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LETTERS TO THE EDITOR

Acetals and *N*,*N*'-Alkylidenebiscarbamates in the Synthesis of *N*-Protected α-Aminophosphinic Acids

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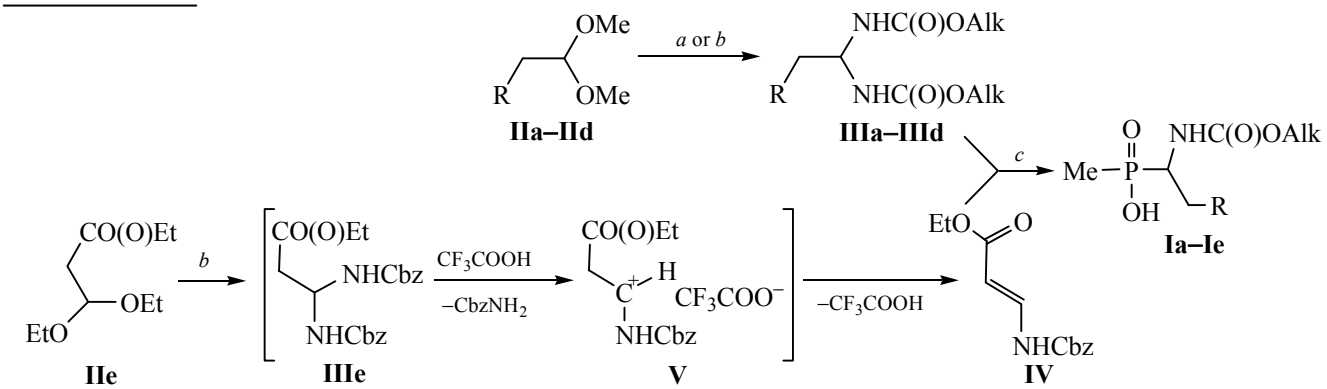
A convenient procedure of the amide version of the Kabachnik–Fields reaction is a promising approach to the synthesis of *N*-protected α-aminophosphorylic compounds. The amidoalkylation originally suggested for the phosphorous chlorides in acetic acid (the Oleksyszyn reaction) [1] was later modified to the amidoalkylation of dialkylphosphites in acetyl chloride or in a mixture of acetic acid and thionyl chloride involving the carbonyl compounds and amides [2].

The study of the amidoalkylation of hydrophosphorylic compounds in acetic anhydride [3] involving amides [4] and alkylcarbamates [5, 6] allowed us developing a new version of the multistep reaction mechanism, comprising the formation of the phosphorus–carbon bond by the Arbuzov reaction via the nucleophilic attack of the phosphorous atom of the POAc-derivative on a positively charged carbon atom of the iminium cation, generated *in situ* in the reaction conditions from starting the hydrophosphorylic compounds and the intermediately forming *N*,*N*'-alkylidenebis(alkylcarbamate) [5, 6]. We failed to introduce some low-stable aldehydes into the three-component amidoalkylation of the hydrophosphorylic compounds. The use of the aldehyde acetals was also unsuccessful or ineffective [6]. In this regard, *N*,*N*'-alkylidenebiscarbamates can be used as the stable synthetic equivalents of the low-stable aldehydes in the reaction with the hydrophosphorylic compounds in accordance with our procedure. The latter includes the generation of an iminium cation and P^{III}-$OC(O)CF_3$-derivative by adding trifluoroacetic anhydride to the mixture of biscarbamate and a PH-component (1:1:1) in an organic solvent [7]. This approach opens the possibility for the synthesis of a number of the low-accessible phosphorylic analogs of amino acids and peptides.

In this work we developed a synthesis of the *N*-alkyloxycarbonyl-α-aminophosphinic acids **I** from acetals **II**, followed by the synthesis of alkylidenebiscarbamates **III**. Their reaction with the hydrophosphorylic component in an organic medium occurs



(*a*) $2EtOC(O)NH_2 + CF_3COOH$; (*b*) $2Cbz\text{-}NH_2 + CF_3COOH$; (*c*) (1) $Me\text{-}PH(O)OH + (CF_3CO)_2O$; (2) H_2O. Alk = Et, Bn; R = H, *i*-Pr, BnO, Ph, C(O)OEt. Cbz – benzyloxycarbonyl.

through the generation *in situ* of the highly reactive intermediates by adding the equivalent of trifluoroacetic anhydride to the reagents mixture to give the mixed P^{IV}-OC(O)CF_3 anhydrides via the Arbuzov reaction. The target compounds **I** were obtained by treating of the obtained anhydrides with water [7].

