Heteroatom Chemistry
Volume 23, Number 3, 2012

Microwave-Assisted Phospha-Michael Addition of Dialkyl Phosphites, a Phenyl-*H*-Phosphinate, and Diphenylphosphine Oxide to Maleic Derivatives

Erika Bálint,[1] Judit Takács,[1] László Drahos,[2] and György Keglevich[1]

[1]*Department of Organic Chemistry and Technology, Budapest University of Technology and Economics, 1521 Budapest, Hungary*

[2]*Hungarian Academy of Sciences, Chemical Research Center, 1525 Budapest, Hungary*

Received 1 December 2011; revised 11 January 2012

ABSTRACT: *A series of new >P(O)-substituted succinic derivatives was synthesized by the microwave-assisted phospha-Michael addition of dialkyl phosphites, ethyl phenyl-H-phosphinate, and diphenylphosphine oxide to N-phenyl and N-methyl maleimide, as well as to maleic acid anhydride.* © 2012 Wiley Periodicals, Inc. Heteroatom Chem 23:235–240, 2012; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.21007

INTRODUCTION

The phospha-Michael addition is an important P—C bond-forming reaction in synthetic organic chemistry. The nucleophiles may include tervalent P-reagents, such as phosphines, phosphinites, and phosphonites, or more often >P(O)H species, such as dialkyl phosphites, secondary phosphine oxides, and other related derivatives [1–6]. The addition of >P(O)H species to α,β-unsaturated ketones and esters is usually carried out under basic conditions to afford γ-ketophosphonates, γ-ketophosphinates, or γ-ketophosphine oxides. Asymmetric phospha-Michael variations using chiral P-reagents are also well known [7–9]. The addition of >P(O)H species to α,β-unsaturated C=O or P=O compounds may lead to derivatives that are of potential biological activity [10,11].

A few maleic derivative-related phospha-Michael reactions are known from the literature. The adduct formed from the interaction of *N*-phenylmaleimide and diphenylphosphine oxide was isolated in 73% after boiling the components in toluene for 8 h [12]. Applying 1,5,7-triazabicyclo[4.4.0]dec-5-ene (TBD) as the catalyst and toluene as the solvent, the addition was already completed after 5 min at 26°C [13]. Besides diphenylphosphine oxide, diphenyl phosphite was also used as the reagent [13]. The $\eta^1 - N$-maleimidato ligand has also been shown to undergo the phospha-Michael reaction with >P(O)H species in the coordination sphere [14]. *N*-Arylmaleimides were reacted with bis(pentafluorophenyl)phosphine oxide in chloroform at the boiling point [15]. It is noteworthy that the addition of silylphosphines to *N*-phenylmaleimide followed by oxidation with 30% hydrogen peroxide also led to maleimide- >P(O)H adduct-type products [16].

One author of this article investigated the addition of >P(O)H species under different conditions to simple model compounds [17,18]. Later

Correspondence to: György Keglevich; e-mail: gkeglevich@mail.bme.hu.

Contract grant sponsor: Hungarian Scientific and Research Fund.
Contract grant sponsor: OTKA K83118.

on, >P(O)H species were added on the double bond of 1-phenyl-2-phospholene 1-oxide and 1-substituted-1,2-dihydrophosphinine oxides to afford a 3-(>P(O)-)phospholane oxide [19] and 3-(>P(O)-) 1,2,3,6-tetrahydrophosphinine oxides, respectively [20–22]. The bis(phosphinoxido) derivatives served as bidentate P-ligands after double deoxygenation and led to *cis* chelate ring Pt complexes on reaction with dichlorodibenzonitrile platinum [19,23]. Because of the unreactivity of the substrates, microwave (MW) irradiation was not useful in the phospha-Michael reaction of 2-phospholene 1-oxides and 1,2-dihydrophosphinine 1-oxides.

In this paper, we investigate the phospha-Michael additions of >P(O)H species to maleic derivatives in which MW irradiation may promote the reaction.

