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(*E*)-4-Methoxy-*N*′-(4-methylbenzylidene)benzohydrazide

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Keywords: crystal structure; hydrazide; benzohydrazide; N—H···O hydrogen bonding..

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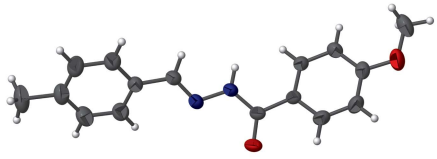
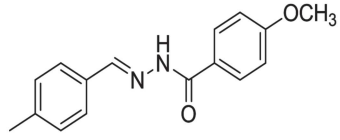
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_{16}N_2O_2$, the dihedral angle between the methoxyphenyl ring and the methylbenzylidene ring is 60.43 (5)°. In the crystal, molecules are linked *via* N—H···O hydrogen bonds, reinforced by C—H···O hydrogen bonds, forming chains propagating along the *c*-axis direction. Inversion-related chains are linked *via* C—H···π interactions, forming ribbons propagating along the *c*-axis direction.

3D view



Chemical scheme



Structure description

Hydrazones have attracted interest due to their versatile applications in various fields, such as biology (Ibrahim *et al.*, 2016), medicine (Velezheva *et al.*, 2016) and catalysis (Selvamurugan *et al.*, 2016). Hydrazone derivatives exhibit antimicrobial (Pieczonka *et al.*, 2013), anti-proliferation (Yadagiri *et al.*, 2014) and antiplatelet (Mashayekhi *et al.*, 2013) activities.

The geometric parameters of the title molecule (Fig. 1) agree well with those reported for similar structures (Maheswari *et al.*, 2016; Nair *et al.*, 2012). The dihedral angle between the methoxyphenyl ring and the methylbenzylidene ring is 60.43 (5)°.

The crystal packing is controlled by N—H···O and C—H···O hydrogen bonds (Fig. 2 and Table 1), which result in the formation of chains along [001]. Inversion-related chains are linked *via* C—H···π interactions, forming ribbons propagating along [001].

Synthesis and crystallization

A few drops of conc. HCl were added to a mixture of 4-methoxybenzohydrazide (1.7 g, 0.01 mol) and *p*-methyl benzaldehyde (1.2 ml, 0.01 mol) in ethanol (15 ml). The reaction

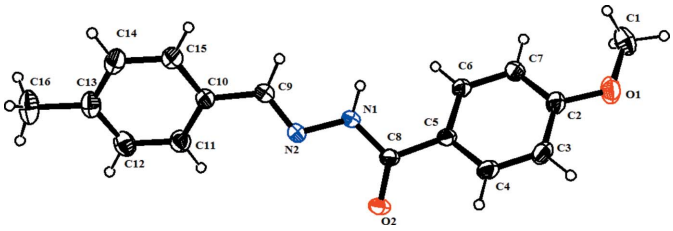


Figure 1
The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

mixture was refluxed for 3 h. The precipitate that formed was filtered and washed with petroleum ether and dried in a vacuum desiccator. The crude solid was recrystallized from DMSO giving colourless block-like crystals (yield 96%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors are grateful to the authorities of Saranathan College of Engineering, Tiruchirappalli, for providing laboratory facilities, and the CAS in Crystallography and Biophysics, University of Madras, Chennai, for the data collection.

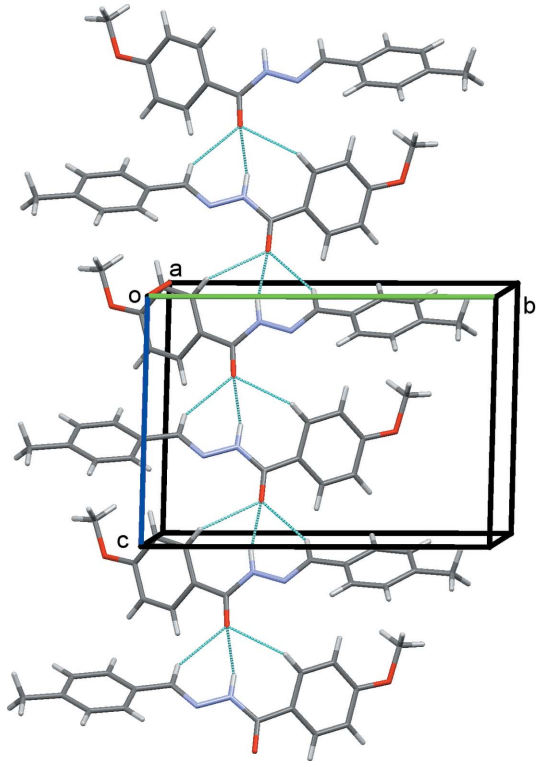


Figure 2
The crystal packing of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines (see Table 1).

