

3,4-Bis(diethoxyphosphorylmethyl)furan in Electrophilic Substitution Reactions

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Received February 26, 2015

Abstract—Procedure for preparation of 3,4-bis(diethoxyphosphorylmethyl)furan is developed and its behavior in the electrophilic substitution reactions is studied. Formylation according to Vilsmeier and acetylation with acetic anhydride in the presence of phosphoric acid lead to the formation of the corresponding 2-formyl and 2-acetyl derivatives. In the reaction with dimethylmethylenammonium chloride in acetonitrile 2-dimethylaminomethyl derivative is formed. The reaction with paraformaldehyde and hydrogen chloride in the presence of zinc chloride gives exclusively 2,5-bis(chloromethyl)-3,4-bis(diethoxyphosphorylmethyl)furan. From this compound by means of the Michaelis–Becker reaction 2,3,4,5-tetrakis(diethoxyphosphorylmethyl)furan was synthesized. Coupling constants between phosphorus atoms in polyphosphonates obtained are evaluated.

Keywords: diphosphonates, furans, formylation, acetylation, aminomethylation, chloromethylation, Michaelis–Becker reaction

DOI: 10.1134/S1070363215070130

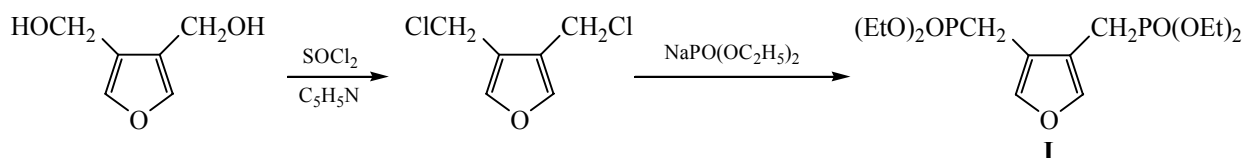
Continuing our studies in the field of bis-phosphorylated furan derivatives where dialkoxyphosphorylmethyl groups form a chelate center, in this work we report on the synthesis and functionalization of 3,4-bisphosphorylated furans. 3,4-Bis(diethoxyphosphorylmethyl)furan **I** was prepared recently [1] in 17% yield, but detailed studies showed that the procedure used can be modified. It occurred that the starting 3,4-bis(hydroxymethyl)furan was significantly less sensitive to the action of acidic agents than it was previously assumed. Besides, it rather slowly reacts with thionyl chloride in the presence of pyridine. Therefore while performing the reaction in ethyl acetate the reaction mixture should be kept for a day. 3,4-Bis(chloromethyl)furan prepared in such a way (71% yield) is a colorless oil retaining its properties after storage for a week at room temperature. A subsequent phosphorylation of this compound by

means of the Michaelis–Becker protocol lead to the formation of target bis(diethoxyphosphorylmethyl)furan **I** in 68% yield. ^1H and ^{31}P NMR data of the compound synthesized were in agreement with the reported data [1] (Scheme 1).

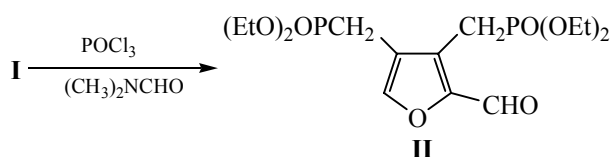
In order to establish the influence of two diethoxyphosphorylmethyl groups on chemical properties of the furan ring phosphonate **I** was brought in the electrophilic substitution reactions characteristic of the furan compounds.

