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To the 100th Anniversary of A.N. Pudovik

Phosphorylation of Betti Base with Diethyl Chlorophosphate

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Abstract—Phosphoric acid derivatives containing chiral Betti base fragment were synthesized by reacting racemic and enantiopure *N*-Boc-protected 1-(α-aminobenzyl)-2-naphthols with diethyl chlorophosphate followed by deprotection.

Keywords: Betti base, chiral shift reagent, phosphoric acid derivatives

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Betti base, 1-(α-aminobenzyl)-2-naphthol prepared in the beginning of the last century [1] now gets popular in asymmetric organic synthesis due to the recently elaborated simple and convenient methods of its isolation in the enantiomerically pure forms [2–4]. Simplicity of the preparation of Betti base and its derivatives as enantiopure species allows regarding them among the limited number of the other accessible enantiopure natural and synthetic compounds widely used in organic asymmetric synthesis. However, this simple, versatile and effective chiral scaffold has been rarely used in organophosphorus chemistry [5–7].

Recently we have showed that Betti base could be used as an effective chiral auxiliary in asymmetric synthesis of enantiopure α-aminophosphonic acids prepared easily from Betti base imines by the Pudovik reaction [8, 9]. Here we report on a possibility to use 1-(α-aminobenzyl)-2-naphthol not only as chiral auxiliary, but in asymmetric synthesis of organophosphorus compounds containing chiral Betti base fragment.

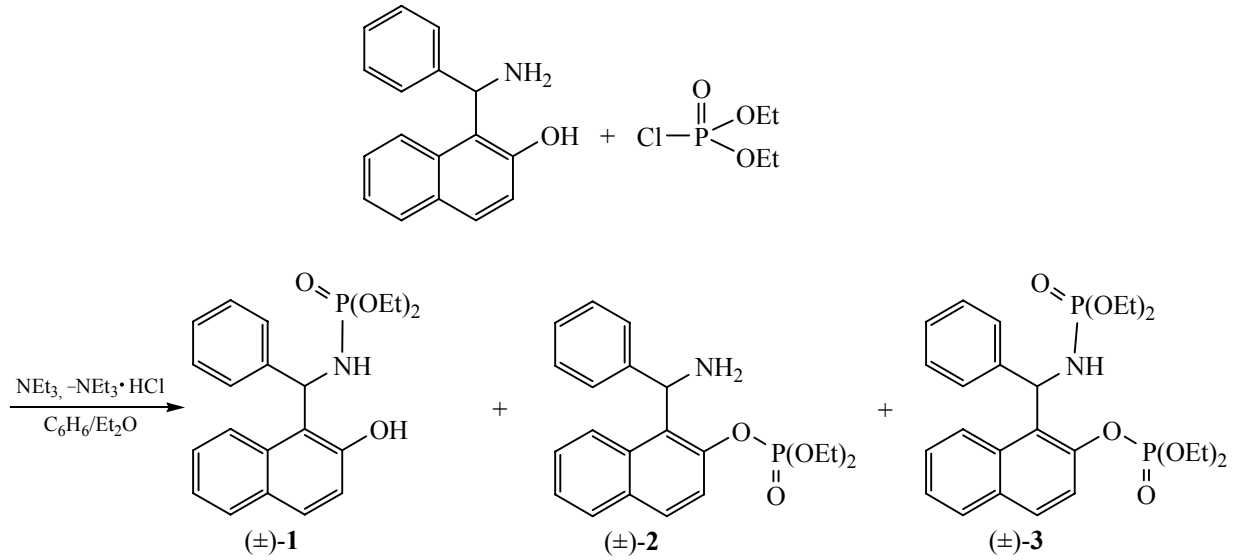
Direct phosphorylation of Betti base presents some difficulties. Thus, 1-(α-aminobenzyl)-2-naphthol possesses two active nucleophilic proton-containing sites, which are able to react with electrophilic phosphorous reagents like phosphorus halides that may vary the reaction pathway.

