

(2-Methylphenoxy)acetic acid

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.078
wR factor = 0.230
Data-to-parameter ratio = 11.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Dimeric hydrogen bonding is present in the crystal structure of (2-methylphenoxy)acetic acid, $\text{C}_9\text{H}_{10}\text{O}_3$, involving the carboxylate groups of centrosymmetrically related pairs of molecules. The structure is further stabilized by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding.

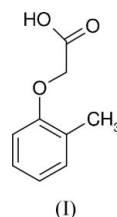
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Comment

The title compound, (I), also known as (*o*-tolylxy)acetic acid, has been described as an expectorant (Negwer, 1996), and several phenoxyacetic acid compounds are used as herbicides (Cserhati & Forgacs, 1998).



The atomic arrangement in (I) is shown in Fig. 1 and selected geometric parameters are given in Table 1. Apart from the H atoms, the molecule is essentially planar and the torsion angle with the greatest deviation from 0, or $\pm 180^\circ$, is $\text{C}2-\text{O}1-\text{C}7-\text{C}8 = 175.6 (2)^\circ$.

Carboxylic acids normally form dimers or catemers and here, as expected for a simple monocarboxylic acid, dimers are formed by intermolecular hydrogen bonding involving the carboxylate groups. The pairs of molecules forming the dimers are related by a centre of symmetry and details of the hydrogen bonding are given in Table 2. As well as the dimeric $R_2^2(8)$ motif, a weak $\text{C}-\text{H}\cdots\text{O}$ contact (Table 2) is present and

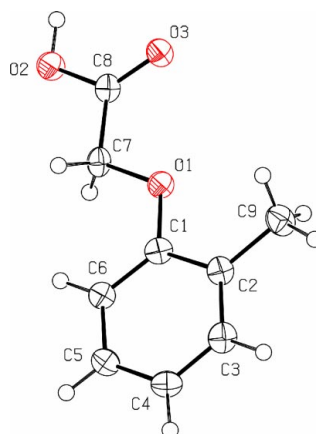


Figure 1

The atomic arrangement in the molecule of (I), with the the atom-numbering scheme and 50% probability displacement ellipsoids.

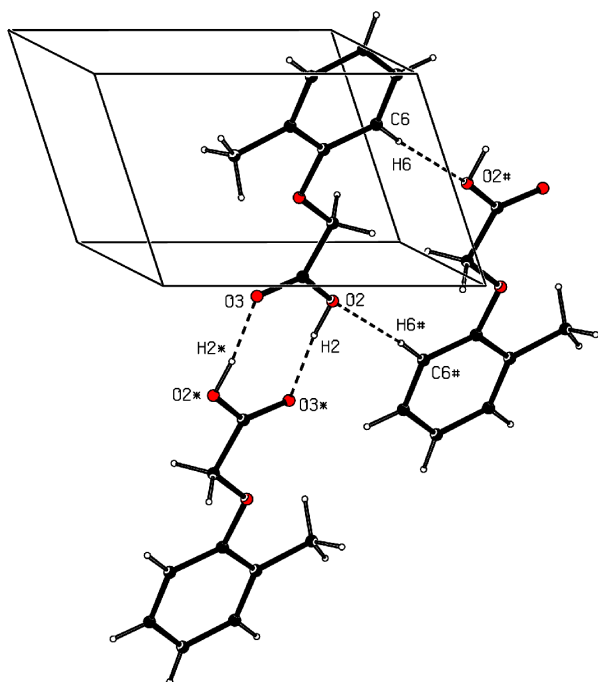


Figure 2

A partial packing diagram of (I), showing $R_2^2(8)$ and $R_2^2(14)$ ring formations. [Symmetry codes: (*) $-1-x, -y, 1-z$; (#) $-x, -y, 2-z$.]

this links two molecules about a centre of symmetry in an $R_2^2(14)$ formation (Fig. 2). Hence, atom O2 acts as both a donor and an acceptor. Indications of C—H $\cdots\pi$ bonding (Table 2) are also present as H7A is close to the centroid (Cg1) of the aromatic ring.

