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ORIGINAL PAPER

Synthesis of some cyclopentenones and crystal structure of 4-hydroxy-3,4-bis(4-methoxyphenyl)-5,5-dimethyl-2-cyclopenten-1-one

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Abstract Sodium-hydroxide-catalyzed condensation of di-*p*-methyl- and di-*p*-methoxybenzil with acetone derivatives was investigated in methanol. Di- and trisubstituted products were obtained as cyclopentenones, while tetraaryl-substituted systems were isolated as cyclopentadienones. The structures of the products were identified by elemental analysis, infrared (IR), nuclear magnetic resonance (^{1}H NMR), and mass spectroscopy. The solid-state structure of 4-hydroxy-3,4-bis(4-methoxyphenyl)-5,5-dimethyl-2-cyclopenten-1-one was further studied by single-crystal X-ray diffraction analysis. The title compound crystallizes in an orthorhombic space group and intermolecular O–H···O and C–H···O hydrogen bonds stabilize the crystal lattice.

Keywords Aldol condensation · Cyclopentenone · Hydrogen bonds · X-ray structure determination

Introduction

Cyclopentenones are not only key building blocks for organic synthesis, but possess many interesting biological properties [1–3]. The cyclopentenone ring is present in a wide array of natural products [4], interesting drug targets, and commercial products [5]. Furthermore the cyclopentenone structure has photochemical properties [6].

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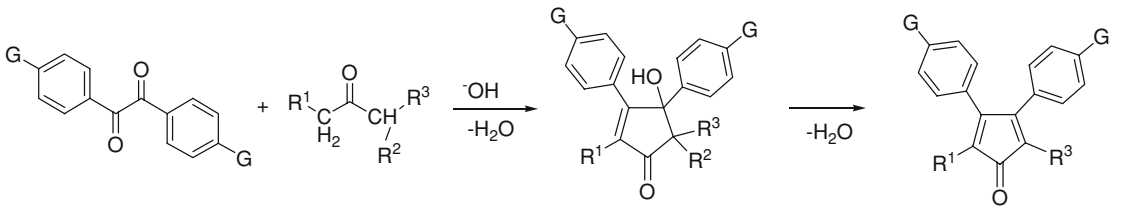
Among a variety of approaches available for preparation of cyclopentenones [7–13], condensation of aromatic α-diketones with acetone derivatives is a simple and efficient method. The aldol condensation is an extremely useful carbon–carbon bond-forming reaction in organic chemistry and this reaction has served as a powerful tool for synthesis of functionalized ring systems [14]. The reactions are usually carried out under classical homogeneous conditions in ethanol [15–18] and catalyzed by acids or bases [19–21].

Condensation of benzil and acetone derivatives was frequently utilized for preparation of diphenylcyclopentenones [22–25]. Recently, we extended the procedure to the synthesis of diarylcyclopentenones from di-*p*-bromo- and di-*p*-chlorobenzils [26]. In this work, we applied the same method to the synthesis of diarylcyclopentenones from di-*p*-methyl- and di-*p*-methoxybenzil.

Results and discussion

Condensations of di-*p*-methyl- and di-*p*-methoxybenzil with five different ketones were performed in methanolic sodium hydroxide under reflux (Scheme 1). The main products were obtained as precipitates, after pouring the mixtures into water. A summary of results is presented in Table 1.

Generally, the reactions of di-*p*-methoxybenzil proceeded faster than the corresponding reactions of di-*p*-methylbenzil. All di- and trisubstituted products were obtained as cyclopentenones (entries 1–4 and 6–9). The yields of cyclopentenones are apparently a function of their ease of dehydration: the cyclopentenones **3** and **8**, which cannot be dehydrated, were isolated with the highest yields. On the other hand, **2**, **4**, **7**, and **9**, which are more susceptible to



