

Biphenyl-3,3'-dicarboxylic acid

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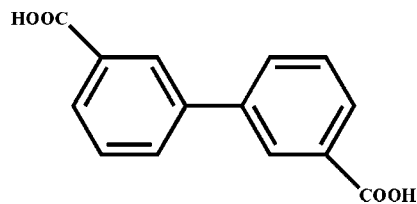
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.052; wR factor = 0.155; data-to-parameter ratio = 15.5.

The asymmetric unit of the title compound, $\text{C}_{14}\text{H}_{10}\text{O}_4$, contains one half molecule, the complete molecule being generated by a twofold axis. The two benzene rings form a dihedral angle of $43.11(5)^\circ$. Intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into one-dimensional zigzag chains. These chains are further connected into two-dimensional supramolecular layers by weak $\pi-\pi$ stacking interactions between neighbouring benzene rings, with centroid-centroid distances of $3.7648(8)$ Å.

Related literature

For general background non-covalent intermolecular interactions, see: Etter *et al.* (1990); Desiraju (2003); Yaghi *et al.* (2003); Li *et al.* (2010). For the structures of related complexes, see: Wang *et al.* (2005); Zhu (2010).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{10}\text{O}_4$
 $M_r = 242.22$
 Monoclinic, $P2_1/n$

$a = 6.6123(9)$ Å
 $b = 3.7648(8)$ Å
 $c = 22.554(3)$ Å

$\beta = 93.14(2)^\circ$
 $V = 560.61(15)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.11$ mm⁻¹
 $T = 296$ K
 $0.21 \times 0.18 \times 0.13$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.978$, $T_{\max} = 0.986$

5212 measured reflections
 1286 independent reflections
 1006 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.107$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.155$
 $S = 1.04$
 1286 reflections

83 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.24$ e Å⁻³
 $\Delta\rho_{\min} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^i$	0.82	1.82	2.6268 (17)	169

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

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2097).

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supplementary materials

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Comment

Non-covalent intermolecular interactions, mainly hydrogen bonding and aromatic stacking, play the key role to perfectly project and regulate the detailed crystal packing of supramolecular materials (Desiraju, 2003). Aromatic carboxylates have also been proved to be effective building blocks in constructing various architectures (Yaghi *et al.*, 2003; Li *et al.*, 2010; Wang *et al.*, 2005; Zhu, 2010). Recently, we obtained the title compound under hydrothermal conditions and we report its crystal structure here.

The asymmetric unit of the title compound, $C_{14}H_{10}O_4$, contains one-half molecule, the complete molecule being generated by a two-fold axis (Fig. 1). The two benzene rings form a dihedral angle of $43.11(5)^\circ$. The carboxylic acid groups form the classic cyclic $R_2^2(8)$ hydrogen-bond motif (Etter *et al.*, 1990) with other acid groups of neighbouring molecules (Table 1). These interactions result into one-dimensional zigzag chains (Fig. 2). The chains are further connected into two-dimensional supramolecular layers by weak π - π stacking interactions between neighbouring benzene rings, with centroid-centroid distances of $3.7648(8) \text{ \AA}$.

Experimental

A mixture of 3,3'-biphenyldicarboxylic acid (0.0242 g, 0.1 mmol), $Pb(CH_3COO)_2$ (0.0379 g, 0.1 mmol), water (8 ml) was stirred vigorously for 30 min and then sealed in a Teflon-lined stainless-steel autoclave. The autoclave was heated and maintained at 413 K for 3 days, and then cooled to room temperature at 5 K h^{-1} to obtain colorless prism crystals suitable for X-ray analysis.

Refinement

All H atoms were positioned geometrically ($C-H = 0.93 \text{ \AA}$ and $O-H = 0.82 \text{ \AA}$) and allowed to ride on their parent atoms, with $U_{iso}(H) = 1.2 U_{eq}(C)$ or $U_{iso}(H) = 1.5 U_{eq}(O)$.