RESULTS AND DISCUSSION

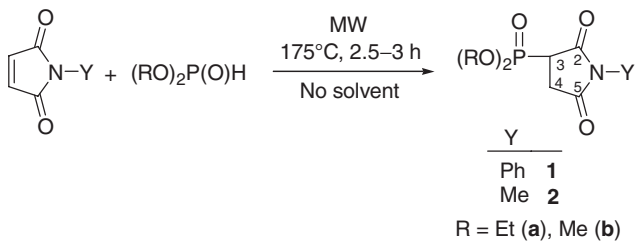
In the first experiments, dialkyl phosphites, such as diethyl phosphite and dimethyl phosphite, were reacted with *N*-phenyl- and *N*-methylmaleimides (NPMI and NMMI) under solventless conditions and MW irradiation. Completion of the additions at 150°C required a reaction time of 6–7 h. Applying a reaction temperature of 175°C, the addition of dialkyl phosphites to NPMI was complete after 2.5 h, whereas with NMMI the reaction time was 3 h. This experience is the consequence of the somewhat more enhanced reactivity of NPMI as compared to NMMI. The 3-(>P(O)-)-substituted succinimide derivatives (**1a**, **1b**, **2a,** and **2b**) were obtained in 71–95% yields after column chromatography (Table 1, entries 1–4; Scheme 1).

The addition of ethyl phenyl-*H*-phosphinate to maleimide derivatives was carried out under similar conditions (MW, 175°C, 0.5-3 h) to afford 3-P(O)PhEtO-succinimides **3** and **4** (Scheme 2). Because of the P-chiral center in the reagent and the prochiral center in the substrate, the Michael adducts (**3** and **4**) were formed as a ~51:49 mixture of two diastereomers. The yields of products **3** and**4** were 92% and 80%, respectively (Table 1, entries 5 and 6).

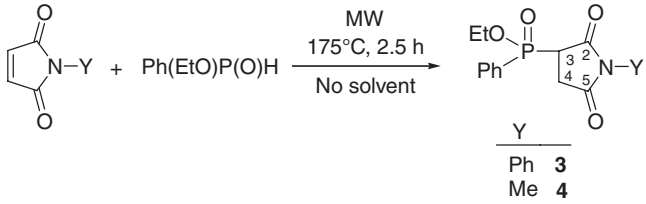
Diphenylphosphine oxide could already be added on the double bond of maleimides at 120°C/3 h on MW irradiation. The 3-$Ph_2P(O)$-succinimides **5** and **6** were obtained in 97% and 98% yields, (Table 1, entries 7 and 8; Scheme 3).

The succinimides (**1a**, **1b**, **2a**, **2b**, **3**, **4**, and **6**) were characterized by ^{31}P, ^{13}C, and ^{1}H NMR, as well as mass spectral methods. Adducts **1a** and **5** are known from the literature [24,12,13], but for compound **1a** no characterization has been provided. For this, compound **1a** was also characterized fully by us. As a consequence of the stereogenic C_3 center, diastereotopy of the P-substituents could be observed in the ^{13}C and ^{1}H NMR spectra of compounds **1a**, **1b**, **2a**, and **2b** and in the^{13}C NMR spectrum of product **6**.

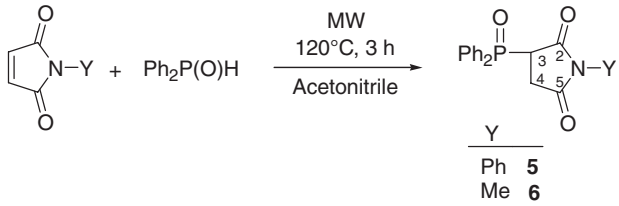
Comparative thermal experiments were also carried out (Table 2). Regarding the addition of diethyl



