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An efficient synthesis of phosphonate derivatives and stable phosphorus ylids by three-component reaction between phosphines or phosphites, benzil hydrazone and dialkyl acetylenedicarboxylates

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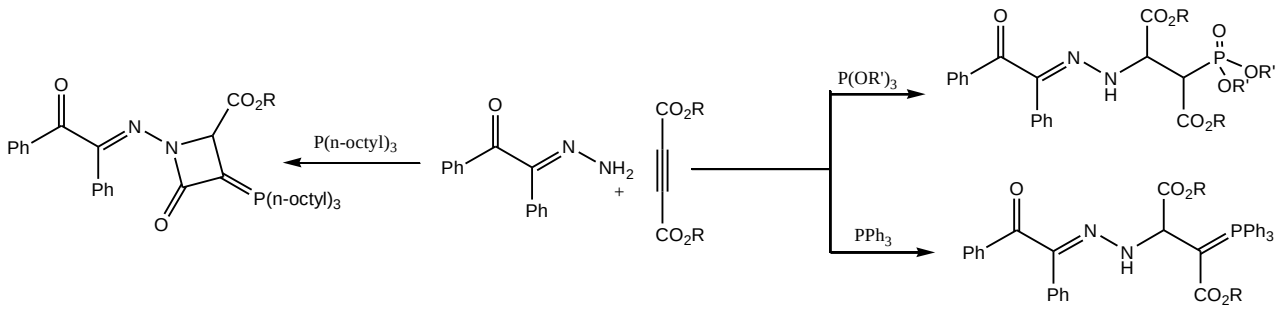
Abstract

Three-component reaction between trialkyl phosphites, dialkyl acetylenedicarboxylates and benzil hydrazone afforded phosphonate derivatives. Similar reaction between triphenylphosphine, dialkyl acetylenedicarboxylates and benzil hydrazone led to stable phosphorus ylides. When tri(n-octyl)phosphine was treated with dialkyl acetylenedicarboxylates and benzil hydrazone cyclic phosphorus ylide derivatives were obtained as the only product. All reactions were conducted in CH_2Cl_2 as solvent at room temperature without using any catalyst and the stable products were obtained in high yields.



Keywords: Phosphonate, phosphorus ylide, phosphines, phosphites, benzil hydrazone, dialkyl acetylenedicarboxylate

Introduction

The importance of organophosphorus compounds as main materials in organic synthesis and synthesis of a number of natural products has been very actively studied and proven in organic laboratories in recent years. Among these, phosphorus ylides are of special importance. They are serious reactive intermediates in synthesis of heterocyclic compounds. Phosphorus ylides, have a special electronic and molecular structure, they are sorted as special zwitterions. Also they are described by electron-rich carbanions, decisively nucleophilic. Therefore, they are used as starting reagents in organic synthesis projects.[1-6] Phosphorus ylides are generally synthesized from deprotonation of phosphonium salts which, in turn are created by the reaction of alkyl halides with phosphine nucleophiles.[7] Phosphonates are other organic compounds that contain phosphorus. Phosphonates have increasingly used as reagents in synthetic organic chemistry and as biologically and industrially important compounds.[8] Phosphonate derivatives also attracted much attention as pesticides, insecticides, antibiotics, blood pressure regulators, antiviral agents, anti-HIV agents, and herbicides.[9] Phosphonate moieties are also found applications as therapeutic drugs.[10,11] They are also increasingly used in medicine to treat disorders associated with bone formation and calcium metabolism.[12,13]