Figures

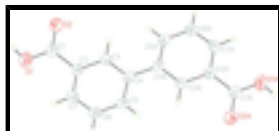


Fig. 1. The molecular structure of the title compound, with atom labels and 50% probability displacement ellipsoids for non-H atoms [symmetry code (A): $-x + 3/2, y, -z + 1/2$].

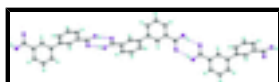


Fig. 2. View of the one-dimensional chains connected by cyclic $R_2^2(8)$ hydrogen-bonds.



Fig. 3. View of the two-dimensional layers formed by weak π - π stacking interactions between neighbouring benzene rings.

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Crystal data

$C_{14}H_{10}O_4$	$F(000) = 252$
$M_r = 242.22$	$D_x = 1.435 \text{ Mg m}^{-3}$
Monoclinic, $P2/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: $-P 2yac$	Cell parameters from 1286 reflections
$a = 6.6123 (9) \text{ \AA}$	$\theta = 3.2\text{--}27.6^\circ$
$b = 3.7648 (8) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 22.554 (3) \text{ \AA}$	$T = 296 \text{ K}$
$\beta = 93.14 (2)^\circ$	Prism, colourless
$V = 560.61 (15) \text{ \AA}^3$	$0.21 \times 0.18 \times 0.13 \text{ mm}$
$Z = 2$	

Data collection

Bruker SMART CCD diffractometer	1286 independent reflections
Radiation source: fine-focus sealed tube graphite	1006 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.107$
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1996)	$\theta_{\text{max}} = 27.6^\circ$, $\theta_{\text{min}} = 3.2^\circ$
$T_{\text{min}} = 0.978$, $T_{\text{max}} = 0.986$	$h = -8 \rightarrow 8$
5212 measured reflections	$k = -4 \rightarrow 4$
	$l = -29 \rightarrow 28$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.052$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.155$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0843P)^2]$
1286 reflections	where $P = (F_o^2 + 2F_c^2)/3$
83 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.73331 (19)	0.2984 (4)	0.48869 (5)	0.0617 (5)
H1	0.6601	0.3923	0.5124	0.093*
C2	0.7741 (2)	0.2032 (4)	0.38631 (6)	0.0340 (4)
O2	0.48155 (17)	0.4652 (4)	0.42503 (5)	0.0527 (4)
C3	0.6866 (2)	0.2063 (4)	0.32849 (6)	0.0323 (4)
H3	0.5542	0.2860	0.3216	0.039*
C4	0.79643 (19)	0.0906 (4)	0.28086 (6)	0.0321 (4)
C1	0.6531 (2)	0.3318 (4)	0.43585 (6)	0.0373 (4)
C5	0.9952 (2)	-0.0282 (4)	0.29269 (7)	0.0384 (4)
H5	1.0701	-0.1063	0.2615	0.046*
C7	0.9722 (2)	0.0838 (4)	0.39736 (7)	0.0402 (4)
H7	1.0301	0.0811	0.4359	0.048*
C6	1.0825 (2)	-0.0316 (4)	0.35005 (8)	0.0421 (4)
H6	1.2149	-0.1110	0.3570	0.050*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0573 (8)	0.1005 (11)	0.0274 (6)	0.0284 (7)	0.0010 (5)	-0.0027 (6)
C2	0.0336 (7)	0.0401 (7)	0.0285 (7)	0.0010 (6)	0.0038 (5)	0.0021 (6)
O2	0.0415 (7)	0.0832 (9)	0.0336 (6)	0.0178 (6)	0.0024 (5)	-0.0019 (5)
C3	0.0270 (7)	0.0397 (7)	0.0305 (7)	0.0014 (5)	0.0030 (5)	0.0024 (5)
C4	0.0297 (7)	0.0374 (7)	0.0295 (8)	-0.0018 (5)	0.0033 (5)	0.0013 (5)
C1	0.0352 (8)	0.0475 (8)	0.0291 (7)	0.0020 (6)	0.0012 (5)	0.0009 (6)
C5	0.0294 (7)	0.0501 (9)	0.0363 (8)	0.0027 (6)	0.0071 (6)	-0.0010 (6)
C7	0.0350 (8)	0.0520 (9)	0.0332 (8)	0.0019 (6)	-0.0028 (5)	0.0037 (6)
C6	0.0272 (7)	0.0571 (9)	0.0418 (9)	0.0075 (6)	0.0014 (6)	0.0046 (7)

