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2-(2-Chlorophenoxy)acetohydrazide

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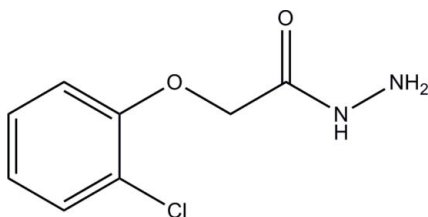
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.050; wR factor = 0.121; data-to-parameter ratio = 20.5.

In the title compound, $\text{C}_8\text{H}_9\text{ClN}_2\text{O}_2$, the acetohydrazide group is approximately planar, with the maximum deviation of 0.031 (2) Å. In the crystal, the molecules are linked by $\text{N}-\text{H}\cdots\text{N}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, with the acetohydrazide O atom accepting two $\text{C}-\text{H}\cdots\text{O}$ links and one $\text{N}-\text{H}\cdots\text{O}$ link. This results in infinite sheets lying parallel to (100).

Related literature

For general background to and biological properties of hydrazine derivatives, see: Rando *et al.* (2008); Kumar *et al.* (2009); Kamal *et al.* (2007); Masunari & Tavares (2007); Rando *et al.* (2002). For related structures, see: Fun *et al.* (2009, 2010). For the preparation, see: Holla & Udupa (1992). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_8\text{H}_9\text{ClN}_2\text{O}_2$ $a = 15.2384$ (5) Å
 $M_r = 200.62$ $b = 3.9269$ (1) Å
 Monoclinic, $P2_1/c$ $c = 16.8843$ (6) Å

$\beta = 117.269$ (2)°
 $V = 898.07$ (5) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.39$ mm⁻¹
 $T = 100$ K
 $0.28 \times 0.10 \times 0.09$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 Absorption correction: multi-scan SADABS (Bruker, 2005)
 $T_{\min} = 0.897$, $T_{\max} = 0.965$

11351 measured reflections
 2662 independent reflections
 2029 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.121$
 $S = 1.05$
 2662 reflections
 130 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.44$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N1}\cdots\text{N2}^{\text{i}}$	0.83 (3)	2.20 (2)	2.930 (3)	148 (2)
$\text{N2}-\text{H1N2}\cdots\text{O2}^{\text{ii}}$	0.91 (3)	2.36 (2)	3.070 (2)	134 (2)
$\text{C1}-\text{H1A}\cdots\text{O2}^{\text{iii}}$	0.93	2.54	3.443 (3)	164
$\text{C7}-\text{H7A}\cdots\text{O2}^{\text{iv}}$	0.97	2.37	3.317 (2)	165

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; 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: HB5257).

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supplementary materials

Acta Cryst. (2010). E66, o31-o32 [doi:10.1107/S1600536809051356]

2-(2-Chlorophenoxy)acetohydrazide

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Comment

Hydrazine derivatives have been reported to possess several biological properties. 5-nitro-2-heterocyclic benzylidene hydrazides were found to possess antileishmanial activities (Rando *et al.*, 2008). Many substituted benzoic acid furan-2-yl-methylene hydrazides showed potent antimicrobial properties (Kumar *et al.*, 2009). Hydrazine derivatives were also associated with remarkable anticancer (Kamal *et al.*, 2007), antibacterial (Masunari & Tavares, 2007) and tuberculostatic (Rando *et al.*, 2002) activities.

The molecular structure is shown in Fig. 1. The acetohydrazide group (C7/C8/N1/N2/O2) is approximately planar, with the maximum deviation of 0.031 (2) Å for atom N1. Bond lengths and angles are within normal ranges, and comparable to closely related structures (Fun *et al.*, 2009, 2010). In the solid state (Fig. 2), the molecules are linked *via* intermolecular N2—H1N2···O2, C1—H1A···O2 and C7—H7A···O2 trifurcated acceptor bonds, together with N1—H1N1···N2 hydrogen bonds, into infinite two-dimensional networks parallel to plane (1 0 0).

