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Homopropargyl alcohol 1,1-diphenylbut-3-yn-1-ol

Christian A. Umaña,[a] Leslie W. Pineda[a,b] and Jorge A. Cabezas[a]*

[a]Escuela de Química, Universidad de Costa Rica, 2060, San José, Costa Rica, and [b]Centro de Electroquímica y Energía Química (CELEQ), Universidad de Costa, Rica, 2060 San José, Costa Rica. *Correspondence e-mail: jorge.cabezas@ucr.ac.cr

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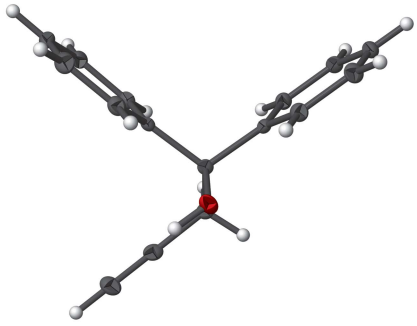
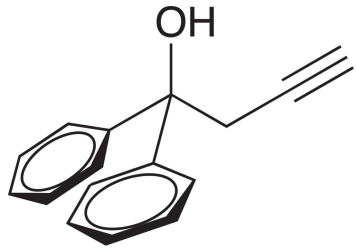
The asymmetric unit of the title compound, $C_{16}H_{14}O$, contains one molecule with a central carbon atom having a distorted tetrahedral geometry made of a propargylic fragment, a hydroxy group and two aromatic rings. Directional interactions such as unusual O—H$\cdots\pi$ contacts are observed between the molecules in the crystal.

3D view



Chemical scheme



Structure description

Homopropargylic alcohols are very useful intermediates in the synthesis of a variety of organic compounds of natural (Kim *et al.*, 2017; Foley & Leighton, 2015; Francais *et al.*, 2010) and synthetic origin (Hosseyni *et al.*, 2016; Gao *et al.*, 2014; Trost & Rhee, 2003; Nicolaou *et al.*, 1990; Yadav & Maiti, 2002). The crystal structure of homopropargyl alcohol 1,1-diphenylbut-3-yn-1-ol is presented herein.

The crystal structure of the title compound comprises a central carbon atom (C7) tetrahedrally bonded to a propargylic moiety, a hydroxy functional group and two phenyl rings (Fig. 1). The bond angles at C7 deviate from the ideal value (109.5°) with angles ranging from 106.13 (11) to 112.06 (11)°, mainly because of the bulky substituent groups attached to this atom. The bond length of the terminal carbon–carbon triple bond (C15≡C16) is 1.190 (2) Å; the propargylic unit (C14–C15–C16) exhibits an angle of 176.26 (15)°, slightly distorted from the linear geometry expected (180°).

In the crystal, the title compound features uncommon O—H$\cdots\pi$ interactions with bond lengths for atom H1 and the C4 and C5 aromatic carbon atoms of 2.72 and 2.80 Å, respectively (symmetry operation $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$) (Fig. 2). Moreover, the hydrogen atom (H16) of the terminal alkyne group has short contacts with carbon atoms of the aromatic ring C9 (2.79 Å) and C10 (2.68 Å), with corresponding symmetry operation $-1 + x, y, z$. Carbon atom C15 of the alkyne fragment accepts an interaction from

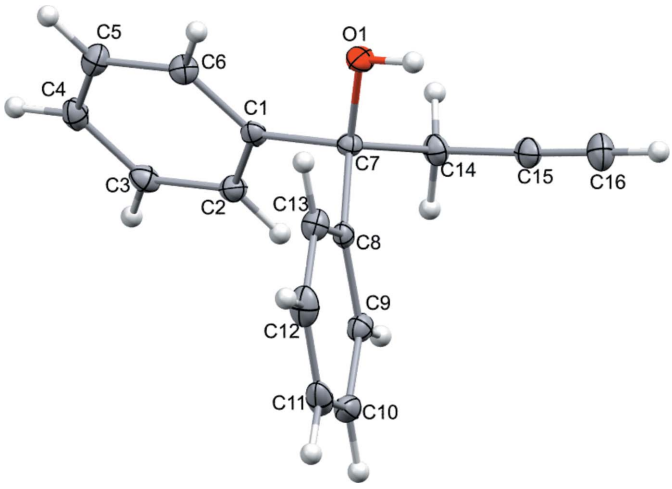


Figure 1
The title molecule with 50% probability ellipsoids.

hydrogen atom H9 bound to aromatic carbon C9 (2.85 Å); symmetry operation $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$.

