

Synthesis of Optically Active Phosphonamino Acids Esters at Microwave Assistance

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Abstract—A preparative synthesis was developed for phosphonamino acids esters proceeding from natural amino acids with the use of microwave assistance. A series of phosphonamino acids esters was prepared from optically active natural α -amino acids.

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Among the enormous multitude of phosphorus compounds containing a P–C bond α -aminophosphonates, analogs of α -amino-carboxylic acids, play a special role and are widely employed as substances endowed with versatile biological activity [1–4]. At present a large number of publications, in particular, reviews, concern the synthesis of this class compounds [4–7]. At the same time the α -aminophosphonates containing fragments of amino acids or peptides (phosphonamino acids, phosphonopeptides) are far less understood. However the well known *N*-(phosphonomethyl)glycine, a strong plant growth regulator extensively used now and commercially available under the trademark *Roundup*, belongs just to this class of compounds [8]. Besides some phosphonamino acids and phosphonopeptides exhibit a strong antibacterial, enzyme-inhibitor and other biological actions and are used as drugs (*Alafosfaline* is an active antibiotic against gram-positive bacteria, *Glu- γ -P* is an inhibitor of glutamine synthetase) [9–12].

The published preparation methods for phosphonamino acids are known to be performed both in a two-component [13] and three-component systems [14, 15]. However the synthesis of these compounds is time-consuming (up to three days). As to the stereoselectivity of the synthesis of organophosphorus compounds of this class, only few examples are known of the preparation of phosphonamino acids esters with a high diastereomer excess [16, 17].

We formerly developed a procedure for the synthesis of α -aminophosphonates based on the reaction of

azomethines with dialkyl phosphites in the presence of Lewis acids (CdI_2) (two-component system) [18] and also on a three-component process carbonyl compound–primary amide–diethyl phosphite under microwave irradiation [19]. It was shown that combining the catalysis with CdI_2 and the microwave assistance we succeeded in a many-fold shortening of the reaction time (from 3–10 h to several minutes, and sometimes, seconds) and in a considerable increase in the yield of the target products [15]. Besides the application of the microwave irradiation made it possible to obtain the α -aminophosphonates from natural compounds, like porphyrins and steroids, that could not be prepared at common heating due to the instability of the initial substances [20, 21].

In extension of these studies we developed a procedure for the synthesis of phosphonamino acids esters in a two-component system azomethine–O,O-diethyl phosphite in the presence of CdI_2 under the microwave radiation.

Initial azomethines **Ia–Ih** were obtained by the reaction of the corresponding carbonyl compounds with the esters of optically active α -amino acids in the presence of K_2CO_3 in benzene at heating to 40–45°C [22].

The reaction of azomethines **Ia–Ih** with diethyl phosphite under the action of the microwave radiation (107 W, 150–160°C) was carried out without solvent, in the presence of 5 mol% of CdI_2 . The reaction progress was monitored by the ^{31}P NMR spectroscopy and by TLC (Silufol, hexane–ethyl acetate, 5:1). Phosphonamino acids esters **IIa–IIh** were obtained in yields from 69 to 86% (see the table).

Compounds **IIa–IIIh** were diastereomers mixtures. The diastereomers ratio was estimated from the ^{31}P NMR spectra. In each of ^{31}P NMR spectra of compounds **IIa–IIIc**, **IIe–IIIh** appeared two signals corresponding to two diastereomers (see the table).

The data of ^1H and ^{13}C NMR spectra confirm the formation of diastereomers mixture in the synthesis of compounds **IIa–IIIh**. Their spectra contained a double set of signals with a close chemical shifts in pairs of signals corresponding to two diastereomers in the ratios indicated in the table. In particular, in each of the ^1H NMR spectra

