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Synthesis and NMR Spectroscopy of 1,3,3,5,5-Pentaalkoxy-1-chlorocyclotriphosphazenes

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Abstract—A series of pentaalkoxychlorocyclotriphosphazenes was synthesized. The spectral characteristics of the synthesized compounds (^{31}P, ^{1}H, ^{13}C NMR) were studied. It was shown that the complexity of the NMR spectra of pentaalkoxychlorocyclotriphosphazenes is associated with the magnetic nonequivalence of the phosphorus atoms in the triphosphazene cycle and the hydrogen and carbon atoms in the alkoxy groups on these phosphorus atoms, as well as the *cis/trans* isomerism of the latter groups.

Keywords: hexachlorotriphosphazene, pentaalkoxychlorocyclotriphosphazenes, NMR spectroscopy, *cis/trans* isomerism, prochirality

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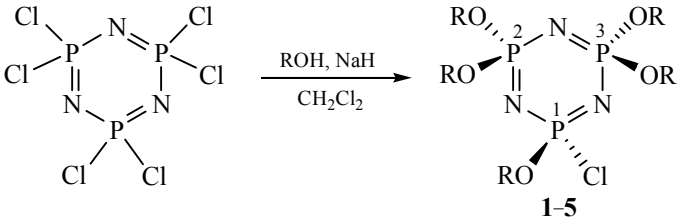
Transport molecules, specifically molecules that are capable of transporting target molecules, are being presently thoroughly investigated [1]. Polyalkoxypolyphosphazenes provide an example of such transport molecules [2]. Being water-soluble compounds, polyalkoxypolyphosphazenes are good candidates for application in medicine and pharmacology as drug carriers [2, 3]. At the same time, no examples of the application of pentaalkoxychlorocyclotriphosphazenes as transport molecules have been reported.

In the present work we synthesized 1,3,3,5,5-substituted 1-chlorocyclotriphosphazenes **1–3** containing 2-methoxyethanol, 2-(2-methoxyethoxy)ethanol, and 1-(2,2-dimethyl-1,3-dioxolan-4-yl)methanol fragments. The ^{31}P, ^{1}H, and ^{13}C NMR spectra of compounds **1–3** proved difficult to assign, and, therefore, as model compounds we synthesized 1,3,3,5,5-pentabenzyl-1-chlorocyclotriphosphazene (**4**) and 1,3,3,5,5-penta(2-phenylethyl)-1-chlorocyclotriphosphazene (**5**), which have simpler spectral patterns (Scheme 1).

1,3,3,5,5-Pentaalkoxy-1-chlorocyclotriphosphazenes **1–5** were synthesized by the reaction of hexachlorocyclotriphosphazene with the corresponding alcohols in methylene chloride at 20°C.

The ^{31}P, ^{1}H, and ^{13}C NMR spectra of chlorocyclotriphosphazenes **1–5** have some special features.

Scheme 1.



1–**5**

R = 2-methoxyethyl (**1**), 2-(2-methoxyethoxy)ethyl (**2**), 1-(2,2-dimethyl-1,3-dioxolan-4-yl)methyl (**3**), 2-phenylethyl (**4**), benzyl (**5**).

The ^{31}P NMR spectra of compounds **1–5** are ABA' systems, where each of the P^1, P^2, and P^3 atoms has its separate signal. Thus, in the spectrum of 1,3,3,5,5-pentakis(2-phenylethoxy)-1-chlorocyclotriphosphazene (**4),** the P^1 atom appears as a doubled doublet at 26.98 ppm ($^2J_{PP}$ = 80.0 and $^2J_{PP}$ = 77.0 Hz) due to spin–spin coupling of P^1 with P^2 and P^3. In their turn, P^2 and P^3 appear as doublets with coincident chemical shifts (δ_P 14.51 ppm) and different coupling constants ($^2J_{PP}$ = 80.0 and $^2J_{PP}$ = 77.0 Hz, respectively).

The ^{31}P NMR spectra of 1,3,3,5,5-pentaalkoxy-1-chlorocyclotriphosphazenes **1**, **2**, and **5** are similar. An exception is the spectrum of 1,3,3,5,5-pentakis[1-(2,2-dimethyl-1,3-dioxolan-4-yl)methoxy]-1-chlorocyclotriphosphazene (**3)**, because of the chirality of the substituents, the phosphorus signals merge into multiplets.

