

-2-(2-Isopropylphenoxy)acetic acid

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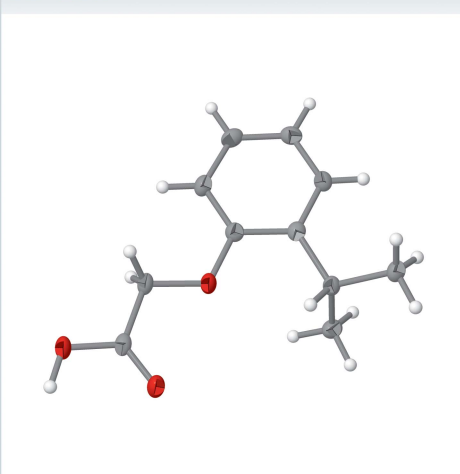
Keywords: crystal structure; phenoxy carboxylic acid; O—H...O hydrogen bonds; inversion dimer.

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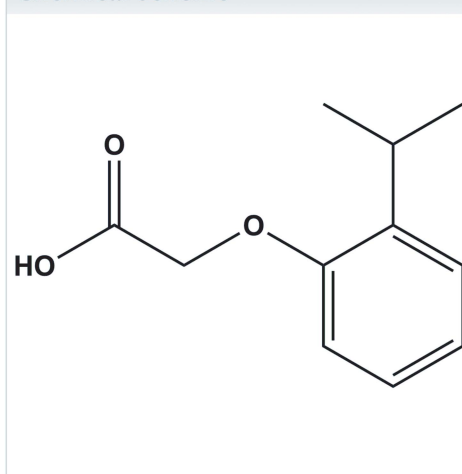
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, C₁₁H₁₄O₃, the pendant —OCH₂COOH chain is in an extended conformation and almost lies in the plane of the benzene ring, as indicated by the dihedral angle of 2.61 (5)°. In the crystal, molecules are linked by a pair of O—H...O hydrogen bonds, forming classical carboxylic acid inversion dimers, with an R₂²(8) loop. The crystal structure of this compound have been reported previously [Smith *et al.* (1992). *Aust. J. Chem.* **45**, 1101–1108], however, in that report, the unit-cell dimensions differ significantly from those of the title structure and the carboxyl H atom was disordered within a cyclic hydrogen-bonded dimer.

3D view



Chemical scheme



Structure description

Phenoxyacetic acid derivatives are interesting building blocks in a variety of natural and synthetic compounds found to possess a variety of pharmacological applications, such as anticancer, analgesic, anti-inflammatory and gastrin receptor antagonistic activities (Gurupadaswamy *et al.*, 2013). Moreover, derivatives of phenoxyacetic acid are characterized by a broad spectrum of physiological activity whose type and strength depends on the substituents in both the aromatic ring and the acid fragment (Okawara *et al.*, 1988). As part of our ongoing research on this class of compounds (Mohammed *et al.*, 2016), the title compound was synthesized and we report herein its crystal structure. The molecule is being assessed for its biological activity.

The molecular structure of the title compound is shown in Fig. 1. The pendant —OCH₂COOH chain [O10/C11/C12/O13/O14; maximum deviation = 0.031 (1) Å for atom C11] is in an extended conformation and almost lies in the plane of the benzene ring (atoms C1–C6), as indicated by the dihedral angle of 2.61 (5)°. The bond lengths and

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O13-H13\cdots O14^i$	0.92 (2)	1.73 (2)	2.6392 (11)	173.5 (17)

Symmetry code: (i) $-x + 3, -y + 2, -z + 1$.



Figure 1
A view of the molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

angles are similar to those reported for the *tert*-butyl analogue 2-(2-*tert*-butylphenoxy)acetic acid (Kennard *et al.*, 1987).

In the crystal, molecules are linked by a pair of $O-H\cdots O$ hydrogen bonds, forming classical carboxylic acid inversion dimers, with an $R_2^2(8)$ loop (Table 1 and Fig. 2). There are no other significant intermolecular interactions present.

