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Synthesis and X-ray structure of bis-[$Re_2(CO)_9$] complex of 3,3′,4,4′-tetramethyl-1,1′-diphosphaferrocene functionalised complex as a potential radiopharmaceutical and metallocarbonyl marker

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Abstract

3,3′,4,4′-Tetramethyl-1,1′-diphosphaferrocene reacts with two mole equivalents of [$Re_2(CO)_{10}$]/Me_3NO to afford bis-[$Re_2(CO)_9$] complex **4**. The X-ray structure of this complex revealed equatorial Re–P bonds. The same reaction with a functionalised derivative of 3,3′4,4′-tetramethyl-1,1′-diphosphaferrocene **1b** afforded *N*-succinimidyl ester **2**, potential reagent for introduction of the large amount of radioactive rhenium into target-specific biomolecules.

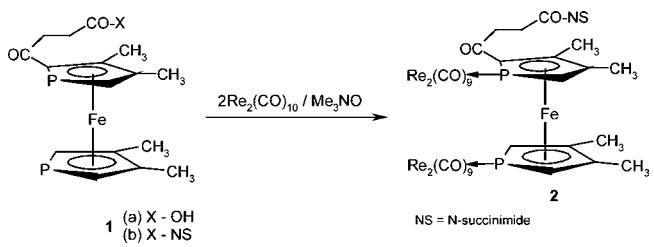
1. Introduction

Labelling of target-specific biomolecules with low-valent *organometallic* complexes of ^{99m}Tc and $^{186/188}Re$ has recently attracted considerable attention as a novel method of preparation of radiopharmaceuticals [1]. Within this context, fast (to avoid a significant loss of radioactivity of these relatively short-lived radioisotopes) and efficient methods of the transformation of $[M(VII)O_4]^-$ (M = Tc, Re), into low oxidation state organometallics such as $[M_2(CO)_{10}]$, $[M(H_2O)_3(CO)_3]^+$ or $[CpM(CO)_3]$ and of the attachment of them to biomolecules have been elaborated.

We have earlier reported [2] that the derivative of 1,1′-diphoshaferrocene, 4-oxo-4-(3,3′,4,4′-tetramethyl-1,1′-diphosphaferrocen-2-yl)butanoic acid (**1a**) (Scheme 1), is a potential carrier of metal moieties to biomolecules since such moieties can be bound to the phosphorus atoms and the carboxylic acid function can be used to attach the complex to a biomolecule (via amido bonds).

We thought that it would be of interest to elaborate a fast and efficient method of binding to the corresponding *N*-succinimidyl ester (**1b**) two [$Re_2(CO)_9$] moieties, as the expected product **2**, containing four rhenium atom per molecule could be an useful carrier for radioactive rhenium, introducing a large amount the radioisotope at a slight chemical modification of the biomolecule. In our approach, the source of the [$Re_2(CO)_9$] moieties is [$Re_2(CO)_{10}$], readily accessible from [ReO_4^-] [3].

We have developed this method using more readily available parent 3,3′,4,4′-tetramethyl-1,1′-diphosphaferrocene (**3**) [4] as a model compound (Scheme 2). The structure of the bis-[$Re_2(CO)_9$] complex **4** was studied by X-ray crystallography.



1 (a) X - OH (b) X - NS

2

Scheme 1.

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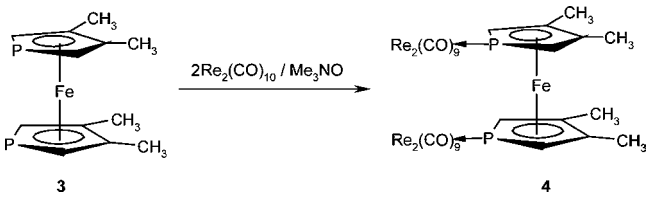


Scheme 2.