The ^1H NMR spectrum of compound **4** contains, along with the phenyl proton multiplet at 7.19–7.13 ppm and the P^1OCH_2 proton signal at 4.139 ppm, signals of the same groups at P^2 and P^3 at 4.140, 4.138 and 4.047, 4.045 ppm, respectively. The signals of the CH_2Ph groups of the alkoxy substituents are observed at 3.01, 3.16, and 2.96 ppm. However, these data were not enough to assign the stereochemistry of compounds **1–5**. Therefore, we measured the ^1H–^1H NOESY NMR spectrum of compound **4**. The spectrum revealed coupling of the P^1OCH_2 protons with the OCH_2 protons that give signals at δ_H 4.047 and 4.045 ppm and no coupling with the OCH_2 protons that give signals at δ_H 4.140 and 4.138 ppm. Therefore, we can suggest that the signals at δ_H 4.047 and 4.045 ppm belong to the corresponding protons of the alkoxy groups on P^2 and P^3, arranged *cis* to the alkoxy group on P^1. The signals at 4.140 and 4.138 ppm belong to protons of the alkoxy groups on P^2 and P^3, arranged *trans* to the alkoxy group on P^1. By analogy, we can suggest that the signal at δ_H 2.96 ppm is associated with the *cis*-CH_2Ph group, and that at 3.16 ppm, to the *trans*-CH_2Ph group.

The ^{13}C NMR spectrum of compound **4** displays signals of both phenyl and alkoxy methylene carbon atoms. To assign the signals of the methylene carbon atoms and protons, we obtained the ^1H–^{13}C HMQC NMR spectrum. It was found that, unlike what is observed in the ^1H NMR spectrum, the carbon signals of the OCH_2 groups on P^2 and P^3 appear in a different order. Specifically, the signals at 66.79 and 66.76 ppm belong to the OCH_2 groups on P^2 and P^3, arranged *cis* to the alkoxy group on P^1, while the signals at 66.75 and 66.72 ppm belong to the *trans* isomer.

The ^1H–^1H NOESY and ^1H–^{13}C HMQC NMR spectra were also obtained for 1,3,3,5,5-pentakis[2-(2-me-thoxyethoxy)ethoxy]-1-chlorocyclotriphosphazene (**2)**. The signals of the alkoxy substituents on P^2 and P^3 in the ^1H and ^{13}C NMR spectra are observed at the positions similar to those of phosphazene **4**. Unfortunately, we could not measure the ^1H–^1H NOESY NMR spectra of compounds **1** and **5**, and, therefore, could not assign the ^1H and ^{13}C NMR spectra of these compounds by analogy with compounds **2** and **4**.

The unexpectedly complicated spectral patterns of compounds **1–5** can be explained by the fact that the planar 1,3,3,5,5-pentaalkoxy-1-chlorocyclotriphosphazene molecules have a prochiral center on P^1. As a result, the P^2 and P^3 atoms in the triphosphazene cycle, as well as the hydrogen and carbon atoms of the alkoxy groups on these phosphorus atoms are magnetically nonequivalent and give separate groups of signals in the ^{31}P, ^1H, and ^{13}C NMR spectra. The spectral patter is even more complicated by the *cis*/*trans* isomerism of the alkoxy groups on P^2 and P^3 with respect to the alkoxy group on P^1, as well as by the long-range ^{31}P– ^1H spin–spin coupling. As a result, the P^1, P^2 and P^3 signals in the ^{31}P NMR spectra of compounds **1–5** appear as an ABA' system; in going from compound **5** to compounds **4** and **1**, **2,** the P^2OCH_2 and

P^3OCH_2 proton signals, which appear as an ABX system in the ^1H NMR spectrum of compound **5** transform into an ABMX system for **4** or hardly interpretable multiplets for **1** and **2**. The ^{13}C NMR spectra are complicated in a similar way.

EXPERIMENTAL

The ^1H, ^{31}P, and ^{13}C NMR spectra were measured on a Bruker Avance 400 spectrometer (400.13, 161.98, and 100.05 MHz, respectively) in $CDCl_3$. The ^1H–^1H NOESY and ^1H–^{13}C HMQC NMR spectra were obtained on a Bruker Avance II 600 spectrometer (600.22 and 150.92 MHz, respectively) in $CDCl_3$.

