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Homopropargyl alcohol 5,5-diphenylpent-2-yne-1,5-diol

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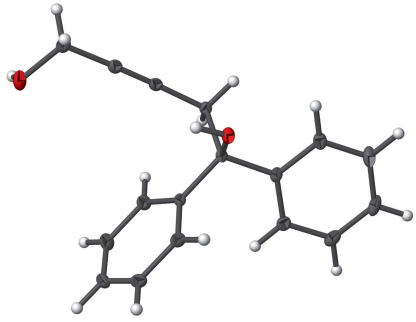
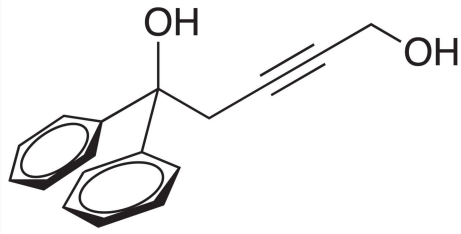
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{16}O_2$, the central carbon atom has a distorted tetrahedral geometry [spread of angles = 105.71 (8)–112.75 (9)°] for its bonds to a homopropargylic but-2-yn-1-ol moiety, a hydroxy group and two phenyl substituents. In the crystal, O—H···O hydrogen-bonding interactions link the molecules into [001] chains and C—H···π(ring) contacts consolidate the packing.

3D view



Chemical scheme



Structure description

Poly-functional homopropargylic alcohols are very useful intermediates in the synthesis of a variety of organic compounds (Kim *et al.*, 2017; Foley & Leighton, 2015; Francais *et al.*, 2010; Hosseyni *et al.*, 2016). Their preparation usually involves multi-step synthesis (Midland *et al.*, 1984). The crystal structure of 5,5-diphenyl-2-pentyn-1,5-diol is reported herein.

The crystal structure of the title compound features a distorted tetrahedral geometry for the central carbon atom (C7) for which the bonding sphere is made of a homopropargylic 2-butyn-1-ol fragment, a hydroxy group and two phenyl groups (Fig. 1). The central carbon atom has angles that deviate from the ideal value (109.4°), mainly because of the bulky phenyl groups attached to it. The dihedral angle between the rings is 80.79 (6)°. The bond length of the carbon-[carbon triple bond (C15≡C16) is 1.190 (2) Å, with the C14—C15—C16 angle being 174.11 (12)°.

In the crystal, O—H···O hydrogen-bonding interactions are observed between molecules whose acceptor atoms are in a different asymmetric unit, forming a ring with an $R(8)$ graph-set motif (Table 1 and Fig. 2) as a component of [001] chains. The packing is consolidated by C—H···π interactions (Table 1 and Fig. 2).

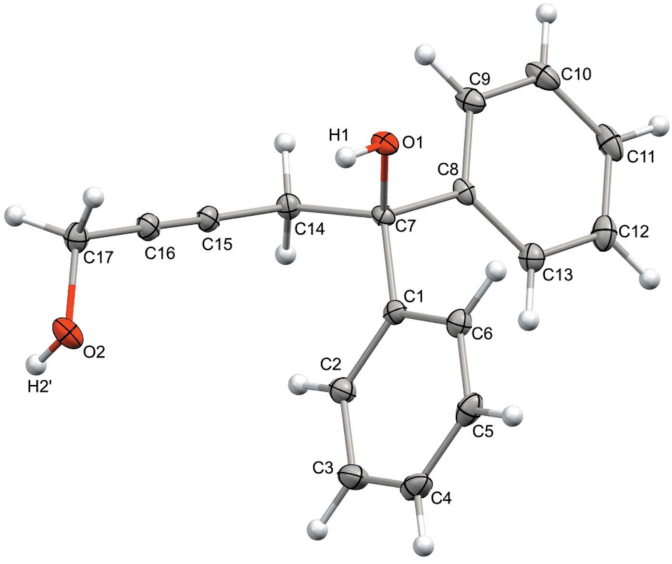


Figure 1
The title molecule with 50% probability ellipsoids.

Synthesis and crystallization

This highly substituted homopropargyl alcohol was synthesized, in a one-pot reaction, by the sequential treatment of propargyl bromide, with *n*-BuLi and TMEDA at −78°C, followed by reaction with benzophenone. The reaction intermediate thus obtained, was treated with paraformaldehyde overnight (Fig. 3), according to the literature procedure (Cabezas *et al.*, 2001). The volatile by-product obtained (2-butyn-1-ol) was removed by Kugelrohr distillation and the residue was purified by column chromatography (ether:hexane) and the product obtained was recrystallized from a mixed ethyl ether:hexanes (1:1) solvent mixture to give colourless block-like crystals.

