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4,4,5,5-Tetramethyl-2-[(*Z*)-1-(3-methylphenoxy)-hex-1-en-2-yl]-1,3,2-dioxaborolane

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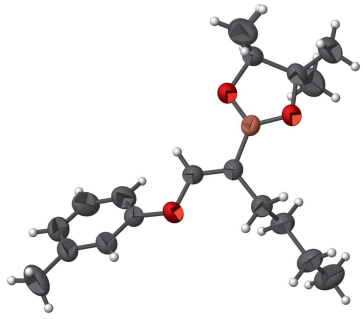
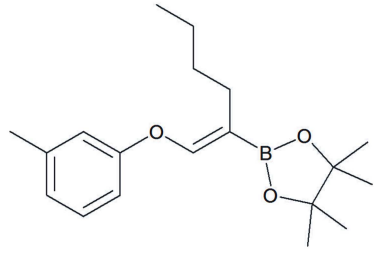
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{19}H_{29}BO_3$, was prepared by the reaction of 1-(hex-1-yn-1-yloxy)-3-methylbenzene in tetrahydrofuran with 4,4,5,5-tetramethyl-1,3,2-dioxaborolane under a nitrogen atmosphere. In the molecule, the butyl group adopts an extended conformation, with a torsion angle of 179.52 (19)°. The dioxaborolane ring has a twisted conformation on the C—C bond, and its mean plane is inclined to the 3-methylphenyl ring by 44.79 (19)°. In the crystal, there are no significant intermolecular interactions present.

3D view



Chemical scheme



Structure description

The title compound was previously prepared by Cui *et al.* (2013), and its derivatives were synthesized by Yang & Cheng (2001) and Whelligan *et al.* (2010). We present here the crystal structure of the compound (Fig. 1). In the molecule, the conformation of the dioxaborolane ring is similar to half chair, and the butyl group adopts an extended conformation with a C10—C11—C12—C13 torsion angle = 179.52 (19)°. No hydrogen bonding is observed in the crystal.

Synthesis and crystallization

To a solution of 1-(hex-1-yn-1-yloxy)-3-methylbenzene (94 mg, 0.5 mmol) in 2.0 ml of tetrahydrofuran (THF) was added neat 4,4,5,5-tetramethyl-1,3,2-dioxaborolane (HBpin) (71 mg, 0.55 mmol) under nitrogen atmosphere. After stirring at room temperature for 1.5 h, the reaction mixture was concentrated and purified by on silica gel (petroleum ether/EtOAc = 60/1).

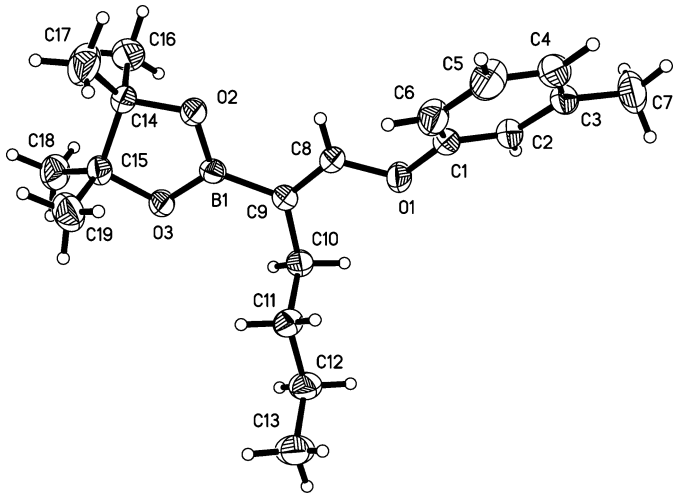


Figure 1
Perspective view of the structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{29}BO_3$
M_r	316.23
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	10.0293 (9), 18.1230 (17), 10.7973 (10)
β (°)	97.475 (6)
V (Å^3)	1945.9 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.07
Crystal size (mm)	0.3 × 0.2 × 0.1
Data collection	
Diffractometer	Bruker APEXII
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 1996)
T_{min}, T_{max}	0.983, 0.993
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30133, 4375, 2665
R_{int}	0.044
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.648
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.168, 1.09
No. of reflections	4375
No. of parameters	208
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.18