Elemental analysis (C, H, N) was performed on a Carlo Erba 1106 analyzer. Analysis for P was performed on a Cary 100 Scan spectrophotometer, and Cl was determined by titrimetry (0.001 M $AgNO_3$).

All syntheses were performed in an inert atmosphere. Methylene chloride was distilled over P_2O_5 and stored over CaH_2.

1-Chloro-1,3,3,5,5-pentakis(2-methoxyethoxy)-cyclotriphosphazene (1). A solution of 2.51 g

(0.033 mol) of 2-methoxyethanol in 10 mL of CH_2Cl_2 was added over the course of 20 min to a vigorously stirred suspension of 0.83 g (0.036 mol) of NaH in 30 mL of a solution of 2 g (0.006 mol) of hexachlorocyclotriphosphazene in CH_2Cl_2; in doing so, the temperature of the reaction mixture was maintained at 0°C. The resulting mixture was stirred at that temperature for 2 h and then at 20°C for 18 h, after which 1 mL of isopropanol was added slowly dropwise under stirring. When gas no longer evolved, 1 mL of water and 2 g of $NaHCO_3$ were added in succession The mixture was stirred for 30 min and the precipitate was filtered off and washed with CH_2Cl_2 (2×10 mL). The filtrate was evaporate, and the residue was chromatographed on silica gel in chloroform–ethyl acetate (1 : 1). Yield 1.15 g (36.6%). ^{1}H NMR spectrum, δ, ppm: 4.22–4.17 m (2H, P^1OCH_2, $^3J_{PH}$ = 9.7 Hz), 4.07–3.99 m (8H, P^2OCH_2, P^3OCH_2), 3.60–3.58 m (2H, $P^1OCH_2C\underline{H}_2OCH_3$), 3.56 t and 3.53 t (8H, $P^2OCH_2C\underline{H}_2OCH_3$, $P^3OCH_2C\underline{H}_2OCH_3$, $^3J_{HH}$ = 5.1 Hz), 3.32 s (3H, $P^1\cdots OCH_3$), 3.31 s and 3.30 s (12H, $P^2\cdots OCH_3$, $P^3\cdots OCH_3$). ^{13}C NMR spectrum, δ_C, ppm: 71.17 d.d (P^2OCH_2, P^3OCH_2, $^2J_{PC}$ = 2.9, 2.5 Hz), 70.79 d (P^1OCH_2, $^2J_{PC}$ = 7.0 Hz), 66.48 d ($P^1OCH_2\underline{C}H_2OCH_3$, $^3J_{PC}$ = 4.8 Hz), 65.33 t ($P^2OCH_2\underline{C}H_2OCH_3$, $^3J_{PC}$ = 1.8 Hz), 65.20 t ($P^3OCH_2\underline{C}H_2OCH_3$, $^3J_{PC}$ = 1.5 Hz), 59.01 ($P^1\cdots CH_3$), 58.98 and 58.95 ($P^2\cdots CH_3$, $P^3\cdots OCH_3$). ^{31}P NMR spectrum, δ_P, ppm: 27.01 d.d (P^1, $^2J_{PP}$ = 81.9, $^2J_{PP}$ = 78.7 Hz), 15.15 d (P^2, $^2J_{PP}$ = 81.9 Hz), 15.14 d (P^3, $^2J_{PP}$ = 78.7 Hz). Found, %: C 33.11; H 6.41; Cl 6.80; N 7.62; P 17.07. $C_{15}H_{35}N_3O_{10}P_3Cl$. Calculated, %: C 33.01; H 6.46; Cl 6.49; N 7.70; P 17.02.

Compounds **2–5** were prepared in a similar way.

