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# 4-Methoxybenzaldehyde 2-methylpropanoylhydrazone

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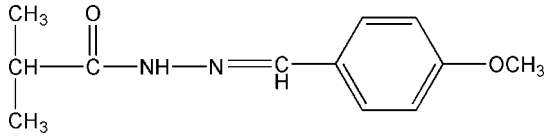
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.047; $wR$ factor = 0.154; data-to-parameter ratio = 17.3.

The title compound, $C_{12}H_{18}N_2O_3$, was prepared by the reaction between *p*-methoxybenzaldehyde and propionylhydrazine. With the exception of the two methyl groups of the $^i$Pr and the H atoms of the OMe group, the molecule is essentially planar. The crystal structure is stabilized by N—H···O, O—H···O and O—H···N hydrogen bonds.

## Related literature

For related literature, see: Allen *et al.* (1987); Sutherland & Hoy (1968); Tucker *et al.* (1975).



## Experimental

### Crystal data

$C_{12}H_{18}N_2O_3\cdot H_2O$
$M_r$ = 238.28
Monoclinic, $P2_1/c$
$a$ = 11.184 (3) Å
$b$ = 12.732 (3) Å
$c$ = 10.634 (3) Å
$\beta$ = 112.636 (4)°
$V$ = 1397.5 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 294 (2) K
0.26 × 0.24 × 0.20 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
7894 measured reflections
2852 independent reflections
1213 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.049

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.154
$S$ = 0.98
2852 reflections
165 parameters
15 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 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 | 1.98 | 2.830 (2) | 170 |
| O3—H3$A$···O2$^{ii}$ | 0.865 (10) | 1.957 (12) | 2.807 (2) | 167 (3) |
| O3—H3$B$···O2 | 0.874 (10) | 2.011 (16) | 2.822 (2) | 154 (2) |
| O3—H3$B$···N1 | 0.874 (10) | 2.570 (19) | 3.246 (3) | 135 (2) |

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2366).

## References

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# supplementary materials

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# 4-Methoxybenzaldehyde 2-methylpropanoylhydrazone

**H.-M. Guo**

## Comment

As an extension of our work on the structural characterization of Schiff base compound, herein we report the crystal and moulecular structures of the title compound (I). The title compound (I) is roughly planar. In (I), the bond lengths are within normal ranges (Allen *et al.*, 1987) (Fig. 1). The C8—N1 distance of 1.274 (3) Å is similar to the reported value of 1.287 Å by Tucker *et al.* (1975). The C9—O2 distance of 1.227 (3) Å is shorter than the distance value of 1.298 Å reported by Sutherland & Hoy (1968).

The crystal structure of (I) is stabilized by N—H···O, O—H···O and O—H···N hydrogen bonds. (Table 1).

## Experimental

A mixture of the *p*-methoxybenzaldehyde (0.1 mol), and propionylhydrazine (0.1 mol) was stirred in refluxing ethanol (30 ml) for 5 h to afford the title compound (I) (0.087 mol, yield 87%). Single crystals of (I) 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 distances = 0.93–0.97 Å, N—H distances = 0.8600 Å and with $U_{iso}$ = 1.2–1.5$U_{eq}$.

## Figures

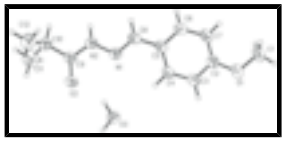

Fig. 1. The structure of the title molecule (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme.

## 4-Methoxybenzaldehyde 2-methylpropanoylhydrazone

*Crystal data*

| | |
|---|---|
| $C_{12}H_{18}N_2O_3·H_2O$ | $Z$ = 4 |
| $M_r$ = 238.28 | $F_{000}$ = 512 |
| Monoclinic, $P2_1/c$ | $D_x$ = 1.133 Mg m$^{-3}$ |
| Hall symbol: -P 2ybc | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 11.184 (3) Å | θ = 2.0–26.5º |
| $b$ = 12.732 (3) Å | μ = 0.08 mm$^{-1}$ |