Table 1
Crystal data and structure refinement details

Chemical formula	$C_{30}H_{16}FeO_{18}P_2Re_4$
Formula weight	1527.02
Temperature (K)	293(2)
Crystal system	Monoclinic
Space group	$C2/c$
a (Å)	18.456(2)
b (Å)	16.359(2)
c (Å)	13.593(1)
β (°)	100.55(1)
V (Å^3)	4034.7(7)
Z	4
Linear absorption coefficient (μ) (mm^{-1})	(Mo–K_α) 12.46
Number of reflections collected	4986
Number of reflections with $F_o > 4\sigma(F_o)$	3875
Number of independent reflections	4657 ($R_{int} = 0.0215$)
Refinement method	Full-matrix least-squares on F^2
H atoms	Individual isotropic refinement
R indices (all data)	
R_1	0.0463
wR_2	0.0954

2. Results and discussion

Bis-[$Mn_2(CO)_9$] complex of **3** was prepared by Mathey et al. in reaction with 2 mole equivalent of [$Mn_2(CO)_{10}$] in the presence of an excess of Me_3NO [5]. We have found that this reaction in the case of rhenium always gave mixtures of mono- and bis-complexes. However, a slight modification of the procedure afforded **4** in 80% yield. It consisted in the use of THF as a solvent and a concentration of the solution of [$Re_2(CO)_{10}$] treated with Me_3NO to around a half of its initial value before adding **3** (we think that in this way the volatile by-product, Me_3N, is removed from the solution). The structure of **4** was confirmed by analytical and spectral data as well as by X-ray crystallography.

2.1. X ray crystal structure of 4

X-ray quality crystals of **4** were grown from layered dichloromethane-hexane. Crystal data and structure refinement details are displayed in Table 1.

An ORTEP of the molecule **4** is given in Fig. 1a. Fig. 1b gives a top view of the phospholyl rings, showing a nearly eclipsed conformation of the metallocene moiety and a C_2 symmetry of the whole molecule. The selected bond lengths and angles are given in Table 2.

An interesting feature of the investigated structure are two equatorial Re–P bonds. This contrasts with the axial Re–P bonds observed in X-ray structures of a series of complexes [$Re_2(CO)_9(PR_3)$] and [$Re_2(CO)_8(PR_3)_2$], where PR_3 is PH_3, PMe_2Ph, $(Ph)_2P$(ferrocen) [6–8] (an exception is [$Re_2(CO)_9(PPh_2H)$] having equatorial Re–P bond [9]. However, bis-equatorial binding has been observed for the relatively small ('needlelike') isocyanide ligands in [$Re_2(CO)_8(CN–Bu^t)_2$] [10] and in [$Re_2(CO)_8(THF)_2$] [11]. The equatorial binding of **3** to [$Re_2(CO)_9$] may suggest relatively small steric requirements of the heteroferrocene ligand. It is also worth noting that Mathey and coworkers [5] suggested on the basis of IR data axial structure for Mn analog of **4**.

In **4** the two halves of the [$Re_2(CO)_9P$] fragments are staggered with respect to each other with the torsion angles OC–Re–Re–CO(P) from 37.1(3)° to −52.6(2)°. The presence of the P of the phospholyl ring on the equatorial position precludes the ideal staggered form. The axial Re–CO bond distance (average value 1.931(6) Å) is shorter than the average equatorial Re–CO bond distance (average value 1.996(4) Å) and exceeds the Re–C10 (CO *trans* to the P atom) (1.948(7) Å). The shortening of the Re–CO (axial) bond distances relative to their equatorial counterparts has been observed in the structures of [$Re_2(CO)_{10}$] [9] and [$Re_2(CO)_9(PR_3)$] and can be ascribed [12] to the competition for d_π electron density between mutually *trans* pairs of equatorial carbonyls. The shorter Re–C10 bond distance reflects the greater π-acceptor ability of the CO group relative to the phospholyl group.

