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## Interaction of Vinylphosphonates with 1,2-Diaminoethane and Ethanolamine

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*The reaction of 1,2-diaminoethane with vinylphosphonate occurs as the addition of the amino group to the  $\beta$ -carbon atom of the unsaturated substrate to give 1:1 and 1:2 adducts. The nucleophilic addition of 2-aminoethanol at the  $\beta$ -position of the double bond of vinylphosphonates involves only the amino group and leads to the formation of hydroxy  $\beta$ -aminoethylphosphonates or zwitterions depending on conditions.*

**Keywords** Addition; 2-aminoethanol,  $\beta$ -aminoethylphosphonates; betains; 1,2-diaminoethane; vinylphosphonate

## INTRODUCTION

Nitrogen-containing phosphonates are known to be potentially useful as biologically active compounds, extragents, and membrane carriers.<sup>1–6</sup> Addition of primary and secondary amines to phosphoryl alkenes is one of the most accessible synthetic routes to  $\beta$ -aminoalkylphosphonates.<sup>7–10</sup> However, only a limited number of articles are devoted to addition reactions of nitrogen containing bi-functional nucleophilic reagents to unsaturated derivatives of tetracoordinated phosphorus.<sup>4,11–13</sup>

## RESULTS AND DISCUSSION

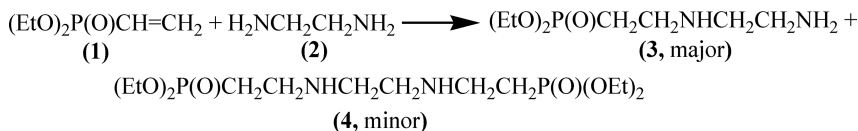
Here we present the results of our investigations on the interaction of vinylphosphonates with 1,2-diaminoethane and 2-aminoethanol.

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Dedicated to Professor Marian Mikołajczyk, CBMiM PAN in Łódź, Poland, on the occasion of his 70th birthday.

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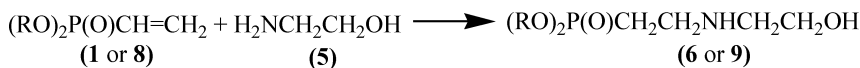
The heating of an equimolar mixture of diethylvinylphosphonate (**1**) with 1,2-diaminoethane (**2**) leads to the formation of adduct **3**. In the IR spectrum of the compound **3**, the absorption band for the double bond ( $1640\text{ cm}^{-1}$ ) is missing. It shows bands characteristic for  $\text{--NH}$  and  $\text{--NH}_2$  groups, as well as a strong  $\text{P=O}$  band ( $1238\text{ cm}^{-1}$ ). The  $^{31}\text{P}$  NMR spectrum of adduct **3** displays a singlet at  $\delta = 31.3$  ppm. In the  $^1\text{H}$  NMR spectrum of adduct **3**, the doublet of triplets at  $\delta = 1.85$  ppm ( $^2J_{\text{PH}} = 18.3\text{ Hz}$ ,  $^3J_{\text{HH}} = 7.2\text{ Hz}$ ) for the methylene group at the phosphorus atom indicates that the aminoorganyl group attached to the  $\beta$ -carbon atom of phosphorylethane fragment. In the EI mass spectrum of compound **3**, an intensive molecular ion peak,  $\text{C}_8\text{H}_{21}\text{N}_2\text{O}_3\text{P}$  ( $[\text{M}^+]$  found,  $m/z$  224.1293;  $[\text{M}^+]$  calculated 224.1290), is observed. Based on the data obtained from IR,  $^1\text{H}$ , and  $^{31}\text{P}$  NMR and mass spectra, adduct **3** can be identified as 1-diethoxyphosphoryl-2-( $\beta$ -aminoethyl)aminoethane (Scheme 1).



### SCHEME 1

In the mass spectra of the reaction mixture, a peak of low intensity corresponding to  $\text{C}_{14}\text{H}_{34}\text{N}_2\text{O}_6\text{P}_2$  ( $[\text{M}^+]$  found,  $m/z$  388.1890;  $[\text{M}^+]$  calculated,  $m/z$  388.1892) is observed, indicating the formation also of the 2:1 addition product 1,2-bis[(diethoxyphosphoryl)-ethyl]diaminoethane (**4**) (Scheme 1). We have reported previously<sup>13</sup> that interaction of 1,2-diaminoethane with 3-methylbuta-1,2-dienylphosphonate leads to the formation of a symmetric adduct consisting of two phosphorylalkane groups connected by a diaminoethane bridge.

