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Solid–Liquid Phase Alkylation of P=O–Functionalized CH Acidic Compounds Utilizing Phase Transfer Catalysis and Microwave Irradiation

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ABSTRACT: *Optimum conditions for the solid–liquid phase alkylation of methylenebis(diphenylphosphine oxide) (MBDPPO) and ethyl cyanomethylphosphonate (ECMP) were explored studying the role of phase transfer catalysis and microwave (MW) irradiation, as well as the effect of the cation of the alkali carbonate. It was found that the alkylation of MBDPPO may be best accomplished in acetonitrile, in the presence of a quaternary ammonium salt and Cs_2CO_3, while that of ECMP in the absence of catalyst and solvent using K_2CO_3. MW irradiation was beneficial in both cases. During the alkylation of ECMP, by-products coming from the alcoholysis of the diethyl ester were also identified.* © 2011 Wiley Periodicals, Inc. Heteroatom Chem 22:174–179, 2011; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20673

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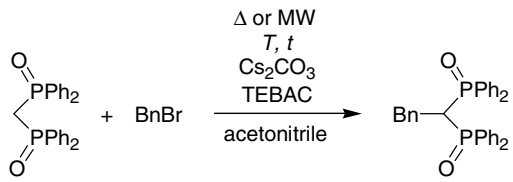
INTRODUCTION

Substituted CH-acidic compounds are important intermediates in the pharmaceutical industry. Substituted malonates are used in the synthesis of barbiturates that are hypnotics, whereas bisphosponic derivatives are applied in the treatment of osteoporosis [1–5]. The C-alkylation of methylenebisphosphonates requires harsher conditions than that for malonic or acetoacetic esters. The potassium salt of tetraethylmethylenebisphosphonate was formed by reaction with potassium in xylene [6], whereas the sodium salt was formed by the interaction with sodium hydride [7]. The salts so obtained were reacted with alkyl halides to afford the alkylated derivatives in variable yields [6,7]. Alkylations with benzylic bromides and propargyl bromide were preceded by salt formation using sodium hydride as shown in recent examples [8–10]. The alkylation with propargyl bromide led to a mixture of mono- and dialkylated products, which were impossible to separate, and an alternative approach had to be elaborated for the monopropargylation [9,10].

With regard to "classical" CH-acidic compounds, alkylation by the phase transfer catalytic (PTC) method is an up-to-date procedure [11]. The

TABLE 1 Benzylation of Bis(Diphenylphosphinyl)methane (**1**)



1 **2a**

RX	*Mode of Heating*	*T (°C)*	*t (h)*	*TEBAC (10%)*	*Conversion (%)*[a]	*Yield of **2a** (%)*	*Entry*
BnBr	Δ	82	24	+	60	44	1
BnBr	MW	120	1.5	–	21		2
BnBr	MW	120	1.5	+	43		3
BnBr	MW	140	1.5	+	38		4
BnBr	MW	120	3	+	57	45	5

[a]On the basis of ^{31}P NMR.

combination of the PTC and the microwave (MW) techniques may offer additional advantages [12]. We found that in the solid–liquid phase alkylation of malonic, acetoacetic, and cyanoacetic esters, the phase transfer catalyst may be substituted by MW irradiation [13,14]. This method could also be extended to the ethylation of tetraethyl methylenebisphosphonate [15].

A few methylenebis(phosphine oxides) were monomethylated in high yields [16]. Bis(diphenylphosphinyl)methane, which is a rather hindered substrate, could be alkylated via the potassium salt [17]. At the same time, the alkylation of ethyl cyanomethylphosphonate (ECMP), which is a mixed CH acidic species, was carried out by forming the salt by sodium hydride in DMF and reacting the salt so formed with an alkyl halide [18], or by liquid–liquid phase transfer catalysis applying aqueous alkali hydroxide as the base [19]. Alternative procedures for the preparation of $NCCHRP(O)(OEt)_2$ were also described [20,21]. 2-Oxoalkanephosphonates were alkylated via the sodium salt formed by reaction with sodium hydride [22,23]. The structure and synthetic use of α-phosphono carbanions (e.g., the phosphonoacetate carbanion) were studied in detail by Seyden-Penne and her coworkers. [24].