The crystal structure of this compound have been reported previously (Smith *et al.*, 1992), however, in that report, the unit-cell dimensions differ significantly from those of the title compound:

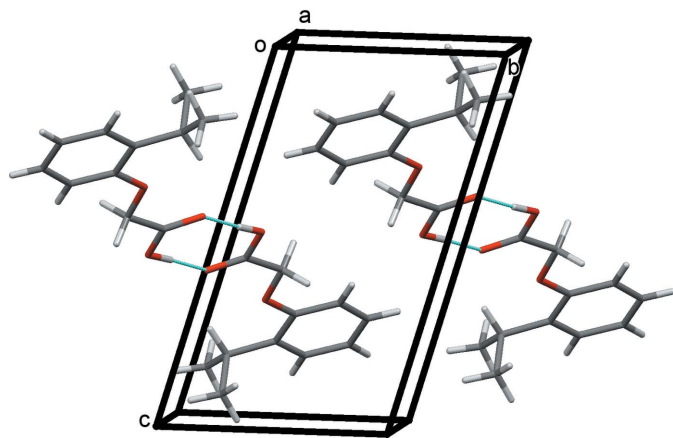


Figure 2
A view along the a axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{14}O_3$
M_r	194.22
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	296
a, b, c (Å)	5.9825 (2), 7.8623 (2), 11.9240 (3)
α, β, γ (°)	104.564 (1), 93.570 (1), 112.302 (1)
V (Å ³)	494.38 (2)
Z	2
Radiation type	Cu $K\alpha$
μ (mm ⁻¹)	0.77
Crystal size (mm)	0.29 × 0.26 × 0.22
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	Multi-scan (SADABS; Bruker, 2013)
T_{min}, T_{max}	0.807, 0.848
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5019, 1617, 1549
R_{int}	0.027
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.585
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.033, 0.090, 1.05
No. of reflections	1617
No. of parameters	134
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.14, -0.17

Computer programs: APEX2 and SAINT (Bruker, 2013), SHELXS97 (Sheldrick, 2008), Mercury (Macrae *et al.*, 2008), SHELXL2014 (Sheldrick, 2015) and PLATON (Spek, 2009).

a, b, c (Å) 5.9946 (8), 7.944 (1), 12.177 (1); α, β, γ (°) 103.666 (9), 94.890 (9), 111.74 (1) (Smith *et al.*, 1992)

cf. a, b, c (Å) 5.9825 (2), 7.8623 (2), 11.9240 (3); α, β, γ (°) 104.564 (1), 93.570 (1), 112.302 (1) (present structural report).

It was also found that the carboxyl H atom was disordered within a cyclic hydrogen-bonded dimer, which is not the case in the present structural report.

Synthesis and crystallization

A mixture of 2-isopropylphenol (0.03 mol), ethyl chloroacetate (0.045 mol) and anhydrous potassium carbonate (0.03 mol) in dry acetone (50 ml) was refluxed for 12 h. The reaction mixture was cooled and the solvent removed by distillation. The residual mass was triturated with cold water to remove potassium carbonate, and extracted with ether (3 × 30 ml). The ether layer was washed with 10% sodium hydroxide solution (3 × 30 ml) followed by water (3 × 30 ml) and then dried over anhydrous sodium sulfate and evaporated to give isopropyl phenoxy ethyl acetate. This compound (0.015 mol) was then dissolved in ethanol (15 ml) and a sodium hydroxide (0.025 mol) solution in water (5 ml) was added. The mixture was refluxed for 9 h and the reaction mixture was cooled and acidified with 5 M hydrochloric acid. The precipitate was filtered off, washed with water and recrystallized from ethanol to yield colourless block-like crystals (yield 88%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The carboxyl H atom (H13) was located in a difference Fourier map and freely refined.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161714 [https://doi.org/10.1107/S2414314616017144]

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2-(2-Isopropylphenoxy)acetic acid

Crystal data

$C_{11}H_{14}O_3$

$M_r = 194.22$

Triclinic, $P\bar{1}$

$a = 5.9825$ (2) Å

$b = 7.8623$ (2) Å

$c = 11.9240$ (3) Å

$\alpha = 104.564$ (1)°

$\beta = 93.570$ (1)°

$\gamma = 112.302$ (1)°

$V = 494.38$ (2) Å³

$Z = 2$

$F(000) = 208$

$D_x = 1.305$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 1549 reflections

$\theta = 6.4$ – 64.4 °

$\mu = 0.77$ mm⁻¹

$T = 296$ K

Block, colourless

$0.29 \times 0.26 \times 0.22$ mm

Data collection

Bruker X8 Proteum
diffractometer

Radiation source: Bruker MicroStar microfocus
rotating anode

Helios multilayer optics monochromator

Detector resolution: 18.4 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2013)

$T_{\min} = 0.807$, $T_{\max} = 0.848$

5019 measured reflections

1617 independent reflections

1549 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 64.4$ °, $\theta_{\min} = 6.4$ °

$h = -6 \rightarrow 6$

$k = -8 \rightarrow 9$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.090$

$S = 1.05$

1617 reflections

134 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.1244P]$

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

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

$\Delta\rho_{\max} = 0.14$ e Å⁻³

$\Delta\rho_{\min} = -0.17$ e Å⁻³

Extinction correction: SHELXL2014
(Sheldrick, 2015),

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

Extinction coefficient: 0.021 (4)

Special details

Experimental. ^1H NMR(400 MHz, CdCl_2 , δ ppm) 1.25(s, 6H, 2CH_3), 4.69 (s, 2H, OCH_2), 6.73-7.26 (m, 4H, Ar-H), 9.18 (s, 1H, OH)

LC-MS m/z 195 (M+1).