Experimental

O-chloro phenol (11 ml, 1.00 mmol), ethyl chloroacetate (10.7 ml, 1.00 mmol) and potassium carbonate (20.75 g, 1.50 mmol) were refluxed in acetone (100 ml) at 80 °C for 18 h. The reaction mixture is then filtered, distilled to remove the acetone and poured into ice cold water with vigorous stirring. The ester, phenoxy ethyl acetate was extracted using ether. The solution was distilled to remove ether. Phenoxy ethyl acetate (8.2 ml, 0.50 mmol) was heated at 100 °C for 10h in an absolute alcohol medium (40 ml) with hydrazine hydrate (2.5 ml, 0.50 mmol). The reaction mixture was allowed to cool, the solid separated was filtered, dried and recrystallized from ethanol. The yield was found to be 7.1 g (71 %). *M. p.* 384-385 K (Holla & Udupa, 1992).

Refinement

Atoms H1N1, H1N2 and H2N2 were located from the difference Fourier map and refined freely. The remaining H atoms were positioned geometrically and refined using a riding model, with C-H = 0.93 and 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Figures

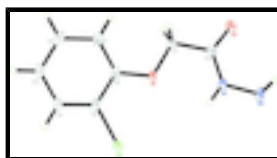


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids.

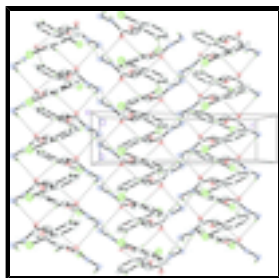


Fig. 2. The crystal structure of (I) viewed along the *a* axis. H atoms not involved in intermolecular interactions (dashed lines) have been omitted for clarity.

2-(2-Chlorophenoxy)acetohydrazide

Crystal data

$C_8H_9ClN_2O_2$

$M_r = 200.62$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 15.2384$ (5) Å

$b = 3.9269$ (1) Å

$c = 16.8843$ (6) Å

$\beta = 117.269$ (2)°

$V = 898.07$ (5) Å³

$Z = 4$

$F(000) = 416$

$D_x = 1.484$ Mg m⁻³

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

Cell parameters from 3388 reflections

$\theta = 2.4$ – 30.1 °

$\mu = 0.39$ mm⁻¹

$T = 100$ K

Block, colourless

$0.28 \times 0.10 \times 0.09$ mm

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

φ and ω scans

Absorption correction: multi-scan
SADABS (Bruker, 2005)

$T_{\min} = 0.897$, $T_{\max} = 0.965$

11351 measured reflections

2662 independent reflections

2029 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\max} = 30.2$ °, $\theta_{\min} = 1.5$ °

