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Reactions of Platinum Nitrile Complexes with Cyclotriphosphazenes Containing Pyridylalkylamino Groups

S. A. Simanova*, T. V. Kuznetsova*, U. Diefenbach, and V. N. Demidov***

** St. Petersburg State Institute of Technology, Moskovskii pr. 26, St. Petersburg, 198013 Russia*
e-mail: tata@TK9173.spb.edu
*** Free University of Berlin, Germany*

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Abstract — Reactions of platinum(II) and platinum(IV) nitrile complexes with polydentate ligands, such as pentaphenoxy(2-pyridylmethylamino)cyclotriphosphazene, pentaphenoxy(3-pyridylmethylamino)cyclotriphosphazene, and pentaphenoxy(2-pyridylethylamino)cyclotriphosphazene, were studied. Platinum(IV) is reduced to platinum(II) upon complex formation; the pyridine and alkylamine nitrogen atoms coordinate to platinum(II) to form chelate rings. The compounds obtained were characterized by ^{1}H and ^{31}P NMR and IR spectroscopy, FAB mass spectrometry, and other methods.
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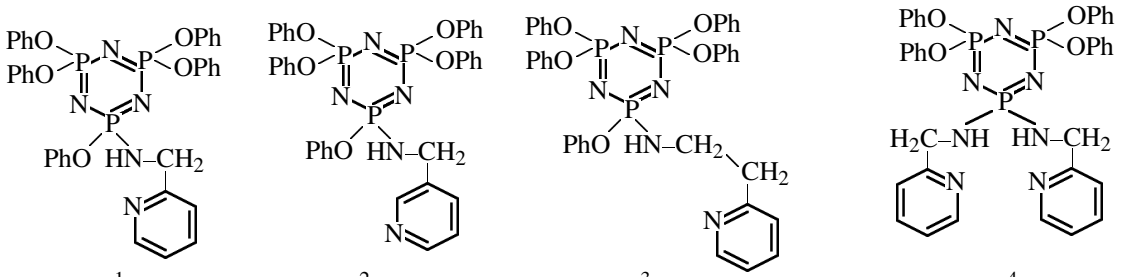
The interest in polymers forming macromolecular metal complexes is caused by the application of these polymers as ion-exchange resins, catalysts, and ionic conductors. They also offer promise as biologically active compounds [1–6]. Phosphazene polymers are not an exception, and, therefore, studying the behavior of small molecular models, such as cyclotri- or cyclotetraphosphazenes, in relation to metal ions becomes an important method of obtaining information on transformations that occur on coordination of polymeric phosphazenes to these ions. The interest in cyclotriphosphazenes is also connected with their polydentate nature, i.e. with a great number of donor atoms potentially capable of coordinating with metal atoms.

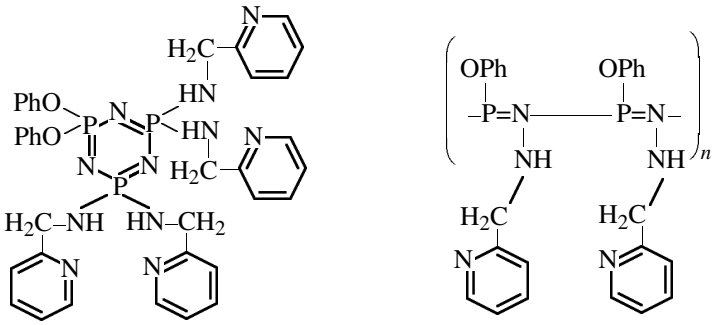
Cyclotriphosphazenes with N-donor side groups, such as pyrazoles and dimethylamine, were studied earlier [6]. Phosphazenes of this type can coordinate both through side-group and phosphazene nitrogens [6]. In particular, recently Thomas et al. [7] obtained complexes $[PtL^{n}Cl_2]$ in which the chelating ligand coordinates to the nitrogen atoms of neighboring pyrazole groups to close a six-membered ring, by reactions of $[Pt(PhCN)_2Cl_2]$ with cyclotriphosphazenes (L^n) containing geminal pyrazole and dimethylpyrazole groups.

