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Synthesis of New Organophosphorus Compounds Using the Atherton–Todd Reaction as a Versatile Tool

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ABSTRACT: *This article discusses the behavior of seven organophosphorus compounds under Atherton–Todd conditions. Therefore, the reactivity and selectivity of different (phen)oxaphosphinines, dioxaphosphinines, dioxaphosphinanes, and diphenylphosphine oxide with three nucleophiles were systematically studied. The results prove the versatility of the Atherton–Todd reaction to a broad range of organophosphorus compounds with different phosphorus environments and reactive P—H bonds. The nucleophiles studied in this article were chosen as model substrates for amines and alcohols. Because organophosphorus molecules are important and versatile compounds, for a broad field of applications, novel synthetic approaches are of interest to both academia and industry. As an example, the single-step synthesis of the bridged 1,3-phenylene bis(diphenylphosphinate) with potential flame-retardant properties was added to this study. In addition, the reaction is utilized for the synthesis of a novel organophosphorus anhydride.* © 2012 Wiley Periodicals, Inc. Heteroatom Chem 23:216–222, 2012; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.21006

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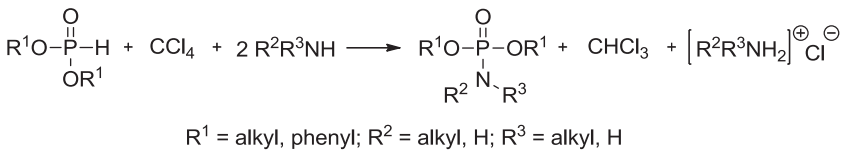
INTRODUCTION

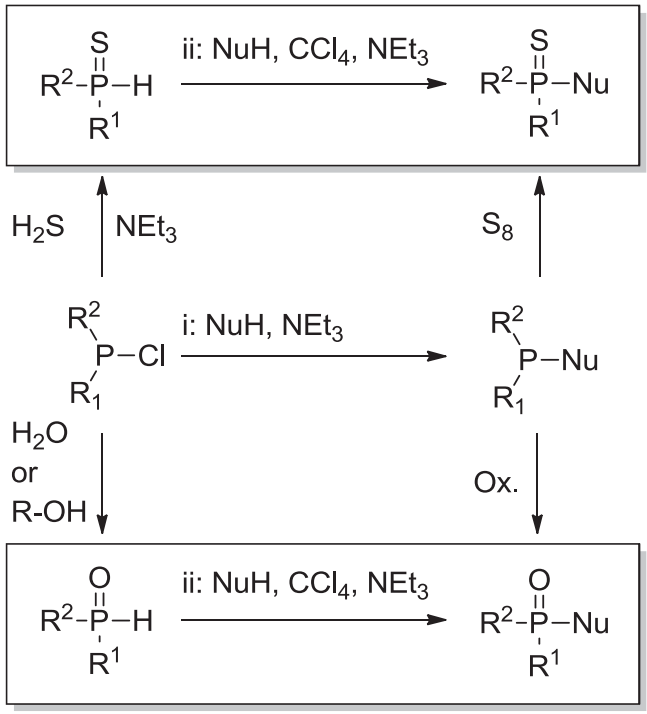
The field of organophosphorus chemistry has been an important subject of both academic and industrial research for several decades [1–3]. A wide range of applications has been reported in different areas of chemistry such as coordination chemistry, material science, homogeneous catalysis, development of biologically active compounds or pesticides, and additives for polymers such as lubricants and antioxidants [4–10]. Recently, there has also been a growing interest from the flame-retardant community regarding phosphorus-containing molecules (e.g., 6*H*-dibenzo[*c*, *e*][1,2]oxaphosphinine 6-oxide (DOPO; **3**) for epoxy resins) as environmentally friendly alternatives to the existing, and often harmful, halogenated systems [11–14]. Several new phosphorus heterocycles with flame-retardant properties have been reported to exhibit similar flame retardancy with reduced toxic effects [15,16]. In our group, we make use of the Atherton–Todd approach as a versatile synthesis tool to access novel derivatives [17,18]. The Atherton–Todd reaction is a classic reaction in organophosphorus chemistry [19–21]. Originally, Atherton and Todd reported the reaction as a very effective single-step route to yield phosphoramidates from diphenyl and dialkyl phosphonates (Scheme 1). Other phosphorus species and phosphorus heterocycles were not investigated under these conditions.