In this article, the effect of MW irradiation on the PTC alkylation of bis(diphenylphosphinyl)methane and ECMP is studied to find easily accomplishable and environment friendly efficient methods for the synthesis of C-alkyl derivatives.

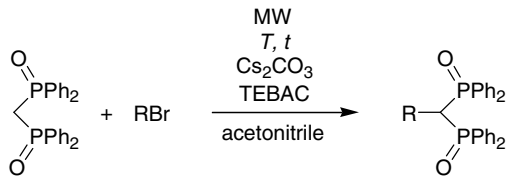
RESULTS AND DISCUSSION

First the benzylation of bis(diphenylphosphinyl) methane (**1**) was investigated. In all cases, acetonitrile was used as the solvent and Cs_2CO_3 as the base. In boiling acetonitrile, in the presence of triethylbenzylammonium chloride (TEBAC), the conversion was 60% after 1 day (Table 1, entry 1). We expected a shorter reaction time under MW conditions. Working at 120°C, the conversion was 21% after 1.5 h (Table 1, entry 2), but if there was 10% TEBAC in the reaction mixture, the conversion was 43% (Table 1, entry 3). Allowing a 3-h reaction time, a conversion of 57% could be achieved (Table 1, entry 5). However, an increase in the reaction temperature (120 → 140°C) did not help (Table 1, entry 4). The hindered model prevented more complete conversions. From one of the best experiments (Table 1, entry 5), the product (**2a**) was isolated by chromatography in a yield of 45%.

Then the alkylation of bis(diphenylphosphinyl) methane **1** by propyl and butyl bromide was studied. The experiments were carried out in the presence of TEBAC and Cs_2CO_3 in acetonitrile under MW irradiation. Using propyl bromide and increasing the temperature from 120 to 180°C, the proportion of the alkylated product (**2b**) was 30 and 47% (after a reaction time of 3 and 4 h, respectively) (Table 2, entries 1 and 4). Entries 2 and 3 of Table 2 represent intermediate cases. It can be seen that the best conversions amounted to ~64%, and unidentified by-products were formed in a quantity of up to 18%. Adopting the best set of parameters (180°C/4 h) for the preparation of the buthylated bis(diphenylphosphinyl)methane (**2c**), its proportion was 52% (Table 2, entry 5). Preparative yields of products **2b** and **2c** were 40 and 46% after chromatography.

Diethyl cyanomethylphosphonate (**3**) was expected to have a more enhanced reactivity toward alkylation. The first experiments were carried out in boiling acetonitrile for 1 day applying propyl

TABLE 2 Alkylation of Bis(Diphenylphosphinyl)Methane (**1**)



R = Pr, Bu

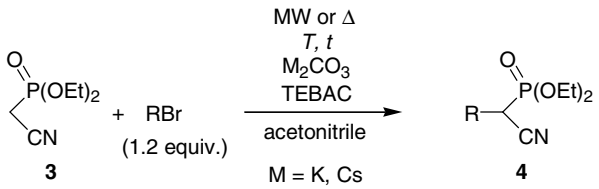
R = Pr (**b**), Bu (**c**)

			Composition (%)[a]				
RX	*T* (°C)	*t* (h)	***1***	***2***	*Other*	*Yield of **2** (%)*	*Entry*
PrBr	120	3	62	30 (**2b**)	8		1
PrBr	140	3	46	37 (**2b**)	17		2
PrBr	160	4	38	44 (**2b**)	18		3
PrBr	180	4	36	47 (**2b**)	17	40	4
BuBr	180	4	26	52 (**2c**)	22	46	5

[a]On the basis of ^{31}P NMR.

bromide. Using K_2CO_3 and Cs_2CO_3, the conversions were 47 and 93%, respectively, providing the propyl product (**4**) in 45 and 84%, respectively (Table 2, entries 1 and 3). The use of phase transfer catalyst (TEBAC) led to decreased proportions of product **4** and 31–37% of other components (Table 2, entries 2 and 4). Hence, the presence of TEBAC is not beneficial in the reaction under discussion. The MW variations were accomplished at temperatures ≥100 for 1–2 h, in the absence of solvent. The best results were obtained at 100°C/2 h using K_2CO_3. In this case, the conversion was 87%, and product **4** was present in 70% in the reaction mixture (Table 3, entry 5). The use of catalyst was disadvantageous (as above)