The formylation of phosphonate **I** according to Vilsmeier reaction was carried out at phosphonate : POCl_3 molar ratio 1.2 in DMF at 40–45°C for 8 h. These conditions are much more rigid than those used for the formylation of furan and furfuryl alcohol esters and ethers. At the same time mono(diethoxyphosphorylmethyl)furans under these conditions de-

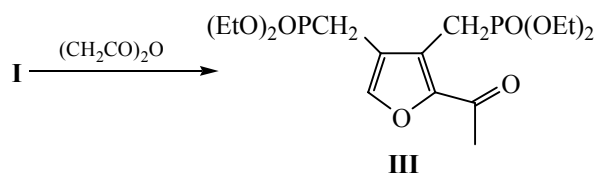
Scheme 1.



compose but do not undergo formylation. After decomposition of the reaction mixture with water and corresponding workup phosphorylated aldehyde **II** was obtained in 23% yield. In its ^1H NMR spectrum the signal of the aldehyde proton was observed at 9.69 ppm, and the signal of the corresponding carbon atom in the ^{13}C NMR spectrum, at 178.76 ppm. Chemical shifts of phosphorus atoms were 23.94 ppm (P^3) and 25.73 ppm (P^4) with $^5J_{\text{PP}}$ coupling constant 3.1 Hz. The signal of the ring carbon atom C^3 was a doublet of doublets with the coupling constants $^2J_{\text{PC}} = 9.5$ Hz and $^3J_{\text{PC}} = 3.7$ Hz. The signal of the ring carbon atom C^4 was also a doublet of doublets with the coupling constants 8.3 and 3.7 Hz respectively.



The acetylation of phosphonate **I** was carried out in the excess of acetic anhydride at elevated temperature. Using magnesium perchlorate as catalyst was not effective. After boiling for 7 h only the starting substance was isolated from the reaction mixture. The reaction in the presence of 85% phosphoric acid permitted obtaining acetyl derivative **III** in 52% yield. Its structure was confirmed by NMR data. The signal of the acetyl group protons was observed at 2.46 ppm, and signals of methyl and carbonyl carbon atoms appeared at 26.81 and 188.89 ppm respectively. In the ^{31}P NMR two signals at 25.19 ppm (P^3) and 26.27 ppm (P^4) with the coupling constant $^5J_{\text{PP}}$ 3.1 Hz were present.

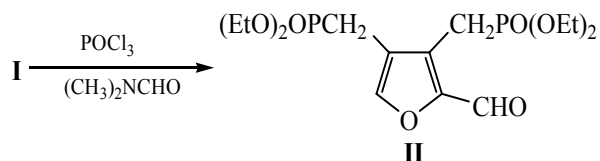


We also tried to carry out nitration of diphosphonate **I** using typical protocol for nitration of furan compounds with acetyl nitrate [2]. It occurred that the compound under study was strongly deactivated because of the influence of phosphorus-containing groups. At diphosphonate **I**–nitric acid molar ratio 1 : 2 and 10-fold excess of acetic anhydride no reaction took place. Meanwhile, 3-(2-furyl)acrylic acid having the furan ring deactivated with a pronounced electron-acceptor group under these conditions forms nitro derivative. When sulfuric acid was added to the solution of acetyl nitrate in acetic anhydride, the

reaction temperature was increased to 3–6°C, and the reaction mixture was kept for 2 h at 6–8°C light orange oil was isolated. According to NMR data this compound contained no furan ring. Therefore under more acidic conditions decomposition of the heterocycle takes place.

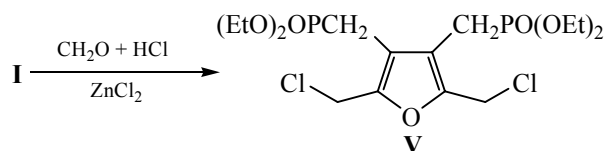
Our attempt to obtain 2-nitro-3,4-bis(chloromethyl)furan by nitration of 3,4-bis(chloromethyl)furan was also unsuccessful. Similarly to the above-described case only decomposition of substrate was observed. No individual products were isolated.

