organic compounds

2-(3-Methoxyphenyl)acetohydrazide

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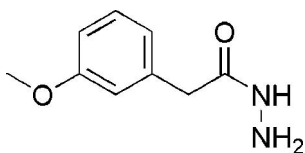
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The title compound, $C_9H_{12}N_2O_2$, is an important intermediate for the synthesis of biologically active heterocyclic compounds. The planar hydrazide group is oriented with respect to the benzene ring at a dihedral angle of 86.66 (3)°. In the crystal structure, intermolecular N—H⋯O hydrogen bonds link the molecules.

Related literature

For related literature, 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); Furniss *et al.* (1978). For bond-length data, see: Allen *et al.* (1987).

Experimental

Crystal data

C9H12N2O2
M_r = 180.21
Monoclinic, $P2_1/c$
a = 13.0330 (8) Å
b = 4.9310 (4) Å
c = 14.604 (1) Å
β = 106.262 (5)°

V = 900.99 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.973, T_{max} = 0.991

7192 measured reflections
1970 independent reflections
1508 reflections with $I > 2\sigma(I)$
R_{int} = 0.090

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.130
S = 1.14
1970 reflections

118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1^i	0.86	2.00	2.863 (2)	175
N2—H2B⋯O1^{ii}	0.94	2.31	3.182 (2)	154

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2377).

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supplementary materials

2-(3-Methoxyphenyl)acetohydrazide

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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, (I), and reported its crystal structure.

In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The dihedral angle between the planar hydrazidic group (C8/O1/N1/N2) and the benzene ring (C1–C6) is 86.66 (3)°.

In the crystal structure, intermolecular N—H⋯O hydrogen bonds (Table 1) link the molecules, in which they seem to be effective in the stabilization of the structure.

Experimental

The title compound, (I), is synthesized by the reaction of methyl ester of 2-(3-methoxyphenyl)acetic acid with hdyrazine hydrate using the reported procedure (Furniss *et al.*, 1978). For the preparation of (I), a mixture of methyl-2-(3-methoxyphenyl)acetate (1.80 g, 10 mmol) and hydrazine hydrate (80%, 15 ml) 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 (30%) to give the title compound (yield; 1.80 g, 83%, m.p. 339–340 K). Colorless single crystals of (I) were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

H atoms of NH_2 group were located in difference syntheses and constrained to ride on their parent atom, [N—H = 0.9239 and 0.9387 Å and $U_{iso}(H) = 1.2U_{eq}(N)$]. The remaining H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C,N)$.
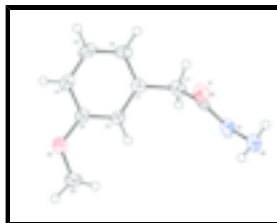
Figures

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

2-(3-Methoxyphenyl)acetohydrazide

Crystal data

$C_9H_{12}N_2O_2$	$F_{000} = 384$
$M_r = 180.21$	$D_x = 1.329$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 339(1) K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 13.0330\ (8)$ Å	Cell parameters from 691 reflections
$b = 4.9310\ (4)$ Å	$\theta = 3.5–23.6°$
$c = 14.604\ (1)$ Å	$\mu = 0.10$ mm^{-1}
$\beta = 106.262\ (5)°$	$T = 294\ (2)$ K
$V = 900.99\ (11)$ Å^3	Block, white
$Z = 4$	$0.30 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	1970 independent reflections
Radiation source: fine-focus sealed tube	1508 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.090$
$T = 294(2)$ K	$\theta_{max} = 27.5°$
φ and ω scans	$\theta_{min} = 2.9°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -16 \rightarrow 13$
$T_{min} = 0.973$, $T_{max} = 0.991$	$k = -6 \rightarrow 6$
7192 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H-atom parameters constrained
$wR(F^2) = 0.130$	$w = 1/[\sigma^2(F_o^2) + (0.0264P)^2 + 0.7351P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.14$	$(\Delta/\sigma)_{max} < 0.001$
1970 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
118 parameters	$\Delta\rho_{min} = -0.33$ 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.50716 (11)	0.5772 (3)	0.62083 (11)	0.0309 (4)
O2	0.91013 (11)	0.4579 (3)	0.56051 (10)	0.0339 (4)
N1	0.45937 (13)	0.1418 (3)	0.58867 (12)	0.0267 (4)
H1	0.4758	−0.0256	0.6015	0.032*
N2	0.36291 (13)	0.2019 (4)	0.51847 (13)	0.0320 (4)
H2A	0.3237	0.3274	0.5416	0.038*
H2B	0.3791	0.2837	0.4661	0.038*
C1	0.72267 (16)	0.3881 (4)	0.70751 (13)	0.0245 (4)
C2	0.76974 (15)	0.3387 (4)	0.63413 (13)	0.0228 (4)
H2	0.7418	0.2063	0.5886	0.027*
C3	0.85827 (15)	0.4880 (4)	0.62976 (13)	0.0246 (4)
C4	0.90129 (16)	0.6834 (4)	0.69845 (15)	0.0308 (5)
H4	0.9609	0.7827	0.6953	0.037*
C5	0.85551 (17)	0.7280 (4)	0.77116 (15)	0.0332 (5)
H5	0.8845	0.8573	0.8175	0.040*
C6	0.76605 (17)	0.5823 (4)	0.77601 (14)	0.0303 (5)
H6	0.7351	0.6149	0.8251	0.036*
C7	0.62388 (16)	0.2311 (4)	0.70976 (14)	0.0286 (5)
H7A	0.6345	0.0409	0.6982	0.034*
H7B	0.6129	0.2468	0.7726	0.034*
C8	0.52577 (16)	0.3328 (4)	0.63582 (14)	0.0237 (4)
C9	0.87378 (18)	0.2483 (5)	0.49202 (15)	0.0355 (5)
H9A	0.9157	0.2484	0.4475	0.043*
H9B	0.8000	0.2784	0.4585	0.043*
H9C	0.8810	0.0763	0.5240	0.043*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0361 (8)	0.0137 (7)	0.0463 (9)	0.0049 (6)	0.0174 (7)	0.0041 (6)
O2	0.0309 (8)	0.0382 (9)	0.0355 (8)	−0.0096 (7)	0.0139 (6)	−0.0046 (7)
N1	0.0318 (9)	0.0142 (8)	0.0366 (9)	0.0030 (7)	0.0139 (7)	0.0053 (7)

