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The Reaction of *Wittig* and *Wittig-Horner* Reagents and Alkyl Phosphites with 1,2-Naphthoquinone-1-benzimide

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Summary. Carbomethoxymethylene and carbethoxymethylene triphenylphosphoranes (**1a**, **b**) react with 1,2-naphthoquinone-1-benzimide (**8**) to give the corresponding 1-benzoylimino-1*H*-naphthalen-2-ylidene-acetic esters **9a**,**b** and 2-(1-benzoylamino-naphthalen-2-yl)-but-2-enedioic esters **10a**,**b** together with 1-benzamido-2-naphthol (**11**). Application of *Wittig-Horner* reagents **4a**,**b** on **8** at room temperature and in the presence of alcoholic sodium alkoxide renders 2-(1-benzoylamino-naphthalen-2-yl)-3-phosphonylsuccinic esters **12a**,**b**. Conducting the reaction in refluxing toluene and in the presence of sodium hydride led to the formation of 2-benzamido-naphthol-1-ethyl ether (**13**). 1-Benzamido-2-naphthol (**11**) was also isolated from both reaction media. Trimethyl and dimethyl phosphites (**7a**,**b**) react with **8** to **14** as the sole product. Mechanisms accounting for the formation of the new products are discussed, and the probable structures of the products are presented based on analytical and spectroscopic data.

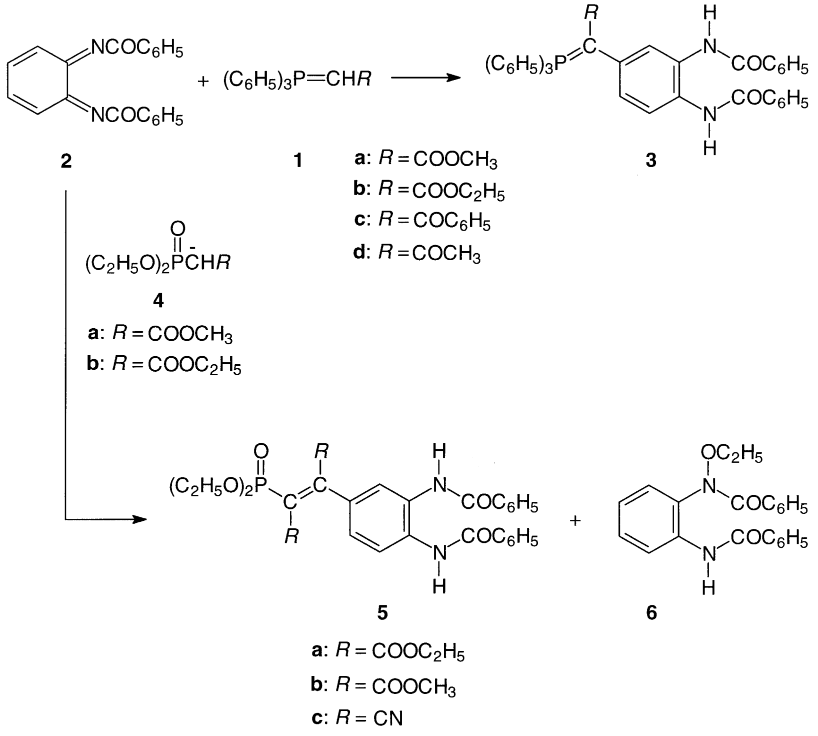
Keywords. *Wittig*; *Wittig-Horner*; Phosphites; Naphthoquinone; Naphthalene; Naphthoxazol derivative.