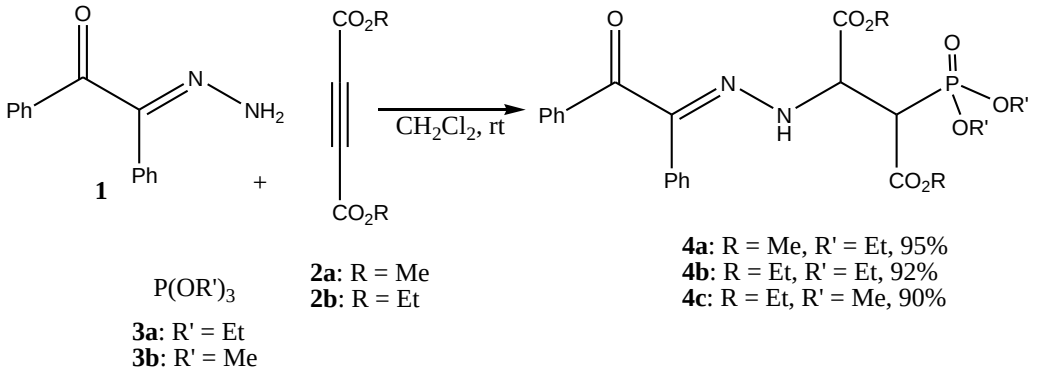
Reaction between trivalent phosphorus nucleophiles and dialkyl acetylenedicarboxylates (DAADs) in the presence of organic compounds with acidic CH, NH, SH or OH groups has been extensively used for synthesis of phosphorus compounds. When triphenylphosphine is used as the phosphorus nucleophile, the product is usually phosphorane derivatives which may be converted to carbocyclic or heterocyclic organic compounds by intramolecular Wittig reaction.[1] When trialkyl phosphites are used as the phosphorus nucleophile, the product is usually the phosphonate derivatives.[14-17] Here we wish to report the results of our study on the reaction of triphenyl or tri(n-octyl)phosphine and trialkyl phosphites with DAADs in the presence of benzil hydrazone.

Results and Discussion

At first, the reaction between trialkyl phosphites, DAADs and benzil hydrazone was studied (Scheme 1). Dimethyl acetylenedicarboxylate (DMAD, **2a**) was added to a mixture of triethyl phosphite (**3a**) and benzil hydrazone (**1**) in CH_2Cl_2 as solvent at room temperature. After 12 hours the TLC analysis of the reaction mixture showed the presence of only one product which was isolated and its structure was proved by IR and NMR spectral and elemental analysis data as phosphonate **4a**. The ^{1}H NMR spectrum of **4a** showed multiplet signals at 1.16-1.28 ppm for two CH_3 of ethoxycarbonyl groups and two singlet signals at 3.66 and 3.75 ppm for two methoxy groups. Two aliphatic CH protons were observed as a dd ($^3J_{HH}$ 5 Hz, $^2J_{PH}$ 25 Hz) signal at 3.81 ppm and a dt ($^3J_{HH}$ 5Hz, $^3J_{HH}$ 5Hz, $^3J_{PH}$ 9Hz) signal at 4.80 ppm. The aromatic and NH protons were observed as multiplet signals at 7.35-8.05 ppm. ^{1}H decoupled ^{13}C NMR spectrum of **4a** showed 20 distinct resonances in agreement with the suggested structure. Structure of **4a** was also confirmed by the IR spectrum showing absorption bands at 3249 and 1726 cm^{-1} for NH and ester carbonyl groups, respectively.

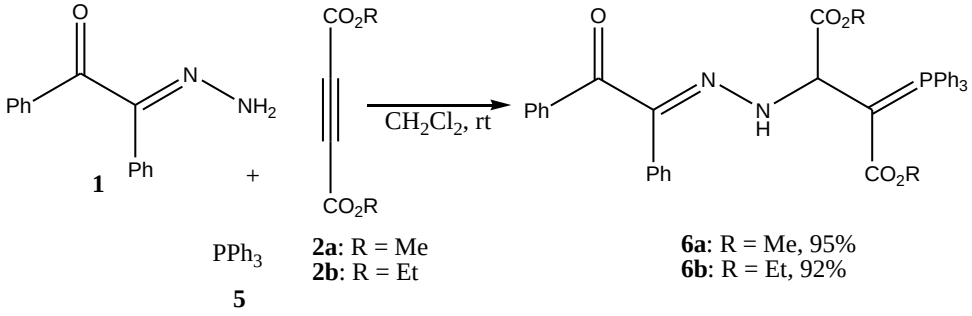
The reaction was also performed with triethyl phosphite (**3a**) or trimethyl phosphite (**3b**), diethyl acetylenedicarboxylate (DEAD, **2b**) and benzil hydrazone and similar phosphonate derivatives **4b** and **4c** were obtained in good yields.

Compounds **4a–c** possess two stereogenic centers in their structure and may exist as two diastereomers. The NMR spectra of these compounds showed that the reaction is diastereoselective and only one diastereomer was observed for these compounds.

