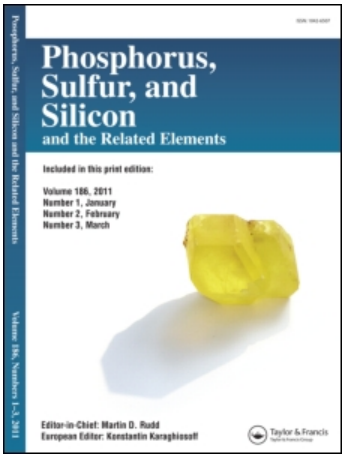

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NEW APPROACH TO THE SYNTHESIS OF HETEROCYCLIC α-AMINOPHOSPHONIC ACIDS

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NEW APPROACH TO THE SYNTHESIS OF HETEROCYCLIC α-AMINOPHOSPHONIC ACIDS

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Heterocyclic α-aminophosphonic acids were obtained by 1,3-dipolar cycloaddition reaction of azides with alkynes.

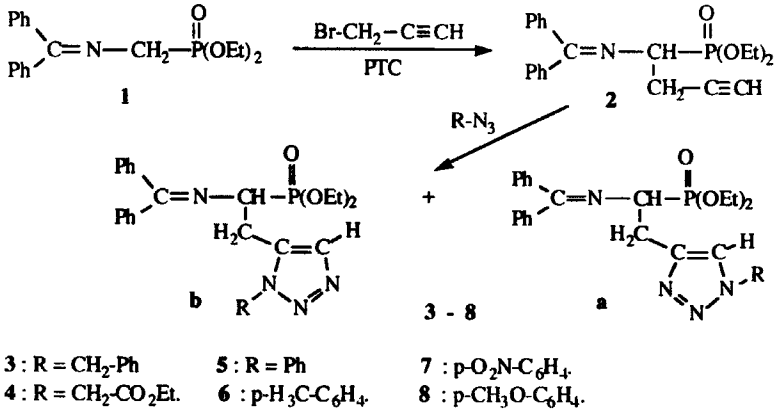
Keywords: α-aminophosphonic acids; cycloaddition; azide; alkyne

INTRODUCTION

α-aminophosphonic acids exhibit interesting biological properties[1]. Their field of action is very large: enzyme inhibitors[2], antibacterials[3], neuroexcitators[4], pesticides, herbicides[5]...

Recently, in our team we have adjusted synthetic methods of heterocyclic α-amino acids[6] and α-amino phosphonic acids[7]. We describe here a new approach to the preparation of triazolic phosphonic analogues of histidine. The key step of the strategy is the reaction of 1,3 dipolar cycloaddition of N-protected acetylenic aminoester **2** on different azides (Scheme 1).

* Correspondence Author.



3 : R = CH_2-Ph **5** : R = Ph **7** : p-O_2N-C_6H_4.
4 : R = CH_2-CO_2Et. **6** : p-H_3C-C_6H_4. **8** : p-CH_3O-C_6H_4.

SCHEME I

RESULT

The Schiff base **1** was obtained by condensation of benzophenone imine with diethyl(1-aminomethyl) phosphonate hydrochloride[8] in 72% yield. Alkylation of this Schiff base with propargylic bromide in the presence of KOH using Aliquat 336 as phase transfer catalyst[9] led to the alkylation product **2** in 75% yield.

The acetylenic Schiff base was submitted to the action of different azides (Scheme 1). The chemical yields of the cycloadditon reactions vary between 51 and 70%. The two regioisomers are separable by chromatography on silica column. The experimental conditions and the results are summarized in Table I.