The acetals of acetic, isovaleric, benzyloxyacetic, phenylacetic, and ethoxycarbonylacetic aldehydes **IIa**–**IIe** were investigated as the starting compounds. They are of interest due to the search for a suitable procedure for constructing the isosteres of serine, phenylalanine, and aspartic fragments to include in future in a molecule of the phosphinic pseudopeptide or phosphorylic analog of the corresponding amino acid. Methylphosphonous acid was studied as a hydrophosphorylic component. We suggest to use the aldehydes generated *in situ* from acetals under the acid catalysis, which is also necessary for the subsequent reaction of aldehydes with alkylcarbamate to give the corresponding *N*,*N*'-alkylidenebiscarbamates **III** [5, 6]. In this case the presence of trifluoroacetic acid provides a satisfactory reaction course. The search for a catalyst as well as the optimal content of CF_3COOH in the reaction mixture was not performed.

As expected, the acetals of acetic, isovaleric, benzyloxyacetic and phenylacetic aldehydes react with 2 equiv of alkylcarbamate in acetic anhydride in the presence of trifluoroacetic acid to give the corresponding *N*,*N*'-alkylidenebiscarbamates **IIIa**–**IIId**. However, the reaction of ethoxycarbonylacetic aldehyde diethylacetal with 2 equiv of benzylcarbamate in the presence of 2.5 equiv of CF_3COOH in acetic anhydride unexpectedly resulted in ethyl β-(*N*-benzyloxycarbonyl)aminoacrylate **IV**. Probably under the acid catalysys the originally formed biscarbamate **IIIe** is converted into *N*-Cbz-β-aminonoacrylate **IV** with the release of the benzylcarbamate molecule and the intermediate formation of *N*-(benzyloxycarbonyl)-iminium cation (salt) **V**. Perhaps the transformation of the latter into the acrylate **IV** is facilitated by means of the electron-withdrawing ethyloxycarbonyl group. The latter result confirms the acid-catalyzed formation of the *N*-alkyloxycarbonyliminium ions from *N*,*N*'-alkylidenebiscarbamates in the multistep amidoalkylation of hydrophosphorylic compounds as we have previously assumed [5–7].

Biscarbamates **IIIa**–**IIId** react with methylphosphonous acid and trifluoroacetic anhydride in toluene or methylene chloride in accordance with the previously suggested procedure for generating the reactive intermediates. The latter react *in situ* via the Arbuzov reaction to form *N*-alkyloxycarbonyl-α-aminophosphinic acids **I** [7]. The reaction of β-*N*-Cbz-aminoacrylate **IV** with methylphosphonous acid and trifluoroacetic anhydride was performed in toluene at room temperature followed by treating with water to give the corresponding *N*-Cbz-protected α-aminophosphinic acid **Ie** in a good yield. This confirm the *in situ* generation of the corresponding bis(trifluoroacetyl)-methylphosphonite and *N*-(benzyloxycarbonyl)iminium ion under the reaction conditions. For comparison, earlier a more reactive analog of β-aminoacrylate, acetamidomethylenemalonate, has been used to obtain the phosphonous and phosphonic analogs of aspartic acid via the addition of the PH-component in the Michael reaction [8].

Thus, in this work we developed a general synthesis of *N*-alkyloxycarbonyl-α-aminophosphinic acids **I** including the compounds containing the structural isostere fragment of serine, phenylalanine, and aspartic acid, which opens the possibility for the synthesis of some low-accessible phosphorylic amino acids analogs and phosphinic pseudopeptides starting from the low-stable aldehydes acetals.