Anal. Calcd. for $\text{C}_{11}\text{H}_{14}\text{O}_6$: C, 68.02; H, 7.27; O, 24.71 Found: C, 68.31; H, 7.06; O, 24.53%.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4051 (2)	0.64683 (15)	0.21029 (9)	0.0187 (3)
C2	0.5345 (2)	0.59261 (15)	0.28716 (9)	0.0197 (3)
C3	0.4231 (2)	0.42232 (16)	0.31556 (10)	0.0230 (3)
H3	0.5119	0.3885	0.3662	0.028*
C4	0.1784 (2)	0.30264 (16)	0.26815 (10)	0.0237 (3)
H4	0.1038	0.1879	0.2863	0.028*
C5	0.0462 (2)	0.35395 (16)	0.19412 (10)	0.0237 (3)
H5	-0.1182	0.2750	0.1632	0.028*
C6	0.1600 (2)	0.52438 (16)	0.16600 (10)	0.0217 (3)
H6	0.0692	0.5575	0.1159	0.026*
C7	0.53617 (19)	0.82750 (15)	0.17505 (9)	0.0200 (3)
H7	0.6538	0.9250	0.2440	0.024*
C8	0.3630 (2)	0.91107 (16)	0.13603 (10)	0.0240 (3)
H8A	0.2581	0.8248	0.0631	0.036*
H8B	0.4572	1.0336	0.1253	0.036*
H8C	0.2647	0.9273	0.1951	0.036*
C9	0.6813 (2)	0.78602 (17)	0.07837 (11)	0.0261 (3)
H9A	0.7877	0.7322	0.1039	0.039*
H9B	0.7777	0.9037	0.0624	0.039*
H9C	0.5696	0.6961	0.0081	0.039*
O10	0.77591 (14)	0.71977 (11)	0.33094 (7)	0.0234 (2)
C11	0.9046 (2)	0.67696 (16)	0.41536 (9)	0.0212 (3)
H11A	0.9080	0.5522	0.3818	0.025*
H11B	0.8225	0.6720	0.4829	0.025*
C12	1.1617 (2)	0.82965 (15)	0.45327 (9)	0.0201 (3)
O13	1.29112 (15)	0.78801 (12)	0.52707 (7)	0.0264 (3)
H13	1.450 (4)	0.878 (3)	0.5439 (16)	0.054 (5)*
O14	1.24080 (14)	0.97255 (11)	0.42015 (7)	0.0237 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0217 (6)	0.0186 (5)	0.0165 (5)	0.0096 (4)	0.0040 (4)	0.0044 (4)
C2	0.0203 (6)	0.0192 (6)	0.0187 (5)	0.0078 (4)	0.0026 (4)	0.0048 (4)
C3	0.0275 (6)	0.0223 (6)	0.0224 (6)	0.0117 (5)	0.0037 (4)	0.0098 (4)
C4	0.0281 (6)	0.0179 (6)	0.0243 (6)	0.0074 (5)	0.0076 (5)	0.0073 (4)

C5	0.0206 (6)	0.0214 (6)	0.0242 (6)	0.0048 (5)	0.0037 (4)	0.0046 (4)
C6	0.0215 (6)	0.0229 (6)	0.0206 (6)	0.0096 (5)	0.0017 (4)	0.0063 (4)
C7	0.0199 (6)	0.0179 (5)	0.0203 (5)	0.0060 (4)	-0.0006 (4)	0.0064 (4)
C8	0.0256 (6)	0.0244 (6)	0.0266 (6)	0.0121 (5)	0.0049 (5)	0.0124 (5)
C9	0.0246 (6)	0.0264 (6)	0.0328 (7)	0.0116 (5)	0.0092 (5)	0.0151 (5)
O10	0.0209 (4)	0.0231 (4)	0.0252 (4)	0.0057 (3)	-0.0031 (3)	0.0121 (3)
C11	0.0250 (6)	0.0227 (6)	0.0189 (6)	0.0112 (5)	0.0012 (4)	0.0093 (4)
C12	0.0260 (6)	0.0225 (6)	0.0155 (5)	0.0135 (5)	0.0027 (4)	0.0063 (4)
O13	0.0244 (5)	0.0274 (5)	0.0279 (5)	0.0087 (4)	-0.0034 (3)	0.0141 (4)
O14	0.0249 (5)	0.0228 (5)	0.0245 (4)	0.0092 (3)	-0.0003 (3)	0.0106 (3)

