Heteroatom Chemistry
Volume 11, Number 2, 2000

C_3-Symmetric Trialkyl Phosphites as Starting Compounds of Asymmetric Synthesis

Oleg I. Kolodiazhnyi, Sergey Sheiko, and Evgeny V. Grishkun

Institute of Bioorganic Chemistry, National Academy of Sciences of Ukraine, Murmanskaya Street, 1, Kiev, 253094, UKRAINE E-mail: oik@biophacs.kiev.ua

Received 15 July 1999

ABSTRACT: *Chiral C_3-symmetric trialkyl phosphites, derivatives, of (−)-(1R,2S,5R)-menthol, and (−)-di-O-isopropylidene-1,2:5,6-α-D-glucofuranose, have been studied as starting reagents for the preparation of chiral organophosphorus compounds. The reactions involve induction at the α-carbon atom of substituted α-alkylphosphonates. The stereoselectivity of the reaction depends on the structure of the starting compounds and the reaction conditions. The configurations of the alkylphosphonates were defined by means of NMR spectroscopy and by transformation into corresponding alkylphosphonic acids.* © 2000 John Wiley & Sons, Inc. Heteroatom Chem 11:138–143, 2000

INTRODUCTION

Chiral C_N symmetric compounds of phosphorus have attracted interest during the last few years as initial compounds of asymmetric synthesis, both as ligands of transition-metal complexes and as analytical derivatizing reagents [1–3].

The symmetric chiral diesters of certain phosphoric acids—diglucofuranosylphosphite, dibornylphosphite, and dimenthylphosphite—have recently been proposed by us to be suitable initial compounds for the synthesis of optically active organophosphorus compounds such as (α-hydroxyalkylphosphonates and α-aminoalkylphosphonic acids) [4, 5]. In the present article, we report that C_3 symmetric trialkyl phosphites, containing a chiral secondary alkoxyl group, can serve as new starting compounds for the asymmetric synthesis of α- and β-substituted phosphonates that have practical importance as pharmaceutical preparations, bioregulators, and agrochemicals [6–12]. Alkoxy groups of trialkyl phosphites are malleable; therefore, these compounds can be used in various reactions for the synthesis of optically active organophosphorus compounds

RESULTS AND DISCUSSION

Chiral phosphites **2a,b** have been synthesized by reaction of optically active secondary alcohols **1,** (−)-(1*R,* 2*S,* 5*R*)-menthol **1a** or (−)-di-*O*-isopropylidene-1,2:5,6-α-D-glucofuranose **1b,** with phosphorus trichloride and triethylamine (as an acceptor of hydrogen chloride) (Scheme 1).

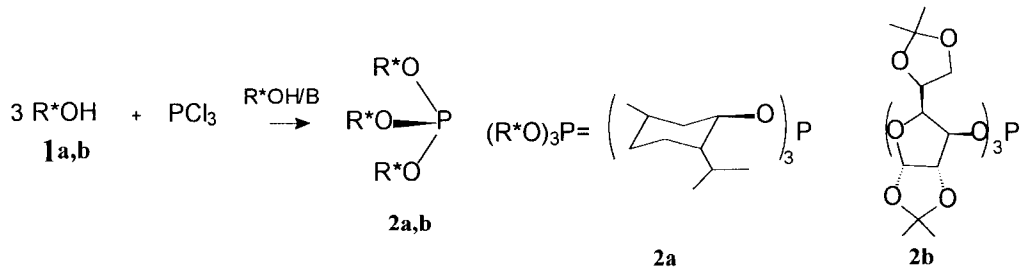
Observance of the optimum reaction conditions allowed us to obtain spectroscopically pure phosphites **2,** which can be used for further reactions without additional purification. Oxidizable triglucofuranosyl phosphite **2b** has been purified by column chromatography with oxygenless silica gel under an inert gas. The structure and purity of compounds **2** have been studied by NMR spectroscopy, TLC, and HPLC. Chiral, nonracemic triesters of phosphoric acid **2** possess an axis of symmetry passing through the phosphorus atom and are typical C_3 symmetric compounds. The computer mod-

Dedicated to Prof. Alfred Schmidpeter on the occasion of his 70th birthday.