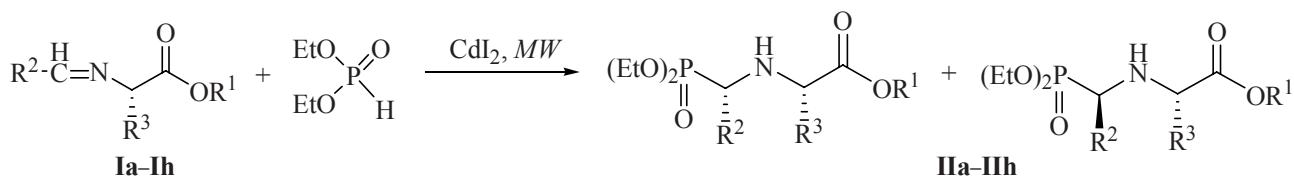
of the obtained phosphonamino acids esters **IIa–IIIh** two clearly resolved doublets of protons of CH groups from the amino acid residues of two diastereomers were observed in the region 2.65–3.63 ppm ($^2J \sim 16$ Hz), in each ^{13}C NMR spectrum appeared two clearly pronounced doublets of the carbon from the same group, and also two doublets corresponding to the carbon atom in the α -position with respect to phosphorus in the region 59–72 ppm, $^2J_{\text{PC}} \sim 150$ Hz.

As seen from the table, the diastereomer excess in most cases was 34–62%. We succeeded in performing

Yields and diastereomer excess (*d.e.*) in reactions of O,O-diethyl phosphite with imines obtained from α -amino acids

Compd. no.	Reaction product	Reaction time, min	Yield, %	Diastereomers ratio (δ_{P} , ppm)	d.e., %
IIa		20	75	29:71 (27.47; 26.20)	42
IIb		30	80	67:33 (22.90; 22.67)	34
IIc		25	86	73:27 (27.68; 27.18)	46
IId		40	81	100 (22.34)	100
IIe		25	73	28:72 (22.50; 22.43)	44
IIf		34	82	27:73 (23.10; 23.00)	46
IIg		42	75	81:19 (23.09; 22.99)	62
IIIh		40	74	76:24(23.17; 23.06)	52

Scheme 1.



$R^1 = \text{Et}$; $R^2 = i\text{-Pr}$, $R^3 = \text{CH}_3$ (**a**); $R^2 = \text{Ph}$, $R^3 = \text{CH}_3$ (**b**); $R^2 = i\text{-Pr}$, $R^3 = \text{CH}_2\text{Ph}$ (**c**); $R^2 = \text{Ph}$, $R^3 = \text{CH}_2\text{Ph}$ (**d**); $R^2 = \text{Ph}$, $R^3 = \text{CH}_2\text{COOEt}$ (**e**); $R^1 = \text{Me}$, $R^2 = \text{Ph}$, $R^3 = i\text{-Pr}$ (**f**); $R^1 = t\text{-Bu}$, $R^2 = i\text{-Pr}$, $R^3 = \text{CH}_2(\text{CH}_2)_3\text{NHC(O)Ph}$ (**g**); $R^2 = i\text{-Pr}$, $R^3 = \text{CH}_2(\text{CH}_2)_3\text{NHC(O)Ph}$ (**h**).

the reaction with a high extent of the diastereomeric purity (*d.e.* 100%) when reacting diethyl phosphite with ethyl *N*-benzylidenephénylalaninate (**Id**). We found that this reaction at the microwave irradiation was completed within 40 min and resulted in a single diastereomer of phosphonamino acid ester (**IIIId**). To prove this fact we additionally registered the ^{31}P NMR spectrum at the maximum peak width 1 Hz. However even in this event we observed in the spectrum a single signal in the region 22.34 ppm.

The formation of a diastereomer excess in reaction of azomethines with *O,O*-diethyl phosphite in the presence of CdI_2 may be rationalized suggesting that the Lewis acid coordinated with imine to give a chelate complex, and presuming that the attack of the phosphite occurred from the side opposite to the complex (Scheme 2). The formation of similar complexes is widely discussed in the literature [7, 16, 23].

As seen from the data obtained the stereochemical result is considerably affected by the nature of substituent R^3 .