Synthesis and crystallization

The title compound was synthesized by treatment of propargyl bromide with *n*-BuLi and TMEDA, at −78°C, followed by addition of benzophenone (Fig. 3), according to a previously reported procedure (Cabezas *et al.*, 2001). It was purified by recrystallization from an ethyl ether:hexanes (1:1) solvent mixture to afford colourless block-shaped crystals.

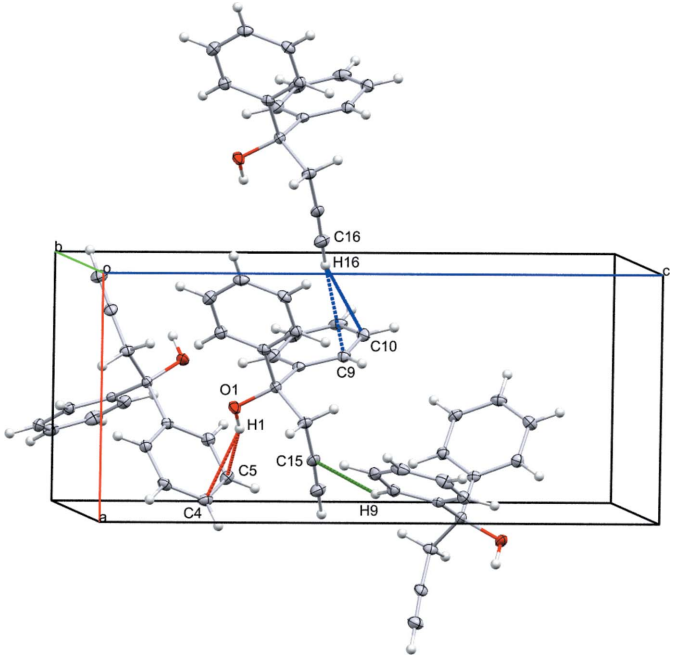


Figure 2
Packing view of the title compound. O–H···π, terminal alkyne hydrogen atom···π and terminal alkyne carbon atom with aromatic hydrogen atom contacts are shown, respectively, as red, blue and green dashed lines.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{14}O$
M_r	222.27
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, b, c (Å)	7.485 (5), 9.173 (7), 16.995 (13)
V (Å^3)	1166.9 (15)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.40 × 0.40 × 0.35
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.701, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	57886, 2688, 2662
R_{int}	0.020
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.652
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.027, 0.075, 1.06
No. of reflections	2688
No. of parameters	157
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.15
Absolute structure	Flack x determined using 1109 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.06 (16)

Computer programs: *APEX3* and *SAINT* (Bruker, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *Mercury* (Macrae *et al.*, 2006).

Refinement

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

Funding information

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References

Bruker (2015). *APEX3*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cabezas, J. A., Pereira, A. & Amey, A. (2001). *Tetrahedron Lett.* **42**, 6819–6822.
Foley, C. N. & Leighton, J. L. (2015). *Org. Lett.* **17**, 5858–5861.
Francais, A., Leyva, A., Etxebarria-Jardi, G. & Ley, S. V. (2010). *Org. Lett.* **12**, 340–343.

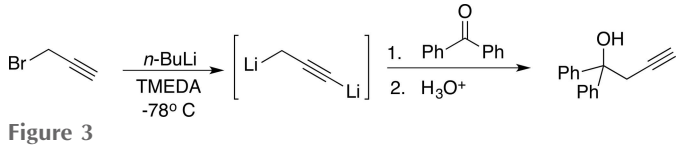

Figure 3
A synthetic scheme for the preparation of the title compound.