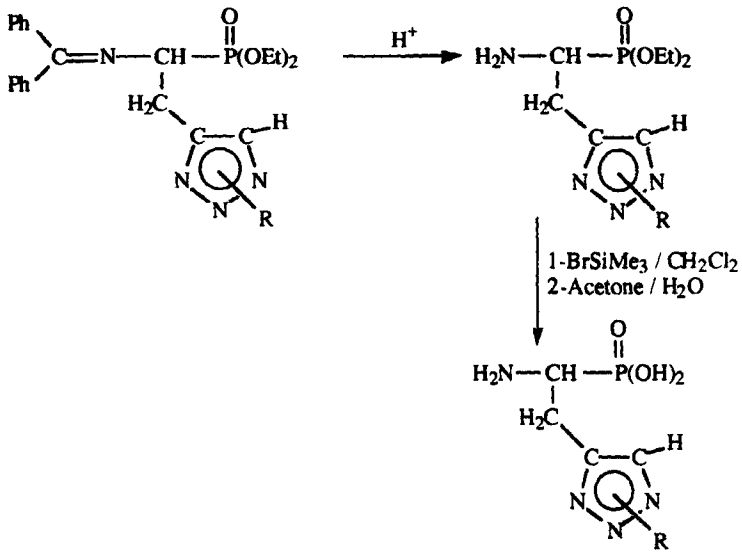
In the case where R = Ph, p-H_3C-C_6H_4, p-O_2N-C_6H_4 and p-CH_3O-C_6H_4, the cycloaddition reaction led exclusively to one regioisomer, confirmed by HPLC and by ^{31}P NMR. In the case where R = Ph-CH_2 and EtO_2C-CH_2, two regioisomers were obtained in the respective proportions of 50/50 and 60/40.

Assignment of the structures to the two regioisomers was done on the basis of the literature data[10] concerning the chemical shifts of triazolic protons (the proton signal for the 1,5 isomer lies downfield from the corresponding signal for the 1,4 isomer) as well as on our results already pub-

lished[6, 7]. The deprotection of the amine function has been realized in acid medium at room temperature to give the heterocyclic α-aminophosphonic esters in satisfactory yields (Scheme 2, Table II).

TABLE I Cycloaddition Products according to Scheme 1

Product	*R*	*Temperature (°C)*	*Time (j)*	*Yield (%)*	*Ratio of isomers*
3a	Ph-CH_2	90	8	51	50
3b					50
4a	EtO_2C-CH_2	90	10	67	60
4b					40
5a	Ph	120	4	68	> 95
6a	p-H_3C-C_6H_4	120	8	52	> 95
7a	p-O_2N-C_6H_4	120	2	64	> 95
8a	p-CH_3O-C_6H_4	120	4	62	> 95

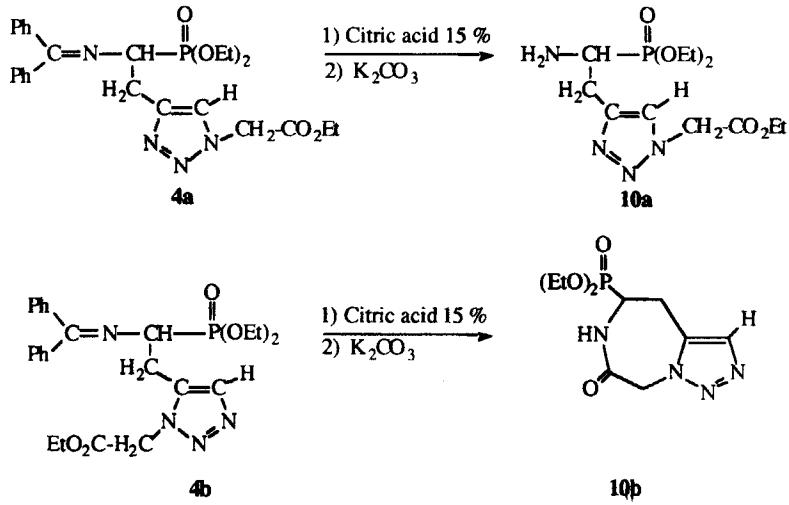


SCHEME 2

TABLE II Heterocyclic aminoesters

Product	*R*	*Time (h)*	*Yield (%)*
9a	Ph-CH 2	48	91
9b		48	90
10a	EtO_2C-CH_2	24	70
11a	Ph	24	75
12a	p-H_3C-C_6H_4	48	86
13a	p-O_2N-C_6H_4	48	72
14a	p-CH_3O-C_6H_4	36	80

In the case of isomers **4a** and **4b**, the deprotection reaction led to two different compounds (Scheme 3). So, the major isomer **4a** gave after hydrolysis the aminoester **10a,** the minor isomer **4b** led, in the same conditions, to cyclised compound **10b** in 75% yield. This cyclisation confirms the proposed structure since only the (1,5) regioisomer can be cyclised.