In this work we studied cyclotriphosphazenes containing pyridylalkylamino groups. Pyridine-substituted phosphazenes were first obtained and characterized by Diefenbach et al. [8, 9]. The structures of Cu(II), Co(II), and Pt(II) complexes with pentaphenoxy(2-pyridylmethylamino)cyclotriphosphazene (L^1) were described in the same works [8, 9]. It was found by IR spectroscopy, FAB mass spectrometry, and elemental and X-ray diffraction analyses that compounds with metal–ligand ratios of 1 : 2 (Cu^{2+}) and 1 : 1 (Pt^{2+}) are formed, and both nitrogen atoms of the pyridylmethylamino ligand are involved in complex formation. The cobalt(II) complex comprises one phosphazene molecule coordinating through the phosphazene and pyridyl nitrogens. The platinum complex $[PtL^1Cl_2]$ was prepared by boiling of $PtCl_2$ with L^1 (molar ratio 1 : 1) in a 1 : 1 chloroform–benzene mixture. The copper and cobalt complexes were obtained by heating of corresponding nitrates with L^1 (molar ratio 1 : 1) in methanol under reflux [9]. Reactions of more complex platinum coordination compounds with pyridine-substituted cyclotriphosphazenes have not been studied before.

The aim of our work was to study complex formation of platinum(II) and platinum(IV) with various pyridine-substituted cyclotriphosphazenes, to identify the resulting complexes, and to study hydrolytic transformations of pentaphenoxy(3-pyridylmethylamino)-cyclotriphosphazene (L^2). As subjects for study we took five various pyridine-substituted cyclotriphosphazenes (L^1-L^5) and one polymeric phosphazene (L^6) of the general formula $N_3P_3(OPh)_xR_y$ [x = 5, y = 1; R = 2-pyridylmethylamino (L^1), 3-pyridylmethylamino (L^2), 2-pyridylethylamino (L^3); x = 4, y = 2, R = 2-pyridylmethylamino (*gem*) (L^4); x = 2, y = 4, R = 2-pyridylmethylamino (*gem*) (L^5); $x = y = n/3$, R = 3-pyridylmethylamino (50%) and phenoxy (50%) (L^6)].



L^1 L^2 L^3 L^4



L^5 L^6

The starting platinum compounds were the platinum(II) and platinum(IV) aceto- and propionitrile complexes $[Pt(EtCN)_2Cl_2]$, $[Pt(MeCN)_2Cl_2]$, $[Pt(EtCN)_2Cl_4]$, and $[Pt(MeCN)_2Cl_4]$. The choice of these very starting compounds was caused by two factors: their high solubility in organic solvents (especially of *cis*-$[Pt(EtCN)_2Cl_2]$) and lability of the nitrile ligands in substitution reactions.

Platinum(II) complexes with R-pentaphenoxycyclotriphosphazenes L^1–L^3 were synthesized. The isolated compounds $[PtL^nCl_2]$ were characterized by NMR and IR spectroscopy, FAB mass spectrometry, thin-layer chromatography, CHN analysis, and analysis for platinum. The complexes were obtained by heating of *cis*-$[Pt(RCN)_2Cl_2]$ (R = Me, Et) with corresponding phosphazene in a 1 : 1.1 ratio in acetonitrile or dichloromethane at 40–50°C for several hours. To increase the yield of the complex, the reaction was performed with the better soluble platinum(II) propionitrile complex.

