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Synthesis and Acylation of *O*,*O*-Dihexyl(dioctyl)-[1-hydroxy-3-(ethyl, diethylamino)-2,2-dimethylpropyl]phosphonates

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Abstract—Reaction of dialkyl phosphites with 3-ethylamino- or 3-diethylamino-substituted aldehydes results in the corresponding 3-amino-1-hydroxyalkylphosphonates. The composition and structure of the obtained compounds were confirmed by elemental analysis, NMR spectroscopy and chemical transformations.

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Previously we have synthesized 3-amino-1-hydroxyalkylphosphonates containing MeO and EtO substituents at the P(IV) atom, which showed weak bactericidal activity [1]. It is known that the complexing ability of organophosphorus compounds including those containing amino group increases significantly as the number of substituents at the atom P(IV) increases [2].

Aiming to synthesize polyfunctional aminophosphoryl organic compounds with more pronounced applied properties, we have studied the interaction of dialkyl phosphites **II** containing long-chain hydrocarbon substituents with 3-ethylamino- or 3-diethylaminoaldehydes **I** and the acylation of the reaction products with different acylating agents.

Reactions of dialkylphosphorous acids **IIa**, **IIb** with 3-alkylamino-substituted aldehydes **Ia**, **Ib** occur in the absence of a catalyst with a small exothermic effect. It is known that the Abramov reaction [3] proceeds in the presence of bases as a catalyst. The formation of phosphonates **III** in the absence of a catalyst is caused by the nucleophilic catalysis by the secondary or tertiary amino group present in the starting aldehyde **I**.

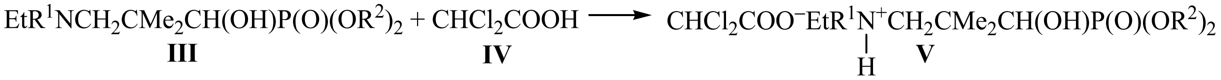
$$\underset{\mathbf{I}}{\mathrm{EtR^1NCH_2CMe_2CHO}} + \underset{\mathbf{II}}{\mathrm{(R^2O)_2PHO}} \longrightarrow \underset{\mathbf{IIIa–IIIc}}{\mathrm{EtR^1NCH_2CMe_2CH(OH)P(O)(OR^2)_2}}$$

I, R^1 = H (**a**), Et (**b**); **II**, R^2 = Hex (**a**), Oct (**b**); **III**, R^1 = Et, R^2 = Hex (**a**), Oct (**b**); R^1 = H, R^2 = Hex (**c**).

The composition and structure of phosphonates **IIIa**–**IIIc** were confirmed by elemental analysis and ^{1}H, ^{31}P NMR spectra. The ^{31}P NMR spectra of compounds **IIIa**–**IIIc** contain the only signal in the range of 24.2–24.5 ppm.

In order to prove the structure of compounds **III** by chemical transformations and to synthesize new *P*- and *N*-containing polyfunctional organic compounds we studied different reactions of phosphonates with acylating agents: dichloroacetic acid **IV**, carboxylic acid chlorides **VI**, and acetic anhydride **X**.

The interaction of phosphonate **III** with dichloroacetic acid **IV** results in stable salts (3-hydroxy-3-*O*,*O*-dialkylphosphoryl-2,2-dimethylpropyl)ethyl- and diethylammonium dichloroacetates **V** as syrupy substances. The acylation on the N–H and O–H bonds does not occur.



$$\underset{\mathbf{III}}{\mathrm{EtR^1NCH_2CMe_2CH(OH)P(O)(OR^2)_2}} + \underset{\mathbf{IV}}{\mathrm{CHCl_2COOH}} \longrightarrow \underset{\mathbf{V}}{\mathrm{CHCl_2COO^{-}EtR^1\underset{\overset{|}{H}}{N^{+}}CH_2CMe_2CH(OH)P(O)(OR^2)_2}}$$

V, R^1 = Et, R^2 = Hex (**a**), Oct (**b**); R^1 = H, R^2 = Hex (**c**).

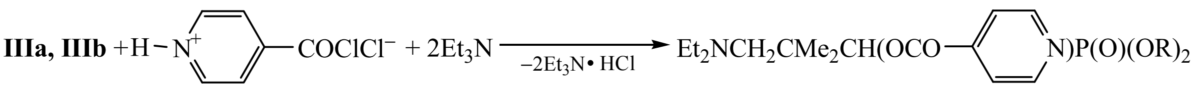
The reactions were carried out by mixing the reactants in anhydrous diethyl ether while cooling and maintaining the reaction mixture at room temperature for 1 day. After removing the solvent and volatile components in a high vacuum salts **V** were obtained as syrupy substances whose composition and structure were confirmed by elemental analysis, IR, ^{1}H, and ^{31}P NMR spectra.