EXPERIMENTAL

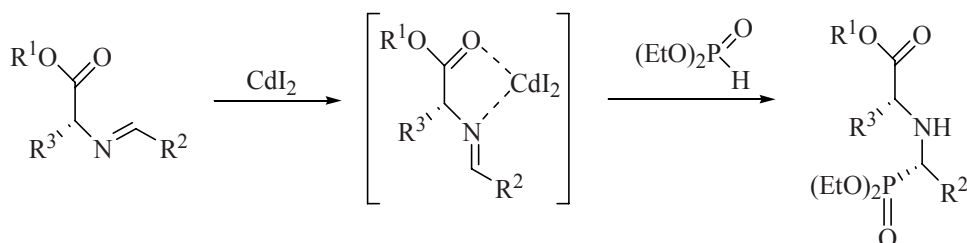
^1H , ^{13}C , and ^{31}P NMR spectra were registered on a spectrometer Bruker Avance-400 (operating frequencies 400, 100.6, and 161.9 MHz respectively) from solutions in CDCl_3 . The chloroform signals (δ_{H} 7.24, δ_{C} 77.10 ppm) served internal references. IR spectra

were recorded on a spectrophotometer UR-20 (690–3600 cm^{-1}). Mass spectra of compounds generated from porphyrins were measured on a Finnigan MAT-LCQ instrument by the method of chemical ionization in electrospray; carrier gas chloroform–acetonitrile. Melting points were measured on an Electrothermal 9100 instrument. The reaction progress was monitored and the purity of the chemical compounds was checked by TLC (Silufol UV-254). In the preparative column chromatography the silica gel Merck (40/60) was used.

Synthesis of phosphonamino acids esters under the conditions of microwave assistance. General procedure. Into an open flat-bottom flask of capacity 25 ml was charged 0.01 mol of *O,O*-diethyl phosphite, 0.0005 mol of cadmium iodide was added, then 0.01 mol of imine **Ia–Ih**. The reaction was carried out under the microwave irradiation (102 W, 155°C) for 20–42 min. The reaction product was isolated by column chromatography on silica gel (eluent hexane–ethyl acetate, 3:2).

Ethyl *N*-[2-methylpropyl-1-(diethoxyphosphoryl)]alaninate (IIa**).** Yield 2.3 g (75%), oily substance (mixture of two diastereomers). ^1H NMR spectrum, δ , ppm: 0.98–1.10 m [12H, $(\text{CH}_3)_2\text{CH}$, for two isomers], 1.20–1.33 m [24H, $\text{P}(\text{OCH}_2\text{CH}_3)_2 + \text{CH}_2\text{CH}_3 + \text{CH}_3$ (alaninate), for two isomers], 1.58–1.62 m [2H, $(\text{CH}_3)_2\text{CH}$, for two isomers], 2.72 d, 2.76 d [1H, CH (alaninate), 2J 16.1 Hz], 3.62–3.65 m (2H, PCHN, for two isomers), 3.99–4.10 m [8H, $\text{P}(\text{OCH}_2\text{CH}_3)_2$, for two

Scheme 2.



isomers], 4.11 q, 4.15 q (2H, OCH₂CH₃, ²J 6.9 Hz). ¹³C NMR spectrum, δ, ppm: 14.02 s, 14.09 s (OCH₂CH₃), 16.37 s, 16.42 s [P(OCH₂CH₃)₂], 18.92 s, 19.03 s [CH₃ (alaninate)], 20.03 s, 20.46 s, 20.33 s, 20.89 s (CH₃CH), 29.61 s, 30.56 s [CH(CH₃)₂], 55.58 d, 56.25 d [CH (alaninate), *J* 6.9 Hz], 59.14 d, 60.90 d (PCHN, ¹J_{CP} 153.5, ¹J_{CP} 150.0 Hz), 60.93 s, 61.02 s (OCH₂CH₃), 62.26 s, 62.33 s [P(OCH₂CH₃)₂], 171.14 s, 171.50 s [C(O)]. ³¹P NMR spectrum, δ, ppm: 27.47, 26.20. Found, %: C 50.31; H 9.05; N 4.45. C₁₃H₂₈NPO₅. Calculated, %: C 50.49; H 9.06; N 4.53.