1 : G = CH_3, $R^1 = R^2 = R^3 = H$
2 : G = CH_3, $R^1 = CH_3$, $R^2 = R^3 = H$
3 : G = CH_3, $R^1 = H$, $R^2 = R^3 = CH_3$
4 : G = CH_3, $R^1 = Ph$, $R^2 = R^3 = H$
6 : G = OCH_3, $R^1 = R^2 = R^3 = H$
7 : G = OCH_3, $R^1 = CH_3$, $R^2 = R^3 = H$
8 : G = OCH_3, $R^1 = H$, $R^2 = R^3 = CH_3$
9 : G = OCH_3, $R^1 = Ph$, $R^2 = R^3 = H$

5 : G = CH_3, $R^1 = R^3 = Ph$
10 : G = OCH_3, $R^1 = R^3 = Ph$

Scheme 1

Table 1 Summary of results

Cpd.	Substituents				Time (min)	Yield[a] (%)
	G	R^1	R^2	R^3		
1	CH_3	H	H	H	180	78[b]
2	CH_3	CH_3	H	H	300	45[b]
3	CH_3	H	CH_3	CH_3	225	85[b]
4	CH_3	C_6H_5	H	H	75	42[b]
5	CH_3	C_6H_5	H	C_6H_5	120	86[c]
6	OCH_3	H	H	H	90	35[b]
7	OCH_3	CH_3	H	H	150	45[b]
8	OCH_3	H	CH_3	CH_3	150	80[b]
9	OCH_3	C_6H_5	H	H	240	41[b]
10	OCH_3	C_6H_5	H	C_6H_5	75	87[c]

[a] Isolated yields

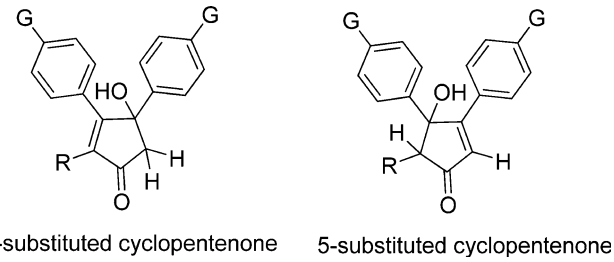
[b] The product is a cyclopentenone derivative

[c] The product is a cyclopentadienone derivative

dehydration, were obtained in relatively lower yields. The condensation of benzils with 1,3-diphenyl-2-propanone (entries 5 and 10), which were expected to form tetraaryl-cyclopentenones, led to the formation of cyclopentadienones **5** and **10** as the main products. The presence of four aryl groups is known to stabilize the cyclopentadienone system, which is inherently unstable. The preparations of **5**, **10**, and some other tetraarylcyclopentadienones have been previously reported [27, 28].

Spectroscopy

The IR spectra of all cyclopentenones exhibit υ_{OH} as a broad band around 3,400 cm^{-1}. This band is absent in the IR spectra of cyclopentadienones **5** and **10**. The spectra of the cyclopentenones present also two bands around 1,690 ($\upsilon_{C=O}$) and 1,605 cm^{-1} ($\upsilon_{C=C}$), in accordance with



Scheme 2

previously reported values for 2-cyclopentenone derivatives [29]. In cyclopentadienones, these bands are shifted to 1,725 and 1,618 cm^{-1}, respectively.

In the ^1H NMR spectra of **1**, **2**, **4**, **6**, **7**, and **9**, two doublets near 2.7 and 2.9 ppm are assigned to the methylene moiety of the cyclopentenone rings. The corresponding *J* constants are in the range 12–18 Hz. The presence of these doublets in the ^1H NMR spectra of **2**, **4**, **7**, and **9**, along with the absence of vinyl signals near 6.7 ppm, indicates the preferred formation of 2-substituted cyclopentenones (Scheme 2).

The ^1H NMR spectra of all compounds show doublets with coupling constants of about 9 Hz in the aromatic region, which are representative of *p*-disubstituted benzene rings. The simplicity of the ^1H NMR spectra of cyclopentadienones **5** and **10** is consistent with their symmetric structures.

