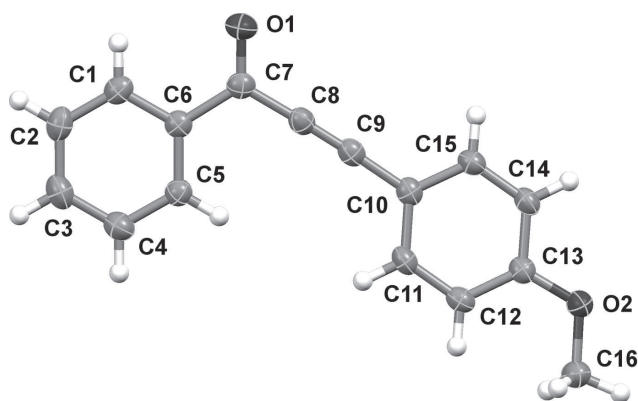


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# Crystal structure of 3-(4-methoxyphenyl)-1-phenylprop-2-yn-1-one, C<sub>16</sub>H<sub>12</sub>O<sub>2</sub>



**Table 1:** Data collection and handling.

Crystal:	Colourless needle
Size:	0.49 × 0.10 × 0.09 mm
Wavelength:	Cu K $\alpha$ radiation (1.54178 Å)
$\mu$ :	0.68 mm <sup>-1</sup>
Diffractometer, scan mode:	Bruker APEX-II, $\varphi$ and $\omega$
$\theta_{\max}$ , completeness:	65.1°, >99%
$N(hkl)_{\text{measured}}$ , $N(hkl)_{\text{unique}}$ , $R_{\text{int}}$ :	18701, 2080, 0.044
Criterion for $I_{\text{obs}}$ , $N(hkl)_{\text{gt}}$ :	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$ , 1857
$N(\text{param})_{\text{refined}}$ :	165
Programs:	Bruker [1], SHELX [2, 3], Mercury [4]

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## Abstract

C<sub>16</sub>H<sub>12</sub>O<sub>2</sub>, monoclinic,  $P2_1/c$  (no. 14),  $a = 15.7947(10)$  Å,  $b = 6.2958(4)$  Å,  $c = 12.2787(8)$  Å,  $\beta = 94.216(2)^\circ$ ,  $V = 1217.69(14)$  Å<sup>3</sup>,  $Z = 4$ ,  $R_{\text{gt}}(F) = 0.0398$ ,  $wR_{\text{ref}}(F^2) = 0.1336$ ,  $T = 150(2)$  K.

CCDC no.: 1879649

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

## Source of material

All reagents were obtained from Sigma-Aldrich and used without further purification. Benzoyl chloride (1.26 mL, 10.8 mmol, 2 eq.), 4-ethylanisole (0.70 mL, 5.4 mmol), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (0.076 g, 0.11 mmol, 0.02 eq.), CuI (0.041 g, 0.22 mmol, 0.04 eq) and Et<sub>3</sub>N (0.92 mL, 6.6 mmol, 1.2 eq.) were stirred in dry THF (20 mL) for one hour at room temperature under an inert atmosphere. Water (1 x 30 mL) was subsequently added and the product extracted into ethyl

acetate (3 x 30 mL). The organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent removed under reduced pressure. Purification of the crude product by column chromatography on silica gel (hexane:ethyl acetate 1:1), followed by recrystallization from ethanol, yielded the title compound (1.23 g, 96%) as off-white rod-like crystals,  $R_f$  0.43 (hexane:ethyl acetate 1:1): <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta_H$  p.p.m.: 8.22 (dd,  $J = 8.3, 1.4$  Hz, 2H, H-2' and H-6'), 7.64 (d,  $J = 8.8$  Hz, 2H, H-2 and H-6), 7.61 (dt,  $J = 7.5, 1.4$  Hz, 1H, H-4'), 7.50 (dd,  $J = 8.3, 7.5$  Hz, 2H, H-3' and H-5'), 6.93 (d,  $J = 8.8$  Hz, 2H, H-3 and H-5), 3.85 (s, 3H, -OMe); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta_C$  p.p.m.: 178.2 (C = O), 161.9 (C-4), 137.2 (C-1'), 135.3 (C2 and C6), 134.0 (C-4'), 129.6 (C-2' and C-6'), 128.7 (C-3' and C-5'), 114.6 (C-3 and C-5), 112.1 (C-1), 94.5 (H- $\beta$ ), 87.0 (H- $\alpha$ ), 55.6 (-OMe); MS (EI)  $m/z$  236 ( $M^+$ , 98.5%).

## Experimental details

The structure was solved by direct methods [2], using the ShelXl [3]. All hydrogen atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, and refined isotropically. The C–H<sub>aromatic</sub> and C–H<sub>methyl</sub> bond distances were restrained to 0.95 Å and 0.95 Å with  $U_{\text{iso}}(\text{H}_{\text{aromatic}}) = 1.2U_{\text{eq}}$  and  $U_{\text{iso}}(\text{H}_{\text{methyl}}) = 1.5U_{\text{eq}}$  of the parent atom, respectively. The highest calculated residual electron density is 0.24 e. Å<sup>-3</sup> at 0.81 Å from C12 and 0.69 Å from C13.