TABLE 3 Alkylation of Diethyl Cyanomethylphosphonate **3**



R = Pr, Bu

R = Pr (**b**), Bu (**c**)

							Composition (%)[a]				
RX	*M*	*Mode of Heating*	*T* (°C)	*t* (h)	*Solvent*	*TEBAC (10%)*	***3***	***4***	*Other*	*Yield of **4** (%)*	*Entry*
PrBr	K	Δ	82	24	MeCN	–	53	45 (**4b**)	2		1
PrBr	K	Δ	82	24	MeCN	+	26	37 (**4b**)	37		2
PrBr	Cs	Δ	82	24	MeCN	–	7	84 (**4b**)	9	75	3
PrBr	Cs	Δ	82	24	MeCN	+	2	67 (**4b**)	31		4
PrBr	K	MW	100	2	–	–	13	70 (**4b**)	17	64	5
PrBr	K	MW	120	1	–	–	20	58 (**4b**)	22		6
PrBr	K	MW	120	1	–	+	2	32 (**4b**)	66		7
PrBr	Cs	MW	120	1	–	–	4	33 (**4b**)	63		8
PrBr	Cs	MW	120	1	–	+	3	24 (**4b**)	73		9
BuBr	Cs	Δ	82	24	MeCN	–	3	90 (**4c**)	7	82	10
BuBr	K	MW	100	2	–	–	45	51 (**4c**)	4		11
BuBr	K	MW	120	2	–	–	20	65 (**4c**)	15	59	12
BuBr	K	MW	110	1	–	+	6	31 (**4c**)	63		13

[a]On the basis of GC.

(Table 3, entries 6 and 7). Switching to the use of Cs_2CO_3, the conversions were almost complete after 1 h, but by-products predominated over expected product **4** (Table 3, entries 8 and 9). Product **4b** was isolated from the best experiments (Table 3, entries 3 and 5) in 75 and 64% yields after chromatography.

The alkylation of diethyl cyanomethylphosphonate **3** with butyl bromide led to similar results. Almost complete conversion could be attained in refluxing acetonitrile, in the presence of Cs_2CO_3, but the butylation was slow (Table 3, entry 10). Under MW and solventless conditions, an irradiation of 120°C/2 h seemed to be more suitable than 100°C/2 h (Table 3, entries 11 and 12). In the latter case, the conversion was 80% and the required product (**4c**) was formed in 65% proportion. In the presence of catalyst, less starting material (**3**) remained in the mixture but the proportion of the by-products increased to 63% (Table 3, entry 13). Preparative yields of **4c** were 82 and 59% from the best experiments (Table 3, entries 10 and 12).

GC-MS analysis of the crude mixture of the experiment covered by Table 3, entry 13 suggested that the major by-products came from the single or double alcoholysis of diethyl ester **4c**. Moreover, applying the butyl bromide in 2.4 and 3.6 equivalent quantities using TEBAC in 20 and 30%, respectively, the relative proportions of monobutyl ester **5** and dibutyl ester **6** were somewhat increased indicating the role of the quantity of the reactant (Table 4, entries 1–3).

Formation of the by-product **5** and **6** can be explained by assuming that water is released in the reaction of hydrogen bromide (formed in the alkylation) with alkali carbonate under the circumstances of the reaction that may lead to the partial hydrolysis of the tetraethyl ester or the alkyl halide. Then, either the acid function may react with the alkyl halide, or the ester itself with the alcohol to furnish mixed ester **5** and **6**.

It can be seen that MW irradiation made possible shorter reaction times. With regard to the alkylation of the hindered bis(diphenylphosphinyl)methane **1** and the more reactive cyanomethylphosphonate, the conditions required are different. In the previous case, the use of solvent and catalyst is appropriate, while in the latter instance, it is better not to apply solvent and catalyst.