The ^{31}P NMR spectra of the salts **V** contain a singlet signals in the range of 20.5–25.4 ppm corresponding to the nearest environment of the phosphorus atom $O_2P(O)C$. The ^{1}H NMR spectra of salts **V** are given in the experimental section. It should be noted that the transformation N(III) → N$^+$(IV) causes a downfield shift of the resonance signals of the hydrogen atoms of methylene groups 2.25–2.75 → 3.15–3.40 ppm. The presence of the absorption bands at 2500 and 2673 cm^{-1} in the IR spectra corresponding to the vibrations of N$^+$H fragment confirms the proposed structure of salts **V** [4].

Studies of acylation of phosphonates **III** with benzoyl chloride **VIa** and 4-(chloroformyl)azinium chloride (isonicotinoyl chloride hydrochloride) **VIb** showed that depending on the structure of aminophosphonate and the reagents ratio occurs *O*-, *N*- and *N*,*O*-acylation.

The interaction of *O*,*O*-dialkyl [1-hydroxy-3-(diethylamino)-2,2-dimethylpropyl]phosphonates **IIIa**, **IIIb** with acylating agents **VI** in a ratio of 1:1 gives rise to the *O*-acylation product **VII**.

$$\mathbf{IIIa,\ IIIb} + \underset{\mathbf{VIa}}{PhCOCl} + Et_3N \xrightarrow[-Et_3N\bullet HCl]{} \underset{\mathbf{VIIa,\ VIIb}}{Et_2NCH_2CMe_2CH(OCOPh)P(O)(OR)_2}$$



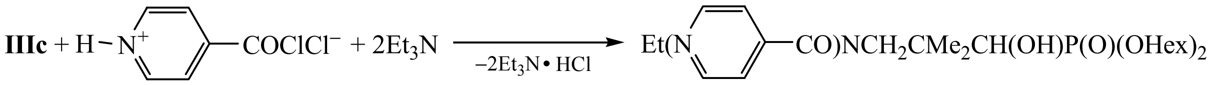
VIb **VIIc, VIId**

R = Hex (**a**, **c**), Oct (**b**, **d**).

The reaction was carried out in a solution of anhydrous diethyl ether or THF at room temperature in the presence of triethylamine. After the removal of triethylamine hydrochloride, distilling off the solvent and volatile components in a high vacuum, the products **VII** were identified in a crude form because of their thermal lability. The composition and structure of compounds **VII** were confirmed by elemental analysis, ^{1}H and ^{31}P NMR spectra. The ^{31}P NMR spectra are characterized by the presence of one singlet signal at 19.58–24.58 ppm, corresponding to phosphonate structure of compounds **VIIa**–**VIId**.

The acylation of phosphonate **IIIc** with compounds **VI** in a 1:1 ratio of the reactants in the presence of triethylamine affords *N*-acylation product **VIII**.

$$\mathbf{IIIc} + \underset{\mathbf{VIa}}{PhCOCl} + Et_3N \xrightarrow[-Et_3N\bullet HCl]{} \underset{\mathbf{VIIIa}}{Et(PhCO)NCH_2CMe_2CH(OH)P(O)(OHex)_2}$$



VIb **VIIIb**

The ^{31}P NMR spectra of compounds **VIIIa**, **VIIIb** are characterized by the presence of a singlet signal at 23.3–24.7 ppm.

Reaction of compound **IIIc** with a double excess of benzoyl chloride **VIa** in THF at reflux results in the *N*,*O*-diacylation product.

$$\mathbf{IIIc} + \underset{\mathbf{VIa}}{2PhCOCl} + 2Et_3N \xrightarrow[-2Et_3N\bullet HCl]{} \underset{\mathbf{IX}}{Et(PhCO)NCH_2CMe_2CH(OCOPh)P(O)(OHex)_2}$$

The *N*,*O*-diacylation product is also formed in the reaction of phosphonate **IIIc** with two equivalents of acetic anhydride **X** at 90°C.

