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SCOPE AND MECHANISM OF THE REACTION OF ALKYLIDENE PHOSPHORANES WITH 10-METHYLENEANTHRONE

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Quinone methide (**1**) reacts with excess carbmethoxymethylenetriphenylphosphorane (**2a**) to afford the new product **3a–6** whereas, with carbethoxymethylenetriphenylphosphorane (**2b**) yielded **3b**, **4b**, **13** and **14**. It was also found that the polarity of the solvent has a limited role in the course of the reaction and in the yield of the condensation products of **1** and ylides **2**. Mechanistic studies suggest that 10-methyleneanthrone (**1**) can react as a Diels-Alder diene and function as a dienophile in the same reaction. When the Wittig reaction was carried out on the new ylides **5** and **6** using benzaldehyde, the new olefins **16** and **17** were isolated.

Key words: Quinone methides; 10-methyleneanthrone; Wittig reaction; Diels-Alder reaction; dimerization.

INTRODUCTION

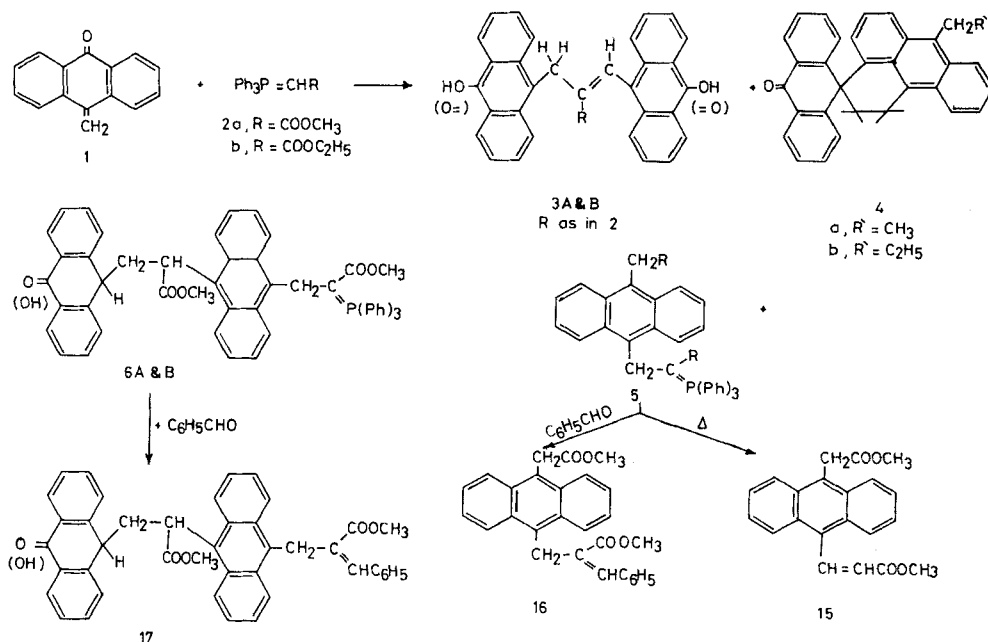
Previously we reported¹ that the condensation of 10-methyleneanthrone with ter-valent phosphorus reagents leads to different pathways depending on the nature of the phosphite ester used, the type of solvent and on the experimental conditions. It also was pointed out that the behavior of the quinone methide (**1**) toward alkyl phosphites is in complete variance with that already reported with other quinone methides^{2–4} and the result was rationalized in terms of steric factors.^{2–5} We have now expanded the scope of this concept by the use of another phosphorus nucleophile, phosphonium ylides, and we have looked further into the mechanism of the reaction.

RESULTS AND DISCUSSION

10-Methyleneanthrone (**1**) reacts with two equiv. (excess ylide leads to better results) of carbmethoxymethylenetriphenylphosphorane (**2a**) after 10 hr in refluxing toluene. Chromatographic separation of the reaction mixture produced successively four different substances **3a–6**. Triphenylphosphine and triphenylphosphine oxide were also isolated in a pure state. Scheme I shows the identified products.