There are many similar examples of dimer formation involving carboxylate groups *e.g.* phenylacetic acid (Hodgson & Asplund, 1991) and 2,4,5-trimethylbenzoic acid (Barcon *et al.*, 1997). The solid-state structures of related compounds, *viz.* (4-methylphenoxy)acetic acid (Kumar & Rao, 1982) and 3-(2-hydroxyphenyl)propionic acid (Begum *et al.*, 1992), have also been determined.

Experimental

The title compound was obtained from Aldrich and was recrystallized from a mixture of methanol and ethanol.

Crystal data

$C_9H_{10}O_3$	$D_x = 1.376 \text{ Mg m}^{-3}$
$M_r = 166.17$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2909 reflections
$a = 5.1062(5) \text{ \AA}$	$\theta = 3.0\text{--}27.5^\circ$
$b = 22.352(2) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 7.4014(9) \text{ \AA}$	$T = 150(2) \text{ K}$
$\beta = 108.235(5)^\circ$	Plate, colourless
$V = 802.33(14) \text{ \AA}^3$	$0.40 \times 0.24 \times 0.06 \text{ mm}$
$Z = 4$	

Data collection

Enraf–Nonius KappaCCD area detector	1304 independent reflections
φ and ω scans	1006 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	$R_{\text{int}} = 0.055$
$T_{\text{min}} = 0.97, T_{\text{max}} = 0.99$	$\theta_{\text{max}} = 27.5^\circ$
2908 measured reflections	$h = -6 \rightarrow 5$
	$k = -28 \rightarrow 29$
	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.078$
 $wR(F^2) = 0.230$
 $S = 1.04$
 1304 reflections
 115 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1765P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.53 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.07 (2)

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

O1—C1	1.381 (3)	O2—C8	1.322 (3)
O1—C7	1.415 (3)	O3—C8	1.219 (3)
C1—O1—C7	116.32 (19)	O3—C8—C7	124.8 (2)
O1—C1—C2	114.7 (2)	O2—C8—C7	110.8 (2)
O3—C8—O2	124.4 (2)		
O1—C1—C2—C9	3.7 (4)	C1—O1—C7—C8	175.6 (2)

Table 2

Hydrogen-bonding geometry ($\text{\AA}, ^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O2—H2 \cdots O3 ⁱ	0.93 (4)	1.69 (4)	2.618 (3)	172 (4)
C6—H6 \cdots O2 ⁱⁱ	0.95	2.56	3.505 (3)	175
C7—H7A \cdots Cg1 ⁱⁱⁱ	0.99	2.60	3.37	134

Symmetry codes: (i) $-1-x, -y, 1-z$; (ii) $-x, -y, 2-z$; (iii) $x-1, y, z$. Cg1 is the centroid of the aromatic ring.

The crystal diffracted very weakly and decomposed during data collection, hence data completeness is only 71%. The hydroxy (O2) H atom was refined isotropically. The C—H H atoms were allowed to ride on their attached C atoms: C—H distances were 0.95 \AA (aromatic), 0.99 \AA (methylene) and 0.98 \AA (methyl), and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{non-methyl C})$ or $1.4U_{\text{eq}}(\text{methyl C})$.

Data collection: DENZO (Otwinowski & Minor, 1997) and COLLECT (Hooft, 1998); cell refinement: DENZO and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: WinGX (Farrugia, 1999).