Correspondence to: Oleg I. Kolodiazhnyi.

Contract Grant Sponsor: INTAS-International Association.
Contract Grant Number: Project INTAS 97-2120.

© 2000 John Wiley & Sons, Inc.



R*O = (*1R,2S,5R*)-menthyl (**a**), (-)-di-O-isopropylidene-1,2:5,6-α–*D*-glucofuranos-2-yl (**b**),

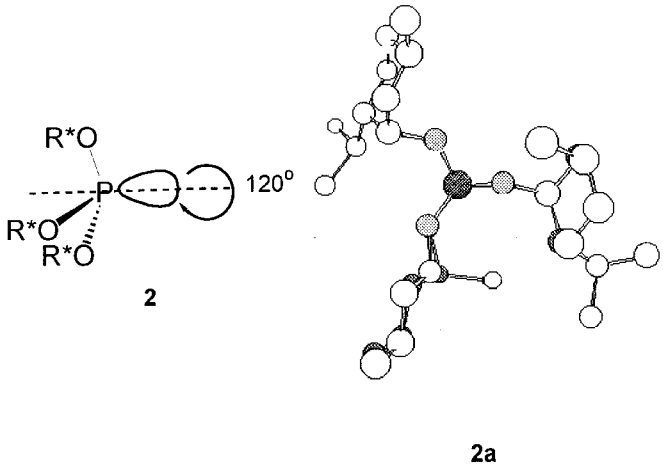
SCHEME 1

eling by means of the MM_1 semi-empirical quantum-chemical program has confirmed the high C_3 symmetry of compounds **2** (Scheme 2).

Addition of an aldehyde to a sterically hindered phosphoric acid triester **2** (R*O = menthyl or di-*O*-isopropylidene-1,2:5,6-α-D-glucofuranos-2-yl) proceeds in the presence of trimethylchlorosilane or boron trifluoride etherate catalysts. The study of the reaction mixtures by ^{31}P-{^{1}H} NMR spectroscopy has shown that the reaction proceeds stereoselectively to result in the formation of α-hydroxyphosphonates in high yields (Scheme 3), which can be isolated as stereochemically pure crystalline compounds. α-Hydroxyalkylphosphonates can easily be obtained in ca. 98–100% stereochemical purity by means of one or two crystallizations from acetonitrile or from hexane. The stereoselectivity of the reaction depends on the structure of the starting compounds and the reaction conditions.

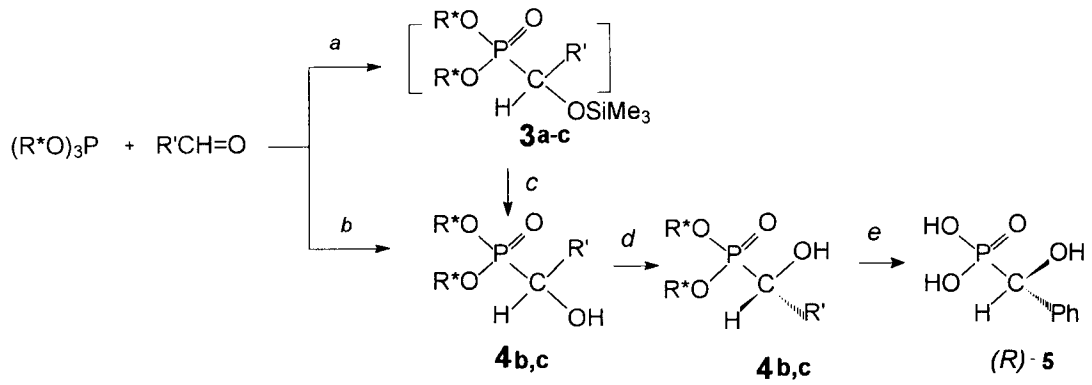
The analysis of reaction mixtures by means of ^{31}P-{^{1}H} NMR spectroscopy showed that the compound **4b** is formed with low diastereomeric excess (de), 35%, whereas the compound **4a** is prepared almost stereochemically pure, de ca. 95%.

