# organic compounds

# N′-[1-(4-Methoxyphenyl)ethylidene]acetohydrazide

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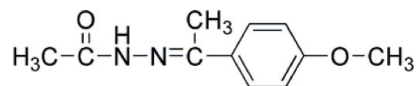Correspondence e-mail: ffjian2008@163.com

The title compound, $C_{11}H_{14}N_2O_2$, was prepared by the reaction of acetohydrazide and 1-(4-methoxyphenyl)-ethanone. In the molecule, all bond lengths and angles are within normal ranges. In the crystal structure, adjacent molecules are linked into a centrosymmetric dimer by intermolecular N—H···O hydrogen bonding.

## Related literature

For related literature, see: Cimerman *et al.* (1997); Girgis (2006). For bond-length data, see: Allen *et al.* (1987).



## Experimental

### Crystal data

| | |
|---|---|
| $C_{11}H_{14}N_2O_2$ | Monoclinic, $P2_1/n$ |
| $M_r$ = 206.24 | $a$ = 13.282 (3) Å |

$b$ = 4.9923 (10) Å
$c$ = 16.854 (3) Å
$\beta$ = 98.88 (3)°
$V$ = 1104.2 (4) Å$^3$
$Z$ = 4

Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 (2) K
$0.25 \times 0.20 \times 0.18$ mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
6830 measured reflections

2681 independent reflections
1228 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.196
$S$ = 0.93
2681 reflections

137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|---|---|---|---|
| N2—H2A···O2$^i$ | 0.86 | 2.12 | 2.956 (3) | 166 |

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2657).

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
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Girgis, A. S. (2006). *J. Chem. Res.* pp. 81–85.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

**supplementary materials**

# *N*'-[1-(4-Methoxyphenyl)ethylidene]acetohydrazide

## Y.-F. Li and F.-F. Jian

## Comment

Schiff bases have received considerable attention in the literature. They are attractive from several points of view, such as the possibility of analytical application (Cimerman *et al.*, 1997). As part of our search for new Schiff base compounds we synthesized the title compound (I), and describe its structure here.

In the title compound (Fig. 1), all bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The C8—N1 bond length of 1.278 (3)Å is comparable with C—N double bond [1.281 (2) Å] reported (Girgis, 2006).

In the crystal structure, adjacent molecules are linked into a centro-symmetric supra-molecular dimer by intermolecular N—H···O hydrogen bonding (Table 1, Fig. 2).

## Experimental

A mixture of the acetohydrazide (0.1 mol), and 1-(4-methoxyphenyl)ethanone (0.1 mol) was stirred in refluxing ethanol (20 mL) for 4 h to afford the title compound (0.080 mol, yield 80%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

## Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H = 0.93-0.96 Å and N—H = 0.86 Å, and with $U_{iso}$=1.2–1.5$U_{eq}$.
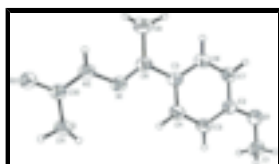
## Figures



Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.
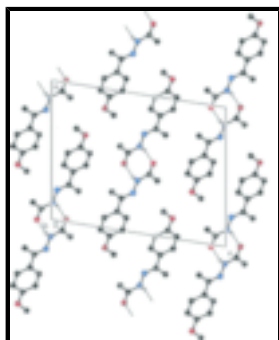


Fig. 2. Part of the crystal structure of the title compound showing the formation of a cyclic centrosymmetric dimer. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted.

# supplementary materials

### *N'*-[1-(4-Methoxyphenyl)ethylidene]acetohydrazide

*Crystal data*

| | |
|---|---|
| $C_{11}H_{14}N_2O_2$ | $F_{000} = 440$ |
| $M_r = 206.24$ | $D_x = 1.241$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| Hall symbol: -P 2yn | Cell parameters from 831 reflections |
| $a = 13.282$ (3) Å | $\theta = 2.4$–24.0º |
| $b = 4.9923$ (10) Å | $\mu = 0.09$ mm$^{-1}$ |
| $c = 16.854$ (3) Å | $T = 293$ (2) K |
| $\beta = 98.88$ (3)º | Block, yellow |
| $V = 1104.2$ (4) Å$^3$ | $0.25 \times 0.20 \times 0.18$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 1228 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.035$ |
| Monochromator: graphite | $\theta_{max} = 28.3$º |
| $T = 273(2)$ K | $\theta_{min} = 1.8$º |
| $\varphi$ and $\omega$ scans | $h = -17 \rightarrow 15$ |
| Absorption correction: none | $k = -6 \rightarrow 6$ |
| 6830 measured reflections | $l = -16 \rightarrow 22$ |
| 2681 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.058$ | H-atom parameters constrained |
| $wR(F^2) = 0.196$ | $w = 1/[\sigma^2(F_o^2) + (0.0986P)^2 + 0.0719P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 0.93$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 2681 reflections | $\Delta\rho_{max} = 0.17$ e Å$^{-3}$ |
| 137 parameters | $\Delta\rho_{min} = -0.17$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*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 ($Å^2$)*