1-Chloro-1,3,3,5,5-pentakis[2-(2-methoxyethoxy)-ethoxy]cyclotriphosphazene (2). Yield 23.6%. ^{1}H NMR spectrum, δ, ppm: 4.12–4.08 m (2H, P^1OCH_2, $^3J_{PH}$ = 10.0 Hz), 4.06–4.03 m (4H, *trans*-P^2OCH_2, *trans*-P^3OCH_2, J_{PH} = 4.7 Hz), 4.02–3.99 m (4H, *cis*-P^2OCH_2, *cis*-P^3OCH_2, $^3J_{PH}$ = 4.0 Hz), 3.69–3.55 m [20H, $P^1OCH_2C\underline{H}_2$, $P^2OCH_2C\underline{H}_2$, $P^3OCH_2C\underline{H}_2$, $P^1\cdots OC\underline{H}_2\cdot CH_2OCH_3$, $P^2\cdots OC\underline{H}_2CH_2OCH_3$, $P^3\cdots OC\underline{H}_2CH_2OCH_3$], 3.47–3.43 m [10H, $P^1\cdots OCH_2C\underline{H}_2OCH_3$, $P^2\cdots OCH_2\cdot C\underline{H}_2OCH_3$, $P^3\cdots OCH_2C\underline{H}_2OCH_3$], 3.30 s (3H, $P^1\cdots OCH_3$), 3.29 s and 3.28 s (12H, $P^2\cdots OCH_3$, $P^3\cdots OCH_3$). ^{13}C NMR spectrum, δ_C, ppm: 71.74 s ($P^1\cdots OCH_3\underline{C}H_3OCH_3$, $P^2\cdots OCH_2\underline{C}H_2OCH_3$, $P^3\cdots OCH_2\underline{C}H_2OCH_3$), 70.48 s ($P^1\cdots O\underline{C}H_2CH_2OCH_3$), 70.37 s ($P^2\cdots O\underline{C}H_2CH_2OCH_3$, $P^3\cdots O\underline{C}H_2CH_2OCH_3$), 69.76 d (*cis*-$P^2OCH_2$, *cis*-$P^3OCH_2$, $^2J_{PC}$ = 4.0 Hz), 69.68 d (*trans*-P^2OCH_2, *trans*-P^3OCH_2, $^2J_{PC}$ = 3.9 Hz), 69.36 d (P^1OCH_2, $^2J_{PC}$ = 10.3 Hz), 66.47 d ($P^1OCH_2\underline{C}H_2$, $^3J_{PC}$ = 7.3 Hz), 65.32 d.d and 65.19 d.d ($P^2CH_2\underline{C}H_2$, $P^3OCH_2\underline{C}H_2$, $^3J_{PC}$ = 2.2, $^3J_{PC}$ = 2.9 Hz), 58.82 (CH_3). ^{31}P NMR spectrum, δ_P, ppm: 26.86 d.d (P^1, $^2J_{PP}$ = 81.9, $^2J_{PP}$ = 78.3 Hz), 15.00 d (P^2, $^2J_{PP}$ = 81.9 Hz), 14.99 d (P^3, $^2J_{PP}$ = 78.3 Hz). Found, %: C 39.35; H 7.51; Cl 4.77; N 5.48; P 12.31. $C_{25}H_{55}N_3O_{15}P_3Cl$. Calculated, %: C 39.19; H 7.24; Cl 4.63; N 5.49; P 12.13.

1-Chloro-1,3,3,5,5-pentakis[1-(2,2-dimethyl-1,3-dioxolan-4-yl)methoxy]cyclotriphosphazene (3). Yield 58.7%. ^{1}H NMR spectrum, δ, ppm: 4.34–4.21 m (5H, $POCH_2C\underline{H}CH_2$), 4.12–3.78 m (20H, $POC\underline{H}_2CHC\underline{H}_2$), 1.38 s [6H, $P^1\cdots C(C\underline{H}_3)_2$], 1.37 s and 1.36 s [12H, *trans*-$P^2\cdots C(C\underline{H}_3)_2$, *trans*-$P^3\cdots C(C\underline{H}_3)_2$], 1.30 s and 1.29 s [12H, *cis*-$PO\cdots C(C\underline{H}_3)_2$]. ^{13}C NMR spectrum, δ_C, ppm: 109.69 [$P^1\cdots\underline{C}(CH_3)_2$], 109.69 [$P^2O\cdots\underline{C}(CH_3)_2$, $P^3O\cdots\underline{C}(CH_3)_2$], 73.68 d.d ($P^2OCH_2$, P^3OCH_2, $^2J_{PC}$ = 5.1, $^2J_{PC}$ = 4.4 Hz), 73.42 d (P^1OCH_2, $^2J_{PC}$ = 11.0 Hz), 67.15 d.d ($P^1OCH_2\underline{C}HCH_2$, $^3J_{PC}$ = 2.9, $^3J_{PC}$ = 7.3 Hz), 66.25–66.09 m ($P^2OCH_2\underline{C}HCH_2$, $P^3OCH_2\underline{C}HCH_2$, $P^2OCH_2CH\underline{C}H_2$, $P^3OCH_2CH\underline{C}H_2$, $P^1OCH_2CH\underline{C}H_2$), 26.67 s ($P^1\cdots C\underline{C}H_3$), 26.64 s ($PO\cdots C\underline{C}H_3$), 25.20 ($P^1\cdots C\underline{C}H_3$), 25.15 ($PO\cdots C\underline{C}H_3$). ^{31}P NMR spectrum, δ_P, ppm: 27.42–26.39 m (P^1), 15.37–15.30 m and 14.87–14.80 m (P^2, P^3). Found, %: C 43.50; H 6.66; Cl 4.36; N 4.98; P 11.17. $C_{30}H_{55}N_3O_{15}P_3Cl$. Calculated, %: C 46.31; H 6.71; Cl 4.29; N 5.09; P 11.25.