SCHEME 1 The reaction reported by Atherton and Todd.

One of the main advantages of this method is the application of phenoxaphosphinines, oxaphosphinines, dioxaphosphinines, dioxaphosphinanes, or phosphine oxides as substrates instead of the highly moisture-sensitive trivalent phosphorus chlorides. Even though phosphorus trichloride remains the main entry point into organophosphorus chemistry, many P—H reactive compounds are commercially available. In addition, the Atherton–Todd reaction is a one-pot reaction and no separate oxidation step is needed. The reaction of carbon tetrachloride, triethylamine, and an organophosphorus compound containing a P—H bond results in the in situ formation of a P(O)—Cl oxychloride species [22]. This intermediate can be observed via ^{31}P NMR spectroscopy. However, the reaction is described to be inefficient when alcohols are used as nucleophiles [19]. On the other hand, the recent literature indicates that some alcohols can react with specific phosphorus species under Atherton–Todd conditions [21]. Moreover, the highly carcinogenic carbon tetrachloride can be replaced by iodoform or bromotrichloromethane; the latter is reported to be the most efficient reagent for this reaction [23,24].

Scheme 2 demonstrates two reaction pathways to obtain the desired products: the nucleophilic substitution of phosphorus chlorides (i) followed by an oxidation step and the single-step Atherton–Todd reaction (ii). The nucleophilic substitution yields trivalent phosphorus species, which are prone to side reactions [25]. The oxidation of such compounds is often accompanied by hydrolysis or transesterification, depending upon the type of method applied [26,27]. The Atherton–Todd reaction starts from a pentavalent, often commercially available, phosphorus species and therefore no additional oxidation step is necessary. To demonstrate the scope of this versatile reaction, we systematically studied the reactivity and selectivity of six different, mostly heterocyclic compounds including: 2,8-dimethyl-10*H*-phenoxaphosphinine 10-oxide (**1**), diphenylphosphine oxide (**2**), 6*H*-dibenzo[*c*, *e*][1,2]oxaphosphinine 6-oxide (**3**), 5,5-dimethyl-1,3,2-dioxaphosphinane 2-oxide (**5**), 5,5-dimethyl-1,3,2-dioxaphosphinane 2-sulfide (**6**), and naphtho[1,8-*de*][1,3,2]dioxaphosphinine 2-oxide (**7**) under



SCHEME 2 (i) Nucleophilic substitution reaction. (ii) Atherton–Todd reaction. NuH represents a nucleophilic species (e.g., alcohols and amines).

Atherton–Todd conditions (Fig. 1). One additional heterocycle, 6*H*-dibenzo[*c*, *e*][1,2]oxaphosphinine 6-sulfide (**4**), recently published by Rakotomalala et al., was also added to this report [16]. To the best of our knowledge, the reactivity of thiophosphorus compounds (e.g., **4** and **6**) under these conditions is only scarcely discussed in the literature. Improved synthetic routes for **1** and **7** are presented in the Supporting Information.

The compounds shown in Fig. 1 represent different phosphorus environments with a decreasing amount of phosphorus–carbon bonds from compounds **1–7**. All organophosphorus molecules (P(X)—H) mentioned above were reacted