Reaktionen von *Wittig*- und *Wittig-Horner*-Reagenzien sowie Alkylphosphiten mit 1,2-Naphthochinon-1-benzimid

Zusammenfassung. Carbomethoxymethylen- und Carbethoxymethylen-triphenylphosphorane (**1a**,**b**) reagieren mit 1,2-Naphthochinon-1-benzimid (**8**) zu den entsprechenden 1-Benzoylimino-1*H*-naphthalin-2-yliden-essigsäureestern **9a**,**b** und zu den 2-(1-Benzoylamino-naphthalin-2-yl)-but-2-endionestern **10a**,**b** sowie zu 1-Benzamido-2-naphthol (**11**). Einwirkung der *Wittig-Horner*-Reagenzien **4a**,**b** auf **8** bei Raumtemperatur in Gegenwart von alkoholischem Natriumalkoxid ergibt die 2-(1-Benzoylamino-naphthalin-2-yl)-3-phosphonyl-bernsteinsäureester **12a**,**b**. Durchführung der Reaktion in Toluol am Rückfluß in Gegenwart von Natriumhydrid führte zur Bildung von 2-Benzamido-naphthol-1-ethylether (**13**). Aus derselben Reaktionsmischung konnte auch 1-Benzamido-2-naphthol (**11**) isoliert werden. Tri- und Dimethylphosphite (**7a**,**b**) reagieren mit **8** zu **14** als einzigem Produkt. Mechanismen, die zur Bildung der neuen Verbindungen führen, werden diskutiert, und die nach analytischen und spektroskopischen Befunden wahrscheinlichen Strukturen werden vorgestellt.

Introduction

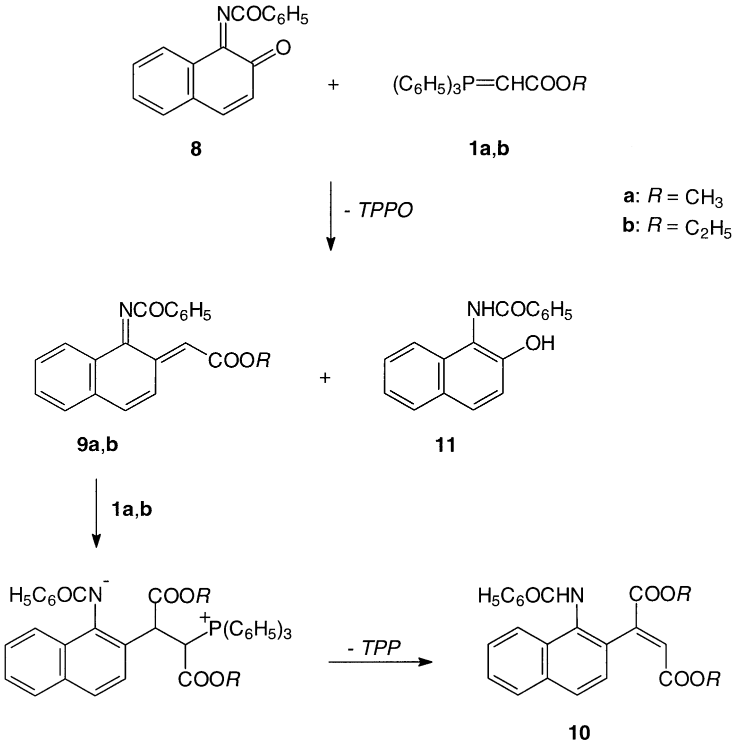
More than a decade ago, we have reported that *Wittig* reagents (**1**) react with *o*-quinone diimines (**2**) to the corresponding ylide-phosphorane adducts **3** [1]. Moreover, it is known that *Wittig-Horner* reagents (**4**) give phosphonate adducts **5** and the alkylated product **6** when reacted with **2** (Scheme 1) [2]. As a continuation of our work on the behavior of organophosphorus reagents towards quinone imines [3–6], we have now investigated the action of phosphonium ylides, phosphonate anions **4** and alkyl phosphites **7** on 1,2-naphthoquinone-1-benzimide (**8**). The purpose of this study was to determine the preferential site of attack by these reagents and to produce new phosphorane adducts.