Geometric parameters (Å, °)

C1—C6	1.3902 (16)	C7—H7	0.9800
C1—C2	1.4071 (15)	C8—H8A	0.9600
C1—C7	1.5182 (15)	C8—H8B	0.9600
C2—O10	1.3802 (14)	C8—H8C	0.9600
C2—C3	1.3895 (16)	C9—H9A	0.9600
C3—C4	1.3897 (17)	C9—H9B	0.9600
C3—H3	0.9300	C9—H9C	0.9600
C4—C5	1.3791 (16)	O10—C11	1.4106 (13)
C4—H4	0.9300	C11—C12	1.4999 (16)
C5—C6	1.3905 (16)	C11—H11A	0.9700
C5—H5	0.9300	C11—H11B	0.9700
C6—H6	0.9300	C12—O14	1.2187 (13)
C7—C8	1.5272 (15)	C12—O13	1.3168 (13)
C7—C9	1.5297 (16)	O13—H13	0.92 (2)
C6—C1—C2	117.03 (10)	C7—C8—H8A	109.5
C6—C1—C7	122.82 (9)	C7—C8—H8B	109.5
C2—C1—C7	120.10 (9)	H8A—C8—H8B	109.5
O10—C2—C3	123.42 (10)	C7—C8—H8C	109.5
O10—C2—C1	115.23 (9)	H8A—C8—H8C	109.5
C3—C2—C1	121.34 (10)	H8B—C8—H8C	109.5
C2—C3—C4	119.81 (10)	C7—C9—H9A	109.5
C2—C3—H3	120.1	C7—C9—H9B	109.5
C4—C3—H3	120.1	H9A—C9—H9B	109.5
C5—C4—C3	120.03 (10)	C7—C9—H9C	109.5
C5—C4—H4	120.0	H9A—C9—H9C	109.5
C3—C4—H4	120.0	H9B—C9—H9C	109.5
C4—C5—C6	119.63 (10)	C2—O10—C11	116.49 (8)
C4—C5—H5	120.2	O10—C11—C12	109.23 (9)
C6—C5—H5	120.2	O10—C11—H11A	109.8
C1—C6—C5	122.15 (10)	C12—C11—H11A	109.8
C1—C6—H6	118.9	O10—C11—H11B	109.8
C5—C6—H6	118.9	C12—C11—H11B	109.8
C1—C7—C8	113.13 (9)	H11A—C11—H11B	108.3
C1—C7—C9	109.82 (9)	O14—C12—O13	124.46 (10)

C8—C7—C9	110.53 (9)	O14—C12—C11	124.79 (10)
C1—C7—H7	107.7	O13—C12—C11	110.74 (9)
C8—C7—H7	107.7	C12—O13—H13	109.0 (11)
C9—C7—H7	107.7		
C6—C1—C2—O10	-178.81 (9)	C4—C5—C6—C1	-0.08 (17)
C7—C1—C2—O10	3.74 (15)	C6—C1—C7—C8	26.38 (14)
C6—C1—C2—C3	1.20 (16)	C2—C1—C7—C8	-156.33 (10)
C7—C1—C2—C3	-176.24 (10)	C6—C1—C7—C9	-97.63 (12)
O10—C2—C3—C4	179.62 (10)	C2—C1—C7—C9	79.66 (12)
C1—C2—C3—C4	-0.40 (17)	C3—C2—O10—C11	-4.86 (15)
C2—C3—C4—C5	-0.69 (17)	C1—C2—O10—C11	175.15 (9)
C3—C4—C5—C6	0.93 (17)	C2—O10—C11—C12	-179.75 (8)
C2—C1—C6—C5	-0.97 (16)	O10—C11—C12—O14	3.41 (15)
C7—C1—C6—C5	176.40 (10)	O10—C11—C12—O13	-176.35 (8)

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>
O13—H13 \cdots O14 ⁱ	0.92 (2)	1.73 (2)	2.6392 (11)	173.5 (17)

Symmetry code: (i) $-x+3, -y+2, -z+1$.