The composition and structure of compounds **IX**, **XI** were proved by elemental analysis, ^{1}H and ^{31}P NMR spectra. The ^{31}P NMR spectra are characterized

$$\textbf{IIIc} + \underset{\textbf{X}}{2(MeCO)_2O} \longrightarrow \underset{\textbf{XI}}{Et(MeCO)NCH_2CMe_2CH(OCOMe)P(O)(OHex)_2}$$

by the presence of only a singlet at 20.9–25.3 ppm, corresponding to the structure of phosphonate.

EXPERIMENTAL

The ^1H NMR spectra were recorded on a Tesla BS-567A spectrometer operating at 100 MHz and on a Bruker MSL-400 instrument operating at 400 MHz. Chemical shifts were measured relative to the residual proton signals of the deuterated solvents ($CDCl_3$, acetone-d_6, and DMSO-d_6). The ^{31}P NMR spectra were recorded on a Bruker MSL-400 (162 MHz) and Bruker WN-250 (101 MHz) spectrometers, external reference 85% H_3PO_4. The IR spectra were registered on a Perkin Elmer Spectrum 65 FT-IR spectrometer in the range of 400–4000 cm^{-1} (NaCl prism, thin layer, mineral oil).

***O*,*O*-Dihexyl (1-hydroxy-3-diethylamino-2,2-dimethylpropyl)phosphonate (IIIa).** To 2.5 g (16 mmol) of 3-diethylamino-2,2-dimethylpropanal **Ia** was added dropwise 4 g (16 mmol) of dihexylphosphorous acid **IIa**. There was a temperature rise to 45°C. The mixture was kept at room temperature for 48 h. Then the volatile components were removed in a high vacuum to give 6.2 g (95.4%) of phosphonate **IIIa**. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.25–1.25 m (34H, $2C_5H_{11}$, $2NCH_2\underline{Me}$, CMe_2), 2.25 m (6H, NCH_2, $2N\underline{CH_2}Me$), 3.7 m (6H, $2OCH_2$, PCH, OH). ^{31}P NMR spectrum: δ_P 24.3 ppm. Found, %: N 3.43; P 7.59. $C_{21}H_{46}NO_4P$. Calculated, %: N 3.36; P 7.51.

***O*,*O*-Dioctyl (1-hydroxy-3-diethylamino-2,2-dimethylpropyl)phosphonate (IIIb)** was prepared analogously from 2.5 g (16 mmol) of 3-diethylamino-2,2-dimethylpropanal **Ia** and 4.87 g (16 mmol) of dioctylphosphorous acid **IIb**. Yield 7 g (95%). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.6–1.7 m (42H, $2C_7H_{15}$, $2NCH_2\underline{Me}$, CMe_2), 2.55 m (6H, NCH_2, $2N\underline{CH_2}Me$), 3.8 m (6H, $2OCH_2$, PCH, OH). ^{31}P NMR spectrum: δ_P 24.5 ppm.

***O*,*O*-Dihexyl (1-hydroxy-3-ethylamino-2,2-dimethylpropyl)phosphonate (IIIc)** was prepared analogously from 5 g (39 mmol) of 3-ethylamino-2,2-dimethylpropanal **Ib** and 9.7 g (39 mmol) of dihexylphosphorous acid **IIa**. Yield 13 g (88.4%). ^1H NMR spectrum $[(CD_3)_2CO]$, δ, ppm: 0.8–1.8 m (31H, $2C_5H_{11}$, $NCH_2\underline{Me}$, CMe_2), 2.75 m (4H, $Me\underline{CH_2}\cdot NH\underline{CH_2}$), 3.60 m (1H, PCH), 4.05 m (4H, $2OCH_2$), 5.4 br.s (2H, OH, NH). ^{31}P NMR spectrum: δ_P 24.18 ppm. Found, %: N 3.75; P 8.12. $C_{19}H_{42}NO_4P$. Calculated, %: N 3.69; P 8.16.

(3-Hydroxy-3-*O*,*O*-dihexylphosphoryl-2,2-dimethylpropyl)diethylammonium dichloroethanoate (Va). To a solution of 1.2 g (3 mmol) of *O*,*O*-dihexyl (1-hydroxy-3-diethylamino-2,2-dimethylpropyl)phosphonate **IIIa** in 10 ml of anhydrous ether while stirring and cooling with cold water was added dropwise 0.387 g (3 mmol) of dichloroacetic acid **IV**. The reaction mixture gradually warmed to room temperature and was left standing for 2 days. Then the solvent and volatile components were removed in a high vacuum. Yield 1.51 g (95.2%). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.8–1.75 m (34H, $2C_5H_{11}$, $2NCH_2\underline{Me}$, CMe_2), 2.9–3.4 m (6H, NCH_2, $2N\underline{CH_2}Me$), 4.0 m (5H, $2OCH_2$, PCH), 5.8 s (1H, $CHCl_2$), 9.05 br. s (2H, N^+H, OH). ^{31}P NMR spectrum: δ_P 25.4 ppm. Found, %: N 2.67; P 5.84; Cl 13.27. $C_{23}H_{48}NO_6Cl_2P$. Calculated, %: N 2.61; P 5.77; Cl 13.22.