Scheme 1

Results and Discussion

When 1,2-naphthoquinone-1-benzimide (**8**) was treated with one equivalent of carbomethoxymethylene triphenylphosphorane (**1a**) in absolute benzene at room temperature for 8 hours, adducts **9a** and **10a**, 1-benzamido-2-naphthol (**11**), triphenylphosphine, and triphenylphosphine oxide were isolated. Carrying out the reaction using two mol of phosphonium ylide **1a** instead of one lead to the formation of compounds **9a**, **10a**, and **11** in good yields (Scheme 2). In many addition reactions of quinoneimines, reduction occurred at varying degree in the presence of reagents which are normally not considered to have reducing properties [7, 8]. It has also been observed that better yields are obtained when the reaction is carried out in a 1:3 molar ratio.



Scheme 2

Compound **9a** was chromatographically pure and exhibited a sharp melting point. The IR spectrum of **9a** revealed the presence of strong absorption bands at 1586 (C=N), 1736 (CO, ester), and 1617 (C=C, Ar)cm^{-1} [9]. The ^{1}H NMR spectrum of **9a** consisted of signals at 3.70 (s, 3H, $COOCH_3$), 6.12 (s, 1H, =CH), 7.38, 7.72 ($J = 8$ Hz), 7.79, 8.34, 8.80, and 7.49–7.60 (arom.H) ppm. The ^{13}C NMR spectrum of **9a** showed signals – among others – at 123.5 (=*C*H), 156.9 (*C*$OOCH_3$), 169.2 (COPh), and 134.4 (C=N) ppm. The mass spectrum of **9a** had its M^{+} peak at $m/z = 317$, thus supporting the proposed structure.

The structure of the other isolated compound (**10a**) was deduced from its analytical, IR, ^{1}H NMR, and mass spectroscopic data. Elemental and mass spectroscopic data of **10a** pointed to an emperical formula of $C_{23}H_{19}NO_5$. Its IR spectrum (KBr) revealed the presence of strong NH absorption band at 3200 cm^{-1}. The carbonyl and C=N absorption bands appearing in the IR spectrum of **8** at 1656 and 1586 cm^{-1}, respectively, are lacking. The ^{1}H NMR spectrum of **10a** showed signals at 3.72 (s, 3H, $COOCH_3$), 3.82 (s, 3H, $COOCH_3$), 6.30 (s, 1H, =CH), 8.72 (s, 1H, NH), 7.38 (d, 1H, $J = 8$ Hz), 7.76–7.94 (m, 4H, aromatic), 7.51–7.56 (m, 5H, COPh), and 8.02 (dd, 1H, $J = 8$ and 1.6 Hz) ppm. The ^{13}C NMR spectrum of **10a** exhibits resonances at 132.5 (*C*=CH), 124.6 (C=*C*H), 167.2, 65.4 (*C*$OOCH_3$) and 170.5 (COPh) ppm. The mass spectrum of **10a** yielded an M^{+} peak of $m/z = 389$ (96%).

A possible explanation for the course of the reaction of the phosphonium ylides **1a,b** with **8** is shown in Scheme 2. Formation of adducts **9a,b** can be explained in terms of carbonyl olefination [10] of **8** by the *Wittig* reagents **1a**,**b** with expulsion of triphenyl phosphine oxide. Another molecule of **9a**,**b** reacts with phosphonium ylide to give adducts **10a**,**b**.