*Crystal structure of **8***

Figure 1 shows an ORTEP view and atom numbering of **8**. The structure of the title compound is composed of two aromatic rings attached to a central cyclopentenone ring. The cyclopentenone ring has an envelope conformation with C(1), C(3), C(4), and C(5) lying in a plane, and C(2)

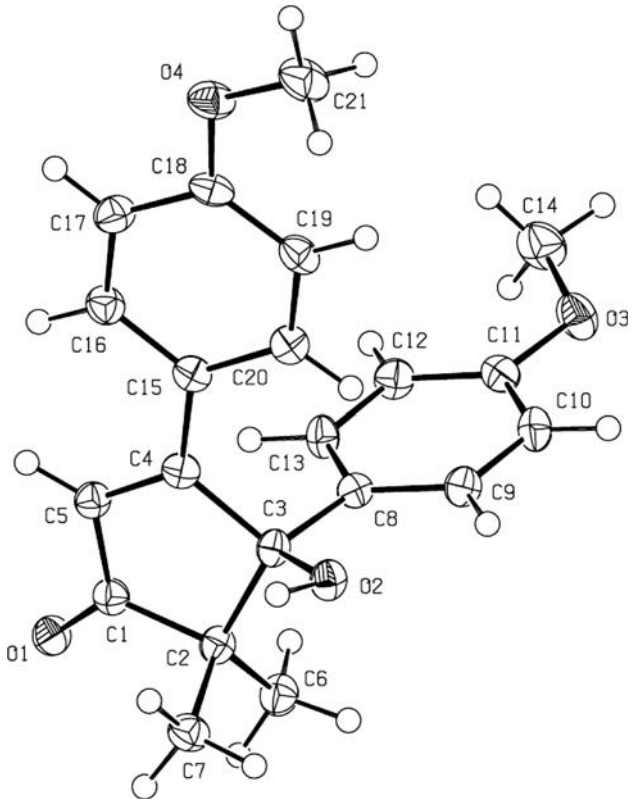


Fig. 1 An ORTEP view and atom numbering of **8** (thermal ellipsoids are drawn at 50% probability level). Selected bond lengths (Å) and angles (°): O(1)–C(1): 1.231(2), C(1)–C(5): 1.451(2), C(3)–C(4): 1.538(2), O(2)–C(3): 1.424(2), C(1)–C(2): 1.526(2), C(4)–C(5): 1.347(2), C(2)–C(3): 1.578(2), C(1)–C(2)–C(3): 102.25(13), C(4)–C(3)–C(2): 103.59(12), O(2)–C(3)–C(4): 112.95(12), C(5)–C(4)–C(3): 110.81(14), C(5)–C(1)–C(2): 108.80(14), O(2)–C(3)–C(2): 112.25(13), C(4)–C(5)–C(1): 111.07(15)

located 0.303 Å out of the plane. The conjugated *p*-methoxyphenyl, C(15)–C(20), is coplanar with the enone moiety of cyclopentenone. The other *p*-methoxyphenyl, C(8)–C(13), is orthogonal to the plane of the main conjugated system (dihedral angle is 88.41°).

The internal bond angles of the five-membered ring range from 102.25(13)° to 111.07(15)°. The bond distances vary according to the bond character: 1.347(2) Å for the C(4)–C(5) double bond to 1.578(2) Å for the C(2)–C(3) single bond. These values are very close to the values reported for similar structures [30, 31].

In the crystal lattice two molecules of **8** are linked to each other by hydrogen bonds. One hydrogen bond is formed from the hydroxyl group of one molecule to the carbonyl group of its next neighbor, O(2)–H(2O)···O(1), to form a chain along the *b* crystallographic axis (Fig. 2; Table 2). A second intermolecular hydrogen bond forms a link between the chains, forming a three-dimensional structure, C(21)–H(21A)···O(3) (Table 2). The molecules are further stabilized by intramolecular C–H···O hydrogen bonds (Fig. 3).