Indeed, the reaction of unsubstituted racemic Betti base with diethyl chlorophosphate in the presence of triethylamine at 1 : 1 : 1.5 ratio of the starting reagents afforded a mixture of the reaction products as shown by ^{31}P NMR spectroscopy. Thus, in the ^{31}P NMR spectrum four signals appeared corresponding most probably to the products of *N*-phosphorylation (±)-**1** (δ_P = 9.8 ppm), *O*-phosphorylation (±)-**2** (δ_P = –4.5 ppm), and *O*,*N*-phosphorylation of Betti base (±)-**3** (δ_P = –5.6, 9.3 ppm). Addition of an excess of diethyl chlorophosphate and triethylamine to the reaction resulted in complete phosphorylation of the starting Betti base and isolation of (±)-**3** (Scheme 1).

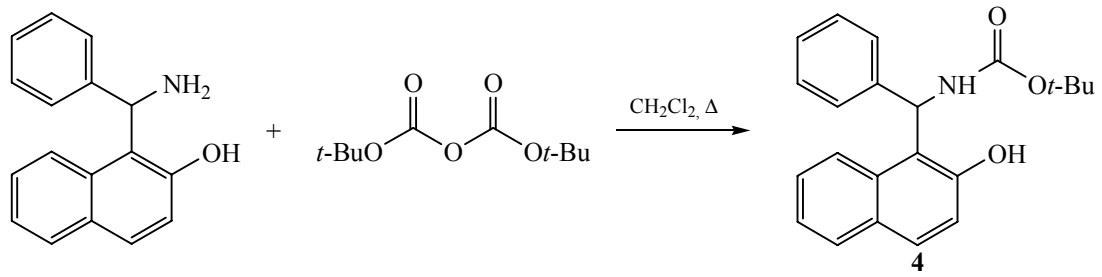
In order to obtain monophosphorylated product, one of the nucleophilic sites of Betti base should be selectively protected. *tert*-Butoxycarbonyl group (Boc) is known to be a convenient protection for amino group [10]. Introduction of the protecting group with subsequent phosphorylation of free naphthol hydroxyl fragment followed by deprotection would lead to the formation of chiral phosphorus-containing amino acids with free primary amino group. Such compounds may be interesting as asymmetric organocatalysts [11].

The analysis of publications revealed that *N*-Boc-protected Betti base has been characterized only as racemic compound. Moreover, it has been prepared not

Scheme 1.



Scheme 2.



by the direct reaction of free base with di-*tert*-butyl dicarbonate, but by reacting of β-naphthol with Boc-benzalimine, which is difficult to obtain [12]. Some researchers tried to prepare enantiopure *N*-Boc-protected Betti base by the same reaction under enantioselective catalysis, but scalemic product was isolated with *ee* 91% [13]. Therefore enantiopure *N*-Boc-protected Betti base has not been described earlier.

Aiming to obtain *N*-Boc-protected Betti bases, we performed the reactions of (±)- and (*S*)-(+)-1-(α-aminobenzyl)-2-naphthols with di-*tert*-butyl dicarbonates. The reactions proceeded under mild conditions with the formation of the target products in 80–86% yield (Scheme 2).

In addition, we elaborated the method of analysis of enantiomeric purity of the prepared compounds by ^{1}H NMR spectroscopy using the (*R*)-(–)-3,5-dinitro-*N*-(1-phenylethyl)benzamide as a chiral shift reagent, which proved to be suitable when analyzing free Betti bases [2]. An addition of the reagent to the solution of compound (±)-**4** in $CDCl_3$ complicated the ^{1}H NMR spectral pattern. The doublet signal of methine proton at the α-carbon of naphthyl fragment is the most informative for the analysis of enantiomeric purity of the product (Fig. 1a). When mixing (*R*)-(–)-3,5-dinitro-*N*-(1-phenylethyl)benzamide with racemic product (±)-**4** at a molar ratio of 1 : 1, the second doublet signal appeared in the same field of the spectrum (Fig. 1b). Complete resolution of the signals succeeded at the molar ratio of the shift reagent and the analyzed compound of 2 : 1 (Fig. 1c). In the case of compound (*S*)-(–)-**4**, under the same conditions the appearance of the second doublet was not observed that evidenced its high enantiomeric purity (*ee* > 98%) (Fig. 2).

Therefore, (*R*)-(–)-3,5-dinitro-*N*-(1-phenylethyl)benzamide was found to be an effective chiral shift reagent allowing estimation of the enantiomeric purity of both Betti base and its *N*-Boc-derivative.

