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## Structure Reports

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## N'-(4-Methoxybenzylidene)-4-nitrobenzohydrazide methanol solvate

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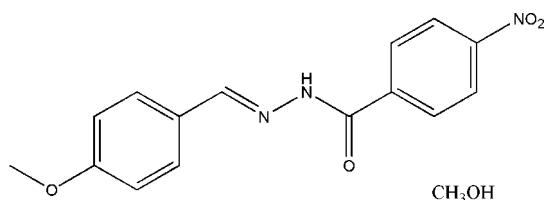
Received 24 February 2008; accepted 1 March 2008

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.058;  $wR$  factor = 0.172; data-to-parameter ratio = 15.0.

The title compound,  $\text{C}_{15}\text{H}_{13}\text{N}_3\text{O}_4 \cdot \text{CH}_4\text{O}$ , was synthesized from the reaction of 4-methoxybenzaldehyde with 4-nitrobenzohydrazide in methanol. The benzene rings of the Schiff base molecule are nearly coplanar, making a dihedral angle of  $7.0(3)^\circ$ . The methanol solvent molecules are linked to the Schiff base molecules by  $\text{N}-\text{H} \cdots \text{O}$ ,  $\text{O}-\text{H} \cdots \text{N}$  and  $\text{O}-\text{H} \cdots \text{O}$  hydrogen bonds, forming chains running parallel to the  $b$  axis.

### Related literature

For related structures, see: Brückner *et al.* (2000); Diao (2007); Diao *et al.* (2007, 2008); Harrop *et al.* (2003); Huang *et al.* (2007); Li *et al.* (2007); Ren *et al.* (2002).



### Experimental

#### Crystal data

$\text{C}_{15}\text{H}_{13}\text{N}_3\text{O}_4 \cdot \text{CH}_4\text{O}$   
 $M_r = 331.33$   
 Monoclinic,  $P2_1/n$   
 $a = 14.719(3)$  Å

$b = 6.631(2)$  Å  
 $c = 18.002(3)$  Å  
 $\beta = 113.17(3)^\circ$   
 $V = 1615.3(7)$  Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>

$T = 298(2)$  K  
 $0.27 \times 0.23 \times 0.23$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2000)  
 $T_{\min} = 0.973$ ,  $T_{\max} = 0.977$

9171 measured reflections  
 3351 independent reflections  
 1493 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.065$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$   
 $wR(F^2) = 0.172$   
 $S = 0.95$   
 3351 reflections  
 224 parameters  
 1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.17$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.17$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N2}-\text{H2A} \cdots \text{O5}$	0.897 (10)	2.049 (13)	2.921 (3)	164 (3)
$\text{O5}-\text{H5} \cdots \text{N3}^{\ddagger}$	0.82	2.56	3.167 (3)	133
$\text{O5}-\text{H5} \cdots \text{O3}^{\ddagger}$	0.82	2.10	2.863 (3)	154

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); 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: RZ2199).

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## supporting information

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## *N'*-(4-Methoxybenzylidene)-4-nitrobenzohydrazide methanol solvate

Yuan-Zhi Wang, Ming-Dong Wang, Yun-Peng Diao and Qian Cai

### S1. Comment

Schiff base compounds have been found to have potential pharmacological and antitumor properties (Brückner *et al.*, 2000; Harrop *et al.*, 2003; Ren *et al.*, 2002). Recently, a few Schiff base compounds derived from the reaction of aldehydes with benzohydrazides have been reported (Diao *et al.*, 2008; Diao *et al.*, 2007; Diao, 2007; Li *et al.*, 2007; Huang *et al.*, 2007). As a further study of such compounds, we report here the structure of the title compound.