The alkylated products **2a–c** and **4b** were mentioned in earlier literature [17,20,25–27], but only **4b** was characterized spectroscopically. Now, alkyl-methylenebis(phosphine oxides) **2a–c** and cyanopentylphosphonate **4c** were characterized by ^{31}P, ^{13}C, and ^{1}H NMR, as well as mass spectral methods.

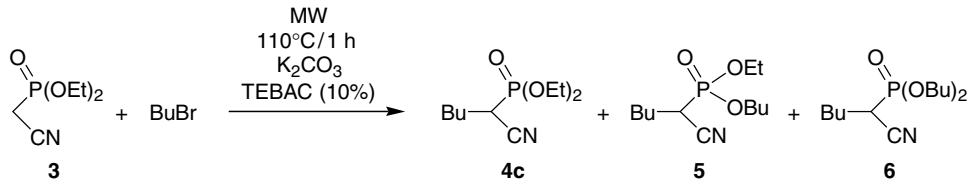
In conclusion, it can be seen that the optimal conditions for the solid–liquid phase C-alkylation of P=O-functionalized CH-acidic compounds are dependent on the nature of the substrate. MW irradiation was beneficial in all cases. The alkylation of bis(diphenylphosphinyl)methane could be best accomplished in the presence of solvent and catalyst using Cs_2CO_3, whereas the alkylation of cyanomethylphosphonate was optimal in the absence of solvent and catalyst using K_2CO_3.

EXPERIMENTAL

General

The ^{31}P, ^{13}C, and ^{1}H NMR spectra were obtained on a Bruker DRX-500 spectrometer operating at 202.4, 125.7, and 500 MHz, respectively. Chemical shifts are downfield relative to 85% H_3PO_4 or TMS. The couplings are given in hertz. Mass spectrometry was performed on a ZAB-2SEQ instrument.

TABLE 4 The Reaction of Diethyl Cyanomethylphosphonate **3** with Butyl Bromide under Solventless MW Conditions



3 **4c** **5** **6**

		Composition (%)[a]					
Quantity of BuBr (equiv)	*Quantity of TEBAC (%)*	***3***	***4c***	***5***	***6***	*Other*	*Entry*
1.2	10	6	31	31	8	24	1
2.4	20	0	20	38	21	21	2
3.6	30	0	14	37	25	24	3

[a]On the basis of GC.

The MW-assisted reactions were carried out in a CEM Discover MW reactor equipped with a pressure controller using ca. 30 W irradiation.

General Procedure for the Alkylation of Bis(diphenylphosphinyl)methane 1 under MW Conditions

The reaction components 0.20 g (0.48 mmol) of bis(phosphinyl)methane **1**, 0.58 mmol of alkyl halide (benzyl bromide: 0.07 mL, propyl bromide: 0.05 mL, butyl bromide: 0.06 mL), 0.16 g (0.48 mmol) of Cs_2CO_3, and 0.01 g (0.05 mmol) of TEBAC along with 5 mL of acetonitrile as the solvent were measured in a tube that was placed in the MW reactor and was irradiated under pressure control by 30–70 W at the appropriate temperature for the appropriate time. Then the mixture was filtered and the solid was washed with 2 mL of ethyl acetate. Combined organic phases were concentrated in vacuum, and the residue so obtained was purified by column chromatography (3% methanol in chloroform, silica gel) to afford products **2a–c**. For details, see Tables 1 and 2 and the list below.

The benzylation of **1** was also carried out using 0.20 g (0.48 mmol) of **1**, 0.07 mL (0.58 mmol) of benzyl bromide, 0.16 g (0.48 mmol) of Cs_2CO_3, 0.01 g (0.05 mmol) of TEBAC, and 5 mL of acetonitrile under traditional thermal conditions according to Table 1, entry 1. The workup procedure was similar to that described above.

