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2-Methoxy-*N'*-(2-methoxybenzylidene)-benzohydrazide

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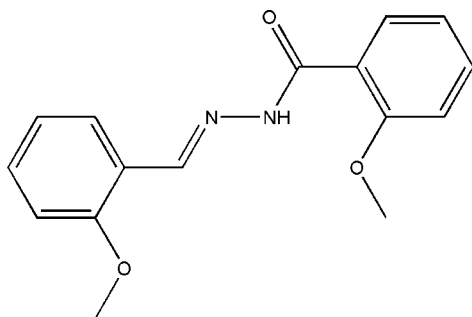
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.051; wR factor = 0.095; data-to-parameter ratio = 8.4.

The title Schiff base compound, $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_3$, was derived from the condensation of 2-methoxybenzaldehyde with 2-methoxybenzohydrazide in an ethanol solution. The dihedral angle between the two aromatic rings is 87.5 (3)°. In the crystal structure, the molecules are linked into chains running parallel to the a axis by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For related literature, see: Lu *et al.* (2008*a,b*); Nie (2008); He (2008); Shi *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_3$
 $M_r = 284.31$

 Monoclinic, $P2_1$
 $a = 4.9998$ (13) Å

 $b = 13.475$ (4) Å
 $c = 10.824$ (3) Å
 $\beta = 93.674$ (4)°
 $V = 727.7$ (4) Å³
 $Z = 2$

 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 298$ (2) K
 $0.30 \times 0.30 \times 0.28$ mm

Data collection

 Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.973$, $T_{\max} = 0.975$

 6081 measured reflections
 1647 independent reflections
 1229 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.095$
 $S = 1.11$
 1647 reflections
 195 parameters
 2 restraints

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.15$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O2}^i$	0.90 (1)	1.99 (1)	2.873 (3)	167 (4)

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); 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: CI2648).

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supplementary materials

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2-Methoxy-*N'*-(2-methoxybenzylidene)benzohydrazide

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Comment

As part of our investigation of the crystal structures of Schiff bases derived from the condensation of aldehydes with benzohydrazides (Lu *et al.*, 2008a,b), we report here the crystal structure of the title new Schiff base compound.

In the title molecule (Fig. 1), the bond lengths have normal values (Allen *et al.*, 1987), and are comparable to those observed in related compounds (Nie, 2008; He, 2008; Shi *et al.*, 2007). The dihedral angle between the two aromatic rings is 87.5 (3)°, indicating that they are almost perpendicular to one another.

In the crystal structure, the molecules are linked into chains (Fig. 2) running parallel to the *a* axis by intermolecular N–H···O hydrogen bonds (Table 1).

Experimental

The title compound was prepared by the Schiff base condensation of 2-methoxybenzaldehyde (0.1 mol) and 2-methoxybenzohydrazide (0.1 mmol) in ethanol (50 ml). The excess ethanol was removed by distillation. The colorless solid obtained was filtered and washed with ethanol. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

The imino H atom was located in a difference map and refined with a N–H distance restraint of 0.90 (1) Å. The other H atoms were positioned geometrically (C–H = 0.93–0.96 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. In the absence of significant anomalous scattering, Friedel pairs were merged.

Figures

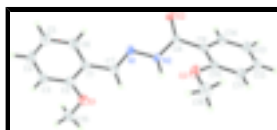


Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate hydrogen bonds.

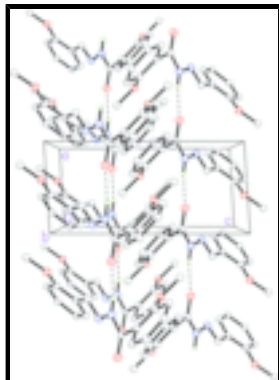


Fig. 2. The crystal packing of the title compound, viewed along the *b* axis. Dashed lines indicate hydrogen bonds.

