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(*E*)-3-(4-Methoxyphenyl)acrylohydrazide

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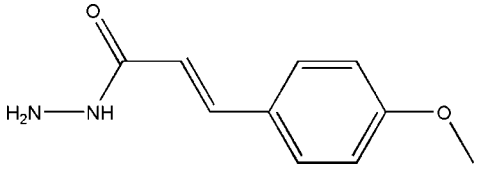
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.143; data-to-parameter ratio = 17.4.

The title compound, $C_{10}H_{12}N_2O_2$, 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 73.93 (3)°.

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); Allen *et al.* (1987); Furniss *et al.* (1978).



Experimental

Crystal data

$C_{10}H_{12}N_2O_2$
M_r = 192.22
Monoclinic, $P2_1/c$
a = 18.661 (5) Å
b = 4.842 (5) Å
c = 12.041 (5) Å
β = 106.774 (5)°
V = 1041.7 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.973, T_{max} = 0.991
5965 measured reflections
2431 independent reflections
1823 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.143
S = 1.04
2431 reflections
140 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003)..

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2254).

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supplementary materials

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(*E*)-3-(4-Methoxyphenyl)acrylohydrazide

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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 the title compound, (I), (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The dihedral angle between the planar hydrazidic group (C10/O2/N1/N2) and benzene ring (C1—C6) is 73.93 (3)°.

Experimental

The title compound, (I), is synthesized by the reaction of methyl ester of (*E*)-3-(4-methoxyphenyl)acrylic acid with hdyrazine hydrate using the reported procedure (Furniss *et al.*, 1978). For the preparation of (I), a mixture of (*E*)-methyl-3-(4-methoxyphenyl)acrylate (1.92 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: 1.74 g, 91%, m.p. 477–499 K). Colorless single crystals of (I) were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

H atoms of NH and NH_2 groups were located in difference syntheses and refined isotropically [N—H = 0.86 (2)–0.91 (2) Å and U_{iso}(H) = 0.065 (5)–0.076 (6) Å^2]. The remaining H atoms were positioned geometrically, with C—H = 0.93, 0.97 and 0.96 Å for aromatic, methine and methyl H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl H, and x = 1.2 for all other H atoms.

Figures

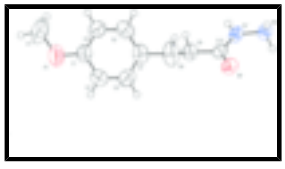


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Fig. 2. The formation of the title molecule.

(*E*)-3-(4-Methoxyphenyl)acrylohydrazide

Crystal data

$C_{10}H_{12}N_2O_2$	F_{000} = 408
	D_x = 1.226 Mg m^{-3}
M_r = 192.22	D_m = 1.213 Mg m^{-3}
	D_m measured by not measured
Monoclinic, $P2_1/c$	Melting point: 477(2) K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation
	λ = 0.71069 Å
a = 18.661 (5) Å	Cell parameters from 691 reflections
b = 4.842 (5) Å	θ = 3.5–23.6º
c = 12.041 (5) Å	μ = 0.09 mm^{-1}
β = 106.774 (5)º	T = 294 (2) K
V = 1041.7 (12) Å^3	Block, colourless
Z = 4	0.30 × 0.20 × 0.10 mm

