

3-(4-Methoxyphenyl)propanohydrazide

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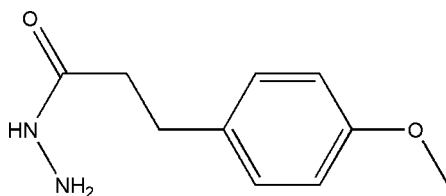
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.066; wR factor = 0.169; data-to-parameter ratio = 17.3.

The title compound, $\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_2$, is an important intermediate for the synthesis of biologically active heterocyclic compounds. The planar hydrazide group is oriented with respect to the benzene ring at a dihedral angle of $81.27(3)^\circ$. The crystal structure is stabilized by $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonding.

Related literature

For general background, see: Zheng *et al.* (2003); Al-Talib *et al.* (1990); Yousif *et al.* (1986); Ahmad *et al.* (2001); Al-Soud *et al.* (2004); El-Emam *et al.* (2004). For synthesis, see: Furniss *et al.* (1978).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_2$
 $M_r = 194.23$
 Monoclinic, $P2_1/c$
 $a = 18.519(9)$ Å
 $b = 4.816(2)$ Å
 $c = 11.884(6)$ Å
 $\beta = 107.521(7)^\circ$

$V = 1010.7(8)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 100(2)$ K
 $0.30 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector
 diffractometer
 Absorption correction: none
 7527 measured reflections

2350 independent reflections
 910 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.126$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.169$
 $S = 0.83$
 2350 reflections
 136 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^{\text{i}}$	0.88	2.04	2.883 (3)	159
$\text{N2}-\text{H2A}\cdots\text{N2}^{\text{ii}}$	0.89 (3)	2.35 (4)	3.174 (4)	154 (3)
$\text{N2}-\text{H2B}\cdots\text{O1}^{\text{iii}}$	0.88 (3)	2.28 (4)	3.112 (4)	158 (3)

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); 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: XU2262).

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supplementary materials

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3-(4-Methoxyphenyl)propanohydrazide

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Comment

Aromatic hydrazides are important intermediates in heterocyclic chemistry and have been used for the synthesis of various biologically active five-membered heterocycles such as 2,5-disubstituted-1,3,4-oxadiazoles (Zheng *et al.*, 2003; Al-Talib *et al.*, 1990) and 5-substituted-2-mercapto-1,3,4-oxadiazoles (Yousif *et al.*, 1986; Ahmad *et al.*, 2001; Al-Soud *et al.*, 2004; El-Emam *et al.*, 2004). In view of the versatility of these compounds, we have synthesized the title compound and reported its crystal structure.

The molecular structure of (I) is shown in Fig. 1. Bond distances and angles are within expected ranges. The dihedral angle between the planar hydrazidic group (C9/O1/N1/N2) and the benzene ring (C1—C6) is 81.27 (3)°. The crystal structure is stabilized by N—H···O and N—H···N hydrogen bonding (Fig. 2).

Experimental

The title compound is synthesized by the reaction of methyl ester of 3-(4-trimethoxyphenyl)propanoic acid with hydrazine hydrate using the reported procedure (Furniss *et al.*, 1978). A mixture of methyl-3-(4-trimethoxyphenyl)propanoate (2.08 g, 10 mmol) and hydrazine hydrate (15 ml, 80%) in absolute ethanol (50 ml) was refluxed for 5 h at 413–423 K. The excess solvent was removed by distillation. The solid residue was filtered off, washed with water and recrystallized from ethanol solution to give the title compound (yield: 1.80 g, 87%). Colorless single crystals were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

Amino H atoms were located in a difference Fourier map and refined isotropically. Other H atoms were placed in calculated positions with N—H = 0.88 Å and C—H = 0.95 (aromatic), 0.99 (methylene) or 0.98 Å (methyl), and refined in riding mode with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl and $1.2U_{\text{eq}}(\text{C}, \text{N})$ for others.

Figures

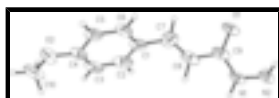


Fig. 1. The molecular structure of (I) with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).



Fig. 2. Crystal packing of (I). Dashed lines indicate hydrogen bonds.



Fig. 3. The synthetic route for the formation of the title compound.