Aminomethylation of diphosphonate **I** with dimethylmethylenammonium chloride was carried out in acetonitrile at 80°C. We failed to obtain crystalline chloride or picrate, and the reaction product **IV** was isolated as a free base in 86% yield. This compound is a light brown viscous syrup. At heating to 70–80°C it decomposes with the liberation of dimethylamine to give a glass-like mass. In the ^{31}P NMR spectrum the signals of both phosphorus atoms have the same chemical shift value of 26.72 ppm. Signals of the methylene group protons near phosphorus atoms appear as doublets at 3.03 ppm (CH_2P^4 , $J_{\text{PH}} = 20.8$ Hz) and 3.11 ppm (CH_2P^3 , $J_{\text{PH}} = 20.4$ Hz). Doublet signals of carbon atoms of these groups are present at 21.42 and 21.47 ppm with the coupling constants $^1J_{\text{PC}} = 142.4$ and 142.3 Hz respectively. C^4 carbon atom of the furan ring gives a doublet of doublets at 113.99 ppm with the coupling constants $^2J_{\text{PC}} = 9.5$ Hz and $^3J_{\text{PC}} = 5.9$ Hz. The signal of C^3 carbon atom at 115.85 ppm is broadened. Its coupling constant $^2J_{\text{PC}}$ is 6.1 Hz. The broadening of the signal is probably due to the interaction between dimethylaminomethyl and phosphoryl groups.

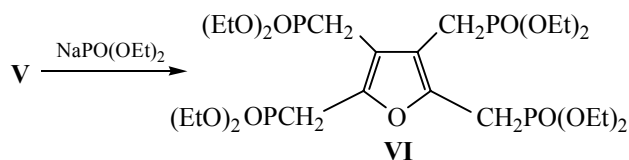


The chloromethylation of diphosphonate **I** was carried out in dichloroethane at 2–3°C by passing hydrogen chloride through the solution of the starting substance containing the suspended paraformaldehyde and zinc chloride. The only reaction product was 2,5-bis(chloromethyl)-3,4-bis(diethoxyphosphorylmethyl)furan **V**. It is interesting to note that no admixture of diphosphonate **I** was observed in the final product despite of the unsufficient amount of paraformaldehyde in the reaction mixture. It may arise from the

acidic hydrolysis of phosphonate groups giving rise to water-soluble compounds. The composition of reaction products shows that the introduction of second chloromethyl group in the furan ring proceeds faster, than the addition of the first one. At the same time the dealkylation of bis(chloromethyl)diphosphonate **V** under the action of hydrogen chloride proceeds probably much slower than that of unsubstituted analog **I**. That is why only the reaction product **V** was isolated while processing the reaction mixture. Under the molar ratio diphosphonate **I**–paraformaldehyde–zinc chloride 1 : 1.5 : 0.25 the yield of compound **V** was 70% with respect to the starting diphosphonate. The product obtained was a crystalline substance with mp 48°C gradually darkening on storage. The structure of compound **V** was confirmed by presence of the signal of the chloromethyl group proton at 4.58 ppm and of the corresponding signal of the carbon atom at 35.59 ppm in the NMR spectra. The chemical shift of phosphorus nuclei was 25.60 ppm.



Compound **V** was involved in the reaction with sodium diethyl phosphite in the 1 : 3 molar ratio. The phosphorylation was carried out in benzene at 80°C to give tetraphosphonate **VI** in 59% yield. Signals of phosphorus atoms were observed as doublets at 23.08 ppm (P^2) and 26.93 ppm (P^3) with the coupling constant $^5J_{PP}$ 14.7 Hz. The signals of carbon atoms of the furan ring were broadened multiplets preventing the evaluation of $^2J_{PC}$ and $^3J_{PC}$ constants.



Hence, 3,4-bis(diethoxyphosphorylmethyl)furan enters in a considerably broad range of the electrophilic substitution reactions, but the reaction conditions are significantly more rigid than in the case of furan compounds with positions 2 and 5 unoccupied by phosphonate groups. In the course of chloromethylation despite of deactivation of the furan ring after entering of the first halomethyl group subsequent chloromethylation proceeds faster than chloromethylation of phosphonate **I**. Due to that within the investigated limits of reagent ratio no formation of 2-chloro-

methyated diphosphonate was found. This effect was not marked in previous investigations of chloroalkylation of the furylmethanephosphonic acids [3, 4].