The first product obtained was the major product (28% yield) and established to have the unsaturated bisphenol structure **3a** by the following physical and struc-

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SCHEME I

tural data: (1) Elemental analysis and MS support the molecular formula C₃₃H₂₄O₄ (484.55). (2) Its IR⁴ spectrum revealed the presence of new absorption bands at 3230 cm⁻¹ (broad, OH's) and at 1710 cm⁻¹ (C=O, ester).

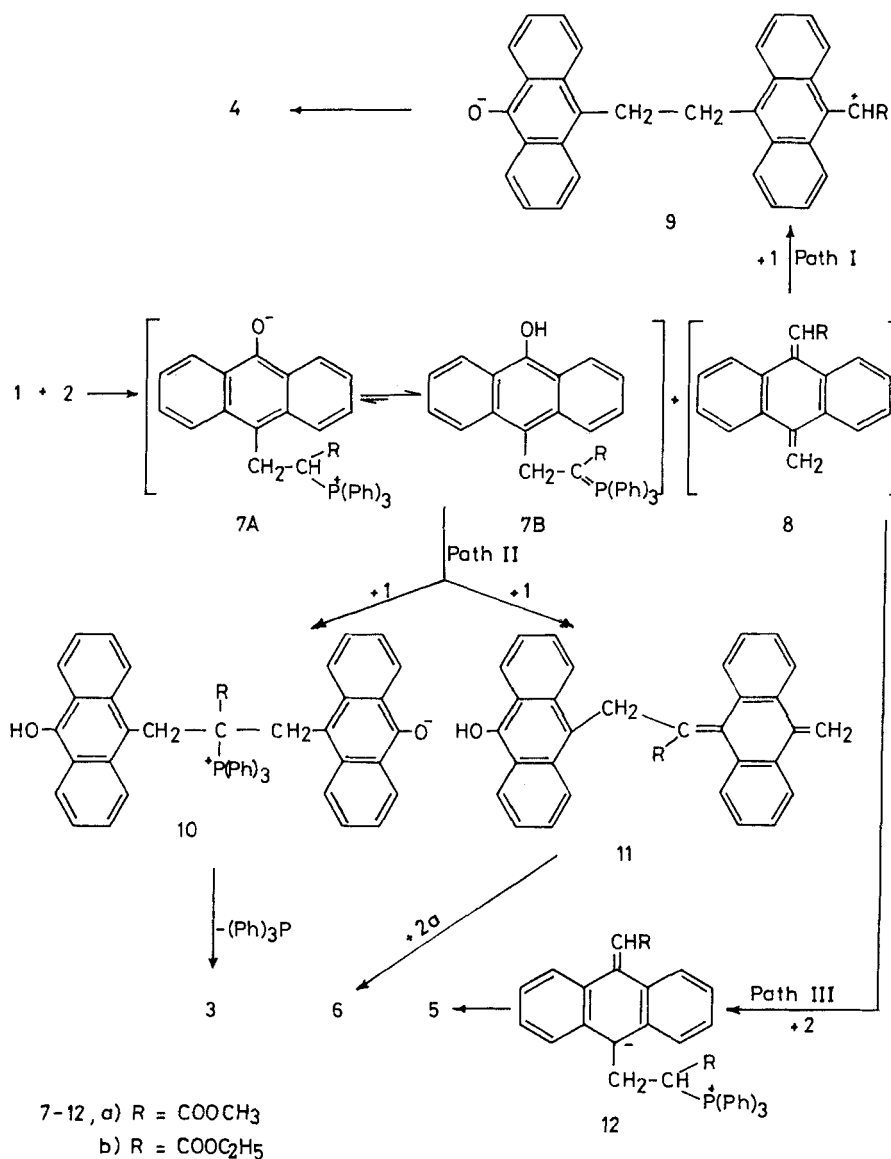
(3) Its PMR spectrum showed signals at: δ 2.81 ppm (s, 2H, —CH₂), 3.86 ppm (s, 3H, OCH₃), 5.23 (broad, 2H, 2OH's), and at 6.73 ppm assigned for the benzylic proton (s, 1H, =CH).⁶

The second product isolated, **4a** was identified as 1,2-dihydro-7-ethyl-3H-benz[de]anthracene-3-spiro-10-anthrone by various spectrometric measurements (Experimental section).