1-Chloro-1,3,3,5,5-pentakis(2-phenylethyl)cyclotriphosphazene (4). Yield 26.3%. ^{1}H NMR spectrum, δ, ppm: 7.19–7.13 m (25H, Ph), 4.28 dt (2H, P^1OCH_2, $^3J_{PH}$ = 8.9, $^3J_{HH}$ = 7.2 Hz), 4.140 m (2H, *trans*-P^2OCH_2, ABMX system, $^2J_{HH}$ = 10.0, $^3J_{HH}$ = 7.2, $^3J_{PH}$ = 4.7, $^3J_{PH}$ = 3.1 Hz), 4.138 m (4H, *trans*-P^3OCH_2, ABMX system, $^2J_{HH}$ = 10.2, $^3J_{HH}$ = 7.2, $^3J_{PH}$ = 3.1, $^3J_{PH}$ = 4.2), 4.047 m (2H, *cis*-P^2OCH_2, ABMX-system, $^2J_{HH}$ = 10.0, $^3J_{HH}$ = 7.2, $^3J_{PH}$ = 4.1, $^3J_{PH}$ = 2.6 Hz), 4.045 m (2H, *cis*-P^3OCH_2, ABMX system, $^2J_{HH}$ = 10.2, $^3J_{HH}$ = 7.2, $^3J_{PH}$ = 3.4, $^3J_{PH}$ = 3.4 Hz), 3.01 t (2H, $P^1OCH_2C\underline{H}_2$, $^3J_{HH}$ = 7.2 Hz), 3.16 t (2H, *trans*-$P^2OCH_2C\underline{H}_2$, *trans*-$P^3OCH_2C\underline{H}_2$, $^3J_{HH}$ = 7.2 Hz), 2.96 t (2H, *cis*-$P^2OCH_2C\underline{H}_2$, *cis*-$P^3OCH_2C\underline{H}_2$, $^3J_{HH}$ = 7.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 137.50 and 137.48 (*ipso*-C, $P^2OCH_2CH_2\underline{Ph}$, $P^3OCH_2CH_2\underline{Ph}$), 137.06 s (*ipso*-C, $P^1OCH_2CH_2\underline{Ph}$), 129.08 s and 129.04 s (*m*-C, $P^2OCH_2CH_2\underline{Ph}$, $P^3OCH_2CH_2\underline{Ph}$), 129.07 s (*m*-C, $P^1OCH_2CH_2\underline{Ph}$), 128.54 s (*o*-C, $P^1OCH_2\cdot CH_2\underline{Ph}$), 128.49 s and 128.48 s (*o*-C, $P^2OCH_2CH_2\underline{Ph}$, $P^3OCH_2CH_2\underline{Ph}$), 126.73 (*p*-C, $P^1OCH_2CH_2\underline{Ph}$), 126.60 s and 126.56 s (*p*-C, $P^2OCH_2CH_2\underline{Ph}$, $P^3OCH_2CH_2\underline{Ph}$), 68.04 d ($P^1OCH_2$, $^2J_{PC}$ = 7.7 Hz), 66.76 t (*cis*-P^2OCH_2, *cis*-P^3OCH_2, $^2J_{PC}$ =

