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Di-*tert*-butyl (1,1′-binaphthyl-2,2′-dioxy)-diacetate

Asra Mustafa,[a] Muhammad Raza Shah,[a] Maimoona Khatoon[a] and Seik Weng Ng[b]*

[a]HEJ Research Institute of Chemistry, International Center for Chemical and Biological Sciences, University of Karachi, Karachi 75270, Pakistan, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

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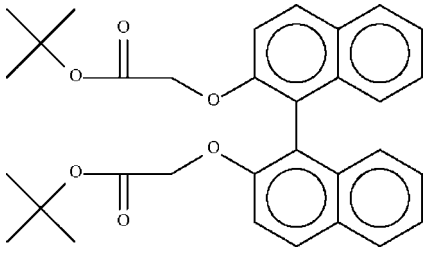
Key indicators: single-crystal X-ray study; T = 133 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.121; data-to-parameter ratio = 18.3.

In the crystal structure of the title compound, $C_{32}H_{34}O_6$, the molecule is located on a twofold rotation axis. The two naphthyl fused-ring systems are aligned at 72.6 (1)°. Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure.

Related literature

For the crystal structure of the parent carboxylic acid, see: Wu *et al.* (2007).



Experimental

Crystal data

$C_{32}H_{34}O_6$
M_r = 514.59
Monoclinic, $C2/c$
a = 18.7604 (3) Å
b = 14.3204 (3) Å
c = 10.9997 (2) Å
β = 110.144 (1)°
V = 2774.37 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 133 K
0.30 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
12968 measured reflections
3198 independent reflections
2514 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.121
S = 1.01
3198 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···O2^{i}	0.95	2.38	3.226 (2)	149

Symmetry code: (i) $x, -y+1, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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, 2009).

We thank the Higher Education Commission of Pakistan and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2499).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2009). *publCIF*. In preparation.
Wu, Y.-M., Cao, G.-Q., Qian, M.-Y. & Zhu, H.-J. (2007). *Acta Cryst.* E**63**, o3446.

supporting information

Acta Cryst. (2009). E**65**, o912 [doi:10.1107/S1600536809010836]

Di-*tert*-butyl (1,1′-binaphthyl-2,2′-dioxy)diacetate

Asra Mustafa, Muhammad Raza Shah, Maimoona Khatoon and Seik Weng Ng

S1. Experimental

Potassium carbonate (0.97 g, 7 mmol) and 1,1′-binaphthyl-2,2′-diol (0.57 mg, 2 mmol) in acetone (20 ml) were stirred for 15 minutes. *tert*-Butyl 2-bromoacetate (1.95 g, 10 mmol) was added and the mixture was stirred at 323 K for 2 h. The solvent was removed and the residue was dissolved in a mixture of water (50 ml) and dichloromethane (50 ml). The two phases were separated and the aqueous layer was extracted with dichloromethane. The combined organic phases were dried and the solvent evaporated. The residue was dissolved recrysstallized from dichloromethane (0.82 mg, 80% yield).

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.99 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C).

