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1-Methoxy-1,1-diphenylbut-2-yne

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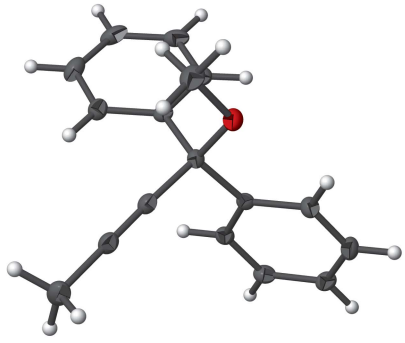
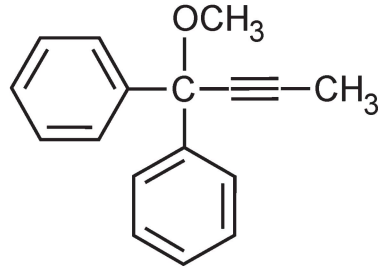
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{16}O$, the phenyl rings are twisted relative to each other at an angle of 85.9 (1)°. The crystal structure features weak C—H$\cdots\pi$ interactions, which connect the molecules into a three-dimensional supramolecular network.

3D view



Chemical scheme



Structure description

The title compound has been prepared as an intermediate for the synthesis of a potential 'wheel-and-axle' molecule (Weber, 1996). Compounds of this latter type are significant crystalline inclusion hosts (Katzsch *et al.*, 2015, 2016) and important examples in the course of the development of the concept of crystal engineering (Hart *et al.*, 1984; Bishop, 2012). Alternative approaches for the synthesis of the title compound have already been reported (Van Rijn *et al.*, 1981; Kostikov *et al.*, 1996; Maraval *et al.*, 2008). One of these, which is closely related to the method we used for the synthesis of the compound, resulted in a yellow oil, while our preparative method yielded the compound as colourless crystals that were used for X-ray crystal structure analysis.

The asymmetric unit of the cell contains one molecule (Fig. 1), the aromatic rings of which are tilted to one another at an angle of 85.9 (1)°. The propyne unit of the molecule slightly deviates from linearity, showing an angle of 177.64 (12)° at C14. In the crystal structure (Fig. 2), the molecules are packed in neither a layered nor a stacked manner, but are connected *via* C—H$\cdots\pi$ interactions (Nishio *et al.*, 2009). These involve the aromatic rings and the C≡C triple bond acting as acceptors (Table 1). The oxygen atom of the molecule does not participate in a comparable weak hydrogen bond (Desiraju & Steiner, 1999), probably due to steric hindrance.

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

*Cg*1 and *Cg*2 are the centroids of the C2–C7 and C8–C13 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17*A*···*Cg*1^{i}	0.98	2.81	3.423 (1)	121
C5—H5···*Cg*2^{ii}	0.95	2.73	3.660 (1)	166

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

Synthesis and crystallization

Under an atmosphere of argon, 1,1-diphenylprop-2-yne-1-ol (5.2 g, 25 mmol) was added to a stirred suspension of sodium hydride (6.0 g, 250 mmol, 10% in paraffin oil) in dry THF (250 ml). After stirring for 30 min, methyl iodide (21 ml, 330 mmol) was added and the mixture was heated to reflux for 16 h. After cooling, the mixture was quenched with water (50 ml) and extracted with diethyl ether. The combined organic layers were dried over sodium sulfate and the solvents evaporated to dryness, thus giving colourless crystals (5.8 g, 98%) with m.p. 329 K. ^{1}H NMR (500.1 MHz, $CDCl_3$): δ = 1.98 (*s*, 3H, C—CH_3), 3.31 (*s*, 3H, OCH_3), 7.26–7.27 (*m*, 8H, Ar—H), 7.52–7.54 (*m*, 2H, Ar—H) p.p.m. ^{13}C NMR (125.8 MHz, $CDCl_3$): δ = 3.7 (C—*C*H_3), 52.1 (O—*C*H_3), 78.6 (Ar—C—*C*≡C), 80.8 (Ar—*C*–-O), 85.7 (C≡*C*—CH_3), 126.7 (Ar), 127.3 (Ar), 127.9 (Ar), 143.9 (Ar) p.p.m. IR (KBr): ν = 2230 cm^{-1} (C≡C); GC—MS: *m*/*z* = 237 $[M]^+$. Colourless plates were grown *via* slow evaporation of solvent from a 1:1 solvent mixture of ethanol and THF.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