The third substance was obtained in 8% yield, assigned the olefinic ylide (**5**) for the following reasons: (1) The elemental analysis and MS are consistent with the constitutional formula. (2) Its IR spectrum reveals a new strong band at 1575 cm⁻¹, typical for the C=P group. (3) The ³¹P-NMR shift = +25.7 ppm.⁷ (4) Its PMR spectrum shows absorptions at: δ 3.81 and 3.60 (2s, 6H, 2OCH₃'s), 2.82 (d, 2H, CH₂—C=P, *J*_{HP} = 8.5 Hz), 2.64 ppm (s, 2H, Ar—CH₂).

The last product obtained in 13% yield, for which the ylide structure **6** was assigned basing on the following evidence: (1) Elemental analysis and molecular weight determination (MS) for **6** corresponds to C₅₄H₄₃O₅P. The ³¹P-NMR shift recorded for compound **6** was δ = 26.8 ppm. Its ¹H-NMR spectrum showed the protons of the methoxyl groups as two singlets at 3.84 ppm (3H) and 3.79 ppm (3H). Protons of the two CH₂ groups appeared in a complex pattern at δ 2.62–2.88 ppm, whereas the methine proton resonated at 4.7 ppm (t, 1H, —CH, *J*_{HH} = 8.5 Hz) which indicates trans to PhCH₂.

Scheme II shows a mechanism for the reaction which is consistent with data presented thus far. Ylide **2** attacks **1**, preferentially,^{1,3,8-11} at the methanide carbon to give intermediates of type **7** (major) and **8** (minor). The products **3-6** are actually accomplished through three pathways (I-III) via these intermediates: I) Addition of the carbanion moiety of **8** to the methylene group of a second quinone methide molecule and internal cyclization of the newly formed betaine **9** followed by or concurrent with its decarboxylation, then gives new spiroanthrone (**4**). This type of intramolecular rearrangement is in accordance with the mechanisms previously reported^{5,12-14} for spiroanthrones.

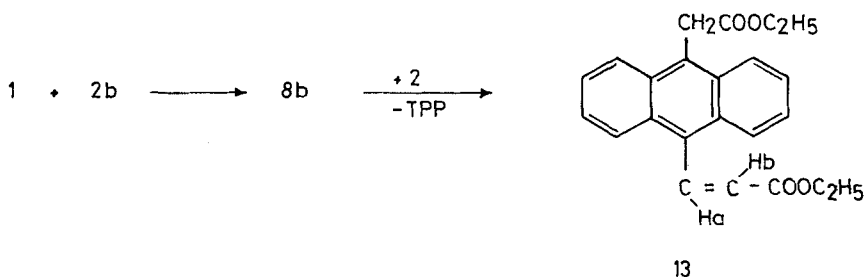


SCHEME II

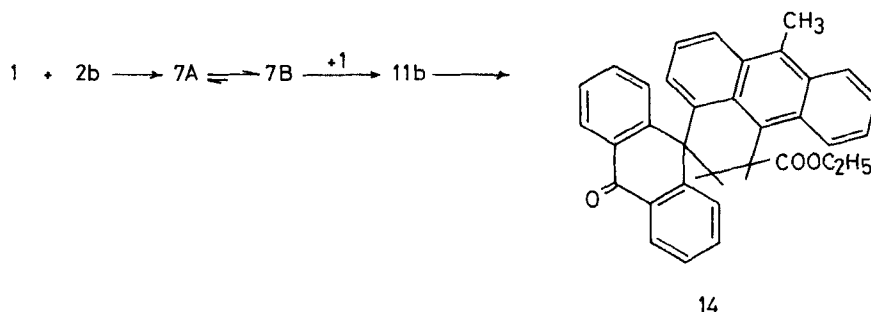
Formation of **3** and **6** during the reaction of **1** with **2a** can be reasonably interpreted by path II (cf. Scheme II) in terms of initial formation of a resonance stabilized phenoxy anion moiety (**7A**) which might be expected to be stabilized through proton migration,^{15,16} giving rise to ylide **7B**. Nevertheless, ylide **7B** should be highly nucleophilic and should therefore undergo quickly further reactions furnishing **3** and **6**. Hoffmann transylidation reaction¹⁷ of betaine **10** (**7B** + **1**) with expulsion of TPP might be the route to bisphenol (**3**). In other instance, **7"B**" undergoes two consecutive Wittig reactions, first **7B** + **1** and then with phosphonium ylide (**2a**) to give **6**. In principle, **8** could condense with resonance hybriide (**7**) leading directly to ylide (**6**). However this route to **6** is ruled out, leaving path II as the only reasonable alternative due to the possible existence of a structure like (**7B**) (—OH) which does not allow further olefination.

