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> SHORT COMMUNICATIONS

Synthesis of Phospholipids on the Basis of 2,2,5,5-Tetrakis(hydroxymethyl)cyclopentanol

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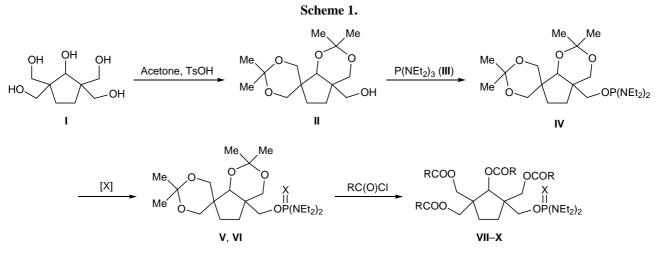
While continuing our studies in the field of synthesis of new lipid structures modified at the alcohol fragment [1], we have prepared for the first time phospholipid derivatives on the basis of 2,2,5,5-tetra-kis(hydroxymethyl)cyclopentanol (I). Compound **I** was treated with acetone in acid medium to obtain diisopropylidene derivative **II** in 68% yield, $n_D^{20} = 1.4792$, R_f 0.40 [chloroform–methanol, 10:1 (A), Silufol UV-254]. Found, %: C 62.83; H 9.06. C₁₅H₂₆O₅. Calculated, %: C 62.91; H 9.15.

Compound **II** was then phosphorylated with hexaethylphosphorous triamide (**III**) at 90–100°C with simultaneous removal of liberated diethylamine by distillation under slightly reduced pressure (380 mm). Crude phosphorodiamidite **IV** [δ_P 134.7 ppm; R_f 0.58, hexane–dioxane, 10:1 (B)] was converted into the corresponding phosphorus(V) derivatives **V** and **VI** by reaction with elemental sulfur or selenium (Scheme 1). Products **V** and **VI** were purified by column chromatography on silica gel using benzene as eluent. **Thiophosphate (V).** Yield 65%, R_f 0.4 (B), $n_D^{20} = 1.5062$, δ_P 79.1 ppm. Found, %: C 55.91; H 9.09; N 5.80; P 6.25. C₂₃H₄₅N₂O₅PS. Calculated, %: C 56.07; H 9.21; N 5.69; P 6.29.

Selenophosphate (VI). Yield 69%, $R_f 0.4$ (B), $n_D^{20} = 1.5204$, $\delta_P 80.4$ ppm (${}^1J_{P,Se} = 845$ Hz). Found, %: C 51.03; H 8.33; N 5.29; P 5.65. C₂₃H₄₅N₂O₅PSe. Calculated, %: C 51.20; H 8.41; N 5.19; P 5.74.

Compounds V and VI were subjected to direct acylation with palmitoyl and stearoyl chlorides at 20°C, following the procedure described in [2]. The resulting tetra-*O*-acyl derivatives VII–X were isolated in up to 52% yield by column chromatography on silica gel using hexane as eluent.

2-Palmitoyloxy-1,1,3-tris(palmitoyloxymethyl)-3-(tetraethyldiaminophosphinothioyloxy)cyclopentane (VII). mp 38–40°C, R_f 0.5 (B). Found, %: C 71.02; H 11.43; N 2.09; P 2.19. $C_{81}H_{157}N_2O_9PS$. Calculated, %: C 71.21; H 11.58; N 2.05; P 2.27.



V, **VII**, **IX**, X = S; **VI**, **VIII**, **X**, X = Se; **VII**, **VIII**, $R = C_{15}H_{31}$; **IX**, **X**, $R = C_{17}H_{35}$.

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2-Palmitoyloxy-1,1,3-tris(palmitoyloxymethyl)-3-(tetraethyldiaminophosphinoselenoyloxy)cyclopentane (VIII). mp 44–46°C, R_f 0.5 (B). Found, %: C 68.66; H 11.05; N 2.07; P 2.11. $C_{81}H_{157}N_2O_9PSe$. Calculated, %: C 68.85; H 11.20; N 1.98; P 2.19.

3-Tetraethyldiaminophosphinothioyloxy-2stearoyloxy-1,1,3-tris(stearoyloxymethyl)cyclopentane (IX). mp 46–47°C, R_f 0.5 (B). Found, %: C 72.13; H 11.69; N 2.01; P 1.98. $C_{89}H_{173}N_2O_9PS$. Calculated, %: C 72.31; H 11.79; N 1.89; P 2.10.

3-Tetraethyldiaminophosphinoselenoyloxy-2stearoyloxy-1,1,3-tris(stearoyloxymethyl)cyclopentane (X). mp 57–58°C, *R*_f 0.5 (B). Found, %: C 69.88; H 11.28; N 1.98; P 1.96. $C_{89}H_{173}N_2O_9PSe$. Calculated, %: C 70.08; H 11.43; N 1.84; P 2.03.

The ¹H NMR spectra of **II** and **V**–**X** were recorded in CDCl₃ (c = 0.5 M) on a Bruker WM-250 spectrometer (250 MHz). The ³¹P NMR spectra of **IV–X** were measured in benzene (c = 1 M) on a Bruker WP-80SY instrument (32.4 MHz) using 85% H₃PO₄ as external reference.

REFERENCES

- 1. Savin, G.A., Kamneva, E.A., and Nifant'ev, E.E., *Russ. J. Org. Chem.*, 2003, vol. 39, p. 1048.
- 2. Nifant'ev, E.E. and Predvoditelev, D.A., Usp. Khim., 1997, vol. 66, p. 47.