In the IR spectra of the compounds, the band of the pyridine CN bond is shifted from 1580–1590 (free phosphazene) to 1600–1650 cm^{-1} (complex), which points, according to [8], to the coordination of the pyridine nitrogen atom to the metal. The characteristic bands in the IR spectra of platinum(II) complexes with cyclotriphosphazenes and of free phosphazenes are shown in Table 1. The ^{31}P NMR spectra display a nonshifted signal of the A_2B type (Table 2), implying that phosphazene nitrogen atoms do not participate in complex formation. Table 2 lists the ^1H and ^{31}P NMR spectra of $[PtL^3Cl_2]$. Evidence for the coordination is provided by the upfield shift of all bands and also by the fact that phosphazene CH_2 protons become non-

Table 1. Characteristic vibration frequencies (cm^{-1}) in the IR spectra of R-pentaphenoxycyclotriphosphazenes and their platinum(II) complexes

Assignment	L^1	$[PtL^1Cl_2]$	L^2	$[PtL^2Cl_2]$	L^3	$[PtL^3Cl_2]$
ν(NH)	3260 w	3293 w	3157 w	3420 w	3350	3428 w
$\nu(CC_{arom})$	1580 s	1590 m	1585 s	1592 m	1590 s	1589 m
$\nu(CC_{arom})$	1476 s	1488 s	1485 s	1490 s	1480 s	1487 s
ν(PN)	1216–1112 v.s	1260–1177 v.s	1220–1120 v.s	1180–1120 v.s	1210–1120 v.s	1179–1164 v.s
$\nu(PO_{arom})$	940 s	945 s	940 s	944 s	940 s	941 s
ν(CN)	1580	1612 m	1585	1652 m	1590	1605 m

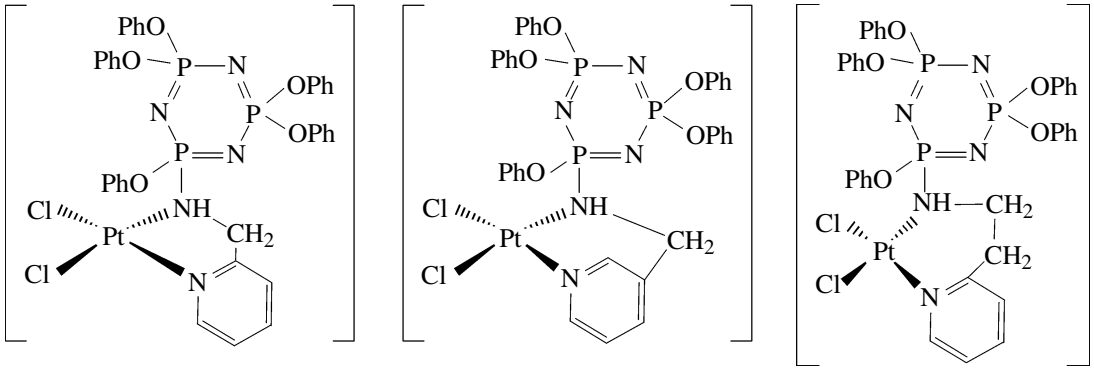
Table 2. NMR spectra of the complex $[PtL^3Cl_2]$ and the phosphaze L^3 (solvent $CDCl_3$)

^1H NMR spectrum, δ, ppm (*J*, Hz)		^{31}P NMR spectrum, δ_P, ppm (*J*, Hz)	
$[PtL^3Cl_2]$	L^3	$[PtL^3Cl_2]$	L^3
2.5 s (1H, $HNCH_2CH_2$), 2.8 t (2H, $HNCH_2CH_2$), 3.1 d (1H, $HNCH_2$, *J* 14.4), 3.4 br.d (1H, NH, *J* 7.1), 4.9 t.d (1H, $HNCH_2$, *J* 4.3, 14.4), 6.9 d.d (4H, CH_{Py}, *J* 7.3, 18.2), 7.7–7.2 m (24H, CH_{arom}/Py), 9.2 d (1H, CH_{Py}, *J* 5.5)	2.8 t (2H, $HNCH_2CH_2$, *J* 7.5), 3.2 (1H, NH), 3.1 m (2H, $HNCH_2$), 6.8 d (1H, CH_{Py}, *J* 9.0), 7.1 t (1H, CH_{Py}, *J* 9.0), 7.3–6.0 m (27H, $CH_{arom}/_{Py}$), 8.5 d (1H, CH_{Py}, *J* 5.4)	7.65 d (*J* 13.1), 7.12 s (P^A), 21.3 d.d (P^B, *J* 74.1, 80.6)	9.4 (P^A), 18.1 (P^B)