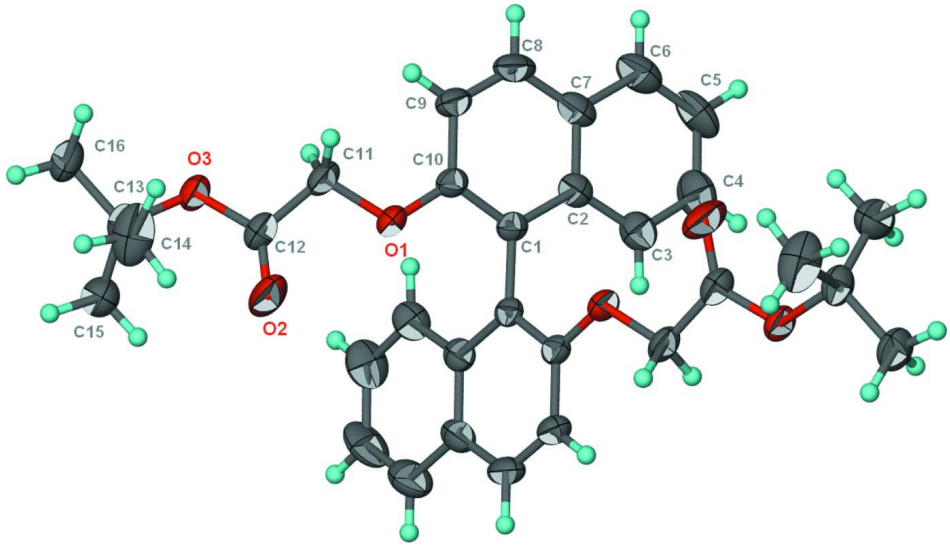


Figure 1

Thermal ellipsoid plot (Barbour, 2001) plot of $C_{32}H_{34}O_6$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Di-*tert*-butyl (1,1′-binaphthyl-2,2′-dioxy)diacetate

Crystal data

$C_{32}H_{34}O_6$
M_r = 514.59
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 18.7604 (3) Å
b = 14.3204 (3) Å
c = 10.9997 (2) Å
β = 110.144 (1)°

V = 2774.37 (9) Å^3
Z = 4
F(000) = 1096
D_x = 1.232 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3672 reflections

θ = 2.3–28.2°
μ = 0.08 mm^{-1}
T = 133 K
Block, colorless
0.30 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
12968 measured reflections
3198 independent reflections

2514 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°, θ_{min} = 1.8°
h = −24→24
k = −18→18
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.121
S = 1.01
3198 reflections
175 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
$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 1.9389P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.38962 (5)	0.53557 (7)	0.23963 (9)	0.0278 (2)
O2	0.28361 (6)	0.45195 (10)	0.03634 (11)	0.0531 (4)
O3	0.19998 (5)	0.45848 (7)	0.14254 (9)	0.0292 (2)
C1	0.49788 (7)	0.63074 (9)	0.31656 (11)	0.0219 (3)
C2	0.55167 (7)	0.68166 (9)	0.41843 (12)	0.0255 (3)
C3	0.61052 (8)	0.73495 (10)	0.39965 (14)	0.0326 (3)
H3	0.6162	0.7352	0.3171	0.039*
C4	0.65937 (10)	0.78615 (12)	0.49836 (16)	0.0441 (4)
H4	0.6980	0.8223	0.4833	0.053*
C5	0.65281 (11)	0.78558 (13)	0.62268 (16)	0.0503 (5)
H5	0.6869	0.8213	0.6908	0.060*
C6	0.59820 (10)	0.73436 (12)	0.64444 (15)	0.0437 (4)
H6	0.5948	0.7336	0.7286	0.052*
C7	0.54571 (8)	0.68154 (10)	0.54431 (13)	0.0314 (3)
C8	0.48693 (8)	0.62974 (12)	0.56391 (13)	0.0350 (3)
H8	0.4833	0.6283	0.6479	0.042*
C9	0.43514 (8)	0.58162 (11)	0.46649 (13)	0.0310 (3)
H9	0.3958	0.5474	0.4824	0.037*
C10	0.44042 (7)	0.58301 (9)	0.34153 (12)	0.0242 (3)
C11	0.32140 (7)	0.50678 (11)	0.25672 (13)	0.0291 (3)
H11A	0.2979	0.5603	0.2857	0.035*