Conclusion

The condensation of aromatic α-diketones with acetone derivatives leads to the formation of cyclopentenones, via two aldol reactions followed by a single dehydration. When nonsymmetric ketones are used, the dehydration proceeds regioselectively to form the more stable cyclopentenones, according to Saytzeff's rule. Dehydration of cyclopentenone to cyclopentadienones may occur in tetraarylated systems.

Experimental

All chemicals were obtained from commercial sources (Merck or Fluka), and used without further purification. Melting points were measured on an Electrothermal 9100 apparatus. IR spectra were measured on a Shimadzu IR-460 spectrometer. ^{1}H NMR spectra were obtained on a Bruker Avance 300 MHz instrument using TMS as internal

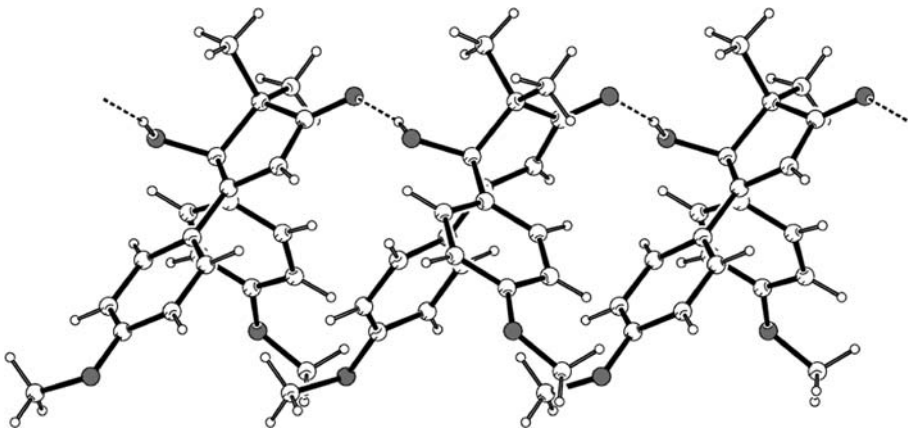


Fig. 2 Fragment of a supramolecular chain formed by intermolecular O–H···O hydrogen bonds in the solid state of **8**

Table 2 Hydrogen-bond geometry for **8**

D–H···A	*d*(D–H) (Å)	*d*(H···A) (Å)	*d*(D···A) (Å)	<(DHA) (°)
O(2)–H(2O)···O(1)[a]	0.94	1.87	2.792 (2)	165
C(9)–H(9A)···O(2)	0.95	2.32	2.685 (2)	102
C(20)–H(20A)···O(2)	0.95	2.44	3.025 (2)	119
C(21)–H(21A)···O(3)[b]	0.98	2.54	3.412 (2)	148

Symmetry transformations used to generate equivalent atoms: [a]1/2 − *x*, −1/2 + *y*, *z*; [b]1/2 + *x*, 3/2 − *y*,−*z*

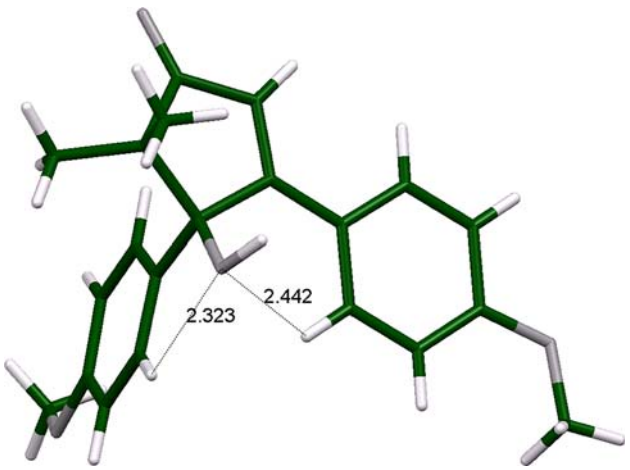


Fig. 3 Intramolecular C–H···O hydrogen bonds in **8**; distances are shown in Å

standard. Mass spectra were recorded on a Shimadzu GC/MS QP1100 EX model. Elemental analyses for C and H were conducted using a Perkin-Elmer 2400 series II analyzer; their results were compatible with the calculated values.

