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Di-*tert*-butyl 2,2′-[(biphenyl-4,4′-diyl)-dioxy]diacetate

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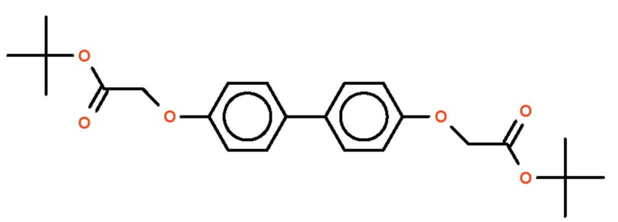
Key indicators: single-crystal X-ray study; *T* = 293 K; mean σ(C–C) = 0.002 Å; *R* factor = 0.041; *wR* factor = 0.120; data-to-parameter ratio = 18.9.

The complete molecule of the title compound, $C_{24}H_{30}O_6$, is generated by a crystallographic inversion centre. In the unique part of the molecule, the four-atom –O–CH_2–C(= O)–O– chain between the benzene ring and the *tert*-butyl group assumes a zigzag conformation [O—C—C—O torsion angle = −162.3 (1)°].

Related literature

For a related structure, see: Shah *et al.* (2010).



Experimental

Crystal data

$C_{24}H_{30}O_6$	V = 1122.88 (13) Å^3
M_r = 414.48	Z = 2
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 9.9390 (7) Å	μ = 0.09 mm^{-1}
b = 12.6247 (8) Å	T = 293 K
c = 9.8458 (7) Å	0.4 × 0.3 × 0.2 mm
β = 114.645 (1)°	

Data collection

Bruker SMART APEX diffractometer	2572 independent reflections
7450 measured reflections	2029 reflections with $I > 2\sigma(I)$
	R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041	136 parameters
$wR(F^2)$ = 0.120	H-atom parameters constrained
S = 1.02	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
2572 reflections	$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5072).

References

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supplementary materials

Acta Cryst. (2010). E**66**, o1739 [doi:10.1107/S1600536810023391]

Di-*tert*-butyl 2,2'-[(biphenyl-4,4'-diyl)dioxy]diacetate

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Comment

We are interested in the solid-state structures of *V*-shaped molecules; recently we reported the crystal structure of 9,9-bis[4-(*tert*-butoxycarbonylmethyloxy)phenyl]fluorene (Shah *et al.*, 2010). For such a shape, the number of carbon atoms making up the kink must be an odd number. In the present compound, the two aromatic rings are directly connected; the molecule lies on a center of symmetry (Fig. 1). The four-atom –O–CH_2–C(═O)–O– chain between the aromatic ring and the *tert*-butyl group assumes a zigzag conformation [O–C–C–O torsion angle 162.3 (1) °].

Experimental

4,4'-Dihydroxybiphenyl (1 g, 2.4 mmol) was dissolved in acetone (25 ml). To the solution was added potassium carbonate (0.67 g, 4.8 mmol) and *t*-butyl bromoacetate (0.75 ml, 4.8 mmol). The mixture was stirred at room temperature for 3 h. The solvent was evaporated under reduced pressure and the residue was dissolved in a mixture of water (50 ml) and dichloromethane (50 ml). The aqueous layer was extracted three times with dichloromethane. The combined organic phases were evaporated under reduced pressure and the solid material was recrystallized from *n*-hexane.

Refinement

H-atoms were placed in calculated positions [C–H 0.93–0.97 Å, U(H) 1.2–1.5U(C)] and were included in the refinement in the riding model approximation.

Figures

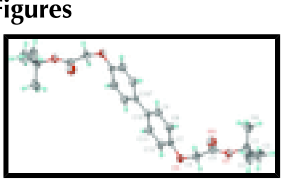

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{24}H_{30}O_6$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. Symmetry code: (i) = 1 - *x*, 1 - *y*, 1 - *z*.

Di-*tert*-butyl 2,2'-[(biphenyl-4,4'-diyl)dioxy]diacetate

Crystal data

$C_{24}H_{30}O_6$	$F(000) = 444$
$M_r = 414.48$	$D_x = 1.226$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2683 reflections
$a = 9.9390$ (7) Å	$\theta = 2.8–28.4°$
$b = 12.6247$ (8) Å	$\mu = 0.09$ mm^{-1}

c = 9.8458 (7) Å	T = 293 K
β = 114.645 (1)°	Block, colorless
V = 1122.88 (13) Å^3	0.4 × 0.3 × 0.2 mm
Z = 2	