2-Methoxy-*N'*-(2-methoxybenzylidene)benzohydrazide

Crystal data

$C_{16}H_{16}N_2O_3$

$M_r = 284.31$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 4.9998$ (13) Å

$b = 13.475$ (4) Å

$c = 10.824$ (3) Å

$\beta = 93.674$ (4)°

$V = 727.7$ (4) Å³

$Z = 2$

$F_{000} = 300$

$D_x = 1.297$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 744 reflections

$\theta = 2.5$ – 24.0 °

$\mu = 0.09$ mm⁻¹

$T = 298$ (2) K

Block, colourless

$0.30 \times 0.30 \times 0.28$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2004)

$T_{\min} = 0.973$, $T_{\max} = 0.975$

6081 measured reflections

1647 independent reflections

1229 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.055$

$\theta_{\max} = 27.0$ °

$\theta_{\min} = 1.9$ °

$h = -6 \rightarrow 6$

$k = -17 \rightarrow 16$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

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

$wR(F^2) = 0.095$	$w = 1/[\sigma^2(F_o^2) + (0.0297P)^2]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
1647 reflections	$(\Delta/\sigma)_{\max} = 0.001$
195 parameters	$\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$
2 restraints	$\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.4130 (5)	0.5063 (2)	1.0258 (2)	0.0579 (8)
O2	1.2673 (4)	0.71624 (19)	0.6930 (2)	0.0436 (6)
O3	0.6361 (5)	0.7245 (2)	0.4683 (2)	0.0513 (6)
N1	0.8931 (5)	0.5916 (2)	0.7771 (2)	0.0357 (7)
N2	0.8339 (5)	0.67811 (19)	0.7116 (3)	0.0354 (7)
C1	0.7337 (7)	0.4561 (2)	0.8904 (3)	0.0371 (9)
C2	0.5766 (7)	0.4321 (3)	0.9892 (3)	0.0426 (9)
C3	0.6008 (7)	0.3398 (3)	1.0439 (3)	0.0501 (10)
H3	0.4953	0.3233	1.1086	0.060*
C4	0.7794 (8)	0.2725 (3)	1.0032 (4)	0.0549 (11)
H4	0.7928	0.2102	1.0400	0.066*
C5	0.9407 (8)	0.2954 (3)	0.9081 (4)	0.0537 (10)
H5	1.0645	0.2498	0.8818	0.064*
C6	0.9142 (7)	0.3876 (3)	0.8529 (3)	0.0455 (9)
H6	1.0216	0.4036	0.7887	0.055*
C7	0.6996 (7)	0.5520 (2)	0.8295 (3)	0.0377 (8)
H7	0.5353	0.5844	0.8291	0.045*
C8	1.0305 (6)	0.7328 (2)	0.6674 (3)	0.0296 (7)
C9	0.9390 (6)	0.8198 (2)	0.5913 (3)	0.0343 (8)
C10	0.7391 (6)	0.8162 (3)	0.4959 (3)	0.0358 (8)
C11	0.6635 (7)	0.9012 (3)	0.4321 (3)	0.0518 (10)
H11	0.5257	0.8990	0.3703	0.062*
C12	0.7913 (8)	0.9890 (3)	0.4597 (4)	0.0606 (12)
H12	0.7384	1.0462	0.4168	0.073*