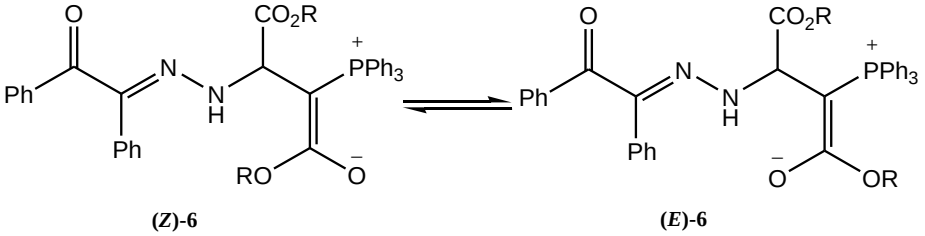


Scheme 1. Three-component reaction between trialkyl phosphites, DAADs and benzil hydrazone.

Three-component reaction between triphenylphosphine, DAADs and benzil hydrazone was also studied (Scheme 2). The reaction between triphenylphosphine, DMAD and benzil hydrazone afforded phosphorane derivative **6a** in 95% yield. Compound **6a** was easily isolated and purified by simple washing of the solid product with diethyl ether and its structure was proved by IR and NMR spectral and elemental analysis data. The NMR spectra of phosphoranes **6a** exhibited a mixture of two rotational isomers. The ylide moiety of these compounds is strongly conjugated with the adjacent carbonyl group and the rotation about the partial double bond in 6-(*E*) and 6-(*Z*) geometrical isomer pairs is slow on the NMR timescale at ambient temperature (Scheme 3). The ^{1}H NMR spectrum of **6a** showed four sharp lines (3.37, 3.67, 3.10 and 3.71 ppm) for methoxy protons along with signals for methine proton at 4.16 and 4,18 ppm, which appear as two triplets for the major and minor geometrical isomers. Similar reaction between PPh_3, DEAD and benzil hydrazone afforded phosphorane **6b** in 90% yield.

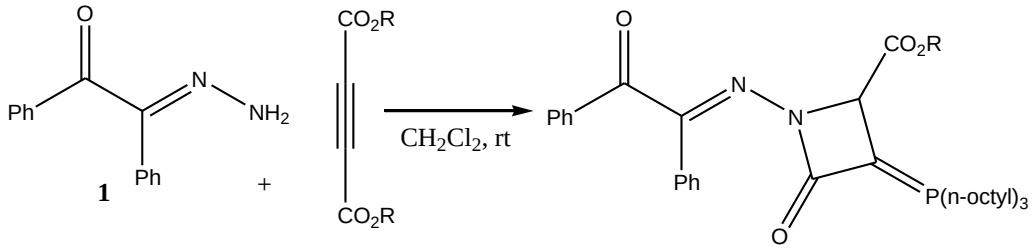


Scheme 2. Three-component reaction between triphenylphosphine, DAADs and benzil hydrazone.



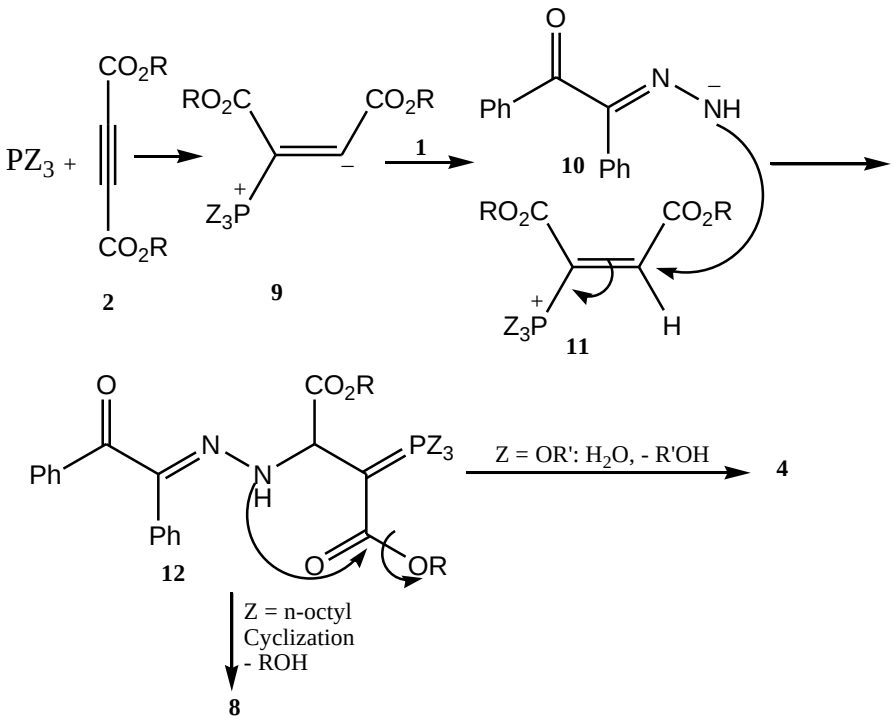
Scheme 3. Geometrical isomers of compounds **6a-b.**