Adduct **5** can be explained by Scheme II, path III. Olefination of **1** by one mole of **2** to give the intermediate **8b**, followed by Michaelis addition of a second phosphorane molecule to afford the stable ylide-phosphorane adduct **5**, via the betaine precursor **12**. Possible attack of **1** spontaneously by two moles of Wittig reagent both on the carbonyl of the quinone and on the activated carbon double bond to give **5** seems less likely for the above mentioned reasons.

The reaction of **1** with carbethoxymethylenetriphenylphosphorane (**2b**) represents another, even more interesting variation of the Wittig reaction (Schemes III and IV). As in the case with ylide **2a**, the primary condensation products **7** and **8** seem to be instable and highly reactive, undergoing consecutive condensations to afford **3b**, **4b**, **13** and **14** as the final products. With respect to **3b** and **4b**, products



SCHEME III



SCHEME IV

were isolated concurrent with those obtained from the reaction of **1** with **2a**, but with **13** and **14** different condensation products were obtained.

Compound **13** (6% yield) was shown to possess the elemental composition $C_{23}H_{22}O_4$. Its 1H -NMR spectrum showed among other signals two doublets due to the exocyclic vinyl-protons (AB) at δ 7.49 ppm (d, 1H, =CHa) and 6.84 ppm (d, 1H, =CHb), having a coupling constant $J = 14$ Hz for each, indicating that the E isomer is the only structure for the alkene **13**. The main features of the spectra is in accord with the assigned structure. Solid evidence for structure **13** came from heating the reaction product **5** which led to the formation of **15** (cf. Experimental section) accompanied by TPP. The mechanism proposed in Scheme II (path III) suggests the mono-olefination of **1** by **2** to give the intermediate **8** which reacts further with a second ylide **2b** to yield, finally, compound **13** and regenerate TPP (Scheme III).

The last product which is unequivocally different from the condensation product **6** is assigned spiroanthrone (**14**). The structure of **14** is assigned from its molecular weight, IR and PMR measurements. The data are consistent with that previously ascribed^{1,5,8,12,14} for spiroanthrones.

It may be considered that **11b** (Scheme II) is the intermediary species of **14**. Intramolecular cyclization, followed by prototropic rearrangement accounts for the formation of the spiroanthrone (**14**) (Scheme IV).

In order to study the solvent effect on the course of the Wittig reaction, the reaction of **1** with **2a** was carried out in several solvents of different polarity (toluene, EtOAc, MeCN, and MeOH), maintaining the general conditions like the first experiment (cf. Table I).

From the data shown in Table I, it is obvious that **1** undergoes Wittig reaction preferentially to give the dimerization product **3**, which dominates always other products **4**–**6**. Moreover, it has been demonstrated that the use of polar solvents increases the attack on the carbonyl group to give the intermediate **8** and consequently, the products **4** and **5** which are derived from its conversions.

It was of interest to explore the synthetic utility of the new phosphoranes **5** and **6**. However, on treating each of them with benzaldehyde, the Wittig reaction occurred, elaborating an olefinic side chain, their structures were assigned **16** and **17**, respectively, on the basis of their origins **5** and **6**. The structures are consistent with their absorptions and their NMR spectra (cf. Experimental section).

In summary, a notable feature of this and previous investigation is the susceptibility of these *p*-quinone methides to react as a Diels-Alder diene and to function

TABLE I
Effect of solvent and the temperature on the Wittig reaction of **1**

Solvent ^b	time/hr	Yield (%) ^a				
		3	4	5	6	15
ArCH ₃	10	28	5	8	13	-
EtOAc	6	37	-	-	17	6
MeCN	18	22	10	12	8	-
MeOH	10	20	12	15	8	-

a) Yields are approximated and based on the amount of the starting material (100% disappearance of **1**, TLC)

b) The reaction was carried out at the reflux temperature.

as a dienophile in the same reaction. In addition, these species are generally attacked at the terminal methylene group by a variety of nucleophilic agents, yielding the appropriate benzyl-derivatives by 1,6-addition.^{1,5,8,14} The results obviously confirmed the tendency of these quinones to undergo combined disproportionation and dimerization. The results revealed the potential synthetic usefulness of ylides **5** and **6**.