H11B	0.3324	0.4575	0.3238	0.035*
C12	0.26778 (8)	0.46984 (10)	0.13024 (14)	0.0304 (3)
C13	0.13800 (8)	0.40721 (10)	0.04369 (14)	0.0315 (3)
C14	0.16385 (11)	0.30839 (13)	0.0342 (2)	0.0556 (5)
H14A	0.1856	0.2817	0.1213	0.083*
H14B	0.2023	0.3087	−0.0075	0.083*
H14C	0.1204	0.2706	−0.0173	0.083*
C15	0.11375 (9)	0.45879 (14)	−0.08469 (15)	0.0439 (4)
H15A	0.1028	0.5241	−0.0710	0.066*
H15B	0.0681	0.4294	−0.1451	0.066*
H15C	0.1547	0.4562	−0.1208	0.066*
C16	0.07523 (8)	0.40965 (12)	0.10104 (15)	0.0394 (4)
H16A	0.0930	0.3799	0.1865	0.059*
H16B	0.0308	0.3760	0.0440	0.059*
H16C	0.0613	0.4746	0.1097	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0218 (5)	0.0396 (6)	0.0254 (5)	−0.0038 (4)	0.0123 (4)	−0.0006 (4)
O2	0.0342 (6)	0.0883 (10)	0.0461 (7)	−0.0154 (6)	0.0255 (5)	−0.0245 (6)
O3	0.0220 (5)	0.0364 (5)	0.0321 (5)	−0.0025 (4)	0.0131 (4)	−0.0021 (4)
C1	0.0220 (6)	0.0255 (6)	0.0194 (6)	0.0059 (5)	0.0088 (5)	0.0020 (5)
C2	0.0265 (7)	0.0256 (6)	0.0225 (6)	0.0064 (5)	0.0062 (5)	−0.0007 (5)
C3	0.0345 (7)	0.0302 (7)	0.0292 (7)	−0.0021 (6)	0.0061 (6)	−0.0016 (6)
C4	0.0416 (9)	0.0373 (8)	0.0456 (9)	−0.0091 (7)	0.0050 (7)	−0.0067 (7)
C5	0.0545 (10)	0.0477 (10)	0.0339 (8)	−0.0049 (8)	−0.0037 (8)	−0.0146 (7)
C6	0.0508 (10)	0.0487 (9)	0.0261 (7)	0.0031 (8)	0.0060 (7)	−0.0089 (7)
C7	0.0340 (7)	0.0357 (7)	0.0214 (6)	0.0105 (6)	0.0054 (6)	−0.0029 (5)
C8	0.0367 (8)	0.0513 (9)	0.0199 (6)	0.0129 (7)	0.0134 (6)	0.0035 (6)
C9	0.0282 (7)	0.0447 (8)	0.0241 (7)	0.0072 (6)	0.0142 (6)	0.0064 (6)
C10	0.0218 (6)	0.0310 (7)	0.0213 (6)	0.0062 (5)	0.0094 (5)	0.0035 (5)
C11	0.0223 (6)	0.0385 (8)	0.0310 (7)	0.0016 (5)	0.0150 (6)	0.0050 (6)
C12	0.0249 (7)	0.0345 (7)	0.0359 (7)	−0.0004 (6)	0.0157 (6)	−0.0007 (6)
C13	0.0240 (6)	0.0332 (7)	0.0377 (8)	−0.0051 (6)	0.0110 (6)	−0.0043 (6)
C14	0.0443 (10)	0.0366 (9)	0.0848 (14)	−0.0032 (8)	0.0207 (10)	−0.0131 (9)
C15	0.0359 (8)	0.0621 (11)	0.0335 (8)	−0.0065 (8)	0.0118 (7)	0.0006 (7)
C16	0.0262 (7)	0.0516 (10)	0.0419 (9)	−0.0063 (7)	0.0138 (6)	0.0024 (7)

Geometric parameters (Å, º)

O1—C10	1.3749 (16)	C8—C9	1.360 (2)
O1—C11	1.4174 (15)	C8—H8	0.9500
O2—C12	1.1966 (17)	C9—C10	1.4118 (17)
O3—C12	1.3347 (15)	C9—H9	0.9500
O3—C13	1.4842 (16)	C11—C12	1.504 (2)
C1—C10	1.3815 (17)	C11—H11A	0.9900
C1—C2	1.4237 (18)	C11—H11B	0.9900