Low temperature slows the reaction but increases the stereochemical yield. For example, the reaction of trimenthyl phosphite with anisaldehyde in the presence of boron trifluoride etherate proceeds at 65°C practically instantaneously, accompanied by heat evolution, to give a diastereomeric mixture in a ratio of 6:4 (de 20%). At room temperature, the reaction also proceeds rapidly accompanied by heat evolution and gives the product with low stereoselectivity (de 30%). However, at −20°C the reaction proceeds over a period of 24 hours to give the α-hydroxyalkylphosphonate with a diastereomeric excess of ca. 50%. The reaction of triesters with an aromatic aldehyde in the presence of chlorotrimethylsilane proceeds with heat evolution at room temperature or with weak cooling. The reaction gives silyl esters that have been characterized by NMR



SCHEME 2

spectroscopy, without isolation in the pure state, because they undergo easy hydrolysis with the formation of hydroxyalkylphosphonates in 85–90% yield and a diastereomeric ratio of 12:1 (**4a**) and 3:1 (**4c**). The stereoselectivity of the reaction in the presence of boron trifluoride diethyl etherate is higher than that in the presence of chlorotrimethylsilane. Subsequent crystallization of the obtained mixtures from hexane or acetonitrile gives α-hydroxyalkylphosphonates with ca. 100% stereochemical purity. The structures of compounds **4** have been confirmed by ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy. The diastereoisomer ratio and stereochemical purity of the compounds have been determined by means of HPLC and ^{31}P-{^{1}H} NMR spectroscopy. The hydrolysis of the dimenthyl ether **4b** by heating it in a water-dioxane solution of 6N hydrochloric acid leads to the formation of α-hydroxybenzylphosphonic acid **5**, which has been isolated as the crystalline dicyclohexylammonium salt. The (*R*) configuration of **5** has been defined by comparison of its optical rotation with the data described earlier by Spilling [13] for



a = Me_3SiCl; b = BF_3 Et_2O; c = H_2O; d = crystallization ; e = H_2/Pd-C;
R*= di-O-isopropylidene-1,2:5,6-α–*D*-glucofuranos-2-yl, R= Ph (**4a**); R*=*(1R,2S,5R)*-menth-2-yl, R=Ph (**4b**); R*=*(1R,2S,5R)*-menth-2-yl, R*=*(1R,2S,5R)*-menth-2-yl, R=C_6H_4OMe-4 (**4c**);

SCHEME 3

the dextrorotatory dicyclohexylammonium salt of (*R*)-α-hydroxybenzylphosphonic acid.

The reaction of the trimenthyl phosphite **2** with benzalimines (Schiff bases) proceeds in the presence of chlorotrimethylsilane or boron trifluoride etherate at room temperature or with mild heating (60–80°C). The reaction results in diesters of α-aminoalkylphosphonic acids in high yields and with moderate stereoselectivity: de = 50%. The crystallization of the aminoalkylphosphonates **6** from hexane or aqueous acetone easily gives stereochemically pure dimenthyl(*N*-benzyl)-α-aminobenzylphosphonate **6**. The hydrolysis of diester **6** with hydrochloric acid in dioxane by heating to 70–80°C over a period of 24 to 48 hours leads to the formation of the optically active (−)-*N*-benzyl-α-aminobenzylphosphonic acid **7**, which has been isolated as a crystalline hydrochloride salt. Catalytic hydrogenolysis of this compound in the presence of palladium gives (+)-(R)-α-aminobenzylphosphonic acid **8** with ca. 95% enantiomeric excess (ee). Racemic **8** has been described previously [14].