SCHEME 1



SCHEME 2



SCHEME 3

TABLE 1 Phospha-Michael Reactions with Maleic Derivatives under MW Conditions

Starting Materials	*T (°C)*	*t (h)*	*Yield (%)*	*Entry*
NPMI + $(EtO)_2P(O)H$	175	2.5	83 (**1a**)	1
NPMI + $(MeO)_2P(O)H$	175	2.5	81 (**1b**)	2
NMMI + $(EtO)_2P(O)H$	175	3	95 (**2a**)	3
NMMI + $(MeO)_2P(O)H$	175	3	71 (**2b**)	4
NPMI + (EtO)PhP(O)H	175	2.5	92 (**3**)[a]	5
NMMI + (EtO)PhP(O)H	175	3	80 (**4**)[a]	6
NPMI + $Ph_2P(O)H$	120	3	97 (**5**)	7
NMMI + $Ph_2P(O)H$	120	3	98 (**6**)	8
MAA + $(EtO)_2P(O)H$	120	3	49 (**7**)	9
MAA + $Ph_2P(O)H$	120	3	66 (**8**)	10

[a]As a ca 1:1 mixture of two isomers.

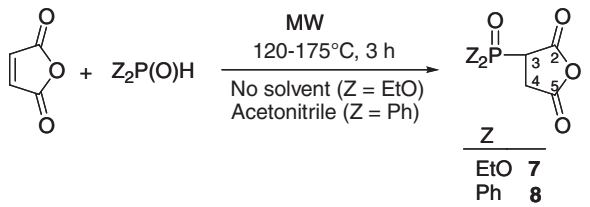
TABLE 2 Phospha-Michael Reactions with Maleimide Derivatives under Thermal Conditions

Starting Materials	*Solvent*	*T (°C)*	*t (h)*	*Conversion (%)*	*Yield (%)*	*Entry*
NPMI + $(EtO)_2P(O)H$	PhMe	110	24	0		1
NPMI + $(EtO)_2P(O)H$	*o*-xylene	144	12	32 (**1a**)		2
NPMI + $(EtO)_2P(O)H$	–	175	3.5	84 (**1a**)[a]	72	3
NMMI + $(EtO)_2P(O)H$	–	175	4	69 (**2a**)[a]		4
NPMI + $Ph_2P(O)H$	PhMe	110	8	100 (**5**)	93 (73 [12])	5

[a]No improvement on further heating.

phosphite to NPMI, there was no reaction at all in boiling toluene after a 24-h reaction time (Table 2, entry 1). Carrying out the reaction in *o*-xylene at the boiling point (144°C) for 12 h, the conversion was 32% (Table 2, entry 2). Applying a reaction temperature of 175°C for 3.5 h in the absence of any solvent, the conversion was 84% that did not increase on additional heating. In this case, product **1a** was isolated in 72% (Table 2, entry 3). The similar reaction of NMMI with diethyl phosphite led to a conversion of 69% after 4 h (Table 2, entry 4). Finally, the addition of diphenylphosphine oxide to NPMI had to be carried out in toluene as the solvent and it was found that at 110°C the reaction took place in a quantitative conversion after 8 h (Table 2, entry 5). It can be seen that the thermal phospha-Michael additions were slower than the MW variations; moreover, except in one case, the final conversions were not quantitative.

Phospha-Michael additions to maleic acid anhydride (MAA) were also investigated under MW irradiation. The addition of diethyl phosphite and diphenylphosphine oxide to MAA could be best accomplished at 120°C and 175°C, respectively, to furnish the corresponding adducts (**7** and **8**) after a reaction time of 3 h (Scheme 4). In these cases, the yield of products **7** and **8** was 49% and 66%, respectively (Table 1, entries 9 and 10). The adducts (**7** and **8**) were characterized by ^{31}P, ^{13}C, and ^{1}H NMR, as well as the mass spectral data. Duplication of the ethoxy and a part of the phenyl carbon atom signals in the ^{13}C NMR spectra of compounds **7** and **8**, respectively, referred to the phenomenon of diastereotopy.