4a **10a**

4b **10b**

SCHEME 3

Last, the action of trimethylsilyl bromide in dichloromethane[11, 12] on the aminoesters **9a** and **9b** (Scheme 2) led to the corresponding aminoacids **15a** and **15b** in a good yield. This deprotection method of the acid function can be generalized to the all prepared aminoesters.

EXPERIMENTAL

Dichloromethane (CH_2Cl_2) was distilled from $CaCl_2$, tetrahydrofuran (THF) was dried by distillation from lithium aluminium hydride. Melting points were determined on Electrothermal melting point apparatus and are uncorrected. Flash chromatography was performed on silica gel 60 F_{254}. ^1H NMR spectra were obtained on VARIAN EM-360 (60 MHz) and BRUCKER (250 MHz) instruments, ^{31}P NMR spectra were obtained on BRUCKER (250 MHz) instruments with TMS as internal standard. Mass spectra were measured on a JEOL – JMS – DX 300 FAB.

Phenylazide was obtained according to the method described by Lindsay and Allen[13]. Arylazides were prepared according to the method described by R. Noelting and O. Michel[14]. Benzyl azide and ethyl azidoacetate were prepared from the corresponding chlorides or bromides via the reaction with sodium azide following the method reported by Henkel and Weygand[15].

Diethyl N-(diphenylmethylene) aminomethyl phosphonate 1

A mixture of diphenylmethanimine (3.75g, 19.7 mmol) and diethyl (1-aminomethyl) phosphonate hydrochloride[8] (4.01g, 19.7 mmol) in CH_2Cl_2 (100 ml) was stirred at room temperature under an inert atmosphere for 24h.

After filtration and evaporation, the residue was taken up in Et_2O (50 ml) and filtered again. The filtrate was washed with water and dried ($MgSO_4$). Evaporation of the solvent gave a solid which was recrystallized from hexane.

1: yield: 72% — m.p = 59–60°C — Rf = 0.29 (ether).
^1H NMR ($CDCl_3$) δ ppm: 1.35 (t, 6H, J = 7Hz); 4.25 (m, 6H); 7.2–7.8 (m, 10H).

Diethyl α-propargyl-N(diphenylmethylene)aminomethylphosphonate 2

A mixture of **1** (331 mg, 1 mmol), Aliquat 336 (20 mg, 0.05 mmol), propargyl bromide (1.5 mmol) and finely ground KOH (300 mg, 5 mmol) was stirred at room temperature. The reaction was monitored by TLC. The mixture was taken up in dichloromethane (10 ml) and silica gel (50 mg) was added in order to retain the catalyst. After filtration, the solvent was

removed in vacuo. The crude product was purified by flash chromatography (silica gel, eluent: ether).

2: yield = 75%. — m.p. = 87°C — Rf = 0.49 (ether).
^{1}H NMR ($CDCl_3$) δ ppm: 1.26 (t, 6H, J = 7Hz); 1.8 (t, 1H, J = 7Hz); 2.5–2.9 (m, 2H); 3.7–4.1 (m, 5H); 7–7.8 (m, 10H).

Cycloaddition reactions, general procedure

The Schiff base **2** (1 mmol) and the azide (1.5 mmol) were magnetically stirred in benzene (5 ml) at reflux for the time indicated (see Table I for reaction conditions and yields). After removing the solvent under reduced pressure, the residue was chromatographed on silica gel.