When tri(n-octyl)phosphine was used as the phosphorus nucleophile in reaction with DAADs and benzil hydrazone *β*-lactam derivatives **8a** and **8b** were obtained in good yields (Scheme 4). The ^{1}H NMR spectrum of **8a** showed a triplet signal at 0.87 ppm for three CH_3 of n-octyl. The other protons of n-octyl groups resonated at 1.14-1.50 ppm and 2.30 ppm as multiplet signals. The methoxy protons resonated as a single signal at 3.80 ppm. The aliphatic CH group was observed at 7.01 ppm as a single signal. The aromatic protons were observed at 7.35-7.50 ppm. Structure **8a** was also confirmed by the IR spectrum showing absorption band at 1726 and 1604 cm^{-1} for ester and amid carbonyl groups, respectively. Conjugation with negative charge accounts for the reduction of the wavenumber of the carbonyl absorption band of amid group. The IR spectra of compounds **8a** and **8b** didn't show the absorption band for the NH group. The ^{1}H and ^{13}C NMR spectral data also confirmed the removal of one of the alkoxy groups of DMAD or DEAD for formation of compounds **8a** or **8b**.



Scheme 4. Three-component reaction between tri(n-octyl)phosphine, DAADs and benzil hydrazone.

On the basis of the well-established chemistry of trivalent phosphorus nucleophiles[1,3-5] a reliable mechanism for formation of phosphonates **4a-c** and phosphoranes **6a-b** and **8a-b** is suggested in Scheme 5. It is reasonable to assume that compounds **4**, **6** and **8** resulted from the initial addition of phosphorus nucleophile to DAAD as a Michael acceptor to generate the inert salt intermediate **9** which was protonated by benzil hydrazone. At the next step, the positively charged ion **11** was attacked by the conjugate base of benzil hydrazone (**10**) to afford phosphorus ylide **12.** In cases that trialkyl phosphites were used, the phosphite ylide **12** hydrolyzed to phosphonate derivatives **4a-c**. In cases when tri(n-octyl)phosphine was used the phosphorane **12** converted to products **8a-b** by a cyclization reaction with removal of an alcohol molecule.



Scheme 5. Suggested mechanism for formation of phosphonates **4a-c** and phosphoranes **6a-b** and **8a-b.**

Conclusions

We report herein a simple route for synthesis of some new phosphonate derivatives by three-component reaction between triethyl or trimethyl phosphite, benzil hydrazone and dialkyl acetylenedicarboxylate. Similar reaction between triphenylphosphine or tri(n-octyl)phosphine, benzil hydrazone and dialkyl acetylenedicarboxylate leads to phosphorane derivatives in good yields. The advantages of the method are readily available starting materials, simple and neutral reaction conditions, and high yields of products.

Experimental Section

General. All solvents and chemicals were purchased from commercial sources and used without further purification. The utilized benzil hydrazone was prepared by reaction of benzil with hydrazine hydrate on the basis of the reported procedure.[18] IR spectra were recorded on a Shimadzu IR-470 spectrometer. NMR spectra were recorded on a Varian model UNITY Inova 500 MHz (1H: 500 13C: 125 MHz) NMR spectrometer. Chemical shifts of ^{1}H, ^{13}C and ^{31}P NMR are reported in parts per million (ppm) from tetramethylsilane (TMS) as an internal standard in $CDCl_3$ as solvent.

General procedure for synthesis of phosphonate derivatives 4a-c. Dialkyl acetylenedicarboxylate (1 mmol) was added dropwise to the mixture of trimethyl or triethyl phosphite (1 mmol) and benzil hydrazone (1 mmol) in CH_2Cl_2 at room temperature. The reaction mixture was then stirred at room temperature for 12 hours. The solvent was removed under reduced pressure, and the residue was purified by silica-gel column chromatography using ethyl acetate-hexane (1:4) mixture as eluent.