SCHEME 4

In summary, a series of >P(O)-substituted maleic derivatives was synthesized by the MW-assisted and, in most cases, solvent-free phospha-Michael addition of dialkyl phosphites, ethyl phenyl-*H*-phosphinate, or diphenylphosphine oxide to *N*-substituted maleimides or maleic anhydride, in most instances with competent (on average 81%) yields and in a short (2.5–3 h) reaction time. The thermal phospha-Michael additions were slower and, in almost all cases, the final conversions were not quantitative. The advantage of the MW-assisted method developed is that there is no need for a solvent and catalyst. At the same time, a temperature of 120–175°C has to be applied. A literature method describes analogous reactions at room temperature in the presence of TBD as the catalyst in a toluene solution in quite a short reaction time [13]. The advantage of this method is that the reactions may be carried out at room temperature; the disadvantage is that there is a need for a solvent and a catalyst.

EXPERIMENTAL

General

The ^{13}C and ^{1}H NMR spectra were obtained in a $CDCl_3$ solution on a Bruker DRX-500 spectrometer operating at 125.7 and 500.1 MHz, respectively. The ^{13}C and ^{1}H chemical shifts are referred to as TMS. ^{31}P NMR spectra were obtained on a Bruker AV-300 spectrometer. Chemical shifts are downfield relative to 85% H_3PO_4. Mass spectrometry was performed on a ZAB-2SEQ instrument. The reactions were carried out in a 300-W CEM Discover focused microwave reactor equipped with a pressure controller applying 30–50 W under isothermal conditions.

General Procedure for the Preparation of 2-(Dialkoxyphosphonyl)succinimide Derivatives **1** *and* **2**

A mixture of 2.0 mmol of maleimide (0.35 g of *N*-phenylmaleimide or 0.22 g of *N*-methylmaleimide) and 2.0 mmol of the >P(O)H species (0.28 mL of

diethyl phosphite or 0.18 mL of dimethyl phosphite) was heated at 175°C in a vial in the MW reactor for 2.5 h (NPMI) and for 3 h (NMMI). The water formed was removed in vacuo. Column chromatography (silica gel 3% methanol in dichloromethane) of the residue afforded the products (**1a**, **1b**, **2a**, and **2b**) as oils.

The following products were thus prepared:

*2-(Diethoxyphosphonyl)-N-phenylsuccinimide (**1a**) [24].* Yield: 0.52 g (83%); ^{31}P NMR ($CDCl_3$) δ: 20.0; ^{13}C NMR ($CDCl_3$) δ: 16.45 (J = 5.8, CH_3), 16.50 (J = 5.9, CH_3), 30.9 (J = 4.0, C_4), 39.7 (J = 141.4, C_3), 63.4 (J = 6.7, OCH_2), 63.9 (J = 6.6, OCH_2), 126.5 ($C_{2'}$)*, 128.9 ($C_{4'}$), 129.3 ($C_{3'}$)*, 131.8 ($C_{1'}$), 171.4 (J = 5.7, C=O), 174.2 (J = 5.7, C=O), *may be reversed; ^{1}H NMR ($CDCl_3$) δ: 1.36 (t, J = 7.3, 3H, CH_3), 1.41 (t, J = 7.3, 3H, CH_3), 3.12–3.22 (m, 2H, C(4)H_2), 3.41–3.53 (m, 1H, C(3)H), 4.20–4.34 (m, 4H, OCH_2), 7.27–7.51 (m, 5H, Ar); $[M + H]^+_{found}$ = 312.1006, $C_{14}H_{19}NO_5P$ requires 312.1001.

*2-(Dimethoxyphosphonyl)-N-phenylsuccinimide (**1b**).* Yield: 0.46 g (81%); ^{31}P NMR ($CDCl_3$) δ: 22.7; ^{13}C NMR ($CDCl_3$) δ: 30.8 (J = 3.9, C_4), 39.4 (J = 143.0, C_3), 53.6 (J = 6.8, OCH_3), 54.5 (J = 6.6, OCH_3), 126.6 ($C_{2'}$)*, 129.1 ($C_{4'}$), 129.4 ($C_{3'}$)*, 131.7 ($C_{1'}$), 171.3 (J = 5.4, C=O), 174.0 (J = 6.2, C=O), *may be reversed; ^{1}H NMR ($CDCl_3$) δ: 3.10–3.27 (m, 2H, C(4)H_2), 3.43–3.56 (m, 1H, C(3)H), 3.87 (J = 10.0, 3H, OCH_3), 3.91 (J = 10.8, 3H, OCH_3), 7.27–7.51 (m, 5H, Ar); $[M + H]^+_{found}$ = 284.0681, $C_{12}H_{15}NO_5P$ requires 284.0688.