3a: Oil — Rf = 0.64 (AcOEt).
^{1}H NMR ($CDCl_3$) δ ppm: 1.31 (t, 3H, J = 7Hz); 1.37(t, 3H, J = 7Hz); 3.07–3.4 (m, 2H); 3.9–4.1 (m, 1H); 4.15–4.3 (m, 4H); 5.35 (AB, 2H, J = 15.6 Hz); 6.6–7.75 (m, 16H).
^{31}P NMR ($CDCl_3$) δ ppm: 22.53.
M.S (FAB) $[M + H]^+ = 503$; $[M + Na]^+ = 525$.

3b: m.p = 112.5°C — Rf = 0.32 (AcOEt).
^{1}H NMR ($CDCl_3$) δ ppm: 1.3 (t, 3H, J = 7Hz); 1.35 (t, 3H, J = 7Hz); 3.3–3.48 (m, 2H); 4.05–4.28 (m, 5H); 5.42 (AB, 2H, J = 14.65 Hz); 6.5–7.55 (m, 16H). ^{31}P NMR ($CDCl_3$) δ ppm: 23.76.
M.S (FAB) $[M + H]^+ = 503$.
Anal. calcd. for $C_{28}H_{31}N_4O_3P$: C, 66.93; H, 6.17; N, 11.15.
Found. C, 66.36; H, 6.39; N, 11.07;

Major isomer **4a**: Oil — Rf = 0.24 (AcOEt).
^{1}H NMR ($CDCl_3$) δ ppm: 1.2 (t, 3H, J = 7Hz); 1.3 (t, 3H, J = 7Hz); 1.4 (t, 3H, J = 7Hz); 3.4–3.55 (m, 2H); 4.1–4. 3 (m, 7H); 5.07 (AB, 2H, J = 17.45 Hz); 6.8–7.7 (m, 11H).
^{31}P NMR ($CDCl_3$) δ ppm: 23.74.
M.S (FAB) $[M + H]^+ = 499$; $[M + Na]^+ = 521$.

Minor isomer **4b**: Oil — Rf = 0.27 (AcOEt).
^{1}H NMR ($CDCl_3$) δ ppm: 1.18 (t, 3H, J = 7Hz); 1.24 (t, 3H, J = 7Hz); 1.28 (t, 3H, J = 7Hz); 3.2–3.35 (m, 2H); 3.9–4.0 (m, 1H); 4.05–4.25 (m, 6H); 4.98 (AB, 2H, J = 17.6 Hz); 6.62–7.6 (m, 11H).
^{31}P NMR ($CDCl_3$) δ ppm: 22.39.
M.S (FAB) $[M + H]^+ = 499$; $[M + Na]^+ = 521$.

Major isomer **5a**: Oil — Rf = 0.2 (Ether).
^{1}H NMR ($CDCl_3$) δ ppm: 1.29 (t, 3H, J = 7Hz); 1.33 (t, 3H, J = 7Hz); 3.1–3.5 (m, 2H); 3.8–4.2 (m, 5H); 6.7–7.7 (m, 16H).
M.S (FAB) $[M + H]^+ = 489$.

Major isomer **6a**: m.p. = 122°C — Rf = 0.63 (AcOEt).
^{1}H NMR ($CDCl_3$) δ ppm: 1.27 (t, 3H, J = 7Hz); 1.32 (t, 3H, J = 7Hz); 2.35 (s, 3H); 3.37–3.45 (m, 2H); 4–4.25 (m, 5H); 6.7–7.59 (m, 15H).
^{31}P NMR ($CDCl_3$) δ ppm: 23.79.
M.S (FAB) $[M + H]^+ = 503$.

Major isomer **7a**: m.p. = 186°C — Rf = 0.68 (AcOEt).
^{1}H NMR (C_6D_6) δ ppm: 1.04 (t, 3H, J = 7Hz); 1. 1 (t, 3H, J = 7Hz); 3.65–3.85 (m, 2H); 3.9–4.2 (m, 4H); 4.58–4.7 (m, 1H); 6.9–7.9 (m, 15H).
^{31}P NMR ($CDCl_3$) δ ppm: 23.47.
M.S (FAB) $[M + H]^+ = 534$.

