

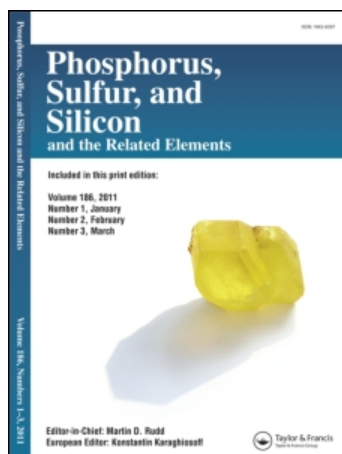
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Synthesis of α -Heterocyclic α -Aminophosphonates,¹ Part II: Morpholine, Piperidine, Pyrrolidine, Tetrahydrofurylmethylamine, *N*-Benzyl-*N*-Methylamine, and Aniline Derivatives

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Synthesis of α -Heterocyclic α -Aminophosphonates,¹ Part II: Morpholine, Piperidine, Pyrrolidine, Tetrahydrofurylmethylamine, *N*-Benzyl-*N*-Methylamine, and Aniline Derivatives

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*α , α -Diaminomethyl phosphonate derivatives were synthesized by *N*-alkylation of nitrogen heterocycles or primary amines with diethyl α -bromo- α -aminomethyl phosphonate.*

Keywords Amine; diethyl α -bromo- α -aminomethyl phosphonate; nitrogen heterocycles; *N*-alkylation

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INTRODUCTION

The study of phosphorus analog of the proteinogenic or non proteinogenic α -amino acids has accelerated in the past twenty years, due to the finding of molecules with useful biological activity as potent antibiotics, enzyme inhibitors, or pharmacological agents.² Amino phosphonic acids are amino acid's surrogates, and the phosphonic acid group is considered an isosteric group of the carboxylic function and also a stable tetrahedral intermediate in reactions including carboxylic acid derivatives.

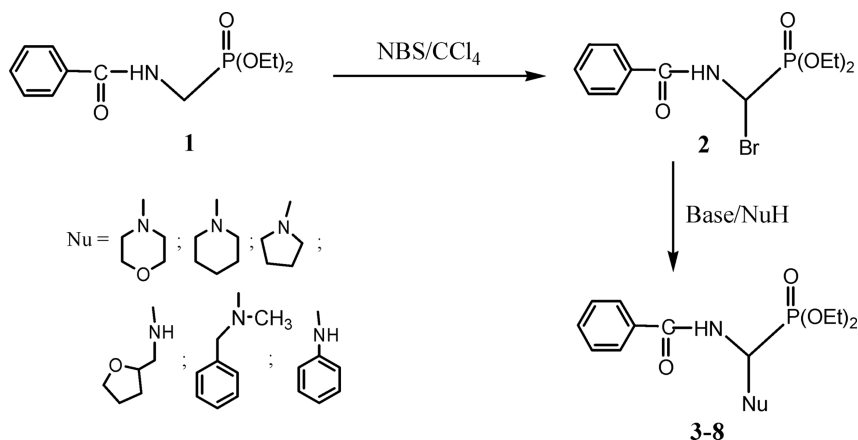
The biological activity of amino phosphonic acids depends on their configuration. However, the number of available methods for the synthesis of heterocyclic aminophosphonates is still limited.³

In a very recent article we described the preparation of α -heterocyclic- α -amino- phosphonates by *N*-alkylation of α -azido- α -aminophosphonates¹ in good to very good yields. Here we describe another part of our investigations concerning carboxylic and phosphonic amino acids⁴ with the aim to have access to new active biomolecules.

RESULTS AND DISCUSSION

Our strategy is based on the *N*-alkylation of amines with diethyl α -bromo- α -amino- methylphosphonate⁴ **2** (Scheme 1).

Compound **2** was obtained by bromination of **1** applying Steglich's procedure^{5,6} (yield = 86%). The reaction of different amines NuH with the bromine derivative **2** results in the formation of the compounds **3–8** carrying saturated heterocyclic or aromatic amines.