Contrary to the diaminoethane (**2**), 2-aminoethanol (**5**) contains two distinct nucleophilic centers, i.e., addition product formation is theoretically possible with both of them. We expected only the formation of the addition product with nitrogen atom bonded to the  $\beta$ -carbon atom of double bond of the vinylphosphonate, analogous to the reaction of phosphoryl containing  $\pi$ -systems with mercaptoethanol.<sup>14</sup> Distillation of the reaction mixture containing the phosphonate **1** and the amine **5** yielded an additional product, the structure of which was found to be 1-diethoxyphosphoryl-2-( $\beta$ -hydroxyethyl)aminoethane (**6**), as indicated by the  $^1\text{H}$  NMR and  $^{31}\text{P}$  NMR spectra (Scheme 2). The IR spectrum of compound **6** displays a broad strong absorption band (at  $3120\text{--}3400\text{ cm}^{-1}$ ) characteristic for the stretching vibrations of  $\text{--OH}$  and  $\text{--NH}$  groups. Absorption bands for  $\text{P=O}$  ( $1240\text{ cm}^{-1}$ ) and  $\text{P--O--C}_2\text{H}_5$

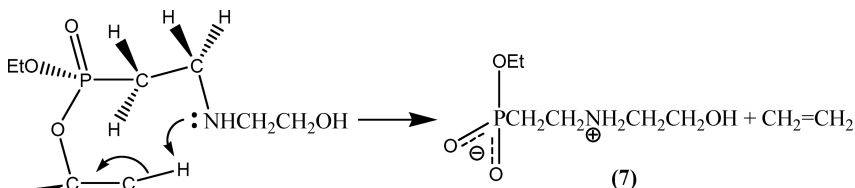


R = Et (1 or 6); Bu (8 or 9)

## SCHEME 2

(1060  $\text{cm}^{-1}$ ) are retained in the IR spectrum of adduct **6**. It should be noted that distillation of compound **6** leads to partial decomposition, resulting in additional signals in the  $^1\text{H}$  NMR spectrum, presumably belonging to the products of the dealkylation of adduct **6**.

Reaction of vinylphosphonate **1** with ethanolamine was carried out at harsh conditions (T 140–160°C) without solvent, resulting in the formation of a white powder with melting temperature of 130°C. The product is insoluble in organic solvents, is soluble in water, and most probably has a zwitterionic structure **7**, as indicated by the  $^1\text{H}$  and  $^{31}\text{P}$  NMR spectroscopic data and elemental analysis (Scheme 3):

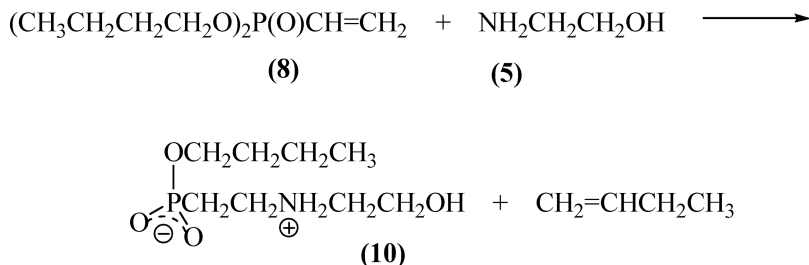


## SCHEME 3

A similar dealkylation of tetracoordinated phosphoric acid esters with alkene elimination was reported previously.<sup>15</sup>

Interaction of the dibutyl ester of vinylphosphonic acid **8** with amine **5** leads to 1-dibutoxyphosphoryl-2-( $\beta$ -hydroxyethyl)amino-ethane (**9**) as the only addition product. The structure **9** is supported by the presence of a doublet of triplets at 1.95 ppm for the protons of methylene group bonded to the phosphorus atom. In this case, the addition would affect the OH group of the nucleophile **5**. A doublet of triplets at 3.6 ppm, corresponding to the protons at  $\beta$ -carbon atom of the adduct, would be expected in the  $^1\text{H}$  NMR spectrum. However in the spectrum of compound **9**, only a triplet at 3.6 ppm, corresponding to the methylene protons of the  $\text{CH}_2\text{OH}$  group, is observed, confirming the absence of spin–spin interaction with the phosphorus nucleus. Therefore, addition of amine **5** to the double bond of vinylphosphonates **1** and **8** proceeds chemoselectively and affects only the amino group of the bifunctional nucleophile.