Geometric parameters (\AA , $^\circ$)

O1—C1	1.2837 (17)	C4—C5	1.400 (2)
O1—H1	0.8200	C4—C4 ⁱ	1.490 (3)

supplementary materials

C2—C7	1.394 (2)	C5—C6	1.388 (2)
C2—C3	1.3975 (19)	C5—H5	0.9300
C2—C1	1.490 (2)	C7—C6	1.394 (2)
O2—C1	1.2520 (18)	C7—H7	0.9300
C3—C4	1.399 (2)	C6—H6	0.9300
C3—H3	0.9300		
C1—O1—H1	109.5	O2—C1—C2	120.13 (13)
C7—C2—C3	120.43 (13)	O1—C1—C2	116.90 (12)
C7—C2—C1	120.56 (13)	C6—C5—C4	121.26 (13)
C3—C2—C1	119.01 (12)	C6—C5—H5	119.4
C2—C3—C4	120.53 (12)	C4—C5—H5	119.4
C2—C3—H3	119.7	C2—C7—C6	119.28 (14)
C4—C3—H3	119.7	C2—C7—H7	120.4
C3—C4—C5	118.33 (13)	C6—C7—H7	120.4
C3—C4—C4 ⁱ	120.83 (14)	C5—C6—C7	120.16 (13)
C5—C4—C4 ⁱ	120.83 (14)	C5—C6—H6	119.9
O2—C1—O1	122.97 (13)	C7—C6—H6	119.9
C7—C2—C3—C4	-0.1 (2)	C3—C2—C1—O1	174.69 (14)
C1—C2—C3—C4	179.39 (13)	C3—C4—C5—C6	-0.1 (2)
C2—C3—C4—C5	0.1 (2)	C4 ⁱ —C4—C5—C6	-179.54 (11)
C2—C3—C4—C4 ⁱ	179.53 (10)	C3—C2—C7—C6	0.2 (2)
C7—C2—C1—O2	174.22 (14)	C1—C2—C7—C6	-179.33 (14)
C3—C2—C1—O2	-5.3 (2)	C4—C5—C6—C7	0.2 (2)
C7—C2—C1—O1	-5.8 (2)	C2—C7—C6—C5	-0.2 (2)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 ⁱⁱ —O2 ⁱⁱ	0.82	1.82	2.6268 (17)	169.

Symmetry codes: (ii) $-x+1, -y+1, -z+1$.

Fig. 1

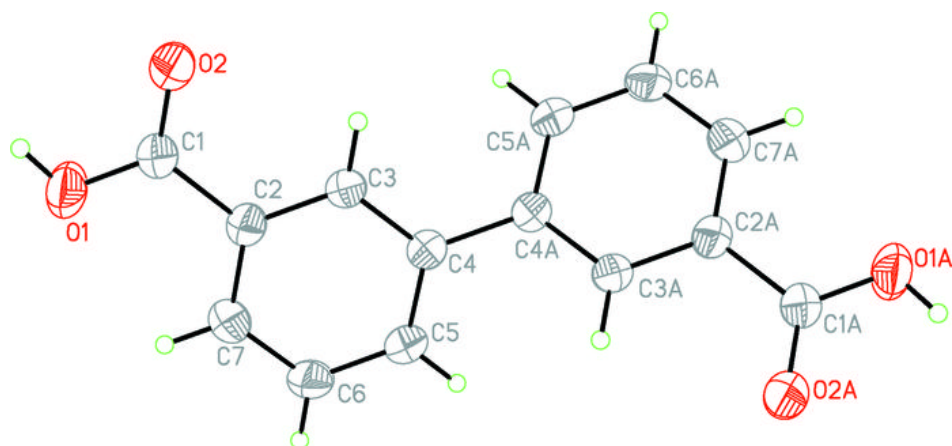


Fig. 2



Fig. 3