General procedure for the synthesis of *N*-alkyloxycarbonyl-α-aminophosphinic acids (I). *a*. To a solution of alkylcarbamate (5.0 mmol) in acetic anhydride (5 ml) were added with stirring at room temperature trifluoroacetic acid (2.5–6.0 mmol) and dropwise the corresponding acetal (2.5 mmol). The reaction mixture was stirred for 3–10 h. The reaction progress was monitored by TLC. Then the reaction mixture was evaporated in vacuo. The residue was crystallized from diethyl ether or hexane and recrystallized from diethyl ether–alcohol mixture to give the corresponding *N*,*N*'-alkylidenebiscarbamates **IIIa**–**IIId** or ethyl β-(*N*-benzyloxycarbonyl) aminoacrylate **IV**.

b. To a mixture of the appropriate *N*,*N*'-alkylidenebiscarbamate **IIIa**–**IIId** (3 mmol) or β-(*N*-benzyloxycarbonyl) aminoacrylate **IV** (3 mmol) and methylphosphonous acid (3 mmol) in toluene or methylene chloride (3.5 ml) was added dropwise trifluoroacetic anhydride (3 mmol) under stirring at room temperature. The reaction mixture was stirred for 3–48 h at room temperature. The reaction progress was monitored by ^{31}P NMR. After the solvent removal, the semi-crystalline residue was partitioned between the

saturated aqueous sodium hydrocarbonate solution (10 ml) and diethyl ether (10 ml) or a hexane–toluene mixture (10:1). The precipitated alkylcarbamate was filtered off. The aqueous layer was separated and acidified to pH ~1–2, and then extracted with chloroform (3×10 ml). The organic extract was dried over $MgSO_4$ and evaporated in vacuo. The residue was chromatographed on a silica gel ($CHCl_3$:*i*-PrOH:AcOH = 95:4:1). The *N*-protected α-aminophosphinic acids **Ia**–**Ie** were obtained as white crystalline substances. Often the residue was crystallized spontaneously or after the treatment with ether or petroleum ether (40–60), and the target compounds were isolated after further crystallization without using the column chromatography.

1-(Benzyloxycarbonylamino)ethylmethylphosphinic acid (Ia). Yield 53%, mp 119–120°C (mp 121–122°C [6]), R_f ~ 0.15 [$CHCl_3$:$CO(CH_3)_2$ = 4:1]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.33 d.d (3H, $\underline{CH}_3CH$, $^3J_{HH}$ 7.1, $^3J_{PH}$ 15.0 Hz), 1.42 d (3H, CH_3P, $^2J_{PH}$ 15.4 Hz), 3.99 m (1H, CHN), 5.09 m (2H, CH_2Ph), 5.37 d (1H, NH, $^3J_{HH}$ 9.7 Hz), 7.32 m (5H, Ph), 10.56 br.s (1H, POOH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 12.4 d ($^1J_{PC}$ 93.3 Hz), 13.9, 46.0 d ($^1J_{PC}$ 107.6 Hz), 67.2, 128.1, 128.2, 128.5, 136.1, 155.9 d ($^3J_{PC}$ 5.1 Hz). ^{31}P NMR spectrum ($CDCl_3$): δ_P 54.8 ppm.

1-Ethoxycarbonylamino-3-methylbutylmethylphosphinic acid (Ib). Yield 55%, mp 144–146°C, R_f ~0.10 [$CHCl_3$:$CO(CH_3)_2$ = 4:1]. ^{1}H NMR spectrum ($CDCl_3$ + CD_3OD, 5:1), δ, ppm: 0.93 d (3H, CH_3, $^3J_{HH}$ 6.6 Hz), 0.97 d (3H, CH_3, $^3J_{HH}$ 6.6 Hz), 1.26 t (3H, CH_3), 1.38 d (3H, CH_3, $^2J_{PH}$ 14.1 Hz,), 1.40–1.55 m (2H, CH_2), 1.60–1.85 m (1H, CH), 3.82 m (1H, PCH), 4.08 q (2H, CH_2). ^{1}H NMR spectrum (CCl_4 + DMSO-d_6, 4:1), δ, ppm: 0.90 d (3H, CH_3, $^3J_{HH}$ 6.2 Hz), 0.95 d (3H, CH_3, $^3J_{HH}$ 6.5 Hz), 1.25 t (3H, CH_3), 1.3 d (3H, CH_3, $^2J_{PH}$ 14.0 Hz), 1.35–1.6 m (2H, CH_2), 1.6–1.85 m (1H, CH), 3.7 m (1H, CH), 4.0 q (2H, CH_2), 6.9 d (1H, NH, $^3J_{HH}$ 9.3 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 12.5 d ($^1J_{PC}$ 89.6 Hz), 14.7, 20.9, 23.3, 24.1 d ($^3J_{PC}$ 11.7 Hz), 36.0, 49.0 d ($^1J_{PC}$ 107.6 Hz), 59.9, 156.4. ^{31}P NMR spectrum (CCl_4 + DMSO-d_6, 4:1): δ_P 49.0 ppm. ^{31}P NMR spectrum (CCl_4 + CD_3OD, 5:1): δ_P 52.0 ppm. Mass spectrum, *m*/*z* (I_{rel}, %): 238 (37) [*M* + H]$^+$, 158 (100) [$(CH_3)_2CHCH_2C^+HNHC(O)OCH_2CH_3$]. Found, %: C 45.70, 45.66; H 8.45, 8.51; N 5.87, 5.82. $C_9H_{20}NO_4P$. Calculated, %: C 45.57; H 8.50; N 5.90.

