenzylidene)-2-nitrobenzohydrazide

Crystal data

$C_{15}H_{13}N_3O_4$	$Z = 2$
$M_r = 299.28$	$F_{000} = 312$
Triclinic, $P\overline{1}$	$D_x = 1.338$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.491$ (2) Å	Cell parameters from 1428 reflections
$b = 9.427$ (3) Å	$\theta = 2.8$–24.9º
$c = 10.977$ (3) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 91.748$ (4)º	$T = 298$ K
$\beta = 106.218$ (4)º	Block, colorless
$\gamma = 92.221$ (4)º	0.23 × 0.23 × 0.22 mm
$V = 743.1$ (4) Å^3	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3140 independent reflections
Radiation source: fine-focus sealed tube	2018 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
$T = 298$ K	$\theta_{max} = 27.0$º
ω scans	$\theta_{min} = 1.9$º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -9 \rightarrow 9$
$T_{min} = 0.978$, $T_{max} = 0.979$	$k = -12 \rightarrow 11$
6232 measured reflections	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.048$	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.132$	$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 0.0142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
3140 reflections	$\Delta\rho_{max} = 0.14$ e Å^{-3}
203 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	0.6095 (2)	0.83513 (13)	0.54829 (12)	0.0763 (4)
O2	0.1921 (2)	0.69215 (17)	0.37644 (16)	0.0874 (5)
O3	0.1059 (2)	0.54863 (19)	0.2124 (2)	0.1155 (7)
O4	0.32257 (18)	1.20069 (12)	0.01171 (11)	0.0635 (4)
N1	0.4749 (2)	0.90208 (15)	0.35161 (13)	0.0609 (4)
N2	0.3966 (2)	0.86740 (14)	0.22397 (12)	0.0526 (4)
N3	0.2239 (2)	0.60480 (19)	0.30207 (19)	0.0714 (5)
C1	0.5624 (2)	0.65719 (17)	0.38490 (14)	0.0479 (4)
C2	0.4158 (2)	0.56387 (18)	0.32220 (16)	0.0504 (4)
C3	0.4423 (3)	0.43069 (18)	0.27844 (17)	0.0620 (5)
H3	0.3408	0.3709	0.2360	0.074*
C4	0.6193 (3)	0.3869 (2)	0.29785 (19)	0.0686 (5)
H4	0.6392	0.2967	0.2690	0.082*
C5	0.7670 (3)	0.4760 (2)	0.35974 (18)	0.0663 (5)
H5	0.8875	0.4463	0.3725	0.080*
C6	0.7390 (2)	0.6095 (2)	0.40340 (16)	0.0590 (5)
H6	0.8411	0.6684	0.4461	0.071*
C7	0.5456 (3)	0.80422 (18)	0.43443 (16)	0.0559 (5)
C8	0.3456 (2)	0.97399 (17)	0.15700 (15)	0.0496 (4)
H8	0.3691	1.0645	0.1958	0.060*
C9	0.2516 (2)	0.95924 (17)	0.02188 (15)	0.0473 (4)
C10	0.2377 (2)	1.07927 (19)	−0.05146 (15)	0.0507 (4)
C11	0.1419 (3)	1.0699 (2)	−0.17899 (17)	0.0675 (5)
H11	0.1328	1.1499	−0.2275	0.081*

