Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(4-Methoxyphenyl)but-3-yn-1-ol

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Key indicators

Single-crystal X-ray study
T = 144 K
Mean σ(C–C) = 0.002 Å
R factor = 0.069
wR factor = 0.139
Data-to-parameter ratio = 29.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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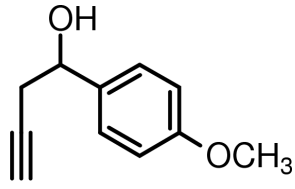
Received 28 May 2002
Accepted 31 May 2002
Online 14 June 2002

Molecules of the title compound, $C_{11}H_{12}O_2$, are connected by intermolecular O—H···O hydrogen bonds to form helices running in the *b* direction. These helices are further stabilized by intermolecular C—H···π(phenyl) interactions.

Comment

As part of attempts to demonstrate the versatility of allenyl ketones as building blocks in transition-metal-catalysed organic reactions, we prepared the precursor 1-(4-methoxyphenyl)but-3-yn-1-ol, (I) (Hashmi *et al.*, 1997). The structure contains two crystallographically independent molecules (Fig. 1). The dimensions of both molecules are very similar: corresponding bond lengths agree within experimental uncertainty, corresponding bond angles within 1°. Corresponding torsion angles in the side groups attached to C5 and C16 differ by up to 3°, while the orientations of the methoxy groups differ by 7°.



(I)

The molecules are connected by intermolecular hydrogen bonding between the hydroxyl groups to form helices running in the crystallographic *b* direction (Fig. 2 and Table 1). One full turn of the helix corresponds to four molecules. The pitch height of the helix corresponds to the length of the *b* axis. The helices are further stabilized by additional C—H···π(phenyl) interactions, C4—H4···*Cg*2 and C14—H14*A*···*Cg*1, where *Cg*1 and *Cg*2 represent the centroids of the phenyl rings of molecules 1 and 2 (Fig. 3). The latter interaction is rather short, with an H···*Cg* distance of 2.58 Å, but is still in the range for such interactions, as reported by Desiraju & Steiner (1999). Neighboring helices are connected by an intermolecular C—H(alkyne)···π(phenyl) interaction (C12—H12···*Cg*2) and four very weak intermolecular C—H···O interactions, with H···O distances between 2.63 and 2.75 Å.

Experimental

The preparation of the title compound by the addition of allenylmagnesium bromide to 4-methoxybenzaldehyde has been reported by Hashmi *et al.* (1997). Single crystals were obtained by recrystallization from diethyl ether at 253 K.

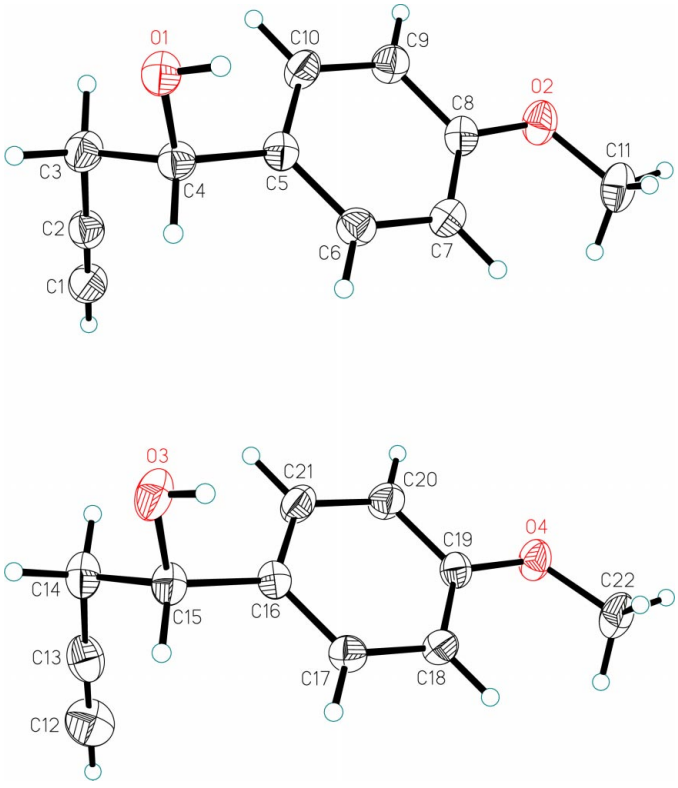


Figure 1
The structure of (I), with 50% probability displacement ellipsoids, for (top) molecule 1 and (bottom) molecule 2.