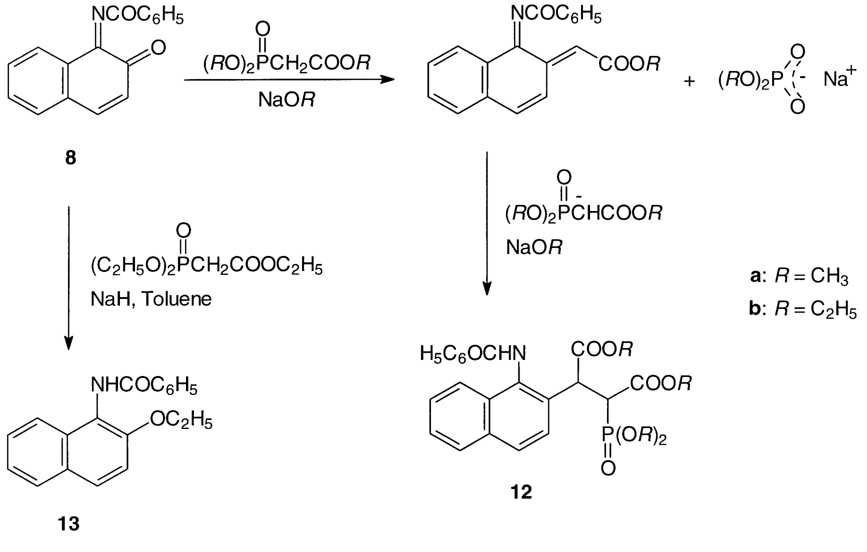
Similarly, carbethoxymethylenetriphenyl phosphorane (**1b**) reacts with 1,2-naphthoquinone-1-benzimide (**8**) in benzene at room temperature (10 hours) to give chromatographically pure adducts **9b** and **10b**, triphenylphosphine, triphenylphosphine oxide, and the reduced form of **8**. On the basis of spectroscopic and analytical data the structures of **9b** and **10b** were deduced (cf. Experimental).

This study was further extended to include the reaction of *Wittig-Horner* reagents **4a**,**b** with quinone monoimine **8** to test whether it would behave in a similar manner. We have found that the reaction of **8** with two molar equivalents of methyl dimethyl phosphonoacetate **4a** in the presence of alcoholic sodium methoxide solution proceeds at room temperature to give adduct **12a** and the corresponding amide **11** (Scheme 3). The structure of **12a** is based on the analytical and spectroscopic evidences given in the Experimental, including ^{31}P NMR spectroscopy [11, 12].

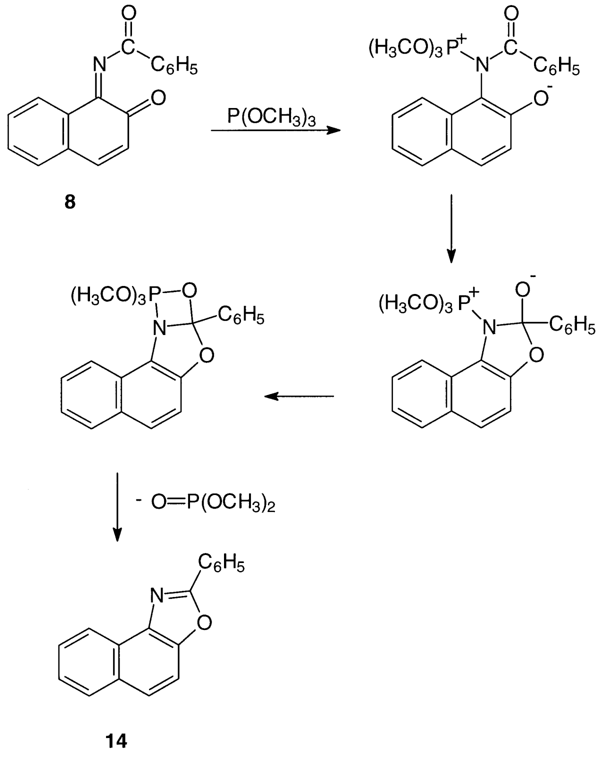
Similarly, triethyl phosphonoacetate **4b** reacts with quinone **8** at room temperature in the presence of sodium ethoxide to give colorless a product which was assigned structure **12b** (Scheme 3).

When **8** was allowed to react with one equivalent of the carbanion reagents **4** in refluxing toluene and in the presence of sodium hydride [13], the alkylated adduct **13** was isolated together with 1-benzamido-2-naphthol (**11**). The spectroscopic data of compound **13** were in good agreement with the proposed structure (cf. Experimental).