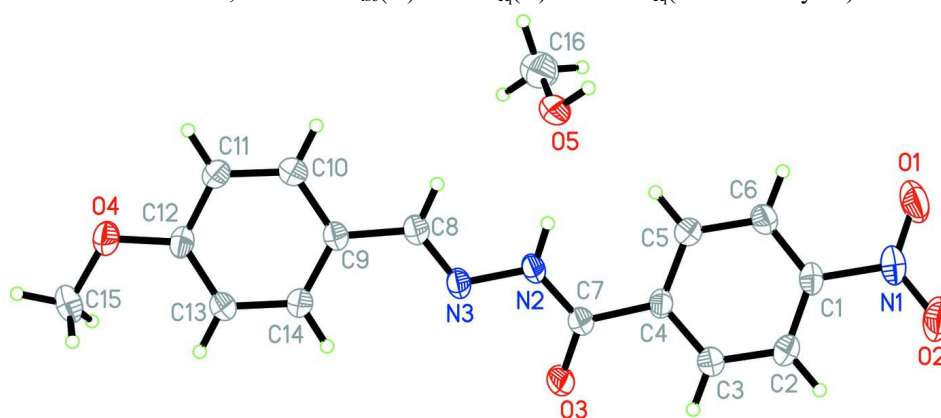
The title compound (Fig. 1) consists of a Schiff base molecule and a lattice methanol molecule. The Schiff base molecule is nearly planar with the dihedral angle between the two phenyl rings of 7.0 (3)°. The dihedral angle between the C1—C6 phenyl ring and the O1/N1/O2 nitryl plane is 5.1 (3)°. The torsion angles C9—C8—N3—N2 and C4—C7—N2—N3 are 1.4 (3) and 1.6 (3)°, respectively. The methanol molecules are linked to the Schiff base molecules by N—H···O, O—H···N and O—H···O hydrogen bonds (Table 1) forming chains running parallel to the *b* axis.

### S2. Experimental

4-Methoxybenzaldehyde (0.1 mmol, 13.6 mg) and 4-nitrobenzohydrazide (0.1 mmol, 18.1 mg) were dissolved in methanol (20 ml). The mixture was stirred at reflux for 1 h and cooled to room temperature. After keeping the solution in air for five days, yellow block-like crystals were formed on slow evaporation of the solvent.

### S3. Refinement

H2A was located from a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. Other H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H distances of 0.93–0.96 Å, O—H distance of 0.82 Å, and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  and  $1.5U_{\text{eq}}(\text{O and methyl C})$ .



**Figure 1**

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

***N'*-(4-Methoxybenzylidene)-4-nitrobenzohydrazide methanol solvate***Crystal data*C<sub>15</sub>H<sub>13</sub>N<sub>3</sub>O<sub>4</sub>·CH<sub>4</sub>O*M<sub>r</sub>* = 331.33Monoclinic, *P*2<sub>1</sub>/*n*Hall symbol: -*P* 2<sub>1</sub> *yn**a* = 14.719 (3) Å*b* = 6.631 (2) Å*c* = 18.002 (3) Å $\beta$  = 113.17 (3)°*V* = 1615.3 (7) Å<sup>3</sup>*Z* = 4*F*(000) = 696*D<sub>x</sub>* = 1.362 Mg m<sup>-3</sup>Mo *K*α radiation,  $\lambda$  = 0.71073 Å

Cell parameters from 670 reflections

 $\theta$  = 2.4–24.5° $\mu$  = 0.10 mm<sup>-1</sup>*T* = 298 K

Block, yellow

0.27 × 0.23 × 0.23 mm

*Data collection*Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega$  scansAbsorption correction: multi-scan  
(*SADABS*; Bruker, 2000)*T<sub>min</sub>* = 0.973, *T<sub>max</sub>* = 0.977

9171 measured reflections

3351 independent reflections

1493 reflections with *I* > 2σ(*I*)*R<sub>int</sub>* = 0.065 $\theta_{\max}$  = 26.5°,  $\theta_{\min}$  = 1.5°*h* = -18→18*k* = -8→7*l* = -17→22*Refinement*Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.057*wR*(*F*<sup>2</sup>) = 0.172*S* = 0.95

