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2-[4-(Carboxymethyl)phenoxy]acetic acid

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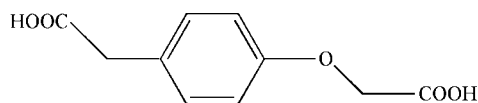
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 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.037; wR factor = 0.099; data-to-parameter ratio = 9.7.

The title compound, $\text{C}_{10}\text{H}_{10}\text{O}_5$, was obtained by the reaction of 4-hydroxyphenylacetic acid with chloroacetic acid. In the crystal, the molecules form a three-dimensional network by way of intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding.

Related literature

For applications of metal-organic frameworks with carboxylic acid ligands, see: Kuppler *et al.* (2009); Jahan *et al.* (2010); Armelao *et al.* (2010); Yashima *et al.* (2009). The title compound was obtained by the reaction of 4-hydroxyphenylacetic acid (Gracin *et al.*, 2005) and chloroacetic acid (Sandhu *et al.*, 1991).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_{10}\text{O}_5$
 $M_r = 210.18$

 Orthorhombic, $P2_12_12_1$
 $a = 5.4640$ (2) Å

 $b = 10.7798$ (5) Å

 $c = 16.2550$ (7) Å

 $V = 957.43$ (7) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.12$ mm⁻¹
 $T = 296$ K

 $0.42 \times 0.38 \times 0.31$ mm

Data collection

Bruker APEXII area-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

 $T_{\min} = 0.95$, $T_{\max} = 0.96$

11814 measured reflections

1315 independent reflections

 1162 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.099$
 $S = 1.07$

1315 reflections

136 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O2}-\text{H2}\cdots\text{O4}^{\text{i}}$	0.82	1.81	2.617 (2)	168
$\text{O5}-\text{H5}\cdots\text{O1}^{\text{ii}}$	0.82	1.85	2.666 (2)	171

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

Data collection: APEX2 (Bruker, 2006); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2074).

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supporting information

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2-[4-(Carboxymethyl)phenoxy]acetic acid

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S1. Comment

Various aromatic carboxylic acid ligands are being used in designing versatile metal-organic frameworks (MOFs) having potential applications (Kuppler *et al.*, 2009; Jahan *et al.*, 2010; Armelao *et al.*, 2010; Yashima *et al.*, 2009). In present work, we report the synthesis and single crystal structure of a new aromatic dicarboxylic acid ligand namely 4-carboxymethoxy phenylacetic acid (I) containing two flexible carboxylic acid groups, obtained by the reaction of 4-hydroxyphenylacetic (Gracin *et al.*, 2005) and chloroacetic acid (Sandhu *et al.*, 1991).

The molecular structure of the title compound is presented in Fig.1. The carboxymethoxy group is almost parallel to the benzene ring with a dihedral angle of $11.4 (1)^\circ$ between the two least-squares planes, while the acetic acid unit is almost perpendicular to the benzene ring, making a dihedral angle of $72.4 (2)^\circ$. In addition, the molecules were linked by the intermolecular O—H \cdots O hydrogen bonding forming a three-dimensional network (Fig.2).

S2. Experimental

The solution of 4-hydroxyphenylacetic acid (1.522 g, 10.0 mmol) in water (10 ml) neutralized with NaOH (0.8 g, 20 mmol) was added to a 1:1 mixture of chloroacetic acid (3.78 g, 40 mmol) and NaOH (1.600 g, 40 mmol) with stirring to adjust the pH value of the mixture to *ca* 11 and refluxed at 90 °C for 3 h. The pH value was adjusted to 2–3 with concentrated hydrochloric acid. While cooling the mixture to room temperature, a white precipitate was appeared rapidly. The solid was filtrated, washed by water. Single crystals suitable for X-ray diffraction were obtained in the mother liquid after evaporation within a few days.

S3. Refinement

The Flack parameter could not be refined to an acceptable value as the title compound is a poor anomalous scatterer even after using MOVE command in Shelxl97. Therefore, the Friedel opposites were merged (using MERG 3 command in Shelxl97) and Flack parameter is not reported. H-atoms were positioned geometrically and included in the refinement using a riding-model approximation [aromatic C–H = 0.93 Å, aliphatic C–H = 0.97 Å, O–H = 0.82 Å] with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{O})$.