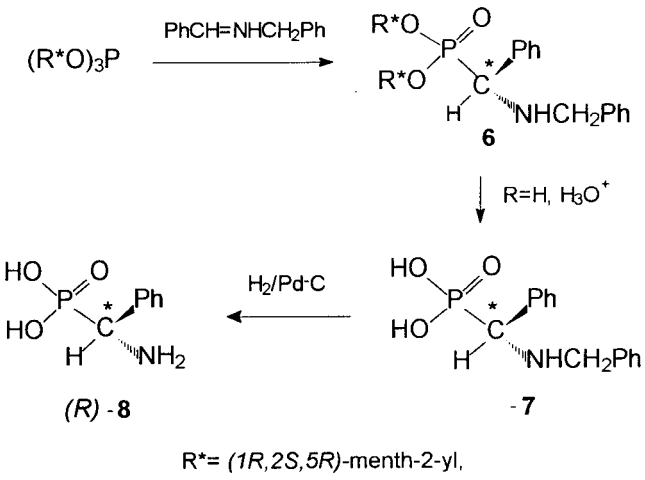
The acid **8** has been obtained as the hydrochloride salt **8a** and the disodium salt **8b**. It is interesting that **8a** and **8b** possess opposite signs of optical rotation (Scheme 4).

8a: $[\alpha]_D$ − 15.5 in water solution.

8b: $[\alpha]_D$ + 17.5 in 0.1 N NaOH.

Earlier, Kafarski et al. have described (+)-(*R*) and (−)-(*S*)-α-aminobenzylphosphonic acids [15], the optical rotation, spectroscopic properties and physical constants of which correspond to the compound **8b**, prepared by us.

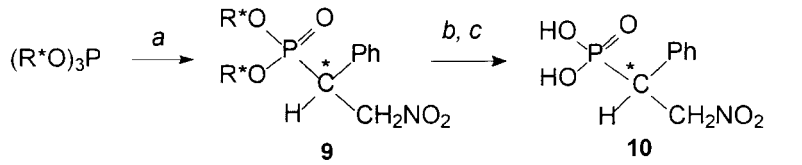
The reaction of the trimenthyl phosphite with β-nitrostyrene in the presence of acid catalysts also proceeds with moderate stereoselectivity to give a mixture of two diastereomers in the ratio of 3:1. Crystallization of the obtained diastereomeric mixture allows us to easily obtain the stereochemically pure major diastereomer of the dimenthyl ester of 1-phenyl-2-nitroethylphosphonic acid as a crystalline compound. The hydrolysis of the diester **9** with hydrochloric acid in aqueous acetic acid during 120 hours results in the homochiral optically active β-nitroalkylphosphonic acid **10** [16].



R*= *(1R,2S,5R)*-menth-2-yl,

SCHEME 4

Thus, preparatively accessible chiral phosphites **2** can be successfully used as initial compounds for asymmetric synthesis of organophosphorus compounds, including the preparation of enantiomerically pure derivatives of α- and β-substituted alkylphosphonic acids. Further studies of these



R* = *(1R,2S,5R)*-menth-2-yl, *a* = $PhCH{=}CHNO_2$, *b* = crystallization; *c*= HCl/HAc/H_2O

SCHEME 5

compounds as starting compounds for the asymmetric synthesis of other optically active α- and β-substituted organophosphorus compounds is now in progress.

EXPERIMENTAL

^1H, ^{13}C, and ^{31}P NMR spectra were taken with Varian VXR-300 (300 MHz) and Bruker WP-200 (200 MHz) instruments in solutions of chloroform-D or benzene-D_6 with the internal standard TMS (^1H, ^{13}C), and also the external standard—85% phosphoric acid (^{31}P). HPLC separations were carried out on the LKB instrument (Sweden) and Milikhrom-1A (Russia) with a Silasorb DEA column. Optical rotations of compounds have been measured by means of the Perkin-Elmer Model 241 spectropolarimeter. The solvents have been carefully purified and by standard methods. Optically active reagents, (−) menthol and (−)-di-*O*-isopropylidene-1,2:5,6-α-D-glucofuranose have been obtained from Fluka and Lancaster.