The reaction of compound (±)-**4** with diethyl chlorophosphate led to the formation of the *O*-phosphory-

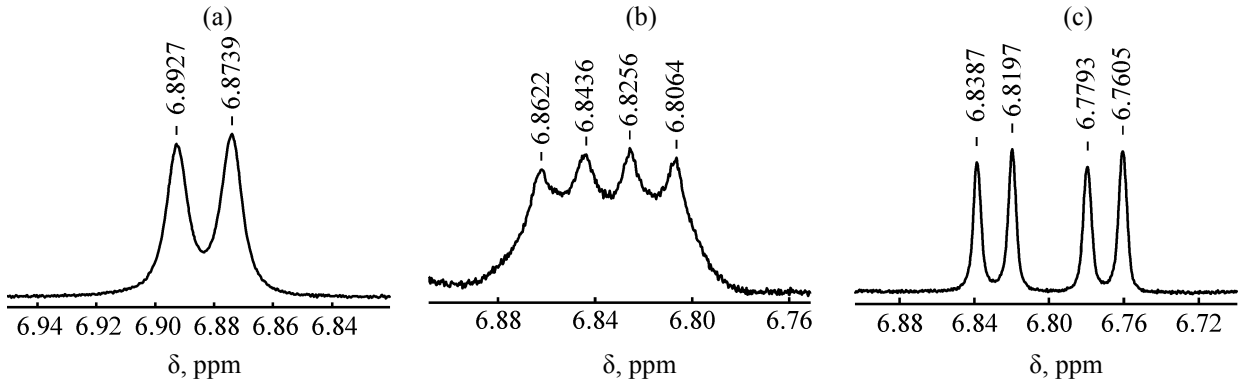


Fig. 1. Fragments of ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of compound (±)-**4** in the absence of the shift reagent (a) and in the presence of equimolar amount (b) and two-fold molar excess of the shift reagent (c).

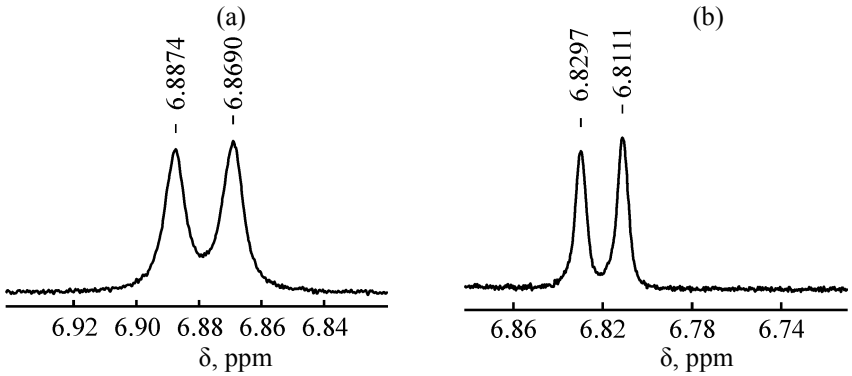
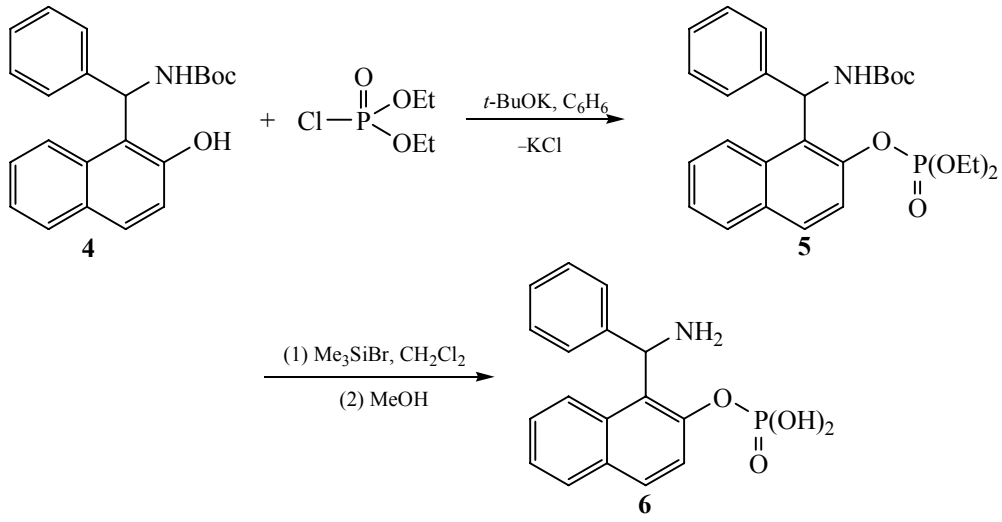