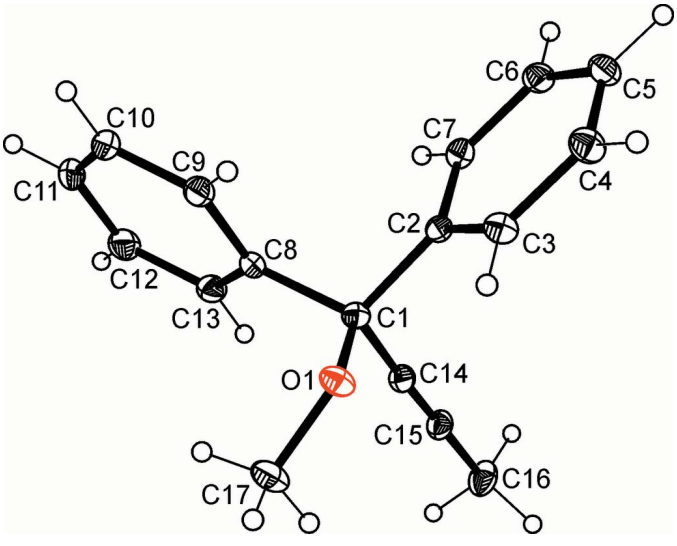


Figure 1
Perspective view of the title compound, showing 50% probability displacement ellipsoids for non-H atoms.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{16}O$
M_r	236.30
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, *b*, *c* (Å)	9.6483 (2), 10.6060 (3), 13.4332 (4)
β (°)	104.034 (1)
V (Å^3)	1333.59 (6)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.07
Crystal size (mm)	0.50 × 0.46 × 0.35
Data collection	
Diffractometer	Bruker Kappa goniometer with an APEXII CCD area detector
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13834, 2939, 2582
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.641
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.040, 0.112, 1.06
No. of reflections	2939
No. of parameters	165
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.31

Computer programs: *APEX2* and *SAINT-NT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

Funding information

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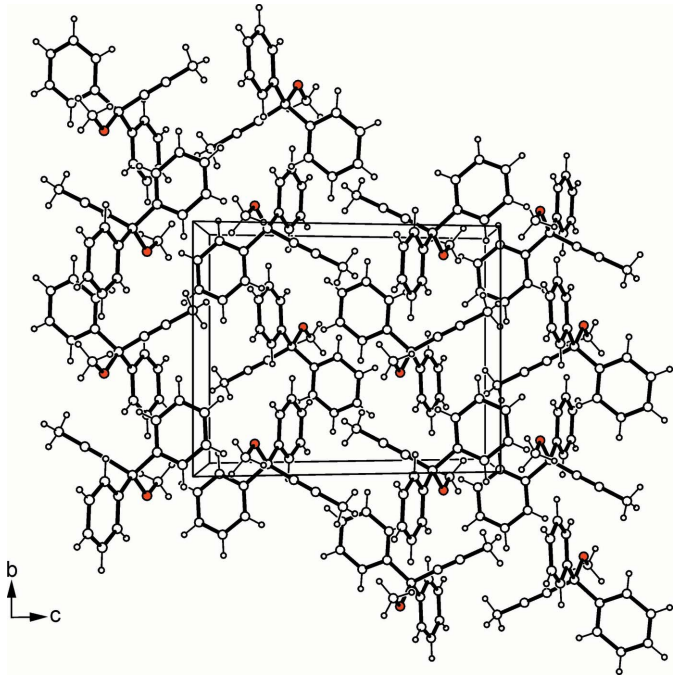

Figure 2
Packing diagram of the title compound viewed down the *a* axis.

References

Bishop, R. (2012). In *Supramolecular Chemistry: From Molecules to Nanomaterials*, edited by P. A. Gale & J. W. Steed, p. 3033. Chichester: Wiley.

Bruker (2008). *APEX2* and *SAINT-NT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond*. IUCr Monographs on Crystallography, Vol. 9, ch. 3. Oxford University Press.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Hart, H., Lin, L. T.-W. & Ward, D. L. (1984). *J. Am. Chem. Soc.* **106**, 4043–4045.