Major isomer **8a**: m.p. = 134.5°C — Rf = 0.6 (AcOEt).
^{1}H NMR (C_6D_6) δ ppm: 1.03 (t, 3H, J = 7Hz); 1.13 (t, 3H, J = 7Hz); 3.15 (s, 3H); 3.7–3.85 (m, 2H); 3.95–4.25 (m, 4H); 4. 6–4.75 (m, 1H); 6.5–7.95 (m, 15H).
^{31}P NMR (C_6D_6) δ ppm: 23.87.
M.S (FAB) $[M + H]^+ = 519$.

Hydrolysis of the imines

Method A

A solution of imine (1 mmol) in Et_2O (4 ml) was stirred with 10% aq HCl (4 ml) at room temperature for 24–48h. The organic phase was separated, and the aqueous phase was extracted with Et_2O (5 × 1 ml) in order to remove benzophenone. The aqueous phase was then stirred with K_2CO_3 (0.3g) and CH_2Cl_2 (4 ml) for 10 min. The organic phase was separated, dried (Na_2SO_4) and evaporated (see Table II).

9a: Oil
^{1}H NMR ($CDCl_3$) δ ppm: 1.04 (t, 6H, J = 7Hz); 1.3 (s, 2H); 2.4–2.6 (m, 1H); 2.7–2.9 (m, 2H); 3.85 (m, 4H); 5.36 (s, 2H); 6.9–7. 1 (m, 5H); 7.45 (s, 1H). ^{31}P NMR ($CDCl_3$) δ ppm: 26.72.
M.S (FAB) $[M + H]^+ = 339$; $[2M + H]^+ = 677$.

9b: Oil
^{1}H NMR ($CDCl_3$) δ ppm: 0.94 (t, 3H, J = 7Hz); 0.96 (t, 3H, J = 7Hz); 1.7 (s, 2H); 2.45–2.6 (m, 1H); 2.7–2.95 (m, 1H); 3.0–3.15 (m, 1H); 3.7–3.9 (m, 4H); 5.2 (s, 2H); 6.9–7.1 (m, 5H); 7.25 (s, 1H).
^{31}P NMR ($CDCl_3$) δ ppm: 27.99.
M.S (FAB) $[M + H]^+ = 339$; $[2M + H]^+ = 677$.

11a: Oil
^{1}H NMR ($CDCl_3$) δ ppm: 1.25(t, 3H, J = 7Hz); 1.3(t, 3H, J = 7Hz); 2.45 (s, 2H); 2.67–3.57 (m, 3H); 4.1 (m, 4H); 7.35–7.55 (m, 4H); 7.7–7.85 (m, 2H). ^{31}P NMR ($CDCl_3$) δ ppm: 23.73.
M.S (FAB) $[M + H]^+ = 325$.

12a: Oil
^{1}H NMR ($CDCl_3$) δ ppm: 1.26 (t, 3H, J = 7Hz); 1.27 (t, 3H, J = 7Hz); 1.5 (s, 2H); 2.35 (s, 3H); 2.75–3.5 (m, 3H); 4–4.2 (m, 4H); 7.22 (d, 2H, J = 8 Hz); 7.51 (d, 2H, J = 8 Hz); 7.85 (s, 1H).
^{31}P NMR ($CDCl_3$) δ ppm: 26.63.
M.S (FAB) $[M + H]^+ = 339$; $[2M + H]^+ = 677$; $[2M + Na]^+ = 699$.

13a: Oil
^{1}H NMR (C_6D_6) δ ppm: 1.03 (t, 3H, J = 7Hz); 1.04 (t, 3H, J = 7Hz); 1.8 (s, 2H); 2.9–3.1 (m, 1H); 3.3–3.5 (m, 2H); 3.9–4.1 (m, 4H); 7.3 (d, 2H, J = 9. 1 Hz); 7.5 (s, 1H); 7.78 (d, 2H, J = 9. 1 Hz).
^{31}P NMR ($CDCl_3$) δ ppm: 27.92.
M.S (FAB) $[M + H]^+ = 370$; $[2M + H]^+ = 739$.