supplementary materials

C13	0.9946 (8)	0.9936 (3)	0.5493 (4)	0.0617 (12)
H13	1.0835	1.0533	0.5659	0.074*
C14	1.0677 (7)	0.9096 (3)	0.6151 (4)	0.0478 (10)
H14	1.2059	0.9131	0.6767	0.057*
C15	0.2563 (8)	0.4878 (4)	1.1267 (4)	0.0653 (12)
H15A	0.1495	0.4295	1.1105	0.098*
H15B	0.1410	0.5435	1.1385	0.098*
H15C	0.3714	0.4778	1.2001	0.098*
C16	0.4372 (8)	0.7167 (4)	0.3692 (3)	0.0712 (12)
H16A	0.2821	0.7542	0.3885	0.107*
H16B	0.3884	0.6483	0.3573	0.107*
H16C	0.5066	0.7422	0.2949	0.107*
H2	0.662 (3)	0.696 (3)	0.696 (3)	0.080*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0577 (16)	0.0581 (19)	0.0601 (19)	0.0064 (15)	0.0196 (15)	0.0132 (15)
O2	0.0265 (12)	0.0451 (14)	0.0586 (14)	0.0050 (12)	-0.0024 (10)	0.0062 (13)
O3	0.0604 (15)	0.0471 (15)	0.0439 (13)	-0.0052 (14)	-0.0149 (11)	0.0053 (14)
N1	0.0391 (16)	0.0318 (15)	0.0356 (15)	0.0028 (13)	-0.0030 (13)	0.0079 (13)
N2	0.0271 (14)	0.0357 (16)	0.0428 (16)	0.0036 (13)	-0.0018 (13)	0.0124 (13)
C1	0.037 (2)	0.038 (2)	0.0354 (19)	-0.0036 (16)	-0.0070 (16)	0.0053 (16)
C2	0.041 (2)	0.046 (2)	0.040 (2)	-0.0060 (18)	-0.0050 (18)	0.0062 (18)
C3	0.054 (2)	0.051 (3)	0.045 (2)	-0.011 (2)	-0.0020 (18)	0.0176 (19)
C4	0.069 (3)	0.035 (2)	0.058 (3)	0.000 (2)	-0.016 (2)	0.018 (2)
C5	0.061 (3)	0.043 (2)	0.056 (3)	0.0023 (19)	-0.001 (2)	0.001 (2)
C6	0.049 (2)	0.042 (2)	0.045 (2)	-0.0021 (19)	0.0002 (18)	0.0085 (18)
C7	0.0336 (18)	0.043 (2)	0.0357 (19)	0.0002 (16)	-0.0008 (16)	0.0078 (16)
C8	0.0279 (17)	0.0279 (18)	0.0332 (16)	-0.0013 (15)	0.0026 (13)	-0.0029 (15)
C9	0.0317 (17)	0.0346 (19)	0.0377 (18)	0.0046 (15)	0.0098 (14)	0.0017 (16)
C10	0.0368 (19)	0.0353 (19)	0.0357 (19)	0.0037 (16)	0.0051 (15)	0.0015 (17)
C11	0.055 (2)	0.056 (3)	0.044 (2)	0.008 (2)	0.001 (2)	0.013 (2)
C12	0.065 (3)	0.046 (3)	0.071 (3)	0.007 (2)	0.009 (2)	0.027 (2)
C13	0.072 (3)	0.039 (2)	0.076 (3)	-0.010 (2)	0.014 (3)	0.008 (2)
C14	0.048 (2)	0.037 (2)	0.058 (2)	-0.0087 (19)	0.0060 (19)	0.003 (2)
C15	0.060 (3)	0.083 (3)	0.054 (3)	-0.002 (3)	0.012 (2)	0.009 (2)
C16	0.077 (3)	0.078 (3)	0.055 (2)	-0.014 (3)	-0.023 (2)	0.009 (3)

Geometric parameters (\AA , $^\circ$)

O1—C2	1.367 (4)	C6—H6	0.93
O1—C15	1.407 (4)	C7—H7	0.93
O2—C8	1.220 (3)	C8—C9	1.488 (4)
O3—C10	1.364 (4)	C9—C14	1.387 (5)
O3—C16	1.419 (4)	C9—C10	1.391 (4)
N1—C7	1.270 (4)	C10—C11	1.378 (5)
N1—N2	1.387 (3)	C11—C12	1.368 (5)
N2—C8	1.341 (4)	C11—H11	0.93