*1,1-Bis(diphenylphosphinyl)-2-phenylethane (***2a***).* Yield: 45%, mp. 218–219°C, mp. [17] 217–218°C; ^{31}P NMR ($CDCl_3$) δ: 31.6; ^{13}C NMR ($CDCl_3$) δ: 31.3 (CH_2), 45.7 (t, J = 56.0, CH), 126.0 (Ar), 128.0 (Ar), 128.1 (J = 12.2, Ar), 128.2 (Ar), 128.4 (J = 12.2, Ar), 131.4 (J = 101.8, Ar), 131.4 (J = 9.2, Ar), 131.7 (dd, J = 100.0, J = 2.9, Ar) 131.5 (Ar), 131.6 (J = 8.3, Ar); ^{1}H NMR ($CDCl_3$) δ: 3.20 (dt, J = 15.5, J = 5.3, 2H, CH_2), 3.71 (tt, J = 15.9, J = 5.3, 1H, CH), 6.69 (d, J = 6.8, 2H, ArH), 6.96 (m, 3H, ArH), 7.14 (m, 4H, ArH), 7.38–7.24 (m, 4H, ArH), 7.61 (d, J = 7.8, 2H, ArH), 7.64 (d, J = 7.9, 2H, ArH), 7.87 (d, J = 7.7, 2H, ArH), 7.89 (d, J = 7.7, 2H, ArH); HRMS, M^+_{found} = 507.1653, $C_{32}H_{29}O_2P_2$ requires 507.1643.

*1,1-Bis(diphenylphosphinyl)butane (***2b***).* Yield: 40%, mp. 185–187°C; ^{31}P NMR ($CDCl_3$) δ: 31.8; ^{13}C NMR ($CDCl_3$) δ: 13.7 (s, CH_3), 23.5 (t, J = 5.5, CH_2), 28.1 (CH_2), 43.3 (t, J = 57.2, CH), 128.1 (J = 12.1, Ar), 128.2 (J = 12.2, Ar), 131.3 (Ar), 131.3 (J = 101.3, Ar), 131.4 (J = 13.7, Ar), 131.4 (J = 15.9, Ar), 131.5 (Ar), 132.2 (dd, J = 102.6, J = 2.8, Ar); ^{1}H NMR ($CDCl_3$) δ: 0.53 (t, J = 7.3, 3H, CH_3), 1.19 (m, 2H, CH_2), 1.86 (m, 2H, CH_2), 3.37–3.20 (m, 1H, CH), 7.36–7.24 (m, 12H, ArH), 7.70 (d, J = 7.4, 2H, ArH), 7.74 (d, J = 7.4, 2H, ArH), 7.87 (d, J = 7.5, 2H, ArH), 7.91 (d, J = 8.1, 2H, ArH); HRMS, M^+_{found} = 459.1640, $C_{28}H_{29}O_2P_2$ requires 459.1643.

*1,1-Bis(diphenylphosphinyl)pentane (***2c***).* Yield: 46%, mp. 204–205°C, mp. [17] 204–206°C; ^{31}P NMR ($CDCl_3$) δ: 31.7; ^{13}C NMR ($CDCl_3$) δ: 13.3 (CH_3), 22.3 (CH_2), 25.9 (CH_2), 32.5 (t, J = 5.3, CH_2), 43.7 (t, J = 57.1, CH), 128.3 (J = 12.1, Ar), 128.4 (J = 12.1, Ar), 131.5 (Ar), 131.6 (J = 101.2, Ar), 131.6 (J = 9.6, Ar), 131.6 (J = 9.4, Ar), 131.7 (Ar), 132.5 (dd, J = 99.8, J = 2.8, Ar); ^{1}H NMR ($CDCl_3$)δ: 0.49 (t, J = 7.3, 3H, CH_3), 0.91 (m, 2H, CH_2), 1.16 (m, 2H, CH_2), 1.87 (m, 2H, CH_2), 3.33–3.20 (m, 1H, CH), 7.35–7.24 (m, 12H, ArH), 7.71 (d, J = 7.9, 2H, ArH), 7.75 (d, J = 7.8, 2H, ArH), 7.88 (d, J = 7.7, 2H, ArH), 7.91 (d, J = 8.3, 2H, ArH); HRMS, M^+_{found} = 473.1800, $C_{29}H_{31}O_2P_2$ requires 473.1799.