(3-Hydroxy-3-*O*,*O*-dioctylphosphoryl-2,2-dimethylpropyl)diethylammonium dichloroethanoate (Vb) was prepared similarly from 1.8 g (4 mmol) *O*,*O*-dioctyl (1-hydroxy-3-diethylamino-2,2-dimethylpropyl)phosphonate **IIIb** and 0.5 g (4 mmol) of dichloroacetic acid **IV**. Yield 2.1 g (91.3%). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.8–1.75 m (42H, $2C_7H_{15}$, $2NCH_2\underline{Me}$, CMe_2), 3.1–3.6 m (6H, NCH_2, $2N\underline{CH_2}Me$), 4.0 m (5H, $2OCH_2$, PCH), 5.8 s (1H, $CHCl_2$), 8.5 br. s (2H, N^+H, OH). ^{31}P NMR spectrum: δ_P 20.5 ppm.

(3-Hydroxy-3-*O*,*O*-dihexylphosphoryl-2,2-dimethylpropyl)ethylammonium dichloroethanoate (Vc) was obtained similarly from 2 g (5 mmol) of *O*,*O*-dihexyl (1-hydroxy-3-ethylamino-2,2-dimethylpropyl)phosphonate **IIIc** and 0.68 g (5 mmol) of dichloroacetic acid **IV**. Yield 2.5 g (94.3%). IR spectrum, ν, cm^{-1}: 1062 (P–O–C), 1213 (P=O), 1650 (C=O), 2500, 2673 (N^+H_2), 3120 (OH). ^1H NMR spectrum $[(CD_3)_2CO]$, δ, ppm: 0.8–1.9 m (31H, $2C_5H_{11}$, $NCH_2\underline{Me}$, CMe_2), 3.2 m (4H, $Me\underline{CH_2}N^+H_2\underline{CH_2}$), 4.15 m (5H, $2OCH_2$, PCH), 5.9 s (1H, $CHCl_2$), 7.0 br.s (3H, N^+H_2, OH). ^{31}P NMR spectrum: δ_P 24.47 ppm. Found, %: N 2.84; P 5.95; Cl 14.00. $C_{21}H_{44}NO_6Cl_2P$. Calculated, %: N 2.75; P 6.09; Cl 13.95

***O*,*O*-Dihexyl [1-(benzoyloxy)-2,2-dimethyl-3-diethylaminopropyl]phosphonate (VIIa).** To a solution of

1.2 g (3 mmol) of *O*,*O*-dihexyl (1-hydroxy-3-diethylamino-2,2-dimethylpropyl)phosphonate **IIIa** and 0.304 g (3 mmol) of triethylamine in 20 ml of anhydrous diethyl ether under constant stirring at room temperature was added a solution of 0.422 g (3 mmol) of benzoyl chloride **VIa** in 5 ml of diethyl ether. The reaction mixture was stirred at room temperature for 1 h. The precipitated triethylamine hydrochloride was filtered off and washed with diethyl ether (5 ml). Then the solvent and volatile components were removed in a high vacuum. Yield 1.72 g (89.3%). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.6–1.6 m (34H, $2C_5H_{11}$, $2NCH_2\underline{Me}$, CMe_2), 2.5 m (6H, NCH_2, $2N\underline{CH_2}Me$), 3.90 m (4H, $2OCH_2$), 5.38 d (1H, PCH, $^2J_{PH}$ 11.1 Hz), 7.2–8.0 m (5H, Ph). ^{31}P NMR spectrum: δ_P 19.58 ppm. Found, %: N 2.83; P 5.92. $C_{30}H_{52}NO_5P$. Calculated, %: N 2.74; P 6.05.