3-(4-Methoxyphenyl)propanohydrazide

Crystal data

$C_{10}H_{14}N_2O_2$

$M_r = 194.23$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 18.519 (9) \text{ \AA}$

$b = 4.816 (2) \text{ \AA}$

$c = 11.884 (6) \text{ \AA}$

$\beta = 107.521 (7)^\circ$

$V = 1010.7 (8) \text{ \AA}^3$

$Z = 4$

$F_{000} = 416$

$D_x = 1.276 \text{ Mg m}^{-3}$

$D_m = 1.253 \text{ Mg m}^{-3}$

D_m measured by not measured

Melting point: 383(2) K

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 691 reflections

$\theta = 3.4\text{--}23.6^\circ$

$\mu = 0.09 \text{ mm}^{-1}$

$T = 100 (2) \text{ K}$

Block, colorless

$0.30 \times 0.20 \times 0.10 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100(2) \text{ K}$

φ and ω scans

Absorption correction: none

7527 measured reflections

2350 independent reflections

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

$R_{\text{int}} = 0.126$

$\theta_{\text{max}} = 28.2^\circ$

$\theta_{\text{min}} = 2.3^\circ$

$h = -24 \rightarrow 24$

$k = -6 \rightarrow 6$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.066$

$wR(F^2) = 0.169$

$S = 0.83$

2350 reflections

136 parameters

Primary atom site location: structure-invariant direct
methods

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

$$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$

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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.74807 (18)	0.8352 (6)	0.4237 (3)	0.0471 (10)
C2	0.7369 (2)	1.0414 (7)	0.3383 (3)	0.0582 (12)
H2	0.6869	1.1033	0.2998	0.070*
C3	0.7965 (2)	1.1595 (6)	0.3074 (3)	0.0500 (10)
H3	0.7868	1.2986	0.2480	0.060*
C4	0.86920 (19)	1.0760 (6)	0.3622 (3)	0.0423 (9)
C5	0.88298 (19)	0.8717 (6)	0.4493 (3)	0.0443 (9)
H5	0.9333	0.8126	0.4881	0.053*
C6	0.82279 (19)	0.7562 (6)	0.4786 (3)	0.0453 (9)
H6	0.8327	0.6180	0.5384	0.054*
C7	0.6831 (2)	0.7041 (8)	0.4551 (4)	0.0615 (12)
H7A	0.6930	0.5029	0.4682	0.074*
H7B	0.6364	0.7257	0.3880	0.074*
C8	0.6703 (2)	0.8297 (7)	0.5645 (3)	0.0487 (10)
H8A	0.7165	0.8033	0.6322	0.058*
H8B	0.6618	1.0319	0.5523	0.058*
C9	0.6042 (2)	0.7035 (6)	0.5935 (3)	0.0453 (9)
C10	0.9193 (3)	1.3676 (7)	0.2403 (4)	0.0759 (14)
H10A	0.8885	1.2778	0.1677	0.114*
H10B	0.9680	1.4244	0.2307	0.114*
H10C	0.8926	1.5315	0.2564	0.114*
N1	0.56013 (17)	0.8802 (5)	0.6305 (2)	0.0478 (8)
H1	0.5721	1.0576	0.6362	0.057*
N2	0.4950 (2)	0.7934 (6)	0.6610 (3)	0.0473 (9)
O1	0.59112 (13)	0.4495 (4)	0.58570 (18)	0.0472 (7)
O2	0.93238 (14)	1.1750 (5)	0.3375 (2)	0.0552 (7)
H2A	0.5135 (17)	0.657 (7)	0.712 (3)	0.044 (10)*
H2B	0.4597 (19)	0.728 (7)	0.599 (3)	0.050 (11)*

Atomic displacement parameters (\AA^2)

U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
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supplementary materials