General synthetic procedure

A magnetically stirred mixture of the substituted benzil (0.3 mmol) and the appropriate ketone (2 mmol) in 2 cm^3 methanolic sodium hydroxide (10%) was heated under reflux. After completion of the reaction, which was monitored by thin-layer chromatography (TLC), the mixture was poured into 200 cm^3 cold water. The precipitate was then filtered off, washed successively with water and a mixture of *n*-hexane/ether, and dried in vacuo.

4-Hydroxy-3,4-di-p-tolyl-2-cyclopenten-1-one (**1**, $C_{19}H_{18}O_2$)

Colorless solid recrystallized from toluene. M.p.: 158–160°C; IR (KBr): $\bar{\nu} = 3{,}394$ (OH), 1,678 (C=O), 1,609 (C=C) cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6): $\delta = 2.26$ (s, 3H, CH_3), 2.27 (s, 3H, CH_3), 2.70 (d, $J = 18.3$ Hz, 1H, methylene), 2.88 (d, $J = 18.3$ Hz, 1H, methylene), 5.49 (s, 1H, OH), 6.69 (s, 1H, CH), 7.08 (d, $J = 8.3$ Hz, 4H, C_6H_4), 7.35 (d, $J = 8.3$ Hz, 2H, C_6H_4), 7.67 (d, $J = 8.3$ Hz, 2H, C_6H_4) ppm; MS (70 eV): *m/z* (%) = 278 (M^+, 28), 129 (59), 119 (100), 115 (88), 91(59).

4-Hydroxy-5-methyl-3,4-di-p-tolyl-2-cyclopenten-1-one (**2**, $C_{20}H_{20}O_2$)

Yellow solid recrystallized from toluene. M.p.: 105–107°C; IR (KBr): $\bar{\nu} = 3{,}400$ (OH), 1,690 (C=O), 1,619 (C=C) cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6): $\delta = 1.83$ (s, 3H, CH_3), 2.21 (s, 3H, CH_3), 2.23 (s, 3H, CH_3), 2.81 (d, $J = 18.3$ Hz, 1H, methylene), 2.91 (d, $J = 18.3$ Hz, 1H, methylene), 5.15 (s, 1H, OH), 7.07 (d, $J = 8.2$ Hz, 4H, C_6H_4), 7.27 (d, $J = 8.2$ Hz, 4H, C_6H_4) ppm; MS (70 eV): *m/z* (%) = 292 (M^+, 20), 129 (59), 119 (100), 115 (88), 91 (59).

4-Hydroxy-5,5-dimethyl-3,4-di-p-tolyl-2-cyclopenten-1-one (**3**, $C_{21}H_{22}O_2$)

Colourless solid recrystallized from toluene. M.p.: 146–148°C; IR (KBr): $\bar{\nu} = 3{,}356$ (OH), 1,678 (C=O), 1,605 (C=C) cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6): $\delta = 0.52$ (s, 3H, CH_3), 1.26 (s, 3H, CH_3), 2.21 (s, 3H, CH_3), 2.22 (s, 3H, CH_3), 5.02 (s, 1H, OH), 6.70 (s, 1H, CH), 7.09 (d, $J = 8$ Hz, 4H, C_6H_4), 7.60 (d, $J = 8$ Hz, 4H, C_6H_4) ppm; MS (70 eV): *m/z* (%) = 306 (M^+, 20), 129 (40), 119 (100), 115 (99), 59 (18).