3351 reflections

224 parameters

1 restraint

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sitesH atoms treated by a mixture of independent  
and constrained refinement*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.07*P*)<sup>2</sup>]where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3(Δ/σ)<sub>max</sub> < 0.001Δρ<sub>max</sub> = 0.17 e Å<sup>-3</sup>Δρ<sub>min</sub> = -0.17 e Å<sup>-3</sup>Extinction correction: *SHELXTL* (Sheldrick,  
2008), *F<sub>c</sub>*\* = *kF<sub>c</sub>*[1 + 0.001*xF<sub>c</sub>*<sup>2</sup>λ<sup>3</sup>/sin(2θ)]<sup>-1/4</sup>

Extinction coefficient: 0.0051 (12)

*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*<sup>2</sup> against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on *F*<sup>2</sup>, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative *F*<sup>2</sup>. The threshold expression of *F*<sup>2</sup> > σ(*F*<sup>2</sup>) 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*<sup>2</sup> 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<sup>2</sup>)*

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>iso</sub></i> */ <i>U<sub>eq</sub></i>
N1	0.3893 (2)	0.1141 (5)	0.43054 (18)	0.0628 (8)
N2	0.37993 (17)	0.4842 (3)	0.09474 (14)	0.0480 (6)
N3	0.38196 (16)	0.5979 (3)	0.03071 (14)	0.0481 (6)

O1	0.35466 (19)	-0.0546 (4)	0.41861 (15)	0.0913 (9)
O2	0.4269 (2)	0.1901 (4)	0.49761 (15)	0.0915 (9)
O3	0.38954 (18)	0.7649 (3)	0.16870 (13)	0.0712 (7)
O4	0.37929 (16)	0.8238 (3)	-0.31725 (12)	0.0643 (6)
O5	0.34226 (17)	0.0632 (3)	0.04415 (13)	0.0643 (6)
H5	0.3646	-0.0390	0.0708	0.096*
C1	0.3883 (2)	0.2315 (4)	0.36036 (17)	0.0474 (8)
C2	0.4211 (2)	0.4269 (5)	0.37219 (18)	0.0587 (8)
H2	0.4442	0.4847	0.4234	0.070*
C3	0.4188 (2)	0.5348 (5)	0.30657 (18)	0.0570 (8)
H3	0.4402	0.6680	0.3136	0.068*
C4	0.38535 (19)	0.4503 (4)	0.23005 (16)	0.0433 (7)
C5	0.3527 (2)	0.2511 (4)	0.22030 (18)	0.0500 (8)
H5A	0.3303	0.1916	0.1695	0.060*
C6	0.3537 (2)	0.1412 (4)	0.28616 (18)	0.0520 (8)
H6	0.3311	0.0088	0.2799	0.062*
C7	0.3851 (2)	0.5799 (4)	0.16214 (17)	0.0484 (7)
C8	0.3756 (2)	0.4971 (4)	-0.03121 (18)	0.0492 (7)
H8	0.3686	0.3578	-0.0305	0.059*
C9	0.37877 (19)	0.5910 (4)	-0.10321 (16)	0.0432 (7)
C10	0.3555 (2)	0.4751 (5)	-0.17273 (17)	0.0525 (8)
H10	0.3393	0.3398	-0.1717	0.063*
C11	0.3559 (2)	0.5571 (4)	-0.24324 (18)	0.0535 (8)
H11	0.3389	0.4783	-0.2895	0.064*
C12	0.3818 (2)	0.7574 (4)	-0.24426 (17)	0.0475 (7)
C13	0.4075 (2)	0.8738 (4)	-0.17570 (17)	0.0497 (8)
H13	0.4263	1.0075	-0.1762	0.060*
C14	0.4054 (2)	0.7901 (4)	-0.10576 (17)	0.0495 (8)
H14	0.4221	0.8695	-0.0597	0.059*
C15	0.3969 (2)	1.0303 (5)	-0.3251 (2)	0.0705 (10)
H15A	0.3480	1.1096	-0.3156	0.106*
H15B	0.3933	1.0560	-0.3787	0.106*
H15C	0.4614	1.0657	-0.2864	0.106*
C16	0.2426 (3)	0.0315 (5)	-0.0070 (2)	0.0854 (11)
H16A	0.2053	0.0016	0.0250	0.128*
H16B	0.2380	-0.0796	-0.0425	0.128*
H16C	0.2166	0.1508	-0.0383	0.128*
H2A	0.377 (2)	0.3496 (16)	0.0892 (19)	0.080*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0707 (18)	0.072 (2)	0.0519 (19)	0.0074 (16)	0.0308 (16)	0.0221 (16)
N2	0.0678 (16)	0.0380 (13)	0.0453 (15)	-0.0013 (12)	0.0298 (13)	0.0086 (12)
N3	0.0634 (15)	0.0436 (14)	0.0426 (15)	0.0012 (12)	0.0266 (13)	0.0089 (12)
O1	0.1079 (19)	0.0897 (19)	0.0742 (18)	-0.0230 (16)	0.0335 (15)	0.0321 (15)
O2	0.137 (2)	0.096 (2)	0.0475 (16)	0.0115 (16)	0.0432 (16)	0.0156 (15)
O3	0.128 (2)	0.0367 (12)	0.0565 (14)	-0.0021 (12)	0.0437 (14)	0.0032 (10)