C1	0.035 (2)	0.0251 (17)	0.069 (3)	-0.0069 (15)	-0.0031 (18)	-0.0161 (17)
C2	0.045 (2)	0.0343 (19)	0.068 (3)	0.0054 (18)	-0.0237 (19)	-0.0168 (19)
C3	0.058 (2)	0.0218 (16)	0.048 (2)	-0.0020 (16)	-0.0172 (19)	-0.0007 (15)
C4	0.046 (2)	0.0263 (17)	0.044 (2)	-0.0077 (15)	-0.0021 (17)	-0.0046 (15)
C5	0.042 (2)	0.0303 (18)	0.049 (2)	0.0050 (16)	-0.0027 (17)	0.0021 (15)
C6	0.046 (2)	0.0245 (16)	0.058 (2)	-0.0002 (15)	0.0055 (18)	0.0052 (15)
C7	0.048 (2)	0.053 (2)	0.079 (3)	-0.0165 (19)	0.012 (2)	-0.030 (2)
C8	0.064 (2)	0.0272 (17)	0.041 (2)	-0.0156 (17)	-0.0050 (18)	-0.0060 (15)
C9	0.068 (3)	0.0252 (16)	0.0290 (18)	-0.0131 (17)	-0.0067 (17)	-0.0032 (15)
C10	0.133 (4)	0.030 (2)	0.067 (3)	-0.020 (2)	0.033 (3)	0.0015 (19)
N1	0.081 (2)	0.0240 (14)	0.0324 (16)	-0.0170 (15)	0.0088 (16)	-0.0044 (11)
N2	0.084 (3)	0.0275 (16)	0.0252 (17)	-0.0105 (16)	0.0086 (18)	0.0009 (13)
O1	0.0653 (16)	0.0243 (11)	0.0417 (13)	-0.0135 (11)	0.0006 (12)	-0.0036 (10)
O2	0.0625 (17)	0.0445 (15)	0.0554 (16)	-0.0158 (13)	0.0129 (13)	0.0019 (12)

Geometric parameters (Å, °)

C1—C2	1.390 (5)	C7—H7B	0.9900
C1—C6	1.393 (4)	C8—C9	1.498 (5)
C1—C7	1.502 (5)	C8—H8A	0.9900
C2—C3	1.385 (5)	C8—H8B	0.9900
C2—H2	0.9500	C9—O1	1.245 (3)
C3—C4	1.368 (4)	C9—N1	1.341 (4)
C3—H3	0.9500	C10—O2	1.445 (4)
C4—O2	1.374 (4)	C10—H10A	0.9800
C4—C5	1.395 (4)	C10—H10B	0.9800
C5—C6	1.381 (4)	C10—H10C	0.9800
C5—H5	0.9500	N1—N2	1.423 (4)
C6—H6	0.9500	N1—H1	0.8800
C7—C8	1.515 (5)	N2—H2A	0.89 (3)
C7—H7A	0.9900	N2—H2B	0.88 (3)
C2—C1—C6	116.4 (3)	H7A—C7—H7B	107.8
C2—C1—C7	121.9 (3)	C9—C8—C7	112.6 (3)
C6—C1—C7	121.7 (3)	C9—C8—H8A	109.1
C3—C2—C1	122.1 (3)	C7—C8—H8A	109.1
C3—C2—H2	118.9	C9—C8—H8B	109.1
C1—C2—H2	118.9	C7—C8—H8B	109.1
C4—C3—C2	120.1 (3)	H8A—C8—H8B	107.8
C4—C3—H3	120.0	O1—C9—N1	121.6 (3)
C2—C3—H3	120.0	O1—C9—C8	122.4 (3)
C3—C4—O2	125.1 (3)	N1—C9—C8	116.0 (3)
C3—C4—C5	119.7 (3)	O2—C10—H10A	109.5
O2—C4—C5	115.2 (3)	O2—C10—H10B	109.5
C6—C5—C4	119.4 (3)	H10A—C10—H10B	109.5
C6—C5—H5	120.3	O2—C10—H10C	109.5
C4—C5—H5	120.3	H10A—C10—H10C	109.5
C5—C6—C1	122.3 (3)	H10B—C10—H10C	109.5
C5—C6—H6	118.8	C9—N1—N2	122.9 (3)
C1—C6—H6	118.8	C9—N1—H1	118.6