| | |
|---|---|
| $c$ = 10.634 (3) Å | $T$ = 294 (2) K |
| β = 112.636 (4)º | Block, colourless |
| $V$ = 1397.5 (6) Å$^3$ | 0.26 × 0.24 × 0.20 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 1213 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int}$ = 0.049 |
| Monochromator: graphite | $\theta_{max}$ = 26.5º |
| $T$ = 294(2) K | $\theta_{min}$ = 2.0º |
| φ and ω scans | $h$ = −13→10 |
| Absorption correction: none | $k$ = −15→14 |
| 7894 measured reflections | $l$ = −12→13 |
| 2852 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 |
| $R[F^2 > 2\sigma(F^2)]$ = 0.047 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.154 | $w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 0.0773P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 0.98 | $(\Delta/\sigma)_{max} < 0.001$ |
| 2852 reflections | $\Delta\rho_{max}$ = 0.13 e Å$^{-3}$ |
| 165 parameters | $\Delta\rho_{min}$ = −0.16 e Å$^{-3}$ |
| 15 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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 $F^2 > 2\text{sigma}(F^2)$ 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 | 0.36412 (18) | 0.09825 (15) | 0.47380 (19) | 0.0995 (6) |

| | | | | |
|---|---|---|---|---|
| O2 | 1.00629 (16) | 0.38689 (14) | 1.11832 (17) | 0.0863 (6) |
| N1 | 0.82345 (19) | 0.23666 (15) | 1.0247 (2) | 0.0638 (5) |
| N2 | 0.91436 (18) | 0.23964 (15) | 1.1570 (2) | 0.0670 (6) |
| H2 | 0.9144 | 0.1918 | 1.2141 | 0.080* |
| C1 | 0.2678 (3) | 0.0190 (2) | 0.4440 (3) | 0.1101 (10) |
| H1A | 0.3086 | −0.0487 | 0.4649 | 0.165* |
| H1B | 0.2125 | 0.0218 | 0.3491 | 0.165* |
| H1C | 0.2170 | 0.0301 | 0.4980 | 0.165* |
| C2 | 0.4509 (2) | 0.1095 (2) | 0.6048 (3) | 0.0722 (7) |
| C3 | 0.5326 (2) | 0.1952 (2) | 0.6278 (3) | 0.0833 (8) |
| H3 | 0.5244 | 0.2406 | 0.5564 | 0.100* |
| C4 | 0.6260 (2) | 0.21393 (19) | 0.7550 (3) | 0.0747 (7) |
| H4 | 0.6795 | 0.2723 | 0.7689 | 0.090* |
| C5 | 0.6414 (2) | 0.14673 (18) | 0.8632 (2) | 0.0617 (6) |
| C6 | 0.5582 (3) | 0.0617 (2) | 0.8380 (3) | 0.0781 (7) |
| H6 | 0.5665 | 0.0157 | 0.9088 | 0.094* |
| C7 | 0.4627 (3) | 0.0433 (2) | 0.7104 (3) | 0.0795 (8) |
| H7 | 0.4071 | −0.0137 | 0.6965 | 0.095* |
| C8 | 0.7410 (2) | 0.16238 (18) | 0.9976 (2) | 0.0644 (7) |
| H8 | 0.7448 | 0.1164 | 1.0670 | 0.077* |
| C9 | 1.0029 (2) | 0.3173 (2) | 1.1967 (3) | 0.0678 (7) |
| C10 | 1.0954 (3) | 0.3134 (2) | 1.3437 (3) | 0.0921 (9) |
| H10 | 1.0901 | 0.2444 | 1.3820 | 0.111* |
| C11 | 1.2333 (3) | 0.3322 (3) | 1.3537 (3) | 0.1332 (13) |
| H11A | 1.2557 | 0.2799 | 1.3015 | 0.200* |
| H11B | 1.2920 | 0.3279 | 1.4473 | 0.200* |
| H11C | 1.2391 | 0.4007 | 1.3186 | 0.200* |
| C12 | 1.0567 (3) | 0.3982 (3) | 1.4220 (3) | 0.1276 (13) |
| H12A | 1.0604 | 0.4657 | 1.3835 | 0.191* |
| H12B | 1.1153 | 0.3970 | 1.5159 | 0.191* |
| H12C | 0.9701 | 0.3852 | 1.4161 | 0.191* |
| O3 | 0.88004 (18) | 0.41283 (14) | 0.83391 (17) | 0.0768 (5) |
| H3A | 0.917 (3) | 0.4731 (13) | 0.837 (3) | 0.121 (11)* |
| H3B | 0.905 (3) | 0.386 (2) | 0.9159 (16) | 0.147 (14)* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0838 (13) | 0.1059 (15) | 0.0950 (15) | −0.0318 (11) | 0.0190 (12) | −0.0025 (11) |