Data collection

Bruker APEXII diffractometer	R_{int} = 0.033
Radiation source: fine-focus sealed tube	θ_{max} = 28.3º
Monochromator: graphite	θ_{min} = 3.4º
T = 294(2) K	h = −12→24
φ and ω scans	k = −5→6
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	l = −15→12
T_{min} = 0.973, T_{max} = 0.991	3 standard reflections
5965 measured reflections	every 120 min
2431 independent reflections	intensity decay: 1%
1823 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.143	$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 0.1851P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
2431 reflections	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
140 parameters	$\Delta\rho_{min}$ = −0.21 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}
O2	0.90820 (6)	−0.54812 (18)	−0.08595 (10)	0.0575 (3)
N1	0.93934 (7)	−0.1228 (2)	−0.12967 (10)	0.0492 (3)
N2	1.00315 (8)	−0.2078 (3)	−0.16148 (13)	0.0537 (3)
C10	0.89604 (8)	−0.2975 (3)	−0.09272 (11)	0.0453 (3)
C9	0.83040 (9)	−0.1707 (3)	−0.06296 (14)	0.0555 (4)
H9	0.8396	0.0252	−0.0491	0.067*
O1	0.56917 (8)	0.1696 (3)	0.16105 (15)	0.0961 (5)
C1	0.63143 (10)	0.0689 (3)	0.13540 (15)	0.0633 (4)
C4	0.75047 (10)	−0.1683 (4)	0.07282 (16)	0.0644 (4)
C5	0.76173 (10)	0.0303 (4)	0.15675 (16)	0.0697 (5)
H5	0.8103	0.0882	0.1934	0.084*
C2	0.61811 (11)	−0.1264 (5)	0.04886 (18)	0.0798 (6)
H2	0.5694	−0.1799	0.0107	0.096*
C6	0.70327 (11)	0.1480 (4)	0.18902 (16)	0.0691 (5)
H6	0.7129	0.2809	0.2472	0.083*
C3	0.67745 (12)	−0.2423 (4)	0.01908 (18)	0.0805 (6)
H3	0.6679	−0.3750	−0.0392	0.097*
C8	0.81568 (13)	−0.2978 (5)	0.0412 (2)	0.0876 (7)
H8	0.8603	−0.2806	0.1065	0.105*
C7	0.58105 (19)	0.3579 (5)	0.2555 (3)	0.1203 (10)
H7A	0.6114	0.2715	0.3249	0.180*
H7B	0.5337	0.4095	0.2658	0.180*
H7C	0.6061	0.5196	0.2393	0.180*
H1N	0.9298 (10)	0.050 (4)	−0.1277 (15)	0.065 (5)*
H3N	0.9888 (11)	−0.346 (4)	−0.2122 (18)	0.076 (6)*
H2N	1.0344 (10)	−0.286 (4)	−0.0967 (17)	0.071 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0706 (7)	0.0264 (5)	0.0784 (7)	0.0057 (4)	0.0259 (5)	0.0040 (4)
N1	0.0671 (8)	0.0271 (5)	0.0591 (7)	0.0064 (5)	0.0274 (6)	0.0023 (5)

N2	0.0680 (8)	0.0395 (6)	0.0603 (8)	0.0053 (6)	0.0291 (6)	0.0019 (6)
C10	0.0571 (8)	0.0288 (6)	0.0488 (7)	0.0031 (5)	0.0133 (6)	0.0007 (5)
C9	0.0633 (9)	0.0360 (7)	0.0707 (9)	0.0076 (6)	0.0247 (7)	0.0060 (6)
O1	0.0852 (10)	0.0967 (11)	0.1238 (12)	0.0098 (8)	0.0578 (9)	−0.0088 (9)
C1	0.0655 (10)	0.0584 (9)	0.0731 (10)	0.0028 (7)	0.0313 (8)	0.0036 (8)
C4	0.0689 (10)	0.0593 (9)	0.0729 (10)	0.0122 (8)	0.0333 (8)	0.0212 (8)
C5	0.0591 (9)	0.0749 (11)	0.0718 (10)	−0.0025 (8)	0.0138 (8)	0.0130 (9)
C2	0.0621 (10)	0.0892 (13)	0.0891 (13)	−0.0140 (9)	0.0233 (9)	−0.0178 (10)
C6	0.0788 (12)	0.0636 (10)	0.0665 (10)	−0.0051 (9)	0.0233 (8)	−0.0061 (8)
C3	0.0920 (14)	0.0720 (12)	0.0866 (13)	−0.0076 (10)	0.0404 (11)	−0.0206 (10)
C8	0.0942 (14)	0.0828 (13)	0.1035 (15)	0.0373 (11)	0.0566 (12)	0.0409 (12)
C7	0.165 (3)	0.0804 (15)	0.153 (2)	0.0165 (16)	0.105 (2)	−0.0104 (16)

Geometric parameters (Å, °)