equivalent. This nonequivalence can be accounted for by closure of a nonplanar six-membered ring.

The reactions of platinum(IV) complexes $[Pt(RCN)_2Cl_4]$ with cyclotriphosphazenes involved reduction of platinum(IV) to platinum(II) to form compounds $[PtL^nCl_2]$ analogous to complexes obtained by the reactions of corresponding phosphazenes and $[Pt(RCN)_2Cl_2]$.

The reaction of *cis*-$[Pt(EtCN)_2Cl_2]$ with pentaphenoxy(2-pyridylmethylamino)cyclotriphosphazene gave the complex $[PtL^1Cl_2]$ which was earlier prepared from $PtCl_2$ and studied by X-ray diffraction [9].



PtL^1Cl_2 PtL^2Cl_2 PtL^3Cl_2

To find out whether the phosphazene ligand is readily replaced by such a typical chelating ligand as *o*-phenanthroline, we mixed solutions of *o*-phenanthroline and $[PtL^1Cl_2]$ (or $[PtL^3Cl_2]$) in chloroform (mole ratio 1 : 1). We expected that the very stable compound $[Pt(Phen)Cl_2]$ insoluble in usual solvents would precipitate. However, no precipitate appeared even when the reaction mixture was heated to the boiling point of the solvent. This result suggests that the phosphazene complex is fairly stable.

According to IR data, when the complexes $[PtL^1Cl_2]$ and $[PtL^3Cl_2]$ were chlorinated in chloroform with the purpose to synthesize platinum(IV) phosphazene complexes, phosphazene was absent from the reaction products, and an aminopyridine complex formed. The IR spectrum of the reaction product contained, instead of a weak amino ν(NH) band in the region of 3300–3400 cm^{-1}, a medium-intensity band at 3201 cm^{-1} and a medium-intensity $\nu(CC_{arom})$ band at 1483 cm^{-1}, whereas strong bands at 1200–1120 (PN) and 940 cm^{-1} (PO_{arom}) were absent.

The destruction of the ligand under the action of molecular chlorine on platinum(II) complexes can be accounted for in light of the review [10] devoted to ligand oxidation and/or cleavage reactions.

Reactions of platinum complexes with cyclotriphosphazenes containing two and more pyridylmethylamino groups were studied. We failed to isolate a

compound with a definite composition in the reaction of diphenoxytetrakis(2-pyridylmethylamino)cyclotriphosphazene (L^5) with *cis*-[$Pt(EtCN)_2Cl_2$].

The reaction of *cis*-[$Pt(EtCN)_2Cl_2$] with *gem*-tetraphenoxy(2-pyridylmethylamino)cyclotriphosphazene L^4 in a 1 : 1 molar ratio involved complete destruction of the phosphazene ring. According to ^1H NMR and ^{31}P data, the reaction products are a platinum(II) complex with 2-pyridylmethylamine coordinated in a bidentate fashion, and phosphoric acid. This It agrees with published data. The complex [$Cu(NH_2CH_2Py)_2 \cdot (NO_3)_2$] was prepared previously [11] by the reaction of *gem*-2-(pyridylmethylamino)tetrachlorocyclotriphosphazene with copper(II) nitrate.