After introduction of formyl and acetyl groups in the position 2 of diphosphonate **I** the phosphorus atoms become nonequivalent. In the ^{31}P NMR spectra the coupling constant between them can be observed. Its value is 3.1 Hz, significantly smaller than in the case of 2,3-bis(phosphonates) [5]. The equality of $^2J_{PC}$ and $^3J_{PC}$ constants found for C^2 and C^3 carbon atoms of the furan ring in 2,3-bis(phosphonates) in the case of C^3 and C^4 carbon atoms of the furan ring of 3,4-bis(phosphonates) is disturbed and the corresponding signals in the ^{13}C NMR spectra acquire the shape of doublets of doublets. Consequently, the magnetic interaction between two phosphorus atoms or phosphorus and carbon atoms significantly depends on the angle between the straight line connecting these atoms and the direction of polarization of the furan ring under the action of oxygen atom.

EXPERIMENTAL

1H , ^{13}C , and ^{31}P NMR spectra were taken on a Bruker DPX-400 spectrometer (400.13 MHz 1H , 161.97 MHz ^{31}P , 100.16 MHz ^{13}C respectively) in $CDCl_3$.

3,4-Bis(chloromethyl)furan. To a solution of 5.9 g of 3,4-bis(hydroxymethyl) furan and 8 mL of pyridine in 50 mL of ethyl acetate a solution of 6.7 mL of thionyl chloride in 7 mL of ethyl acetate was added dropwise with stirring. The temperature of the reaction mixture was maintained no higher than 35°C. The reaction mixture was stirred for 3 h and left overnight. On the next day pyridine hydrochloride was filtered off, the filtrate was washed with 5% hydrochloric acid, then with water, and finally with sodium bicarbonate solution. The organic phase was dried over calcium chloride and distilled in a vacuum to give 4.9 g (71%) of the target product, a colorless oil of bp 86°C (1 mmHg). 1H NMR spectrum, δ , ppm: 4.61 s (4H, CH_2Cl), 7.47 s (2H, $H^{2,5}$ -furan). ^{13}C NMR spectrum, δ_C , ppm: 35.66 (CH_2Cl), 121.58 ($C^{3,4}$ -furan), 142.50 ($C^{2,5}$ -furan).

3,4-Bis(diethoxyphosphorylmethyl)furan (I). To a solution of sodium diethyl phosphite prepared from 0.6 g of sodium and 4.7 mL of diethyl hydrogen phosphite in 50 mL of benzene a solution of 1.9 g of 3,4-bis(chloromethyl)furan in 5 mL of benzene was

added at 50–60°C under vigorous stirring. The mixture formed was refluxed with stirring for 19 h, then washed with 20 mL of water, and dried over sodium sulfate. After removing the solvent the residue was kept in a vacuum (1 mmHg) at room temperature for 1 h. Yield of phosphonate **I** 2.9 (68%), viscous syrup gradually crystallizing on storage, mp 63–65°C. ¹H NMR spectrum, δ , ppm: 1.22 t (12H, CH₃, J_{HH} 7.0 Hz), 3.01 d (4H, CH₂P, J_{PH} 20.4 Hz), 4.01 m (8H, CH₂OP J_{HH} 7.0 Hz, J_{PH} 14.4 Hz), 7.33 d (2H, H⁵-furan, J_{PH} 3.2 Hz). ¹³C NMR spectrum, δ_{C} , ppm: 16.32 d (CH₃, $^3J_{\text{PC}}$ 6.0 Hz), 21.29 d (CH₂P, $^1J_{\text{PC}}$ 142.7 Hz), 62.04 d (CH₂OP, $^2J_{\text{PC}}$ 6.8 Hz), 115.31 d.d (C^{3,4}-furan, $^2J_{\text{PC}}$ 8.9 Hz, $^3J_{\text{PC}}$ 6.1 Hz), 141.41 d (C^{2,5}-furan, $^3J_{\text{PC}}$ 8.4 Hz). ³¹P NMR spectrum, δ_{P} , ppm: 26.62.