$h = -21 \rightarrow 20$

$k = -5 \rightarrow 4$

$l = -22 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.121$

$S = 1.05$

2662 reflections

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

$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.3771P]$

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

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

130 parameters

$$\Delta\rho_{\max} = 0.44 \text{ e } \text{\AA}^{-3}$$

0 restraints

$$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.69094 (3)	0.22956 (13)	0.86423 (3)	0.02146 (15)
O1	0.83867 (8)	0.0410 (4)	0.81001 (8)	0.0174 (3)
O2	1.09342 (9)	-0.0760 (4)	0.87110 (8)	0.0183 (3)
N1	1.01406 (11)	0.2382 (4)	0.93081 (10)	0.0158 (3)
N2	1.10162 (11)	0.3687 (5)	1.00234 (10)	0.0175 (3)
C1	0.72527 (14)	-0.1775 (5)	0.66271 (12)	0.0192 (4)
H1A	0.7768	-0.2398	0.6506	0.023*
C2	0.62775 (14)	-0.2331 (5)	0.59963 (12)	0.0232 (4)
H2A	0.6144	-0.3314	0.5452	0.028*
C3	0.55039 (14)	-0.1436 (6)	0.61715 (13)	0.0236 (4)
H3A	0.4855	-0.1815	0.5746	0.028*
C4	0.57002 (12)	0.0024 (5)	0.69829 (12)	0.0197 (4)
H4A	0.5185	0.0633	0.7105	0.024*
C5	0.66699 (13)	0.0571 (5)	0.76107 (12)	0.0177 (4)
C6	0.74518 (12)	-0.0283 (5)	0.74390 (11)	0.0161 (4)
C7	0.91785 (12)	-0.0685 (5)	0.79289 (11)	0.0154 (4)
H7A	0.9113	0.0356	0.7383	0.018*
H7B	0.9149	-0.3137	0.7851	0.018*
C8	1.01638 (12)	0.0305 (5)	0.86985 (11)	0.0147 (4)
H1N1	0.9633 (17)	0.307 (5)	0.9322 (14)	0.014 (5)*
H1N2	1.1345 (16)	0.478 (6)	0.9761 (14)	0.018 (6)*
H2N2	1.1333 (17)	0.195 (6)	1.0302 (16)	0.022 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0139 (2)	0.0305 (3)	0.0168 (2)	0.00075 (17)	0.00427 (15)	-0.00393 (19)
O1	0.0092 (5)	0.0272 (8)	0.0117 (6)	-0.0005 (5)	0.0012 (4)	-0.0029 (5)

supplementary materials

O2	0.0137 (6)	0.0242 (8)	0.0163 (6)	0.0013 (5)	0.0063 (5)	-0.0006 (5)
N1	0.0092 (6)	0.0219 (8)	0.0123 (7)	0.0002 (6)	0.0016 (5)	-0.0024 (6)
N2	0.0113 (6)	0.0238 (9)	0.0114 (7)	-0.0012 (6)	-0.0002 (6)	-0.0012 (6)
C1	0.0177 (8)	0.0214 (10)	0.0142 (8)	-0.0010 (7)	0.0035 (7)	-0.0001 (7)
C2	0.0226 (9)	0.0242 (10)	0.0136 (8)	-0.0030 (8)	0.0003 (7)	-0.0023 (8)
C3	0.0145 (8)	0.0259 (11)	0.0201 (9)	-0.0035 (7)	-0.0011 (7)	-0.0010 (8)
C4	0.0113 (7)	0.0224 (10)	0.0205 (9)	-0.0013 (7)	0.0029 (7)	0.0006 (8)
C5	0.0154 (8)	0.0184 (10)	0.0140 (8)	-0.0007 (7)	0.0023 (6)	0.0002 (7)
C6	0.0115 (7)	0.0175 (9)	0.0140 (8)	-0.0011 (7)	0.0012 (6)	0.0019 (7)
C7	0.0124 (7)	0.0195 (10)	0.0116 (7)	-0.0005 (7)	0.0031 (6)	-0.0007 (7)
C8	0.0142 (7)	0.0162 (9)	0.0115 (7)	0.0002 (7)	0.0041 (6)	0.0032 (6)

Geometric parameters (Å, °)

