Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(3,4-Dimethoxybenzylidene)-acetohydrazide

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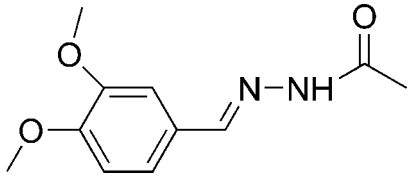
Received 11 July 2009; accepted 12 July 2009

Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.133; data-to-parameter ratio = 13.9.

In the title molecule, $C_{11}H_{14}N_2O_3$, the acetohydrazide group is planar 0.084 (1) Å and forms a dihedral angle of 19.7 (1)° with the benzene ring. One of the methoxy groups is coplanar with the attached benzene ring within 0.052 (3) Å, whereas the other is slightly twisted [C—O—C—C = 6.3 (3)°]. The molecule adopts a *trans* configuration with respect to the C=N bond. In the crystal, the molecules are linked into chains along the *a* axis by N—H···O hydrogen bonds and the chains are cross-linked into a three-dimensional network by C—H···O hydrogen bonds.

Related literature

For general background to Schiff bases, see: Cimerman *et al.* (1997); Offe *et al.* (1952); Richardson *et al.* (1988). For related structures, see: Li & Jian (2008); Tamboura *et al.* (2009).



Experimental

Crystal data

$C_{11}H_{14}N_2O_3$ M_r = 222.24
Orthorhombic, *Pbca*
a = 8.794 (3) Å
b = 10.920 (3) Å
c = 24.418 (7) Å
V = 2345.0 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 223 K
0.24 × 0.21 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.977, T_{max} = 0.979
11332 measured reflections
2070 independent reflections
1819 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.133
S = 1.10
2070 reflections
149 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O3^{i}	0.86	1.97	2.803 (2)	164
C1—H1*C*···O2^{ii}	0.96	2.55	3.435 (3)	153
C11—H11*C*···O3^{iii}	0.96	2.47	3.425 (3)	174

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{5}{2}, -z$; (ii) $x+\frac{1}{2}, y, -z+\frac{1}{2}$; (iii) $-x, -y+3, -z$.

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*.

The authors thank the Science and Technology Project of Zhejiang Province (grant No. 2007 F70077) and Hangzhou Vocational and Technical College for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2855).

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supporting information

Acta Cryst. (2009). E**65**, o1989 [doi:10.1107/S1600536809027366]

N′-(3,4-Dimethoxybenzylidene)acetohydrazide

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S1. Comment

Schiff bases have attracted much attention due to the possibility of their analytical applications (Cimerman *et al.*, 1997). They are also important ligands, which have been reported to have mild bacteriostatic activity and are used as potential oral iron-chelating drugs for genetic disorders such as thalassemia (Offe *et al.*, 1952; Richardson *et al.*, 1988). Metal complexes based on Schiff bases have received considerable attention because they can be utilized as model compounds of active centres in various complexes (Tamboura *et al.*, 2009). We report here the crystal structure of the title compound (Fig. 1).

The acetohydrazide group is planar and it forms a dihedral angle of 19.7 (1)° with the benzene ring. One of the methoxy groups is coplanar with the attached benzene ring [C1—O1—C4—C5 = -1.7 (3)°] whereas the other is slightly twisted [C2—O2—C3—C8 = 6.3 (3)°]. The molecule adopts a *trans* configuration with respect to the C═N bond. Bond lengths and angles are comparable to those observed for *N*′-[1-(4-methoxyphenyl)ethylidene]acetohydrazide (Li *et al.*, 2008).

The molecules are linked into a chain along the *a* axis by N—H···O hydrogen bonds (Table 1). The chains are cross-linked into a three-dimensional network by C—H···O hydrogen bonds (Fig.2).

S2. Experimental

3,4-Dimethoxybenzaldehyde (1.66 g, 0.01 mol) and acetohydrazide (0.74 g, 0.01 mol) were dissolved in stirred methanol (25 ml) and left for 2.5 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 90% yield. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature (m.p. 470–472 K).

S3. Refinement

H atoms were positioned geometrically (N-H = 0.86 Å and C-H = 0.93 or 0.96Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,N) and 1.5U_{eq}(C_{methyl}). A rotating group model was used for the methyl groups.