Tris[(1R, 2S, 5R)-menth-2-yl] Phosphite (**2a**)

To a solution of phosphorus trichloride (2.74 g, 0.02 mol) in 30 mL of toluene a solution of (−)-menthol (9.4 g, 0.06 mol) and 10 mL of triethylamine in 50 mL toluene were added with stirring, dropwise, and with cooling to −20° C. When warmed to ambient temperature, the reaction mixture was stirred for 2 hours. The precipitate of triethylamine hydrochloride was filtered off and the solvent was removed under vacuum. The residue was spectroscopically pure trimenthyl phosphite, which can be used for the various chemical transformations described herein.

NMR spectra (δ, $CDCl_3$): δ_p 148.0 (q, $^3J_{PH}$ 8.0 Hz).

Mass-spectrum: *m/z* 497 ($M^+ + 1$).

Tris(*di-O-isopropylidene-1,2:5,6-α-D-glucofuranos-2-yl*) *Phosphite* (**2b**)

A solution of phosphorus trichloride (2.74 g, 0.20 mol) in 30 mL of toluene was added dropwise to a solution of di-*O*-isopropylidene-1,2:5,6-α-D-glucofuranose (14.4 g, 0.6 mol) and triethylamine (10 mL) in 50 mL of toluene, with stirring and cooling to −20° C. Then the reaction mixture was stirred for 2 hours at +20°C. The triethylamine hydrochloride was filtered off, and the solvent removed under reduced pressure. The residue was purified by flash chromatography (silica gel and 1:1 mixture of ethyl acetate-hexane mixture as eluent., R_f 0.61)

NMR spectra (δ, ppm, $CDCl_3$): δ_P 144.5 (q, $^3J_{PH}$ 8.0 Hz)

Mass-spectrum: m/z 809 (M^+).

Calcd. for $C_{36}H_{57}O_{18}P$: P, 3.83. Found: P, 3.70.

(−)-*[(1R,2S,5R)-menth-2-yl] 1-hydroxybenzylphosphonate* (**4b**)

Benzaldehyde (0.55 g (0.05 mol) and 1.5 ml of trimethylchlorosilane were added to trimenthyl phosphite **2a** (2.5 g, 0.05 mol) with stirring and at 0° C. The mixture was stirred for 1 hour at 0° C, then warmed to ambient temperature and left for 1 to 2 hours. The ^{31}P NMR spectrum showed the presence of only two signals at δ_P 20.36 and 20.08 ppm in the ratio of 3:1. The solvent was removed under reduced pressure. The residue was mixed with silica gel that was then washed with a 1:1 mixture of ethyl acetate-hexane. The solvent was removed under vacuum, the residue dissolved in hexane and the solution placed in a refrigerator. After 2 days a crystalline product was obtained. Yield: 60%, m.p. 139° C, $[\alpha]_D^{20}$ −88.9 (c 1, toluene).

NMR spectra ($CDCl_3$): δ_H 0.7–1.0 (m, CH_3); 1.1–1.23 (m, CH_2 + CH); 3.7 (br, OH); 4.2 (dt, OCH, J_{HH} 2.3, J_{HH} 4.1); 4.92 (d, J_{HP} 11, CHP); 7.2–7.3 (m, C_6H_5); 7.3–7.5 (m, C_6H_5). d_C: 127.99 (d, *J* 2.5); 127.8 (d, J_{CP} 2.5); 127.3 (d, C_6H_5, J_{CP} 9.6); 71.6 (d, PC, J_{CP} 160); 48.6 (d, J_{CP}14, CHO[a]); 48.5 (d, J_{CP} 13.2, CHO[b]); 45.66,s; 42.53,s; 34,s; 31.5,s; 25.31,s; 22.7,s; 21.97,s; 21.13,s; 21.03,s; 15,74, 15.60.