C12	0.0606 (3)	0.9428 (3)	−0.2335 (2)	0.0777 (6)
H12	−0.0041	0.9371	−0.3193	0.093*
C13	0.0727 (3)	0.8231 (2)	−0.1637 (2)	0.0735 (6)
H13	0.0170	0.7370	−0.2020	0.088*
C14	0.1680 (3)	0.8317 (2)	−0.03657 (18)	0.0618 (5)
H14	0.1763	0.7508	0.0107	0.074*
C15	0.3219 (4)	1.3252 (2)	−0.0573 (2)	0.0835 (7)
H15A	0.3822	1.3086	−0.1227	0.125*
H15B	0.3874	1.4019	−0.0009	0.125*
H15C	0.1958	1.3497	−0.0953	0.125*
H1	0.462 (3)	0.9897 (13)	0.3841 (18)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1187 (12)	0.0584 (8)	0.0431 (8)	0.0124 (8)	0.0071 (7)	0.0026 (6)
O2	0.0841 (11)	0.0920 (11)	0.1047 (12)	0.0271 (9)	0.0521 (9)	0.0251 (10)
O3	0.0594 (10)	0.1026 (13)	0.1606 (18)	−0.0046 (9)	−0.0059 (11)	−0.0071 (12)
O4	0.0816 (9)	0.0535 (7)	0.0498 (7)	−0.0058 (6)	0.0095 (6)	0.0122 (6)
N1	0.0937 (12)	0.0441 (8)	0.0412 (8)	0.0091 (8)	0.0119 (8)	0.0035 (6)
N2	0.0670 (9)	0.0485 (8)	0.0426 (8)	0.0070 (7)	0.0149 (7)	0.0036 (6)
N3	0.0590 (11)	0.0633 (11)	0.0941 (14)	0.0018 (9)	0.0236 (10)	0.0200 (10)
C1	0.0598 (11)	0.0455 (9)	0.0382 (9)	0.0046 (8)	0.0122 (7)	0.0101 (7)
C2	0.0515 (10)	0.0488 (10)	0.0536 (10)	0.0057 (8)	0.0180 (8)	0.0126 (8)
C3	0.0713 (13)	0.0459 (10)	0.0673 (12)	−0.0034 (9)	0.0175 (10)	0.0045 (9)
C4	0.0838 (15)	0.0526 (11)	0.0747 (13)	0.0144 (10)	0.0292 (11)	0.0060 (10)
C5	0.0629 (12)	0.0700 (13)	0.0697 (13)	0.0201 (10)	0.0213 (10)	0.0160 (10)
C6	0.0552 (11)	0.0629 (12)	0.0541 (11)	0.0023 (9)	0.0067 (8)	0.0105 (9)
C7	0.0734 (12)	0.0496 (10)	0.0427 (10)	0.0033 (9)	0.0127 (8)	0.0069 (8)
C8	0.0599 (10)	0.0437 (9)	0.0454 (10)	0.0036 (8)	0.0148 (8)	0.0032 (8)
C9	0.0486 (9)	0.0490 (10)	0.0459 (9)	0.0047 (7)	0.0153 (7)	0.0012 (8)
C10	0.0483 (10)	0.0598 (11)	0.0436 (9)	0.0040 (8)	0.0118 (7)	0.0036 (8)
C11	0.0670 (12)	0.0854 (15)	0.0466 (11)	0.0043 (11)	0.0096 (9)	0.0082 (10)
C12	0.0665 (13)	0.1108 (18)	0.0486 (11)	0.0036 (12)	0.0059 (9)	−0.0106 (12)
C13	0.0657 (13)	0.0799 (15)	0.0706 (14)	−0.0082 (11)	0.0166 (10)	−0.0269 (12)
C14	0.0641 (12)	0.0577 (11)	0.0635 (12)	0.0007 (9)	0.0191 (9)	−0.0070 (9)
C15	0.1165 (18)	0.0631 (13)	0.0682 (14)	−0.0017 (12)	0.0200 (12)	0.0229 (11)

Geometric parameters (Å, °)

O1—C7	1.2283 (19)	C5—C6	1.377 (3)
O2—N3	1.218 (2)	C5—H5	0.9300
O3—N3	1.215 (2)	C6—H6	0.9300
O4—C10	1.358 (2)	C8—C9	1.453 (2)
O4—C15	1.416 (2)	C8—H8	0.9300
N1—C7	1.333 (2)	C9—C14	1.385 (2)
N1—N2	1.3828 (19)	C9—C10	1.399 (2)
N1—H1	0.910 (9)	C10—C11	1.382 (2)
N2—C8	1.270 (2)	C11—C12	1.364 (3)