3,4-Bis(diethoxyphosphorylmethyl)furan-2-carbaldehyde (II). Phosphorus oxychloride, 0.4 mL, was added in one portion with stirring to 3 mL of DMF. The reaction mixture was kept for 20 min at room temperature, and then the solution of 1.2 g of diphosphonate **I** in 3 mL of DMF was added in one portion. After that the mixture obtained was stirred for 8 h at 40–45°C and then poured in 30 mL of ice water. The solution obtained was treated with small portions of sodium carbonate to pH = 10 and then kept at room temperature for 4 h. After that it was extracted with chloroform (3 × 15 mL), washed with saturated solution of sodium chloride, and dried over sodium sulfate. After removing the solvent the residue was kept in a vacuum (1 mmHg) for 1.5 h at room temperature. Yield of compound **II** 0.3 g (23%), yellowish brown syrup. ¹H NMR spectrum, δ , ppm: 1.18–1.24 m (12H, CH₃), 3.13 d (2H, CH₂P⁴, J_{PH} 20.4 Hz), 3.53 d (2H, CH₂P³, J_{PH} 22.4 Hz), 3.97–4.06 m (8H, CH₂OP), 7.56 d (1H, H⁵-furan, J_{PH} 3.2 Hz), 9.69 s (1H, CHO). ¹³C NMR spectrum, δ_{C} , ppm: 16.22 d (CH₃, $^3J_{\text{PC}}$ 6.4 Hz), 16.31 d (CH₃, $^3J_{\text{PC}}$ 6.2 Hz), 21.05 d (CH₂P⁴, $^1J_{\text{PC}}$ 142.5 Hz), 21.75 d (CH₂P³, $^1J_{\text{PC}}$ 138.7 Hz), 62.24 d (CH₂OP, $^2J_{\text{PC}}$ 6.6 Hz), 62.40 d (CH₂OP, $^2J_{\text{PC}}$ 6.7 Hz), 119.51 d.d (C⁴-furan, $^2J_{\text{PC}}$ 8.9 Hz, $^3J_{\text{PC}}$ 3.0 Hz), 126.64 d.d (C³-furan, $^2J_{\text{PC}}$ 9.5 Hz, $^3J_{\text{PC}}$ 3.7 Hz), 145.97 d (C⁵-furan, $^3J_{\text{PC}}$ 6.3 Hz), 149.29 d (C²-furan, $^3J_{\text{PC}}$ 8.0 Hz), 178.76 (CHO). ³¹P NMR spectrum, δ_{P} , ppm: 25.73 d (P⁴), 23.94 d (P³), $^5J_{\text{PP}}$ 3.1 Hz.

2-Acetyl-3,4-bis(diethoxyphosphorylmethyl)furan (III). A mixture of 1.2 g of diphosphonate **I**, 5 mL of acetic anhydride, and 2 drops of 85% phosphoric acid was heated for 7 h with stirring at 90–95°C. Then the reaction mixture was poured in 25 mL of water, neutralized with sodium bicarbonate, extracted with