δ_P 23.71.

Mass-spectrum: *m/z* 464 (M^+).

Calcd. for $C_{27}H_{45}O_4P$: P, 6.67. Found: P, 6.55.

(−)-*[(1R,2S,5R)-menth-2-yl] 1-(4-methoxyphenyl)-1-hydroxymethylphosphonate* (**4c**)

Method a. The compound **4c** was prepared similarly to **4b** from trimenthyl phosphite (2.5 g, 0.05 mol), 4-methoxybenzaldehyde (0.55 g, 0.05 mol) and 1.5 mL trimethylchlorosilane. The ^{31}P NMR spectrum revealed the presence of two diastereomers δ_P 18.17; 17.83 in a 3:1 ratio. The solvent was removed under vacuum, and the residue was purified by flash chromatography (SiO_2, 1:1 ethyl acetate-hexane as eluent. The solvent was removed under vacuum and the residue was recrystallized from hexane at 0°C over 2 days.

Method b. The trimenthyl phosphite **2a** (2.5 g, 0.05 mol) and anisic aldehyde (0.68 g 0.05 mol) was dissolved in 2 mL toluene at 0° C. The reaction mixture was cooled to −20°C, the boron trifluoride etherate (0.1–0.2 mL) was added, and the reaction mixture was left overnight at this temperature. Then, the mixture was warmed to ambient temperature and left for 3 to 4 hours at that temperature to complete the reaction. The reaction mixture was washed with water, and the desired product was extracted with methylene chloride. The solvent was removed under reduced pressure and the residue was recrystallized from acetonitrile.

Yield 60%, de 50%, m.p. 150°C, $[\alpha]_D^{20}$ − 77.1 (c 0.1, toluene).

NMR spectra ($CDCl_3$): δ_H 0.7–1.0 (m,CH_3); 1.1–1.23 (m, CH_2 + CH); 3.1 (bs, OH); 3.78 (s, CH_3); 4.18 (m, OCH,); 4.9 (d, CHP, J_{HP} 11); 6.74 (d); 6.89 (d, C_6H_4). δ_P 20.09 (major); 19.98 (minor).

(*R*)-*1-Phenyl(hydroxymethyl)phosphonic Acid* (**5**)

Method a. The solution of dimenthyl 1-phenylhydroxymethylphosphonate **4b** (1 g) in 50 mL of dioxane was placed into a flask and 25 mL of 6 N hydrochloric acid was added. Then the reaction mixture was left for 3 to 4 days at 80°C, the course of hydrolysis being monitored by ^{31}P NMR spectroscopy. When the reaction had been completed, the solvent was evaporated, and the residue was dissolved in alcohol and excess cyclohexylamine was added (ca. 1.5 mL). The precipitate of the dicyclohexylammonium salt of (*R*)-(+)-1-phenyl(hydroxymethyl)phosphonic acid **5** was filtered off. The yield was 70%, m.p. 226° C, $[\alpha]_D^{20}$ + 14.0 (c = 1, 50% aqueous MeOH) that corresponds to the (*R*)-configuration of the acid **5**. The dicyclohexylammonium salt of (−)-(*S*)-phenyl(hydroxymethyl)phosphonic acid has been described.

Method b. Tris(1.2:5.6-di-*O*-Isopropylidene-α-D-glucofuranos-2-yl) phosphite **2b** (2.8 g, 0.005 mol), and benzaldehyde (0.55 g, 0.005 mol) were placed into a flask, and 1.5 mL of chlorotrimethylsilane was added at 0°C. The reaction mixture was warmed to ambient temperature, and the mixture was left for 1–2 hours. The ^{31}P NMR-{^1H} spectrum showed the presence of only two signals, d_P 23.02 and 22.86 ppm in the ratio of 10:1, belonging to *bis*(1.2:5.6-di-*O*-isopropylidene-α-D-glucofuranos-2-yl) 1-phenylhydroxymethylphosphonate **4a.** The solvent was removed under reduced pressure, and the residue was passed through a short column of silica gel with elution by a mixture of ethyl acetate-hexane in a 1:1 ratio. The solvent was removed under vacuum, and the residue was hydrolyzed with a solution of hydrochloric acid in dioxane as previously described. The yield of 1-phenyl(hydroxymethyl)phosphonic acid was 60%.

