1,4-Phenylenediacetic acid

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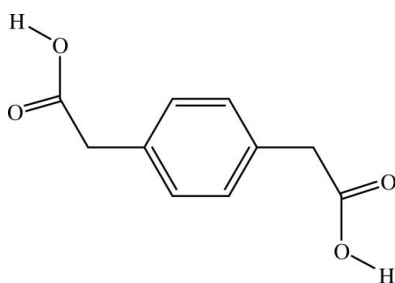Correspondence e-mail: hkfun@usm.my, suchada.c@psu.ac.th

The title molecule, $C_{10}H_{10}O_4$, possesses a crystallographically imposed centre of symmetry. The acetic acid unit is almost perpendicular to the benzene ring, making a dihedral angle of 84.46 (6)°. In the crystal structure, molecules are linked by intermolecular O—H···O hydrogen bonds into chains running along the [101] direction, and these chains are stacked along the b axis.

Related literature

For general background on molecular recognition and supramolecular chemistry, see: Desiraju (2003); Garcia-Tellado *et al.* (1991); Lehn (1995); Leiserowitz (1976); Goswami *et al.* (2000, 2001, 2005, 2006). For standard values of bond lengths, see: Allen *et al.* (1987).

Experimental

Crystal data

$C_{10}H_{10}O_4$	$V = 442.57$ (3) Å³
$M_r = 194.18$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.2747$ (4) Å	$\mu = 0.11$ mm⁻¹
$b = 4.7218$ (2) Å	$T = 100.0$ (1) K
$c = 10.1686$ (4) Å	$0.56 \times 0.23 \times 0.04$ mm
$\beta = 116.220$ (2)°	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	6558 measured reflections
	1293 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	1047 reflections with $I > 2\sigma(I)$
$T_{min} = 0.939$, $T_{max} = 0.995$	$R_{int} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$	84 parameters
$wR(F^2) = 0.107$	All H-atom parameters refined
$S = 1.10$	$\Delta\rho_{max} = 0.32$ e Å⁻³
1293 reflections	$\Delta\rho_{min} = -0.18$ e Å⁻³

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1O2···O1[i]	0.908 (19)	1.771 (19)	2.6766 (12)	175 (2)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2261).

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supplementary materials

1,4-Phenylenediacetic acid

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Comment

The recognition of dicarboxylic acids (Leiserowitz, 1976; Garcia-Tellado *et al.*, 1991) plays a very important role in the field of molecular recognition and supramolecular chemistry due to its nice donor-acceptor arrays. We have used 1,4-phenylene-diacetic acid (the title compound) in our continuing work for the recognition of dicarboxylic acids by designed synthetic receptors (Goswami *et al.*, 2000; 2001). It has produced both *syn-syn* and anti-anti polymeric hydrogen bonded complexes with pyridine amide based receptors in solid phase (Goswami *et al.*, 2005; 2006). Here we disclose the arrangement of the 1,4-phenylenediacetic acid itself in solid phase, which will help us in designing new hydrogen bonding motifs in the field of supramolecular chemistry as well as crystal engineering (Lehn, 1995; Desiraju, 2003).

Molecules of the title compound, lie across crystallographic inversion centres and the asymmetric unit therefore contain one-half of a molecule (Fig. 1). The orientation of acetic acid [C4/C5/O1/O2] with respect to the benzene ring, shown by the torsion angle C1/C2/C4/C5 = 95.59 (14)°, indicates a (+)-anticlinal conformation. The dihedral angle between the mean plane of acetic acid and benzene ring is 84.46 (6)°. All bond lengths and angles are in normal values (Allen *et al.*, 1987).

In the crystal packing in Fig. 2, the molecules are linked by O—H⋯O intermolecular hydrogen bonds (Table 1) into chains along the [1 0 1] direction and these chains are stacked along the *b* axis.

Experimental

Commercially available 1,4-phenylenediacetic acid (19.42 mg, 0.1 mmol) was dissolved in methanol–chloroform (*v/v* 2:1). Single crystals were grown by slow evaporation of the solvent.

Refinement

All H atoms were located in a difference map and isotropically refined.
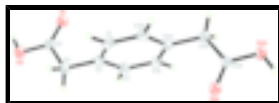
Figures

Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. [Symmetry code: (A) 2 − *x*, −*y*, 1 − *z*].
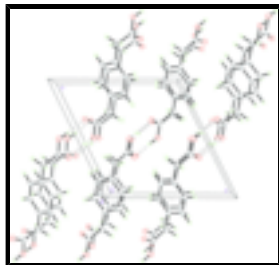
Fig. 2. The crystal packing of (I), viewed along the *b* axis. Hydrogen bonds were shown as dash lines.