4-Hydroxy-2-phenyl-3,4-di-p-tolyl-2-cyclopenten-1-one (**4**, $C_{25}H_{22}O_2$)

Slightly yellow solid recrystallized from toluene. M.p.: 163–165°C; IR (KBr): $\bar{\nu} = 3{,}439$ (OH), 1,697 (C=O), 1,605 (C=C) cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6): $\delta = 2.18$ (s, 3H, CH_3), 2.27 (s, 3H, CH_3), 2.91 (d, $J = 18.2$ Hz, 1H, methylene), 3.07 (d, $J = 18.2$ Hz, 1H, methylene), 5.37 (s, 1H, OH), 6.90 (d, $J = 8$ Hz, 2H, C_6H_4), 7.06 (d, $J = 8$ Hz, 2H, C_6H_4), 7.13 (d, $J = 8$ Hz, 2H, C_6H_4), 7.26–7.31 (m, 5H, Ph), 7.40 (d, $J = 8$ Hz, 2H, C_6H_4) ppm; MS (70 eV): *m/z* (%) = 354 (M^+, 20), 295 (26), 189 (100), 91(56).

2,5-Diphenyl-3,4-di-p-tolyl-2,4-cyclopentadien-1-one (**5**)

Deep red solid recrystallized from methanol. M.p.: 220–222°C (221.8–222°C [28]); IR (KBr): $\bar{\nu} = 1{,}725$ (C=O), 1618 (C=C) cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6): $\delta = 2.28$ (s, 6H, 2 CH_3), 6.87 (d, $J = 8$ Hz, 4H, C_6H_4), 7.03 (d, $J = 8$ Hz, 4H, C_6H_4), 7.22–7.23 (m, 10H, Ph) ppm.

4-Hydroxy-3,4-bis(4-methoxyphenyl)-2-cyclopenten-1-one (**6**, $C_{19}H_{18}O_4$)

Yellow solid recrystallized from toluene. M.p.: 220–222°C; IR (KBr): $\bar{\nu} = 3{,}390$ (OH), 1,678 (C=O), 1,600 (C=C) cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6): $\delta = 2.88$

(d, $J = 18$ Hz, 1H, methylene), 2.91 (d, $J = 18$ Hz, 1H, methylene), 3.71 (s, 3H, OCH_3), 3.73 (s, 3H, OCH_3), 5.27 (s, 1H, OH), 6.64 (s, 1H, CH), 6.82 (d, $J = 9$ Hz, 4H, C_6H_4), 7.41 (d, $J = 9$ Hz, 2H, C_6H_4), 7.81 (d, $J = 9$ Hz, 2H, C_6H_4) ppm.

4-Hydroxy-3,4-bis(4-methoxyphenyl)-2-methyl-2-cyclopenten-1-one (**7**, $C_{20}H_{20}O_4$)

Yellow solid recrystallized from toluene. M.p.: 108–110°C; IR (KBr): $\bar{\nu} = 3{,}396$ (OH), 1,688 (C=O), 1,601 (C=C) cm^{-1}; ^1H NMR (300 MHz, acetone-d_6): $\delta = 1.89$ (s, 3H, CH_3), 2.69 (d, $J = 18$ Hz, 1H, methylene), 2.89 (d, $J = 18$ Hz, 1H, methylene), 3.73 (s, 3H, OCH_3), 3.76 (s, 3H, OCH_3), 5.16 (s, 1H, OH), 6.83 (d, $J = 8.9$ Hz, 4H, C_6H_4), 7.32 (d, $J = 8.9$ Hz, 2H, C_6H_4), 7.39 (d, $J = 8.9$ Hz, 2H, C_6H_4) ppm; MS: *m/z* (%) = 324 (M^+, 14), 149 (34), 112 (64), 57 (100), 43 (88).