Ethyl *N*-[(phenyl)(diethoxyphosphoryl)methyl]-alaninate (IIb). Yield 2.7 g (80%), oily substance (mixture of two diastereomers). ¹H NMR spectrum, δ, ppm: 0.87–0.99 m [12H, P(OCH₂CH₃)₂, for two isomers], 3.98–4.05 m (2H, PCHN), 4.08–4.15 m [12H, P(OCH₂CH₃)₂ + OCH₂CH₃, for two isomers], 7.28–7.64 m (10H, 2C₆H₅), 1.24 t, 1.26 t (3H, OCH₂CH₃, ²J 6.8 Hz), 1.36 d, 1.40 d [3H, CH₃ (alaninate), ²J 7.7 Hz], 3.24 d, 3.31 d [1H, CH (alaninate), *J* 16.5 Hz]. ¹³C NMR spectrum, δ, ppm: 13.68 s, 14.16 s (OCH₂CH₃), 18.64 s, 18.86 s [P(OCH₂CH₃)₂], 19.50 s, 20.97 (CH₃, alaninate), 61.02 s, 62.11 s (OCH₂CH₃), 63.07 s, 63.15 s [P(OCH₂CH₃)₂], 64.04 d, 64.32 d [CH (alaninate), *J* 7.1 Hz], 69.71 d, 71.83 d (PCHN, ¹J_{CP} 155.0, ¹J_{CP} 154.1 Hz), 125.19 s, 126.69 s, 128.08 s, 128.09 s, 129.51 s, 129.73 s, 134.45 s, 136.85 s (C₆H₅), 171.27 s, 172.68 s [C(O)]. NMR spectrum ³¹P, δ, ppm: 22.90, 22.67. Found, %: C 55.69; H 7.75; N 4.02. C₁₆H₂₆NPO₅P. Calculated, %: C 55.98; H 7.58; N 4.08.

Ethyl *N*-[2-methylpropyl-1-(diethoxyphosphoryl)]phenylalaninate (IIc). Yield 3.3 g (86%), oily substance (mixture of two diastereomers). ¹H NMR spectrum, δ, ppm: 0.77 d, 0.86 d (3H, CH₃CH, ²J 6.6 Hz), 0.91 d, 0.97 d (3H, CH₃CH, ²J 6.6 Hz), 1.10–1.34 m [18H, P(OCH₂CH₃)₂ + OCH₂CH₃, for two isomers], 2.59–2.70 m [2H, (CH₃)₂CH, for two isomers], 2.81–2.86 m [4H, CH₂ (Ph alaninate), for two isomers], 2.89–2.93 m (2H, PCHN, for two isomers), 3.61 d, 3.63 [1H, CH (Ph alaninate), *J* 15.6 Hz], 3.90–4.07 m [12H, P(OCH₂CH₃)₂ + OCH₂CH₃, for two isomers], 7.13–7.30 m (10H, C₆H₅, for two isomers). ¹³C NMR spectrum (CDCl₃), δ, ppm: 13.43 s, 13.85 s (OCH₂CH₃), 16.16 s, 16.21 s [P(OCH₂CH₃)₂], 20.00 d, 20.12 d (CH₃CH, *J* 25.0, *J* 24.2 Hz), 20.28 d, 20.68 d (CH₃CH, *J* 24.2, *J* 25.0 Hz), 29.06 d, 30.37 d [CH(CH₃)₂, *J* 31.8, *J* 32.0 Hz], 39.76 s [CH₂ (Ph alaninate)], 40.26 s [CH₂ (Ph alaninate)], 59.09 d, 59.49 d (PCHN, ¹J_{CP} 150.7, ¹J_{CP} 149.3 Hz), 60.34 s (OCH₂CH₃), 61.46 d, 61.72 d

[CH (Ph alaninate), *J* 7.3 Hz], 61.81 s [P(OCH₂CH₃)₂], 62.49 s (OCH₂CH₃), 64.03 s [P(OCH₂CH₃)₂], 126.25 s, 126.35 s, 127.95 s, 128.05 s, 129.10 s, 129.21 s, 137.01 s, 137.43 s (C₆H₅); 173.67 s, 173.96 s [C(O)]. ³¹P NMR spectrum, δ, ppm: 27.63, 27.10. Found, %: C 59.32; H 8.45; N 3.34. C₁₉H₃₂NO₅P. Calculated, %: C 59.21; H 8.37; N 3.63.