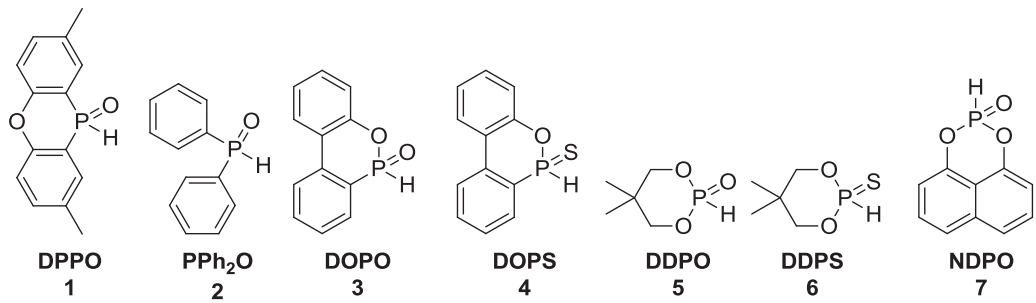


FIGURE 1 Organophosphorus compounds (and their acronyms) applied in this study.

with phenylethylamine ($PhCH_2CH_2NH_2$), piperidine ($C_5H_{10}NH$), as well as with 2-phenylethanol ($PhCH_2CH_2OH$), which can be considered as model substrates (NuH) for primary and secondary amines, and primary aliphatic alcohols.

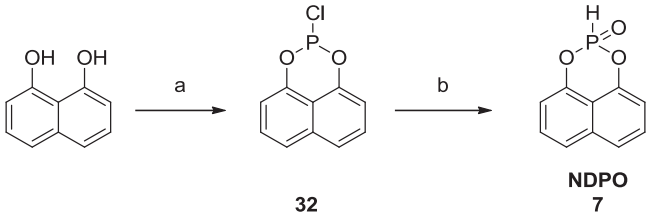
The formation of anhydrides via the Atherton–Todd reaction was also performed. The results of this study can be extrapolated to other nucleophiles, thus allowing us to access a broad range of new compounds. As a result, the one-step synthesis of a bridged compound with a rod-like geometry and potential flame-retardant properties was included in this study [28].

RESULTS AND DISCUSSION

2,8 - Dimethyl - 10*H* - phenoxaphosphinine 10 - oxide (DPPO, **1**) was already reported in the literature [17]. However, we have developed an improved synthetic route and workup procedure. The molecule is highly prone to oxidation (yielding the acid) and therefore must be handled in dry and degassed solvents. Compounds **2** and **3** are commercially available, and compounds **4–6** were already described in the literature as mentioned above [16,29,30].

In addition, we synthesized naphtho[1,8-*de*] [1,3,2] dioxaphosphinine-2-oxide (NDPO, **7**) using a novel synthetic approach developed in our laboratory. The precursor (**32**) was obtained by a reaction of 1,8-dihydroxynaphthaline in phosphorus trichloride as a solvent at 70°C; upon completion, excess PCl_3 was removed and purified for reuse. The application of additional solvents (chloroform) resulted in reduced yields. Treatment of the obtained product (**32**) with *tert*-butanol in dichloromethane generated **7** in 98% yield (Scheme 3).

Table 1 presents the results of the Atherton–Todd reaction between the P(X)—H reactive organophosphorus molecules **1** to **7** with each nucleophile (NuH: $PhCH_2CH_2NH_2$, $PhCH_2CH_2OH$, and $C_5H_{10}NH$). All reactions were performed with carbon tetrachloride as an oxidation agent.



SCHEME 3 Synthesis of NDPO (**7**). (a) PCl_3, 70°C, 20 h. (b) DCM, *t*-BuOH, 5°C, 2 h.

Phenoxaphosphinine **1**, diphenylphosphine oxide **2**, and the oxaphosphinine **3** were generally found to react easily with all three nucleophiles under Atherton–Todd conditions, yielding molecules **8–16**. This is a pleasant result because there is a strong demand for novel derivatives of **3** by the flame-retardant industry.