A white powder, insoluble in organic solvents, was obtained from the reaction of vinylphosphonate **8** with ethanolamine at harsh conditions (150°C). Based on  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{31}\text{P}$  NMR, as well as on elemental analysis, the product most probably has the structure **10** (Scheme 4). Formation of zwitterion **10** occurs in analogy to the formation of compound **7**.



#### SCHEME 4

In summary, the reaction of 1,2-diaminoethane with vinylphosphonates proceeds with the attack of the amino group to the  $\beta$ -carbon atom of the unsaturated substrate to give 1:1 (major) and 1:2 (minor) adducts. The reaction of vinylphosphonates with ethanolamine leads either to  $\beta$ -aminoethylphosphonates containing a hydroxy group (**6** and **9**) or to zwitterions (**7** or **10**), depending on the conditions. The presence of a hydroxy group allows the synthesis of salts with good water solubility, which is important for biologically active compounds.

#### EXPERIMENTAL

The IR spectra were recorded with a UR-20 spectrometer. The  $^1\text{H}$  and  $^{31}\text{P}$  NMR spectra were obtained with a Varian Unity 300 spectrometer at 300 MHz and 121.42 MHz, respectively.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were measured with a Bruker instrument at 400 MHz and 100.56 MHz, respectively. The  $^1\text{H}$  NMR chemical shifts are given with respect to the residual signal of  $\text{CHCl}_3$  and  $\text{DMSO}-d_5$  as internal references; the  $^{13}\text{C}$  NMR chemical shifts referred to the signal of the solvent  $\text{DMSO}-d_6$ . The  $^{31}\text{P}$  NMR chemical shifts are given relative to 85%  $\text{H}_3\text{PO}_4$  as the external standart. The mass spectra were obtained with a MAT 212 mass spectrometer (ionizing voltage 60 eV, emission current 0.1 mA, direct inlet of the sample into the ion source, gradual increase in the temperature of the evaporator). The exact masses for the ions were determined by peak matching at 10,000 resolution. All operations were carried out under argon.

## Addition of Amines 2 and 5 to Vinylphosphonates 1 and 8: General Procedure

### Method A

The amine **2** or **5** (1 mmol) was added to the corresponding vinylphosphonate **1** or **8** (1 mmol). The reaction mixture was heated at 85–95°C for 2 h and was subsequently distilled under vacuum to yield compounds **3**, **6**, or **9**.

**1-Diethoxyphosphoryl-2( $\beta$ -aminoethyl)amino-ethane (3).** Yield 1.4 g (63%), bp 105–107°C (0.03 mm Hg),  $n_D^{20}$  1.4574. IR ( $\nu$ , cm<sup>-1</sup>): 1023 (P-O-C), 1238 (P=O), 3180–3380 (NH, NH<sub>2</sub>). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 1.20 (t, <sup>3</sup> $J_{HH}$  = 7.1 Hz, 6H, CH<sub>3</sub>CH<sub>2</sub>OP), 1.3 (br s, 2H, -HNCH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>), 1.85 (dt, <sup>2</sup> $J_{PH}$  = 18.3 Hz, <sup>3</sup> $J_{HH}$  = 7.2 Hz, 2H, P(O)CH<sub>2</sub>CH<sub>2</sub>N), 2.54 (m, 2H, -HNCH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>), 2.66 (m, 2H, -HNCH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>), 2.78 (dt, <sup>3</sup> $J_{PH}$  = 15.5 Hz, <sup>3</sup> $J_{HH}$  = 7.2 Hz, 2H, P(O)CH<sub>2</sub>CH<sub>2</sub>N), 3.99 (m, 4H, CH<sub>3</sub>CH<sub>2</sub>OP). <sup>31</sup>P NMR (DMSO-*d*<sub>6</sub>):  $\delta$  = 31.3. Mass spectrum of the reaction mixture:  $m/z$  224.1293, C<sub>8</sub>H<sub>21</sub>N<sub>2</sub>O<sub>3</sub>P, [M<sup>+</sup>]; 388.1890, C<sub>14</sub>H<sub>34</sub>N<sub>2</sub>O<sub>6</sub>P<sub>2</sub>, [M<sup>+</sup>].