*2-(Diethoxyphosphonyl)-N-methylsuccinimide (**2a**).* Yield: 0.47 g (95%); ^{31}P NMR ($CDCl_3$) δ: 20.1; ^{13}C NMR ($CDCl_3$) δ: 16.58 (J = 5.9, CH_2CH_3), 16.62 (J = 5.8, CH_2CH_3), 25.6 (NCH_3), 30.9 (J = 3.8, C_4), 39.7 (J = 143.2, C_3), 63.3 (J = 6.7, OCH_2), 63.7 (J = 6.6, OCH_2), 172.4 (J = 5.1, C=O, 175.2 (J = 6.2, C=O); ^{1}H NMR ($CDCl_3$) δ: 1.36 (t, J = 7.0, 3H, CH_2CH_3), 1.38 (t, J = 7.0, 3H, CH_2CH_3), 2.97–3.05 (m, 2H, C(4)H_2), 3.03 (s, 3H, NCH_3), 3.25–3.38 (m, 1H, C(3)H), 4.17–4.28 (m, 4H, OCH_2); $[M + H]^+_{found}$ = 250.0843, $C_9H_{17}NO_5P$ requires 250.0844.

*2-(Dimethoxyphosphonyl)-N-methylsuccinimide (**2b**).* Yield: 0.31 g (71%); ^{31}P NMR ($CDCl_3$) δ: 20.7; ^{13}C NMR ($CDCl_3$) δ: 25.4 (NCH_3), 30.4 (J = 3.8, C_4), 38.8 (J = 144.7, C_3), 53.4 (J = 6.8, OCH_3), 54.2 (J = 6.6, OCH_3), 171.9 (J = 5.1, C=O), 174.8 (J = 6.7, C=O); ^{1}H NMR ($CDCl_3$) δ: 2.94–2.98 (m, 2H, C(4)H_2), 3.01 (s, 3H, NCH_3), 3.29–3.42 (m, 1H, C(3)H), 3.84 (J = 11.0, 3H, OCH_3), 3.87 (J = 10.9, 3H, OCH_3); $[M + H]^+_{found}$ = 222.0531, $C_7H_{13}NO_5P$ requires 222.0531.

3-(Ethyl phenylphosphinyl)succinimide Derivatives **3** *and* **4**

These compounds were prepared similarly using the same amount of maleimide with 2.0 mmol (0.30 mL) of ethyl phenylphosphonate. The products were obtained as oils.

*3-(Ethylphenylphosphinyl)-N-phenylsuccinimide (**3**).* Yield: 0.63 g (92%); ^{31}P NMR ($CDCl_3$) δ: 35.1 (51%) and 35.5 (49%); for the two isomers: ^{13}C NMR ($CDCl_3$) δ: 16.61 and 16.70 (CH_2CH_3), 30.3 (J = 1.8) and 30.5 (J = 2.8) (C_4), 42.6 (J = 92.7) and 43.3 (J = 92.5) (C_3), 62.56 (J = 5.7) and 62.64 (J = 6.2) (OCH_2), 126.5 and 126.7 ($C_{3'}$)[a], 129.0 ($C_{4'}$), 129.05 (J = 11.5) and 129.11 (J = 9.9) ($C_{3''}$)[b], 129.3 and 129.4 ($C_{2'}$)[a], 131.7 and 131.9 ($C_{1'}$), 132.7 (J = 10.2) ($C_{2''}$)[b], 133.6 (J = 2.9) and 133.7 (J = 2.8) ($C_{4''}$), 171.7 (J = 2.6) and 171.9 (J = 5.5) (C=O), 173.9 (J = 4.6) and 174.2 (J = 4.2) (C=O) [a,b]may be reversed, $C_{1'}$, $C_{2'}$, $C_{3'}$, and $C_{4'}$ refer to the corresponding carbon atoms of the *N*-phenyl ring, whereas $C_{2''}$, $C_{3''}$, and $C_{4''}$ refer to the respective carbon atoms of the *P*-phenyl ring; ^{1}H NMR ($CDCl_3$) δ: 1.36 (t, J = 7.1) (CH_2CH_3), 1.42 (t, J = 7.1) (CH_2CH_3) total intensity 3H, 2.98–3.37 (m, 2H, C(4)H_2), 3.50–3.73 (m, 1H, C(3)H), 4.00–4.34 (m, 2H, OCH_2), 6.98–7.94 (m, 10H, Ar); $[M + H]^+_{found}$ = 344.1045, $C_{18}H_{19}NO_4P$ requires 344.1052.