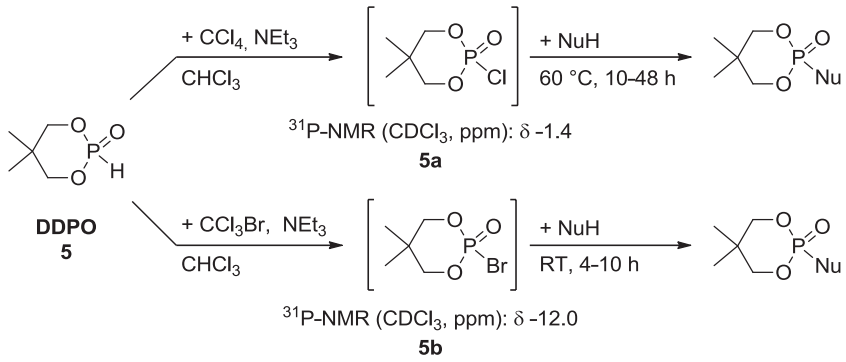
All studied phosphorus molecules **1–7** were successfully reacted with $PhCH_2CH_2NH_2$ and $C_5H_{10}NH$ (Table 1). When $PhCH_2CH_2OH$ was used the yields were lower, and in the case of the thiodioxaphosphinane **6** and dioxaphosphinine **7** the reaction was not successful. Even though **5** successfully reacted with $PhCH_2CH_2OH$, dioxaphosphinane and dioxaphosphinine structures seem to be inefficient for the reaction with alcohols.

This is supporting our assumption that the successful application of alcohols strongly depends on the phosphorus species. The formation of anhydrides was observed in all cases when traces of water were present. Hence, dry solvents and substrates are necessary.

For all substrates but **4**, **6**, and **7**, the formation of a P(X)—Cl intermediate was observed instantly via ^{31}P NMR spectroscopy when triethylamine was added to the reaction mixture (Table 1). In this context, it should be mentioned that chlorothiophosphates and their analogues are much more stable than its chlorophosphate equivalents as a result of the low polarity of the P=S bond. The preparation and purification of

TABLE 1 Products of the Atherton–Todd Reaction between Compounds **1–7** (P(X)—H) and $PhCH_2CH_2OH$, $PhCH_2CH_2NH_2$, and $C_5H_{10}NH$ (NuH). The intermediates Were Observed by ^{31}P NMR (250 MHz) Spectroscopy in $CDCl_3$ and Not Isolated

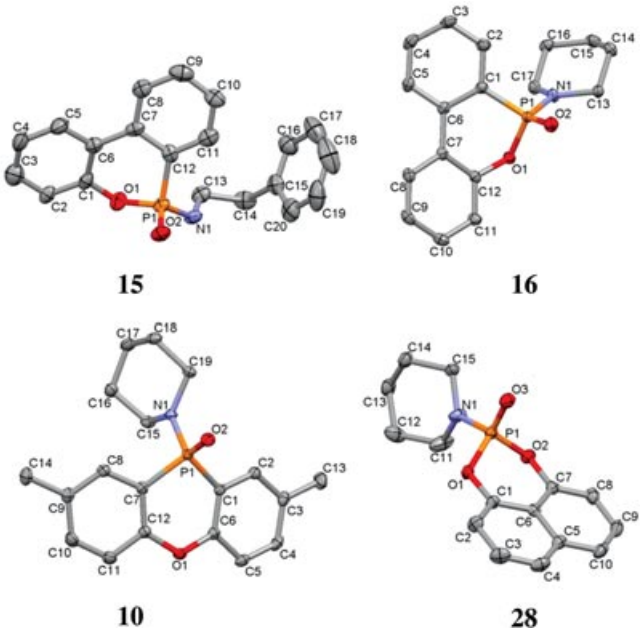
#	*Organophosphorus Compound (P(X)—H)*	*Nucleophile (NuH)*	*Intermediate (ppm)*	*Yield (%)*
8	1	$PhCH_2CH_2OH$	16.2	56
9		$PhCH_2CH_2NH_2$		96
10		$C_5H_{10}NH$		86
11	2	$PhCH_2CH_2OH$	30.1	59
12		$PhCH_2CH_2NH_2$		73
13		$C_5H_{10}NH$		51
14	3	$PhCH_2CH_2OH$	21.3	85
15		$PhCH_2CH_2NH_2$		80
16		$C_5H_{10}NH$		87
17	4	$PhCH_2CH_2OH$	–	60
18		$PhCH_2CH_2NH_2$		74
19		$C_5H_{10}NH$		87
20	5	$PhCH_2CH_2OH$	−1.4	76
21		$PhCH_2CH_2NH_2$		85
22		$C_5H_{10}NH$		74
23	6	$PhCH_2CH_2OH$	–	–
24		$PhCH_2CH_2NH_2$		79
25		$C_5H_{10}NH$		76
26	7	$PhCH_2CH_2OH$	–	–
27		$PhCH_2CH_2NH_2$		65
28		$C_5H_{10}NH$		70