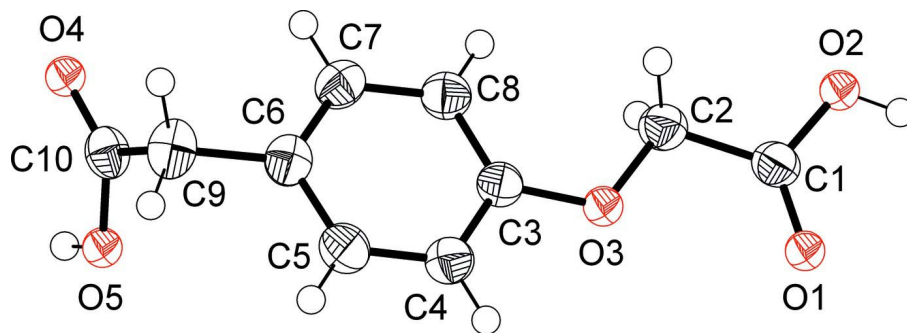


Figure 1

View of the molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

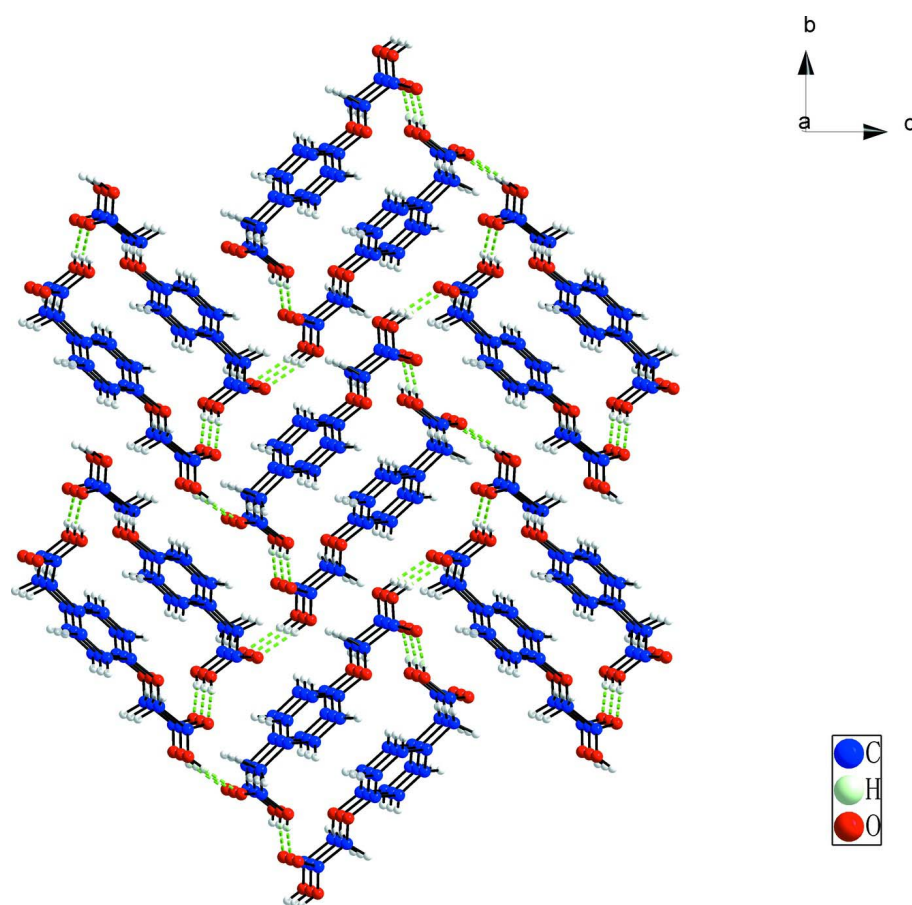


Figure 2

A packing diagram of (I), the hydrogen bonds are depicted as green dashed lines.