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

*Cg*1 is the centroid of the C2–C7 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.09	2.9280 (12)	164
C6—H6···O2^{i}	0.93	2.58	3.3093 (14)	136
C9—H9···O2^{i}	0.93	2.50	3.3051 (15)	145
C1—H1*C*···*Cg*1^{ii}	0.96	2.77	3.6515 (5)	152

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{16}N_2O_2$
M_r	268.31
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, *b*, *c* (Å)	11.7678 (7), 13.0072 (7), 9.9025 (6)
β (°)	112.371 (2)
V (Å^3)	1401.66 (14)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.15 × 0.13 × 0.11
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 1996)
T_{min}, T_{max}	0.987, 0.991
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13430, 3517, 2752
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.669
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.042, 0.136, 1.00
No. of reflections	3517
No. of parameters	184
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.18

Computer programs: *APEX2* (Bruker, 2008), *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *PLATON* (Spek, 2009).

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full crystallographic data

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Crystal data

$C_{16}H_{16}N_2O_2$
M_r = 268.31
Monoclinic, $P2_1/c$
a = 11.7678 (7) Å
b = 13.0072 (7) Å
c = 9.9025 (6) Å
β = 112.371 (2)°
V = 1401.66 (14) Å^3
Z = 4
$F(000)$ = 568
D_x = 1.271 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3517 reflections
θ = 1.9–28.4°
μ = 0.09 mm^{-1}
T = 296 K
Block, colourless
0.15 × 0.13 × 0.11 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.987, T_{max} = 0.991
13430 measured reflections
3517 independent reflections
2752 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 28.4°, θ_{min} = 1.9°
h = −15→15
k = −17→17
l = −13→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.136
S = 1.00
3517 reflections
184 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
$w = 1/[\sigma^2(F_o^2) + (0.0794P)^2 + 0.199P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C16	0.5536 (2)	0.86088 (13)	0.1367 (3)	0.0842 (6)

H16A	0.6252	0.9031	0.1805	0.126*
H16B	0.5046	0.8633	0.1952	0.126*
H16C	0.5062	0.8858	0.0404	0.126*
O2	0.76856 (10)	0.20548 (7)	0.35951 (9)	0.0538 (3)
N2	0.70263 (10)	0.37061 (7)	0.17516 (11)	0.0427 (3)
C8	0.77468 (11)	0.19967 (8)	0.23869 (12)	0.0385 (3)
C5	0.82056 (11)	0.10506 (8)	0.19119 (11)	0.0376 (3)
N1	0.74327 (10)	0.27769 (7)	0.14161 (10)	0.0431 (3)
H1	0.7485	0.2697	0.0580	0.052*
C6	0.79284 (11)	0.07902 (9)	0.04612 (12)	0.0401 (3)
H6	0.7425	0.1221	−0.0273	0.048*
O1	0.96488 (10)	−0.16261 (7)	0.09212 (12)	0.0629 (3)
C7	0.83872 (12)	−0.00995 (9)	0.00857 (13)	0.0440 (3)
H7	0.8183	−0.0267	−0.0892	0.053*
C2	0.91488 (12)	−0.07376 (9)	0.11683 (14)	0.0451 (3)
C4	0.89715 (14)	0.03958 (10)	0.29838 (13)	0.0517 (3)
H4	0.9164	0.0553	0.3962	0.062*
C9	0.70520 (12)	0.44398 (9)	0.09164 (13)	0.0446 (3)
H9	0.7339	0.4308	0.0179	0.054*
C10	0.66494 (11)	0.54833 (9)	0.10661 (12)	0.0423 (3)
C3	0.94501 (15)	−0.04806 (10)	0.26235 (14)	0.0559 (4)
H3	0.9975	−0.0900	0.3356	0.067*
C13	0.59258 (13)	0.75127 (10)	0.12739 (16)	0.0541 (3)
C11	0.58724 (12)	0.57071 (10)	0.17829 (15)	0.0482 (3)
H11	0.5589	0.5179	0.2207	0.058*
C12	0.55146 (13)	0.67065 (11)	0.18738 (18)	0.0561 (4)
H12	0.4985	0.6840	0.2350	0.067*
C1	0.93554 (16)	−0.19275 (12)	−0.0552 (2)	0.0667 (4)
H1A	0.8487	−0.2048	−0.1015	0.100*
H1B	0.9792	−0.2547	−0.0576	0.100*
H1C	0.9589	−0.1392	−0.1062	0.100*
C15	0.70586 (16)	0.62896 (11)	0.04561 (18)	0.0595 (4)
H15	0.7576	0.6156	−0.0036	0.071*
C14	0.67073 (16)	0.72906 (11)	0.05695 (19)	0.0636 (4)
H14	0.7002	0.7822	0.0165	0.076*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C16	0.0873 (12)	0.0478 (9)	0.1117 (16)	0.0202 (8)	0.0314 (11)	−0.0069 (9)
O2	0.0900 (7)	0.0450 (5)	0.0398 (5)	−0.0012 (4)	0.0399 (5)	−0.0007 (4)
N2	0.0585 (6)	0.0357 (5)	0.0368 (5)	0.0042 (4)	0.0215 (4)	−0.0034 (4)
C8	0.0520 (6)	0.0355 (6)	0.0326 (5)	−0.0045 (5)	0.0213 (5)	−0.0017 (4)
C5	0.0510 (6)	0.0325 (5)	0.0322 (5)	−0.0032 (4)	0.0192 (5)	0.0006 (4)
N1	0.0666 (6)	0.0358 (5)	0.0329 (5)	0.0059 (4)	0.0258 (4)	−0.0008 (4)
C6	0.0505 (6)	0.0366 (6)	0.0323 (5)	0.0033 (5)	0.0145 (5)	0.0014 (4)
O1	0.0764 (7)	0.0416 (5)	0.0668 (7)	0.0149 (5)	0.0228 (5)	−0.0052 (4)
C7	0.0552 (7)	0.0401 (6)	0.0354 (6)	0.0009 (5)	0.0159 (5)	−0.0056 (4)