Data collection

Bruker SMART APEX diffractometer	2029 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.024
graphite	θ_{max} = 27.5°, θ_{min} = 2.3°
ω scans	$h = -9\rightarrow12$
7450 measured reflections	$k = -15\rightarrow16$
2572 independent reflections	$l = -12\rightarrow12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.041	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.120	H-atom parameters constrained
S = 1.02	$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.1713P]$ where $P = (F_o^2 + 2F_c^2)/3$
2572 reflections	$(\Delta/\sigma)_{max}$ = 0.001
136 parameters	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.16964 (10)	1.03985 (7)	0.42919 (10)	0.0453 (2)
O2	0.24672 (13)	0.91302 (9)	0.31464 (12)	0.0643 (3)
O3	0.49943 (10)	0.88751 (7)	0.57432 (12)	0.0538 (3)
C1	0.03883 (16)	1.07454 (11)	0.29402 (15)	0.0507 (4)
C2	0.0877 (2)	1.11440 (17)	0.1761 (2)	0.0784 (5)
H2A	0.1256	1.0563	0.1397	0.118*
H2B	0.1636	1.1669	0.2193	0.118*
H2C	0.0047	1.1452	0.0949	0.118*
C3	−0.0216 (2)	1.16473 (14)	0.3541 (2)	0.0726 (5)
H3A	−0.0548	1.1376	0.4260	0.109*
H3B	−0.1032	1.1973	0.2733	0.109*
H3C	0.0548	1.2163	0.4010	0.109*
C4	−0.07095 (18)	0.98421 (15)	0.2398 (2)	0.0708 (5)
H4A	−0.0985	0.9629	0.3184	0.106*
H4B	−0.0266	0.9255	0.2119	0.106*
H4C	−0.1574	1.0070	0.1549	0.106*

C5	0.25888 (15)	0.96225 (10)	0.42366 (15)	0.0434 (3)
C6	0.37853 (15)	0.94408 (10)	0.57882 (16)	0.0474 (3)
H6A	0.4129	1.0118	0.6274	0.057*
H6B	0.3377	0.9046	0.6374	0.057*
C7	0.48857 (14)	0.77893 (10)	0.55146 (14)	0.0430 (3)
C8	0.40501 (16)	0.71184 (11)	0.59620 (17)	0.0501 (3)
H8	0.3454	0.7391	0.6398	0.060*
C9	0.41070 (15)	0.60337 (10)	0.57559 (15)	0.0465 (3)
H9	0.3543	0.5589	0.6066	0.056*
C10	0.49740 (13)	0.55858 (9)	0.51049 (13)	0.0372 (3)
C11	0.58025 (16)	0.62869 (11)	0.46692 (16)	0.0485 (3)
H11	0.6399	0.6020	0.4230	0.058*
C12	0.57629 (16)	0.73697 (11)	0.48722 (17)	0.0513 (4)
H12	0.6332	0.7817	0.4573	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0429 (5)	0.0464 (5)	0.0447 (5)	0.0080 (4)	0.0165 (4)	−0.0013 (4)
O2	0.0713 (7)	0.0657 (7)	0.0547 (6)	0.0123 (5)	0.0250 (5)	−0.0130 (5)
O3	0.0412 (5)	0.0355 (5)	0.0814 (7)	0.0021 (4)	0.0224 (5)	−0.0019 (4)
C1	0.0454 (7)	0.0560 (8)	0.0483 (7)	0.0088 (6)	0.0171 (6)	0.0094 (6)
C2	0.0807 (12)	0.0946 (14)	0.0670 (11)	0.0121 (10)	0.0378 (10)	0.0271 (9)
C3	0.0677 (11)	0.0677 (11)	0.0821 (11)	0.0265 (9)	0.0309 (9)	0.0127 (9)
C4	0.0509 (9)	0.0787 (11)	0.0664 (10)	−0.0030 (8)	0.0082 (8)	0.0053 (8)
C5	0.0447 (7)	0.0381 (6)	0.0503 (7)	−0.0006 (5)	0.0227 (6)	−0.0037 (5)
C6	0.0470 (7)	0.0381 (6)	0.0538 (8)	0.0051 (5)	0.0177 (6)	−0.0026 (5)
C7	0.0361 (6)	0.0364 (6)	0.0503 (7)	0.0024 (5)	0.0117 (5)	0.0010 (5)
C8	0.0493 (8)	0.0448 (7)	0.0637 (8)	−0.0005 (6)	0.0312 (7)	−0.0061 (6)
C9	0.0489 (7)	0.0417 (7)	0.0549 (8)	−0.0054 (6)	0.0277 (6)	−0.0014 (5)
C10	0.0341 (6)	0.0379 (6)	0.0359 (6)	0.0010 (5)	0.0109 (5)	0.0033 (5)
C11	0.0487 (7)	0.0415 (7)	0.0645 (8)	0.0046 (6)	0.0327 (7)	0.0058 (6)
C12	0.0485 (8)	0.0399 (7)	0.0715 (9)	0.0004 (6)	0.0310 (7)	0.0085 (6)