The reaction of **8** with alkyl phosphites was also investigated. We have found that reaction with trimethyl and/or dimethyl phosphite in dry benzene at room temperature gave naphthoxazol **14** as the sole product [14].



Scheme 3



Scheme 4

From the results of the present investigation it can be concluded that the reaction of **8** with phosphonium ylides **1**, phosphonate anions **4**, and alkyl phosphites **7** leads to different products, depending on the nature of the phosphorus reagents as well as on the stability of the addition products. The significance of these findings lies in the discovery of a new pattern of *Wittig* reagents and in the establishment of a novel method for the synthesis of new phosphonate products using *Wittig-Horner* reagents. It is noteworthy that **8** behaves towards alkyl phosphites in a manner different from that of *Wittig* and *Wittig-Horner* reagents.

Experimental

All melting points are uncorrected. Benzene (thiophene-free) and petroleum ether (60–80°C) were dried over sodium. Carbomethoxymethylene and carbethoxymethylene triphenylphosphoranes were prepared according to established methods [15]. Trimethyl and triethyl phosphonoacetates were prepared by *Michaelis-Arbuzov* reactions [16, 17]. Alkyl phosphites were prepared according to known procedures and purified by fractional distillation [18, 19]. The IR spectra were measured in KBr pellets on a Perkin-Elmer Infracord spectrophotometer Model 157 (Grating). The ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Varian NMR spectrometer at 200 and 50 MHz, respectively, using *TMS* as internal standard. The ^{13}P NMR spectra were recorded in $CDCl_3$ with a JNM-PS-100Fa spectrometer at 81 MHz (δ relative to external 85% H_3PO_4). The mass spectra were run at 70 eV using Kratos MS equipment and/or a Varian MAT 311 A Spectrometer.

Reaction of phosphoranes **1a**,**b** *with 1,2-naphthoquinone-1-benzimide* (**8**)

To a solution of **8** (0.26 g, 0.001 mol) [20] in dry benzene (20 cm^3), a solution of **1a** (0.99 g, 0.03 mol) in benzene was added and the reaction mixture was left at room temperature under stirring for 8 h. The volatile materials were evaporated under reduced pressure, and the residual substance was chromatographed on a silica gel column using ethyl acetate/petroleum ether (5:95 v/v) as eluent to give **9a** as pale yellow crystals.

Yield: 55%; m.p.: 135–136°C; $C_{20}H_{15}NO_3$ (317.34); calcd.: C 75.70, H 4.76, N 4.41; found: C 75.67, H 4.70, N 4.43; ^{1}H NMR: $\delta = 3.70$ (s, 3H, $COOCH_3$), 6.12 (s, 1H, =CH), 7.38 (d, 1H, $J = 8$ Hz), 7.72 (dd, 1H, $J = 8$ Hz, 1.6 Hz), 7.79, 8.34, 8.80, 7.79–7.60 (arom. H) ppm; ^{13}C NMR: $\delta = 123.5$ (=CH), 156.9 ($\textit{C}OOCH_3$), 169.2 (COPh), 54.0 (CH_3), 139.4 ppm; (C-1), 132.5 (C-2), 122.8 (C-3), 126.5 (C-4), 128.2, 126.7, 127.5, 128.7, 115.1, 128.9 (C_6H_4), 134.4, 129.5, 128.9, 132.3 (C_6H_5) ppm; MS: *m*/*z*(%) = 317 (M^+, 100), 266 (35); IR: $\tilde{\nu} = 1586$ (C=N), 1736 (C=O, ester), 1617 (C=C, Ar) cm^{-1}.