The comparison of the average bond lengths in the sole known equatorial complex [$Re_2(CO)_9(PPh_2H)$] [9] and in **4** shows shortening in **4** of the Re–P bond (2.443(3) and 2.406(2) Å, respectively), elongation of the Re–Re bond (3.0526(7) and 3.0685(4) Å, respectively), and practically identical Re–C_{eq} (1.998(6) and 1.992(4) Å) Re–C_{eq} (trans to P atom) (1.947(14) and 1.948(7) Å) and Re–C_{ax} (1.924(14) and 1.931(6) Å) bond lengths. In the axial [$Re_2(CO)_9(PR_3)$] and [$Re_2(CO)_8(PR_3)_2$] [6–8] the observed average bond lengths Re–Re (3.0464(6) Å) and Re–P (2.375(3) Å) are a little shorter, than in the cited above equatorial complexes. In **4**, similarly as in [$Re_2(CO)_{10}$] [9] and in other its substituted complexes, the CO ligands bend in towards the metal–metal bond. The observed in **4** Re1–Re2–C_{eq} average angle is 86.3(2)°, C_{ax}–Re–C_{eq}, 93.6(2)°. The P atom is not distorted from the Re–CO equatorial plane (Re2–Re1–P1 angle is 90.90(4)° and $C8_{ax}$–Re1–P1 is 90.3(2)°).

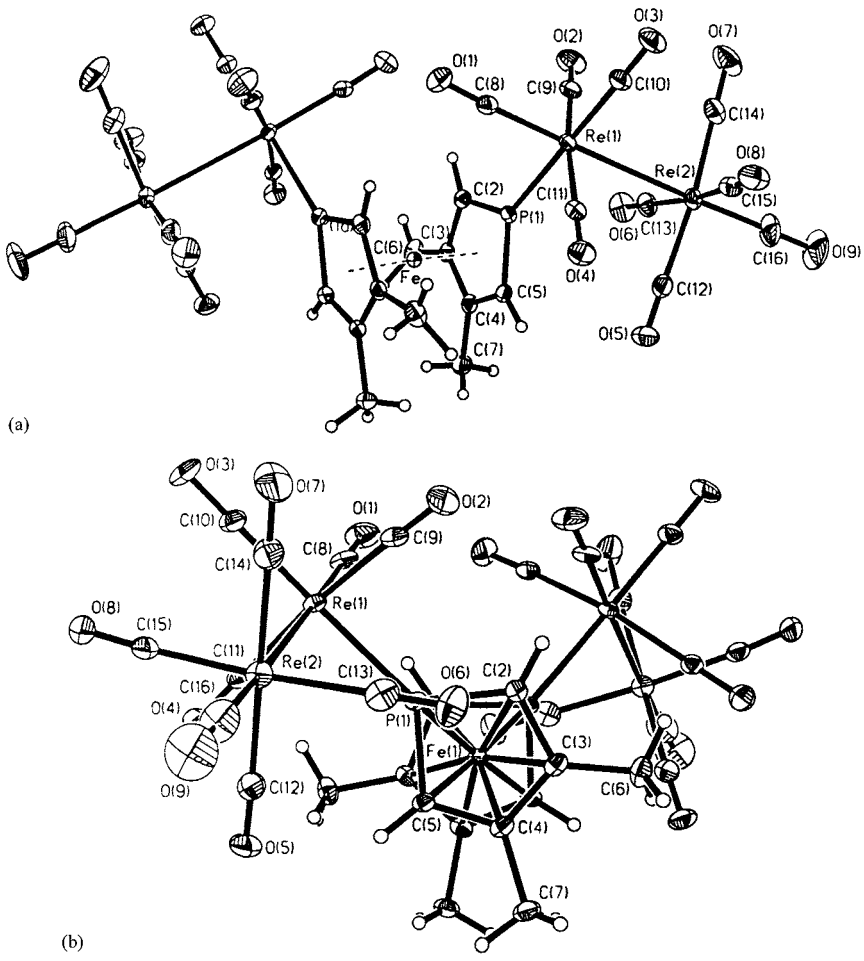


Fig. 1. (a) A view of the molecule **4**. (b) A top view of the phospholyl rings in **4**.

