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2-(4-Methylphenoxy)acetohydrazide

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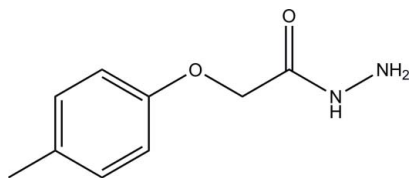
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.067; wR factor = 0.149; data-to-parameter ratio = 16.4.

In the title compound, $\text{C}_9\text{H}_{12}\text{N}_2\text{O}_2$, the acetohydrazide group is approximately planar [maximum deviation = 0.034 (2) Å]. In the crystal, molecules are linked *via* intermolecular N—H...O, N—H...N and C—H...O hydrogen bonds into infinite two-dimensional networks parallel to (001).

Related literature

For general background to and the biological activity of hydrazide derivatives, see: Isloor *et al.* (2009); Holla & Udupa (1992); Ozdemir *et al.* (2009); Khattab (2005); Yale *et al.* (1953). For the preparation of title compound, see: Conti (1964). For bond-length data, see: Allen *et al.* (1987). For related structures, see: Fun *et al.* (2009, 2010a,b).



Experimental

Crystal data

$\text{C}_9\text{H}_{12}\text{N}_2\text{O}_2$	$V = 935.87$ (5) Å ³
$M_r = 180.21$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 6.3833$ (2) Å	$\mu = 0.09$ mm ⁻¹
$b = 4.0755$ (1) Å	$T = 296$ K
$c = 35.9741$ (12) Å	$0.46 \times 0.33 \times 0.10$ mm
$\beta = 90.018$ (2)°	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

* Thomson Reuters ResearcherID: A-3561-2009.

§ Thomson Reuters ResearcherID: A-5525-2009.

Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
 $T_{\min} = 0.959$, $T_{\max} = 0.991$
 15060 measured reflections

2150 independent reflections
 1747 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.149$
 $S = 1.14$
 2150 reflections
 131 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N1}\cdots\text{N2}^i$	0.92 (3)	2.17 (3)	2.982 (3)	147 (2)
$\text{N2}-\text{H2N2}\cdots\text{O2}^{ii}$	0.90 (3)	2.14 (3)	3.022 (3)	168 (3)
$\text{N2}-\text{H1N2}\cdots\text{O2}^{iii}$	0.98 (3)	2.47 (3)	3.166 (3)	128 (2)
$\text{C1}-\text{H1A}\cdots\text{O2}^{iv}$	0.93	2.53	3.410 (3)	157

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5765).

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supplementary materials

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2-(4-Methylphenoxy)acetohydrazide

H.-K. Fun, C. K. Quah, S. Malladi, V. A. M. and A. M. Isloor

Comment

The full therapeutic possibilities of hydrazides were realized after the discovery of isonicotinic acid hydrazide (INH). Hydrazides and their derivatives have been described as useful synthons of various heterocyclic rings (Isloor *et al.*, 2009; Holla & Udupa, 1992). A large number of hydrazides and their derivatives are reported to possess a broad spectrum of biological activities (Ozdemir *et al.*, 2009; Khattab, 2005). The most widely used method to prepare hydrazides is hydrazinolysis of the corresponding esters with hydrazine hydrate (Yale *et al.*, 1953). Prompted by the diverse activities of hydrazides and its derivatives, we have synthesized the title compound to study its crystal structure.

The molecular structure is shown in Fig. 1. The acetohydrazide group (C7/C8/N1/N2/O2) is approximately planar, with the maximum deviation of 0.034 (2) Å at atom C7. Bond lengths and angles are within normal ranges, and comparable to closely related structures (Fun *et al.*, 2009, 2010*a,b*). In the solid state (Fig. 2), the molecules are linked *via* intermolecular N2–H1N2···O2, N2–H2N2···O2 and C1–H1A···O2 trifurcated acceptor bonds, together with N1–H1N1···N2 hydrogen bonds, into infinite two-dimensional networks parallel to plane (001).