[(1R, 2S, 5R)-Menth-2-yl]1-phenyl(benzylamine)methylphosphonate (**6**)

Triimenthyl phosphite **2a** (3.5 g, 0.1 mol) maintained at 0°C was added to benzylbenzaldimine (1.8 g, 0.10 mol), and the reaction mixture was left for 12 hours at 60–80°C. ^{31}P NMR spectroscopy revealed the presence of two signals at δ_P 21.91 and 21.67 in the of ratio 3:1. The reaction mixture was separated by flash chromatography [SiO_2, mixture of hexane-ethylacetate 2:1 (300 mL) and 1:1 (300 mL) as eluent]. The solvent was removed under vacuum to afford the crystalline product in the residue, which was recrystallized from acetonitrile or from hexane to afford stereochemically pure **6**. Yield, 60%. m.p. 86–87° C. $[\alpha]_D^{20}$ − 57.9 (c 1, toluene).

NMR spectra ($CDCl_3$): δ_H 0.5 (s, CH_3); 0.6–0.95 (m, CH_3); 1.1–2.2 (m, CH_2 + CH); 3.55 (d, $PhCH^a$, J_{HH} 12 Hz); 3.75 (d, $PhCH^b$, J_{HH} 12 Hz); 3.98 (d, CHP, J_{HP} 20.6 Hz); 4.43 (m, NH); 7.2–7.4 (m, C_6H_5).

NMR spectra ($CDCl_3$): δ_P 21.95.

Calcd. for $C_{34}H_{52}NO_3P$: P, 5.59. Found: P, 5.32.

1-phenyl-1-(N-benzyl)-aminomethylphosphonic acid (**7**)

To a solution of dimenthyl 1-phenyl-1-(*N*-benzylamino)-methylphosphonate **6** (1 g) in 60 mL of dioxane, hydrochloric acid (25 mL 6 N) was added, and the reaction mixture was left for 2 to 3 days at 80°C, the course of hydrolysis being monitored by

means of ^{31}P NMR spectroscopy. When the reaction had been completed, the solvent was removed under reduced pressure to result in 1-phenyl-1-(N-benzylamin)methylphosphonic acid, which was recrystallized from a mixture of water and alcohol.

m.p. 219°C, $[\alpha]_D$ + 29.9 (c 1, DMSO).

NMR spectra (DMSO) δ_H 7.35 (m, 10H, C_6H_5); 3.74–4.07, m (3H, NCH + NCH_2); 3.48 s (NH^{2+}). Racemic 1-phenyl-1-(N-benzyl)-aminomethylphosphonic acid has been described [14]

(*R*)*1-phenyl-1-aminomemylphosphonic acid* (**8**)

The hydrochloride of 1-phenyl-1-(*N*-benzylamino)-methylphosphonic acid (156 mg) was dissolved in 7 mL of water and Pd/C was added. Gaseous hydrogen was passed through the mixture at +20°C to the end of reaction. The catalyst was filtered off and the solvent was evaporated. (R)=(+)1-phenyl-1-aminomethylphosphonic acid **8** was obtained. Yield, 70%; m.p. 226° C; $[\alpha]_D^{20}$ + 15.5 (c 0.32, in a 1N NaOH solution), corresponding to the earlier described (+)-(R)-1-phenyl-1-aminomethylphosphonic acid: $[\alpha]_{578}^{20}$ = +19 (c 1, in 1N NaOH solution) [15].