2-[4-(Carboxymethyl)phenoxy]acetic acid

Crystal data

$C_{10}H_{10}O_5$

$M_r = 210.18$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 5.4640 (2) \text{ \AA}$

$b = 10.7798 (5) \text{ \AA}$

$c = 16.2550 (7) \text{ \AA}$
 $V = 957.43 (7) \text{ \AA}^3$
 $Z = 4$
 $F(000) = 440$
 $D_x = 1.458 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 6173 reflections
 $\theta = 2.3\text{--}27.7^\circ$
 $\mu = 0.12 \text{ mm}^{-1}$
 $T = 296 \text{ K}$
 Block, colourless
 $0.42 \times 0.38 \times 0.31 \text{ mm}$

Data collection

Bruker APEXII area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 phi and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.95, T_{\max} = 0.96$

11814 measured reflections
 1315 independent reflections
 1162 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$
 $\theta_{\max} = 27.7^\circ, \theta_{\min} = 2.3^\circ$
 $h = -7 \rightarrow 7$
 $k = -13 \rightarrow 14$
 $l = -20 \rightarrow 21$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.099$
 $S = 1.07$
 1315 reflections
 136 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.2342P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.14 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e \AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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	0.4626 (4)	0.67278 (16)	0.64905 (10)	0.0651 (5)
O2	0.7484 (4)	0.79234 (17)	0.59564 (10)	0.0645 (5)
H2	0.7132	0.8367	0.6349	0.077*
O3	0.4929 (3)	0.51339 (14)	0.52607 (9)	0.0489 (4)
O4	0.8002 (3)	0.06923 (15)	0.22690 (11)	0.0536 (4)
O5	0.6100 (4)	-0.00299 (16)	0.33749 (9)	0.0589 (5)
H5	0.7257	-0.0515	0.3374	0.071*
C1	0.6127 (4)	0.6927 (2)	0.59700 (12)	0.0440 (5)
C2	0.6695 (4)	0.6081 (2)	0.52593 (12)	0.0443 (5)
H2A	0.6632	0.6538	0.4746	0.053*

H2B	0.8320	0.5731	0.5320	0.053*
C3	0.4856 (4)	0.43389 (19)	0.46020 (12)	0.0401 (4)
C4	0.2906 (4)	0.3526 (2)	0.45967 (13)	0.0449 (5)
H4A	0.1741	0.3559	0.5013	0.054*
C5	0.2681 (4)	0.2661 (2)	0.39733 (13)	0.0467 (5)
H5A	0.1357	0.2119	0.3972	0.056*
C6	0.4405 (4)	0.2592 (2)	0.33498 (12)	0.0427 (5)
C7	0.6354 (4)	0.3416 (2)	0.33640 (13)	0.0457 (5)
H7A	0.7520	0.3381	0.2948	0.055*
C8	0.6606 (4)	0.4294 (2)	0.39878 (12)	0.0445 (5)
H8A	0.7925	0.4839	0.3991	0.053*
C9	0.4187 (4)	0.1613 (2)	0.26811 (13)	0.0502 (5)
H9A	0.2651	0.1171	0.2741	0.060*
H9B	0.4198	0.2009	0.2145	0.060*
C10	0.6279 (4)	0.07182 (19)	0.27412 (13)	0.0431 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0757 (12)	0.0593 (11)	0.0602 (9)	-0.0208 (10)	0.0218 (10)	-0.0149 (8)
O2	0.0736 (12)	0.0572 (10)	0.0627 (10)	-0.0251 (10)	0.0144 (10)	-0.0129 (8)
O3	0.0598 (9)	0.0426 (8)	0.0444 (7)	-0.0102 (8)	0.0096 (8)	-0.0050 (6)
O4	0.0479 (8)	0.0490 (9)	0.0639 (9)	0.0009 (8)	0.0033 (8)	0.0069 (8)
O5	0.0625 (10)	0.0639 (10)	0.0503 (8)	0.0177 (10)	0.0026 (8)	0.0099 (8)
C1	0.0475 (11)	0.0409 (10)	0.0437 (10)	-0.0063 (10)	-0.0009 (10)	0.0025 (9)
C2	0.0446 (11)	0.0461 (11)	0.0423 (10)	-0.0056 (10)	0.0009 (10)	0.0003 (9)
C3	0.0448 (10)	0.0376 (9)	0.0378 (9)	0.0010 (9)	0.0000 (9)	0.0026 (8)
C4	0.0447 (11)	0.0452 (11)	0.0448 (10)	-0.0008 (10)	0.0034 (10)	0.0012 (9)
C5	0.0435 (11)	0.0452 (11)	0.0515 (11)	-0.0016 (10)	-0.0029 (10)	0.0017 (9)
C6	0.0436 (11)	0.0428 (10)	0.0416 (9)	0.0092 (10)	-0.0067 (9)	0.0012 (8)
C7	0.0436 (11)	0.0512 (12)	0.0422 (10)	0.0051 (10)	0.0030 (9)	0.0011 (9)
C8	0.0412 (10)	0.0465 (11)	0.0457 (10)	-0.0023 (10)	0.0002 (9)	0.0018 (9)
C9	0.0493 (12)	0.0541 (12)	0.0473 (11)	0.0129 (11)	-0.0122 (10)	-0.0060 (10)
C10	0.0455 (11)	0.0389 (10)	0.0451 (10)	0.0010 (10)	-0.0084 (10)	-0.0040 (9)