1-(Benzyloxycarbonylamino)-2-benzyloxyethylmethylaminophosphinic acid (Ic). Yield 53%, mp 98–99°C, R_f ~0.10 [$CHCl_3$:$CO(CH_3)_2$ = 4:1]. ^{1}H NMR spectrum (CD_3OD + DMSO-d_6, 4:1), δ, ppm: 1.47 d (3H, CH_3P, $^2J_{PH}$ 14.2 Hz), 3.79 d (1H, $OC\underline{H}_2CHP$, $^3J_{PH}$ 10.8 Hz), 3.81 d (1H, $OC\underline{H}_2CHP$, $^3J_{PH}$ 9.3 Hz), 4.17 d. t (1H, CHP, $^2J_{PH}$ 14.2 Hz), 4.55 br.s (2H, $OC\underline{H}_2Ph$), 5.14 br.s [2H, $C(O)OC\underline{H}_2Ph$], 7.34 m (10H, Ph). ^{13}C NMR spectrum (CD_3OD + DMSO-d_6, 4:1), δ_C, ppm: 13.8 d (CH_3P, $^1J_{PC}$ 92.2 Hz), 53.0 d (PCHN, $^1J_{PC}$ 104.7 Hz), 67.7, 69.0 d ($PC\underline{H}_2CH_2$, $^2J_{PC}$ 7.0 Hz), 74.0, 128.8, 128.9, 129.1, 129.5, 129.6, 138.3, 139.4, 158.5 d (C=O, $^3J_{PC}$ 5.0 Hz). ^{31}P NMR spectrum (CD_3OD + DMSO-d_6, 4:1): δ_P 49.0 ppm. Found, %: C 59.31, 59.43; H 6.23, 6.15; P 8.73, 8.67. $C_{18}H_{22}NO_5P$. Calculated, %: C 59.50; H 6.10; P 8.52%.

1-(Ethoxycarbonylamino)-2-phenylethylmethylphosphinic acid (Id). Yield 51%, mp 163–164°C, R_f ~0.15 [$CHCl_3$:$CO(CH_3)_2$ = 5:1]. ^{1}H NMR spectrum ($CDCl_3$ + CD_3OD, 1:2), δ, ppm: 0.98 t (3H, CH_3, $^3J_{HH}$ 7.1 Hz), 1.33 d (3H, CH_3P, $^2J_{PH}$ 13.7 Hz), 2.70 m (1H, $C\underline{H}_2Ph$), 3.16 m (1H, $C\underline{H}_2Ph$), 3.90 q (CH_2O, $^3J_{HH}$ 7.1 Hz), 4.05 m (1H, CHP), 7.19 m (5H, Ph). ^{13}C NMR spectrum (CD_3OD), δ_C, ppm: 12.4 d (CH_3P, $^1J_{PC}$ 90.9 Hz), 14.9 (CH_3), 34.5 ($\underline{C}H_2Ph$), 53.8 d (PCHN, $^1J_{PC}$ 108.6 Hz), 62.1 ($O\underline{C}H_2CH_3$), 127.6 (CH), 129.4 (CH), 130.2 (CH), 139.0 d ($^3J_{PC}$ 13.4 Hz), 158.7 d (C=O, $^3J_{PC}$ 3.3 Hz). ^{31}P NMR spectrum ($CDCl_3$ + + CD_3OD, 1:2), δ_P, ppm: 51.1, 50.0 (15%) [7]. ^{31}P NMR spectrum ($CDCl_3$ + CF_3COOH): δ_P 59.7 ppm. Found, %: C 53.01, 52.93; H 6.93, 6.95; P 11.53, 11.78. $C_{12}H_{18}NO_4P$. Calculated, %: C 53.14; H 6.69; P 11.42.