Fig. 2. Fragments of ^{1}H NMR spectrum (500 MHz, $CDCl_3$) of compound (*S*)-(–)-**4** in the absence of the shift reagent (a) and in the presence of two-fold molar excess of the shift reagent (b).

lated product (±)-**5**; the composition and structure of which was established by elemental analysis, IR and NMR spectroscopy data. The *O*-phosphorylation was confirmed by the presence of a doublet signal of the methine proton of the Betti base residue in ^{1}H NMR spectrum of compound (±)-**5**; the signal did not split into doublet of doublets by the phosphorus nuclei that would happen at the *N*-phosphorylation.

Scheme 3.



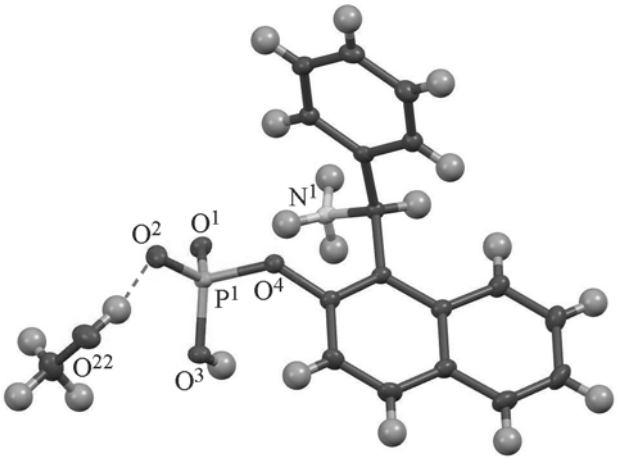


Fig. 3. Crystal structure of (*S*)-(–)-*O*-1-[amino(phenyl)methyl]naphth-2-yl phosphate (*S*)-**6** (hydrogen bond with solvate molecule of methanol is shown as dashed line).

To prepare phosphorylated Betti base derivative containing free amino group it was necessary to deprotect the nitrogen atom in the molecule of **5** and to remove the ethyl group in the phosphoryl fragment. Bromotrimethylsilane is known to be an effective reagent, which allow to carry out both reactions under mild conditions [14]. Indeed, the treatment of compound (±)-**5** with this reagent followed by an addition of methanol led to the formation of acid (±)-**6** (Scheme 3).

The total yield of (±)-**6** was 48%. We succeeded to improve it up to 68% in the case of one-pot synthesis starting from compounds (±)-**4** and (*S*)-**4** without isolation of intermediate **5**.

Like other aminocarboxylic and aminophosphonic acids, compounds (±)-**6** and (*S*)-**6** exist as inner salts (betaines); their structure was confirmed by NMR and IR spectra. Recrystallization of enantiopure betaine (*S*)-**6** from methanol allowed to obtain the solvate crystals suitable for X-ray single-crystal diffraction study.

In the crystal of (*S*)-**6** a complex system of hydrogen bonds was present: intramolecular N–H···O bonds and numerous intermolecular hydrogen bonds with participation of both the target compound and methanol were detected (Fig. 3, Table 1).

In summary, we accomplished the synthesis of *N*-Boc-protected 1-(α-aminobenzyl)-2-naphthols in racemic and enantiopure forms. The method of the optical purity determination of these compounds by ^{1}H NMR spectroscopy with the use of (*R*)-(–)-3,5-dinitro-*N*-(1-phenylethyl)benzamide as a chiral shift reagent was elaborated. Simple and effective approach to synthesis of racemic and enantiopure phosphoric acids containing chiral Betti base fragment with free amino group was developed.