Katzsch, F., Gruber, T. & Weber, E. (2015). *Cryst. Growth Des.* **15**, 5047–5061.

Katzsch, F., Gruber, T. & Weber, E. (2016). *J. Mol. Struct.* **1114**, 48–64.

Kostikov, R. R., Varakin, G. S., Molchanov, A. P. & Oglobin, K. A. (1996). *Russ. J. Org. Chem.* **32**, 31–35.

Maraval, V., Duhayon, C., Coppel, Y. & Chauvin, R. (2008). *Eur. J. Org. Chem.* pp. 5144–5156.

Nishio, M., Umezawa, Y., Honda, K., Tsuboyama, S. & Suezawa, H. (2009). *CrystEngComm*, **11**, 1757–1788.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Van Rijn, P. E., Mommers, S., Visser, R. G., Verkruijsse, H. D. & Brandsma, L. (1981). *Synthesis*, pp. 459–460.

Weber, E. (1996). In *Comprehensive Supramolecular Chemistry*, edited by D. D. MacNicol, F. Toda & R. Bishop, Vol. 6, pp. 535–592. Oxford, UK: Wiley.

full crystallographic data

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Crystal data

$C_{17}H_{16}O$
M_r = 236.30
Monoclinic, $P2_1/c$
a = 9.6483 (2) Å
b = 10.6060 (3) Å
c = 13.4332 (4) Å
β = 104.034 (1)°
V = 1333.59 (6) Å^3
Z = 4
$F(000)$ = 504
D_x = 1.177 Mg m^{-3}
Melting point: 329 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8020 reflections
θ = 2.4–33.1°
μ = 0.07 mm^{-1}
T = 100 K
Irregular, colourless
0.50 × 0.46 × 0.35 mm

Data collection

Bruker Kappa goniometer with an APEXII CCD area detector diffractometer
Radiation source: Sealed X-ray tube
φ and ω scans
13834 measured reflections
2939 independent reflections
2582 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.1°, θ_{min} = 2.2°
h = −12→11
k = −13→9
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.112
S = 1.06
2939 reflections
165 parameters
0 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.6393P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Special details

Refinement. The non-hydrogen atoms were refined anisotropically. The hydrogen atoms were positioned geometrically and refined isotropically using the riding model with C—H = 0.98 Å and U_{iso}(H) = 1.5 U_{eq}(C) for methyl groups, and C—H = 0.95 Å and U_{iso}(H) = 1.2 U_{eq}(C) for phenyl groups.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.44623 (8)	−0.10133 (8)	0.17564 (6)	0.0189 (2)
C1	0.36399 (11)	−0.01102 (11)	0.21645 (8)	0.0148 (2)
C2	0.24889 (11)	−0.08676 (11)	0.25168 (8)	0.0146 (2)
C3	0.25175 (12)	−0.21765 (11)	0.25575 (9)	0.0183 (2)
H3	0.3230	−0.2629	0.2326	0.022*
C4	0.14980 (13)	−0.28244 (12)	0.29387 (9)	0.0221 (3)
H4	0.1520	−0.3720	0.2964	0.027*
C5	0.04549 (12)	−0.21788 (12)	0.32810 (9)	0.0217 (3)
H5	−0.0230	−0.2626	0.3546	0.026*
C6	0.04201 (12)	−0.08689 (12)	0.32339 (9)	0.0205 (3)
H6	−0.0295	−0.0418	0.3464	0.025*
C7	0.14275 (12)	−0.02197 (11)	0.28520 (8)	0.0176 (2)
H7	0.1395	0.0675	0.2818	0.021*
C8	0.29562 (11)	0.08338 (11)	0.13200 (8)	0.0158 (2)
C9	0.19931 (12)	0.03667 (12)	0.04470 (9)	0.0200 (3)
H9	0.1779	−0.0509	0.0394	0.024*
C10	0.13505 (13)	0.11747 (14)	−0.03405 (9)	0.0245 (3)
H10	0.0689	0.0854	−0.0929	0.029*
C11	0.16720 (13)	0.24534 (14)	−0.02706 (10)	0.0269 (3)
H11	0.1226	0.3008	−0.0808	0.032*
C12	0.26422 (13)	0.29179 (13)	0.05828 (10)	0.0256 (3)
H12	0.2873	0.3790	0.0625	0.031*
C13	0.32831 (12)	0.21100 (11)	0.13811 (9)	0.0200 (3)
H13	0.3945	0.2434	0.1968	0.024*
C14	0.45375 (12)	0.05219 (11)	0.30794 (9)	0.0173 (2)
C15	0.52478 (12)	0.09973 (11)	0.38375 (9)	0.0194 (2)
C16	0.56975 (13)	−0.05142 (13)	0.14930 (10)	0.0246 (3)
H16A	0.5419	0.0189	0.1013	0.037*
H16B	0.6149	−0.1174	0.1170	0.037*
H16C	0.6374	−0.0214	0.2115	0.037*
C17	0.61243 (14)	0.15804 (13)	0.47669 (10)	0.0264 (3)
H17A	0.6757	0.0944	0.5170	0.040*
H17B	0.5503	0.1929	0.5177	0.040*
H17C	0.6700	0.2258	0.4576	0.040*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0169 (4)	0.0170 (4)	0.0252 (4)	0.0001 (3)	0.0099 (3)	−0.0019 (3)
C1	0.0141 (5)	0.0138 (5)	0.0163 (5)	−0.0004 (4)	0.0036 (4)	−0.0010 (4)
C2	0.0139 (5)	0.0168 (5)	0.0121 (5)	−0.0017 (4)	0.0013 (4)	−0.0006 (4)
C3	0.0171 (5)	0.0173 (6)	0.0211 (5)	−0.0008 (4)	0.0056 (4)	−0.0023 (4)
C4	0.0229 (6)	0.0172 (6)	0.0265 (6)	−0.0041 (5)	0.0066 (5)	−0.0011 (5)
C5	0.0189 (6)	0.0262 (6)	0.0207 (6)	−0.0057 (5)	0.0063 (4)	0.0007 (5)
C6	0.0176 (5)	0.0259 (6)	0.0190 (5)	0.0022 (4)	0.0063 (4)	−0.0008 (5)