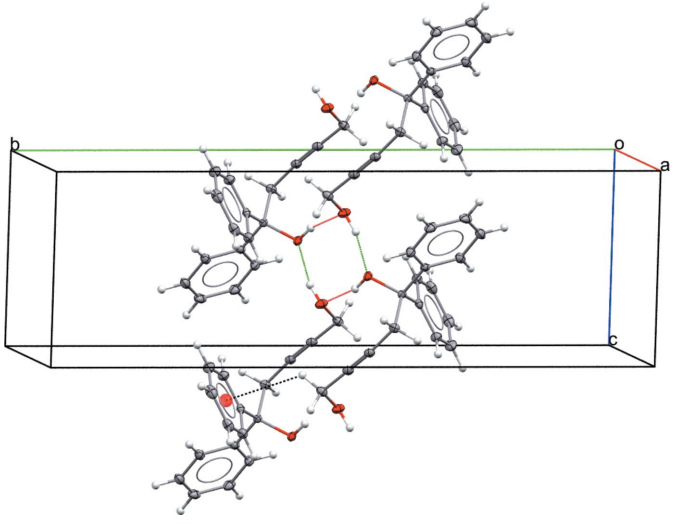


Figure 2
The crystal packing of the title compound. O–H···O hydrogen bonds and the C–H···π(ring) interactions are shown, respectively, as black, green and red dashed lines.

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C8–C13 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.86	1.91	2.7493 (12)	164
O2—H2′···O1^{ii}	0.86	1.87	2.7056 (12)	164
C5—H5···*Cg*2^{iii}	0.95	2.80	3.7122 (13)	160
C17—H17*A*···*Cg*1^{i}	0.99	3.00	3.6172 (13)	122

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{16}O_2$
M_r	252.30
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, *b*, *c* (Å)	7.9772 (3), 22.8528 (8), 7.3075 (3)
β (°)	92.791 (1)
V (Å^3)	1330.59 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.50 × 0.34 × 0.25
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.727, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	65030, 3060, 2668
R_{int}	0.050
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.045, 0.104, 1.03
No. of reflections	3060
No. of parameters	176
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −0.25

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 2.

Funding information

We thank the Sistema de Estudios de Posgrado (SEP), Universidad de Costa Rica (UCR) for a stipend to CAU, and the Vicerrectoría de Investigación (UCR) for financial support.

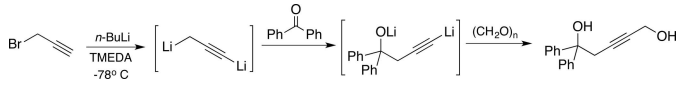


Figure 3
A synthetic scheme for the preparation of the title compound.

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full crystallographic data

IUCrData (2018). **3**, x181618 [https://doi.org/10.1107/S2414314618016188]

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5,5-Diphenylpent-2-yne-1,5-diol