Experimental

Ethyl(4-methylphenoxy)acetate (1.94 g, 0.01 mol) and hydrazine hydrate (99%, 0.02 mol) in ethanol (15 ml) was heated on a water-bath for 6 h. Excess ethanol was removed by distillation. On cooling, colourless needle-shaped crystals of 2-(4-methylphenoxy)acetohydrazide begin to separate. It was collected by filtration and recrystallized from ethanol. Yield: 1.2 g, 67.0 %, *M.p.*: 411–413K. (Conti, 1964).

Refinement

H1N1, H1N2 and H2N2 were located in a difference Fourier map and allowed to refined freely. The remaining H atoms were positioned geometrically and refined using a riding model with C–H = 0.93 –0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$. The highest residual electron density peak is located at 0.94 Å from H9C and the deepest hole is located at 0.94 Å from C8.

Figures

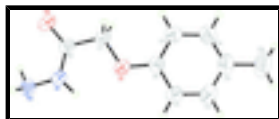


Fig. 1. The molecular structure of the title compound showing 50% probability displacement ellipsoids for non-H atoms and the atom-numbering scheme.

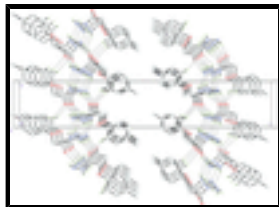


Fig. 2. The crystal structure of the title compound, viewed along the *b* axis. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity.

2-(4-Methylphenoxy)acetohydrazide

Crystal data

$C_9H_{12}N_2O_2$

$M_r = 180.21$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 6.3833$ (2) Å

$b = 4.0755$ (1) Å

$c = 35.9741$ (12) Å

$\beta = 90.018$ (2)°

$V = 935.87$ (5) Å³

$Z = 4$

$F(000) = 384$

$D_x = 1.279$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4756 reflections

$\theta = 3.2$ – 25.4 °

$\mu = 0.09$ mm⁻¹

$T = 296$ K

Block, colourless

$0.46 \times 0.33 \times 0.10$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

ϕ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2009)

$T_{\min} = 0.959$, $T_{\max} = 0.991$

15060 measured reflections

2150 independent reflections

1747 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.3$ °

$h = -8 \rightarrow 8$

$k = -5 \rightarrow 5$

$l = -46 \rightarrow 45$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.149$

$S = 1.14$

2150 reflections

131 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

$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2 + 0.8025P]$

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

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

$\Delta\rho_{\max} = 0.21$ e Å⁻³

0 restraints

$$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.8895 (2)	0.9310 (5)	0.16293 (4)	0.0486 (5)
O2	0.5021 (2)	0.4131 (5)	0.20181 (5)	0.0518 (5)
N1	0.8051 (3)	0.6119 (5)	0.22475 (5)	0.0386 (5)
N2	0.7875 (3)	0.4400 (6)	0.25893 (6)	0.0424 (5)
C1	1.1646 (4)	1.1960 (7)	0.13257 (7)	0.0492 (6)
H1A	1.2259	1.2295	0.1557	0.059*
C2	1.2626 (4)	1.3059 (7)	0.10089 (8)	0.0568 (7)
H2A	1.3912	1.4113	0.1030	0.068*
C3	1.1765 (5)	1.2649 (7)	0.06605 (8)	0.0581 (7)
C4	0.9852 (5)	1.1122 (8)	0.06415 (7)	0.0614 (8)
H4A	0.9226	1.0843	0.0410	0.074*
C5	0.8816 (4)	0.9975 (7)	0.09561 (7)	0.0529 (7)
H5A	0.7514	0.8967	0.0935	0.063*
C6	0.9740 (3)	1.0352 (6)	0.12983 (6)	0.0407 (5)
C7	0.7042 (3)	0.7393 (6)	0.16147 (6)	0.0405 (5)
H7A	0.7183	0.5731	0.1423	0.049*
H7B	0.5864	0.8784	0.1550	0.049*
C8	0.6637 (3)	0.5776 (6)	0.19811 (6)	0.0374 (5)
C9	1.2895 (6)	1.3842 (10)	0.03148 (9)	0.0890 (11)
H9A	1.3602	1.2033	0.0199	0.133*
H9B	1.3900	1.5489	0.0383	0.133*
H9C	1.1897	1.4761	0.0144	0.133*
H1N1	0.919 (4)	0.745 (7)	0.2202 (7)	0.058 (8)*
H2N2	0.712 (5)	0.572 (8)	0.2738 (8)	0.066 (9)*
H1N2	0.705 (5)	0.243 (9)	0.2537 (8)	0.070 (9)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0452 (9)	0.0592 (11)	0.0415 (9)	-0.0185 (8)	0.0003 (7)	0.0002 (8)
O2	0.0347 (8)	0.0604 (11)	0.0604 (11)	-0.0179 (8)	-0.0005 (7)	0.0015 (9)