Analysis of the NMR spectra of the free cyclotriphosphazenes and above-described complexes showed that the integral intensity of phenyl signals is much lower than expected by calculations. The ^{31}P NMR spectra of the phosphazenes contain two groups of signals instead of two expected A_2B peaks. In the chromatogram of solutions of the complexes in chloroform and dichloromethane, there are several spots with R_f values so close to each other that they are extremely difficult to separate (eluent acetone–chloroform in various ratios). Elemental analysis gave slightly overestimated platinum contents and underestimated carbon contents. Such discrepancy can be connected with the known tendency of pyridine-substituted phosphazenes for hydrolysis [8, 9]; however, hydrolysis products for the phosphazenes under discussion have not been described in the literature. Our data point to cleavage of phenol in the presence of traces of moisture.

$$N_3P_3(OPh)_5(NHCH_2Py) + H_2O \longrightarrow N_3P_3(OPh)_x(OH)_y(NHCH_2Py) + PhOH,$$
$$x = 0\text{–}5,\ y = 5 - x.$$

By ^{31}P NMR spectroscopy we found that even starting phosphazenes dissolved in deuterochloroform dried over molecular sieves partially hydrolyzed on storage.

We tried to isolate products of complete hydrolysis using phosphazene L^2 as an example. To this end, we dissolved 50 mg of L^2 in acetone-d_6, added a little of water, and recorded ^{31}P NMR spectra every 10 min. The intensity of spectral signals corresponding to hydroxyphenoxyphosphazene derivatives increased within 3 h, and further the spectral pattern remained unchanged. Even when the solution was let to stand for a month, phenoxy groups were not completely replaced by hydroxyls, and the phosphazene rings did not decompose. Under these conditions, coordinated phosphazenes are not hydrolyzed, and the ^{31}P NMR spectrum of the complex [PtL^1Cl_2] in acetone-d_6 does not change with time.

EXPERIMENTAL

The IR spectra were measured on a Nicolet SXC-5 spectrometer in KBr. The NMR spectra were recorded on Jeol LA-400 (^{31}P, 162 MHz, external standard), Bruker AM-250 (^1H and ^{13}C), and Bruker WM-360 (^{31}P, 145 MHz, external standard). The mass spectra were taken on EI-Varian MAT-711 and Varian MAT-112 mass spectrometers. The FAB(+) spectra were obtained on a Varian MAT-CH5 instrument. The elemental analysis (C, H, N) was carried out on a Perkin–Elmer CHN-Analyzer-240 instrument. Analysis for platinum was performed by a known procedure involving sulfuric acid digestion.

The phosphazene ligands were obtained by the procedure in [8] and platinum nitrile complexes [$Pt(RCN)_2Cl_2$], by the known procedure [12]. Chloroform was dried over molecular sieves according to the procedure in [13].

To develop the procedure for preparing complexes, we tried to heat the ligand L^1 with the complexes [$Pt(MeCN)_2Cl_2$], [$Pt(QuinS)_2$] (QuinS = 8-mercaptoquinoline), [$Pt(PPh3)_2Cl_2$], and [$Pt(Py)_2Cl_2$] to substitute their ligands by L^1. The starting complexes (10 mg) were dissolved in acetonitrile, chloroform, acetonitrile–chloroform (1 : 1), and chloroform, respectively. A solution of phosphazene (10 mg in a few drops of chloroform) was added to the resulting solution, and the reaction mixture was heated for ~30 min at 50°C. It is only with the starting platinum(II) acetonitrile complex that noticeable reaction occurred (the solution changed color and a new spot appeared in the chromatogram). Nevertheless, all solutions were evaporated, washed out with CCl_4 to remove unreacted phosphazene, and dried. The IR spectra gave evidence to show that phosphazene coordination took place with [$Pt(MeCN)_2Cl_2$] only.

All further reactions were carried out with platinum(II) and platinum(IV) nitrile complexes [14, 15].