chloroform (3 × 15 mL), and the extract was dried over sodium sulfate. After removing chloroform the residue was kept in a vacuum (1 mmHg) for 1.5 h at room temperature. Yield of compound **III** 0.7 g (52%), light brown glass. ¹H NMR spectrum, δ , ppm: 1.24 t (6H, CH₃, J_{HH} 7.2 Hz), 1.28 t (6H, CH₃, J_{HH} 7.2 Hz), 2.46 s (3H, CH₃-acetyl), 3.18 d (2H, CH₂P⁴, J_{PH} 20.4 Hz), 3.67 d (2H, CH₂P³, J_{PH} 22.4 Hz), 4.02–4.11 m (8H, CH₂OP), 7.48 d (1H, H⁵-furan, J_{PH} 3.2 Hz). ¹³C NMR spectrum, δ_{C} , ppm: 16.24 d (CH₃, $^3J_{\text{PC}}$ 6.2 Hz), 16.36 d (CH₃, $^3J_{\text{PC}}$ 5.9 Hz), 21.11 d (CH₂P⁴, $^1J_{\text{PC}}$ 142.1 Hz), 22.13 d (CH₂P³, $^1J_{\text{PC}}$ 137.5 Hz), 26.81 (CH₃), 62.23 d (CH₂OP, $^2J_{\text{PC}}$ 4.8 Hz), 119.30 d.d (C⁴-furan, $^2J_{\text{PC}}$ 8.5 Hz, $^3J_{\text{PC}}$ 3.0 Hz), 124.10 d.d (C³-furan, $^2J_{\text{PC}}$ 10.7 Hz, $^3J_{\text{PC}}$ 6.0 Hz), 143.37 d (C⁵-furan, $^3J_{\text{PC}}$ 8.0 Hz), 149.03 d (C²-furan, $^3J_{\text{PC}}$ 8.5 Hz), 188.89 (C=O). ³¹P NMR spectrum, δ_{P} , ppm: 26.27 d (P⁴), 25.19 d (P³), $^5J_{\text{PP}}$ 3.1 Hz.

2-(N,N-Dimethylaminomethyl)-3,4-bis(diethoxyphosphorylmethyl)furan (IV). A mixture of 0.6 g of diphosphonate **I**, 0.2 g of dimethylmethylenammonium chloride, and 6 mL of acetonitrile was refluxed with stirring at 80°C for 6 h. After that the reaction mixture was treated with 4.5 mL of sodium ethylate solution prepared from 0.1 g of sodium and 10 mL of ethanol. Sodium chloride was filtered off, the filtrate was evaporated at reduced pressure, and the residue was kept in a vacuum (1 mmHg) at room temperature for 1 h. Yield of amine **IV** 0.6 g (86%), light brown syrup. ¹H NMR spectrum, δ , ppm: 1.21 t (12H, CH₃, J_{HH} 7.2 Hz), 2.26 s [6H, N(CH₃)₂], 3.02 d (2H, CH₂P⁴, J_{PH} 20.8 Hz), 3.11 d (2H, CH₂P³, J_{PH} 20.4 Hz), 3.53 d (2H, N-CH₂-furan, J_{PH} 2.0 Hz), 4.02–4.11 m (8H, CH₂OP), 7.31 d (1H, H⁵-furan, J_{PH} 3.6 Hz). ¹³C NMR spectrum, δ_{C} , ppm: 16.32 d (CH₃, $^3J_{\text{PC}}$ 4.9 Hz), 21.43 d (CH₂P, $^1J_{\text{PC}}$ 142.3 Hz), 21.47 d (CH₂P³, $^1J_{\text{PC}}$ 142.3 Hz), 44.61 [N(CH₃)₂], 53.55 (N-CH₂-furan), 62.08 d (CH₂OP, $^2J_{\text{PC}}$ 6.0 Hz), 113.99 d.d (C⁴-furan, $^2J_{\text{PC}}$ 9.5 Hz, $^3J_{\text{PC}}$ 5.9 Hz), 115.85 br.d (C³-furan, $^2J_{\text{PC}}$ 6.1 Hz), 140.51 d (C⁵-furan, $^3J_{\text{PC}}$ 7.7 Hz), 148.89 d (C²-furan, $^3J_{\text{PC}}$ 10.1 Hz). ³¹P NMR spectrum, δ_{P} , ppm: 26.72 (P^{3,4}).