Geometric parameters (Å, °)

O1—C1	1.198 (3)	C4—C5	1.383 (3)
O2—C1	1.305 (3)	C4—H4A	0.9300
O2—H2	0.8200	C5—C6	1.386 (3)
O3—C3	1.372 (2)	C5—H5A	0.9300
O3—C2	1.405 (3)	C6—C7	1.387 (3)
O4—C10	1.215 (3)	C6—C9	1.519 (3)
O5—C10	1.312 (3)	C7—C8	1.394 (3)
O5—H5	0.8200	C7—H7A	0.9300
C1—C2	1.504 (3)	C8—H8A	0.9300
C2—H2A	0.9700	C9—C10	1.499 (3)
C2—H2B	0.9700	C9—H9A	0.9700

C3—C4	1.380 (3)	C9—H9B	0.9700
C3—C8	1.383 (3)		
C1—O2—H2	109.5	C6—C5—H5A	119.6
C3—O3—C2	118.19 (16)	C5—C6—C7	118.39 (19)
C10—O5—H5	109.5	C5—C6—C9	120.5 (2)
O1—C1—O2	123.3 (2)	C7—C6—C9	121.11 (19)
O1—C1—C2	125.1 (2)	C6—C7—C8	121.5 (2)
O2—C1—C2	111.64 (18)	C6—C7—H7A	119.2
O3—C2—C1	107.31 (17)	C8—C7—H7A	119.2
O3—C2—H2A	110.3	C3—C8—C7	118.7 (2)
C1—C2—H2A	110.3	C3—C8—H8A	120.6
O3—C2—H2B	110.3	C7—C8—H8A	120.6
C1—C2—H2B	110.3	C10—C9—C6	109.89 (17)
H2A—C2—H2B	108.5	C10—C9—H9A	109.7
O3—C3—C4	115.09 (18)	C6—C9—H9A	109.7
O3—C3—C8	124.41 (19)	C10—C9—H9B	109.7
C4—C3—C8	120.47 (19)	C6—C9—H9B	109.7
C3—C4—C5	120.1 (2)	H9A—C9—H9B	108.2
C3—C4—H4A	119.9	O4—C10—O5	122.6 (2)
C5—C4—H4A	119.9	O4—C10—C9	124.4 (2)
C4—C5—C6	120.8 (2)	O5—C10—C9	112.97 (19)
C4—C5—H5A	119.6		
C3—O3—C2—C1	171.19 (18)	C5—C6—C7—C8	0.3 (3)
O1—C1—C2—O3	8.2 (3)	C9—C6—C7—C8	-178.2 (2)
O2—C1—C2—O3	-171.83 (18)	O3—C3—C8—C7	178.09 (19)
C2—O3—C3—C4	-172.77 (18)	C4—C3—C8—C7	0.2 (3)
C2—O3—C3—C8	9.3 (3)	C6—C7—C8—C3	-0.2 (3)
O3—C3—C4—C5	-178.35 (19)	C5—C6—C9—C10	-114.2 (2)
C8—C3—C4—C5	-0.3 (3)	C7—C6—C9—C10	64.2 (3)
C3—C4—C5—C6	0.4 (3)	C6—C9—C10—O4	-106.1 (2)
C4—C5—C6—C7	-0.3 (3)	C6—C9—C10—O5	72.4 (2)
C4—C5—C6—C9	178.15 (19)		

Hydrogen-bond geometry (\AA , $^\circ$)

<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 O4 ⁱ	0.82	1.81	2.617 (2)	168
O5—H5 \cdots O1 ⁱⁱ	0.82	1.85	2.666 (2)	171

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