C1—C7—C8	112.9 (3)	N2—N1—H1	118.6
C1—C7—H7A	109.0	N1—N2—H2A	102 (2)
C8—C7—H7A	109.0	N1—N2—H2B	111 (2)
C1—C7—H7B	109.0	H2A—N2—H2B	110 (3)
C8—C7—H7B	109.0	C4—O2—C10	116.5 (3)
C6—C1—C2—C3	1.2 (5)	C2—C1—C7—C8	-98.0 (4)
C7—C1—C2—C3	-178.8 (3)	C6—C1—C7—C8	82.1 (4)
C1—C2—C3—C4	-0.7 (5)	C1—C7—C8—C9	178.4 (3)
C2—C3—C4—O2	178.9 (3)	C7—C8—C9—O1	41.3 (5)
C2—C3—C4—C5	0.0 (5)	C7—C8—C9—N1	-139.8 (3)
C3—C4—C5—C6	0.2 (5)	O1—C9—N1—N2	-1.4 (5)
O2—C4—C5—C6	-178.9 (3)	C8—C9—N1—N2	179.7 (3)
C4—C5—C6—C1	0.4 (5)	C3—C4—O2—C10	-4.6 (5)
C2—C1—C6—C5	-1.0 (5)	C5—C4—O2—C10	174.4 (3)
C7—C1—C6—C5	179.0 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O1 ⁱ	0.88	2.04	2.883 (3)	159
N2—H2A...N2 ⁱⁱ	0.89 (3)	2.35 (4)	3.174 (4)	154 (3)
N2—H2B...O1 ⁱⁱⁱ	0.88 (3)	2.28 (4)	3.112 (4)	158 (3)

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, y-1/2, -z+3/2$; (iii) $-x+1, -y+1, -z+1$.

Fig. 1

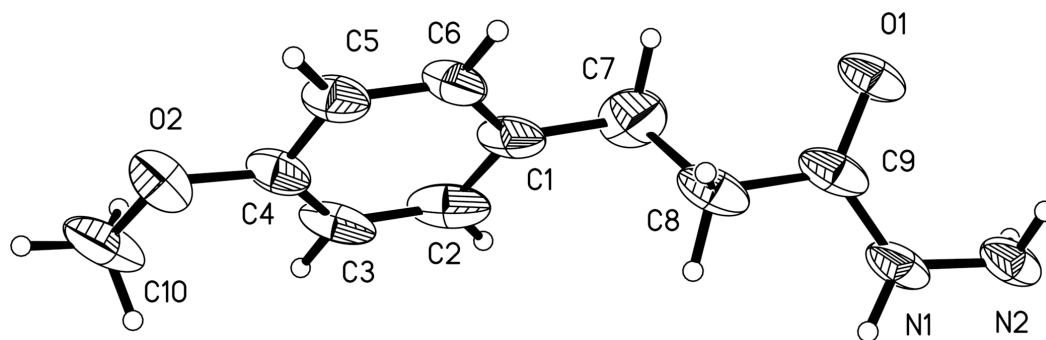


Fig. 2

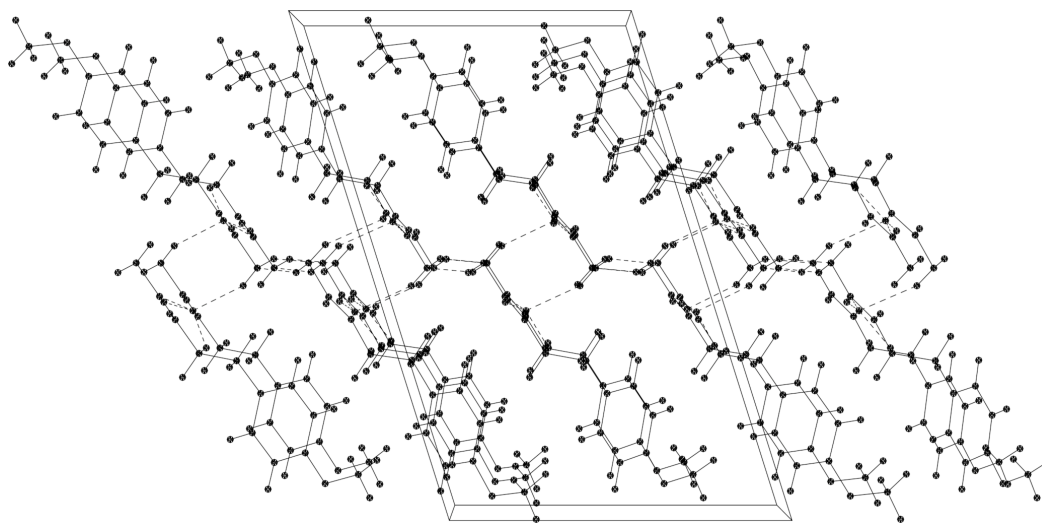


Fig. 3