***O*,*O*-Dioctyl [1-(benzoyloxy)-2,2-dimethyl-3-diethylaminopropyl]phosphonate (VIIb)** was prepared similarly from 1.05 g (2.3 mmol) of *O*,*O*-dioctyl (1-hydroxy-3-diethylamino-2,2-dimethylpropyl)phosphonate **IIIb** and 0.32 g (2.3 mmol) of benzoyl chloride **VIa**. Yield 1.1 g (85.5%). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.8–1.8 m (42H, $2C_7H_{15}$, $2NCH_2\underline{Me}$, CMe_2), 2.7 m (6H, NCH_2, $2N\underline{CH_2}Me$), 4.0 m (4H, $2OCH_2$), 5.45 d (1H, PCH, $^2J_{PH}$ 11.0 Hz), 7.35– 8.2 m (5H, Ph). ^{31}P NMR spectrum: δ_P 24.8 ppm.

***O*,*O*-Dihexyl [3-(benzoylethylamino)-1-hydroxy-2,2-dimethylpropyl]phosphonate (VIIIa)** was prepared similarly from 2.5 g (6.6 mmol) of *O*,*O*-dihexyl (1-hydroxy-3-ethylamino-2,2-dimethylpropyl)phosphonate **IIIc**, 0.67 g (6.6 mmol) of triethylamine in 20 ml of anhydrous diethyl ether, and 0.93 g (6.6 mmol) of benzoyl chloride **VIa** in 10 ml of diethyl ether. Yield 2.7 g (80%). ^{1}H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 0.8–1.8 m (31H, $2C_5H_{11}$, $NCH_2\underline{Me}$, CMe_2), 3.6 m (4H, $Me\underline{CH_2}N\underline{CH_2}$), 4.1 m (5H, $2OCH_2$, PCH), 7.5 s (5H, Ph). ^{31}P NMR spectrum: δ_P 24.66 ppm. Found, %: N 2.81; P 6.02. $C_{28}H_{46}NO_5P$. Calculated, %: N 2.76; P 6.10.

***O*,*O*-Dihexyl [1-(4-azinecarbonyloxy)-3-diethylamino-2,2-dimethylpropyl]phosphonate (VIIc).** To a solution of 2 g (5 mmol) of *O*,*O*-dihexyl (1-hydroxy-3-diethylamino-2,2-dimethylpropyl)phosphonate **IIIa** and 1.06 g (10.3 mmol) of triethylamine in 20 ml of tetrahydrofuran under cooling and stirring was added a solution of 0.9 g (5 mmol) of 4-(chlorocarbonyl)-azinium chloride **VIb** in 10 ml of tetrahydrofuran. The reaction mixture was warmed to room temperature and kept overnight. The precipitated triethylamine hydrochloride was filtered off and washed with tetrahydrofuran (15 ml). Then the solvent and volatile components were removed in a high vacuum. Yield 2.05 g (75%). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.7–1.9 m (34H, $2C_5H_{11}$, $2NCH_2\underline{Me}$, CMe_2), 2.7 m (6H, NCH_2, $2N\underline{CH_2}Me$), 4.1 m (4H, $2OCH_2$), 5.5 d (1H, PCH, $^2J_{PH}$ 10.0 Hz), 7.9 d, 8.8 d (4H, Py). ^{31}P NMR spectrum: δ_P 20.6 ppm.

***O*,*O*-Dioctyl [1-(4-azinecarbonyloxy)-3-diethylamino-2,2-dimethylpropyl]phosphonate (VIId)** was prepared similarly from 2.5 g (5.4 mmol) of *O*,*O*-dioctyl (1-hydroxy-3-diethylamino-2,2-dimethylpropyl)-phosphonate **IIIb** and 0.96 g (5.4 mmol) of 4-(chlorocarbonyl)azinium chloride **VIb**. Yield 2.6 g (80%). ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 0.8–1.8 m (42H, $2C_7H_{15}$, $2NCH_2\underline{Me}$, CMe_2), 2.5 m (6H, NCH_2, $2N\underline{CH_2}Me$), 4.0 m (4H, $2OCH_2$), 5.4 d (1H, PCH, $^2J_{PH}$ 10.0 Hz), 7.8 d, 8.75 d (4H, Py). ^{31}P NMR spectrum: δ_P 20.1 ppm.