Ethyl *N*-[(phenyl)(diethoxyphosphoryl)methyl]-phenylalaninate (II d). Yield 3.4 g (81%), oily substance. ¹H NMR spectrum, δ, ppm: 1.16 t [3H, P(OCH₂CH₃)₂, ²J 6.7 Hz], 1.19 t [3H, P(OCH₂CH₃)₂, ²J 6.7 Hz], 1.33 t [3H, OCH₂CH₃, ²J 7.2 Hz], 2.17 br.s (1H, NH), 3.05 d [2H, CH₂ (Ph alaninate), *J* 6.2 Hz], 3.29–3.43 m [4H, P(OCH₂CH₃)₂], 3.56 d [1H, CH (Ph alaninate), *J* 17.0 Hz], 4.12 q (2H, OCH₂CH₃, ²J 6.9 Hz), 4.25 d (1H, PCHN, *J* 19.6 Hz), 7.12–7.37 m (10H, 2C₆H₅). ¹³C NMR spectrum, δ, ppm: 13.84 s (OCH₂CH₃), 16.26 s [P(OCH₂CH₃)₂], 38.48 s [CH₂ (Ph alaninate)], 61.31 s (OCH₂CH₃), 62.49 s [P(OCH₂CH₃)₂], 63.26 d [CH (Ph alaninate), *J* 7.8 Hz], 70.52 d (PCHN, ¹J_{CP} 158.4 Hz), 126.80 s, 127.08 s, 128.37 s, 128.87 s, 129.43 s, 129.65 s, 133.96 s, 136.77 s (2C₆H₅), 172.78 s [C(O)]. ³¹P NMR spectrum, δ, ppm: 22.34, 22.16. Found, %: C 63.06; H 7.21; N 3.42. C₂₂H₃₀NO₅P. Calculated, %: C 63.01; H 7.16; N 3.34.

Diethyl *N*-[(phenyl)(diethoxyphosphoryl)-methyl]aspartate (II e). Yield 3.0 g (73%), oily substance (mixture of two diastereomers). ¹H NMR spectrum, δ, ppm: 1.09 t, 1.13 t [6H, P(OCH₂CH₃)₂, ²J 6.8 Hz], 1.19–1.22 m (6H, OCH₂CH₃, for two diastereomers), 1.27–1.31 m (6H, OCH₂CH₃, for two diastereomers), 2.67–2.74 m (4H, CH₂ for two diastereomers), 3.90–4.02 m [8H, P(OCH₂CH₃)₂, for two diastereomers], 4.10–4.14 m (4H, OCH₂CH₃, for two diastereomers), 4.29–4.33 m (2H, PCHN, for two isomers), 7.26–7.43 m (10H, C₆H₅, for two isomers). ¹³C NMR spectrum, δ, ppm: 13.93 s, 14.11 (OCH₂CH₃), 16.15 s, 16.22 s [P(OCH₂CH₃)₂], 16.38 s, 16.45 s [P(OCH₂CH₃)₂], 37.86 s, 38.42 s (CH₂), 54.76 d, 56.23 d [CH (aspartate), *J* 8.4 Hz], 59.20 d, 59.82 d (PCN, ¹J_{CP} 151.7, ¹J_{CP} 151.4 Hz), 60.60 s, 60.68 s (OCH₂CH₃), 61.04 s, 61.16 s (OCH₂CH₃), 62.89 s, 62.95 s [P(OCH₂CH₃)₂], 63.11 s, 63.18 s [P(OCH₂CH₃)₂], 128.09 s, 128.44 s, 128.65 s, 128.77 s, 128.97 s, 129.04 s, 135.72 s, 135.87 s (C₆H₅), 170.56 s, 170.72 s [C(O)], 172.84 s, 173.15 s [C(O)]. ³¹P NMR spectrum, δ, ppm: 22.50, 22.43. Found, %: C 54.93; H 7.28; N 3.37. C₁₉H₃₀NO₇P. Calculated, %: C 54.99; H 7.58; N 3.51.