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supporting information

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*Crystal data*C₉H₁₀O₃ $M_r = 166.17$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 5.1062$ (5) Å $b = 22.352$ (2) Å $c = 7.4014$ (9) Å $\beta = 108.235$ (5)° $V = 802.33$ (14) Å³ $Z = 4$ $F(000) = 352$ $D_x = 1.376$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2909 reflections

 $\theta = 3.0$ – 27.5 ° $\mu = 0.10$ mm⁻¹ $T = 150$ K

Plate, colourless

 $0.40 \times 0.24 \times 0.06$ mm*Data collection*Enraf–Nonius KappaCCD area detector
diffractometerRadiation source: Enraf–Nonius FR591 rotating
anode

Graphite monochromator

Detector resolution: 9.091 pixels mm⁻¹ φ and ω scansAbsorption correction: multi-scan
(SORTAV; Blessing, 1995) $T_{\min} = 0.97$, $T_{\max} = 0.99$

2908 measured reflections

1304 independent reflections

1006 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.055$ $\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.0$ ° $h = -6 \rightarrow 5$ $k = -28 \rightarrow 29$ $l = -9 \rightarrow 9$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.078$ $wR(F^2) = 0.230$ $S = 1.04$

1304 reflections

115 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.1765P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.36$ e Å⁻³ $\Delta\rho_{\min} = -0.53$ e Å⁻³

Extinction correction: SHELXL97,

 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.07 (2)

Special details

Experimental. Please note cell_measurement_fields are not relevant to area detector data, the entire data set is used to refine the cell, which is indexed from all observed reflections in a 10 degree phi range.

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 $F^2 > \sigma(F^2)$ 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	0.2301 (3)	0.09458 (8)	0.7120 (2)	0.0268 (6)
O2	-0.2795 (4)	-0.00989 (9)	0.7344 (3)	0.0307 (7)
H2	-0.443 (7)	-0.022 (2)	0.645 (5)	0.062 (12)*
O3	-0.2447 (3)	0.04691 (8)	0.4924 (3)	0.0267 (6)
C1	0.4626 (5)	0.12234 (11)	0.8299 (4)	0.0240 (7)
C2	0.5808 (5)	0.16514 (11)	0.7399 (4)	0.0256 (7)
C3	0.8208 (6)	0.19291 (12)	0.8493 (4)	0.0288 (8)
H3	0.9040	0.2221	0.7915	0.035*
C4	0.9440 (5)	0.17961 (12)	1.0406 (4)	0.0278 (7)
H4	1.1116	0.1984	1.1112	0.033*
C5	0.8177 (5)	0.13824 (12)	1.1272 (4)	0.0268 (7)
H5	0.8958	0.1296	1.2589	0.032*
C6	0.5776 (5)	0.10956 (11)	1.0210 (4)	0.0246 (7)
H6	0.4924	0.0811	1.0799	0.030*
C7	0.1007 (5)	0.05243 (11)	0.7989 (4)	0.0247 (7)
H7A	0.0566	0.0714	0.9067	0.030*
H7B	0.2272	0.0185	0.8489	0.030*
C8	-0.1568 (5)	0.03024 (11)	0.6572 (4)	0.0236 (7)
C9	0.4458 (6)	0.18060 (13)	0.5338 (4)	0.0334 (8)
H9A	0.5338	0.2163	0.5017	0.047*
H9B	0.2495	0.1886	0.5114	0.047*
H9C	0.4662	0.1470	0.4541	0.047*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0295 (11)	0.0223 (10)	0.0275 (13)	-0.0086 (7)	0.0072 (9)	0.0024 (8)
O2	0.0338 (12)	0.0250 (10)	0.0308 (13)	-0.0092 (8)	0.0066 (10)	0.0048 (8)
O3	0.0329 (12)	0.0200 (9)	0.0258 (13)	-0.0051 (7)	0.0070 (10)	0.0006 (7)
C1	0.0270 (14)	0.0150 (12)	0.0297 (16)	0.0000 (9)	0.0083 (12)	-0.0019 (10)
C2	0.0322 (15)	0.0170 (12)	0.0280 (15)	-0.0010 (10)	0.0100 (12)	-0.0004 (11)
C3	0.0372 (15)	0.0157 (12)	0.0344 (18)	-0.0038 (10)	0.0126 (13)	-0.0005 (11)
C4	0.0282 (14)	0.0192 (13)	0.0331 (18)	-0.0046 (9)	0.0055 (13)	-0.0062 (11)
C5	0.0305 (15)	0.0225 (14)	0.0257 (15)	0.0002 (10)	0.0063 (12)	-0.0035 (11)