Parameters of hydrogen bonds in the crystal of compound (*S*)-**6**

D–H	*d*(D–H), Å	*d*(H···A), Å	∠DHA, deg	*d*(D···A), Å	A
N^1–H^{1A}	0.883	1.881	174.48	2.762	O^1
N^1–H^{1A}	0.883	2.501	116.75	3.004	O^4
N^1–H^{1B}	0.907	1.836	169.00	2.731	$O^{2'}$ (*x*, *y*–1, *z*)
N^1–H^{1C}	0.947	1.852	158.74	2.757	$O^{1'}$ (–*x*+2, *y*–1/2, –*z*+2)
O^3–H^3	0.856	1.724	165.05	2.561	$O^{22'}$ (*x*+1, *y*–1, *z*)
O^{22}–H^{22}	0.955	1.705	167.65	2.646	$O^{2'}$ (*x*–1, *y*, *z*)

EXPERIMENTAL

NMR spectra were recorded on a Bruker Avance-400 [400.13 (^{1}H) and 161.97 MHz (^{31}P)] and Avance-500 [500.13 (^{1}H) and 202.45 MHz (^{31}P)] instruments relative to the signals of residual protons of deuterated solvents ($CDCl_3$, D_2O) as an internal reference or to the external standard (85% H_3PO_4). IR spectra were recorded on a Bruker Tensor 27 spectrometer from KBr pellets. Optical rotations were measured on a Perkin Elmer (Model 341) polarimeter at 20°C. Melting points were measured on a Boetius melting point microscope.

(±)-1-(α-Aminobenzyl)-2-naphthol was synthesized by hydrolysis of 1,3-diphenylnaphthoxazine [4] prepared by the known procedure [15]. (*S*)-(+)-1-(α-Aminobenzyl)-2-naphthol was prepared by the method described in [3]. Free bases were obtained by treating with sodium carbonate.

(±)-*O*,*O*-Diethyl [2-(diethoxyphosphoryloxy)-naphth-1-yl](phenyl)methylphosphoramidate (±)-3. A solution of triethylamine (0.61 g, 6 mmol) in 1 mL of anhydrous benzene and a solution of diethyl chlorophosphate (0.69 g, 4 mmol) in 4 mL of anhydrous benzene were successively added under argon to a solution of (±)-1-(α-aminobenzyl)-2-naphthol (1 g, 4 mmol) in a mixture of anhydrous benzene (10 mL) and diethyl ether (10 mL). The reaction mixture was stirred at room temperature under argon for 24 h following by sampling for the analysis by ^{31}P NMR spectroscopy. To synthesize of the diphosphorylated Betti base, a solution of triethylamine (1 g, 10 mmol) in 2 mL of anhydrous benzene and a solution of diethyl chlorophosphate (1.385 g, 8 mmol) in 8 mL of anhydrous benzene were added to the reaction mixture. The obtained mixture was stirred at room temperature under argon for 24 h. Then triethylamine hydrochloride was filtered off, and washed with 10 mL of anhydrous diethyl ether. The filtrate was evaporated, and the residue was crystalized from acetonitrile. Yield 0.91 g (43.5%), mp 135–137°C (CH_3CN). IR spectrum, ν, cm^{-1}: 1026 s (P–O–C_{Alk}), 1240 s (P=O), 1599 w (C=C_{Ar}), 1625 w (C=C_{Ar}), 3211 br (NH). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm: 1.00 t (3H, $OCH_2\underline{CH_3}$, $^3J_{HH}$ = 7.0 Hz), 1.17–1.27 m (9H, $OCH_2\underline{CH_3}$), 3.55–3.64 m (1H, $O\underline{CH_2}CH_3$), 3.84–4.09 m (7H, $O\underline{CH_2}CH_3$), 6.48 t (1H, PhCH, $^3J_{PH}$ = 11.3, $^3J_{HH}$ = 11.3 Hz), 7.19–7.90 m (11H, H_{Ar}), 8.12 br.s (1H, NH). ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: –6.97, 8.08. Found, %: C 57.71; H 6.48; N 2.81; P 11.67. $C_{25}H_{33}NO_7P_2$. Calculated, %: C 57.58; H 6.38; N 2.69; P 11.88.

(±)-*tert*-Butyl (2-hydroxynaphth-1-yl)(phenyl)methylcarbamate (±)-4. A solution of di-*tert*-butyl dicarbonate (2.45 g, 11.2 mmol) in 10 mL of methylene chloride was added to a solution of (±)-1-(α-aminobenzyl)-2-naphthol (2 g, 8 mmol) in 25 mL of methylene chloride. The obtained mixture was stirred at room temperature for 1 h followed by refluxing for 5 h. After the solvent removal the residue was crystallized from acetonitrile. Yield 2.25 g (80.3%), mp 217–219°C (CH_3CN) (mp = 218–219.1°C [12]). IR spectrum, ν, cm^{-1}: 1519 s (C=O), 1581 w (C=C_{Ar}), 1627 w (C=C_{Ar}), 1673 s (C=O), 3420 m (NH). ^{1}H NMR spectrum (500 MHz, $CDCl_3$), δ, ppm: 1.50 s [9H, $C(CH_3)_3$], 6.88 d (1H, PhCH, $^3J_{HH}$ = 9.4 Hz), 7.17–7.42 m (9H, H_{Ar}), 7.73–7.82 m (2H, H_{Ar}), 7.84 br.s (1H, NH). Found, %: C 75.40; H 6.53; N 4.22. $C_{22}H_{23}NO_3$. Calculated, %: C 75.62; H 6.63; N 4.01.