4-Hydroxy-3,4-bis(4-methoxyphenyl)-5,5-dimethyl-2-cyclopenten-1-one (**8**, $C_{21}H_{22}O_4$)

Colourless solid recrystallized from toluene. M.p.: 143–145°C; IR (KBr): $\bar{\nu} = 3{,}356$ (OH), 1,678 (C=O), 1,605 (C=C) cm^{-1}; ^1H NMR (300 MHz, acetone-d_6): $\delta = 0.52$ (s, 3H, CH_3), 1.25 (s, 3H, CH_3), 3.76 (s, 3H, OCH_3), 3.78 (s, 3H, OCH_3), 5.01 (s, 1H, OH), 6.64 (s, 1H, CH), 6.83 (d, $J = 9$ Hz, 4H, C_6H_4), 7.69 (d, $J = 9$ Hz, 4H, C_6H_4) ppm; MS (70 eV): *m/z* (%) = 338 (M^+, 95), 251 (55), 135 (100), 77 (30).

4-Hydroxy-3,4-bis(4-methoxyphenyl)-5-phenyl-2-cyclopenten-1-one (**9**, $C_{25}H_{22}O_4$)

Colourless solid recrystallized from toluene. M.p.: 166–168°C; IR (KBr): $\bar{\nu} = 3{,}427$ (OH), 1,706 (C=O), 1,607 (C=C) cm^{-1}; ^1H NMR (300 MHz, acetone-d_6): $\delta = 2.89$ (d, $J = 18$ Hz, 1H, methylene), 2.91 (d, $J = 18$ Hz, 1H, methylene), 3.69 (s, 3H, OCH_3), 3.67 (s, 3H, OCH_3), 5.40 (s, 1H, OH), 6.65 (d, $J = 9$ Hz, 2H, C_6H_4), 6.89 (d, $J = 9$ Hz, 2H, C_6H_4), 7.15 (d, $J = 9$ Hz, 2H, C_6H_4), 7.24–7.30 (m, 5H, Ph), 7.45 (d, $J = 9$ Hz, 2H, C_6H_4) ppm.

3,4-Bis(4-methoxyphenyl)-2,5-diphenyl-2,4-cyclopentadien-1-one (**10**)

Deep-red solid recrystallized from methanol. M.p.: 223–225°C (226.8–227°C [28]); IR (KBr): $\bar{\nu} = 1{,}725$ (C=O), 1,618 (C=C) cm^{-1}; ^1H NMR (300 MHz, acetone-d_6): $\delta = 3.78$ (s, 6H, 2 CH_3), 6.79 (d, $J = 9$ Hz, 4H, C_6H_4), 6.89 (d, $J = 9$ Hz, 4H, C_6H_4), 7.20–7.29 (m, 10H, Ph) ppm.

X-ray crystallography

Diffraction data were collected on a Bruker SMART 1000 CCD area detector diffractometer with graphite monochromated Mo-K_α radiation ($\lambda = 0.71073$ Å). A total of 27,913 reflections were collected, which reduced to 4,724 unique reflections ($R_{int} = 0.0537$). The structure was solved by direct methods, and refined on F^2 using all data by full-matrix least-square procedures using SHELXTL ver. 5.1 software [32]. The hydrogen atoms of OH groups were found in the difference Fourier synthesis. The H(C) atom positions were calculated.

Crystallographic data for **8** are as follows: $C_{21}H_{22}O_4$, molecular weight 338.39, orthorhombic, *Pbca*, $a = 15.869(4)$ Å, $b = 11.470(2)$ Å, $c = 19.548(3)$ Å, $V = 3558.2(13)$ $Å^3$, $Z = 8$, $T = 120(2)$ K, $F(000) = 1{,}440$, $\mu = 0.087$ mm^{-1}, $D_{calc} = 1.263$ Mg m^{-3}, $2\theta_{max} = 58.00°$, 230 parameters, goodness of fit = 0.999, $wR(F^2) = 0.1265$ (all data), $wR(F^2) = 0.1154$ [1,634 data with $I > 2\sigma(I)$], $R = 0.0889$ (all data), $R = 0.0534$ [1634 data with $I > 2\sigma(I)$].

CCDC 692901 contains the supplementary crystallographic data for **8**. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: 44-1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

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