O4	0.0934 (16)	0.0610 (14)	0.0450 (13)	-0.0019 (12)	0.0342 (12)	0.0065 (11)
O5	0.0883 (16)	0.0432 (13)	0.0554 (15)	-0.0041 (12)	0.0218 (13)	0.0050 (10)
C1	0.0540 (18)	0.0532 (19)	0.0393 (18)	0.0052 (15)	0.0230 (15)	0.0101 (15)
C2	0.081 (2)	0.060 (2)	0.0378 (18)	-0.0057 (18)	0.0263 (16)	-0.0037 (15)
C3	0.080 (2)	0.0474 (18)	0.0464 (19)	-0.0068 (16)	0.0277 (17)	0.0011 (15)
C4	0.0526 (17)	0.0442 (17)	0.0360 (17)	0.0037 (14)	0.0208 (14)	0.0049 (13)
C5	0.0615 (19)	0.0465 (18)	0.0440 (18)	-0.0040 (15)	0.0229 (16)	-0.0025 (14)
C6	0.0664 (19)	0.0466 (18)	0.050 (2)	-0.0042 (15)	0.0306 (16)	0.0039 (15)
C7	0.0634 (19)	0.0404 (18)	0.0434 (18)	-0.0016 (15)	0.0231 (16)	0.0035 (15)
C8	0.0605 (19)	0.0452 (17)	0.0458 (18)	-0.0016 (15)	0.0253 (16)	0.0052 (15)
C9	0.0489 (16)	0.0420 (16)	0.0404 (17)	-0.0004 (14)	0.0194 (14)	0.0058 (13)
C10	0.0657 (19)	0.0420 (17)	0.052 (2)	-0.0042 (14)	0.0247 (16)	-0.0003 (15)
C11	0.072 (2)	0.0487 (19)	0.0405 (18)	-0.0036 (16)	0.0231 (16)	-0.0042 (15)
C12	0.0551 (18)	0.0543 (19)	0.0362 (17)	0.0042 (15)	0.0214 (15)	0.0052 (15)
C13	0.0638 (19)	0.0410 (17)	0.0476 (18)	-0.0050 (14)	0.0254 (16)	0.0036 (14)
C14	0.0620 (19)	0.0472 (19)	0.0417 (18)	-0.0036 (15)	0.0231 (15)	0.0004 (14)
C15	0.087 (2)	0.066 (2)	0.067 (2)	-0.0062 (19)	0.040 (2)	0.0200 (18)
C16	0.092 (3)	0.075 (3)	0.087 (3)	-0.002 (2)	0.032 (2)	0.000 (2)