supplementary materials

N1	0.0297 (9)	0.0437 (11)	0.0424 (10)	-0.0053 (8)	0.0015 (7)	0.0008 (9)
N2	0.0350 (10)	0.0473 (12)	0.0448 (11)	0.0032 (9)	0.0032 (8)	0.0029 (10)
C1	0.0411 (12)	0.0547 (15)	0.0519 (14)	-0.0064 (11)	-0.0038 (10)	0.0032 (12)
C2	0.0443 (13)	0.0590 (17)	0.0671 (18)	-0.0089 (12)	0.0074 (12)	0.0027 (14)
C3	0.0676 (17)	0.0532 (16)	0.0534 (16)	-0.0053 (14)	0.0135 (13)	0.0042 (13)
C4	0.0749 (19)	0.0663 (19)	0.0431 (14)	-0.0118 (16)	-0.0020 (13)	0.0015 (13)
C5	0.0507 (14)	0.0592 (17)	0.0487 (14)	-0.0118 (13)	-0.0026 (11)	-0.0002 (12)
C6	0.0405 (11)	0.0396 (12)	0.0418 (12)	-0.0001 (10)	0.0020 (9)	-0.0010 (10)
C7	0.0329 (11)	0.0430 (13)	0.0455 (13)	-0.0068 (10)	-0.0008 (9)	-0.0037 (10)
C8	0.0283 (10)	0.0385 (11)	0.0453 (12)	0.0015 (9)	0.0027 (8)	-0.0053 (10)
C9	0.107 (3)	0.091 (3)	0.070 (2)	-0.022 (2)	0.0300 (19)	0.010 (2)

Geometric parameters (Å, °)