Dichloropentaphenoxy(2-pyridylmethylamino)-cyclotriphosphazeneplatinum(II) [PtL^1Cl_2]. *a.* A solution of 85 mg of pentaphenoxy(2-pyridylmethylamino)cyclotriphosphazene in 2 ml of dichloromethane was added to a solution of 40 mg of [$Pt(EtCN)_2Cl_2$] in 2 ml of dichloromethane. The reaction mixture was heated for 4 h at 50°C. After cooling, the solution was filtered, and the solvent was removed. The oily orange residue was recrystallized from dichloromethane–ether mixture. Yield 39.2 mg (37%), yellow transparent

crystals, mp 190°C. Mass spectrum (*M* 973.6), *m/z*: 974 [*M* + H], 938 [*M* – HCl], 902 [*M* – 2HCl], 708, 600, 507, 383, 307, 217, 154, 107, 77. Found, %: C 42.27; H 3.25; N 7.13; Pt 20.98. $C_{36}H_{32}Cl_2N_5O_5P_3Pt$. Calculated, %: C 44.39; H 3.28; N 7.19; Pt 20.04.

b. To 0.3 g of the complex $[Pt(MeCN)_2Cl_2]$ in 4 ml of acetonitrile, a solution of 0.761 g of pentaphenoxy-(2-pyridylmethylamino)cyclotriphosphazene in 6 ml of acetonitrile was added. The reaction mixture was heated for 1.5 h at 55–60°C. The solution was evaporated to a minimal volume, its color changed from dark red from lemon yellow. To the resulting oily substance, 10 ml of chloroform–benzene (1 : 1) was added, and the solution was left for crystallization for several days. The crystals that formed were washed with chloroform–benzene. Yield 120 mg (25%).

c. A solution of 0.258 g of pentaphenoxy(2-pyridylmethylamino)cyclotriphosphazene in 10 ml of acetonitrile was gradually added with stirring and heating at 60°C to a solution of 0.125 g of $[Pt(MeCN)_2Cl_4]$ in 5 ml of acetonitrile. The resulting solution was heated for 3 h. The pale yellow color of the solution changed to dark red. Crystals of the complex were isolated from dichloromethane–hexane. Yield 52 mg (16%).

d. $[Pt(EtCN)_2Cl_4]$, 0.1 g, was dissolved in 1 ml of chloroform and mixed with a solution of 0.2 g of pentaphenoxy(2-pyridylmethylamino)cyclotriphosphazene in 0.5 ml of chloroform. Within a few minutes, a yellow precipitate appeared, and the solution turned orange. The reaction mixture was thoroughly stirred and held without heating for one day. The next day the precipitate was filtered off, washed with ether, and dried in air (yellow powder of $[PtCl_2L^1]$). Yield 15 mg (6%).

Dichloropentaphenoxy(3-pyridylmethylamino)-cyclotriphosphazeneplatinum(II) $[PtL^2Cl_2]$. *a*. Attempts to obtain this complex by the reaction of $[Pt(MeCN)_2Cl_2]$ with the corresponding phosphazene were unsuccessful.

b. $[Pt(EtCN)_2Cl_2]$, 0.15 g, was dissolved in 2 ml of chloroform, and 0.364 g of pentaphenoxy(3-pyridylmethylamino)cyclotriphosphazene in 3 ml of chloroform was added to the resulting solution. The mixture was heated for 4 h at 50°C, the solution turned intensively yellow. A little of acetone was added to the solution, and the mixture was left for crystallization (from acetone–toluene).

c. A heated solution of 0.743 g of pentaphenoxy(3-pyridylmethylamino)cyclotriphosphazene in 15 ml of acetonitrile was added to a solution of 0.36 g of $[Pt(MeCN)_2Cl_4]$ in 10 ml of acetonitrile. The reaction mixture was heated for 4 h, then evaporated, and the oily residue was recrystallized from acetone–toluene (1 : 1). Yield 56 mg (17%). Found, %: C 44.01; H 3.45; N 7.64. $C_{36}H_{32}Cl_2N_5O_5P_3Pt$. Calculated, %: C 44.39; H 3.28; N 7.19.