Gao, P., Li, H. X., Hao, X. H., Jin, D. P., Chen, D. Q., Yan, X. B., Wu, X. X., Song, X. R., Liu, X. Y. & Liang, Y. M. (2014). *Org. Lett.* **16**, 6298–6301.
Hosseyni, S., Wojtas, L., Li, M. & Shi, X. (2016). *J. Am. Chem. Soc.* **138**, 3994–3997.
Kim, J., Jeong, W. & Rhee, Y. H. (2017). *Org. Lett.* **19**, 242–245.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Nicolaou, K. C., Skokotas, G., Furuya, S., Suemune, H. & Nicolaou, D. C. (1990). *Angew. Chem. Int. Ed. Engl.* **29**, 1064–1067.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Trost, B. M. & Rhee, Y. H. (2003). *J. Am. Chem. Soc.* **125**, 7482–7483.
Yadav, J. S. & Maiti, A. (2002). *Tetrahedron*, **58**, 4955–4961.

full crystallographic data

IUCrData (2018). **3**, x181619 [https://doi.org/10.1107/S241431461801619X]

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1,1-Diphenylbut-3-yn-1-ol

Crystal data

$C_{16}H_{14}O$
M_r = 222.27
Orthorhombic, $P2_12_12_1$
a = 7.485 (5) Å
b = 9.173 (7) Å
c = 16.995 (13) Å
V = 1166.9 (15) Å^3
Z = 4
F(000) = 472
D_x = 1.265 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 115 reflections
θ = 3.2–24.4°
μ = 0.08 mm^{-1}
T = 100 K
Block, colourless
0.40 × 0.40 × 0.35 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: Incoatec Microsource
Mirrors monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2015)
T_{min} = 0.701, T_{max} = 0.746
57886 measured reflections
2688 independent reflections
2662 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.6°, θ_{min} = 2.5°
h = −9→9
k = −11→11
l = −20→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.075
S = 1.06
2688 reflections
157 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 0.2544P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015b)
Extinction coefficient: 0.038 (5)
Absolute structure: Flack x determined using 1109 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.06 (16)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.57457 (13)	0.42888 (11)	0.27540 (5)	0.0168 (2)
H1	0.664 (3)	0.481 (2)	0.2894 (4)	0.025*
C1	0.32959 (17)	0.28343 (14)	0.31357 (8)	0.0126 (3)
C2	0.24610 (18)	0.17965 (14)	0.36076 (8)	0.0153 (3)
H2	0.2889	0.1624	0.4126	0.018*
C3	0.10087 (19)	0.10126 (15)	0.33270 (8)	0.0164 (3)
H3	0.0453	0.0305	0.3654	0.02*
C4	0.03638 (18)	0.12534 (15)	0.25746 (9)	0.0173 (3)
H4	−0.0627	0.0712	0.2383	0.021*
C5	0.1179 (2)	0.22922 (16)	0.21040 (8)	0.0183 (3)
H5	0.0738	0.247	0.1589	0.022*
C6	0.26348 (19)	0.30746 (14)	0.23819 (8)	0.0154 (3)
H6	0.3186	0.3782	0.2054	0.018*
C7	0.48752 (18)	0.37335 (14)	0.34344 (7)	0.0122 (3)
C8	0.41558 (17)	0.49758 (14)	0.39480 (8)	0.0121 (3)
C9	0.37060 (18)	0.47642 (15)	0.47354 (8)	0.0151 (3)
H9	0.3922	0.3846	0.4976	0.018*
C10	0.29437 (19)	0.58841 (16)	0.51711 (8)	0.0188 (3)
H10	0.2627	0.5721	0.5705	0.023*
C11	0.2641 (2)	0.72385 (16)	0.48333 (9)	0.0210 (3)
H11	0.2119	0.8002	0.5133	0.025*
C12	0.3107 (2)	0.74623 (16)	0.40560 (9)	0.0219 (3)
H12	0.2921	0.8391	0.3822	0.026*
C13	0.38473 (19)	0.63376 (16)	0.36129 (8)	0.0166 (3)
H13	0.4145	0.6501	0.3077	0.02*
C14	0.62155 (18)	0.27596 (15)	0.38847 (8)	0.0159 (3)
H14A	0.5632	0.2354	0.4359	0.019*
H14B	0.6585	0.1935	0.3546	0.019*
C15	0.77909 (19)	0.35999 (16)	0.41165 (8)	0.0170 (3)
C16	0.90564 (19)	0.43325 (17)	0.42685 (9)	0.0214 (3)
H16	1.0067	0.4917	0.439	0.026*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0153 (4)	0.0212 (5)	0.0138 (4)	−0.0062 (4)	0.0024 (4)	0.0000 (4)
C1	0.0115 (6)	0.0112 (6)	0.0151 (6)	0.0008 (5)	0.0008 (5)	−0.0032 (5)
C2	0.0157 (6)	0.0161 (6)	0.0140 (6)	−0.0008 (5)	0.0010 (5)	−0.0011 (5)
C3	0.0155 (6)	0.0148 (6)	0.0189 (6)	−0.0023 (5)	0.0038 (5)	−0.0005 (5)