| O2 | 0.0979 (13) | 0.0678 (11) | 0.0771 (12) | −0.0194 (9) | 0.0160 (10) | 0.0196 (9) |
| N1 | 0.0636 (12) | 0.0576 (12) | 0.0679 (14) | 0.0022 (10) | 0.0228 (11) | 0.0047 (10) |
| N2 | 0.0715 (13) | 0.0582 (13) | 0.0657 (14) | −0.0021 (10) | 0.0203 (12) | 0.0131 (10) |
| C1 | 0.088 (2) | 0.116 (2) | 0.112 (2) | −0.0408 (19) | 0.0237 (18) | −0.023 (2) |
| C2 | 0.0594 (16) | 0.0748 (17) | 0.0794 (19) | −0.0090 (13) | 0.0234 (15) | −0.0017 (15) |
| C3 | 0.0695 (17) | 0.0817 (19) | 0.089 (2) | −0.0175 (14) | 0.0195 (16) | 0.0179 (15) |
| C4 | 0.0640 (16) | 0.0633 (16) | 0.0880 (19) | −0.0132 (12) | 0.0196 (15) | 0.0106 (14) |
| C5 | 0.0605 (15) | 0.0534 (14) | 0.0760 (17) | 0.0002 (11) | 0.0315 (14) | 0.0012 (12) |
| C6 | 0.0850 (18) | 0.0704 (17) | 0.0829 (19) | −0.0131 (14) | 0.0365 (17) | 0.0089 (14) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C7 | 0.0737 (18) | 0.0677 (17) | 0.097 (2) | −0.0193 (13) | 0.0331 (17) | −0.0017 (16) |
| C8 | 0.0677 (16) | 0.0557 (15) | 0.0751 (18) | 0.0040 (12) | 0.0331 (15) | 0.0060 (12) |
| C9 | 0.0714 (17) | 0.0540 (15) | 0.0742 (17) | −0.0011 (13) | 0.0239 (14) | 0.0095 (14) |
| C10 | 0.093 (2) | 0.081 (2) | 0.081 (2) | −0.0171 (16) | 0.0100 (17) | 0.0248 (16) |
| C11 | 0.081 (2) | 0.137 (3) | 0.146 (3) | 0.007 (2) | 0.004 (2) | 0.033 (2) |
| C12 | 0.149 (3) | 0.148 (3) | 0.088 (2) | −0.042 (2) | 0.049 (2) | −0.013 (2) |
| O3 | 0.1092 (15) | 0.0565 (11) | 0.0653 (12) | −0.0040 (10) | 0.0342 (10) | −0.0018 (9) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.365 (3) | C5—C8 | 1.448 (3) |
| O1—C1 | 1.421 (3) | C6—C7 | 1.386 (3) |
| O2—C9 | 1.227 (3) | C6—H6 | 0.9300 |
| N1—C8 | 1.274 (3) | C7—H7 | 0.9300 |
| N1—N2 | 1.383 (2) | C8—H8 | 0.9300 |
| N2—C9 | 1.347 (3) | C9—C10 | 1.507 (3) |
| N2—H2 | 0.8600 | C10—C11 | 1.524 (4) |
| C1—H1A | 0.9600 | C10—C12 | 1.524 (4) |
| C1—H1B | 0.9600 | C10—H10 | 0.9800 |
| C1—H1C | 0.9600 | C11—H11A | 0.9600 |
| C2—C7 | 1.369 (3) | C11—H11B | 0.9600 |
| C2—C3 | 1.383 (3) | C11—H11C | 0.9600 |
| C3—C4 | 1.375 (3) | C12—H12A | 0.9600 |
| C3—H3 | 0.9300 | C12—H12B | 0.9600 |
| C4—C5 | 1.390 (3) | C12—H12C | 0.9600 |
| C4—H4 | 0.9300 | O3—H3A | 0.865 (10) |
| C5—C6 | 1.385 (3) | O3—H3B | 0.874 (10) |
| C2—O1—C1 | 118.9 (2) | C2—C7—H7 | 120.2 |
| C8—N1—N2 | 115.8 (2) | C6—C7—H7 | 120.2 |
| C9—N2—N1 | 120.18 (19) | N1—C8—C5 | 122.7 (2) |
| C9—N2—H2 | 119.9 | N1—C8—H8 | 118.7 |
| N1—N2—H2 | 119.9 | C5—C8—H8 | 118.7 |
| O1—C1—H1A | 109.5 | O2—C9—N2 | 121.9 (2) |
| O1—C1—H1B | 109.5 | O2—C9—C10 | 122.6 (2) |
| H1A—C1—H1B | 109.5 | N2—C9—C10 | 115.4 (2) |
| O1—C1—H1C | 109.5 | C9—C10—C11 | 109.9 (2) |
| H1A—C1—H1C | 109.5 | C9—C10—C12 | 108.5 (2) |
| H1B—C1—H1C | 109.5 | C11—C10—C12 | 110.1 (3) |
| O1—C2—C7 | 125.3 (2) | C9—C10—H10 | 109.4 |
| O1—C2—C3 | 115.2 (2) | C11—C10—H10 | 109.4 |
| C7—C2—C3 | 119.5 (3) | C12—C10—H10 | 109.4 |
| C4—C3—C2 | 120.7 (2) | C10—C11—H11A | 109.5 |
| C4—C3—H3 | 119.6 | C10—C11—H11B | 109.5 |
| C2—C3—H3 | 119.6 | H11A—C11—H11B | 109.5 |
| C3—C4—C5 | 120.9 (2) | C10—C11—H11C | 109.5 |
| C3—C4—H4 | 119.6 | H11A—C11—H11C | 109.5 |
| C5—C4—H4 | 119.6 | H11B—C11—H11C | 109.5 |
| C6—C5—C4 | 117.4 (2) | C10—C12—H12A | 109.5 |
| C6—C5—C8 | 120.2 (2) | C10—C12—H12B | 109.5 |

