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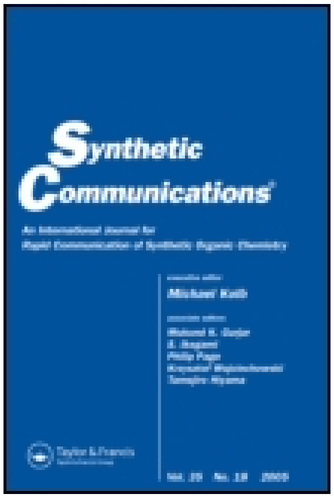

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A FACILE SYNTHESIS OF 1,4-BIS- (3-ARYL-3-OXO-1-PROPENYL)BENZENES UNDER MICROWAVE IRRADIATION

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A FACILE SYNTHESIS OF 1,4-*BIS*-(3-ARYL-3-OXO-1-PROPENYL)BENZENES UNDER MICROWAVE IRRADIATION

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ABSTRACT

1,4-*bis*(3-Aryl-3-oxo-1-propenyl)benzenes were synthesized by 1,4-benzenedicarboxaldehyde with substituted acetophenones in the presence of sodium hydroxide under microwave irradiation with good yields.

1,4-*bis*(3-Aryl-3-oxo-1-propenyl)benzenes are important intermediates in organic synthesis,[1,2] which are usually obtained by the condensation of 1,4-benzenedicarboxaldehyde and substituted acetophenones in the presence of potassium hydroxide solution.[3,4] Also, we had previously reported that synthesis of 1,4-*bis*(3-aryl-3-oxo-1-propenyl)benzenes from 1,4-benzenedicarboxaldehyde and substituted acetophenones using *bis*(4-methoxphenyl)telluroxide (BMPTO) as catalyst in DMSO.[5] However, the

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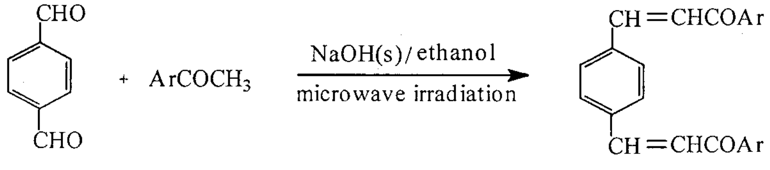
substituted acetophenones were easily self-condensed in base solution and the BMPTO was not readily available in the above synthetic methods.

The recent years, the wide applicability of microwave irradiation in chemical reaction enhancement is due to high reaction rate with formation of cleaner products and the operation simplicity.[6–8] We found that the reaction between 1,4-benzenedicarboxaldehyde and substituted acetophenones using sodium hydroxide as catalyst in ethanol under microwave irradiation was easy and the reaction was finished in short time with good yields. Here, we would like to report a facile synthesis of 1,4-*bis*(3-aryl-3-oxo-1-propenyl)benzenes from 1,4-benzenedicarboxaldehyde and substituted acetophenones in the presence of a catalytic quantity of sodium hydroxide in ethanol under microwave irradiation, **3d**, **3e**, **3f** and **3g** were not accumulated in literature. The reactions were generally finished in 2–4 min with 66–86% yields and easy work-up.

The synthetic route is shown as following:



1 **2a∼g** **3a∼g**

The results obtained are shown in Table 1.

Table 1. Reaction Times and Yields of Products **3a–g** Under Microwave Irradiation

Products	Ar	Reaction Time (min)	Yield[a] (%)
3a	C_6H_5	4	81
3b	4-ClC_6H_4	2	79
3c	4-$CH_3OC_6H_4$	2	80
3d	3-BrC_6H_4	2	66
3e	4-$CH_3C_6H_4$	3	81
3f	3,4-$(CH_3)_2C_6H_3$	3	85
3g	2-Naphthyl	2	68

[a] Yields of the isolated products.

EXPERIMENTAL

Melting points were determined in open capillaries and uncorrected. IR spectra were recorded on a Nicolet FT-IR 5DX instrument. ^{1}H NMR were measured on a Bruker DPX 300 MHz spectrometer or Bruker QC-P 200 MHz spectrometer in DMSO with TMS as internal standard. Elemental analyses were carried on a Perkin-Elmer 240C elemental analyzer. The reactions were carried out with a modified commercial microwave oven (Sanle WP650D 650 W) under atmospheric pressure.