SCHEME 1

TABLE I Synthesis of α,α -Diaminomethyl Phosphonates 3–8

Product	Nu-H	M.P. ($^{\circ}$ C)	Reaction Time (h)	Yield (%)
3	Morpholine	112–114	2	90
4	Piperidine	103–105	8	92
5	Pyrrolidine	123–125	0.5	85
6	Tetrahydrofurylmethylamine	115–117	16	80
7	<i>N</i> -Benzyl- <i>N</i> -methylamine	105–107	16	75
8	Aniline	130–132	16	80

The literature reports numerous methods of *N*-alkylation of amines.⁷ After several attempts of reactions by phase transfer catalysis or in the presence of bases such as triethylamine or diethylamine, the reaction with diisopropylethylamine gave the best results. It was carried out in dry acetone at r.t. The results are summarized in Table I.

Products **3–8** were obtained in a 75% to 92% overall yield from **2** and were analyzed by MS and ^1H NMR spectroscopy.

Comparison of these results with those obtained in the reaction of **2** with aromatic heterocyclic compounds like triazole, pyrazole, or imidazole indicated that saturated cyclic amines are the better reagents.

The structure of compound **4** was also confirmed by single crystal X-ray diffraction as shown in Figure 1. Experimental details and crystallographic data are reported in Table II. Atomic coordinates are reported

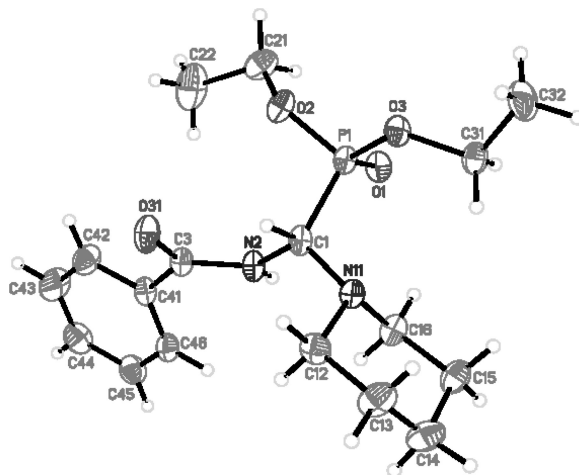
**FIGURE 1** ORTEP representation of the molecular structure of **4** in the crystal; displacement ellipsoids are drawn at the 50% probability level.

TABLE II Crystal Data and Structure Refinement for 4 at 173(2) K

Empirical formula	C ₁₇ H ₂₇ N ₂ O ₄ P	
Formula weight	354.38	
Wavelength ($\lambda_{\text{MoK}\alpha}$)	0.71073 Å	
Crystal system, (S.G.)	Monoclinic (P 2 ₁ /n)	
Unit cell dimensions (Å, °)	a = 9.8118(5) b = 16.2571(11) c = 12.2775(6)	$\alpha = 90$ $\beta = 106.384(4)$ $\gamma = 90$
Volume (Å ³), Z	1878.88(18), 4	
Absorption coefficient (mm ⁻¹)	0.169	
Theta range for (°)	3.68 to 25.64	
Index ranges	h:[-11, 11], k:[-19, 19], l:[-14, 14]	
Reflections [I > 2sigma(I)]; Final R indices	34190; R1/wR2: 0.0488/0.1216	

in Table III, while Table IV reports the most significant bond distances and angles. The heterocyclic ring adopts chair conformation.

In contrast to the amines in Table I, which give the alkylation products in very good yields, other amines like 2-aminomethyl pyridine, 2-imidazolidone, and *N*-ethylbenzylamine induced a cleavage of the C α –N bond, and benzamide was the major product. It was identified by ¹H NMR spectroscopy and MS FAB ([M+H]⁺ = 122).

In conclusion, the method described in this article provides a general and convenient access to a wide range of α,α -diaminomethyl phosphonate derivatives starting from the appropriate bromide **2**. The *N*-alkylation of different amines with compound **2** occurs under very mild conditions and leads to amino phosphonates **3–8** in good yields.