C2	0.0537 (7)	0.0322 (6)	0.0495 (7)	0.0008 (5)	0.0197 (5)	−0.0010 (5)
C4	0.0775 (9)	0.0442 (7)	0.0309 (6)	0.0032 (6)	0.0176 (6)	0.0032 (5)
C9	0.0617 (7)	0.0391 (6)	0.0381 (6)	0.0040 (5)	0.0245 (5)	−0.0016 (5)
C10	0.0526 (7)	0.0369 (6)	0.0370 (6)	0.0030 (5)	0.0165 (5)	−0.0019 (4)
C3	0.0772 (9)	0.0417 (7)	0.0410 (7)	0.0117 (6)	0.0137 (6)	0.0095 (5)
C13	0.0533 (7)	0.0416 (7)	0.0604 (8)	0.0082 (5)	0.0137 (6)	−0.0043 (6)
C11	0.0495 (7)	0.0439 (7)	0.0546 (7)	−0.0022 (5)	0.0235 (6)	−0.0017 (5)
C12	0.0500 (7)	0.0554 (8)	0.0678 (9)	0.0057 (6)	0.0278 (7)	−0.0081 (6)
C1	0.0732 (10)	0.0504 (8)	0.0802 (11)	0.0034 (7)	0.0333 (8)	−0.0223 (7)
C15	0.0824 (10)	0.0452 (7)	0.0675 (9)	0.0082 (7)	0.0470 (8)	0.0068 (6)
C14	0.0824 (10)	0.0398 (7)	0.0774 (10)	0.0039 (7)	0.0401 (9)	0.0076 (7)

Geometric parameters (Å, º)