Computer programs: *APEX2* and *SAINT* (Bruker, 2006), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160611 [doi:10.1107/S2414314616006118]

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4,4,5,5-Tetramethyl-2-[(*Z*)-1-(3-methylphenoxy)hex-1-en-2-yl]-1,3,2-dioxaborolane

Crystal data

$C_{19}H_{29}BO_3$
M_r = 316.23
Monoclinic, $P2_1/c$
a = 10.0293 (9) Å
b = 18.1230 (17) Å
c = 10.7973 (10) Å
β = 97.475 (6)°
V = 1945.9 (3) Å^3
Z = 4
$F(000)$ = 688
D_x = 1.079 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4375 reflections
θ = 2.1–27.4°
μ = 0.07 mm^{-1}
T = 296 K
Block, colourless
0.3 × 0.2 × 0.1 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.983, T_{max} = 0.993
30133 measured reflections
4375 independent reflections
2665 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.4°, θ_{min} = 2.1°
h = −13→12
k = −23→21
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.168
S = 1.09
4375 reflections
208 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
$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2 + 0.1148P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
B1	0.66602 (17)	0.11935 (10)	0.19395 (16)	0.0479 (4)
O1	0.49825 (11)	0.27876 (7)	0.01441 (12)	0.0662 (4)
O2	0.56737 (11)	0.06699 (6)	0.18524 (11)	0.0620 (4)
O3	0.78374 (10)	0.09596 (6)	0.25964 (11)	0.0573 (3)
C1	0.36387 (17)	0.29473 (9)	−0.02574 (16)	0.0558 (4)
C2	0.33922 (18)	0.33782 (9)	−0.13101 (16)	0.0587 (4)
H2A	0.4104	0.3531	−0.1721	0.070*
C3	0.2087 (2)	0.35875 (10)	−0.17633 (18)	0.0664 (5)
C4	0.1053 (2)	0.33435 (12)	−0.1136 (2)	0.0820 (7)
H4A	0.0170	0.3466	−0.1438	0.098*
C5	0.1308 (2)	0.29256 (12)	−0.0081 (3)	0.0882 (7)
H5A	0.0597	0.2772	0.0331	0.106*
C6	0.26130 (19)	0.27263 (11)	0.0386 (2)	0.0732 (5)
H6A	0.2789	0.2450	0.1115	0.088*
C7	0.1814 (3)	0.40687 (14)	−0.2895 (2)	0.1007 (8)
H7C	0.0864	0.4152	−0.3082	0.121*
H7B	0.2137	0.3830	−0.3592	0.121*
H7A	0.2266	0.4533	−0.2738	0.121*
C8	0.52791 (17)	0.21201 (9)	0.07200 (15)	0.0548 (4)
H8A	0.4622	0.1755	0.0649	0.066*
C9	0.64616 (15)	0.19761 (9)	0.13719 (14)	0.0493 (4)
C10	0.75572 (16)	0.25522 (10)	0.15952 (15)	0.0563 (4)
H10B	0.8418	0.2317	0.1547	0.068*
H10A	0.7419	0.2917	0.0933	0.068*
C11	0.76153 (18)	0.29400 (10)	0.28296 (16)	0.0632 (5)
H11B	0.7786	0.2579	0.3494	0.076*
H11A	0.6747	0.3163	0.2891	0.076*
C12	0.8690 (2)	0.35332 (11)	0.30207 (18)	0.0763 (6)
H12B	0.9557	0.3308	0.2964	0.092*
H12A	0.8523	0.3890	0.2349	0.092*
C13	0.8758 (3)	0.39323 (13)	0.4244 (2)	0.1006 (8)
H13C	0.9454	0.4299	0.4295	0.121*
H13B	0.8954	0.3587	0.4917	0.121*
H13A	0.7911	0.4167	0.4304	0.121*
C14	0.62832 (19)	−0.00170 (9)	0.23442 (18)	0.0635 (5)
C15	0.75651 (16)	0.02563 (10)	0.31787 (16)	0.0565 (4)
C16	0.6612 (3)	−0.04561 (13)	0.1223 (2)	0.1110 (9)
H16C	0.5792	−0.0618	0.0741	0.133*
H16B	0.7147	−0.0877	0.1505	0.133*
H16A	0.7103	−0.0150	0.0715	0.133*