1-(Benzyloxycarbonylamino)-2-(ethyloxycarbonyl)ethylmethylphosphinic acid (Ie). Yield 68%, mp 97–98°C. R_f ~0.2 [$CHCl_3$:$CO(CH_3)_2$ = 5:1]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.18 t (3H, CH_3, $^3J_{HH}$ 7.0 Hz), 1.48 d (3H, CH_3P, $^2J_{PH}$ 14.0 Hz), 2.66 m [1H, $CH_2C(O)$], 2.77 m [1H, $CH_2C(O)$], 4.11 q (2H, CH_2O, $^3J_{HH}$ 7.0 Hz), 4.39 m (1H, CHP), 5.10 br.s (2H, $PhC\underline{H}_2O$), 5.63 m (1H, NH), 7.32 m (5H, Ph), 8.00 br.s (1H, POOH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 12.8 d (CH_3P, $^1J_{PC}$ 92.6 Hz), 14.0 (CH_3), 33.4 [$\underline{C}H_2C(O)$], 47.6 d (PCHN, $^1J_{PC}$ 110.2 Hz), 61.1 ($CH_3\underline{C}H_2O$), 67.2 ($Ph\underline{C}H_2O$), 128.0 (CH), 128.2 (CH), 128.5 (CH), 136.1, 155.8 (C=O), 170.3 d (C=O, $^3J_{PC}$ 11.7 Hz). ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 53.6, 52.4 (13%) [7]. Found, %: C 50.92, 50.93; H 6.23, 6.32; P 9.52, 9.68. $C_{14}H_{20}NO_6P$. Calculated, %: C 51.07; H 6.12; P 9.41.

***N*,*N*'-Ethylidenebis(benzylcarbamate) (IIIa).** Yield 71%, mp 200–201°C (mp 203–204°C [6]), R_f 0.8 [$CHCl_3$:$CO(CH_3)_2$ = 4:1].

***N*,*N*'-Isoamylidenebis(ethylcarbamate) (IIIb).** Yield 67%, mp 129–130°C, R_f 0.7 [$CHCl_3$:$CO(CH_3)_2$ = 4:1]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.90 d (6H, CH_3, $^3J_{HH}$ 6.6 Hz), 1.21 t (6H, CH_3, $^3J_{HH}$ 7.1 Hz), 1.52–1.81 m (3H, $CHCH_2$), 4.08 q (4H, CH_2O, $^3J_{HH}$ 7.1 Hz), 4.97 br.s (1H, CH), 5.50 br.s (2H, NH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 0.80 d (6H, CH_3, $^3J_{HH}$ 7.0 Hz), 1.10 t (6H, CH_3, $^3J_{HH}$ 7.1 Hz), 1.30–1.55 m (3H, $CHCH_2$), 3.60–4.05 q (4H, CH_2O, $^3J_{HH}$ 7.1 Hz), 5.04 m (1H, CH), 7.17 d (2H, NH, $^3J_{HH}$ 6.6 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 14.6 ($\underline{C}H_3CH_2$), 22.2 ($\underline{C}H_3CH$), 24.1 ($\underline{C}HCH_3$), 43.5 ($\underline{C}H_2CH$), 57.8 ($\underline{C}HN$), 59.5 ($\underline{C}H_2O$), 155.1 (C=O). Mass spectrum, *m*/*z* (I_{rel}, %): 247 (<1) $[M + H]^+$, 189 (100) $[CH^+(NHC(O)\cdot OCH_2CH_3)_2]$, 158 (51) $[(CH_3)_2CHCH_2CH^+NHC(O)\cdot OCH_2CH_3]$. Found, %: C 53.53, 53.44; H 8.89, 8.88; N 11.21, 11.23. $C_{11}H_{22}N_2O_4$. Calculated, %: C 53.64; H 9.00; N 11.37.

