SHORT PAPER

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A facile synthesis of 10-arylmethylene anthracenones under microwave irradiation[†]

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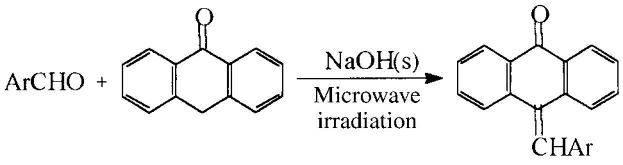
10-Arylmethylene anthracenones were synthesised by the condensation of aromatic aldehydes with anthracenone using sodium hydroxide as catalyst under microwave irradiation in good yields.

Keywords: arylmethylene anthracenanes

10-Arylmethylene anthracenones are important intermediate in organic synthesis. They have been employed for synthesis of 9-(α-bromoarylmethylene) anthracenone and its derivatives.[1–5] Derivatives of some arylmethylene anthracenones, such as 10-(4-acetamidobenzylidene)-9(10H)anthracenone (DK-V-47), 10-aminomethylene-1,8-dihydroxy-9(10H) anthracenone and 10-benzoyl-1,8-dihydroxy-9(10H) anthracenone *etc.* possess biological activity. They have the ability to inhibit the growth of the human keratinocyte cell line HaCaT and the 5-lipoxygenase enzyme in bovine polymorphonuclear leukocytes.[6–8] In general, 10-arylmethylene anthracenones were synthesised by the condensation of aromatic aldehyde with anthracenone using piperidine as catalyst in ethanol, acetic anhydride, pyridine or xylene.[1–3, 9–10] Tewari and Gupta[11] reported that 10-arylmethylene anthracenones were synthesised by reaction of aromatic aldehydes with the ylide of the 10-anthracenonyl triphenyl phosphonium or arsonium salt. Very recently, we[12] have reported that 10-arylmethylene anthracenones were obtained by the condensation of aromatic aldehydes with anthracenone using bis-(*p*-methoxyphenyl)telluroxide (BMPTO) as catalyst in DMSO. However, the BMPTO was not readily available and reaction time was long by classical heating methods.

Microwave irradiation is a very useful technique in organic synthesis. In our laboratory, we have reported the synthesis of substituted 2,5-dioxo-1,2,3,4,5,6,7,8-octahydroquinolines[13] and 2-amino-5,6,7,8-tetrahydro-5-oxo-4-aryl-7,7-dimethyl-4*H*-benzo-[b]-pyran-3-ethylcarboxylates[14] by the condensation of aromatic aldehydes with active methylene compounds under microwave irradiation. Here, we would like to report a fast, efficient and simple method for the synthesis of 10-arylmethylene anthracenones by the condensation of aromatic aldehydes with anthracenone in the presence of sodium hydroxide using ethanol as energy transfer medium under microwave irradiation. The reaction was generally finished in 5–15 minutes with good yield and easy work-up is environmental friendly and has low cost.



1a–h **2** **3a–h**

Ar = **a:** C_6H_5 **b:** $4\text{-}ClC_6H_4$ **c:** $4\text{-}O_2NC_6H_4$
d: $3\text{-}O_2NC_6H_4$ **e:** $4\text{-}HOC_6H_4$ f: $4\text{-}CH_3OC_6H_4$
g: $3,4\text{-}(OCH_2O)C_6H_3$ **h:** $3,4\text{-}(CH_3O)_2C_6H_3$

The results obtained are shown in Table 1.

Experimental

Melting points were determined in open capillaries and uncorrected. IR spectra were recorded on a Nicolet FT-IR 5DX instrument. ^{1}HNMR were measured on a Burke 300 MHz spectrometer in $CDCl_3$ with TMS as internal standard. The reactions were carried out with a modified commercial microwave oven (Sanle WP650D 650w) under atmospheric pressure.

10-Benzalanthracenone **(3a)***; typical procedure:* Into an Erlenmeyer flask (50 ml) equipped with reflux condenser were introduced the benzaldehyde (5 mmol), anthracenone (5 mmol), anhydrous ethanol (15 ml) and a catalytic quantity of sodium hydroxide (4–5 mol%). After irradiation by microwave for 12 minutes (as indicated by TLC), the reaction mixture was cooled by an ice bath, the crude product was collected by filtration and washed with cooled ethanol, then washed with water till the washings were neutral. The crude product was recrystallised from acetic acid to yellowish crystals, m.p.117~118 °C. ^{1}H NMR($CDCl_3$)δ: 7.49 (s, 1H, CH), 7.21~8.39 (m, 13H, Ar). IR (cm^{-1}): 1650 (C=O), 1600 (C=C).

Table 1 Condensation of aromatic aldehydes with anthracenone

Products[a]	Reaction time MW[b]/min	Standard/h	Yield[c]/% MW	Standard	m.p./°C	Lit. m.p. /°C[1, 5, 9, 11]
3a	12	7	72	69	117–8	118
3b	7	8	77	60	175–6	175–8
3c	5	7	83	43	178–9	181
3d	7	7	78	54	170–1	170–2
3e	14	6	71	42	140–2	139–42
3f	13	7	62	65	160–2	162
3g	7	7	79	48	140–2	142
3h	15	12	60	40	180–2	182–3

[a]All products were characterised by IR and ^{1}HNMR spectra [b]Microwave irradiation [c]Isolated yield.

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[†] This is a Short Paper, there is therefore no corresponding material in *J Chem. Research (M).*

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