2.9 Hz), 66.71 t (*trans*-P^2OCH_2, *trans*-P^3OCH_2, $^2J_{PC}$ = 2.9 Hz), 36.58 t ($P^2OCH_2\underline{C}H_2$, $P^3OCH_2\underline{C}H_2$, $^3J_{PC}$ = 4.0 Hz), 36.32 d ($P^1OCH_2\underline{C}H_2$, $^3J_{PC}$ = 9.5 Hz). ^{31}P NMR spectrum, δ_P, ppm: 26.98 d.d (P^1, $^2J_{PP}$ = 80.0, $^2J_{PP}$ = 77.0 Hz), 14.51 d (P^2, $^2J_{PP}$ = 80.0 Hz), 14.51 d (P^3, $^2J_{PP}$ = 77.0 Hz). Found, %: C 61.73; H 5.74; Cl 5.49; N 5.30; P 12.38. $C_{40}H_{45}N_3O_5P_3Cl$. Calculated, %: C 61.90; H 5.84; Cl 4.57; N 5.41; P 11.97.

1-Chloro-1,3,3,5,5-pentabenzylcyclotriphosphazene (5). Yield 18.4%. ^1H NMR spectrum, δ, ppm: 7.33–7.24 m (25H, Ph), 5.11 d (2H, P^1OCH_2, $^3J_{PH}$ = 9.36 Hz), 5.06 m (2H, *trans*-P^2OCH_2, ABX system, $^2J_{HH}$ = 11.4, $^3J_{PH}$ = 4.3, $^3J_{PH}$ = 4.1 Hz), 5.02 m (2H, *trans*-P^3OCH_2, ABX system, $^2J_{HH}$ = 11.4, $^3J_{PH}$ = 4.4, $^3J_{PH}$ = 4.2 Hz), 4.96 m (2H, *cis*-P^2OCH_2, ABX system, $^2J_{HH}$ = 12.0, $^3J_{PH}$ = 4.3, $^3J_{PH}$ = 4.3 Hz), 4.93 m (2H, *cis*-P^3OCH_3, ABX system, $^2J_{HH}$ = 12.0, $^3J_{PH}$ = 4.6, $^3J_{PH}$ = 4.4 Hz). ^{13}C NMR spectrum, δ_C, ppm: 136.32 d and 136.28 d (*ipso*-C, $P^2OCH_3\underline{Ph}$, $P^3OCH_3\underline{Ph}$, $^3J_{PC}$ = 3.7, $^3J_{PC}$ = 4.4 Hz), 135.42 d (*ipso*-C, $P^1OCH_2\underline{Ph}$, $^3J_{PC}$ = 9.5 Hz), 128.56 s (*m*-C, $P^1OCH_2\underline{Ph}$), 128.48 s (*o*-C, $P^1OCH_2\underline{Ph}$), 128.47 s and 128.46 s (*m*-C, $P^2OCH_2\underline{Ph}$, $P^3OCH_2\underline{Ph}$), 128.20 s and 128.46 s (*p*-C, $P^2OCH_2\underline{Ph}$, $P^3OCH_2\underline{Ph}$), 127.96 s (*o*-C, $P^1OCH_3\underline{Ph}$), 127.77 s and 127.65 s (*o*-C, $P^2OCH_3\underline{Ph}$, $P^3OCH_2\underline{Ph}$), 69.09 d (P^1OCH_2, $^2J_{PC}$ = 9.5 Hz), 67.95 t and 67.91 t (P^2OCH_2, P^3OCH_2, $^2J_{PC}$ = 2.8 Hz). ^{31}P NMR spectrum, δ_P, ppm: 26.98 d.d (P^1, $^2J_{PP}$ = 80.7, $^2J_{PP}$ = 77.0), 15.13 d (P^2, $^2J_{PP}$ = 80.7 Hz), 15.13 d (P^3, $^2J_{PP}$ = 77.0 Hz). Found, %: C 59.54; H 5.11; Cl 5.07; N 5.91; P 13.61. $C_{35}H_{35}N_3O_5P_3Cl$. Calculated, %: C 59.54; H 5.00; Cl 5.02; N 5.95; P 13.16.

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