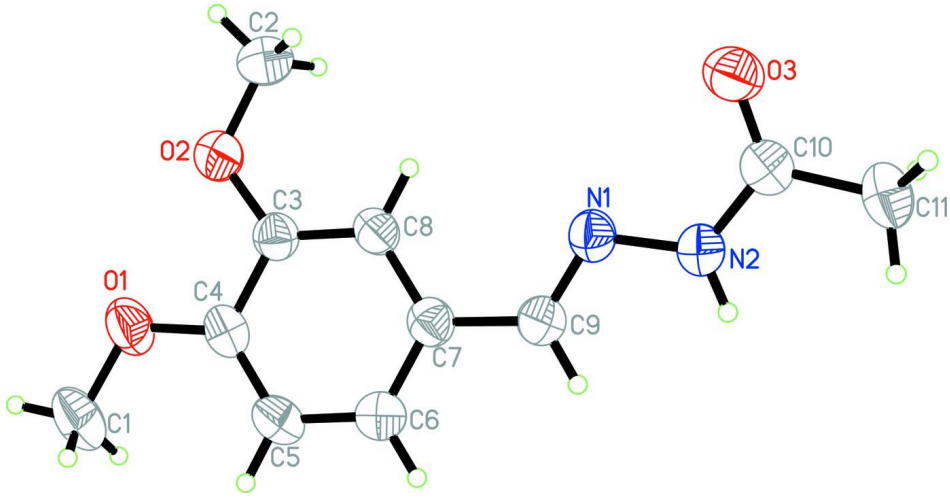

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 40% probability level.

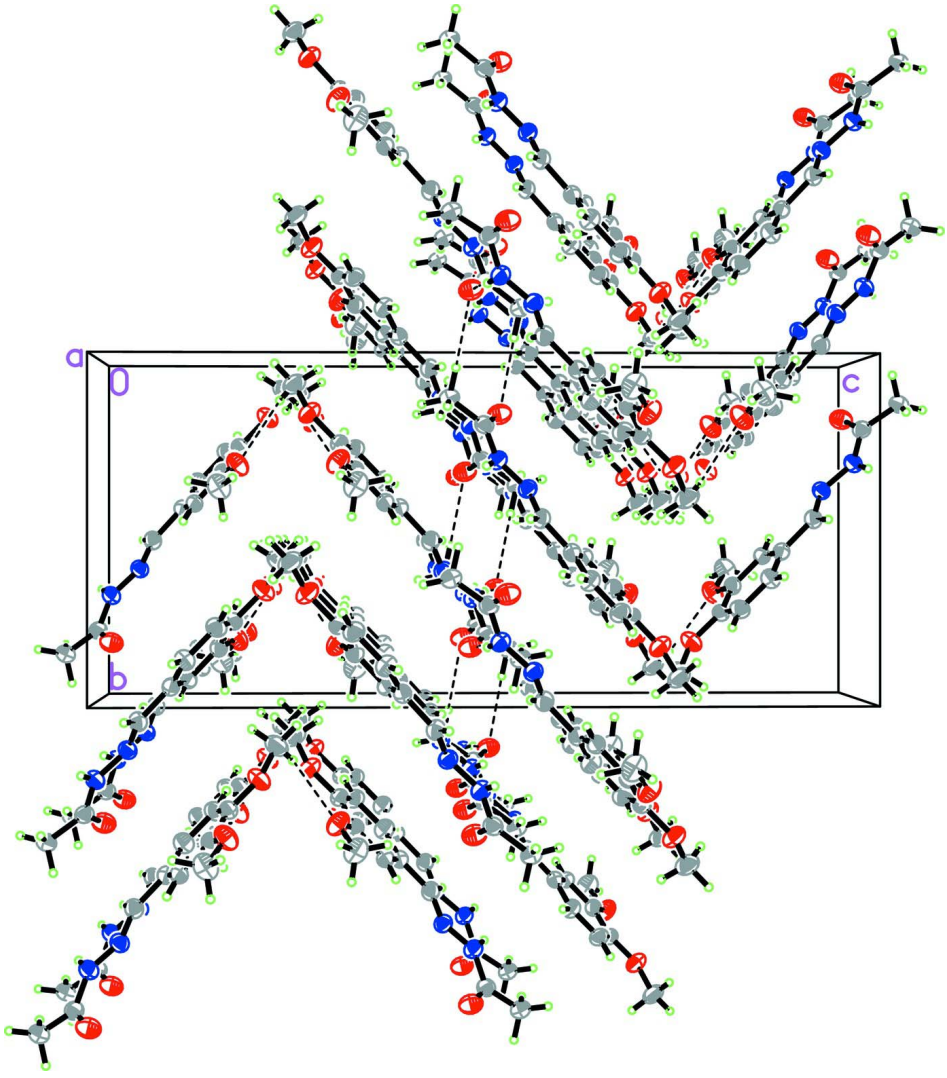

Figure 2

Part of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines.