2,5-Bis(chloromethyl)-3,4-bis(diethoxyphosphorylmethyl)furan (V). Through the suspension of 0.32 g of paraformaldehyde and 0.24 g of finely pulverized zinc chloride in the solution of 2.6 g of diphosphonate **I** in 30 mL of dichloroethane hydrogen chloride was passed at 2–3°C for 2 h under the intense stirring. After that the reaction mixture was washed with water (2 × 10 mL), with 10 mL of sodium bicarbonate solution, and with 10 mL of saturated sodium chloride solution. The organic phase obtained

was dried over sodium sulfate, and the solvent was removed at a reduced pressure to give 2.3 g (70%) of dichloride **V**, colorless crystals of mp 48°C. ^1H NMR spectrum, δ , ppm: 1.24 t (12H, CH_3 , J_{HH} 7.2 Hz), 3.10 d (4H, CH_2P , J_{PH} 20.4 Hz), 4.02 m (8H, CH_2OP , J_{HH} 7.2 Hz, J_{PH} 14.4 Hz), 4.58 s (4H, CH_2Cl). ^{13}C NMR spectrum, δ_{C} , ppm: 16.32 d (CH_3 , $^3J_{\text{PC}}$ 5.5 Hz), 21.77 d (CH_2P , $^1J_{\text{PC}}$ 142.5 Hz), 35.59 (CH_2Cl), 62.32 d (CH_2OP , $^2J_{\text{PC}}$ 6.6 Hz), 115.91 d.d ($\text{C}^{3,4}$ -furan, $^2J_{\text{PC}}$ 9.7 Hz, $^3J_{\text{PC}}$ 2.2 Hz), 147.97 d ($\text{C}^{2,5}$ -furan, $^3J_{\text{PC}}$ 8.9 Hz). ^{31}P NMR spectrum, δ_{P} , ppm: 25.60.

2,3,4,5-Tetrakis(diethoxyphosphorylmethyl)furan (VI). To a solution of sodium diethyl phosphite prepared from 0.2 g of sodium and 1.4 mL of diethyl hydrogen phosphite in 30 mL of benzene the solution of 1.2 g of dichloride **V** in 5 mL of benzene was added. The reaction mixture was refluxed with stirring for 9 h at 80°C, diluted with 20 mL of ethyl acetate, and washed with 15 mL of water, and then with 15 mL of saturated sodium chloride solution. The organic solution obtained was dried over sodium sulfate. After that the solvent was removed at a reduced pressure, and the residue was kept in a vacuum (1 mmHg) for 2 h at room temperature to give 1.0 g (59%) of tetraphosphonate **VI** as a viscous syrup. ^1H NMR

spectrum, δ , ppm: 1.19–1.21 m (12H, CH_3), 3.08 d (4H, $\text{CH}_2\text{P}^{3,4}$, J_{PH} 20.0 Hz), 3.25 d (4H, $\text{CH}_2\text{P}^{2,5}$, J_{PH} 19.2 Hz), 3.92–4.00 m (8H, CH_2OP). ^{13}C NMR spectrum, δ_{C} , ppm: 16.33 br.s (CH_3), 21.29 d ($\text{CH}_2\text{P}^{3,4}$, $^1J_{\text{PC}}$ 141.8 Hz), 25.34 d ($\text{CH}_2\text{P}^{2,5}$, $^1J_{\text{PC}}$ 142.8 Hz), 61.98 d (CH_2OP , $^2J_{\text{PC}}$ 6.6 Hz), 62.33 br.s (CH_2OP), 113.85 br.s ($\text{C}^{3,4}$ -furan), 142.60 br.s ($\text{C}^{2,5}$ -furan). ^{31}P NMR spectrum, δ_{P} , ppm: 23.08 d ($\text{P}^{2,5}$), 26.93 d ($\text{P}^{3,4}$), $^5J_{\text{P}^{2,5}\text{P}^{3,4}}$ 14.7 Hz.

ACKNOWLEDGMENTS

The work was carried out within the frame of State Project of Ministry of Science and Education of Russia

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