Crystal data

$C_{17}H_{16}O_2$
M_r = 252.30
Monoclinic, $P2_1/c$
a = 7.9772 (3) Å
b = 22.8528 (8) Å
c = 7.3075 (3) Å
β = 92.791 (1)°
V = 1330.59 (9) Å^3
Z = 4
$F(000)$ = 536
D_x = 1.259 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 117 reflections
θ = 3.6–23.1°
μ = 0.08 mm^{-1}
T = 100 K
Block, colourless
0.50 × 0.34 × 0.25 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} = 0.727, T_{max} = 0.746
65030 measured reflections
3060 independent reflections
2668 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 27.5°, θ_{min} = 2.6°
h = −8→10
k = −29→29
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.104
S = 1.03
3060 reflections
176 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
$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.6384P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.27538 (10)	0.54181 (3)	0.43375 (11)	0.01258 (19)
H1	0.345 (2)	0.5263 (5)	0.3615 (17)	0.019*
O2	0.46943 (11)	0.51425 (4)	−0.26458 (12)	0.0171 (2)
H2′	0.4133 (13)	0.5299 (7)	−0.355 (2)	0.026*
C1	0.34385 (14)	0.63310 (5)	0.28443 (15)	0.0115 (2)
C2	0.33084 (15)	0.66221 (5)	0.11643 (17)	0.0159 (2)
H2	0.2316	0.6583	0.0402	0.019*
C3	0.46201 (17)	0.69690 (6)	0.05955 (18)	0.0206 (3)
H3	0.4515	0.7166	−0.0549	0.025*
C4	0.60738 (17)	0.70284 (5)	0.16880 (18)	0.0202 (3)
H4	0.6968	0.7264	0.1294	0.024*
C5	0.62206 (15)	0.67418 (5)	0.33639 (18)	0.0178 (3)
H5	0.722	0.678	0.4116	0.021*
C6	0.49087 (15)	0.63986 (5)	0.39436 (16)	0.0149 (2)
H6	0.5013	0.6208	0.5099	0.018*
C7	0.20382 (14)	0.59370 (5)	0.35016 (15)	0.0104 (2)
C8	0.10203 (14)	0.62267 (5)	0.49755 (15)	0.0117 (2)
C9	−0.01385 (15)	0.58905 (5)	0.58940 (17)	0.0159 (2)
H9	−0.0277	0.5488	0.5593	0.019*
C10	−0.10905 (16)	0.61360 (6)	0.72402 (17)	0.0196 (3)
H10	−0.1869	0.5902	0.7857	0.024*
C11	−0.09031 (16)	0.67244 (6)	0.76825 (17)	0.0201 (3)
H11	−0.1553	0.6893	0.8601	0.024*
C12	0.02357 (16)	0.70637 (6)	0.67798 (17)	0.0192 (3)
H12	0.0364	0.7466	0.7079	0.023*
C13	0.11950 (15)	0.68163 (5)	0.54327 (16)	0.0150 (2)
H13	0.1974	0.7052	0.4822	0.018*
C14	0.08274 (14)	0.57347 (5)	0.19017 (15)	0.0127 (2)
H14A	−0.0068	0.5488	0.2391	0.015*
H14B	0.0291	0.6081	0.1309	0.015*
C15	0.17153 (14)	0.54009 (5)	0.05346 (16)	0.0127 (2)
C16	0.25424 (14)	0.51196 (5)	−0.04534 (16)	0.0138 (2)
C17	0.36115 (15)	0.47781 (5)	−0.16380 (16)	0.0153 (2)
H17A	0.43	0.4502	−0.0875	0.018*
H17B	0.2892	0.4545	−0.2507	0.018*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0132 (4)	0.0123 (4)	0.0125 (4)	0.0038 (3)	0.0025 (3)	0.0022 (3)
O2	0.0142 (4)	0.0248 (5)	0.0124 (4)	0.0041 (3)	0.0021 (3)	0.0033 (3)
C1	0.0112 (5)	0.0105 (5)	0.0130 (5)	0.0007 (4)	0.0032 (4)	−0.0014 (4)
C2	0.0163 (6)	0.0165 (5)	0.0149 (6)	−0.0008 (4)	0.0010 (4)	0.0016 (4)
C3	0.0261 (7)	0.0181 (6)	0.0183 (6)	−0.0035 (5)	0.0070 (5)	0.0029 (5)
C4	0.0191 (6)	0.0173 (6)	0.0252 (7)	−0.0058 (5)	0.0109 (5)	−0.0044 (5)

C5	0.0122 (5)	0.0179 (6)	0.0234 (6)	−0.0019 (4)	0.0021 (5)	−0.0071 (5)
C6	0.0147 (5)	0.0148 (5)	0.0152 (6)	0.0004 (4)	0.0006 (4)	−0.0018 (4)
C7	0.0101 (5)	0.0108 (5)	0.0102 (5)	0.0009 (4)	0.0008 (4)	0.0013 (4)
C8	0.0107 (5)	0.0150 (5)	0.0095 (5)	0.0031 (4)	−0.0004 (4)	0.0004 (4)
C9	0.0174 (6)	0.0148 (5)	0.0160 (6)	0.0029 (4)	0.0043 (4)	0.0030 (4)
C10	0.0201 (6)	0.0240 (6)	0.0154 (6)	0.0057 (5)	0.0067 (5)	0.0054 (5)
C11	0.0211 (6)	0.0272 (6)	0.0122 (5)	0.0094 (5)	0.0034 (5)	−0.0015 (5)
C12	0.0212 (6)	0.0183 (6)	0.0180 (6)	0.0042 (5)	−0.0004 (5)	−0.0057 (5)
C13	0.0139 (5)	0.0160 (5)	0.0151 (6)	0.0010 (4)	0.0005 (4)	−0.0014 (4)
C14	0.0103 (5)	0.0156 (5)	0.0122 (5)	−0.0006 (4)	0.0009 (4)	−0.0013 (4)
C15	0.0108 (5)	0.0153 (5)	0.0118 (5)	−0.0017 (4)	−0.0016 (4)	0.0000 (4)
C16	0.0116 (5)	0.0170 (5)	0.0126 (5)	−0.0015 (4)	−0.0015 (4)	−0.0002 (4)
C17	0.0134 (5)	0.0173 (5)	0.0151 (6)	0.0006 (4)	0.0009 (4)	−0.0029 (4)

Geometric parameters (Å, º)