C16—C13	1.5113 (19)	C4—C3	1.3772 (19)
C16—H16A	0.9600	C4—H4	0.9300
C16—H16B	0.9600	C9—C10	1.4636 (16)
C16—H16C	0.9600	C9—H9	0.9300
O2—C8	1.2279 (14)	C10—C15	1.3849 (18)
N2—C9	1.2707 (15)	C10—C11	1.3851 (18)
N2—N1	1.3860 (13)	C3—H3	0.9300
C8—N1	1.3491 (14)	C13—C14	1.380 (2)
C8—C5	1.4901 (15)	C13—C12	1.380 (2)
C5—C6	1.3886 (15)	C11—C12	1.3799 (18)
C5—C4	1.3914 (17)	C11—H11	0.9300
N1—H1	0.8600	C12—H12	0.9300
C6—C7	1.3864 (16)	C1—H1A	0.9600
C6—H6	0.9300	C1—H1B	0.9600
O1—C2	1.3606 (15)	C1—H1C	0.9600
O1—C1	1.421 (2)	C15—C14	1.384 (2)
C7—C2	1.3820 (17)	C15—H15	0.9300
C7—H7	0.9300	C14—H14	0.9300
C2—C3	1.3875 (19)		
C13—C16—H16A	109.5	N2—C9—H9	118.6
C13—C16—H16B	109.5	C10—C9—H9	118.6
H16A—C16—H16B	109.5	C15—C10—C11	118.12 (12)
C13—C16—H16C	109.5	C15—C10—C9	118.58 (12)
H16A—C16—H16C	109.5	C11—C10—C9	123.30 (11)
H16B—C16—H16C	109.5	C4—C3—C2	120.03 (12)
C9—N2—N1	113.77 (10)	C4—C3—H3	120.0
O2—C8—N1	123.11 (11)	C2—C3—H3	120.0
O2—C8—C5	121.40 (10)	C14—C13—C12	117.95 (12)
N1—C8—C5	115.48 (9)	C14—C13—C16	120.30 (15)
C6—C5—C4	117.96 (11)	C12—C13—C16	121.75 (15)
C6—C5—C8	123.87 (10)	C12—C11—C10	120.58 (13)
C4—C5—C8	118.17 (10)	C12—C11—H11	119.7
C8—N1—N2	120.74 (9)	C10—C11—H11	119.7

C8—N1—H1	119.6	C11—C12—C13	121.47 (13)
N2—N1—H1	119.6	C11—C12—H12	119.3
C7—C6—C5	121.26 (10)	C13—C12—H12	119.3
C7—C6—H6	119.4	O1—C1—H1A	109.5
C5—C6—H6	119.4	O1—C1—H1B	109.5
C2—O1—C1	117.80 (11)	H1A—C1—H1B	109.5
C2—C7—C6	119.81 (11)	O1—C1—H1C	109.5
C2—C7—H7	120.1	H1A—C1—H1C	109.5
C6—C7—H7	120.1	H1B—C1—H1C	109.5
O1—C2—C7	124.60 (12)	C14—C15—C10	120.86 (14)
O1—C2—C3	115.75 (11)	C14—C15—H15	119.6
C7—C2—C3	119.66 (11)	C10—C15—H15	119.6
C3—C4—C5	121.27 (11)	C13—C14—C15	121.01 (14)
C3—C4—H4	119.4	C13—C14—H14	119.5
C5—C4—H4	119.4	C15—C14—H14	119.5
N2—C9—C10	122.80 (11)		

O2—C8—C5—C6	157.52 (12)	N1—N2—C9—C10	179.27 (11)
N1—C8—C5—C6	−23.71 (17)	N2—C9—C10—C15	159.59 (14)
O2—C8—C5—C4	−23.46 (17)	N2—C9—C10—C11	−20.7 (2)
N1—C8—C5—C4	155.32 (12)	C5—C4—C3—C2	−1.5 (2)
O2—C8—N1—N2	0.57 (19)	O1—C2—C3—C4	−178.89 (13)
C5—C8—N1—N2	−178.18 (10)	C7—C2—C3—C4	1.3 (2)
C9—N2—N1—C8	163.64 (12)	C15—C10—C11—C12	0.4 (2)
C4—C5—C6—C7	0.69 (18)	C9—C10—C11—C12	−179.28 (12)
C8—C5—C6—C7	179.72 (11)	C10—C11—C12—C13	−0.8 (2)
C5—C6—C7—C2	−0.86 (19)	C14—C13—C12—C11	0.3 (2)
C1—O1—C2—C7	−0.9 (2)	C16—C13—C12—C11	179.89 (15)
C1—O1—C2—C3	179.30 (14)	C11—C10—C15—C14	0.5 (2)
C6—C7—C2—O1	−179.95 (12)	C9—C10—C15—C14	−179.86 (14)
C6—C7—C2—C3	−0.1 (2)	C12—C13—C14—C15	0.6 (2)
C6—C5—C4—C3	0.5 (2)	C16—C13—C14—C15	−179.04 (16)
C8—C5—C4—C3	−178.61 (13)	C10—C15—C14—C13	−1.0 (3)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C2–C7 ring.

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