C1—C1^{i}	1.494 (2)	C13—C14	1.511 (2)
C2—C3	1.415 (2)	C13—C16	1.5158 (19)
C2—C7	1.4270 (18)	C13—C15	1.518 (2)
C3—C4	1.368 (2)	C14—H14A	0.9800
C3—H3	0.9500	C14—H14B	0.9800
C4—C5	1.415 (2)	C14—H14C	0.9800
C4—H4	0.9500	C15—H15A	0.9800
C5—C6	1.347 (3)	C15—H15B	0.9800
C5—H5	0.9500	C15—H15C	0.9800
C6—C7	1.417 (2)	C16—H16A	0.9800
C6—H6	0.9500	C16—H16B	0.9800
C7—C8	1.406 (2)	C16—H16C	0.9800
C10—O1—C11	116.08 (10)	O1—C11—H11A	109.9
C12—O3—C13	121.30 (11)	C12—C11—H11A	109.9
C10—C1—C2	119.15 (11)	O1—C11—H11B	109.9
C10—C1—C1^{i}	120.24 (12)	C12—C11—H11B	109.9
C2—C1—C1^{i}	120.60 (12)	H11A—C11—H11B	108.3
C3—C2—C1	122.59 (12)	O2—C12—O3	126.15 (14)
C3—C2—C7	117.92 (13)	O2—C12—C11	126.00 (13)
C1—C2—C7	119.47 (12)	O3—C12—C11	107.83 (11)
C4—C3—C2	121.09 (14)	O3—C13—C14	108.95 (12)
C4—C3—H3	119.5	O3—C13—C16	102.04 (11)
C2—C3—H3	119.5	C14—C13—C16	111.35 (14)
C3—C4—C5	120.45 (16)	O3—C13—C15	110.43 (12)
C3—C4—H4	119.8	C14—C13—C15	113.09 (15)
C5—C4—H4	119.8	C16—C13—C15	110.43 (13)
C6—C5—C4	119.97 (15)	C13—C14—H14A	109.5
C6—C5—H5	120.0	C13—C14—H14B	109.5
C4—C5—H5	120.0	H14A—C14—H14B	109.5
C5—C6—C7	121.34 (15)	C13—C14—H14C	109.5
C5—C6—H6	119.3	H14A—C14—H14C	109.5
C7—C6—H6	119.3	H14B—C14—H14C	109.5
C8—C7—C6	122.29 (13)	C13—C15—H15A	109.5
C8—C7—C2	118.50 (13)	C13—C15—H15B	109.5
C6—C7—C2	119.20 (14)	H15A—C15—H15B	109.5
C9—C8—C7	122.11 (12)	C13—C15—H15C	109.5
C9—C8—H8	118.9	H15A—C15—H15C	109.5
C7—C8—H8	118.9	H15B—C15—H15C	109.5
C8—C9—C10	119.30 (13)	C13—C16—H16A	109.5
C8—C9—H9	120.3	C13—C16—H16B	109.5
C10—C9—H9	120.3	H16A—C16—H16B	109.5
O1—C10—C1	116.85 (10)	C13—C16—H16C	109.5
O1—C10—C9	121.70 (12)	H16A—C16—H16C	109.5
C1—C10—C9	121.44 (12)	H16B—C16—H16C	109.5
O1—C11—C12	109.05 (10)		
C10—C1—C2—C3	−177.72 (12)	C7—C8—C9—C10	0.3 (2)

C1^{i}—C1—C2—C3	0.71 (18)	C11—O1—C10—C1	−164.92 (11)
C10—C1—C2—C7	0.70 (18)	C11—O1—C10—C9	16.29 (18)
C1^{i}—C1—C2—C7	179.14 (11)	C2—C1—C10—O1	179.49 (11)
C1—C2—C3—C4	177.29 (13)	C1^{i}—C1—C10—O1	1.05 (16)
C7—C2—C3—C4	−1.2 (2)	C2—C1—C10—C9	−1.72 (19)
C2—C3—C4—C5	1.1 (2)	C1^{i}—C1—C10—C9	179.83 (11)
C3—C4—C5—C6	0.0 (3)	C8—C9—C10—O1	179.96 (12)
C4—C5—C6—C7	−1.0 (3)	C8—C9—C10—C1	1.2 (2)
C5—C6—C7—C8	−178.13 (16)	C10—O1—C11—C12	171.26 (11)
C5—C6—C7—C2	0.9 (2)	C13—O3—C12—O2	9.7 (2)
C3—C2—C7—C8	179.26 (13)	C13—O3—C12—C11	−168.85 (11)
C1—C2—C7—C8	0.76 (19)	O1—C11—C12—O2	12.1 (2)
C3—C2—C7—C6	0.2 (2)	O1—C11—C12—O3	−169.43 (11)
C1—C2—C7—C6	−178.34 (13)	C12—O3—C13—C14	60.23 (17)
C6—C7—C8—C9	177.77 (14)	C12—O3—C13—C16	178.05 (13)
C2—C7—C8—C9	−1.3 (2)	C12—O3—C13—C15	−64.54 (16)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···O2^{ii}	0.95	2.38	3.226 (2)	149

Symmetry code: (ii) *x*, −*y*+1, *z*+1/2.