C17	0.5277 (2)	−0.04152 (15)	0.3024 (3)	0.1157 (10)
H17C	0.4526	−0.0567	0.2436	0.139*
H17B	0.4971	−0.0091	0.3630	0.139*
H17A	0.5694	−0.0841	0.3438	0.139*
C18	0.8788 (2)	−0.02308 (12)	0.3206 (2)	0.0831 (6)
H18C	0.8927	−0.0352	0.2367	0.100*
H18B	0.8653	−0.0675	0.3656	0.100*
H18A	0.9562	0.0025	0.3612	0.100*
C19	0.7334 (3)	0.04403 (14)	0.45048 (18)	0.0917 (7)
H19C	0.6552	0.0747	0.4487	0.110*
H19B	0.8103	0.0697	0.4918	0.110*
H19A	0.7201	−0.0007	0.4948	0.110*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B1	0.0444 (10)	0.0490 (10)	0.0490 (9)	−0.0016 (8)	0.0007 (8)	−0.0027 (8)
O1	0.0523 (7)	0.0530 (7)	0.0881 (9)	−0.0010 (5)	−0.0104 (6)	0.0185 (6)
O2	0.0509 (7)	0.0507 (7)	0.0791 (8)	−0.0044 (5)	−0.0114 (6)	0.0111 (6)
O3	0.0468 (6)	0.0527 (7)	0.0689 (7)	−0.0018 (5)	−0.0065 (5)	0.0086 (6)
C1	0.0504 (10)	0.0473 (9)	0.0661 (10)	0.0005 (7)	−0.0056 (8)	−0.0005 (8)
C2	0.0585 (11)	0.0536 (10)	0.0615 (10)	0.0058 (8)	−0.0012 (8)	0.0016 (8)
C3	0.0666 (12)	0.0567 (11)	0.0705 (11)	0.0123 (9)	−0.0117 (9)	−0.0080 (9)
C4	0.0536 (12)	0.0669 (13)	0.1195 (18)	0.0088 (10)	−0.0116 (12)	−0.0070 (13)
C5	0.0605 (13)	0.0714 (14)	0.136 (2)	0.0021 (11)	0.0232 (12)	0.0120 (14)
C6	0.0678 (12)	0.0633 (12)	0.0886 (14)	0.0002 (10)	0.0104 (10)	0.0164 (10)
C7	0.1099 (18)	0.0981 (18)	0.0862 (15)	0.0364 (15)	−0.0170 (13)	0.0121 (13)
C8	0.0571 (10)	0.0424 (9)	0.0620 (10)	−0.0049 (7)	−0.0037 (8)	0.0053 (7)
C9	0.0471 (9)	0.0484 (9)	0.0506 (9)	−0.0010 (7)	−0.0004 (7)	0.0011 (7)
C10	0.0530 (10)	0.0553 (10)	0.0593 (10)	−0.0056 (8)	0.0020 (7)	0.0041 (8)
C11	0.0690 (12)	0.0538 (10)	0.0662 (11)	−0.0074 (8)	0.0065 (9)	−0.0014 (8)
C12	0.0868 (14)	0.0681 (13)	0.0716 (12)	−0.0258 (11)	0.0013 (10)	−0.0003 (10)
C13	0.136 (2)	0.0864 (16)	0.0765 (14)	−0.0390 (15)	0.0029 (14)	−0.0076 (12)
C14	0.0657 (11)	0.0445 (9)	0.0749 (12)	−0.0028 (8)	−0.0108 (9)	0.0092 (8)
C15	0.0546 (10)	0.0541 (10)	0.0586 (9)	0.0042 (8)	−0.0011 (7)	0.0093 (8)
C16	0.160 (2)	0.0626 (14)	0.0960 (17)	0.0129 (15)	−0.0365 (16)	−0.0184 (12)
C17	0.0818 (16)	0.0952 (18)	0.164 (3)	−0.0247 (14)	−0.0082 (16)	0.0607 (18)
C18	0.0690 (13)	0.0736 (14)	0.1030 (16)	0.0160 (11)	−0.0028 (11)	0.0132 (12)
C19	0.1162 (18)	0.0980 (17)	0.0586 (12)	0.0114 (14)	0.0023 (11)	0.0079 (11)