EXPERIMENTAL

Melting points were determined with an Electrothermal melting point apparatus and are uncorrected. ¹H NMR spectra were recorded on a Bruker AC-250 MHz spectrometer in CDCl₃ with TMS as an internal standard; coupling constants are reported in Hz. Mass spectra were recorded on a JEOL-JMS- DX 300 mass spectrometer (FAB in glycerol) or on a Polaris Thermoelectron mass spectrometer (CI by desorption in methane). Compound **2** was prepared using Steglich's procedure.⁵

The X-ray diffraction data for a selected crystal were collected with a STOE IPDS II two-circle diffractometer using monochromatic MoK α radiation ($\lambda = 0.71073$ Å). Data were corrected for Lorentz and polarization effect, and for adsorption effect.⁸ The structure was solved by

TABLE III Atomic Coordinates and Equivalent Isotropic Displacement Parameters ($\times 10^3$) for 4

	x	y	z	$U_{eq} (\text{\AA}^2)$
P(1)	0.5912(1)	0.6154(1)	0.3916(1)	20(1)
O(1)	0.6412(1)	0.5450(1)	0.4687(1)	27(1)
O(2)	0.5705(2)	0.5979(1)	0.2617(1)	28(1)
C(21)	0.6064(2)	0.5206(1)	0.2168(2)	35(1)
C(22)	0.4722(3)	0.4808(2)	0.1460(3)	55(1)
O(3)	0.6940(1)	0.6922(1)	0.4120(1)	25(1)
C(31)	0.7644(2)	0.7222(1)	0.5272(2)	27(1)
C(32)	0.8907(3)	0.7728(2)	0.5206(2)	42(1)
C(1)	0.4149(2)	0.6558(1)	0.3865(2)	21(1)
N(11)	0.4163(2)	0.7084(1)	0.4831(1)	22(1)
C(12)	0.2946(2)	0.7655(1)	0.4580(2)	30(1)
C(13)	0.3151(3)	0.8291(1)	0.5532(2)	38(1)
C(14)	0.3329(3)	0.7879(1)	0.6674(2)	39(1)
C(15)	0.4529(2)	0.7243(1)	0.6888(2)	32(1)
C(16)	0.4287(2)	0.6638(1)	0.5897(2)	27(1)
N(2)	0.3113(2)	0.5876(1)	0.3656(1)	23(1)
C(3)	0.2112(2)	0.5791(1)	0.2638(2)	22(1)
O(31)	0.2034(2)	0.6247(1)	0.1821(1)	32(1)
C(41)	0.1074(2)	0.5090(1)	0.2556(2)	23(1)
C(42)	0.0596(2)	0.4684(1)	0.1517(2)	35(1)
C(43)	-0.0380(3)	0.4035(2)	0.1389(2)	44(1)
C(44)	-0.0894(2)	0.3812(1)	0.2287(2)	37(1)
C(45)	-0.0442(2)	0.4224(1)	0.3313(2)	34(1)
C(46)	0.0550(2)	0.4863(1)	0.3453(2)	29(1)

the Direct Methods procedure of SIR97,⁹ and refined by the full matrix least square technique of SHELX-L 97.¹⁰ All the hydrogen atoms were localized from difference Fourier maps. Data of crystal structures and refinements, such as a full list of bond lengths and angles and anisotropic thermal parameters, have been deposited with the Cambridge Crystallographic Database. These data can be obtained free of charge on quoting the depository no. CCDC 621832 using the link <http://www.ccdc.cam.ac.uk> or from CCDC, 12 Union Road Cambridge CB2 1EZ, UK; fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk.