EXPERIMENTAL

All melting points are uncorrected. The IR spectra were recorded in KBr, with Perkin-Elmer Infracord Model 137 and Beckmann Infracord Model 4220. The PMR spectra were taken in CDCl_3 or DMSO, with a Varian Spectrometer at 90 MHz, using TMS as an internal reference. ^{31}P -NMR spectra were recorded, relative to external H_3PO_4 (85%), with a Varian FT-80 Spectrometer. The mass spectra were performed at 70 eV on MS-50 Kratos (A.E.I.) Spectrometer.

Reaction of 10-methyleneanthrone (1) with ylides (2a,b). A solution of the anthrone (**1**)³ (2.06 g, 0.010 mol) and **2a**¹³ (6.8 g, ~ 0.02 mol) in 100 ml toluene was refluxed for 10 hr, the solvent then evaporated, and the solid product was redissolved in methanol (100 ml) and evaporated to dryness in the presence of silica gel (10 g). The mixture was then added to a column previously charged with silica gel in petroleum ether (b.p. 40–60°C). The column was developed with petroleum ether containing increasing amounts of chloroform then with pure ethylacetate. Petroleum ether elution afforded colorless needles mp 80°C, identified to be TPP.

Fraction (up to 8.5:1.5 v/v) eluted 677 mg (28%) of **3a** as yellow crystals mp 283°C (chloroform). Calcd. for: $\text{C}_{33}\text{H}_{24}\text{O}_4$ (484.555): C, 81.79; H, 4.99. Found: C, 81.76; H, 4.98. IR (KBr) cm^{-1} : 3230 (2OH's; broad and weak), 1710 (C=O, ester). ^1H -NMR (DMSO) δ 2.81 (s, 2H, CH_2), 3.86 (s, 3H, OCH_3), 5.23 (broad, 2H, 2OH's), 6.73 (s, 1H, $=\text{CHAr}$), 6.85–8.4 ppm (m, 16H, Ar—H). MS: m/z = 484 (M^+ , 18%).

The fraction (up to 7:3 v/v) yielded yellow crystals of **4a** (106 mg, 5%) mp 178°C (benzene). Anal. calcd. for $\text{C}_{32}\text{H}_{25}\text{O}$ (425.552): C, 90.32; H, 5.92. Found: C, 90.27; H, 5.90. IR (KBr) cm^{-1} : 1668 (anthrone C=O), no OH. ^1H -NMR (CCl_4) δ 1.15 (t, 3H, Ar—C— CH_3 , J = 6.0 Hz), 2.22–2.41 (m, 4H, Ar— CH_2), 3.31 (t, 2H, Ar— CH_2 , J = 8 Hz), 6.8–8.4 (m, 16H, Ar—H). MS: m/z = 425 (M^+ , 4%).

The fraction (up to 6:4 v/v) yielded colorless needles, mp 156°C of triphenylphosphine oxide (mp, mixed mps, and comparative IR spectra).

The fraction (up to 5:5 v/v) yielded colorless substance, mp 168°C of carbmethoxytriphenylphosphorane (**2a**) (mp, mixed mps, and comparative IR spectra).¹³

The fraction (up to 3:6 v/v) gave 476 mg (8%) of yellow product, recrystallized from acetone to give a straw yellow material of mp 254°C and identified as the ylide **5** calcd. for: $\text{C}_{42}\text{H}_{37}\text{O}_4\text{P}$ (624.727): C, 78.50; H, 5.57, P, 5.19. Found: C, 78.77; H, 5.52, P, 5.23. IR (KBr) cm^{-1} 1715

$\left(\begin{array}{c} \diagup \\ \text{C=O, ester} \\ \diagdown \end{array} \right)$, 1575 $\left(\begin{array}{c} \diagup \\ \text{C=P} \\ \diagdown \end{array} \right)$. ^1H -NMR (DMSO) δ 2.64 (s, 2H, Ar— CH_2), 2.82 (d, 2H, CH_2),

2.82 (d, 2H, CH_2 —C=P, J_{HP} = 8.5 Hz), 3.60 and 3.8, (2s, 6H, 2 OCH_3 's) 6.8–8.4 ppm (m, 23H, Ar—H). ^{31}P -NMR (DMSO) δ = +25.7 ppm. MS: m/z = 624 (M^+ , 12%).