***N*,*N*'-2-Benzyloxyethylidenebis(benzylcarbamate) (IIIc).** Yield 53%, mp 111–112°C, R_f 0.6 [$CHCl_3$:$CO(CH_3)_2$ = 4:1]. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.47 d (2H, $O\underline{CH_2}CH$, $^3J_{HH}$ 6.4 Hz), 4.47 br.s (2H, $OC\underline{H_2}Ph$), 5.01 br.s [4H, $C(O)OC\underline{H_2}Ph$], 5.28 t (1H, $C\underline{H}CH_2O$, $^3J_{HH}$ 6.4 Hz), 7.32 m (15H, Ph), 7.68 d (2H, NH, $^3J_{HH}$ 7.3 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 58.8, 65.4, 70.2, 72.0, 127.5, 127.8, 128.2, 128.3, 136.9, 138.2, 155.2 (C=O). Found, %: C 68.94, 69.04; H 5.96, 6.00; N 6.24, 6.33. $C_{25}H_{26}N_2O_5$. Calculated, %: C 69.11; H 6.03; N 6.45.

***N*,*N*'-2-Phenylethylidenebis(ethylcarbamate) (IIId).** Yield 61%, mp 155–156°C, R_f 0.7 [$CHCl_3$:$CO(CH_3)_2$ = 4:1]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.25 t (6H, $2CH_3$), 3.18 m (2H, CH_2Ph), 4.13 q (4H, CH_2O), 5.21 m (1H, CHN), 5.56 m (2H, 2NH), 7.28 m (5H, Ph). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.10 t (6H, CH_3, $^3J_{HH}$ 7.1 Hz), 2.84 d (2H, $\underline{CH_2}CH$, $^3J_{HH}$ 7.1 Hz), 3.91 q (4H, CH_2O, $^3J_{HH}$ 7.1 Hz), 5.10 t (1H, CHN, $^3J_{HH}$ 7.5 Hz), 7.23 m (5H, Ph). 7.47 br. d (2H, 2NH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 14.4, 40.1, 61.0, 126.8, 128.6, 129.3, 136.5, 155.6 (C=O). Found, %: C 60.21, 60.13; H 7.11, 7.17; N 10.15, 10.04. $C_{14}H_{20}N_2O_4$. Calculated, %: C 59.99; H 7.19; N 9.99.

Ethyl β-(*N*-benzyloxycarbonyl)aminoacrylic acid (IV). Yield 47%, mp 115–116°C, R_f ~0.5 [$CHCl_3$:$CO(CH_3)_2$ = 7:1]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.25 t (3H, CH_3, $^3J_{HH}$ 7.1 Hz), 4.16 q (2H, CH_2O, $^3J_{HH}$ 7.1 Hz), 5.19 br.s (2H, $Ph\underline{CH_2}O$), 5.36 d [1H, CHC(O), $^3J_{HH}$ 14.1 Hz], 7.07 d (1H, NH, $^3J_{HH}$ 11.9 Hz), 7.36 m (5H, Ph), 7.80 d.d (1H, $\underline{CH}NH$, $^3J_{HH}$ 14.1, $^3J_{HH}$ 11.9 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 14.2 (CH_3), 60.1 ($CH_3\underline{C}H_2O$), 68.1 ($Ph\underline{C}H_2O$), 100.1 [$C(O)\underline{C}^{\alpha}H$=], 128.4 (CH), 128.6 (CH), 135.1, 139.4 ($NH\underline{C}^{\beta}H$=), 152.9 (C=O), 167.3 (C=O). Found, %: C 62.34, 62.33; H 6.13, 6.20. $C_{13}H_{15}NO_4$. Calculated, %: C 62.64; H 6.07.

The ^{1}H, ^{31}P and ^{13}C NMR spectra were recorded on Bruker DPX-200 and Bruker CXP-300 spectrometers relative to internal TMS and external 85% H_3PO_4. The melting points were measured on a Boetius-PHMK instrument or in an open capillary.

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