**1-Diethoxyphosphoryl-2( $\beta$ -hydroxyethyl)amino-ethane (6).** Yield 0.96 g (43%), bp 127–144°C (0.7 mm Hg),  $n_D^{20}$  1.4482. IR ( $\nu$ , cm<sup>-1</sup>): 1060 (P-O-C), 1240 (P=O), 3120–3400 (NH, OH). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 1.33 (t, <sup>3</sup> $J_H$  7.1 Hz, 6H, CH<sub>3</sub>CH<sub>2</sub>OP), 1.97 (dt, <sup>2</sup> $J_{PH}$  = 18.4 Hz, <sup>3</sup> $J_{HH}$  = 7.1 Hz, 2H, P(O)CH<sub>2</sub>CH<sub>2</sub>); 2.7 – 2.9 (m, 4H, P(O)CH<sub>2</sub>CH<sub>2</sub>NH- and P(O)CH<sub>2</sub>CH<sub>2</sub>NHCH<sub>2</sub>-); 3.53 (t, <sup>3</sup> $J_{HH}$  = 5.3 Hz, 2H, -NHCH<sub>2</sub>CH<sub>2</sub>OH); 4.10 (m, 4H, CH<sub>3</sub>CH<sub>2</sub>OP). <sup>31</sup>P NMR (DMSO-*d*<sub>6</sub>):  $\delta$  = 31.0. Mass spectra of adduct (**6**):  $m/z$  225.0663, C<sub>8</sub>H<sub>20</sub>NO<sub>4</sub>P, [M<sup>+</sup>].

**1-Dibutoxyphosphoryl-2( $\beta$ -hydroxyethyl)amino-ethane (9).** Yield 1.6 g (55%), bp 147–148°C (0.03 mm Hg),  $n_D^{20}$  1.4598. IR ( $\nu$ , cm<sup>-1</sup>): 1060 (P-O-C), 1240 (P=O), 3200–3450 (NH, OH). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 0.91 (t, <sup>3</sup> $J_{HH}$  = 7.3 Hz, 6H, CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OP), 1.37 (m, 4H, CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OP), 1.62 (m, <sup>3</sup> $J_{HH}$  = 6.7 Hz, 4H, CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OP), 1.95 (dt, <sup>2</sup> $J_{PH}$  = 18.1 Hz, <sup>3</sup> $J_{HH}$  = 7.2 Hz, 2H, P(O)CH<sub>2</sub>CH<sub>2</sub>NH-), 2.74 (t, <sup>3</sup> $J_{HH}$  = 5.2 Hz, 2H, -NHCH<sub>2</sub>CH<sub>2</sub>OH), 2.90 (dt, <sup>2</sup> $J_{PH}$  = 15.0 Hz, <sup>3</sup> $J_{HH}$  = 7.2 Hz, 2H, P(O)CH<sub>2</sub>CH<sub>2</sub>NH-), 3.61 (t, <sup>3</sup> $J_{HH}$  = 5.2 Hz, 2H, -NHCH<sub>2</sub>CH<sub>2</sub>OH); 4.00 (m, 4H, CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OP). <sup>31</sup>P NMR (DMSO-*d*<sub>6</sub>):  $\delta$  = 31.2. Found: C 51.03, H 9.81, P 11.44; Calcd. for C<sub>12</sub>H<sub>28</sub>NO<sub>4</sub>P: C 51.25, H 9.96, P 11.03%.

## Method B

The reaction mixture of vinylphosphonate (**1** or **8**, 1 mmol) with ethanolamine (**5**) was heated at 140–160°C for 6 h. After the addition of benzene to the reaction mixture, the zwitterions (**7** or **10**) were obtained, each as a white powder.