O2—C10	1.233 (2)	C4—C3	1.377 (3)
N1—C10	1.3319 (19)	C4—C8	1.512 (2)
N1—N2	1.4132 (17)	C5—C6	1.382 (3)
N1—H1N	0.86 (2)	C5—H5	0.9300
N2—H3N	0.89 (2)	C2—C3	1.378 (3)
N2—H2N	0.91 (2)	C2—H2	0.9300
C10—C9	1.503 (2)	C6—H6	0.9300
C9—C8	1.492 (2)	C3—H3	0.9300
C9—H9	0.9700	C8—H8	0.9700
O1—C7	1.424 (3)	C7—H7A	0.9600
C1—C6	1.364 (3)	C7—H7B	0.9600
C1—C2	1.376 (3)	C7—H7C	0.9600
C4—C5	1.367 (3)		
C10—N1—N2	123.06 (12)	C4—C5—H5	118.9
C10—N1—H1N	117.4 (12)	C6—C5—H5	118.9
N2—N1—H1N	119.2 (12)	C1—C2—C3	119.60 (18)
N1—N2—H3N	107.1 (12)	C1—C2—H2	120.2
N1—N2—H2N	105.7 (12)	C3—C2—H2	120.2
H3N—N2—H2N	105.4 (16)	C1—C6—C5	119.92 (18)
O2—C10—N1	121.96 (14)	C1—C6—H6	120.0
O2—C10—C9	122.24 (13)	C5—C6—H6	120.0
N1—C10—C9	115.78 (12)	C4—C3—C2	122.16 (19)
C8—C9—C10	113.09 (13)	C4—C3—H3	118.9
C8—C9—H9	109.0	C2—C3—H3	118.9
C10—C9—H9	109.0	C9—C8—C4	113.42 (14)
C1—O1—C7	117.35 (19)	C9—C8—H8	108.9
C6—C1—O1	124.95 (17)	C4—C8—H8	108.9
C6—C1—C2	119.31 (16)	O1—C7—H7A	109.5
O1—C1—C2	115.74 (16)	O1—C7—H7B	109.5
C5—C4—C3	116.80 (17)	H7A—C7—H7B	109.5
C5—C4—C8	121.01 (18)	O1—C7—H7C	109.5
C3—C4—C8	122.19 (19)	H7A—C7—H7C	109.5
C4—C5—C6	122.18 (17)	H7B—C7—H7C	109.5
N2—N1—C10—O2	−1.4 (2)	O1—C1—C6—C5	179.34 (16)

N2—N1—C10—C9	−179.85 (13)	C2—C1—C6—C5	−0.5 (3)
O2—C10—C9—C8	40.0 (2)	C4—C5—C6—C1	−0.9 (3)
N1—C10—C9—C8	−141.58 (17)	C5—C4—C3—C2	−0.9 (3)
C7—O1—C1—C6	−4.1 (3)	C8—C4—C3—C2	179.27 (18)
C7—O1—C1—C2	175.79 (19)	C1—C2—C3—C4	−0.4 (3)
C3—C4—C5—C6	1.6 (3)	C10—C9—C8—C4	179.08 (16)
C8—C4—C5—C6	−178.65 (15)	C5—C4—C8—C9	−97.3 (2)
C6—C1—C2—C3	1.1 (3)	C3—C4—C8—C9	82.5 (3)
O1—C1—C2—C3	−178.76 (18)		

Fig. 1

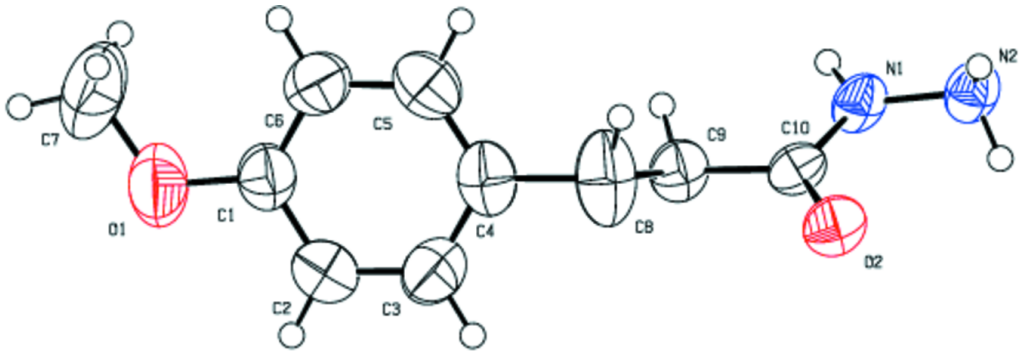
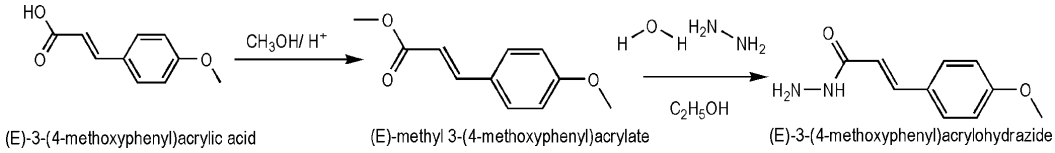


Fig. 2



(E)-3-(4-methoxyphenyl)acrylic acid (E)-methyl 3-(4-methoxyphenyl)acrylate (E)-3-(4-methoxyphenyl)acrylohydrazide