Conformation of the 1,1′-diphoshaferrocene moiety is usually described by the value of the dihedral angle θ, defined as the angle between the planes normal to each phospholyl ring that contain both Fe and P atoms [13]. When two P atoms are eclipsed $\theta = 0°$, whereas in the antiperiplanar conformation $\theta = 180°$. In **4** the observed value of θ is 85.2(2)°, which is larger than that observed in the X-ray structure of 3,3′,4,4′,-tetramethyl-1,1′-diphosphaferrocenyl-2 carboxylic acid ($\theta = 4.7(1)°$) and in its bis-[$W(CO)_5$] complex ($\theta = 72.6(1)°$) [14]. This may reflect higher steric hindrance of [$Re_2(CO)_9$] moieties. On the other hand, it is worth noting that **3** displays a larger value of θ (ca. 140°) [13]. Apparently, conformational preferences of the 1,1′-diphosphaferrocene system are more complicated than it was believed on the basis of theoretical calculations [13], indicating destabilisation of the eclipsed conformation with respect to conformations with $\theta > 100°$.

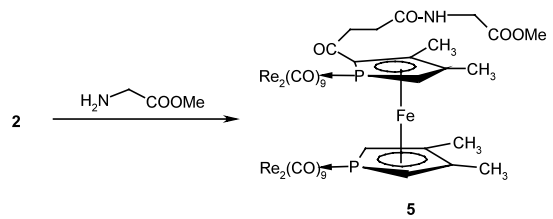
2.2. Synthesis of **2** and testing its reactivity

The above method was then applied to prepare **2**. Reaction of **1b** with four equivalents of [$Re_2(CO)_{10}$] pretreated with Me_3NO gave this compound in 61% yield. Compound **2** was purified by flash chromatography and gave correct elemental analysis and spectral data. The infrared spectrum of this compound in the region 2200–1900 $cm^{-1}$is closely similar to that of **4**, suggesting the same co-ordination mode of the $Re_2(CO)_9$ moieties in both compounds. The total reaction time was 1 h. In order to test reactivity of the active ester function in **2** we reacted this compound with glycine methyl ester in dichloromethane–triethylamine at room temperature for 30 min. We found that the conjugate **5** is formed in 71% isolated yield (after flash chromatography) (Scheme 3). The formation of the amide bond proceeds therefore efficiently, which is a good promise for eventual conjugation of **5** with target-specific biomolecules. Compounds **2** and **5** displays in their IR spectra ($CHCl_3$ solutions) very intense absorption bands at 2105, 2045, 1989 and 1940 cm^{-1} [1] making them easily detectable by this technique. Compound **2** is soluble in polar organic solvents (*N*,*N*-dimethylformamide, dimethylsulfoxide etc.) and in their mixtures with water, usually used for covalent labelling of proteins with water-insoluble reagents. Aerated solu-