N2—H2	0.901 (10)	C12—C13	1.360 (5)
C1—C6	1.370 (5)	C12—H12	0.93
C1—C2	1.405 (5)	C13—C14	1.375 (5)
C1—C7	1.456 (4)	C13—H13	0.93
C2—C3	1.380 (5)	C14—H14	0.93
C3—C4	1.365 (5)	C15—H15A	0.96
C3—H3	0.93	C15—H15B	0.96
C4—C5	1.383 (5)	C15—H15C	0.96
C4—H4	0.93	C16—H16A	0.96
C5—C6	1.381 (5)	C16—H16B	0.96
C5—H5	0.93	C16—H16C	0.96
C2—O1—C15	118.0 (3)	C14—C9—C10	118.1 (3)
C10—O3—C16	118.0 (3)	C14—C9—C8	117.5 (3)
C7—N1—N2	115.9 (3)	C10—C9—C8	124.3 (3)
C8—N2—N1	120.5 (2)	O3—C10—C11	123.8 (3)
C8—N2—H2	120 (3)	O3—C10—C9	115.9 (3)
N1—N2—H2	120 (3)	C11—C10—C9	120.3 (3)
C6—C1—C2	118.8 (3)	C12—C11—C10	120.0 (4)
C6—C1—C7	121.6 (3)	C12—C11—H11	120.0
C2—C1—C7	119.6 (3)	C10—C11—H11	120.0
O1—C2—C3	125.0 (3)	C13—C12—C11	120.8 (4)
O1—C2—C1	115.3 (3)	C13—C12—H12	119.6
C3—C2—C1	119.7 (4)	C11—C12—H12	119.6
C4—C3—C2	120.1 (4)	C12—C13—C14	119.6 (4)
C4—C3—H3	120.0	C12—C13—H13	120.2
C2—C3—H3	119.9	C14—C13—H13	120.2
C3—C4—C5	121.1 (3)	C13—C14—C9	121.2 (4)
C3—C4—H4	119.5	C13—C14—H14	119.4
C5—C4—H4	119.5	C9—C14—H14	119.4
C6—C5—C4	118.6 (4)	O1—C15—H15A	109.5
C6—C5—H5	120.7	O1—C15—H15B	109.5
C4—C5—H5	120.7	H15A—C15—H15B	109.5
C1—C6—C5	121.6 (4)	O1—C15—H15C	109.5
C1—C6—H6	119.2	H15A—C15—H15C	109.5
C5—C6—H6	119.2	H15B—C15—H15C	109.5
N1—C7—C1	120.3 (3)	O3—C16—H16A	109.5
N1—C7—H7	119.9	O3—C16—H16B	109.5
C1—C7—H7	119.9	H16A—C16—H16B	109.5
O2—C8—N2	122.8 (3)	O3—C16—H16C	109.5
O2—C8—C9	122.0 (3)	H16A—C16—H16C	109.5
N2—C8—C9	115.1 (3)	H16B—C16—H16C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 \cdots O2 ⁱ	0.90 (1)	1.99 (1)	2.873 (3)	167 (4)

Symmetry codes: (i) $x-1, y, z$.

Fig. 1

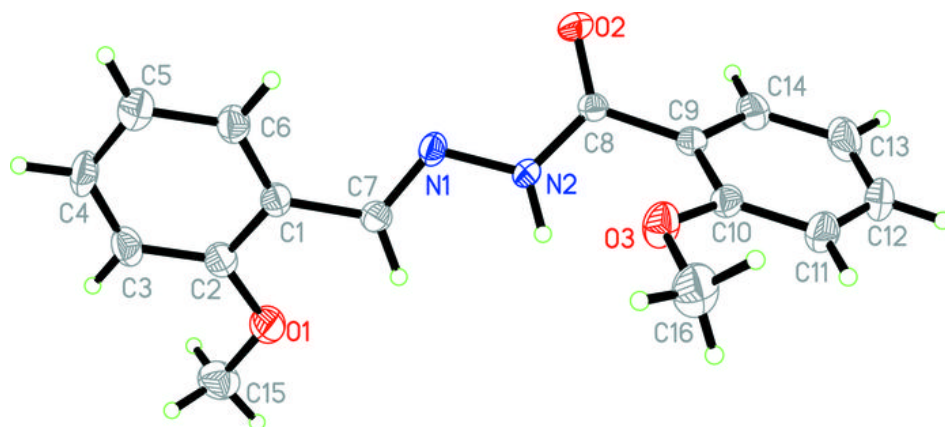


Fig. 2