O1—C7	1.4391 (13)	C8—C13	1.3936 (16)
O1—H1	0.860 (17)	C8—C9	1.3985 (16)
O2—C17	1.4296 (14)	C9—C10	1.3900 (16)
O2—H2′	0.857 (18)	C9—H9	0.95
C1—C2	1.3957 (16)	C10—C11	1.3892 (19)
C1—C6	1.3974 (16)	C10—H10	0.95
C1—C7	1.5302 (15)	C11—C12	1.3856 (19)
C2—C3	1.3923 (17)	C11—H11	0.95
C2—H2	0.95	C12—C13	1.3956 (16)
C3—C4	1.3822 (19)	C12—H12	0.95
C3—H3	0.95	C13—H13	0.95
C4—C5	1.3888 (19)	C14—C15	1.4665 (15)
C4—H4	0.95	C14—H14A	0.99
C5—C6	1.3903 (16)	C14—H14B	0.99
C5—H5	0.95	C15—C16	1.1900 (17)
C6—H6	0.95	C16—C17	1.4690 (16)
C7—C8	1.5307 (15)	C17—H17A	0.99
C7—C14	1.5503 (15)	C17—H17B	0.99
C7—O1—H1	109.5	C10—C9—C8	120.97 (11)
C17—O2—H2′	109.5	C10—C9—H9	119.5
C2—C1—C6	118.51 (11)	C8—C9—H9	119.5
C2—C1—C7	122.17 (10)	C11—C10—C9	119.94 (12)
C6—C1—C7	119.32 (10)	C11—C10—H10	120.0
C3—C2—C1	120.61 (12)	C9—C10—H10	120.0
C3—C2—H2	119.7	C12—C11—C10	119.77 (11)
C1—C2—H2	119.7	C12—C11—H11	120.1
C4—C3—C2	120.34 (12)	C10—C11—H11	120.1
C4—C3—H3	119.8	C11—C12—C13	120.25 (11)
C2—C3—H3	119.8	C11—C12—H12	119.9
C3—C4—C5	119.69 (11)	C13—C12—H12	119.9
C3—C4—H4	120.2	C8—C13—C12	120.59 (11)

C5—C4—H4	120.2	C8—C13—H13	119.7
C4—C5—C6	120.15 (12)	C12—C13—H13	119.7
C4—C5—H5	119.9	C15—C14—C7	111.41 (9)
C6—C5—H5	119.9	C15—C14—H14A	109.3
C5—C6—C1	120.69 (11)	C7—C14—H14A	109.3
C5—C6—H6	119.7	C15—C14—H14B	109.3
C1—C6—H6	119.7	C7—C14—H14B	109.3
O1—C7—C1	109.72 (9)	H14A—C14—H14B	108.0
O1—C7—C8	105.71 (8)	C16—C15—C14	174.11 (12)
C1—C7—C8	112.75 (9)	C15—C16—C17	178.17 (13)
O1—C7—C14	107.11 (9)	O2—C17—C16	112.13 (10)
C1—C7—C14	112.10 (9)	O2—C17—H17A	109.2
C8—C7—C14	109.10 (9)	C16—C17—H17A	109.2
C13—C8—C9	118.48 (11)	O2—C17—H17B	109.2
C13—C8—C7	122.46 (10)	C16—C17—H17B	109.2
C9—C8—C7	119.06 (10)	H17A—C17—H17B	107.9

C6—C1—C2—C3	0.37 (17)	C14—C7—C8—C13	−115.62 (12)
C7—C1—C2—C3	−178.85 (11)	O1—C7—C8—C9	−50.95 (13)
C1—C2—C3—C4	0.18 (19)	C1—C7—C8—C9	−170.83 (10)
C2—C3—C4—C5	−0.24 (19)	C14—C7—C8—C9	63.93 (13)
C3—C4—C5—C6	−0.26 (18)	C13—C8—C9—C10	−0.35 (18)
C4—C5—C6—C1	0.83 (18)	C7—C8—C9—C10	−179.92 (11)
C2—C1—C6—C5	−0.88 (17)	C8—C9—C10—C11	0.34 (19)
C7—C1—C6—C5	178.37 (10)	C9—C10—C11—C12	−0.10 (19)
C2—C1—C7—O1	138.56 (11)	C10—C11—C12—C13	−0.11 (19)
C6—C1—C7—O1	−40.66 (13)	C9—C8—C13—C12	0.13 (17)
C2—C1—C7—C8	−103.91 (12)	C7—C8—C13—C12	179.69 (10)
C6—C1—C7—C8	76.87 (13)	C11—C12—C13—C8	0.09 (18)
C2—C1—C7—C14	19.69 (14)	O1—C7—C14—C15	−60.24 (11)
C6—C1—C7—C14	−159.53 (10)	C1—C7—C14—C15	60.16 (12)
O1—C7—C8—C13	129.49 (11)	C8—C7—C14—C15	−174.22 (9)
C1—C7—C8—C13	9.62 (15)		

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C1–C6 and C8–C13 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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C17—H17*A*···*Cg*1^{i}	0.99	3.00	3.6172 (13)	122

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