Dimethyl 2-(ethoxyphosphono)-3-[2-(2-oxo-1,2-diphenylethylidene)hydrazinyl]succinate (4a). Yield: (95%); White powder; mp 100–103° C. IR (KBr) (ν_{max}, cm^{-1}): 3249 (NH), 1726, 1636 (C=O). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.16-1.28 (m, 6H, $2CH_3$), 3,66, 3.75 (6H, 2s, $2OCH_3$), 3.81(dd,1H, $^3J_{PH}$ 25.0, $^3J_{HH}$ 5 Hz, CH), 4.0-4.12 (m, 4H, $2CH_2$), 4.80 (dt ($^3J_{HH}$ 5Hz, $^3J_{HH}$ 5Hz, $^3J_{PH}$ 9Hz)), 7.39-7.52 (m, 9H, arom-H, NH), 8.00 (d, $^3J_{HH}$ 7 Hz). ^{13}C NMR ($CDCl_3$): δ 16.18, 16.20 ($2CH_3$), 47.14 (d, $^1J_{PC}$ 134.8Hz, C=P), 52.6, 52.8 ($2OCH_3$), 61.3 (d, $^2J_{PC}$ 4 Hz), 63.10, 63.14 ($2OCH_2$), 127.5, 128.8, 129.1, 129.2, 130.4, 130.6, 131.4, 138.2 (arom-C) , 143.3 (C=N), 167.9 (d, *J* 4Hz, C=O of ester), 170.1 (d, *J* 12Hz, C=O of ester), 191.2 (C=O of ketone). ^{31}P NMR ($CDCl_3$): δ 8.32. Anal. calcd for $C_{24}H_{29}N_2O_8P$: C 57.14, H 5.79, N 5.55. Found: C 57.22, H 5.83, N 5.50.

Diethyl 2-(ethoxyphosphono)-3-[2-(2-oxo-1,2-diphenylethylidene)hydrazinyl]succinate (4b). Yield: (92%); White powder; mp 90–92°C. IR (KBr) (ν_{max}, cm^{-1}): 3265 (NH), 1724, 1636 (C=O). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.19-1.26 (m, 12H, $4CH_3$), 3.74 (dd,1H, $^3J_{PH}$ 22, $^3J_{HH}$ 8 Hz, CH), 3.96-4.21 (m,8 H, $4CH_2$), 4.85 (ddd, $^3J_{HH}$ 8Hz, $^3J_{HH}$ 5 Hz, $^3J_{HP}$ 15Hz, CH), 7.15 (1H, d $^3J_{HH}$ 5Hz, NH), 7.95 (2H, d, $^3J_{HH}$ 8Hz, arom-H).^{13}C NMR (125 MHz, $CDCl_3$): δ 13.9, 14.0, 16.1, 16.2 (4 CH_3), 47.3 (d, $^1J_{PC}$ 135.8Hz, C=P), 60.9 (d, $^2J_{PC}$ 5 Hz), 61.0, 61.8, 62.9, 63.1 (4 OCH_2), 127.5, 128.9, 129.0, 129.2, 130.2, 130.4, 131.4, 138.0 (aromatic), 143.0 (C=N), 166.7 (d, *J* 4Hz, C=O of ester), 169.7 (d, *J* 12Hz, C=O of ester), 190.9 (C=O of keton). Anal. calcd for $C_{26}H_{33}N_2O_8P$: C 58.64, H 6.25, N 5.26. Found : C 58.77, H 6.40, N 5.16.