(*S*)-(–)-*tert*-Butyl (2-hydroxynaphth-1-yl)(phenyl)-methylcarbamate (*S*)-(–)-4 was prepared similarly from (*S*)-(+)-1-(α-aminobenzyl)-2-naphthol. Yield 2.4 g (85.8%), mp 224–226°C (CH_3CN), $[\alpha]_D^{20}$ = –69 (*c* 0.25, $CHCl_3$). IR spectrum, ν, cm^{-1}: 1515 s (C=O), 1586 w (C=C_{Ar}), 1630 w (C=C_{Ar}), 1671 s (C=O), 3428 m (NH). ^{1}H NMR spectrum (500 MHz, $CDCl_3$), δ, ppm: 1.50 s [9H, $C(CH_3)_3$], 6.88 d (1H, PhCH, $^3J_{HH}$ = 9.2 Hz), 7.16–7.41 m (9H, H_{Ar}), 7.73–7.81 m (2H, H_{Ar}), 7.83 br.s (1H, NH). Found, %: C 75.67; H 6.79; N 4.17. $C_{22}H_{23}NO_3$. Calculated, %: C 75.62; H 6.63; N 4.01.

(±)-*tert*-Butyl [2-(diethoxyphosphoryloxy)naphth-1-yl](phenyl)methylcarbamate (±)-5. Potassium *tert*-butylate (0.42 g, 3.75 mmol) was added to a suspension of compound (±)-**4** (1 g, 2.87 mmol) in 20 mL of anhydrous benzene. The reaction mixture was stirred for 20 min under argon followed by addition of a solution of diethyl chlorophosphate (0.545 g, 3.16 mmol) in 4 mL of anhydrous benzene. The obtained reaction mixture was stirred under argon for 24 h, centrifuged, decanted, and the solvent was distilled off. The residue was dissolved at heating in a mixture of cyclohexane and ethyl acetate (4 : 1) and kept for 2 days at 10°C. The precipitate was filtered off and dried. Yield 0.9 g (64.8%), mp = 131–132°C (cyclohexane–ethyl acetate). IR spectrum, ν, cm^{-1}: 1036 s (P–O–C_{Alk}), 1221 s (P=O), 1527 s (C=O), 1599 w (C=C_{Ar}), 1626 w (C=C_{Ar}), 1703 s (C=O), 3316 br (NH). ^{1}H NMR spectrum (400 MHz, $CDCl_3$), δ, ppm: 1.23 t and 1.28 2t (6H, $OCH_2\underline{CH_3}$, $^3J_{HH}$ = 7.1 Hz), 1.49 s [9H, $C(CH_3)_3$], 3.92–4.11 m (4H, $O\underline{CH_2}CH_3$), 6.27 br.s (1H, PhCH), 7.08–7.90 m (11H, H_{Ar}), 8.22 br.s

(1H, NH). ^{31}P NMR spectrum ($CDCl_3$): δ_P –6.96 ppm. Found, %: C 64.57; H 6.75; N 2.97; P 6.58. $C_{26}H_{32}NO_6P$. Calculated, %: C 64.32; H 6.64; N 2.88; P 6.38.