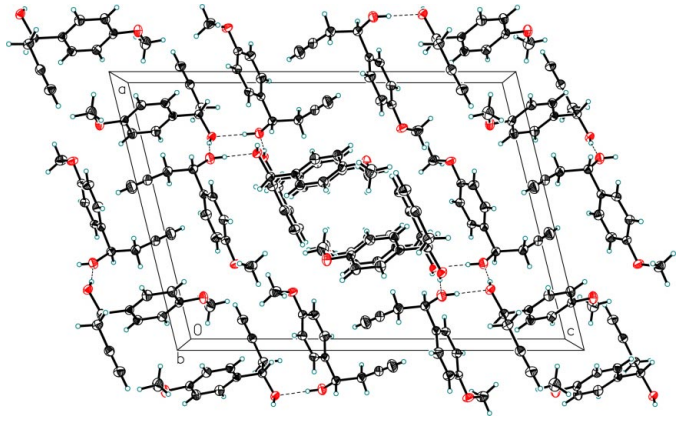


Figure 2
The crystal packing of (I), viewed down *b*.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H01···O3^{i}	0.84	1.90	2.7290 (17)	170
O3—H03···O1^{ii}	0.84	1.90	2.7372 (16)	179
C4—H4···*Cg*2^{iii}	1.00	2.80	3.704 (2)	151
C12—H12···*Cg*2^{iv}	0.95	2.86	3.791 (2)	166
C14—H14*A*···*Cg*1^{v}	0.99	2.58	3.559 (2)	169

Symmetry codes: (i) $x, 1+y, z$; (ii) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iv) $1-x, -y, -z$; (v) $x, y-1, z$.

The H atoms were obtained from difference Fourier syntheses. They were constrained with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C), U_{iso}(H) = 1.5U_{eq}(C_{methyl}), U_{iso}(H) = 1.5U_{eq}(O)], using a riding model with fixed distances: H—C = 0.98 Å for methyl, 0.99 Å for secondary, 1.00 Å for primary, 0.95 Å for aromatic and

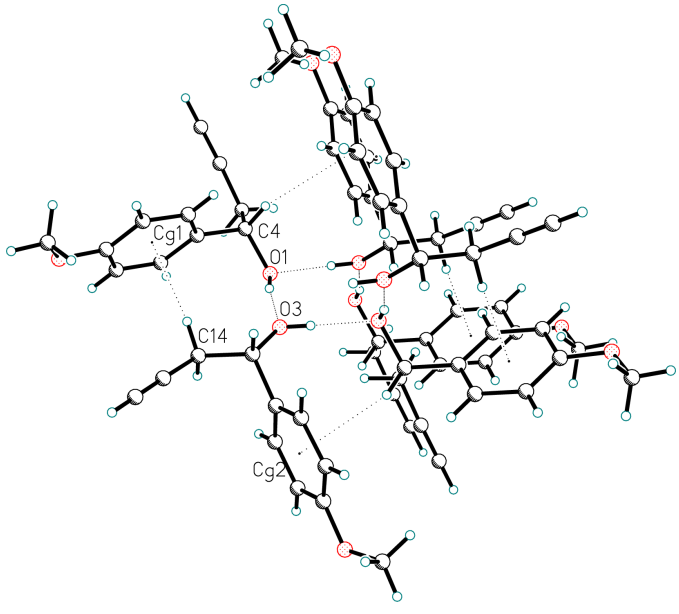


Figure 3
Detailed view of the intermolecular interactions which stabilize the helix structure of (I).

Crystal data

$C_{11}H_{12}O_2$
M_r = 176.21
Monoclinic, $P2_1/n$
a = 14.734 (3) Å
b = 6.5429 (9) Å
c = 20.907 (3) Å
β = 103.793 (9)°
V = 1957.4 (6) Å^3
Z = 8
D_x = 1.196 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 155 reflections
θ = 3–23°
μ = 0.08 mm^{-1}
T = 144 (2) K
Parallelepiped, pale yellow
0.45 × 0.27 × 0.14 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: numerical (*SHELXTL*; Sheldrick, 1996)
T_{min} = 0.971, T_{max} = 0.990
41566 measured reflections
7056 independent reflections
4779 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 33.0°
$h = -20 \rightarrow 22$
$k = -9 \rightarrow 10$
$l = -32 \rightarrow 30$
358 standard reflections
frequency: 600 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.139
S = 1.21
7056 reflections
240 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0034 (9)

alkyne H atoms and H—O = 0.84 Å. The torsion angles about the C—O bonds of the methoxy groups and the hydroxyl groups were refined.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXL*97.

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