N2	0.0279 (9)	0.0257 (9)	0.0431 (10)	0.0020 (7)	0.0112 (8)	0.0072 (8)
C1	0.0298 (10)	0.0186 (9)	0.0243 (9)	0.0057 (8)	0.0064 (8)	0.0060 (8)
C2	0.0255 (10)	0.0184 (9)	0.0226 (9)	−0.0005 (8)	0.0039 (7)	0.0011 (7)
C3	0.0238 (9)	0.0228 (10)	0.0247 (9)	0.0026 (8)	0.0026 (7)	0.0024 (8)
C4	0.0248 (10)	0.0252 (11)	0.0363 (11)	−0.0016 (8)	−0.0016 (8)	0.0009 (9)
C5	0.0353 (12)	0.0256 (11)	0.0316 (11)	0.0035 (9)	−0.0021 (9)	−0.0082 (9)
C6	0.0385 (12)	0.0278 (11)	0.0235 (10)	0.0094 (9)	0.0068 (8)	−0.0004 (9)
C7	0.0381 (11)	0.0219 (10)	0.0293 (10)	0.0038 (9)	0.0153 (8)	0.0074 (9)
C8	0.0306 (10)	0.0162 (9)	0.0316 (10)	0.0021 (8)	0.0210 (8)	0.0041 (8)
C9	0.0395 (12)	0.0393 (13)	0.0313 (11)	−0.0040 (10)	0.0157 (9)	−0.0056 (10)

Geometric parameters (Å, °)

O1—C8	1.237 (2)	C3—C4	1.391 (3)
O2—C3	1.373 (2)	C4—H4	0.9300
O2—C9	1.424 (3)	C5—C4	1.373 (3)
N1—C8	1.333 (3)	C5—C6	1.388 (3)
N1—N2	1.413 (2)	C5—H5	0.9300
N1—H1	0.8601	C6—H6	0.9301
N2—H2A	0.9239	C7—H7A	0.9700
N2—H2B	0.9387	C7—H7B	0.9700
C1—C6	1.386 (3)	C8—C7	1.510 (3)
C1—C2	1.397 (3)	C9—H9A	0.9599
C1—C7	1.510 (3)	C9—H9B	0.9600
C2—C3	1.385 (3)	C9—H9C	0.9600
C2—H2	0.9300		
C3—O2—C9	117.41 (16)	C4—C5—H5	119.8
C8—N1—N2	122.91 (16)	C6—C5—H5	119.6
C8—N1—H1	118.7	C1—C6—C5	120.0 (2)
N2—N1—H1	118.4	C1—C6—H6	119.9
N1—N2—H2A	110.3	C5—C6—H6	120.1
N1—N2—H2B	108.9	C8—C7—C1	111.76 (16)
H2A—N2—H2B	106.4	C8—C7—H7A	109.3
C6—C1—C2	119.74 (19)	C1—C7—H7A	109.1
C6—C1—C7	121.08 (19)	C8—C7—H7B	109.3
C2—C1—C7	119.16 (18)	C1—C7—H7B	109.4
C3—C2—C1	119.53 (18)	H7A—C7—H7B	107.9
C3—C2—H2	120.3	O1—C8—N1	122.04 (19)
C1—C2—H2	120.1	O1—C8—C7	122.35 (19)
O2—C3—C2	124.38 (18)	N1—C8—C7	115.61 (17)
O2—C3—C4	115.10 (18)	O2—C9—H9A	109.6
C2—C3—C4	120.52 (19)	O2—C9—H9B	109.5
C5—C4—C3	119.6 (2)	H9A—C9—H9B	109.5
C5—C4—H4	120.3	O2—C9—H9C	109.3
C3—C4—H4	120.1	H9A—C9—H9C	109.5
C4—C5—C6	120.64 (19)	H9B—C9—H9C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1[i]	0.86	2.00	2.863 (2)	175
N2—H2B···O1[ii]	0.94	2.31	3.182 (2)	154

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

Fig. 1

Fig. 2