*Geometric parameters (Å, °)*

N1—O1	1.213 (3)	C5—H5A	0.9300
N1—O2	1.222 (3)	C6—H6	0.9300
N1—C1	1.479 (4)	C8—C9	1.455 (4)
N2—C7	1.345 (3)	C8—H8	0.9300
N2—N3	1.388 (3)	C9—C14	1.383 (4)
N2—H2A	0.897 (10)	C9—C10	1.392 (4)
N3—C8	1.271 (3)	C10—C11	1.383 (4)
O3—C7	1.232 (3)	C10—H10	0.9300
O4—C12	1.372 (3)	C11—C12	1.384 (4)
O4—C15	1.411 (3)	C11—H11	0.9300
O5—C16	1.407 (4)	C12—C13	1.376 (4)
O5—H5	0.8200	C13—C14	1.388 (4)
C1—C6	1.366 (4)	C13—H13	0.9300
C1—C2	1.370 (4)	C14—H14	0.9300
C2—C3	1.370 (4)	C15—H15A	0.9600
C2—H2	0.9300	C15—H15B	0.9600
C3—C4	1.386 (4)	C15—H15C	0.9600
C3—H3	0.9300	C16—H16A	0.9600
C4—C5	1.392 (4)	C16—H16B	0.9600
C4—C7	1.493 (4)	C16—H16C	0.9600
C5—C6	1.387 (4)		
O1—N1—O2	123.4 (3)	C9—C8—H8	118.7
O1—N1—C1	118.2 (3)	C14—C9—C10	118.0 (3)
O2—N1—C1	118.4 (3)	C14—C9—C8	123.2 (3)
C7—N2—N3	118.7 (2)	C10—C9—C8	118.8 (3)
C7—N2—H2A	123 (2)	C11—C10—C9	121.3 (3)

N3—N2—H2A	118 (2)	C11—C10—H10	119.3
C8—N3—N2	115.1 (2)	C9—C10—H10	119.3
C12—O4—C15	118.0 (2)	C10—C11—C12	119.3 (3)
C16—O5—H5	109.5	C10—C11—H11	120.3
C6—C1—C2	122.6 (3)	C12—C11—H11	120.3
C6—C1—N1	118.6 (3)	O4—C12—C13	125.0 (3)
C2—C1—N1	118.7 (3)	O4—C12—C11	114.6 (3)
C1—C2—C3	118.2 (3)	C13—C12—C11	120.4 (3)
C1—C2—H2	120.9	C12—C13—C14	119.5 (3)
C3—C2—H2	120.9	C12—C13—H13	120.2
C2—C3—C4	121.6 (3)	C14—C13—H13	120.2
C2—C3—H3	119.2	C9—C14—C13	121.3 (3)
C4—C3—H3	119.2	C9—C14—H14	119.3
C3—C4—C5	118.6 (3)	C13—C14—H14	119.3
C3—C4—C7	117.8 (3)	O4—C15—H15A	109.5
C5—C4—C7	123.6 (3)	O4—C15—H15B	109.5
C6—C5—C4	120.2 (3)	H15A—C15—H15B	109.5
C6—C5—H5A	119.9	O4—C15—H15C	109.5
C4—C5—H5A	119.9	H15A—C15—H15C	109.5
C1—C6—C5	118.7 (3)	H15B—C15—H15C	109.5
C1—C6—H6	120.7	O5—C16—H16A	109.5
C5—C6—H6	120.7	O5—C16—H16B	109.5
O3—C7—N2	122.6 (3)	H16A—C16—H16B	109.5
O3—C7—C4	120.8 (3)	O5—C16—H16C	109.5
N2—C7—C4	116.6 (2)	H16A—C16—H16C	109.5
N3—C8—C9	122.6 (3)	H16B—C16—H16C	109.5
N3—C8—H8	118.7		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N2—H2A $\cdots$ O5	0.90 (1)	2.05 (1)	2.921 (3)	164 (3)
O5—H5 $\cdots$ N3 <sup>i</sup>	0.82	2.56	3.167 (3)	133
O5—H5 $\cdots$ O3 <sup>i</sup>	0.82	2.10	2.863 (3)	154

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