C11—C5	1.7443 (19)	C1—H1A	0.9300
O1—C6	1.3756 (19)	C2—C3	1.386 (3)
O1—C7	1.430 (2)	C2—H2A	0.9300
O2—C8	1.237 (2)	C3—C4	1.385 (3)
N1—C8	1.326 (2)	C3—H3A	0.9300
N1—N2	1.4231 (19)	C4—C5	1.385 (2)
N1—H1N1	0.83 (2)	C4—H4A	0.9300
N2—H1N2	0.91 (2)	C5—C6	1.390 (3)
N2—H2N2	0.84 (2)	C7—C8	1.519 (2)
C1—C6	1.391 (3)	C7—H7A	0.9700
C1—C2	1.393 (2)	C7—H7B	0.9700
C6—O1—C7	115.63 (14)	C5—C4—H4A	120.3
C8—N1—N2	122.15 (15)	C3—C4—H4A	120.3
C8—N1—H1N1	125.4 (15)	C4—C5—C6	121.25 (17)
N2—N1—H1N1	112.5 (15)	C4—C5—C11	119.09 (15)
N1—N2—H1N2	105.5 (13)	C6—C5—C11	119.65 (13)
N1—N2—H2N2	104.8 (16)	O1—C6—C5	116.80 (16)
H1N2—N2—H2N2	110 (2)	O1—C6—C1	124.09 (17)
C6—C1—C2	119.63 (18)	C5—C6—C1	119.11 (16)
C6—C1—H1A	120.2	O1—C7—C8	110.19 (14)
C2—C1—H1A	120.2	O1—C7—H7A	109.6
C3—C2—C1	120.73 (18)	C8—C7—H7A	109.6
C3—C2—H2A	119.6	O1—C7—H7B	109.6
C1—C2—H2A	119.6	C8—C7—H7B	109.6
C4—C3—C2	119.78 (17)	H7A—C7—H7B	108.1
C4—C3—H3A	120.1	O2—C8—N1	123.86 (16)
C2—C3—H3A	120.1	O2—C8—C7	119.11 (16)
C5—C4—C3	119.50 (18)	N1—C8—C7	117.00 (15)
C6—C1—C2—C3	-0.5 (3)	C4—C5—C6—C1	-1.3 (3)
C1—C2—C3—C4	-0.1 (3)	C11—C5—C6—C1	177.58 (15)
C2—C3—C4—C5	0.0 (3)	C2—C1—C6—O1	-179.16 (18)
C3—C4—C5—C6	0.7 (3)	C2—C1—C6—C5	1.1 (3)
C3—C4—C5—C11	-178.13 (16)	C6—O1—C7—C8	178.73 (15)
C7—O1—C6—C5	176.26 (16)	N2—N1—C8—O2	2.4 (3)
C7—O1—C6—C1	-3.5 (3)	N2—N1—C8—C7	-175.28 (16)

C4—C5—C6—O1	179.00 (18)	O1—C7—C8—O2	172.20 (16)
C11—C5—C6—O1	-2.1 (2)	O1—C7—C8—N1	-10.0 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N1 \cdots N2 ⁱ	0.83 (3)	2.20 (2)	2.930 (3)	148 (2)
N2—H1N2 \cdots O2 ⁱⁱ	0.91 (3)	2.36 (2)	3.070 (2)	134 (2)
C1—H1A \cdots O2 ⁱⁱⁱ	0.93	2.54	3.443 (3)	164
C7—H7A \cdots O2 ^{iv}	0.97	2.37	3.317 (2)	165

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

Fig. 1

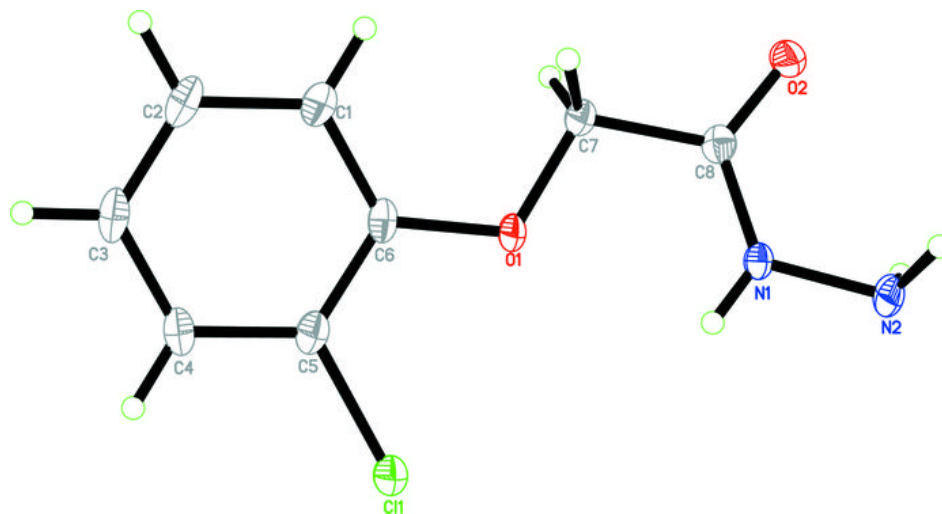


Fig. 2