Typical Procedure for *N*-alkylation

To a stirred solution of 2.86 mmol of the corresponding amine and 3.12 mmol of diisopropylethylamine in 10 mL of dry acetone, 2.6 mmole of diethyl 1-bromo-1-(benzoyl- amino)methyl phosphonate were added. The mixture was stirred at r.t. and the reaction was followed by TLC

TABLE IV Selected Bond Distances (Å) and Angles (°) for **4**

P(1)—O(1)	1.4780(13)	P(1)—O(1)	1.4780(13)
P(1)—O(2)	1.5768(14)	P(1)—O(2)	1.5768(14)
P(1)—O(3)	1.5800(13)	P(1)—O(3)	1.5800(13)
P(1)—C(1)	1.8340(19)	P(1)—C(1)	1.8340(19)
O(3)—C(31)	1.471(2)	O(3)—C(31)	1.471(2)
C(1)—N(11)	1.460(2)	C(1)—N(11)	1.460(2)
C(1)—N(2)	1.477(2)	C(1)—N(2)	1.477(2)
N(11)—C(16)	1.470(2)	N(11)—C(16)	1.470(2)
N(11)—C(12)	1.475(2)	N(11)—C(12)	1.475(2)
C(12)—C(13)	1.530(3)	C(12)—C(13)	1.530(3)
C(13)—C(14)	1.520(3)	C(13)—C(14)	1.520(3)
C(14)—C(15)	1.533(3)	C(14)—C(15)	1.533(3)
C(15)—C(16)	1.529(3)	C(15)—C(16)	1.529(3)
N(2)—C(3)	1.361(2)	N(2)—C(3)	1.361(2)
C(3)—O(31)	1.232(2)	C(3)—O(31)	1.232(2)
C(3)—C(41)	1.514(2)	C(3)—C(41)	1.514(2)
C(41)—C(42)	1.394(3)	C(41)—C(42)	1.394(3)
C(42)—C(43)	1.403(3)	C(42)—C(43)	1.403(3)
C(43)—C(44)	1.384(4)	C(43)—C(44)	1.384(4)
C(44)—C(45)	1.384(3)	C(44)—C(45)	1.384(3)
C(45)—C(46)	1.401(3)	C(45)—C(46)	1.401(3)

(Kiesegel Merck 60F524). The solvent was evaporated under reduced pressure. The residue was quenched with saturated aqueous solution of ammonium chloride (20 mL) and extracted with dichloromethane (3 × 20 mL). The organic phase was dried with sodium sulfate (Na₂SO₄), and the solvent was removed under reduced pressure. The product was purified, wherever necessary, by column chromatography on silica gel using ether/hexane or ether/methanol as an eluent to afford pure N-alkylated phosphonates.

3: Rf: 0.6 (ether). ¹H NMR (CDCl₃): 1.19 (t, *J* = 7.0 Hz, 3H, CH₃); 1.38 (t, *J* = 7.0 Hz, 3H, CH₃); 2.64 (m, 2H, NCH₂ morpholine); 2.94 (m, 2H, NCH₂ morpholine); 3.64 (m, 4H, OCH₂ morpholine); 4.05 (m, 2H, OCH₂); 4.22 (m, 2H, OCH₂); 5.35 (dd, ²*J*_{PH} = 20.3 Hz, ³*J*_{HH} = 9.8 Hz, 1H, P—CH<); 6.95 (m, 1H, NH); 7.40 (m, 2H, arom-H); 7.50 (m, 1H, arom-H); 7.78 (m, 2H, arom-H). MS (FAB⁺): 356 [M]⁺ C₁₆H₂₅PN₂O₅.

4: Rf: 0.7 (ether). ¹H NMR (CDCl₃): 1.23 (t, *J* = 7.0 Hz, 3H, CH₃); 1.39 (m, 2H, CH₂ piperidine); 1.40 (t, *J* = 7.0 Hz, 3H, CH₃); 1.59 (m, 4H, CH₂ piperidine); 2.60 (m, 2H, NCH₂ piperidine); 2.95 (m, 2H, NCH₂ piperidine); 4.07 (m, 2H, OCH₂); 4.30 (m, 2H, OCH₂); 5.40 (dd, ²*J*_{PH} = 20.4 Hz, ³*J*_{HH} = 9.9 Hz, 1H, P—CH<); 7.08 (m, 1H, NH); 7.48 (m, 2H, arom-H); 7.55 (m, 1H, arom-H); 7.85 (m, 2H, arom-H). MS (FAB⁺): 354 [M]⁺ C₁₇H₂₇PN₂O₄.