14a: Oil
^{1}H NMR (C_6D_6) δ ppm: 1.06 (t, 3H, J = 7Hz); 1.07 (t, 3H, J = 7Hz); 2.9–3.1 (m, 1H); 3.2(s, 3H); 3.4–3.6 (m, 2H); 3.95–4.1 (m, 4H); 6.64 (d, 2H, J = 9Hz); 7.26 (s, 1H); 7.41 (d, 2H, J = 9Hz).
^{31}P NMR (C_6D_6) δ ppm: 28.25.
M.S (FAB) $[M + H]^+ = 355$; $[M + Na]^+ = 377$; $[2M + H]^+ = 709$.

Method B: Hydrolysis of the imines 4a and 4b

The imine (1 mmol) was dissolved in THF (7 ml) and hydrolysed at 25°C with 15% aqueous citric acid (6 ml). The solvent was evaporated at room temperature and the aqueous layer extracted with Et_2O. The organic layer containing the ketol was extracted with 15% aqueous citric acid. The combined aqueous layer was basified with sodium carbonate and extracted

with diethyl ether. The organic extracts were dried ($MgSO_4$) and evaporated.

10a: Oil
^{1}H NMR ($CDCl_3$) δ ppm: 1.25(t, 3H, J = 7Hz); 1.29 (t, 3H, J = 7Hz); 1.3 (t, 3H, J = 7Hz); 2.75–2.9 (m, 1H); 3.2–3.5 (m, 2H); 4.05–4.19 (m, 4H); 4.22 (q, 2H, J = 7Hz); 5. 1(s, 2H); 7.6 (s, 1H).
^{31}P NMR ($CDCl_3$) δ ppm: 27.91.
IR υ (cm^{-1}): 3381. and 3400(NH); 1748.9(C=O); 1219.6(P=O); 1024. 1(C-O).
M.S (FAB) $[M + H]^+ = 335$; $[2M + H]^+ = 669$; $[2M + Na]^+ = 691$.

10b: Oil
^{1}H NMR ($CDCl_3$) δ ppm: 1.33 (t, 6H, J = 7Hz); 3.1–3.3 (m, 1H); 3.4–3.6 (m, 1H) 3.9–4.15 (m, 1H); 4.15–4.3 (m, 4H); 5.26 (AB, 2H, J = 16.05 Hz); 6.35 (s, 1H); 7.5 (s, 1H).
^{31}P NMR ($CDCl_3$) δ ppm: 20.1.
IR υ (cm^{-1}) = 3051(N-H); 1736 (C=O); 1693.5; 1263.5 (P=O).
M.S (FAB) $[M + H]^+ = 289$; $[2M + H]^+ = 577$; $[2M + Na]^+ = 599$.

Synthesis of 15a and 15b

A solution of **9a** or **9b** (1.9 mmol) in dichloromethane (40 ml) was carefully dried on $MgSO_4$ before stirring under argon. Trimethyl silyl bromide (45 mmol, 2.4 eq) was introduced dropwise from a syringe through the rubber cap. The mixture was stirred for 4h at room temperature, then evaporated in vacuo. The oily residue was dissolved in acetone and water (1 ml) was added, the mixture was kept under stirring 3 additionnal hours, and about half of the solvent was evaporated. The white precipitate was collected by filtration, and dried under reduced pressure.

15a: yield = 69%
^{1}H NMR (CD_3OD) δ ppm: 3.1–3.7 (m, 3H); 5.8 (s, 2H); 7.1–7.5(m, 5H); 8.06 (s, 1H).
M.S (FAB) $[M + H]^+ = 283$; $[2M + H]^+ = 565$.

15b: m.p. = 248°C yield = 71%
^{1}H NMR (DMSO-d_6) δ ppm: 2.85–3.0 (m, 1H); 3.05–3.25 (m, 1H); 3.4–3.6 (m, 1H); 5.54 (s, 2H); 7.2–7.45 (m, 5H); 7.99 (s, 1H); 8.2 (se, 2H).
^{31}P NMR (DMSO-d_6) δ ppm: 15.68.
M.S (FAB) $[M + H]^+ = 283$; $[2M + H]^+ = 565$.

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