***O*,*O*-Dihexyl [3-(4-azinecarbonylamino)-1-hydroxy-2,2-dimethylpropyl]phosphonate (VIIIb).** To a solution of 2 g (5 mmol) of *O*,*O*-dihexyl (1-hydroxy-3-ethylamino-2,2-dimethylpropyl)phosphonate **IIIc** and 1.06 g (10.5 mmol) of triethylamine in 20 ml of tetrahydrofuran under cooling to 0°C was added a solution of 0.89 g (5 mmol) of 4-(chlorcarbonyl) azinium chloride **VIb** in 10 ml of tetrahydrofuran. The reaction mixture was warmed to room temperature and left overnight. The precipitated triethylamine hydrochloride was filtered off and washed with tetrahydrofuran (10 ml). Then the solvent and volatile components were removed in a high vacuum. Yield 2.1 g (70%). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.8–1.9 m (31H, $2C_5H_{11}$, $NCH_2\underline{Me}$, CMe_2), 2.8–3.4 m (5H, $Me\underline{CH_2}N\underline{CH_2}$, PCH), 4.1 m (4H, $2OCH_2$), 7.8 d, 8.6 d (4H, Py), 8.2 br.s (1H, OH). ^{31}P NMR spectrum: δ_P 23.27 ppm. Found, %: N 5.43; P 6.18. $C_{27}H_{45}N_2O_5P$. Calculated, %: N 5.50; P 6.09.

***O*,*O*-Dihexyl [3-(benzoylethylamino)-1-(benzoyloxy)-2,2-dimethylpropyl]phosphonate (IX).** To a solution of 2 g (5 mmol) of *O*,*O*-dihexyl (1-hydroxy-3-ethylamino-2,2-dimethylpropyl)phosphonate **IIIc** and 1.06 g (10.5 mmol) of triethylamine in 20 ml of anhydrous ether at room temperature was added dropwise a solution of 1.4 g (10 mmol) of benzoyl chloride **VIa** in 10 ml of diethyl ether. The reaction mixture was heated on a water bath for 5–6 h. The precipitated triethylamine hydrochloride was filtered off and washed with ether (10 ml). Then the solvent and volatile components were removed in a high vacuum. Yield 1.9 g (56%). ^{1}H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 0.8–1.8 m (31H, $2C_5H_{11}$, $NCH_2\underline{Me}$, CMe_2), 3.5 m (4H, $Me\underline{CH_2}N\underline{CH_2}$), 4.1 m (4H, $2OCH_2$), 5.35 d, 5.55 d (1H, PCH, $^2J_{PH}$ 11.5 Hz), 7.4–8.2 m (10H, 2Ph). ^{31}P NMR spectrum: δ_P 25.3 ppm. Found, %: N 2.41; P 4.92. $C_{35}H_{50}NO_6P$. Calculated, %: N 2.29; P 5.06.

***O*,*O*-Dihexyl [-2,2-dimethyl-3-(ethanoylethylamino)-1-acetoxypropyl]phosphonate (XI).** To 1.5 g (4 mmol) of *O*,*O*-dihexyl (1-hydroxy-3-ethylamino-2,2-dimethylpropyl)phosphonate **IIIc** with stirring was added dropwise 1.02 g (10 mmol) of acetic anhydride (**X**). The reaction mixture was heated at a bath temperature of 80–90°C for 8 h. Then the volatiles were removed in a high vacuum. Yield 1.3 g (77.4%). ^{1}H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 0.8–1.8 m (31H, $2C_5H_{11}$, $NCH_2\underline{Me}$, CMe_2), 2.0 s, 2.1 s [6H, $CH_3C(O)$], 3.5 m (4H, $Me\underline{CH_2}N\underline{CH_2}$), 4.1 m (4H, $2OCH_2$), 5.1 d (1H, PCH, $^2J_{PH}$ 11.5 Hz). ^{31}P NMR spectrum: δ_P 24.5 ppm. Found, %: N 3.13; P 6.49. $C_{23}H_{46}NO_6P$. Calculated, %: N 3.02; P 6.68.

REFERENCES

1. Gazizov, M.B., Khairullin, R.A., Bagauva, L.R., Sinyashin, O.G., Gaisin, L.G., Karimova, R.F., and Shaikhutdinov, R.M., *Russ. J. Gen. Chem.*, 2003, vol. 73, no. 9, p. 1579.
2. Dyatlova, N.M., Temnikova, V.Ya., and Popov, K.I., *Kompleksony i kompleksonaty metallov* (Metal Chelators and Complexonates), Moscow: Khimiya, 1988, p. 544.
3. Pudovik, A.N., Gur'yanova, I.V., and Ishmaeva, E.A., *Reaktsii i metody issledovaniya organicheskikh soedinenii*, 1968, vol. 19, p. 11.
4. Kazitsyna,, L.A. and Kupletskaya, I.B., *Primenenie UF, IK- i YaMR-spektroskopii v organicheskoi khimii* (Application of UV, IR, and NMR Spectroscopy in Organic Chemistry), Moscow: Vysshaya Shkola, 1971, p. 264.