*3-(Ethylphenylphosphinyl)-N-methylsuccinimide (**4**).* Yield: 0.45 g (80%); ^{31}P NMR ($CDCl_3$) δ: 35.60 (51%) and 35.63 (49%); for the two isomers: ^{13}C NMR ($CDCl_3$) δ: 16.46 and 16.53 (CH_2CH_3), 25.2 and 25.4 (N-CH_3), 29.9 (J = 2.2) and 30.2 (J = 2.2) (C_4), 42.4 (J = 94.9) and 42.8 (J = 94.3) (C_3), 62.2 (J = 6.2) and 62.5 (J = 6.4) (OCH_2), 128.92 (J = 13.4) and 128.94 (J = 13.2) ($C_{3''}$)*, 132.37 (J = 10.2) and 132.41 (J = 10.0) ($C_{2''}$)*, 133.4 (J = 2.8) and 133.5 (J = 2.9) ($C_{4''}$), 172.5 (broad, d, J = 5.3) (C=O), 174.9 (J = 4.9) and 175.0 (J = 5.0) (C=O) *tentative assignment, $C_{2''}$, $C_{3''}$, and $C_{4''}$ refer to the corresponding carbon atoms of the *P*-phenyl ring; ^{1}H NMR ($CDCl_3$) δ: 1.33 (t, J = 7.1) (CH_2CH_3), 1.39 (t, J = 7.0) (CH_2CH_3) total intensity 3H, 2.72–3.15 (m, C(4)H_2), 2.85 (s, NCH_3), and 3.00 (s, NCH_3) overlapped, total intensity 5H, 3.35–3.55 (m, 1H, C(3)H), 3.99–4.30 (m, 2H, OCH_2), 7.49–7.90 (m, 5H, Ar); $[M + H]^+_{found}$ = 282.0898, $C_{13}H_{17}NO_4P$ requires 282.0895.

3-(Diphenylphosphinoyl)succinimide Derivatives **5** *and* **6**

A mixture of 2.0 mmol of maleimide (0.35 g of *N*-phenylmaleimide or 0.22 g of *N*-methylmaleimide) and 2.0 mmol (0.40 g) of diphenylphosphine oxide in 3 mL of acetonitrile was heated at 120°C in a vial in the MW reactor for 3 h. The workup was similar as mentioned above. Yield: 0.75 g (97%) of compound **5** and 0.61 g (98%) of compound **6**, both as white crystals.

*3-(Diphenylphosphinoyl)-N-phenylsuccinimide (**5**).* mp: 132–134°C, mp [12,14]: 134–136°C; ^{31}P NMR ($CDCl_3$) δ: 30.4, δ_P [25,14]: 31.2; $[M + H]^+_{found}$ = 376.0934, $C_{22}H_{19}NO_3P$ requires 376.0922.