Elution with chloroform (or ethylacetate) furnished 521 mg (13%) of yellow crystals, mp 310°C (methanol), which was proved to be ylide **6**. Calcd. for: $\text{C}_{34}\text{H}_{43}\text{O}_3\text{P}$ (802.918): C, 80.77; H, 5.39; P, 3.85. Found: C, 80.72; H, 5.28, P, 3.56. IR (KBr) cm^{-1} : 3350 (OH, weak), 1695 (C=O), 1670 and

1525 $\left(\begin{array}{c} \diagup \\ \text{C=P} \\ \diagdown \end{array} \right)$. ^1H -NMR (DMSO) δ 2.62–2.88 (m, 4H, Ar— CH_2), 3.79 and 3.84 (2s, 6H, 2 OCH_3 's),

4.7 (t, 1H, Ar—CH, J = 6 Hz), 6.4–8.5 ppm (m, 31H, Ar—H). ^{31}P -NMR (DMSO) δ = +26.8 ppm. MS: m/z = 640 (M^+ 25%) which corresponds to the exact M. Wt. after the expulsion of TPP.

Similarly, the products **3b**, **4b**, **13** and **14** were obtained upon reacting **2b** with **1** and working-up of the reaction mixture as mentioned above. TPP, TPPO and excess ylides (**2b**) were also isolated and identified.

Bisphenol **3b** was obtained as yellow crystals (771 mg, 31%), mp 274°C (chloroform). Anal. calcd. for $\text{C}_{34}\text{H}_{26}\text{O}_4$ (498.582): C, 81.90; H, 5.25. Found: C, 81.84; H, 5.22. IR (KBr) cm^{-1} : 3245 (2OH's,

broad and weak), 1720 (C=O, ester). ¹H-NMR (DMSO) δ 1.33 (t, 3H, OC—CH₃, *J* = 7 Hz), 2.76 (s, 2H, CH₂), 4.1 (q, 2H, OCH₂, *J* = 7 Hz), 5.09 (broad, 2H, 2OH's), 6.68 (s, 1H, =CH—Ar), 6.83–8.41 ppm (m, 16H, Ar—H). MS: *m/z* 498 (M⁺, 33%).

Spiroanthrone (**4b**), yellow crystals (153 mg, 7%) of mp 196°C (benzene). Calcd. for C₃₃H₂₇O (439.579): C, 90.07; H, 6.19. Found: 89.87; H, 6.17. IR (KBr) cm⁻¹: 1668 (anthrone C=O), no OH. ¹H-NMR (CCl₄) δ 1.15 (t, 3H, C—CH₃), 2.25–2.74 (m, 8H, 4—CH₂'s), 6.4–8.8 (m, 16H, Ar—H). MS: *m/z* = 439 (M⁺, 8%).

Compound **13**, yellow needles (217 mg, 6%), mp 210°C (ethyl alcohol). Calcd. for C₂₃H₂₂O₄ (362.429): C, 76.22; H, 6.12. Found: C, 76.20; H, 6.08. IR (KBr) cm⁻¹: 1700 and 1715 $\left(\begin{array}{c} \diagup \quad \diagdown \\ \text{C=O, esters} \end{array} \right)$, 1622

$\left(\begin{array}{c} \diagup \quad \diagdown \\ \text{C=C} \end{array} \right)$. ¹H-NMR (DMSO) δ 1.33–1.37 (two triplets, superimposed, 6H, 2—C—CH₃), 2.66 (s, 2H, Ar—CH₂), 3.82–4.2 (2q, 4H, 2OCH₂'s), 6.84 (d, 1H, =CH, *J* = 14 Hz), 7.49 (d, 1H, =CH, *J* = 14 Hz), 6.67–8.12 (m, 4H, Ar—H). MS: *m/z* = 362 (M⁺, 28%).