Diethyl 2-(methoxyphosphono)-3-[2-(2-oxo-1,2-diphenylethylidene)hydrazinyl]succinate (4c). Yield: (90%); White powder; mp 93–95°C. IR (KBr) (ν_{max}, cm^{-1}): 3336 (NH), 1726, 1640 (C=O). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.16-1.28 (m, 6H, $2CH_3$), 3.74 (dd,1H, $^3J_{PH}$ 20, $^3J_{HH}$ 5 Hz,CH), 3.72, 3.75 (6H, 2d, $^3J_{HP}$ 11Hz, $2OCH_3$), 4.10-4.25 (m, 4H, $2OCH_2$), 4.77-4.83 (1H, m, CH), 7.10 (1H, d, $^3J_{HH}$ 5Hz, NH), 7.30 -7.55 (m, 8H, aromatic), 8.02 (2H, d $^3J_{HH}$ 8Hz, aromatic) .^{13}C NMR ($CDCl_3$): δ 13.7, 14.0 ($2CH_3$), 46.8 (d, $^1J_{PC}$ 135Hz, C=P), 53.3, 53.6 (2d, $^2J_{PC}$ 8Hz, $2OCH_3$), 61.1 (d $^2J_{CP}$ 5Hz, CH), 62.1, 62.2 ($2OCH_2$), 127.5, 128.8, 129.1, 130.4, 130.5, 131.4, 138.1 (aromatic), 143.3 (C=N), 167.0 (d, JCP 4Hz, C=O of ester), 170.3 (d, J_{CP} 16Hz, C=O of ester), 191.2(C=O of keton). ^{31}P NMR ($CDCl_3$): δ 20.9. Anal. calcd for $C_{24}H_{29}N_2O_8P$: C 57.14, H 5.79, N 5.55. Found C 57.22, H 5.71, N 5.47.

General procedure for synthesis of phosphorane derivatives 6a-b and 8a-b. Dialkyl acetylenedicarboxylate (1 mmol) was added dropwise to the mixture of phosphine derivatives (1 mmol) and benzil hydrazone (1 mmol) in CH_2Cl_2 at room temperature. The reaction mixture was then stirred at room temperature for 12 hours. The solvent was removed under reduced pressure, and the solid residue was washed by diethyl ether to afford the pure product.

Dimethyl 2-(triphenylphosphoranyliden)-3-[2-(2-oxo-1,2-diphenylethylidene)hydrazinyl]succinate (6a). Yield: (95%); Orange powder; mp 156–158° C. IR (KBr) (ν_{max}, cm^{-1}): 3280 (NH), 1743, 1629 (C=O). ^{1}H NMR (500MHz, $CDCl_3$): δ 3.37(3.10) (s, 3H, OCH_3), 3.67(3.71) (s, 3H, OCH_3), 4.16(4.18) (1H, t, 3JHH=8Hz, 3JHP=8Hz, CH), 7.20-7.60 (25H, m, aromatic), 7.66(7.83) (1H, d $^3J_{HH}$ 8Hz, NH). ^{13}C NMR (125 MHz, $CDCl_3$): δ 44.6(43.7) (d, J_{CP} 126Hz, C=P), 49.0(49.7), 52.1(52.3) (2 OCH_3), 61.9(63.4) (d, J_{CP} 16Hz) (NCH), 125.5 (d, $^1J_{PC}$ =91 Hz), 128.3(128.4) (d, $^2J_{PC}$ 12 Hz), 131.8(132.1) (d, $^4J_{PC}$ 2 Hz), 133.7(133.6) (d, $^3J_{PC}$ 10 Hz), 127.6, 128.1, 128.4, 128.5, 128.6, 129.1, 129.2, 130.04, 130.3, 131.9, 133.5, 133.6 (aromatic), 140.7(C=N), 169.4(169.1) (d, J_{CP} 17Hz, CO of ester), 173.4(173.2) (d, J_{CP} 10Hz, C=O of ester), 191.4(192.1) (C=O of keton). Anal. calcd for $C_{38}H_{33}N_2O_5P$: C 72.60, H 5.29, N 4.46. Found: 72.66, H 5.23, N 4.50.

Diethyl 2-(triphenylphosphoranyliden)-3-[2-(2-oxo-1,2-diphenylethylidene)hydrazinyl]succinate (6b). Yield: (92%); Orange powder; mp 168–1170°C. IR (KBr) (ν_{max}, cm^{-1}): 3300 (NH), 1735, 1624 (C=O). ^{1}H NMR (500MHz, $CDCl_3$): δ 1.25(0.88) (3H, t $^3J_{HH}$ 7Hz, CH_3), 1.25(1.21) (3H, t $^3J_{HH}$ 7Hz, CH_3), 3.55-4.30 (m, 5H, $2OCH_2$ and CH), 7.11 -7.94 (m, 26H, aromatic and NH).^{13}C NMR(125MHZ, $CDCl_3$): δ 14.1(14.3) (CH_3), 44.1(43.1) (d, J_{CP} 124Hz, C=P), 60.9 (OCH_2) 58.0(57.6) (d, $^1J_{PC}$ 15 Hz, NCH), 61.3(61.2), 61.7(61.8) ($2OCH_2$), 125.7 (d, $^1J_{PC}$ =90 Hz), 128.4(128.2) (d, $^2J_{PC}$ 12 Hz), 131.7(132.0) (d, $^4J_{PC}$ 2 Hz), 133.7(133.5) (d, $^3J_{PC}$ 10 Hz), 127.8, 128.0, 128.5, 128.6,