N3—C2	1.461 (2)	C11—H11	0.9300
C1—C6	1.376 (2)	C12—C13	1.375 (3)
C1—C2	1.386 (2)	C12—H12	0.9300
C1—C7	1.496 (2)	C13—C14	1.377 (3)
C2—C3	1.372 (2)	C13—H13	0.9300
C3—C4	1.365 (3)	C14—H14	0.9300
C3—H3	0.9300	C15—H15A	0.9600
C4—C5	1.366 (3)	C15—H15B	0.9600
C4—H4	0.9300	C15—H15C	0.9600
C10—O4—C15	118.70 (14)	N1—C7—C1	118.62 (15)
C7—N1—N2	121.79 (14)	N2—C8—C9	122.23 (15)
C7—N1—H1	117.1 (13)	N2—C8—H8	118.9
N2—N1—H1	120.3 (13)	C9—C8—H8	118.9
C8—N2—N1	113.81 (14)	C14—C9—C10	118.43 (16)
O3—N3—O2	124.2 (2)	C14—C9—C8	122.47 (16)
O3—N3—C2	117.72 (19)	C10—C9—C8	119.04 (15)
O2—N3—C2	118.09 (18)	O4—C10—C11	124.23 (17)
C6—C1—C2	116.74 (16)	O4—C10—C9	115.40 (14)
C6—C1—C7	117.41 (16)	C11—C10—C9	120.37 (17)
C2—C1—C7	125.85 (16)	C12—C11—C10	119.7 (2)
C3—C2—C1	122.53 (17)	C12—C11—H11	120.2
C3—C2—N3	117.31 (17)	C10—C11—H11	120.2
C1—C2—N3	120.16 (16)	C11—C12—C13	121.14 (19)
C4—C3—C2	119.23 (18)	C11—C12—H12	119.4
C4—C3—H3	120.4	C13—C12—H12	119.4
C2—C3—H3	120.4	C12—C13—C14	119.44 (19)
C3—C4—C5	119.76 (18)	C12—C13—H13	120.3
C3—C4—H4	120.1	C14—C13—H13	120.3
C5—C4—H4	120.1	C13—C14—C9	120.9 (2)
C4—C5—C6	120.57 (19)	C13—C14—H14	119.5
C4—C5—H5	119.7	C9—C14—H14	119.5
C6—C5—H5	119.7	O4—C15—H15A	109.5
C1—C6—C5	121.16 (18)	O4—C15—H15B	109.5
C1—C6—H6	119.4	H15A—C15—H15B	109.5
C5—C6—H6	119.4	O4—C15—H15C	109.5
O1—C7—N1	121.37 (16)	H15A—C15—H15C	109.5
O1—C7—C1	119.77 (15)	H15B—C15—H15C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.910 (9)	1.943 (10)	2.844 (2)	170.3 (18)

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1.

Fig. 1

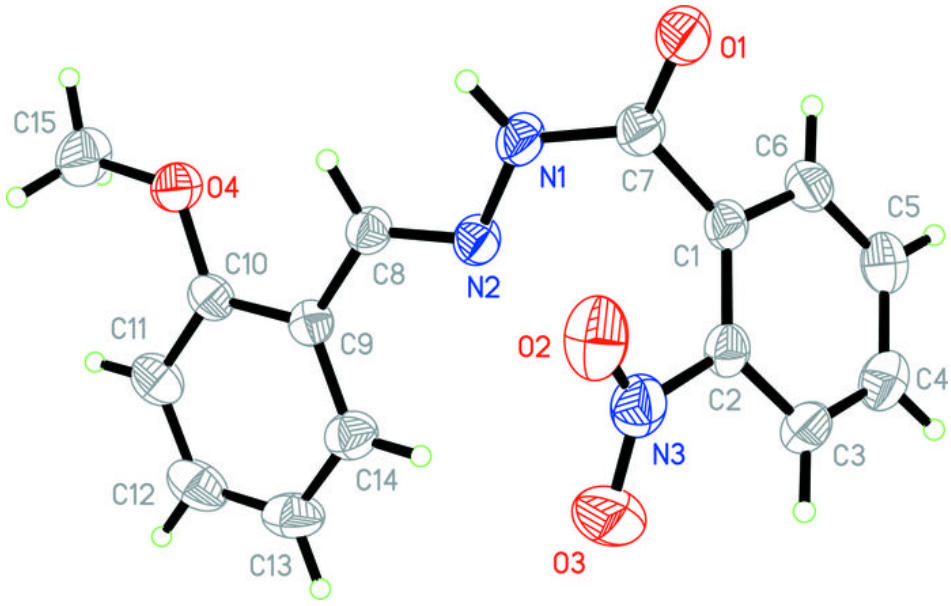
O1
C15
O4
N1
C7
C6
C5
C10
C8
N2
C1
C11
C9
O2
C4
C2
N3
C12
C14
C3
C13
O3

Fig. 2