Spiroanthrone (**14**), yellow substance (306 mg, 15%), mp 240°C (acetonitrile). Calcd. for C₃₄H₂₇O₃ (483.59): C, 84.44; H, 5.63. Found: C, 84.41; H, 5.59. IR (KBr) cm⁻¹: 1705 (C=O, ester), 1672 (C=O, anthrone), 1605 (C=C, aryl). ¹H-NMR (DMSO) δ 1.13 (s, 3H, Ar—CH₃), 1.37 (t, 3H, OC—CH₃, *J* = 7 Hz), 2.25 (d, 2H, Ar—CH₂, *J* = 6 Hz), 3.73 (t, 1H, —CHR, *J* = 6 Hz), 4.23 (q, 2H, OCH₂, *J* = 7 Hz), 6.8–8.8 (m, 16H, Ar—H). MS: *m/z* = 483 (M⁺, 5%).

Thermal decomposition of 5. The ylide adduct **5** (500 mg, 0.8 mmol) was heated (bath temp. 230°C) for one hour under reduced pressure (5 mm/Hg) in a cold sublimator. The reaction vessel was left to cool and ethyl alcohol (5 ml) was added. The crystals that separated were recrystallized from benzene to give yellow crystals (18 mg, 78% yield) mp 202°C and identified as **15**. Calcd. for: C₂₁H₁₈O₄ (334.375) C, 75.43, H, 5.42. Found: C, 75.38; H, 5.41. IR (KBr) cm⁻¹: 1715 (C=O, ester), 1630 (HC=CH). ¹H-NMR (DMSO) δ 3.74 and 3.85 (2s, 6H, 2OCH₃'s), 6.68 and 7.58 (2d, 2H, —CH=CH; *J* = 16.5 Hz), 6.8–8.72 ppm (m, 8H, Ar—H). MS: *m/z* = 334 (M⁺, 35%).

TPP was also isolated and identified from the ethanol filtrate (mp, mixed mps and comparative IR spectra).

Significantly, heating the ylide product **5** in ethylacetate for 6 hr at the reflux temperature, yielded the adduct **15** and TPP in a quantitative yield.

Wittig reaction of 1 in different solvents. In three parallel experiments, the reaction of **1** with **2a** was carried out in ethylacetate (EtOAc), acetonitrile (MeCN) and methanol (MeOH) (similar to the reaction of **1** with **2** in toluene), employing the same conditions. The reaction mixture was worked up in the usual way and chromatography on silica gel gave products **3a**, **4a**, **5**, **6** and **15** (in case of using EtOAc). For comparative yields see Table I.

Reaction of the new ylides 5 and 6 with benzaldehyde. To a suspension of **5** (or **6**, 0.001 mol.) in toluene (50 ml), benzaldehyde (0.55 g, 0.005 mol.) was added. The reaction mixture was refluxed for 18 hr (TLC) and the solvent evaporated. Extraction of the residual substance with hot petroleum ether, gave on cooling TPPO, mp 156°C. Crystallization of the residue from a suitable solvent gave the olefinic side chain products **16** and/or **17**, respectively.

Compound **16**, yellow crystals (62%) mp. 222°C (CH₂Cl₂) calcd. for C₂₈H₂₄O₄ (424.5): C, 79.22; H, 5.69. Found: C, 79.09; H, 5.68. IR (KBr) cm⁻¹: 1715 $\left(\begin{array}{c} \diagup \quad \diagdown \\ \text{C=O, ester} \end{array} \right)$, 1620 (C=C). ¹H-NMR

(DMSO) δ 2.64 (s, 2H, Ar—CH₂), 3.62 and 3.84 (2s, 6H, 2OCH₃'s), 6.85 and 7.65 (2d, 2H, —CH=CH(AB), *J*_{HH} = 16 Hz), 7.2–8.4 (m, 8H, Ar—H). MS: *m/z* = 424 (M⁺, 18%).

Compound **17**, yellow crystals (55%) mp 265°C (Chloroform). Calcd. for: C₄₃H₃₄O₅ (630.745): C, 81.88; H, 5.43. Found: C, 81.85; H, 5.41. MS: *m/z* = 630 (M⁺, <5%).

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