Methyl *N*-[(phenyl)(diethoxyphosphoryl)-methyl]valinate (IIf). Yield 2.9 g (82%), oily substance (mixture of two diastereomers). ^1H NMR spectrum, δ , ppm: 0.89–0.94 m [12H, $(\text{CH}_3)_2\text{CH}$, for two isomers], 1.11–1.18 m [12H, $\text{P}(\text{OCH}_2\text{CH}_3)_2$, for two isomers], 3.42 d, 3.38 d [1H, CH (valinate), 2J 17.2 Hz], 3.66 s, 3.69 s (3H, OCH_3), 3.75–3.86 m [8H, $\text{P}(\text{OCH}_2\text{CH}_3)_2$, for two isomers], 4.31 d, 4.35 d (1H, PCHN, J 18.6 Hz), 7.26–7.41 m (10H, C_6H_5 , for two isomers). ^{13}C NMR spectrum, δ , ppm: 16.11 s, 16.24 s [$\text{P}(\text{OCH}_2\text{CH}_3)_2$], 18.30 s, 18.34 s (CH_3CH), 19.15 s, 19.20 s (CH_3CH), 29.18 s, 29.21 s [$\text{CH}(\text{CH}_3)_2$], 50.46 s, 50.51 s (OCH_3), 56.26 d, 57.79 d (PCN, $^1J_{\text{CP}}$ 152.1, $^1J_{\text{CP}}$ 150.9 Hz), 62.91 d, 63.07 d [CH (valinate), J 7.0 Hz], 63.18 s, 63.24 s [$\text{P}(\text{OCH}_2\text{CH}_3)_2$], 127.03 s, 127.15 s, 127.49 s, 127.54 s, 128.10 s, 128.19 s, 137.22 s, 137.91 s (C_6H_5), 176.15 s, 176.23 s [C(O)]. ^{31}P NMR spectrum, δ , ppm: 23.10, 23.00. Found, %: C 57.13; H 7.90; N 3.92. $\text{C}_{17}\text{H}_{28}\text{NO}_5\text{P}$. Calculated, %: C 57.27; H 7.81; N 3.83.

***tert*-Butyl *N*⁶-benzoyl-*N*²-[2-methylpropyl-1-(diethoxyphosphoryl)]lysinate (IIg).** Yield 3.7 g (75%), oily substance (mixture of two diastereomers). ^1H NMR spectrum, δ , ppm: 1.12–1.16 m [12H, $(\text{CH}_3)_2\text{CH}$ for two isomers], 1.21–1.30 m [12H, $\text{P}(\text{OCH}_2\text{CH}_3)_2$, for two diastereomers], 1.39 s, 1.41 s [9H, $\text{C}(\text{CH}_3)_3$], 1.60–1.84 m (12H, 3CH_2 , for two diastereomers), 3.09 m (2H, PCHN, for two diastereomers), 4.00–4.12 m [8H, $\text{P}(\text{OCH}_2\text{CH}_3)_2$, for two isomers], 7.31–7.42 m (10H, C_6H_5 , for two diastereomers). ^{31}P NMR spectrum, δ , ppm: 23.09, 22.99. Found, %: C 60.22; H 8.69; N 5.62. $\text{C}_{25}\text{H}_{43}\text{NO}_6\text{P}$. Calculated, %: C 60.29; H 8.74; N 5.81.

Ethyl *N*⁶-benzoyl-*N*²-[2-methylpropyl-1-(diethoxyphosphoryl)]lysinate (IIh). Yield 3.5 g (74%), oily substance (mixture of two diastereomers). ^1H NMR spectrum, δ , ppm: 1.04–1.11 m [12H, $(\text{CH}_3)_2\text{CH}$, for two isomers], 1.20–1.33 m [12H + 6H, $\text{P}(\text{OCH}_2\text{CH}_3)_2$ + OCH_2CH_3 , for two diastereomers], 1.56–1.72 m (12H, 3CH_2 , for two diastereomers), 3.15 m (2H, PCHN, for two diastereomers), 4.10–4.18 m [8H, $\text{P}(\text{OCH}_2\text{CH}_3)_2$, for two isomers], 7.28–7.44 m (10H, C_6H_5 , for two diastereomers). ^{31}P NMR spectrum, δ , ppm: 23.17, 23.06. Found, %: C 58.60; H 8.28; N 5.94. $\text{C}_{23}\text{H}_{39}\text{NO}_6\text{P}$. Calculated, %: C 58.63; H 8.41; N 5.81.

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