C4	0.0133 (6)	0.0155 (6)	0.0231 (7)	−0.0011 (5)	−0.0010 (5)	−0.0035 (5)
C5	0.0179 (6)	0.0194 (6)	0.0176 (6)	0.0003 (5)	−0.0043 (5)	0.0001 (5)
C6	0.0162 (6)	0.0136 (6)	0.0165 (6)	−0.0006 (5)	0.0000 (5)	0.0013 (5)
C7	0.0120 (6)	0.0129 (6)	0.0117 (6)	−0.0002 (5)	0.0006 (4)	0.0005 (4)
C8	0.0090 (5)	0.0129 (6)	0.0145 (6)	−0.0010 (4)	−0.0016 (4)	−0.0016 (5)
C9	0.0145 (6)	0.0153 (6)	0.0155 (6)	−0.0021 (5)	−0.0015 (5)	−0.0005 (5)
C10	0.0157 (6)	0.0248 (7)	0.0160 (6)	−0.0027 (6)	0.0004 (5)	−0.0063 (5)
C11	0.0141 (6)	0.0192 (6)	0.0297 (8)	0.0033 (5)	−0.0034 (5)	−0.0111 (6)
C12	0.0209 (7)	0.0135 (6)	0.0313 (8)	0.0032 (5)	−0.0068 (6)	−0.0002 (6)
C13	0.0165 (6)	0.0158 (6)	0.0173 (6)	−0.0010 (5)	−0.0030 (5)	0.0010 (5)
C14	0.0139 (6)	0.0143 (6)	0.0195 (6)	0.0020 (5)	−0.0013 (5)	0.0002 (5)
C15	0.0156 (6)	0.0187 (6)	0.0166 (6)	0.0046 (5)	−0.0014 (5)	0.0022 (5)
C16	0.0164 (6)	0.0231 (7)	0.0249 (7)	0.0000 (6)	−0.0046 (5)	0.0024 (6)

Geometric parameters (Å, º)