**Zwitterion (7).** Yield 0.63 g (32%), mp 130°C. IR ( $\nu$ ,  $\text{cm}^{-1}$ ): 1060 (P-O-C), 1200 ( $\text{PO}_2^-$ ), 2878 ( $\text{H}_2\text{N}^+$ ), 3200–3250 (OH).  $^1\text{H}$  NMR (300 MHz,  $\text{D}_2\text{O}$  +  $\text{DMSO}-d_6$ ):  $\delta$  = 1.23 (t,  $^3J_{\text{HH}}$  = 7.2 Hz, 3H,  $\text{CH}_3\text{CH}_2\text{OP}$ ), 1.98 (dt,  $^2J_{\text{PH}}$  = 18.4 Hz,  $^3J_{\text{HH}}$  = 8.1 Hz, 2H,  $\text{P}(\text{O}_2^-)\text{-CH}_2\text{CH}_2\text{N}$ ), 3.16 (m, 2H,  $-\text{N}^+\text{H}_2\text{CH}_2\text{CH}_2\text{OH}$ ), 3.23 (m, 2H,  $\text{P}(\text{O})\text{CH}_2\text{CH}_2\text{N}$ ), 3.81 (t,  $^3J_{\text{HH}}$  = 5.1 Hz, 2H,  $\text{N}^+\text{H}_2\text{CH}_2\text{CH}_2\text{OH}$ ), 3.93 (m, 2H,  $\text{CH}_3\text{CH}_2\text{OP}$ ).  $^{31}\text{P}$  NMR ( $\text{DMSO}-d_6$ ):  $\delta$  = 21.2. Found: C 36.12, H 7.93, P 15.91; Calcd. for  $\text{C}_6\text{H}_{16}\text{NO}_4\text{P}$ :  $\tilde{\text{N}}$  36.55, H 8.12, P 15.74%.

**Zwitterion (10).** Yield 0.75 g (33.4%), mp 153°C. IR ( $\nu$ ,  $\text{cm}^{-1}$ ): 1048 (P-O-C), 1197 ( $\text{PO}_2^-$ ), 2858 ( $\text{H}_2\text{N}^+$ ), 3200–3300 (OH).  $^1\text{H}$  NMR (400 MHz,  $\text{D}_2\text{O}$  +  $\text{DMSO}-d_6$ ):  $\delta$  = 0.78 (t,  $^3J_{\text{HH}}$  = 7.2 Hz, 3H,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OP}$ ), 1.24 (tq,  $^3J_{\text{HH}}$  =  $^3J_{\text{HH}}$  = 7.4 Hz, 2H,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OP}$ ), 1.47 [tt,  $^3J_{\text{HH}}$  = 7.4 Hz,  $^3J_{\text{HH}}$  = 6.7 Hz, 2H,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OP}$ ), 1.89 (m,  $^2J_{\text{PH}}$  = 18.4 Hz,  $^3J_{\text{HH}}$  = 16.3 Hz, 2H,  $\text{P}(\text{O}_2^-)\text{CH}_2\text{CH}_2\text{N}$ ), 3.07 (m,  $^3J_{\text{HH}}$  = 10.3 Hz, 2H,  $-\text{N}^+\text{H}_2\text{CH}_2\text{CH}_2\text{OH}$ ), 3.13 (m,  $^3J_{\text{HH}}$  = 16.3 Hz,  $^3J_{\text{PH}}$  = 9.6 Hz, 2H,  $\text{P}(\text{O}_2^-)\text{CH}_2\text{CH}_2\text{N}$ ), 3.71 (m,  $^3J_{\text{HH}}$  = 10.3 Hz, 2H,  $-\text{N}^+\text{H}_2\text{CH}_2\text{CH}_2\text{OH}$ ), 3.75 (dt,  $^3J_{\text{HH}}$  = 6.7 Hz,  $^3J_{\text{PH}}$  = 6.7 Hz, 2H,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OP}$ ).  $^{13}\text{C}$  NMR (100.56 MHz,  $\text{D}_2\text{O}$  +  $\text{DMSO}-d_6$ ):  $\delta$  = 12.3 (s,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OP}$ ), 18.0 (s,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OP}$ ), 23.0 (d,  $^1J_{\text{PC}}$  = 132.8 Hz,  $\text{PCH}_2\text{CH}_2\text{N}$ ), 31.8 (d,  $^2J_{\text{PC}}$  = 6.0 Hz,  $\text{PCH}_2\text{CH}_2\text{N}$ ); 42.7 (s,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OP}$ ), 48.5 (s,  $-\text{N}^+\text{H}_2\text{CH}_2\text{CH}_2\text{OH}$ ), 56.2 (s,  $-\text{N}^+\text{H}_2\text{CH}_2\text{CH}_2\text{OH}$ ), 64.3 (d,  $^2J_{\text{PC}}$  = 5.9 Hz,  $\text{POCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$ ).  $^{31}\text{P}$  NMR ( $\text{DMSO}-d_6$ ):  $\delta$  = 21.7. Found:  $\tilde{\text{N}}$  43.12, H 9.44, N 5.82. Calcd. for  $\text{C}_8\text{H}_{20}\text{NO}_4\text{P}$ : C 42.66, H 8.88, N 6.22%.

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