The Best Procedure for the Propylation of Diethyl Cyanomethylphosphonate **3** *under Traditional Conditions (Table 3, entry 3).* The mixture of 0.25 g (1.4 mmol) phosphonate **3**, 0.15 mL (1.7 mmol) of propyl bromide, 0.52 g (1.4 mmol) of Cs_2CO_3 in 5 mL acetonitrile was heated at reflux for 24 h. The contents of the flask were filtrated, the solid washed with 2 mL of acetonitrile, and the combined organic phases evaporated. The residue so obtained was purified by column chromatography (3% methanol in chloroform, silica gel) to give 0.23 g (75%) of **4b**.

The Best Procedure for the Propylation of Diethyl Cyanomethylphosphonate **3** *under MW Conditions (Table 3, entry 5).* The mixture of 0.12 g (0.68 mmol) of phosphonate **3**, 0.07 mL (0.81 mmol) of propyl bromide, and 0.09 g (0.68 mmol) of K_2CO_3 in a tube was placed in the MW reactor and was irradiated under pressure control by 30 W at 100°C for 2 h. The workingup procedure was similar described above to give 0.096 g (64%) of **4b**.

*Diethyl 1-Cyanobutylphosphonate (***4b***).* ^{31}P NMR ($CDCl_3$) δ: 18.3, δ_P [20] 18.7; ^{13}C NMR ($CDCl_3$) δ: 13.2 (CH_2CH_3), 16.4 (J = 4.9, OCH_2CH_3), 21.2 (J = 12.6, CH_2), 28.9 (J = 4.4, CH_2), 29.8 (J = 143.8, CH), 63.7 (J = 6.9, OCH_2), 64.0 (J = 7.0, OCH_2), 116.3 (J = 9.4, CN); ^{1}H NMR ($CDCl_3$) δ: 1.00 (dt, J = 7.2, J = 1.6, 3H, CH_3), 1.39 (t, J = 7.1, 3H, CH_3), 1.40 (t, J = 7.1, 3H, CH_3), 1.48–1.57 and 1.69–1.77 (m, 2H, CH_2), 1.82–1.93 (m, 2H, CH_2), 2.87–3.00 (m, 1H, CH), 4.18–4.32 (m, 4H, OCH_2); HRMS, M^+_{found} = 220.1103, $C_9H_{19}NO_3P$ requires 220.1103.

*Diethyl 1-Cyanopentylphosphonate (**4c**).* Product **4c** was prepared by the MW method (Table 3, entry 12). Yield: 59%; ^{31}P NMR ($CDCl_3$) δ: 18.23; ^{13}C NMR ($CDCl_3$) δ 13.7 (CH_2CH_3), 16.40 (J = 5.8, OCH_2CH_3), 16.42 (J = 5.8, OCH_2CH_3), 21.9 (CH_2CH_3), 26.7 (J = 4.4, CH_2), 30.0 (J = 143.8, CH), 30.0 (J = 12.2, CH_2), 63.7 (J = 6.9, OCH_2), 64.1 (J = 7.9, OCH_2), 116.3 (J = 9.3, NC); ^{1}H NMR ($CDCl_3$) δ: 0.92 (t, J = 7.3, 3H, $CH_2CH_2CH_3$), 1.37 (t, J = 7.0, 6H, OCH_2CH_3), 1.416–1.52 and 1.59–1.69 (m, 4H, CH_2) 1.78–1.95 (m, 2H, CH_2), 2.83–2.92 (m, 1H, CH), 4.19–4.25 (m, 4H, OCH_2CH_3); $[M + Na]^+_{found}$ = 256.1084, $C_{10}H_{20}O_3NPNa$ requires 256.1079.

*Butyl, Ethyl 1-Cyanopentylphosphonate (**5**).* ^{31}P NMR ($CDCl_3$) δ: 18.22; $[M + H]^+_{found}$ = 262.1577, $C_{12}H_{25}O_3NP$ requires 262.1572.

*Dibutyl 1-Cyanopentylphosphonate (**6**).* ^{31}P NMR ($CDCl_3$)δ: 18.29; $[M + H]^+_{found}$ = 290.1892, $C_{14}H_{29}O_3NP$ requires 290.1885.

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