(±)-*O*-1-[Amino(phenyl)methyl]naphth-2-yl phosphate (±)-6. A mixture of compound (±)-**5** (0.5 g, 1.03 mmol) and bromotrimethylsilane (0.788 g, 5.15 mmol) in 5 mL of methylene chloride was stirred at room temperature under argon for 24 h. The solvent was distilled off, and methanol (5 mL) was added. The obtained mixture was stirred for 1 h. The precipitate was filtered off, washed with 2 mL of methanol, and dried. Yield 0.28 g (75.3%), solvate with methanol, mp = 191–192°C (CH_3OH). IR spectrum, ν, cm^{-1}: 1231 s (P=O), 1597 w (C=C_{Ar}), 1624 w (C=C_{Ar}), 2850 br (NH_3^+). ^{1}H NMR spectrum (500 MHz, D_2O, K_2CO_3), δ, ppm: 3.33 s (3H, $\underline{CH_3}OH$), 6.28 s (1H, PhCH), 7.21–7.90 m (14H, H_{Ar}, NH_3). ^{31}P NMR spectrum (D_2O, K_2CO_3): δ_P 0.07 ppm. Found, %: C 59.99; H 5.66; N 3.63; P 8.38. $C_{17}H_{16}NO_4P{\cdot}CH_4O$. Calculated, %: C 59.83; H 5.58; N 3.88; P 8.57.

The total yield of compound (±)-**6** after two steps was 47.9%. To improve the yield of the target product the synthesis was repeated with the same loading in the first step, but without isolation of intermediately formed carbamate (±)-**5**. A solution of bromotrimethylsilane (2.19 g, 14.3 mmol) in 10 mL of methylene chloride was immediately added to a viscous substance obtained from the reaction of compound (±)-**4** with diethyl chlorophosphate. The reaction mixture was stirred at room temperature for 24 h under argon. After the solvent removal methanol (10 mL) was added, and the mixture was stirred for 1 h. The precipitate was filtered off, washed with methanol (5 mL), and dried. Yield of compound (±)-**6** is 0.7 g (67.7%).

(*S*)-(–)-*O*-1-[Amino(phenyl)methyl]naphth-2-yl phosphate (*S*)-(-)-6 was prepared similarly from compound (*S*)-(–)-**4** without isolation of the intermediate product. Yield 0.72 g (69.6%), mp = 189–191°C (CH_3OH), $[\alpha]_D^{20}$ = –134 (*c* 0.1, DMSO), –109 [*c* 0.5, H_2O + K_2CO_3, potassium carbonate was added in three folder excess regarding to phosphate (*S*)-(–)-**6**]. IR spectrum, ν, cm^{-1}: 1232 s (P=O), 1597 w (C=C_{Ar}), 1624 w (C=C_{Ar}), 2824 br (NH_3^+). ^{1}H NMR spectrum (500 MHz, D_2O, K_2CO_3), δ, ppm: 3.32 s (3H, $\underline{CH_3}OH$), 6.26 s (1H, PhCH), 7.21–7.92 m (14H, H_{Ar}, NH_3). ^{31}P NMR spectrum (D_2O, K_2CO_3): δ_P 0.08 ppm. Found, %: C 59.96; H 5.58; N 3.62; P 8.48. $C_{17}H_{16}NO_4P{\cdot}CH_3OH$. Calculated, %: C 59.83; H 5.58; N 3.88; P 8.57.

X-Ray diffraction study of the crystals of compound (*S*)-6 was done on a Bruker SMART Apex II diffractometer (graphite monochromator, MoK_α-irradiation, λ = 0.71073 Å) at 150(2) K. Empirical absorption correction was carried out using a SADABS program [16]. The structures were solved by the direct method applying SHELXS software [17]. Non-hydrogen atoms were refined isotropically followed by anisotropic approximation using SHELXS program package [17]. Hydrogen atoms were placed into the calculated positions and refined in a *riding* model. The hydrogen atoms of hydroxyl and ammonium groups were revealed from the difference Fourier synthesis, their positions were refined isotropically in the final step of the refinement. All calculations were performed with the help of APEX2 program [18].

Crystals of compound (*S*)-**6** are monoclinic, $C_{17}H_{16}NO_4P{\cdot}CH_4O$, M = 361.32, space group $P2_1$, a = 10.560(1), b = 5.997(1), c = 13.558(2) Å, β = 97.656(2)°, V = 850.9(1) Å^3, Z = 2, d_{calc} = 1.410 g/cm^3, μ = 0.191 mm^{-1}, θ_{max} = 29°; 10819 reflections were collected, 4347 from them were independent (R_{int} = 0.028) and 4103 were observed with [$I > 2\sigma(I)$], 262 parameters of refinement, R_1 = 0.031, wR_2 = 0.079 [$I > 2\sigma(I)$], residual electron density 0.379/–0.210 e/Å^3, GoF = 1.030.

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