Table 2
Selected bond lengths (Å) and angles (°) and their average values

bond lengths		average values		bond angles		average values	
Re Coordination							
Re(1)-Re(2)	3.0685(4)			P(1)-Re(1)-Re(2)	90.90(4)		
Re(1)-P(1)	2.4063(16)			P(1)-Re(1)-C(8)	90.3(2)		
Re(1)-C(10)	1.948(7)			P(1)-Re(1)-C(9)	86.8(2)		
Re(1)-C(8)	1.937(7)	$Re-C_{ax}$	1.931(6)	P(1)-Re(1)-C(10)	177.9(2)	C_{ax}-Re-Re	178.3(2)
Re(2)-C(16)	1.925(10)			P(1)-Re(1)-C(11)	89.9(2)	C_{ax}-Re-C_{eq}	93.6(2)
Re(1)-C(9)	1.981(8)			C(9)-Re(1)-C(11)	170.0(3)	C_{eq}-Re-Re	86.3(2)
Re(1)-C(11)	1.995(7)			C(12)-Re(2)-C(14)	173.4(4)	C_{eq}-Re-C_{eq}	89.8(2)
Re(2)-C(12)	2.004(9)			C(13)-Re(2)-C(15)	171.6(4)		
Re(2)-C(13)	1.988(10)	$Re-C_{eq}$	1.996(4)				
Re(2)-C(14)	1.994(9)						
Re(2)-C(15)	1.989(9)						
		O-C	1.135(3)			O-C-Re	178.4(3)
Phospholyl Ring							
P(1)-C(5)	1.739(7)			C(5)-P(1)-C(2)	91.4(3)		
P(1)-C(2)	1.754(7)			C(5)-P(1)-Re(1)	138.0(2)		
C(2)-C(3)	1.429(9)			C(2)-P(1)-Re(1)	130.0(2)		
C(3)-C(4)	1.407(9)			C(3)-C(2)-P(1)	112.0(5)		
C(3)-C(6)	1.483(10)			C(4)-C(3)-C(2)	111.6(6)		
C(4)-C(5)	1.426(9)			C(4)-C(3)-C(6)	125.3(7)		
C(4)-C(7)	1.500(10)			C(2)-C(3)-C(6)	123.2(7)		
				C(3)-C(4)-C(5)	113.5(6)		
				C(3)-C(4)-C(7)	123.1(7)		
				C(5)-C(4)-C(7)	123.4(7)		
				C(4)-C(5)-P(1)	111.6(5)		
Fe Coordination							
Fe-P(1)	2.257(2)	Fe-C	2.086(2)				

tions of **2** are stable and do not show any traces of decomposition on a few days standing at room temperature. This stability can be (at least in part) associated with π-accepting properties of the 1,1′-diphosphaferrocene moiety [5], stabilising a low oxidation state of Re centres. The intense yellow colour of **2** significantly simplifies chromatographic separation of this compound and its bioconjugates.



Scheme 3.

3. Experimental

All operations were performed under an atmosphere of dry pure argon. Solvents were freshly distilled over the appropriate drying agents immediately prior to use. Compounds **1a** and **3** were prepared according to the literature methods [2,4]. All other reagents were commercially available (Fluka, Aldrich) and were used as

received. Chromatographic separations were carried out on Kieselgel 60 (230–400 mesh ASTM) purchased from Merck, using $CHCl_3$ as eluent. NMR spectra were recorded on a Varian Gemini 200 BB spectrometer (200 MHz for ^1H) and are referenced to internal Me_4Si (^1H and ^{13}C) or to external H_3PO_4 (^{31}P). IR spectra were measured on a Biorad FTIR spectrometer.

3.1. Syntheses

3.1.1. [{$Re_2(CO)_9(PC_6H_8)$}$_2$Fe] (4)

A solution of [$Re_2(CO)_{10}$] (652 mg, 1 mmol) and trimethylamine *N*-oxide (94 mg, 1.25 mmol) in THF (100 ml) was stirred at room temperature (r.t.), for 30 min and concentrated in vacuo to ca. 1/2 of its initial volume. 3,3′,4,4′-Tetramethyl-1,1′-diphosphaferrocene 3 (70 mg, 0.25 mmol) was added and the resulting solution was heated under reflux for 0.5 h. After evaporation to dryness pale yellow **4** was isolated by column chromatography and crystallisation from $CHCl_3$–C_6H_{14}. Yield: 80% ^1H-NMR ($CDCl_3$, δ ppm): 4.05 (d, 4H, $J_{P-H} = 33$ Hz, phospholyl H's), 2.10 (s, 12H, Me); ^{31}P-NMR ($CDCl_3$, δ ppm): -53.1(s). IR (KBr, cm^{-1}): 2101, 2042, 1970, 1933. Anal. Calc. for $C_{30}H_{16}FeO_{18}P_2Re_4$: C, 23.60; H, 1.06. Found: C, 24.11; H, 1.03%.