Adduct **10a** was separated (25:75) as colorless crystals; yield: 25%; m.p.: 206°C; $C_{23}H_{19}NO_5$ (389.41); calcd.: C 70.94, H 4.92, N 3.60; found: C 70.90, H 4.89, N 3.57; ^{1}H NMR: $\delta = 3.72$ (s, 3H, $COOCH_3$), 3.82 (s, 3H, $COOCH_3$), 6.30 (s, 1H, =CH), 8.72 (s, 1H, NH), 7.38 (d, 1H, $J = 8$ Hz), 7.76–7.94 (m, 4H, aromatic), 7.51–7.56 (m, 5H, COPh), 8.02 (dd, 1H, $J = 8$ Hz, 1.6 Hz) ppm; ^{13}C NMR $\delta = 132.5$ (*C*=CH), 124.6 (C=*C*H), 53.4 (CH_3), 52.5 (CH_3), 167.2 (CO, ester), 165.4 (CO, ester), 170.5 (COPh), 134.3 (C-1), 128.8 (C-2), 127.7 (C-3), 124.9 (C-4), 128.1, 126.5, 126.4, 128.4, 129.3, 134.2 (C_6H_4), 135.1, 130.3, 128.1, 132.7 (C_6H_5) ppm; MS: *m*/*z*(%) = 389 (M^+, 100), 358 (35) 285 (55), 105 (90); IR: $\tilde{\nu} = 1635$ (C=O, ester), 1616 (C=C, Ar), 3200 (NH) cm^{-1}.

Compound **11** was also isolated (10%) and proved to be the corresponding amide (mixed m.p. and comparative IR spectra) [21]. Triphenylphosphine and triphenyl phosphine oxide were isolated and identified (mixed m.p. and comparative IR spectra).

Similarly, carbethoxymethylene triphenylphosphorane (**1b**) reacts with **8** in dry benzene at room temperature for 10 h to give compound **9b** (eluent: ethyl acetate/petroleum ether 10:90 (v/v)) as pale yellow crystals.

Yield: 60%; m.p.: 112–113°C; $C_{21}H_{17}NO_3$ (331.371): calcd.: C 76.12, H 5.17, N 4.23; found: C 76.09, H 5.14, N 4.20; ^{1}H NMR: $\delta = 1.20$ (t, 3H, ethoxy-CH_3), 4.20 (q, 2H, ethoxy-CH_2), 6.15 (s, 1H, =CH), 7.41 (d, 1H, $J = 8$ Hz), 7.71 (d, 1H, $J = 8$ Hz, 1.6 Hz), 7.85 (br, 1H, aromatic), 7.51–7.62 (m, 5H, COPh), 8.36 (2H, aromatic), 8.81 (br, 1H, aromatic) ppm; ^{13}C NMR: $\delta = 156.4$ (CO, ester), 169.0 (COPh), 123.8 (=CH), 61.5 ($O\textit{C}H_2CH_3$), 13.9 ($OCH_2\textit{C}H_3$), 139.8 (C-1), 132.3 (C-2), 122.7 (C-3), 123.5 (C-4), 128.5, 126.7, 127.3, 126.4, 115.3, 128.9 (C_6H_4), 134.3, 129.3, 128.9, 131.6 (C_6H_5) ppm; IR: $\tilde{\nu} = 1580$ (C=N), 1740 (C=O, ester), 1617 (C=C, Ar) cm^{-1}; MS: *m*/*z*(%) = 331 (M^+, 55), 258 (100), 127 (40).