O1—C6	1.375 (3)	C3—C4	1.372 (4)
O1—C7	1.418 (3)	C3—C9	1.518 (4)
O2—C8	1.238 (3)	C4—C5	1.392 (4)
N1—C8	1.323 (3)	C4—H4A	0.9300
N1—N2	1.420 (3)	C5—C6	1.374 (3)
N1—H1N1	0.92 (3)	C5—H5A	0.9300
N2—H2N2	0.90 (3)	C7—C8	1.496 (3)
N2—H1N2	0.98 (3)	C7—H7A	0.9700
C1—C2	1.375 (4)	C7—H7B	0.9700
C1—C6	1.385 (3)	C9—H9A	0.9600
C1—H1A	0.9300	C9—H9B	0.9600
C2—C3	1.378 (4)	C9—H9C	0.9600
C2—H2A	0.9300		
C6—O1—C7	117.73 (17)	C6—C5—H5A	120.4
C8—N1—N2	121.39 (19)	C4—C5—H5A	120.4
C8—N1—H1N1	118.1 (16)	C5—C6—O1	125.0 (2)
N2—N1—H1N1	120.5 (16)	C5—C6—C1	119.6 (2)
N1—N2—H2N2	105.3 (19)	O1—C6—C1	115.4 (2)
N1—N2—H1N2	106.3 (17)	O1—C7—C8	110.74 (17)
H2N2—N2—H1N2	108 (2)	O1—C7—H7A	109.5
C2—C1—C6	119.7 (2)	C8—C7—H7A	109.5
C2—C1—H1A	120.2	O1—C7—H7B	109.5
C6—C1—H1A	120.2	C8—C7—H7B	109.5
C1—C2—C3	122.2 (2)	H7A—C7—H7B	108.1
C1—C2—H2A	118.9	O2—C8—N1	123.2 (2)
C3—C2—H2A	118.9	O2—C8—C7	118.52 (19)
C4—C3—C2	117.1 (2)	N1—C8—C7	118.25 (19)
C4—C3—C9	121.8 (3)	C3—C9—H9A	109.5
C2—C3—C9	121.1 (3)	C3—C9—H9B	109.5
C3—C4—C5	122.3 (3)	H9A—C9—H9B	109.5
C3—C4—H4A	118.8	C3—C9—H9C	109.5
C5—C4—H4A	118.8	H9A—C9—H9C	109.5
C6—C5—C4	119.2 (2)	H9B—C9—H9C	109.5
C6—C1—C2—C3	0.7 (4)	C7—O1—C6—C1	-174.4 (2)
C1—C2—C3—C4	0.9 (5)	C2—C1—C6—C5	-2.3 (4)

C1—C2—C3—C9	-179.1 (3)	C2—C1—C6—O1	179.2 (2)
C2—C3—C4—C5	-0.9 (5)	C6—O1—C7—C8	165.76 (19)
C9—C3—C4—C5	179.1 (3)	N2—N1—C8—O2	4.7 (3)
C3—C4—C5—C6	-0.6 (5)	N2—N1—C8—C7	-173.8 (2)
C4—C5—C6—O1	-179.4 (2)	O1—C7—C8—O2	176.8 (2)
C4—C5—C6—C1	2.2 (4)	O1—C7—C8—N1	-4.6 (3)
C7—O1—C6—C5	7.2 (4)		

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—H1N1...N2 ⁱ	0.92 (3)	2.17 (3)	2.982 (3)	147 (2)
N2—H2N2...O2 ⁱⁱ	0.90 (3)	2.14 (3)	3.022 (3)	168 (3)
N2—H1N2...O2 ⁱⁱⁱ	0.98 (3)	2.47 (3)	3.166 (3)	128 (2)
C1—H1A...O2 ^{iv}	0.93	2.53	3.410 (3)	157.

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

Fig. 1

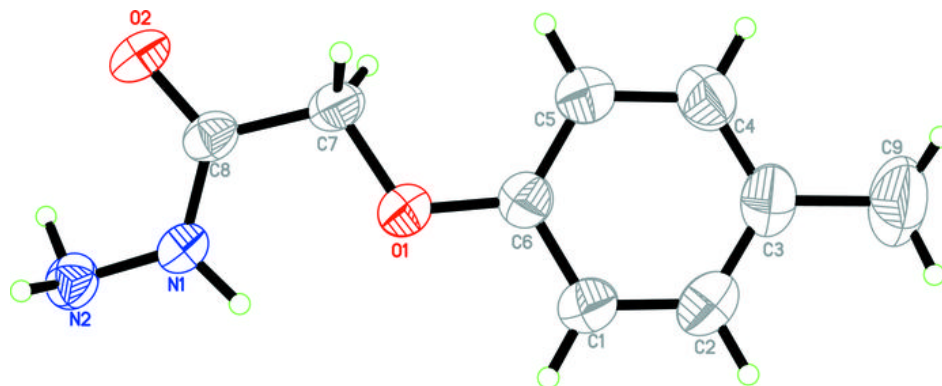


Fig. 2

