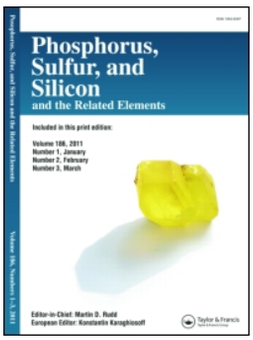

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Synthesis of polyphosphorylated diaminoalkanes

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ABSTRACT

Heating a mixture of 2 2:1 molar ratio of vinylphosphonate with 1,3-diaminopropane leads to the formation N,N-bis-[(dialkoxyphosphoryl)ethyl]-1,3-diaminopropane. The tetraphosphorylated diaminoalkanes were obtained on the basis of the Kabachnik-Fields reaction.

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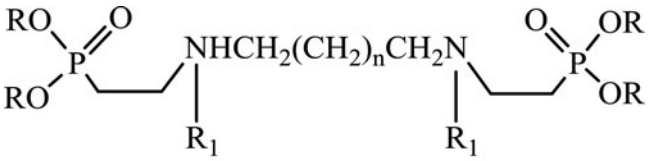
1,3-Diaminopropane; N,N-bis-[(dialkoxyphosphoryl)ethyl]-1,3-diaminopropane; tetraphosphorylated diaminoalkane; vinylphosphonate

GRAPHICAL ABSTRACT



R_1= H; R = Et; *i*-Pr

$R_1 = (p\text{-Tol})_2P(O)CH_2$; R = Et; n = 0; 1

Introduction

Synthesis of the new nitrogen-containing polyphosphonates and polyphosphineoxides is an important field of the modern organoelement chemistry. Stable interest to the creation of new synthetic methods leading to these compounds is due to their potential application as biological active substances, extractants and membrane transporters.[1,2]

Results and discussion

Having the goal of obtaining the new bisphosphorylated diamines we have studied the interaction of two-fold abundance of dialkylvinylphosphonates with 1,3-diaminopropane. Reactants heating at 85°C for 1 h leads to the formation of N,N-bis-[(dialkoxyphosphoryl)-ethyl]-1,3-diaminopropanes **1**, which consist of two phosphorylethane groups symmetrically linked to each other via the 1,3-diaminopropane bridge (Scheme 1).

The molecular structure of adducts **1** was investigated via IR, ^{1}H, ^{13}C, ^{31}P NMR spectroscopy. In the ^{1}H NMR spectrum of the products **1** we have observed signals having the following δ_H ppm: 2.1dt (P(O)C$\underline{H}_2$, $^2J_{PH}$ 14.0, $^3J_{HH}$ 7.6 Hz), 2.9 m (NC$\underline{H}_2$CH$_2$C$\underline{H}_2$N), 3.15 m (P(O)CH$_2$C$\underline{H}_2$). ^{31}P NMR spectrum has δ_P at 30.6 ppm. It was found that the compounds **1** show antibacterial activity.

The introduction of the more two phosphorus-containing fragments into molecule of compounds **1** can explain the appearance of the new practically useful property of these derivatives. For the synthesis of the tetraphosphorylated diaminoalkanes we have used the reaction of the compound **1** with formaldehyde and di-(*p*-toluene) phosphineoxide (the Kabachnik-Fields reaction) (Scheme 2). In the ^{31}P NMR spectrum of the product **2** there are two phosphorus signals having the values δ_P 29.3 and 30.9 ppm.

$$(RO)_2P(O)CH{=}CH_2 + NH_2(CH_2)_3NH_2 \longrightarrow [(RO)_2P(O)CH_2CH_2NH(CH_2)_2]CH_2$$

R = Et (1a); *i*-Pr (1b) — **1**

Scheme 1. The reaction of vinylphosphonates with 1,3-diaminopropane.

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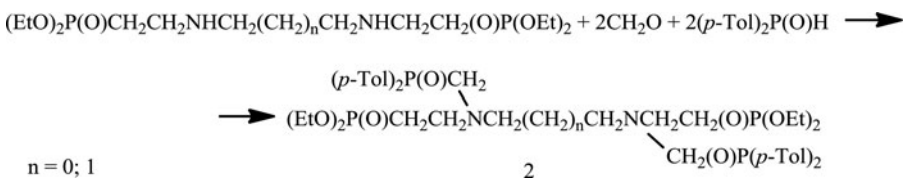

Scheme 2. The Kabachnik-Fields reaction.

Conclusions

Employing the 2:1 reactant ratio (vinylphosphonate:diamine) resulted in the double phosphorylation of the diamine. Heating a mixture of N,N-bis-[(dialkoxyphosphoryl)-ethyl] diaminoalkane with paraform and di-(*p*-toluene)phosphine-oxide in the presence of the p-toluenesulfonic acid leads to the formation of tetraphosphorylated diaminoalkanes.

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