N′-(3,4-Dimethoxybenzylidene)acetohydrazide

Crystal data

$C_{11}H_{14}N_2O_3$
$M_r = 222.24$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 8.794\ (3)$ Å
$b = 10.920\ (3)$ Å
$c = 24.418\ (7)$ Å
$V = 2345.0\ (12)$ Å^3
$Z = 8$
$F(000) = 944$
$D_x = 1.259$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2070 reflections
$\theta = 1.7–25.0°$
$\mu = 0.09$ mm^{-1}
$T = 223$ K
Block, colourless
0.24 × 0.21 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.977, T_{max} = 0.979
11332 measured reflections
2070 independent reflections
1819 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.0°, θ_{min} = 1.7°
h = −10→10
k = −12→12
l = −27→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.133
S = 1.10
2070 reflections
149 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
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 0.6946P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0113 (16)

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 $F^2 > 2\text{sigma}(F^2)$ 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	−0.00337 (17)	0.67057 (13)	0.22113 (6)	0.0725 (5)
O2	−0.21387 (16)	0.81158 (15)	0.18477 (6)	0.0738 (5)
O3	−0.07522 (17)	1.32783 (14)	0.01901 (7)	0.0735 (5)
C3	−0.0722 (2)	0.83356 (17)	0.16379 (7)	0.0516 (5)
C7	0.1114 (2)	0.93669 (17)	0.10716 (7)	0.0519 (5)
C4	0.0432 (2)	0.75598 (17)	0.18390 (8)	0.0537 (5)
C6	0.2227 (2)	0.8587 (2)	0.12649 (8)	0.0621 (5)
H6	0.3216	0.8663	0.1135	0.074*
C8	−0.0381 (2)	0.92289 (17)	0.12603 (7)	0.0507 (5)
H8	−0.1143	0.9742	0.1130	0.061*
C10	0.0460 (2)	1.29542 (18)	−0.00180 (8)	0.0556 (5)
C5	0.1896 (2)	0.76932 (19)	0.16500 (9)	0.0625 (6)
H5	0.2663	0.7184	0.1780	0.075*
C9	0.1527 (2)	1.03131 (19)	0.06757 (8)	0.0564 (5)
H9	0.2501	1.0303	0.0528	0.068*

C11	0.1217 (3)	1.3670 (2)	−0.04662 (9)	0.0702 (6)
H11A	0.0706	1.3517	−0.0807	0.105*
H11B	0.2261	1.3423	−0.0496	0.105*
H11C	0.1169	1.4528	−0.0382	0.105*
C2	−0.3390 (2)	0.8771 (3)	0.16274 (10)	0.0825 (7)
H2A	−0.3234	0.9633	0.1680	0.124*
H2B	−0.4307	0.8524	0.1810	0.124*
H2C	−0.3476	0.8600	0.1243	0.124*
C1	0.1076 (3)	0.5863 (2)	0.24102 (11)	0.0827 (8)
H1A	0.1518	0.5429	0.2108	0.124*
H1B	0.0601	0.5291	0.2655	0.124*
H1C	0.1857	0.6303	0.2602	0.124*
N2	0.12042 (19)	1.19408 (15)	0.01359 (6)	0.0569 (4)
H2	0.2071	1.1778	−0.0010	0.068*
N1	0.06138 (18)	1.11504 (14)	0.05240 (6)	0.0544 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0779 (10)	0.0661 (9)	0.0736 (10)	−0.0030 (7)	−0.0097 (8)	0.0243 (8)
O2	0.0572 (9)	0.0863 (11)	0.0779 (10)	0.0039 (8)	0.0052 (7)	0.0262 (8)
O3	0.0634 (9)	0.0642 (10)	0.0929 (11)	0.0057 (7)	−0.0005 (8)	0.0069 (8)
C3	0.0533 (11)	0.0536 (11)	0.0479 (10)	−0.0003 (9)	−0.0020 (8)	−0.0001 (8)
C7	0.0581 (11)	0.0515 (11)	0.0462 (10)	0.0034 (9)	−0.0006 (8)	−0.0017 (8)
C4	0.0650 (12)	0.0477 (10)	0.0486 (10)	−0.0021 (9)	−0.0085 (9)	0.0030 (8)
C6	0.0545 (11)	0.0661 (13)	0.0656 (12)	0.0041 (10)	0.0016 (10)	0.0063 (10)
C8	0.0560 (11)	0.0488 (10)	0.0473 (10)	0.0068 (8)	−0.0032 (8)	0.0010 (8)
C10	0.0587 (12)	0.0505 (11)	0.0576 (11)	−0.0049 (9)	−0.0092 (9)	−0.0037 (9)
C5	0.0624 (12)	0.0574 (12)	0.0679 (13)	0.0097 (10)	−0.0096 (10)	0.0071 (10)
C9	0.0559 (11)	0.0605 (12)	0.0529 (10)	0.0042 (10)	0.0022 (9)	0.0027 (9)
C11	0.0918 (16)	0.0530 (12)	0.0659 (13)	−0.0018 (11)	−0.0030 (12)	0.0071 (10)
C2	0.0548 (12)	0.1042 (19)	0.0886 (17)	0.0095 (13)	0.0055 (12)	0.0167 (15)
C1	0.0994 (18)	0.0651 (14)	0.0836 (16)	0.0000 (13)	−0.0244 (14)	0.0224 (13)
N2	0.0577 (9)	0.0551 (10)	0.0578 (9)	0.0008 (8)	0.0040 (8)	0.0078 (8)
N1	0.0572 (9)	0.0525 (9)	0.0536 (9)	−0.0042 (8)	0.0005 (7)	0.0052 (7)