128.8, 129.1, 129.3, 130.1, 130.3, 131.7, 133.5, 133.9 (aromatic), 140.5(C=N), 169.5(170.1) (d, J_{CP} 15Hz, CO of ester), 172.4(173.1) (d, J_{CP} 11Hz, C=O of ester), 191.4(192.0) (C=O of keton). ^{31}P NMR ($CDCl_3$): δ 22.4(21.5). Anal. calcd for $C_{40}H_{37}N_2O_5P$: C 73.16, H 5.68, N 4.27. Found: C 73.22, H 5.73, N 4.33.

Methyl 1-(2-oxo-1,2-diphenylethylideneamino)-4-oxoazetidine-3-(tri-n-octylphosphoranyliden)-2-carboxylate (8a). Yield: (95%); Yellow powder; mp 76–78°C. IR (KBr) (ν_{max}, cm^{-1}): 1701, 1604 (C=O). ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.88 (9H, t $3J_{HH}$ 7Hz, 3CH3), 1.05-1.50 (36H, m, 18CH2), 2.32 (6H, m, 3CH2), 3.79 (3H, s, OCH_3), 7.00 (1H, s, NCH), 7.32 -7.48 (8H, m, aromatic), 7.96 (2H, d $^3J_{HH}$ 8Hz, aromatic).^{13}C NMR(125MHz, $CDCl_3$): δ 13.9, 20.5 (d, $^1J_{PC}$ 55.7, C=P), 21.7 (d, J_{PC} 4 Hz), 22.5, 28.9(d, J_{PC} 9 Hz), 29.1 (d, $^3J_{PC}$ 8 Hz), 30.7(d, J_{PC} 15Hz), 31.7, 51.8 (OCH_3), 62.8 (NCH), 127.5, 127.7, 128.1, 128.3, 128.8, 129.5, 132.3, 136.0 (aromatic), 141.0 (C=N), 164.3 (C=O of amide), 167.1 (d, J_{CP}=22Hz, C=O of ester), 194.4(C=O of keton). Anal. calcd for $C_{43}H_{65}N_2O_5P$: C 73.26, H 9.29, N 3.97. Found: C 73.20, H 9.25, N 4.03.

Ethyl 1-(2-oxo-1,2-diphenylethylideneamino)-4-oxoazetidine-3-(tri-n-octylphosphoranyliden)-2-carboxylate (8b). Yield: (90%); Yellow powder; mp 89–91°C. IR (KBr) (ν_{max}, cm^{-1}): 1693, 1624 (C=O). ^{1}H NMR ($CDCl_3$): δ 0.88 (t, 3H, $^3J_{HH}$ 7 Hz , $3CH_3$) 1.19-1.36 (m,39 H, m, $18CH_2$ and CH_3), 2.29 (6H, m, $3CH_2$), 4.25(1H, q $3J_{HH}$ 8Hz, OCH2), 6.36 (1H, s, NCH), 7.22 -7.68 (8H, m, aromatic), 7.94 (2H, d $^3J_{HH}$ 8Hz, aromatic)..^{13}C NMR($CDCl_3$): δ 13.8, 14.3, 20.4 (d, $^1J_{PC}$ 55.7, C=P), 21.5 (d, J_{PC} 4 Hz), 22.7, 28.9 (d, J_{PC} 9 Hz), 29.2 (d, $^3J_{PC}$ 8 Hz), 30.7(d, J_{PC} 15Hz), 31.3, 60.9 (OCH_2), 62.8 (NCH), 127.6, 127.8, 128.0, 128.3, 128.7, 129.3, 132.3, 136.2 (aromatic), 141.4 (C=N), 164.1 (C=O of amide), 167.9 (d, J_{CP}=22Hz, C=O of ester), 194.4 (C=O of ketone). ^{31}P NMR ($CDCl_3$): δ 19.8. Anal. calcd for $C_{44}H_{67}N_2O_5P$: C 73.50, H 9.39, N 3.90. Found: C 73.55, H 9.46, N 3.84.

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