C7	0.0193 (5)	0.0173 (6)	0.0160 (5)	0.0016 (4)	0.0038 (4)	0.0001 (4)
C8	0.0139 (5)	0.0190 (6)	0.0154 (5)	−0.0001 (4)	0.0052 (4)	0.0014 (4)
C9	0.0184 (5)	0.0241 (6)	0.0180 (5)	−0.0032 (4)	0.0050 (4)	−0.0013 (5)
C10	0.0184 (6)	0.0394 (8)	0.0152 (5)	−0.0024 (5)	0.0029 (4)	0.0026 (5)
C11	0.0215 (6)	0.0366 (7)	0.0235 (6)	0.0031 (5)	0.0068 (5)	0.0143 (5)
C12	0.0229 (6)	0.0218 (6)	0.0328 (7)	−0.0006 (5)	0.0078 (5)	0.0089 (5)
C13	0.0171 (5)	0.0201 (6)	0.0226 (6)	−0.0022 (4)	0.0040 (4)	0.0013 (5)
C14	0.0166 (5)	0.0163 (5)	0.0185 (5)	−0.0009 (4)	0.0035 (4)	0.0033 (4)
C15	0.0204 (5)	0.0179 (6)	0.0186 (5)	−0.0014 (4)	0.0026 (4)	0.0038 (4)
C16	0.0191 (6)	0.0259 (6)	0.0325 (7)	−0.0004 (5)	0.0134 (5)	0.0008 (5)
C17	0.0300 (7)	0.0262 (7)	0.0185 (6)	−0.0062 (5)	−0.0025 (5)	0.0020 (5)

Geometric parameters (Å, º)