SCHEME 4 Formation of the intermediates **5a** and **5b** (observed via ^{31}P NMR). CCl_3Br increases the reaction rate. NuH: $C_5H_{10}NH$, $PhCH_2CH_2NH_2$ and $PhCH_2CH_2OH$.

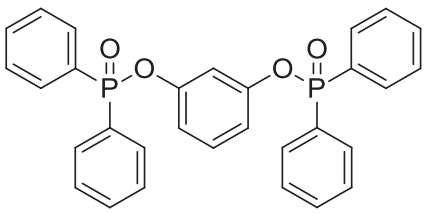
these compounds were recently illustrated by Fraix et al. [31]. For a better understanding of the mechanism, the intermediates were monitored via ^{31}P NMR spectroscopy but not isolated. In the case of compound **5**, the formed intermediate **5a** only reacted with the nucleophilic species at higher temperatures (up to 60°C) and demanded longer reaction time for all three reagents (up to 48 h, Scheme 4). Thus, the reaction of the intermediate **5a** with the nucleophile (amine or alcohol) is the rate-limiting step. The reaction speed could be increased when bromotrichloromethane was applied. The formation of the P(O)—Br species **5b** was observed in this case. The ^{31}P NMR signal of **5b** was shifted to higher fields as a result of the shielding effect of bromine. The relative bond strength, and therefore reactivity, of **5a** (P(O)—Cl bond) compared to **5b** (P(O)—Br bond) played a crucial role in reaction times and selectivity. The weaker phosphorus–bromine bond is readily replaced by a nucleophilic species. Other reactive species were reported or proposed in the literature [32].

As mentioned above, $C_5H_{10}NH$ and $PhCH_2CH_2NH_2$ were effective reagents for all organophosphorus substrates (**1–7**). Dioxaphosphinanes and dioxaphosphinines, on the other hand, seem to be less applicable for the Atherton–Todd reaction, especially with aliphatic alcohols. This is



15 16 10 28

FIGURE 2 Crystal structures of compounds **10**, **15**, **16**, and **28**.



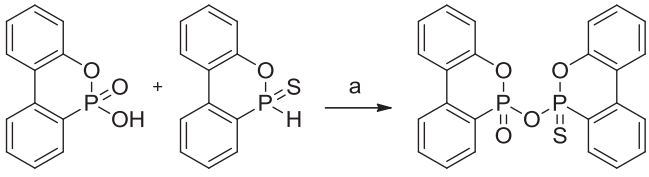
29

FIGURE 3 Organophosphorus compound **29**.

in agreement with the results reported by Atherton and Todd that the reaction is an efficient tool to access phosphoramidates. Crystal structures of selected compounds are included in Fig. 2.

To verify the versatility of the method presented in this paper, we have synthesized the bridged molecule **29** from compound **2** in 95% yield (Fig. 3). The aromatic diol resorcinol was used as a nucleophile instead of the monofunctional, aliphatic $PhCH_2CH_2OH$. Because of its bridged structure, **29** could be used as a polymer additive with a rod-like geometry and therefore only have a negligible impact on the material properties [28].

The Atherton–Todd reaction can also be exploited to access novel anhydride derivatives. The formation of anhydrides is common when traces of water cause hydrolysis of the P(X)—Cl intermediate. Commercially available **30** readily reacted with **4** to form the mixed anhydride **31** in 64% yield



DOPO-OH 30 DOPS 4 DOPS-O-DOPO 31

SCHEME 5 Synthesis of the mixed anhydride **31**. a: CCl_4, NEt_3, $CHCl_3$, 5°C, 2 h.