C6	0.0278 (14)	0.0182 (12)	0.0274 (16)	-0.0001 (9)	0.0081 (12)	0.0002 (11)
C7	0.0341 (16)	0.0164 (12)	0.0261 (15)	-0.0045 (9)	0.0133 (13)	-0.0001 (10)
C8	0.0310 (14)	0.0147 (12)	0.0268 (16)	-0.0006 (9)	0.0114 (13)	-0.0005 (10)
C9	0.0412 (16)	0.0241 (14)	0.0319 (19)	-0.0037 (11)	0.0072 (14)	0.0070 (11)

Geometric parameters (Å, °)

O1—C1	1.381 (3)	C4—C5	1.393 (4)
O1—C7	1.415 (3)	C4—H4	0.9500
O2—C8	1.322 (3)	C5—C6	1.389 (3)
O2—H2	0.93 (4)	C5—H5	0.9500
O3—C8	1.219 (3)	C6—H6	0.9500
C1—C6	1.381 (4)	C7—C8	1.487 (3)
C1—C2	1.405 (4)	C7—H7A	0.9900
C2—C3	1.386 (4)	C7—H7B	0.9900
C2—C9	1.504 (4)	C9—H9A	0.9800
C3—C4	1.390 (4)	C9—H9B	0.9800
C3—H3	0.9500	C9—H9C	0.9800
C1—O1—C7	116.32 (19)	C1—C6—H6	120.0
C8—O2—H2	109 (2)	C5—C6—H6	120.0
O1—C1—C6	124.1 (2)	O1—C7—C8	109.7 (2)
O1—C1—C2	114.7 (2)	O1—C7—H7A	109.7
C6—C1—C2	121.2 (2)	C8—C7—H7A	109.7
C3—C2—C1	117.4 (2)	O1—C7—H7B	109.7
C3—C2—C9	122.0 (2)	C8—C7—H7B	109.7
C1—C2—C9	120.6 (2)	H7A—C7—H7B	108.2
C2—C3—C4	122.4 (3)	O3—C8—O2	124.4 (2)
C2—C3—H3	118.8	O3—C8—C7	124.8 (2)
C4—C3—H3	118.8	O2—C8—C7	110.8 (2)
C3—C4—C5	118.9 (2)	C2—C9—H9A	109.5
C3—C4—H4	120.5	C2—C9—H9B	109.5
C5—C4—H4	120.5	H9A—C9—H9B	109.5
C6—C5—C4	120.0 (3)	C2—C9—H9C	109.5
C6—C5—H5	120.0	H9A—C9—H9C	109.5
C4—C5—H5	120.0	H9B—C9—H9C	109.5
C1—C6—C5	120.1 (3)		
C7—O1—C1—C6	2.6 (4)	C2—C3—C4—C5	-2.0 (4)
C7—O1—C1—C2	-178.2 (2)	C3—C4—C5—C6	2.1 (4)
O1—C1—C2—C3	-177.6 (2)	O1—C1—C6—C5	177.7 (2)
C6—C1—C2—C3	1.5 (4)	C2—C1—C6—C5	-1.4 (4)
O1—C1—C2—C9	3.7 (4)	C4—C5—C6—C1	-0.5 (4)
C6—C1—C2—C9	-177.2 (2)	C1—O1—C7—C8	175.6 (2)
C1—C2—C3—C4	0.2 (4)	O1—C7—C8—O3	-1.3 (4)
C9—C2—C3—C4	178.8 (3)	O1—C7—C8—O2	179.1 (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>
O2—H2 \cdots O3 ⁱ	0.93 (4)	1.69 (4)	2.618 (3)	172 (4)
C6—H6 \cdots O2 ⁱⁱ	0.95	2.56	3.505 (3)	175
C7—H7A \cdots Cg1 ⁱⁱⁱ	0.99	2.60	3.37	134

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