| | | | |
|---|---|---|---|
| C4—C5—C8 | 122.4 (2) | H12A—C12—H12B | 109.5 |
| C5—C6—C7 | 121.9 (2) | C10—C12—H12C | 109.5 |
| C5—C6—H6 | 119.0 | H12A—C12—H12C | 109.5 |
| C7—C6—H6 | 119.0 | H12B—C12—H12C | 109.5 |
| C2—C7—C6 | 119.6 (2) | H3A—O3—H3B | 110.1 (15) |
| C8—N1—N2—C9 | −177.8 (2) | C3—C2—C7—C6 | −1.3 (4) |
| C1—O1—C2—C7 | 7.3 (4) | C5—C6—C7—C2 | 1.1 (4) |
| C1—O1—C2—C3 | −173.5 (2) | N2—N1—C8—C5 | −178.47 (19) |
| O1—C2—C3—C4 | −178.7 (2) | C6—C5—C8—N1 | 177.0 (2) |
| C7—C2—C3—C4 | 0.5 (4) | C4—C5—C8—N1 | −2.3 (3) |
| C2—C3—C4—C5 | 0.7 (4) | N1—N2—C9—O2 | −0.1 (3) |
| C3—C4—C5—C6 | −1.0 (4) | N1—N2—C9—C10 | 179.0 (2) |
| C3—C4—C5—C8 | 178.3 (2) | O2—C9—C10—C11 | −46.6 (4) |
| C4—C5—C6—C7 | 0.1 (4) | N2—C9—C10—C11 | 134.3 (3) |
| C8—C5—C6—C7 | −179.2 (2) | O2—C9—C10—C12 | 73.9 (3) |
| O1—C2—C7—C6 | 177.8 (2) | N2—C9—C10—C12 | −105.3 (3) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O3$^{i}$ | 0.86 | 1.98 | 2.830 (2) | 170 |
| O3—H3A···O2$^{ii}$ | 0.865 (10) | 1.957 (12) | 2.807 (2) | 167 (3) |
| O3—H3B···O2 | 0.874 (10) | 2.011 (16) | 2.822 (2) | 154 (2) |
| O3—H3B···N1 | 0.874 (10) | 2.570 (19) | 3.246 (3) | 135 (2) |

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) −*x*+2, −*y*+1, −*z*+2.

**Fig. 1**