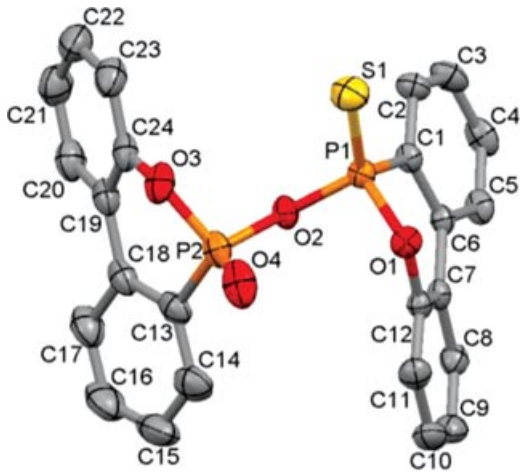


FIGURE 4 Crystal structure of compound **31**.

(Scheme 5). In this reaction, the acid functionality of **30** acts as a nucleophile.

A crystal structure of **31** was successfully obtained, proving the ability to obtain anhydrides with help of the Atherton–Todd reaction (Fig. 4). Moreover, the product was observed to be very stable against moisture, which is not common for this type of organophosphorus anhydride.

CONCLUSION

In this article, we further studied the scope of the Atherton–Todd reaction. To this end, we have systematically reacted seven organophosphorus molecules, with different phosphorus environments, with three model substrates, including two different amines and an alcohol. The results demonstrate that the Atherton–Todd reaction can be applied to almost any P—H reactive compound and nucleophile. However, the successful reaction with aliphatic alcohols strongly depends on the environment of phosphorus atom. Reaction times could be decreased by replacement of carbon tetrachloride with the less toxic bromotrichloromethane, as a result of the formation

of the more reactive phosphorus–bromine bond. The phenoxaphosphinine **1** and the oxaphosphinines **3**, **4** and diphenylphosphine oxide reacted readily with amines and alcohols in good yields. On the other hand, dioxaphosphinanes **5–6** and dioxaphosphinine **7** often required higher reaction temperatures and time and did not react with alcohols in all presented cases except compound **5**. Because oxaphosphinines such as DOPO (**3**) are industrially relevant compounds with flame-retardant properties, the Atherton–Todd reaction represents an easy, one-step synthetic tool to access novel derivatives.

EXPERIMENTAL

Materials and Instruments

Unless stated otherwise, solvents and chemicals were obtained from commercial sources and used as such without further purification. Compound **2** was supplied by BASF SE (Ludwigshafen, Germany), and compounds **3** and **30** were supplied by Schill + Seilacher (Böblingen, Germany). NMR spectra were recorded with a Bruker-Analytical BZH 250/52 (250 MHz) and a Varian INOVA-400 (400 MHz). Chemical shifts are reported as δ values relative to the solvent peak. Tetramethylsilane was used as a standard. All ^{31}P NMR spectra were measured proton decoupled. All ^{13}C NMR spectra were measured proton decoupled and phosphorus coupled. ^{1}H proton spectra were measured phosphorus coupled. Melting points are uncorrected and measured with a Büchi B-545. High-resolution mass spectrometry (HR-MS) analyses were performed on a MicroMass GCT (time of flight (TOF); electron ionization (EI), 70 eV) and Bruker micrOTOF (Nano ESI Offline). IR spectra were recorded with a Varian 660-IR (FT-IR). Elemental analysis was performed using a Vario EL III from Elementar Analysensysteme GmbH.

Crystallographic Data

X-ray diffraction measurements were performed on a Siemens SMART CCD 1000 diffractometer with monochromated MoKα-irradiation collecting a full sphere of data in the θ—ranging from 1.57° to 28.34°. The data were corrected for Lorentz and polarization effects and an empirical absorption correction with SADABS was applied [33]. The structures were solved by direct methods and refined to an optimum R_1 value with SHELX-97 [34]. Visualization for evaluation was performed with XPMA and figures were created with ORTEP [35,36].