NMR spectra (D_2O): δ_H 7.28 (s, 5H, C_6H_5); 4.25 (d J_{HP} 17 Hz, 1H, PCH).

Di[(1R,2S,5R)-Menth-2-yl] 1-phenyl-2-nitroethylphosphonate (**9**)

A solution of trimenthyl phosphite (1 g) and β-nitrostyrene (0.3 g) in 10 mL of benzene was placed into a flask and a solution of dimethylaniline hydrochloride (0.35 g) in acetonitrile was added. The reaction mixture was left overnight at ambient temperature. The solvent was removed under vacuum, and the residue was purified by flash-chromatography with ethyl acetate-hexane as eluent and the product recrystallized from aqueous acetone. Yield: 50%, m.p. 140°C.

$[\alpha]_D$ −67.86 (c 1.3, C_6H_6)

NMR spectra (δ; *J*, Hz, $CDCl_3$): δ_H 0.7–1.0 (m, CH_3); 1.1–1.23 (m, CH_2 + CH); 3.45 (dt, J_{HP} 8, J_{HH} 8, PCH); 4.1 (dt, OCH, J_{HH} 2.3, J_{HH} 4.1); 4.93 (dd, CHP, J_{HP} 7, J_{HH} 8); 7.3 (m, C_6H_5).

NMR ^{31}P (C_6D_6): δ_P 19.85.

ACKNOWLEDGMENTS

We thank professor Vladimir Kibirev for his assistance in carrying out the spectropolarimetric analyses and for his useful discussions.

REFERENCES

[1] Kolodiazhnyi, O. I. Tetrahedron: Asymmetry 1998, 9, 1279.
[2] Kolodiazhnyi, O. I.; New Achievements in Asymmetric Synthesis of Organophosphorus Compounds. In Advances of Asymmetric Synthesis. Hassner, A., Ed.; JAI Press Inc. Stamford-London, 1998, Vol. 3, 273–357.
[3] Whitessell, J. K. Chem Rev 1989, 89, 1581.
[4] (a) Kolodiazhnyi, O. I.; Grishkun, E. V.; Sheiko, S.; Demchuk, O.; Thoennessen, H.; Jones, P.; Schmutzler, R. Tetrahedron: Asymmetry 1998, 9, 1645; (b) Kolodiazhnyi, O. I.; Grishkun, E. V. Tetrahedron: Asymmetry 1996, 7, 967.
[5] Kolodiazhnyi, O. I.; Demchuk, O.; Gershkovitch, A. Tetrahedron: Asymmetry 1999, 10, 1729–1732.
[6] Valentine, D., Jr. Asymmetric Synthesis. Morrison, J. D., Scott, J. W., Eds.; Academic Press: New York, 1984; Vol. 4, pp 267–312.
[7] Tasz, M. K.; Gamliel, A.; Rodriguez, O. P.; Lane, T. M.; Cremer, S. E.; Bennett, D. W. J Org Chem 1995, 60, 6281.
[8] Blazis, V. J.; Koeller, K. J.; Spilling, S. D. Tetrahedron: Asymmetry 1994, 5, 499.
[9] Rath, P.; Spilling, C. D. Tetrahedron Lett 1994, 35, 227.
[10] Sum, V.; Kee, T. P. J Chem Soc Perkin Trans I 1993, 1778.
[11] Patel, D. V.; Rielly-Gauvin, D. E. Tetrahedron Lett 1990, 31, 5587.
[12] Denmark, S. E.; Chatani, N.; Pansare, S. V. Tetrahedron 1992, 48, 2191.
[13] Blazis, V. J.; Koeller, K. J.; Spilling, S. D. J Org Chem 1995, 60, 931.
[14] Tyka, B. Tetrahedron Lett 1970, 677.
[15] Kafarski, P.; Lejczak, B.; Szewczyk, J. Can J Chem 1983, 61, 2425.
[16] Kolodiazhnyi, O. I.; Sheiko, O. Zh Obshch Khim (in press).