O1—C16	1.4248 (14)	C9—C10	1.3855 (17)
O1—C1	1.4347 (13)	C9—H9	0.9500
C1—C14	1.4811 (15)	C10—C11	1.389 (2)
C1—C2	1.5354 (15)	C10—H10	0.9500
C1—C8	1.5367 (15)	C11—C12	1.3830 (19)
C2—C3	1.3893 (16)	C11—H11	0.9500
C2—C7	1.3955 (15)	C12—C13	1.3947 (17)
C3—C4	1.3951 (16)	C12—H12	0.9500
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.3848 (17)	C14—C15	1.1928 (17)
C4—H4	0.9500	C15—C17	1.4643 (16)
C5—C6	1.3908 (18)	C16—H16A	0.9800
C5—H5	0.9500	C16—H16B	0.9800
C6—C7	1.3871 (16)	C16—H16C	0.9800
C6—H6	0.9500	C17—H17A	0.9800
C7—H7	0.9500	C17—H17B	0.9800
C8—C13	1.3877 (16)	C17—H17C	0.9800
C8—C9	1.3984 (15)		
C16—O1—C1	114.85 (9)	C10—C9—H9	119.9
O1—C1—C14	110.67 (9)	C8—C9—H9	119.9
O1—C1—C2	106.07 (9)	C9—C10—C11	120.07 (11)
C14—C1—C2	107.59 (9)	C9—C10—H10	120.0
O1—C1—C8	109.33 (8)	C11—C10—H10	120.0
C14—C1—C8	112.19 (9)	C12—C11—C10	119.93 (11)
C2—C1—C8	110.81 (8)	C12—C11—H11	120.0
C3—C2—C7	119.23 (10)	C10—C11—H11	120.0
C3—C2—C1	121.74 (10)	C11—C12—C13	120.21 (12)
C7—C2—C1	118.95 (10)	C11—C12—H12	119.9
C2—C3—C4	119.85 (11)	C13—C12—H12	119.9
C2—C3—H3	120.1	C8—C13—C12	120.12 (11)
C4—C3—H3	120.1	C8—C13—H13	119.9
C5—C4—C3	120.81 (11)	C12—C13—H13	119.9
C5—C4—H4	119.6	C15—C14—C1	177.64 (12)

C3—C4—H4	119.6	C14—C15—C17	179.79 (14)
C4—C5—C6	119.36 (11)	O1—C16—H16A	109.5
C4—C5—H5	120.3	O1—C16—H16B	109.5
C6—C5—H5	120.3	H16A—C16—H16B	109.5
C7—C6—C5	120.09 (11)	O1—C16—H16C	109.5
C7—C6—H6	120.0	H16A—C16—H16C	109.5
C5—C6—H6	120.0	H16B—C16—H16C	109.5
C6—C7—C2	120.66 (11)	C15—C17—H17A	109.5
C6—C7—H7	119.7	C15—C17—H17B	109.5
C2—C7—H7	119.7	H17A—C17—H17B	109.5
C13—C8—C9	119.37 (11)	C15—C17—H17C	109.5
C13—C8—C1	122.75 (10)	H17A—C17—H17C	109.5
C9—C8—C1	117.86 (10)	H17B—C17—H17C	109.5
C10—C9—C8	120.29 (12)		

C16—O1—C1—C14	−56.68 (12)	C1—C2—C7—C6	−176.11 (10)
C16—O1—C1—C2	−173.08 (9)	O1—C1—C8—C13	−116.64 (11)
C16—O1—C1—C8	67.39 (11)	C14—C1—C8—C13	6.52 (15)
O1—C1—C2—C3	9.86 (13)	C2—C1—C8—C13	126.80 (11)
C14—C1—C2—C3	−108.60 (11)	O1—C1—C8—C9	61.74 (12)
C8—C1—C2—C3	128.42 (11)	C14—C1—C8—C9	−175.10 (10)
O1—C1—C2—C7	−173.33 (9)	C2—C1—C8—C9	−54.83 (13)
C14—C1—C2—C7	68.21 (12)	C13—C8—C9—C10	−1.25 (17)
C8—C1—C2—C7	−54.77 (12)	C1—C8—C9—C10	−179.68 (10)
C7—C2—C3—C4	−0.53 (16)	C8—C9—C10—C11	0.66 (17)
C1—C2—C3—C4	176.27 (10)	C9—C10—C11—C12	0.46 (18)
C2—C3—C4—C5	−0.15 (17)	C10—C11—C12—C13	−0.98 (19)
C3—C4—C5—C6	0.59 (17)	C9—C8—C13—C12	0.73 (17)
C4—C5—C6—C7	−0.33 (17)	C1—C8—C13—C12	179.08 (10)
C5—C6—C7—C2	−0.35 (17)	C11—C12—C13—C8	0.38 (18)
C3—C2—C7—C6	0.78 (16)		

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C2–C7 and C8–C13 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17*A*···*Cg*1^{i}	0.98	2.81	3.423 (1)	121
C5—H5···*Cg*2^{ii}	0.95	2.73	3.660 (1)	166

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