Compound **10b**: eluent as above (15:85, v/v); colorless crystals; yield: 20%; m.p..: 172–173°C; $C_{25}H_{23}NO_5$ (417.46); calcd.: C 71.93, H 5.55, N 3.36; found: C 71.90, H 5.50, N 3.33%; ^{1}H NMR: $\delta = 1.24$ (t, 3H, CH_3, $J = 7.2$ Hz), 1.18 (t, 3H, CH_3, $J = 7.2$ Hz), 4.22 (m, 4H, 2(CH_2)), 6.25 (s, 1H, =CH), 8.73 (s, 1H, NH), 7.45 (d, 1H, $J = 8$ Hz), 7.85–7.94 (m, 4H, aromatic), 7.51–7.56 (m, 5H, COPh), 8.04 (dd, 1H, $J = 8$ Hz, 1.6 Hz) ppm; ^{13}C NMR: $\delta = 62.6$, 61.4 (2(CH_2)), 13.1 (CH_3), 14.2 (CH_3), 168.2 (CO, ester), 169.4 ppm (CO, ester), 123.7 (C=*C*H), 170.2 (COPh), 134.8 (C-1), 128.3 (C-2), 127.9 (C-3), 125.2 (C-4), 128.1, 126.5, 126.4, 128.4, 129.3, 133.2 (C_6H_4), 135.3, 130.3, 128.2, 132.1 (C_6H_5) ppm; MS: *m*/*z*(%) = 417 (M^+, 50), 312 (20), 372 (15), 193 (22); IR: $\tilde{\nu} = 3250$ (NH), 1730 (C=O, ester), 1745 (C=O, ester), 1600 (C=C, Ar) cm^{-1}.

Reaction of phonoacetates **4a**,**b** *with 1,2-naphthoquinone-1-benzimide* (**8**)

A solution of 2 mol of sodium methoxide in absolute methanol was treated with an equimolar amount of phosphonoacetate anion **4a** (0.42 g, 002 mol). After 5 min, 1 mol of **8** (0.26 g, 0.001 mol) was added, and the resulting mixture was stirred at room temperature for 4 h. The reaction mixture was extracted with ethyl acetate, and the extracts were evaporated under reduced pressure. The residue was subjected to silica gel column chromatography using ethyl acetate/petroleum ether (70:30 (v/v)) as eluent to give compound **12a** as colorless crystals (cyclohexane).

Yield: 50%; m.p.: 123–124°C; $C_{25}H_{26}NO_8P$ (499.46); calcd.: C 60.12, H 5.25, N 2.80, P 6.20; found: C 60.08, H 5.23, N 2.82, P 6.18; ^{1}H NMR: $\delta = 3.68$ (s, 3H, OCH_3), 3.71 (s, 3H, OCH_3), 2.97 (dd, 1H, $J_{HP} = 12$ Hz, $J_{HH} = 7.5$ Hz), 3.13 (dd, 1H, $J_{HP} = 10$ Hz, $J_{HH} = 7.5$ Hz), 3.74, 3.82 (2s, 6H, $2COOCH_3$), 7.18 (d, 1H, $J = 8$ Hz), 7.43–7.65 (m, 5H, COPh), 7.76–7.88 (m, 4H, aromatic), 8.04 (dd, 1H, $J = 8$ Hz, 1.6 Hz), 8.75 (s, 1H, NH) ppm; ^{13}C NMR: $\delta = 167.5$, 165.0 (2(COOCH$_3$)), 50.4, 52.3 (2(COOCH$_3$)), 45.3 (d, $^1J_{CP} = 140$ Hz, HC-P(O)), 53.1, 55.1 (2d, $^2J_{PC} = 7.0$ Hz, $O = P(OCH_3)_2$) ppm; ^{31}P NMR: $\delta = 20.7$ ppm; MS: m/z(%) = 499 (M^+, 12), 390 (20), 331 (18); IR: $\tilde{\nu} = 3290$ (NH), 1230 (P=O), 1056 (P–OCH_3) cm^{-1}.