General Procedure for the Synthesis of 3a–g

Into an Erlenmeyer flask (25 mL) equipped with reflux condenser were introduced the 1,4-benzenedicarboxaldehyde **1** (1 mmol), substituted acetophenone **2** (2 mmol), a catalytic quantity of powder sodium hydroxide (10 mol%), dry ethanol (3 mL). After irradiation for 2–4 min (monitored by TLC) at 130 W (i.e., 20% microwave power). The mixture were cooled and then filtered, washed with cooled ethanol followed by water till the washings were neutral. The crude product was recrystallized from ethanol to give a pure sample. Specific details on each product are as follows:

3a: M.p. 192–193°C (194–195°C).[5] ^{1}H NMR (DMSO-d$_6$) δ: 7.79 (2H, d, $J=15.6$ Hz), 8.06 (2H, d, $J=15.6$ Hz), 8.00–7.75 (10H, m), 8.20–8.17 (4H, m). IR (KBr): 1658, 979 cm^{-1}.

3b: M.p. 252–253°C (253–254°C).[5] ^{1}H NMR (DMSO-d$_6$) δ: 7.37 (2H, d, $J=15.6$ Hz), 7.75 (4H, d, $J=9.0$ Hz), 7.98 (4H, d, $J=9.0$ Hz), 8.25 (2H, d, $J=15.6$ Hz). IR (KBr): 1658, 974 cm^{-1}.

3c: M.p. 241–242°C (243–244°C).[5] ^{1}H NMR (DMSO-d$_6$) δ: 3.91 (6H, s), 7.03 (4H, d, $J=9.0$ Hz), 7.31 (4H, d, $J=12.4$ Hz), 7.76 (4H, m), 8.09 (2H, d, $J=12.4$ Hz), 8.30 (4H, d, $J=9.0$ Hz). IR (KBr): 1660, 971 cm^{-1}.

3d: M.p. 204–206°C. ^{1}H NMR (DMSO-d$_6$) δ: 7.79–7.54 (4H, m), 7.82 (2H, d, $J=15.6$ Hz), 8.17–8.03 (6H, m), 8.25 (2H, d, $J=15.6$ Hz), 8.35 (2H, s). IR (KBr): 1661, 971 cm^{-1}. Anal. calcd. (%) for $C_{24}H_{16}Br_2O_2$: C, 57.95; H, 3.22. Found: C, 57.72; H, 3.09.

3e: M.p. 220–222°C. ^{1}H NMR (DMSO-d$_6$) δ: 2.42 (6H, s), 7.41–7.39 (4H, d), 7.76 (2H, d, $J=15.6$ Hz), 8.04 (2H, d, $J=15.6$ Hz), 8.12–7.99 (8H, m). IR (KBr): 1655, 984 cm^{-1}. Anal. calcd. (%) for $C_{26}H_{22}O_2$: C, 85.25; H, 6.01. Found: C, 84.99; H, 6.04.

3f: M.p. 214–216°C. ^{1}H NMR (DMSO-d$_6$) δ: 2.33 (6H, s), 2.35 (6H, s), 7.76 (2H, d, $J=15.6$ Hz), 7.99–7.34 (10H, m), 8.03 (2H, d, $J=15.6$ Hz). IR (KBr): 1657, 980 cm^{-1}. Anal. calcd. (%) for $C_{28}H_{26}O_2$: C, 85.28; H, 6.60. Found: C, 85.01; H, 6.53.

3g: M.p. 232–234°C. ^{1}H NMR (DMSO-d_6) δ: 7.65–8.10 (14H, m), 7.88 (2H, d, $J = 15.6$ Hz), 8.16–8.21 (4H, m), 8.26 (2H, d, $J = 15.6$ Hz). IR (KBr): 1659, 979 cm^{-1}. Anal. calcd. (%) for $C_{32}H_{22}O_2$: C, 87.67; H, 5.02. Found: C, 87.46; H, 5.04.

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