*3-(Diphenylphosphinoyl)-N-methylsuccinimide (**6**).* mp: 164°C; ^{31}P NMR ($CDCl_3$) δ: 30.4; ^{13}C NMR ($CDCl_3$) δ: 25.3 (CH_3), 30.1 (J = 2.0, C_4), 42.6 (J = 63.1, C_3), 128.9 (J = 12.4, $C_{3'}$)[a], 129.2 (J = 12.4, $C_{3''}$)[a], 130.8 (J = 108.7, $C_{1'}$)[b], 130.9 (J= 107.3, $C_{1''}$)[b], 131.6 (J = 20.4, $C_{2'}$)[a], 131.7 (J = 20.2, $C_{2''}$)[a], 132.9 (J = 2.7, $C_{4'}$)[c], 132.9 (J = 2.7, $C_{4''}$)[c], 173.0 (J = 3.6, C=O), 174.9 (J = 4.3, C=O), [a]tentative assignment, [b,c]may be reversed; ^{1}H NMR ($CDCl_3$) δ: 2.83 (s, CH_3), 2.88–3.22 (m, 2H, C(4)H_2), 3.91–4.00 (m, 1H, C(3)H), 7.47–7.96 (m, 10H, ArH); $[M + H]^+_{found}$ = 314.0946, $C_{17}H_{17}NO_3P$ requires 314.0946.

*2-(Diethoxyphosphonyl)succinanhydride (**7**)*

A mixture of 6.0 mmol (0.60 g) of maleic anhydride and 6.0 mmol (0.78 mL) of diethyl phosphite was heated at 120°C in a vial in the MW reactor for 3 h. The water formed was removed in vacuo. Column chromatography (silica gel, 3% methanol in dichloromethane) of the residue afforded 0.69 g (49%) of adduct **7** as an oil. ^{31}P NMR ($CDCl_3$) δ: 21.8; ^{13}C NMR ($CDCl_3$) δ: 16.56 (J = 6.1, CH_3), 16.59 (J = 6.0, CH_3), 31.6 (J = 4.4, C_4), 41.4 (J = 131.1, C_3), 64.2 (J = 2.7, OCH_2), 64.3 (J = 2.9, OCH_2), 171.5 (J = 5.0, C=O), 175.4 (J = 19.2, C=O); 1H NMR ($CDCl_3$) δ: 1.33 (t, J = 7.0, 3H, CH_3), 1.34 (t, J = 7.0, 3H, CH_3), 2.76–3.12 (m, 2H, C(4)H_2), 3.45–3.58 (m, 1H, C(3)H), 4.13–4.23 (m, 4H, OCH_2); $[M + H]^+_{found}$ = 237.0533, $C_8H_{14}O_6P$ requires 237.0528.

*3-(Diphenylphosphinoyl)succinanhydride (**8**)*

A mixture of 6.0 mmol (0.60 g) of maleic anhydride and 6.0 mmol (1.2 g) of diphenylphosphine oxide in 3 mL of acetonitrile was heated at 120°C in a vial in the MW reactor for 3 h. The workup was similar as mentioned above. Yield: 1.2 g (66%) of compound **8** as a dense oil. ^{31}P NMR ($CDCl_3$) δ: 34.6; ^{13}C NMR ($CDCl_3$) δ: 31.1 (C_4), 44.4 (J = 59.8, C_3), 128.9 (J = 12.0, $C^*_{3'}$ and $C^*_{3''}$), 130.8 (J = 9.7, $C_{2'}$)*, 130.9 (J = 88.3, $C_{1'}$ and $C_{1''}$), 131.3 (J = 9.7, $C_{2''}$)*, 132.3 (J = 2.8, $C_{4'}$), 132.5 (J = 2.9, $C_{4''}$), 168.2 (J = 2.9, C=O), 171.1 (J = 14.7, C=O), *tentative assignments regarding the C_2 and C_3 carbon atoms of the phenyl rings; ^{1}H NMR ($CDCl_3$) δ: 2.78–2.90 and 3.22–3.32 (m, 2H, C(4)H_2), 3.99–4.17 (m, 1H, C(3)H), 7.45–7.90 (m, 10H, Ar); $[M + H]^+_{found}$ = 301.0610, $C_{16}H_{14}O_4P$ requires 301.0630.