O1—C7	1.4217 (17)	C8—C13	1.392 (2)
O1—H1	0.86 (2)	C8—C9	1.393 (2)
C1—C6	1.391 (2)	C9—C10	1.389 (2)
C1—C2	1.3928 (19)	C9—H9	0.95
C1—C7	1.5283 (19)	C10—C11	1.387 (2)
C2—C3	1.388 (2)	C10—H10	0.95
C2—H2	0.95	C11—C12	1.382 (2)
C3—C4	1.385 (2)	C11—H11	0.95
C3—H3	0.95	C12—C13	1.392 (2)
C4—C5	1.386 (2)	C12—H12	0.95
C4—H4	0.95	C13—H13	0.95
C5—C6	1.388 (2)	C14—C15	1.463 (2)
C5—H5	0.95	C14—H14A	0.99
C6—H6	0.95	C14—H14B	0.99
C7—C8	1.5332 (19)	C15—C16	1.190 (2)
C7—C14	1.5460 (19)	C16—H16	0.95
C7—O1—H1	109.5	C13—C8—C7	119.50 (12)
C6—C1—C2	118.63 (12)	C9—C8—C7	121.87 (12)
C6—C1—C7	119.71 (12)	C10—C9—C8	120.54 (13)
C2—C1—C7	121.65 (12)	C10—C9—H9	119.7
C3—C2—C1	120.51 (13)	C8—C9—H9	119.7
C3—C2—H2	119.7	C11—C10—C9	120.60 (14)
C1—C2—H2	119.7	C11—C10—H10	119.7
C4—C3—C2	120.52 (13)	C9—C10—H10	119.7
C4—C3—H3	119.7	C12—C11—C10	119.17 (13)
C2—C3—H3	119.7	C12—C11—H11	120.4
C3—C4—C5	119.30 (13)	C10—C11—H11	120.4
C3—C4—H4	120.3	C11—C12—C13	120.50 (14)
C5—C4—H4	120.3	C11—C12—H12	119.8
C4—C5—C6	120.33 (13)	C13—C12—H12	119.8
C4—C5—H5	119.8	C8—C13—C12	120.65 (14)

C6—C5—H5	119.8	C8—C13—H13	119.7
C5—C6—C1	120.72 (12)	C12—C13—H13	119.7
C5—C6—H6	119.6	C15—C14—C7	110.60 (12)
C1—C6—H6	119.6	C15—C14—H14A	109.5
O1—C7—C1	106.13 (11)	C7—C14—H14A	109.5
O1—C7—C8	110.96 (12)	C15—C14—H14B	109.5
C1—C7—C8	108.58 (11)	C7—C14—H14B	109.5
O1—C7—C14	108.20 (11)	H14A—C14—H14B	108.1
C1—C7—C14	110.76 (11)	C16—C15—C14	176.26 (15)
C8—C7—C14	112.06 (11)	C15—C16—H16	180.0
C13—C8—C9	118.54 (12)		

C6—C1—C2—C3	0.50 (19)	C14—C7—C8—C13	142.93 (12)
C7—C1—C2—C3	178.85 (12)	O1—C7—C8—C9	−161.69 (12)
C1—C2—C3—C4	−0.2 (2)	C1—C7—C8—C9	82.05 (15)
C2—C3—C4—C5	−0.3 (2)	C14—C7—C8—C9	−40.62 (17)
C3—C4—C5—C6	0.5 (2)	C13—C8—C9—C10	0.9 (2)
C4—C5—C6—C1	−0.2 (2)	C7—C8—C9—C10	−175.59 (12)
C2—C1—C6—C5	−0.29 (19)	C8—C9—C10—C11	−0.9 (2)
C7—C1—C6—C5	−178.67 (12)	C9—C10—C11—C12	0.0 (2)
C6—C1—C7—O1	−20.69 (16)	C10—C11—C12—C13	1.0 (2)
C2—C1—C7—O1	160.98 (12)	C9—C8—C13—C12	0.1 (2)
C6—C1—C7—C8	98.64 (14)	C7—C8—C13—C12	176.63 (12)
C2—C1—C7—C8	−79.68 (15)	C11—C12—C13—C8	−1.0 (2)
C6—C1—C7—C14	−137.91 (12)	O1—C7—C14—C15	59.66 (14)
C2—C1—C7—C14	43.77 (16)	C1—C7—C14—C15	175.60 (11)
O1—C7—C8—C13	21.86 (17)	C8—C7—C14—C15	−62.99 (15)
C1—C7—C8—C13	−94.40 (14)		