Geometric parameters (Å, º)

B1—O3	1.363 (2)	C11—C12	1.517 (2)
B1—O2	1.365 (2)	C11—H11B	0.9700
B1—C9	1.548 (2)	C11—H11A	0.9700
O1—C8	1.3749 (19)	C12—C13	1.500 (3)
O1—C1	1.3914 (19)	C12—H12B	0.9700
O2—C14	1.456 (2)	C12—H12A	0.9700

O3—C15	1.463 (2)	C13—H13C	0.9600
C1—C6	1.374 (3)	C13—H13B	0.9600
C1—C2	1.375 (2)	C13—H13A	0.9600
C2—C3	1.390 (2)	C14—C17	1.506 (3)
C2—H2A	0.9300	C14—C16	1.520 (3)
C3—C4	1.383 (3)	C14—C15	1.552 (2)
C3—C7	1.498 (3)	C15—C18	1.508 (2)
C4—C5	1.364 (3)	C15—C19	1.517 (3)
C4—H4A	0.9300	C16—H16C	0.9600
C5—C6	1.388 (3)	C16—H16B	0.9600
C5—H5A	0.9300	C16—H16A	0.9600
C6—H6A	0.9300	C17—H17C	0.9600
C7—H7C	0.9600	C17—H17B	0.9600
C7—H7B	0.9600	C17—H17A	0.9600
C7—H7A	0.9600	C18—H18C	0.9600
C8—C9	1.324 (2)	C18—H18B	0.9600
C8—H8A	0.9300	C18—H18A	0.9600
C9—C10	1.512 (2)	C19—H19C	0.9600
C10—C11	1.501 (2)	C19—H19B	0.9600
C10—H10B	0.9700	C19—H19A	0.9600
C10—H10A	0.9700		

O3—B1—O2	112.99 (15)	C13—C12—C11	114.32 (18)
O3—B1—C9	123.30 (15)	C13—C12—H12B	108.7
O2—B1—C9	123.70 (14)	C11—C12—H12B	108.7
C8—O1—C1	117.89 (13)	C13—C12—H12A	108.7
B1—O2—C14	107.64 (12)	C11—C12—H12A	108.7
B1—O3—C15	107.28 (12)	H12B—C12—H12A	107.6
C6—C1—C2	121.19 (17)	C12—C13—H13C	109.5
C6—C1—O1	122.72 (16)	C12—C13—H13B	109.5
C2—C1—O1	116.00 (15)	H13C—C13—H13B	109.5
C1—C2—C3	120.40 (18)	C12—C13—H13A	109.5
C1—C2—H2A	119.8	H13C—C13—H13A	109.5
C3—C2—H2A	119.8	H13B—C13—H13A	109.5
C4—C3—C2	118.19 (18)	O2—C14—C17	108.13 (16)
C4—C3—C7	121.14 (19)	O2—C14—C16	106.33 (15)
C2—C3—C7	120.7 (2)	C17—C14—C16	111.4 (2)
C5—C4—C3	121.04 (19)	O2—C14—C15	102.45 (13)
C5—C4—H4A	119.5	C17—C14—C15	115.27 (17)
C3—C4—H4A	119.5	C16—C14—C15	112.36 (18)
C4—C5—C6	120.9 (2)	O3—C15—C18	108.75 (14)
C4—C5—H5A	119.6	O3—C15—C19	105.91 (15)
C6—C5—H5A	119.6	C18—C15—C19	109.50 (16)
C1—C6—C5	118.26 (19)	O3—C15—C14	102.45 (12)
C1—C6—H6A	120.9	C18—C15—C14	115.77 (16)
C5—C6—H6A	120.9	C19—C15—C14	113.67 (16)
C3—C7—H7C	109.5	C14—C16—H16C	109.5
C3—C7—H7B	109.5	C14—C16—H16B	109.5