1,4-phenylenediacetic acid

Crystal data

$C_{10}H_{10}O_4$	$F_{000} = 204$
$M_r = 194.18$	$D_x = 1.457$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1293 reflections
$a = 10.2747$ (4) Å	$\theta = 4.0–30.0º$
$b = 4.7218$ (2) Å	$\mu = 0.11$ mm^{-1}
$c = 10.1686$ (4) Å	$T = 100.0$ (1) K
$\beta = 116.220$ (2)º	Slab, colourless
$V = 442.57$ (3) Å^3	$0.56 \times 0.23 \times 0.04$ mm
$Z = 2$	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	1293 independent reflections
Radiation source: fine-focus sealed tube	1047 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 30.0º$
$T = 100.0(1)$ K	$\theta_{min} = 4.0º$
ω scans	$h = -14\rightarrow14$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -6\rightarrow6$
$T_{min} = 0.939$, $T_{max} = 0.995$	$l = -14\rightarrow14$
6558 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	All H-atom parameters refined
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 0.1424P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$

1293 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
84 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The data were collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.66050 (9)	0.05389 (19)	0.13965 (9)	0.0257 (2)
O2	0.50586 (9)	−0.29448 (19)	0.11918 (10)	0.0257 (2)
C1	0.88216 (13)	0.0593 (3)	0.52686 (13)	0.0225 (3)
C2	0.86705 (12)	−0.1392 (2)	0.42009 (12)	0.0206 (3)
C3	0.98642 (13)	−0.1978 (3)	0.39426 (13)	0.0231 (3)
C4	0.72489 (14)	−0.2887 (3)	0.33517 (14)	0.0253 (3)
C5	0.62940 (12)	−0.1559 (2)	0.18932 (13)	0.0203 (3)
H1	0.7984 (17)	0.095 (3)	0.5453 (17)	0.028 (4)*
H3	0.9756 (16)	−0.336 (3)	0.3220 (18)	0.026 (4)*
H4A	0.6654 (18)	−0.288 (4)	0.3894 (19)	0.037 (5)*
H4B	0.7381 (19)	−0.477 (4)	0.3108 (19)	0.035 (4)*
H1O2	0.453 (2)	−0.204 (4)	0.033 (2)	0.052 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0212 (4)	0.0278 (5)	0.0206 (4)	−0.0035 (3)	0.0024 (3)	0.0053 (3)
O2	0.0207 (4)	0.0271 (5)	0.0201 (4)	−0.0046 (3)	0.0007 (3)	0.0026 (3)
C1	0.0197 (5)	0.0265 (6)	0.0191 (6)	0.0034 (4)	0.0066 (5)	0.0024 (4)
C2	0.0190 (5)	0.0212 (5)	0.0149 (5)	0.0002 (4)	0.0015 (4)	0.0041 (4)
C3	0.0254 (6)	0.0227 (6)	0.0167 (5)	0.0018 (4)	0.0052 (5)	−0.0014 (4)
C4	0.0228 (6)	0.0251 (6)	0.0195 (6)	−0.0040 (4)	0.0017 (5)	0.0043 (4)
C5	0.0188 (5)	0.0219 (6)	0.0174 (5)	0.0001 (4)	0.0055 (4)	−0.0012 (4)

Geometric parameters (Å, °)

O1—C5	1.2169 (14)	C2—C4	1.5043 (16)

O2—C5	1.3222 (14)	C3—C1^i	1.3919 (17)
O2—H1O2	0.91 (2)	C3—H3	0.952 (17)
C1—C2	1.3901 (17)	C4—C5	1.5065 (17)
C1—C3^i	1.3919 (17)	C4—H4A	0.988 (18)
C1—H1	0.973 (15)	C4—H4B	0.950 (18)
C2—C3	1.3909 (18)		
C5—O2—H1O2	107.9 (13)	C2—C4—C5	114.50 (10)
C2—C1—C3^i	120.80 (11)	C2—C4—H4A	111.2 (10)
C2—C1—H1	117.7 (9)	C5—C4—H4A	104.7 (10)
C3^i—C1—H1	121.5 (9)	C2—C4—H4B	112.0 (11)
C1—C2—C3	118.57 (10)	C5—C4—H4B	103.9 (11)
C1—C2—C4	120.65 (11)	H4A—C4—H4B	110.1 (14)
C3—C2—C4	120.78 (11)	O1—C5—O2	122.99 (11)
C2—C3—C1^i	120.64 (11)	O1—C5—C4	124.83 (10)
C2—C3—H3	118.0 (9)	O2—C5—C4	112.17 (10)
C1^i—C3—H3	121.3 (9)		
C3^i—C1—C2—C3	0.36 (19)	C1—C2—C4—C5	95.59 (14)
C3^i—C1—C2—C4	179.93 (11)	C3—C2—C4—C5	−84.85 (15)
C1—C2—C3—C1^i	−0.36 (19)	C2—C4—C5—O1	0.34 (19)
C4—C2—C3—C1^i	−179.93 (11)	C2—C4—C5—O2	−179.89 (11)

Symmetry codes: (i) −*x*+2, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1O2···O1^{ii}	0.908 (19)	1.771 (19)	2.6766 (12)	175 (2)

Symmetry codes: (ii) −*x*+1, −*y*, −*z*.

Fig. 1

Fig. 2