3.1.2. Active ester 1b

A solution of 4-oxo-4-3,3′,4,4′-tetramethyl-1,1′-(diphosphaferrocene-2-yl)butanoic acid (**1a**) (378 mg, 1 mmol), *N*-hydroxysuccinimide (115 mg, 1 mmol) and *N*,*N*′-dicyclohexylcarbodiimide (1 ml of 1 M solution in CH_2Cl_2, 1 mmol) in CH_2Cl_2 (5 ml) was stirred at r.t. for 45 min. The precipitated *N*,*N*′-dicyclohexylurea was filtered off and the filtrate evaporated to dryness. Column chromatography afforded **1b** as a deep red oil (440 mg, 93%); ^1H-NMR ($CDCl_3$, δ ppm): 3.98 (1H, d, $J_{P-H} = 36.9$ Hz, H2), 3.73 (d, 1H, $J_{P-H} = 36.0$ Hz, H2′ or H5), 3.66 (d, 1H, $J_{P-H} = 38.0$ Hz, H2′ or H5), 3.08 (m, 2H, CH_2), 3.02 (m, 2H, CH_2), 2.83 (s, 4H, succinimidyl H's), 2.38, 2.09, 2.03, 1.93 (singlets, each 3H, Me's).

*3.1.2.1. [{$Re_2(CO)_9$}$_2$-**1b**] (2).* A solution of [$Re_2(CO)_{10}$] (652 mg, 1 mmol) and trimethylamine *N*-oxide (94 mg, 1.25 mmol) in THF (100 ml) was strirred at r.t., for 30 min and concentrated in vacuo to ca. 1/2 of its initial volume. Compound **1b** (119 mg, 0.25 mmol) was added and the resulting solution was heated under reflux for 0.5 h. After evaporation to dryness pale yellow **4** was isolated by column chromatography and crystallisation from CH_2Cl_2–C_6H_{14}. Yield: 272 mg (62%). Anal. Calc. for $C_{38}H_{23}FeNO_{23}P_2Re_4 \cdot 1/6C_6H_{14}$: C, 26.95; H, 1.5; N, 0.8. Found: C, 27.3; H, 1.4; N, 0.9%. ^1H-NMR ($CDCl_3$, δ ppm): 4.66 (1H, dd, $J_{P-H} =$ 32.6 Hz, $J_{H-H} = 1.7$ Hz, H2′ or H5′), 4.52 (d, $J_{P-H} =$ 33.2 Hz, 1H, H5), 3.55 (dd, $J_{P-H} = 32.6$ Hz, $J_{H-H} = 1.7$ Hz, 1H, H2′ or H5′), 3.20–3.50 (m, 4H, CH_2CH_2 in the lateral chain), 2.84 (s, 4H, succinimide), 2.33, 2.17, 2.07, 2.01 (singlets, each 3H, $4 \times CH_3$). ^{13}C-NMR ($CDCl_3$, δ ppm): 199.6 (d, $J_{P-C} = 13.0$ Hz, C=O), 194.7–193.2 (m, C≡O), 192.1 (d, $J_{P-C} = 5.3$ Hz, C≡O), 190.8 (s, C≡O), 186.5–186.0 (m, C≡O), 168.8 (s, C=O), 168.3 (s, C=O), {99.5 (d, $J_{P-C} = 1.9$ Hz), 98.6 (s), 98.1 (d, $J_{P-C} = 1.6$ Hz), 90.9 (d, $J_{P-C} = 57$ Hz), 86.6 (s), 75.3 (d, $J_{P-C} = 8.7$ Hz), 72.6 (d, $J_{P-C} = 8.9$ Hz), 72.4 (d, $J_{P-C} = 8.0$ Hz) phospholyl C's}, 38.5 (s, CH_2), 25.6 (s, $2 \times CH_2$ succinimide), 25.1 (s, CH_2), 16.0 (d, $J_{P-C} =$ 4.9 Hz), 14.2 (d, $J_{P-C} = 3.5$ Hz), 14.0 (d, $J_{P-C} = 5.7$ Hz), 13.4 (d, $J_{P-C} = 5.9$ Hz), $4 \times CH_3$}.^{13}C-NMR ($CDCl_3$, δ ppm): -23.7 (d, $J_{P-H} = 33.2$ Hz, P1), -43.0 (*t*, $J_{P-H} = 32.6$ Hz, P1′). IR (KBr, cm^{-1}): 2105, 2045, 1989, 1940.