ACKNOWLEDGMENTS

This work is connected to the scientific program of the "Development of quality-oriented and harmonized R+D+I strategy and functional model at BME" project. This project is supported by the New Széchenyi Plan (Project ID: TÁMOP-4.2.1/B-09/1/KMR-2010-0002).

REFERENCES

[1] Enders, D.; Saint-Dizier, A.; Lannou, M.-I.; Lenzen, A. Eur J Org Chem 2006, 29–49.
[2] Tanaka, M. J. Top Curr Chem 2004, 232, 25–54.
[3] Alonso, F.; Beletskaya, I. P.; Yus, M. Chem Rev 2004, 104, 3079–3159.
[4] Pudovik, A. N.; Konovalova, I. V. Synthesis 1979, 81–96.
[5] Enders, D.; Tedeschi, L.; Bats, J. W. Angew Chem, Int Ed 2000, 39, 4605–4607.
[6] Stockland, R. A.; Taylor, R. I.; Thompson, L. E.; Patel, P. B. Org Lett 2005, 7, 851–853.
[7] Tedeschi, L.; Enders, D. Org Lett 2001, 3, 3515–3517.
[8] Enders, D.; Tedeschi, L.; Bats, J. W. Angew Chem 2000, 112, 4774–4776.
[9] Enders, D.; Tedeschi, L.; Bats, J. W. Angew Chem, Int Ed 2000, 39, 4605–4607.
[10] Jackson, P. F.; Slusher, B. S. Curr Med Chem 2001, 8, 949–957.
[11] Harsányi, K.; Domány, Gy.; Greiner, I.; Forintos, H.; Keglevich, G. Heteroatom Chem 2005, 16, 562–565.
[12] Van Es, J. J. G. S.; Jaarsveld, K.; Gen, A. J Org Chem 1990, 55, 4063–4069.
[13] Jiang, Z.; Zhang, Y.; Ye, W.; Tan, C.-H. Tetrahedron Lett 2007, 48, 51–54.
[14] Rudolf, B.; Salmai, M.; Palusiak, M.; Zakrzewski, J. J Organomet Chem 2009, 694, 908–915.
[15] Frolova, N. G.; Savin, E. D.; Goryunov, E. I.; Lysenko, K. A.; Nelyubina, Yu. V.; Petrovskii, P. V.; Nifant'ev, E. E. Dokl Chem 2010, 430, 18–23.
[16] Trepohl, V. T.; Mori, S.; Itami, K.; Oestreich, M. Org Lett 2009, 11, 1091–1094.

[17] Keglevich, G.; Sipos, M.; Takács, D.; Greiner, I. Heteroatom Chem 2007, 18, 226–229.
[18] Keglevich, G.; Sipos, M.; Takács, D.; Ludányi, K. Heteroatom Chem 2008, 19, 288–292.
[19] Pietrusiewicz, K. M.; Flis, A.; Ujj, V.; Körtvélyesi, T.; Drahos, L.; Pongrácz, P.; Kollár, L.; Keglevich, G. Heteroatom 2011, 22, 730–736.
[20] Keglevich, G.; Sipos, M.; Imre, T.; Ludányi, K.; Szieberth, D.; Tőke, L. Tetrahedron Lett 2002, 43, 8515–8518.
[21] Keglevich, G.; Sipos, M.; Szieberth, D.; Nyulászi, L.; Imre, T.; Ludányi, K.; Tőke, L. Tetrahedron 2004, 60, 6619–6627.
[22] Sipos, M.; Körtvélyesi, T.; Ujj, V.; Ludányi, K.; Vékey, K.; Tőke, L.; Keglevich, G. Heteroatom Chem 2007, 18, 747–753.
[23] Keglevich, G.; Sipos, M.; Szieberth, D.; Petöcz, G.; Kollár, L. J Organomet Chem 2004, 689, 3158–3162.
[24] Pudovik, A. N.; Batyeva, E. S.; Grifanova, Y. N. J Gen Chem USSR 1973, 43, 1681–1683.