CCDC numbers 842383–842387 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033; or deposit@ccdc.cam.ac.uk).

Syntheses

The Supporting Information can be found in the online version of this article, including a general procedure for the Atherton–Todd reaction, the synthesis of all compounds and starting materials, and spectroscopic details. The synthesis and experimental data of compound **14** is given as a general procedure for an Atherton–Todd reaction involving a P—H reactive compound.

*6 - Phenethoxy - 6H - dibenzo[c,e][1,2]oxaphosphinine 6-oxide (**14**).* A flame-dried three-neck flask fitted with a condenser, a thermometer, and an addition funnel was charged with DOPO (**3**) (5.00 g, 23.14 mmol), carbon tetrachloride (3.91 g, 25.44 mmol), and 30 mL of dry chloroform. The reaction mixture was cooled with an ice bath to 5°C. The additional funnel was charged with triethylamine (2.57 g, 25.44 mmol) and 2-phenyl ethanol (2.82 g, 23.14 mmol) dissolved in 20 mL of dry chloroform. The triethylamine and alcohol mixture were added dropwise under vigorous stirring. The reaction temperature was not allowed to exceed 10°C. After 1 h, the addition was complete, and NMR analysis indicated complete conversion of the starting material. The reaction mixture was washed three times with 50 mL water to remove the triethylamine hydrochloride. The organic phase was isolated, dried over $MgSO_4$, filtered, and the solvent was removed in vacuo. The spectroscopically pure product was obtained as a colorless oil. Yield: 6.57 g, 19.53 mmol, 85%. ^{31}P NMR (101 MHz, $CDCl_3$) δ 11.5 ppm (s, 1P); ^{13}C NMR (101 MHz, $CDCl_3$) δ 45.0 (d, J = 5.7 Hz, 2C), 135.1 (s, 2C), 127.9 (s, 2C), 124.2 (s, 2C), 119.7 (s, 2C), 113.4 ppm (d, J = 8.1 Hz, 2C); ^{1}H NMR (250 MHz, $CDCl_3$) δ 7.95–7.82 (m, 2H), 7.83 (ddd, J = 14.6 Hz, J = 7.5 Hz, J = 1.25 Hz, 1H), 7.68 (d, J = 7.5 Hz, 1H), 7.45 (td, J = 7.5 Hz, J = 3.6 Hz, 1H), 7.39–7.29 (m, 1H), 7.29–7.17 (m, 6H), 7.06–7.02 (m, 1H), 4.38–4.28 (m, 2H), 2.91 ppm (t, J = 7.2 Hz, 2H); IR (KBr) $\tilde{\nu}$: 3061 (w, C_{Aryl}—H), 3028 (w, C_{aryl}—H), 2957 (w, C_{aryl}—H), 2918 (w, C_{alkyl}—H), 2895, 1597 (s, P—C_{aryl}), 1583, 1476 (m, O—CH_2), 1431 (m, —CH_2), 1384, 1300 (s, P=O), 1155 (vs, C-O—P), 1087 (s, P—O—C), 868 (m, P—O), 798, 702, 602, 543, 488 cm^{-1}; MS (ESI) m/z [M + H]$^+$ 337, [2M + H]$^+$ 673, [2M + Na]$^+$ 695; HR-MS (EI) calcd. for [$^{12}C_{12}H_9PO_3$]$^+$ 232.0289,

found 232.0228 $[M-C_8H_8]^+$ calcd for $[^{12}C_8H_8]^+$ 104.0626, found 104.0550 $[M-C_{12}H_9PO_3]^+$; HR-MS (ESI) calcd for $[^{12}C_{20}H_{17}PO_3]^+$ 337.09153, found 337.09886 $[M + H]^+$.

SUPPORTING INFORMATION

Supporting information concerning the synthesis and analytical data (including X-ray data) for all compounds may be found in the online version of this article.

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