3.2. Reaction of 2 with glycine methyl ester

A solution of **2** (51 mg, 0.03 mmol), glycine methyl ester chlorohydride (7 mg, 0.06 mmol) in CH_2Cl_2 (5 ml) containing two drops of triethylamine was stirred at r.t., for 30 min. After shaking with 2 M aq. HCl (5 ml) the organic layer was separated, dried (Na_2SO_4), evaporated to dryness and chromatographed. The analytical sample of **5** was crystallised from CH_2Cl_2–C_5H_{12} at -80 °C. Yield: 31 mg (61%). Anal. Calc. for $C_{37}H_{25}FeNO_{22}P_2Re_4 \cdot 2/3C_6H_{14}$: C, 28.1; H, 2.0; N, 0.80. Found: C, 28.1; H, 2.1; N, 1.0%. ^1H-NMR ($CDCl_3$, δ ppm): 6.10 (t, $J_{H-H} = 5.0$ Hz, 1H, NH), 4.71 (dd, $J_{P-H} = 32.5$ Hz, $J_{H-H} = 0.8$ Hz, 1H, H2′ or H5′), 4.56 (d, $J_{P-H} = 33.2$ Hz, 1H, H5), 4.03 (t, $J_{H-H} = 5.0$ Hz, 2H, CH_2 of glycine), 3.77 (s, 3H, CH_3 of glycine), 3.48 (dd, $J_{P-H} = 32.5$ Hz, $J_{H-H} = 0.8$ Hz, 1H, H2′ lub H5′), 3.25–3.08 (m, 1H, CH_{2-}), 2.78–2.62 (m, 2H, CH_2), 2.45–2.27 (m, 1H, CH_2), 2.37 (s, 3H, CH_3), 2.16 (s, 3H, CH_3), 2.08 (s, 3H, CH_3), 1.87 (s, 3H, CH_3). ^{13}C-NMR ($CDCl_3$, δ ppm): 199.6 (d, $J_{P-C} = 13.0$ Hz, C=O), 194.6–190.8 (m, C≡O), 186.3 (d, $J_{P-C} = 9.4$ Hz, C=O), 171.7 (s, C=O), 170.2 (s, C=O), {99.6 (d, $J_{P-C} =$ 2.6 Hz), 98.4 (s), 98.0 (s), 91.0 (s), 86.5 (s), 75.4 (d, $J_{P-C} = 9.0$ Hz), 72.3 (d, $J_{P-C} = 8.6$ Hz), 72.1 (d, $J_{H-H} = 7.2$ Hz), H2′ phospholyl C's}, 52.4 (s, CH_3, ester), 41.3 (s, CH_2), 38.9 (s, CH_2), 30.9 (s, CH_2), {16.0 (d, $J_{P-C} = 5.2$ Hz), 14.2 (d, $J_{P-C} = 3.7$ Hz), 13.9 (d, $J_{P-C} = 5.6$ Hz), 13.2 (d, $J_{H-H} = 6.1$ Hz, methyls}. ^{31}P-NMR ($CDCl_3$, δ ppm): -23.5 (d, $J_{P-H} = 33.2$ Hz, P1), -44.5 (t, $J_{P-H} = 32.5$ Hz, P1′). IR (KBr, cm^{-1}): 2105, 2045, 1989, 1940.

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