H7C—C7—H7B	109.5	H16C—C16—H16B	109.5
C3—C7—H7A	109.5	C14—C16—H16A	109.5
H7C—C7—H7A	109.5	H16C—C16—H16A	109.5
H7B—C7—H7A	109.5	H16B—C16—H16A	109.5
C9—C8—O1	122.69 (15)	C14—C17—H17C	109.5
C9—C8—H8A	118.7	C14—C17—H17B	109.5
O1—C8—H8A	118.7	H17C—C17—H17B	109.5
C8—C9—C10	122.06 (15)	C14—C17—H17A	109.5
C8—C9—B1	116.84 (14)	H17C—C17—H17A	109.5
C10—C9—B1	121.07 (13)	H17B—C17—H17A	109.5
C11—C10—C9	114.10 (14)	C15—C18—H18C	109.5
C11—C10—H10B	108.7	C15—C18—H18B	109.5
C9—C10—H10B	108.7	H18C—C18—H18B	109.5
C11—C10—H10A	108.7	C15—C18—H18A	109.5
C9—C10—H10A	108.7	H18C—C18—H18A	109.5
H10B—C10—H10A	107.6	H18B—C18—H18A	109.5
C10—C11—C12	113.41 (15)	C15—C19—H19C	109.5
C10—C11—H11B	108.9	C15—C19—H19B	109.5
C12—C11—H11B	108.9	H19C—C19—H19B	109.5
C10—C11—H11A	108.9	C15—C19—H19A	109.5
C12—C11—H11A	108.9	H19C—C19—H19A	109.5
H11B—C11—H11A	107.7	H19B—C19—H19A	109.5

O3—B1—O2—C14	−8.47 (19)	O3—B1—C9—C10	−4.3 (2)
C9—B1—O2—C14	172.46 (15)	O2—B1—C9—C10	174.71 (15)
O2—B1—O3—C15	−9.78 (18)	C8—C9—C10—C11	96.8 (2)
C9—B1—O3—C15	169.30 (15)	B1—C9—C10—C11	−81.07 (19)
C8—O1—C1—C6	35.4 (2)	C9—C10—C11—C12	−178.12 (16)
C8—O1—C1—C2	−148.01 (16)	C10—C11—C12—C13	179.52 (19)
C6—C1—C2—C3	−1.4 (3)	B1—O2—C14—C17	143.74 (18)
O1—C1—C2—C3	−178.00 (15)	B1—O2—C14—C16	−96.49 (19)
C1—C2—C3—C4	−0.7 (3)	B1—O2—C14—C15	21.59 (17)
C1—C2—C3—C7	178.83 (18)	B1—O3—C15—C18	145.33 (15)
C2—C3—C4—C5	1.8 (3)	B1—O3—C15—C19	−97.07 (16)
C7—C3—C4—C5	−177.8 (2)	B1—O3—C15—C14	22.30 (17)
C3—C4—C5—C6	−0.7 (3)	O2—C14—C15—O3	−26.26 (17)
C2—C1—C6—C5	2.4 (3)	C17—C14—C15—O3	−143.43 (17)
O1—C1—C6—C5	178.81 (18)	C16—C14—C15—O3	87.46 (18)
C4—C5—C6—C1	−1.4 (3)	O2—C14—C15—C18	−144.43 (16)
C1—O1—C8—C9	−164.95 (16)	C17—C14—C15—C18	98.4 (2)
O1—C8—C9—C10	3.5 (3)	C16—C14—C15—C18	−30.7 (2)
O1—C8—C9—B1	−178.53 (15)	O2—C14—C15—C19	87.53 (18)
O3—B1—C9—C8	177.75 (15)	C17—C14—C15—C19	−29.6 (2)
O2—B1—C9—C8	−3.3 (2)	C16—C14—C15—C19	−158.76 (18)