Geometric parameters (Å, °)

O1—C5	1.3379 (15)	C4—H4C	0.9600
O1—C1	1.4867 (16)	C5—C6	1.5118 (19)
O2—C5	1.2020 (16)	C6—H6A	0.9700
O3—C7	1.3861 (15)	C6—H6B	0.9700
O3—C6	1.4143 (16)	C7—C12	1.3787 (19)
C1—C4	1.513 (2)	C7—C8	1.3807 (19)
C1—C2	1.519 (2)	C8—C9	1.3890 (19)
C1—C3	1.518 (2)	C8—H8	0.9300
C2—H2A	0.9600	C9—C10	1.3905 (18)
C2—H2B	0.9600	C9—H9	0.9300
C2—H2C	0.9600	C10—C11	1.3929 (18)
C3—H3A	0.9600	C10—C10^{i}	1.497 (2)

C3—H3B	0.9600	C11—C12	1.3845 (19)
C3—H3C	0.9600	C11—H11	0.9300
C4—H4A	0.9600	C12—H12	0.9300
C4—H4B	0.9600		

C5—O1—C1	121.76 (10)	O2—C5—C6	124.59 (12)
C7—O3—C6	119.66 (10)	O1—C5—C6	108.99 (10)
O1—C1—C4	109.05 (11)	O3—C6—C5	111.44 (11)
O1—C1—C2	110.05 (12)	O3—C6—H6A	109.3
C4—C1—C2	113.18 (14)	C5—C6—H6A	109.3
O1—C1—C3	102.28 (12)	O3—C6—H6B	109.3
C4—C1—C3	110.99 (14)	C5—C6—H6B	109.3
C2—C1—C3	110.73 (14)	H6A—C6—H6B	108.0
C1—C2—H2A	109.5	C12—C7—C8	119.36 (12)
C1—C2—H2B	109.5	C12—C7—O3	115.67 (12)
H2A—C2—H2B	109.5	C8—C7—O3	124.85 (12)
C1—C2—H2C	109.5	C7—C8—C9	119.45 (13)
H2A—C2—H2C	109.5	C7—C8—H8	120.3
H2B—C2—H2C	109.5	C9—C8—H8	120.3
C1—C3—H3A	109.5	C8—C9—C10	122.62 (12)
C1—C3—H3B	109.5	C8—C9—H9	118.7
H3A—C3—H3B	109.5	C10—C9—H9	118.7
C1—C3—H3C	109.5	C9—C10—C11	116.31 (11)
H3A—C3—H3C	109.5	C9—C10—C10^{i}	121.94 (14)
H3B—C3—H3C	109.5	C11—C10—C10^{i}	121.75 (14)
C1—C4—H4A	109.5	C12—C11—C10	121.79 (13)
C1—C4—H4B	109.5	C12—C11—H11	119.1
H4A—C4—H4B	109.5	C10—C11—H11	119.1
C1—C4—H4C	109.5	C7—C12—C11	120.47 (13)
H4A—C4—H4C	109.5	C7—C12—H12	119.8
H4B—C4—H4C	109.5	C11—C12—H12	119.8
O2—C5—O1	126.41 (13)		

C5—O1—C1—C4	64.56 (16)	C12—C7—C8—C9	−0.1 (2)
C5—O1—C1—C2	−60.14 (17)	O3—C7—C8—C9	−175.79 (13)
C5—O1—C1—C3	−177.86 (12)	C7—C8—C9—C10	−0.3 (2)
C1—O1—C5—O2	0.4 (2)	C8—C9—C10—C11	0.3 (2)
C1—O1—C5—C6	−178.70 (11)	C8—C9—C10—C10^{i}	179.82 (14)
C7—O3—C6—C5	−77.86 (15)	C9—C10—C11—C12	−0.1 (2)
O2—C5—C6—O3	18.60 (19)	C10^{i}—C10—C11—C12	−179.55 (14)
O1—C5—C6—O3	−162.30 (10)	C8—C7—C12—C11	0.3 (2)
C6—O3—C7—C12	153.70 (12)	O3—C7—C12—C11	176.43 (12)
C6—O3—C7—C8	−30.43 (19)	C10—C11—C12—C7	−0.3 (2)

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

Fig. 1