Similarly, the anion of triethylphosphonoacetate **4b** reacts with **8** in absolute ethanol to give colorless adduct **12b**, (acetone: petroleum ether, 70:30 (v/v)); yield: 60%; m.p.: 125–126°C; $C_{29}H_{34}NO_8P$ (555.56); calcd.: C 62.70, H 6.16, N 2.52, P 5.58; found: C 62.68, H 6.12, N 2.50, P 5.54; ^{1}H NMR: $\delta = 1.15$, 1.28 (2t, 6H, $O{=}P(OCH_2CH_3)_2$), 0.85 (t, 3H, $COOCH_2CH_3$), 1.45 (t, 3H, $COOCH_2CH_3$), 3.60 (dd, 1H, $J_{HP} = 12$ Hz, $J_{HH} = 7.5$ Hz), 3.73 (dd, 1H, $J_{HP} = 10$ Hz, $J_{HH} = 7.5$ Hz), 4.42 (m, 4H, $P(OCH_2CH_3)$)), 4.20 (m, 4H, $2(COOCH_2CH_3)$)), 8.50 (s, 1H, NH), 7.05 (d, 1H, $J = 8$ Hz), 7.43–7.62 (m, 5H, COPh), 7.72–7.87 (m, 4H, aromatic), 8.09 (dd, 1H, $J = 8$ Hz, 1.6 Hz) ppm; ^{31}P NMR: $\delta = 21.02$ ppm; MS: m/z(%) = 555 (M^+, 10), 465 (12), 328 (16), 246 (30); IR: $\tilde{\nu} = 3200$ (NH), 1235 (P=O), 1752 (C=O, ester), 1740 (C=O, ester), 1620 (C=C, Ar) cm^{-1}.

The reaction of **4b** (0.001 mol) with **8** (0.001 mol) in refluxing toluene using sodium hydride (0.001 mol) as a base instead of sodium ethoxide gave rise to the corresponding alkylated product **13** as colorless crystals; yield: 60%; m.p.: 220-221°C; $C_{19}H_{17}NO_2$ (291.35); calcd.: C 78.33, H 5.88, N 4.81; found: C 78.30, H 5.84, N 4.78; ^{1}H NMR: $\delta = 1.47$ (t, 3H, OCH_2CH_3 $J = 7.2$ Hz), 4.75 (q, 2H, OCH_2CH_3, $J = 7.2$ Hz), 7.48–7.73 (m, 5H, CO, Ph), 7.81–7.84 (m, 6H, aromatic), 8.80 (s, 1H, NH) ppm; ^{13}C NMR: $\delta = 169.7$ (COPh), 67.9 (CH_2), 14.9 (CH_3), 124.6 (C-1), 150.4 (C-2), 112.6 (C-3), 123.5 (C-4), 128.1 126.2, 127.6, 128.7, 126.4 128.8 (C_6H_4), 133.3 129.2, 128.5, 132.2 (C_6H_5) ppm; MS: m/z(%) = 291 (10), 245 (100); IR: $\tilde{\nu} = 3262$ (NH), 1671 (C=O) cm^{-1}.

Reaction of alkyl phosphites **7** *with 1,2-naphthoquinone-1-benzimide* (**8**)

A mixture of freshly distilled trimethyl phosphite and/or dimethyl phosphite **7** (0.03 mol), **8**, and dry benzene (30 cm^3) was stirred at room temperature for 4 h. The volatile materials were evaporated under reduced pressure, and the residue was applied on a silica gel column using ethyl acetate/petroleum ether (5:95, (v/v)) as eluent to give **14** as colorless crystals.

Yield: 80%; m.p.: 126°C; $C_{17}H_{11}NO$ (245.28); calcd.: C 83.25, H 4.52, N 5.71; found: C: 83.20, H 4.50, N 5.67; ^{1}H NMR: $\delta = 7.43$–7.95 (m, 6H, aromatic), 8.32–8.60 (m, 5H, COPh) ppm; ^{13}C NMR: $\delta = 162.2$ (N=C), 137.5 (C-1), 147.9 (C-2), 110.7 (C-3), 125.3 (C-4), 128.1, 126.9, 126.5, 127.4, 128.8, 131.0 (C_6H_4), 122.2, 127.2, 128.5, 131.1 (C_6H_5) ppm; MS: m/z(%) = 245 (90).

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