5: Rf: 0.48 (ether). ^1H NMR (CDCl_3): 1.29 (t, $J = 7.0$ Hz, 3H, CH_3); 1.40 (t, $J = 7.0$ Hz, 3H, CH_3); 1.75 (m, 4H, CH_2); 2.80 (m, 2H, NCH_2); 2.90 (m, 2H, NCH_2); 4.10 (m, 2H, OCH_2); 4.20 (m, 2H, OCH_2); 5.65 (dd, $^2J_{\text{PH}} = 19.2$ Hz, $^3J_{\text{HH}} = 9.9$ Hz, 1H, $\text{P}-\text{CH}<$); 7.08 (m, 1H, NH); 7.48 (m, 2H, arom-H); 7.55 (m, 1H, arom-H); 7.85 (m, 2H, arom-H). MS (FAB^+): 340 $[\text{M}]^+$ $\text{C}_{16}\text{H}_{25}\text{PN}_2\text{O}_4$.

6: Rf: 0.43 (ether). ^1H NMR (CDCl_3): 1.27 (t, $J = 7.0$ Hz, 3H, CH_3); 1.32 (t, $J = 7.0$ Hz, 3H, CH_3); 1.40–1.60 (m, 2H, CH_2); 1.70–1.90 (m, 2H, CH_2); 2.70–2.95 (m, 2H, NCH_2); 3.65–3.80 (m, 3H, NH and OCH_2); 3.90–4.00 (m, 1H, OCH); 4.10 (m, 2H, OCH_2); 4.20 (m, 2H, OCH_2); 5.45 (dd, $^2J_{\text{PH}} = 20.4$ Hz, $^3J_{\text{HH}} = 9.8$ Hz, 1H, $\text{P}-\text{CH}<$); 7.20 (m, 1H, NH); 7.35–7.60 (m, 3H, arom-H); 7.85 (m, 2H, arom-H). MS DCI (methane): 371 $[\text{M} + \text{H}]^+$ $\text{C}_{17}\text{H}_{28}\text{PN}_2\text{O}_5$.

7: Rf: 0.7 (ether). ^1H NMR (CDCl_3): 1.25 (t, $J = 7.0$ Hz, 3H, CH_3); 1.40 (t, $J = 7.0$ Hz, 3H, CH_3); 2.50 (s, 3H, CH_3); 3.80 (d, $J = 13.4$ Hz, 1H, $\text{CH}_a\text{H}_b\text{-Ph}$); 3.95 (d, $J = 13.4$ Hz, 1H, $\text{CH}_a\text{H}_b\text{-Ph}$); 4.10 (m, 2H, OCH_2); 4.25 (m, 2H, OCH_2); 5.55 (dd, $^2J_{\text{PH}} = 20.4$ Hz, $^3J_{\text{HH}} = 9.8$ Hz, 1H, $\text{P}-\text{CH}<$); 7.08 (m, 1H, NH); 7.25–7.60 (m, 8H, arom-H); 7.85 (m, 2H, arom-H). MS (FAB^+): 390 $[\text{M}]^+$ $\text{C}_{20}\text{H}_{27}\text{PN}_2\text{O}_4$.

8: Rf: 0.53 (ether/MeOH 5%). ^1H NMR (CDCl_3): 1.27 (t, $J = 7.0$ Hz, 3H, CH_3); 1.32 (t, $J = 7.0$ Hz, 3H, CH_3); 4.10 (m, 2H, OCH_2); 4.20 (m, 2H, OCH_2); 6.10 (dd, $^2J_{\text{PH}} = 20.4$ Hz, $^3J_{\text{HH}} = 9.8$ Hz, 1H, $\text{P}-\text{CH}<$); 7.08 (m, 1H, NH); 7.25–7.60 (m, 9H, NH and arom-H); 7.85 (m, 2H, arom-H). MS DCI (methane): 363 $[\text{M} + \text{H}]^+$ $\text{C}_{18}\text{H}_{24}\text{PN}_2\text{O}_4$.

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