## Comment

Ongoing research in  $\alpha,\beta$ -acetylenic ketones is driven by their versatile use as valuable precursors in organic synthesis with

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**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>).

Atom	x	y	z	<i>U</i> <sub>iso</sub> <sup>*</sup> / <i>U</i> <sub>eq</sub>
O1	0.30002(7)	0.09694(16)	0.92779(7)	0.0444(3)
C1	0.38601(8)	-0.1680(2)	0.78750(10)	0.0333(3)
H1	0.380064	-0.217515	0.859620	0.040*
C3	0.43856(9)	-0.2168(2)	0.61146(12)	0.0425(4)
H3	0.468531	-0.299890	0.562418	0.051*
O2	0.06190(6)	1.21510(15)	0.57473(7)	0.0380(3)
C2	0.42935(8)	-0.2892(2)	0.71605(12)	0.0389(4)
H2	0.452794	-0.422271	0.738880	0.047*
C4	0.40412(9)	-0.0227(2)	0.57783(11)	0.0403(4)
H4	0.410855	0.026687	0.505877	0.048*
C5	0.36000(8)	0.0993(2)	0.64849(10)	0.0321(3)
H5	0.336088	0.231534	0.625011	0.039*
C6	0.35091(7)	0.02705(19)	0.75414(9)	0.0288(3)
C7	0.30202(8)	0.1491(2)	0.83230(10)	0.0314(3)
C8	0.25590(8)	0.3332(2)	0.78984(10)	0.0328(3)
C9	0.21843(8)	0.4864(2)	0.75198(10)	0.0312(3)
C10	0.17710(7)	0.66929(19)	0.70483(9)	0.0295(3)
C11	0.19710(7)	0.7405(2)	0.60202(10)	0.0316(3)
H11	0.236425	0.662438	0.562835	0.038*
C12	0.16062(8)	0.9227(2)	0.55628(9)	0.0311(3)
H12	0.175628	0.970479	0.486886	0.037*
C13	0.10166(7)	1.03584(19)	0.61263(9)	0.0289(3)
C14	0.08046(8)	0.9644(2)	0.71430(10)	0.0328(3)
H14	0.039707	1.040133	0.752222	0.039*
C15	0.11788(8)	0.7852(2)	0.76063(10)	0.0313(3)
H15	0.103541	0.739764	0.830718	0.038*
C16	0.08442(10)	1.2943(2)	0.47176(11)	0.0416(4)
H16A	0.071163	1.187205	0.415153	0.062*
H16B	0.145329	1.325893	0.475810	0.062*
H16C	0.052227	1.424252	0.453660	0.062*

the syntheses of polycyclic and heterocyclic compounds [5]. These compounds include isoxazoles, chromones, triazoles, quinolones, indenones, thiazoles, 1,5-benzodiazepines, and spirocyclic compounds [5–7]. Apart from the plethora of fine chemicals attainable from these acetylenic ketones, their increasing use as electron-rich ligands in organometallic chemistry [8] warrants continued research into their syntheses, properties, and molecular structure.

A complete molecule of the title compound is contained within the asymmetric unit, which reveals the *para*-methoxyphenyl, alkynyl, and carbonyl moieties. Furthermore, the *para*-methoxyphenyl-alkynyl and corresponding phenone functionalities are not co-planar with C8–C7–C6–C5 and C8–C9–C10–C11 torsion angles of 7.15(17)° and 28.42(18)°, respectively. The dihedral angle between the

least-squares plane through the C1 to C6 arene and the C10 to C15 arene rings is 11.16(6)°. There are also no indications of  $\pi$ – $\pi$  interactions between the *para*-methoxyphenyl and phenyl ring systems, with a distance between their ring centroids of 4.04(6) Å. There are also several types of relatively weak intermolecular contacts among neighbouring molecules present, which include two unique bifurcate interactions. The first is between C5–H5···O1 (H5–O1 = 2.67 Å) and C11–H11···O1 (H11–O1 = 2.58 Å), with angles of 162.1° (C11–H11···O1) and 133.0° (C5–H5···O1, O1 symmetry operation:  $x, 1/2 - y, -1/2 + z$ ). The second is between C16–H16···O2 (H16–O2 = 2.90 Å) and C15–H15···O2 (H15–O2 = 2.94 Å), with angles of 170.7° (C16–H16···O2, H16 symmetry operation:  $-x, 3 - y, 1 - z$ ) and 130.7° (C15–H15···O2, H15 symmetry operation:  $-x, 1/2 + y, 3/2 - z$ ). All geometric parameters are within the expected ranges.

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