|       | $x$           | $y$         | $z$           | $U_{iso}*/U_{eq}$ |
|-------|---------------|-------------|---------------|-------------------|
| O2    | 0.98272 (13)  | 0.2016 (4)  | 0.91643 (10)  | 0.0761 (6)        |
| O1    | 1.64324 (12)  | 0.8045 (4)  | 1.19345 (10)  | 0.0788 (6)        |
| N2    | 1.12429 (14)  | 0.2219 (4)  | 1.00666 (11)  | 0.0631 (6)        |
| H2A   | 1.1037        | 0.0925      | 1.0338        | 0.076*            |
| N1    | 1.21780 (14)  | 0.3415 (4)  | 1.03226 (11)  | 0.0608 (5)        |
| C5    | 1.36778 (17)  | 0.4053 (4)  | 1.12310 (13)  | 0.0559 (6)        |
| C10   | 1.06577 (19)  | 0.3085 (5)  | 0.93934 (14)  | 0.0606 (6)        |
| C8    | 1.26860 (17)  | 0.2683 (4)  | 1.09940 (13)  | 0.0572 (6)        |
| C2    | 1.55227 (16)  | 0.6811 (5)  | 1.16686 (13)  | 0.0588 (6)        |
| C7    | 1.51710 (18)  | 0.5048 (5)  | 1.21885 (14)  | 0.0675 (7)        |
| H7A   | 1.5547        | 0.4775      | 1.2695        | 0.081*            |
| C3    | 1.49509 (19)  | 0.7215 (5)  | 1.09293 (15)  | 0.0739 (8)        |
| H3A   | 1.5174        | 0.8408      | 1.0570        | 0.089*            |
| C4    | 1.40476 (19)  | 0.5860 (6)  | 1.07177 (14)  | 0.0752 (8)        |
| H4A   | 1.3671        | 0.6163      | 1.0213        | 0.090*            |
| C6    | 1.42740 (18)  | 0.3683 (5)  | 1.19720 (13)  | 0.0632 (7)        |
| H6A   | 1.4061        | 0.2477      | 1.2333        | 0.076*            |
| C11   | 1.1030 (2)    | 0.5331 (5)  | 0.89359 (15)  | 0.0754 (7)        |
| H11A  | 1.0532        | 0.5729      | 0.8475        | 0.113*            |
| H11B  | 1.1660        | 0.4828      | 0.8764        | 0.113*            |
| H11C  | 1.1137        | 0.6886      | 0.9273        | 0.113*            |
| C9    | 1.2343 (2)    | 0.0599 (6)  | 1.15380 (17)  | 0.0931 (10)       |
| H9A   | 1.1689        | 0.1090      | 1.1668        | 0.159 (16)*       |
| H9B   | 1.2828        | 0.0482      | 1.2022        | 0.239*            |
| H9C   | 1.2292        | −0.1105     | 1.1271        | 0.239*            |
| C1    | 1.68123 (19)  | 0.9957 (5)  | 1.14314 (16)  | 0.0807 (8)        |
| H1B   | 1.7453        | 1.0639      | 1.1695        | 0.121*            |
| H1C   | 1.6335        | 1.1404      | 1.1324        | 0.121*            |
| H1D   | 1.6906        | 0.9120      | 1.0935        | 0.121*            |

*Atomic displacement parameters ($Å^2$)*

|       | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$     | $U^{23}$      |
|-------|--------------|--------------|--------------|---------------|--------------|---------------|
| O2    | 0.0790 (12)  | 0.0801 (12)  | 0.0670 (11)  | −0.0227 (10)  | 0.0048 (9)   | 0.0087 (9)    |
| O1    | 0.0687 (11)  | 0.0908 (13)  | 0.0735 (12)  | −0.0172 (10)  | 0.0003 (9)   | 0.0073 (10)   |
| N2    | 0.0729 (13)  | 0.0604 (12)  | 0.0562 (12)  | −0.0161 (10)  | 0.0109 (10)  | 0.0038 (9)    |
| N1    | 0.0651 (12)  | 0.0615 (12)  | 0.0562 (12)  | −0.0126 (9)   | 0.0107 (9)   | −0.0025 (9)   |