Dichloropentaphenoxy(2-pyridylethylamino)-cyclotriphosphazeneplatinum(II) $[PtL^3Cl_2]$. The complex $[Pt(MeCN)_2Cl_2]$, 0.3 g, was dissolved with slight heating (~30°C) in 10 ml of acetonitrile, and a heated solution of 0.762 g of pentaphenoxy(2-pyridylethylamino)cyclotriphosphazene in 20 ml of acetonitrile was added dropwise to the resulting solution. The mixture was heated for ~30 min at 60°C; therewith, its color changed from pale yellow to bright yellow. The solution was evaporated to a minimal volume. The residue was separated on a column of silica gel Chemapol L 40/100 μm, eluent acetone–chloroform (1 : 5). Crystals were grown from the middle fraction (R_f 0.8). Yield 194 mg (22.8%), mp 145°C. Mass spectrum (*M* 987.1), *m/z*: 988 [*M* + H], 951 [*M* – HCl], 915 [*M* – 2 HCl], 722, 600, 307, 217, 154, 136, 107, 89, 77. Found, %: C 45.15; H 3.50; N 6.94; Pt 19.60. $C_{37}H_{34}Cl_2N_5O_5P_3Pt$. Calculated, %: C 45.00; H 3.47; N 7.09; Pt 19.75.

Dichloropentaphenoxy(2-pyridylmethoxy)cyclotriphosphazeneplatinum(II) $[PtL^6Cl_2]$. The complex $[Pt(EtCN)_2Cl_4]$, 0.1 g, and 0.189 g of dichloropentaphenoxy(2-pyridylmethoxy)cyclotriphosphazene were mixed, and 3 ml of chloroform was added to the mixture. It was thoroughly stirred until both components dissolved completely and then heated for 1.5 h at 50°C. The solvent was then removed, and the oily residue was crystallized from dichloromethane–acetone–chloroform–toluene–carbon tetrachloride (1 : 1 : 1 : 1 : 1); R_f 0.75 (acetone–chloroform, 1 : 3). Yield 48 mg (19%), mp 161°C. Mass spectrum (*M* 974.6), *m/z*: 1969 [2*M* – 2H + Na], 1911 [2*M* – HCl], 1875 [2*M* – 2HCl], 1801 [2*M* – 4HCl], 1459 [2*M* – 4HCl – L^6], 1097 [2*M* – 4HCl – $2L^6$], 996 [*M* – H + Na], 974 [*M*], 938 [*M* – HCl], 901 [*M* – 2HCl]. Found, %: C 44.28; H 3.23; N 5.67. $C_{36}H_{31}Cl_2N_4O_6P_3Pt$. Calculated, %: C 44.37; H 3.21; N 5.75.

Modification of the polymer $[NP(OPh)R]_n$ with the complex $Pt(EtCN)_2Cl_2$. The complex $[Pt(EtCN)_2 \cdot Cl_2]$, 0.1 g, and polymeric phosphazene $[NP(OPh)R]_n$, 0.2 g, were mixed, and 2 ml dichloromethane was added to the mixture with permanent stirring of the resulting suspension. More dichloromethane (2 ml) and 3 drops of ether were then added, and the mixture was thoroughly stirred until complete dissolution, and left. In a few minutes, a dense viscous precipitate, probably of a polymeric complex, started to precipitate. It was filtered off, washed with dichlorome-

thane and ether, and dried. The compound is insoluble in usual organic solvents and represents a light yellow amorphous material. Yield 0.23 g. A broad band with its maximum at ~330 cm^{-1} is observed in the far IR spectrum, which seems to point to the presence of a Pt(II)–Cl coordination bond in the complex. Found, %: C 41.04; H 3.96; N 12.04; Pt 36.37.

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