Geometric parameters (Å, º)

O1—C4	1.365 (2)	C10—C11	1.501 (3)
O1—C1	1.427 (3)	C5—H5	0.93
O2—C3	1.368 (2)	C9—N1	1.272 (2)
O2—C2	1.419 (3)	C9—H9	0.93
O3—C10	1.233 (2)	C11—H11A	0.96
C3—C8	1.375 (3)	C11—H11B	0.96
C3—C4	1.410 (3)	C11—H11C	0.96
C7—C6	1.381 (3)	C2—H2A	0.96
C7—C8	1.401 (3)	C2—H2B	0.96
C7—C9	1.461 (3)	C2—H2C	0.96

C4—C5	1.375 (3)	C1—H1A	0.96
C6—C5	1.386 (3)	C1—H1B	0.96
C6—H6	0.93	C1—H1C	0.96
C8—H8	0.93	N2—N1	1.383 (2)
C10—N2	1.340 (2)	N2—H2	0.86
C4—O1—C1	117.52 (18)	N1—C9—H9	118.5
C3—O2—C2	118.41 (16)	C7—C9—H9	118.5
O2—C3—C8	125.05 (17)	C10—C11—H11A	109.5
O2—C3—C4	114.81 (17)	C10—C11—H11B	109.5
C8—C3—C4	120.14 (18)	H11A—C11—H11B	109.5
C6—C7—C8	119.11 (18)	C10—C11—H11C	109.5
C6—C7—C9	119.09 (18)	H11A—C11—H11C	109.5
C8—C7—C9	121.80 (17)	H11B—C11—H11C	109.5
O1—C4—C5	125.20 (18)	O2—C2—H2A	109.5
O1—C4—C3	115.24 (18)	O2—C2—H2B	109.5
C5—C4—C3	119.55 (18)	H2A—C2—H2B	109.5
C7—C6—C5	121.14 (19)	O2—C2—H2C	109.5
C7—C6—H6	119.4	H2A—C2—H2C	109.5
C5—C6—H6	119.4	H2B—C2—H2C	109.5
C3—C8—C7	120.12 (17)	O1—C1—H1A	109.5
C3—C8—H8	119.9	O1—C1—H1B	109.5
C7—C8—H8	119.9	H1A—C1—H1B	109.5
O3—C10—N2	122.96 (19)	O1—C1—H1C	109.5
O3—C10—C11	122.33 (19)	H1A—C1—H1C	109.5
N2—C10—C11	114.71 (19)	H1B—C1—H1C	109.5
C4—C5—C6	119.93 (19)	C10—N2—N1	121.62 (17)
C4—C5—H5	120.0	C10—N2—H2	119.2
C6—C5—H5	120.0	N1—N2—H2	119.2
N1—C9—C7	122.97 (18)	C9—N1—N2	114.28 (17)
C2—O2—C3—C8	6.3 (3)	C6—C7—C8—C3	0.7 (3)
C2—O2—C3—C4	−173.8 (2)	C9—C7—C8—C3	−179.28 (17)
C1—O1—C4—C5	−1.7 (3)	O1—C4—C5—C6	179.13 (18)
C1—O1—C4—C3	177.38 (18)	C3—C4—C5—C6	0.1 (3)
O2—C3—C4—O1	0.1 (2)	C7—C6—C5—C4	1.1 (3)
C8—C3—C4—O1	−179.94 (16)	C6—C7—C9—N1	−171.95 (19)
O2—C3—C4—C5	179.25 (18)	C8—C7—C9—N1	8.1 (3)
C8—C3—C4—C5	−0.8 (3)	O3—C10—N2—N1	3.4 (3)
C8—C7—C6—C5	−1.5 (3)	C11—C10—N2—N1	−176.62 (17)
C9—C7—C6—C5	178.54 (18)	C7—C9—N1—N2	−178.40 (17)
O2—C3—C8—C7	−179.67 (18)	C10—N2—N1—C9	−171.88 (17)
C4—C3—C8—C7	0.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3^{i}	0.86	1.97	2.803 (2)	164

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