| | | | | | | |
|---|---|---|---|---|---|---|
| C5 | 0.0640 (13) | 0.0541 (13) | 0.0515 (13) | −0.0032 (11) | 0.0153 (11) | −0.0005 (10) |
| C10 | 0.0718 (16) | 0.0557 (14) | 0.0558 (14) | −0.0084 (12) | 0.0145 (12) | −0.0020 (11) |
| C8 | 0.0674 (15) | 0.0560 (14) | 0.0506 (13) | −0.0047 (12) | 0.0162 (11) | −0.0009 (11) |
| C2 | 0.0598 (14) | 0.0620 (14) | 0.0549 (13) | 0.0000 (11) | 0.0097 (11) | 0.0000 (11) |
| C7 | 0.0722 (15) | 0.0773 (16) | 0.0510 (13) | −0.0031 (14) | 0.0033 (11) | 0.0059 (12) |
| C3 | 0.0746 (16) | 0.0863 (19) | 0.0598 (15) | −0.0177 (15) | 0.0072 (13) | 0.0196 (14) |
| C4 | 0.0759 (16) | 0.0917 (19) | 0.0542 (14) | −0.0197 (15) | −0.0016 (12) | 0.0178 (13) |
| C6 | 0.0768 (16) | 0.0603 (14) | 0.0533 (14) | −0.0024 (12) | 0.0130 (12) | 0.0081 (11) |
| C11 | 0.0877 (17) | 0.0683 (16) | 0.0696 (16) | −0.0160 (14) | 0.0099 (13) | 0.0132 (13) |
| C9 | 0.101 (2) | 0.097 (2) | 0.0798 (19) | −0.0350 (18) | 0.0105 (16) | 0.0259 (17) |
| C1 | 0.0726 (16) | 0.0842 (19) | 0.0872 (18) | −0.0190 (15) | 0.0183 (14) | 0.0019 (16) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O2—C10 | 1.232 (3) | C7—H7A | 0.9300 |
| O1—C2 | 1.369 (3) | C3—C4 | 1.376 (3) |
| O1—C1 | 1.420 (3) | C3—H3A | 0.9300 |
| N2—C10 | 1.344 (3) | C4—H4A | 0.9300 |
| N2—N1 | 1.386 (2) | C6—H6A | 0.9300 |
| N2—H2A | 0.8600 | C11—H11A | 0.9600 |
| N1—C8 | 1.278 (3) | C11—H11B | 0.9600 |
| C5—C6 | 1.384 (3) | C11—H11C | 0.9600 |
| C5—C4 | 1.391 (3) | C9—H9A | 0.9600 |
| C5—C8 | 1.483 (3) | C9—H9B | 0.9600 |
| C10—C11 | 1.488 (3) | C9—H9C | 0.9600 |
| C8—C9 | 1.503 (3) | C1—H1B | 0.9600 |
| C2—C3 | 1.370 (3) | C1—H1C | 0.9600 |
| C2—C7 | 1.374 (3) | C1—H1D | 0.9600 |
| C7—C6 | 1.372 (3) | | |
| C2—O1—C1 | 118.96 (19) | C3—C4—H4A | 118.8 |
| C10—N2—N1 | 119.9 (2) | C5—C4—H4A | 118.8 |
| C10—N2—H2A | 120.1 | C7—C6—C5 | 121.7 (2) |
| N1—N2—H2A | 120.1 | C7—C6—H6A | 119.1 |
| C8—N1—N2 | 118.54 (19) | C5—C6—H6A | 119.1 |
| C6—C5—C4 | 116.1 (2) | C10—C11—H11A | 109.5 |
| C6—C5—C8 | 122.8 (2) | C10—C11—H11B | 109.5 |
| C4—C5—C8 | 121.0 (2) | H11A—C11—H11B | 109.5 |
| O2—C10—N2 | 119.9 (2) | C10—C11—H11C | 109.5 |
| O2—C10—C11 | 121.1 (2) | H11A—C11—H11C | 109.5 |
| N2—C10—C11 | 119.0 (2) | H11B—C11—H11C | 109.5 |
| N1—C8—C5 | 115.6 (2) | C8—C9—H9A | 109.5 |
| N1—C8—C9 | 124.7 (2) | C8—C9—H9B | 109.5 |
| C5—C8—C9 | 119.7 (2) | H9A—C9—H9B | 109.5 |
| O1—C2—C3 | 124.7 (2) | C8—C9—H9C | 109.5 |
| O1—C2—C7 | 116.7 (2) | H9A—C9—H9C | 109.5 |
| C3—C2—C7 | 118.6 (2) | H9B—C9—H9C | 109.5 |
| C6—C7—C2 | 121.0 (2) | O1—C1—H1B | 109.5 |
| C6—C7—H7A | 119.5 | O1—C1—H1C | 109.5 |
| C2—C7—H7A | 119.5 | H1B—C1—H1C | 109.5 |

| | | | |
|---|---|---|---|
| C2—C3—C4 | 120.1 (2) | O1—C1—H1D | 109.5 |
| C2—C3—H3A | 119.9 | H1B—C1—H1D | 109.5 |
| C4—C3—H3A | 119.9 | H1C—C1—H1D | 109.5 |
| C3—C4—C5 | 122.4 (2) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2A···O2$^i$ | 0.86 | 2.12 | 2.956 (3) | 166 |
| C4—H4A···N1 | 0.93 | 2.44 | 2.755 (3) | 100 |

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

**Fig. 1**

**Fig. 2**