

Synthesis and Molecular Structure of Platinum(II) Complexes with Cyclotriphosphazenes Containing Pyridylalkylamino and Pyridylmethoxy Groups

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Abstract—The interaction of platinum(II) nitrile complexes with polydentate ligands, pentaphenoxy(2-pyridylmethylamino)cyclotriphosphazene (L¹), pentaphenoxy(3-pyridylmethylamino)cyclotriphosphazene (L²), pentaphenoxy[2-(2-pyridyl)ethylamino]cyclotriphosphazene (L³), and pentaphenoxy(2-pyridylmethoxy)-cyclotriphosphazene (L⁴), was studied. The synthesized complexes were characterized by single-crystal X-ray diffraction, ¹H and ³¹P NMR spectroscopy, IR spectroscopy, FAB mass spectrometry, and other methods. In complexation of phosphazenes L¹–L³ with Pt(II) ions, nitrogen atoms of the pyridine ring and alkylamino fragment participate in the coordination to form chelate rings. In the complex with L⁴, the substituted phosphazene is coordinated via nitrogen atoms of the pyridyl group and cyclotriphosphazene ring to form a seven-membered ring.

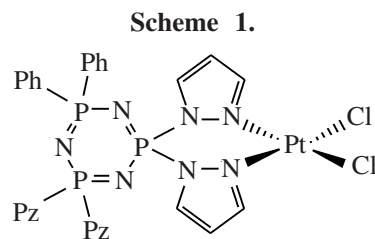
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Published data on coordination chemistry of cyclotriphosphazenes [1–6] show that phosphazenes with a large number of donor atoms such as N, P, and O are of most interest. These ligands can coordinate with metal ions in different fashions depending on the steric conditions and nature of the central ion. A systematic study of the reactivity of phosphazenes with pyridylalkylamino and pyridylalkoxy groups was made in [4, 7]. As shown by Chandrasekhar and Nagendran [6], cyclotriphosphazenes with pyrazole and dimethylamino groups coordinate with the metal ion via nitrogen atoms of the pyrazole ring and/or nitrogen atoms of the phosphazene ring. Depending on the type of the complexing ion and nature of substituents R¹ and R² in polydentate cyclotriphosphazenes (N₃P₃R_x¹R_y², x + y = 6), in each particular case the formation of diverse coordination compounds can be expected, including polynuclear complexes which are of great interest.

Platinum complexes of *cis* configuration with various cyclo- and polyphosphazene ligands containing pyridylalkylamino groups were described in [2–4]. Particularly promising is complexation of platinum with polymeric phosphazenes, because such macromolecular structures find use as catalysts and ionic

conductors. In addition, phosphazene polymers can be used as sorbents for recovering platinum from organic media. In turn, data obtained for small molecular models such as cyclotriphosphazenes give certain insight into processes occurring in reactions with polymeric ligands.

Justin Thomas et al. [5] prepared platinum(II) complexes with cyclotriphosphazenes L containing geminal pyrazole and dimethylpyrazole groups by reaction with [Pt(PhCN)₂Cl₂]. This reaction yields complexes of the composition [PtLCl₂] in which the ligand L is coordinated via nitrogen atoms of the adjacent pyrazole groups to form a six-membered chelate ring (Scheme 1).



It was found previously that cyclotriphosphazene N₃P₃(OC₆H₅)₅R, where R is 2-pyridylmethylamino

Table 1. Characteristic vibration frequencies (cm^{-1}) of R-pentaphenoxycyclotriphosphazenes and Pt(II) phosphazene complexes

Assignment	L^1	$[\text{Pt}L^1\text{Cl}_2]$	L^2	$[\text{Pt}L^2\text{Cl}_2]$	L^3	$[\text{Pt}L^3\text{Cl}_2]$	L^4	$[\text{Pt}L^4\text{Cl}_2]$
$\nu(\text{NH})$	3260 w	3293 w	3157 w	3420 w	3350	3428 w	–	–
$\nu(\text{CC}_{\text{aryl}})$	1580 s	1590 m	1585 s	1592 m	1590 s	1589 m	1590 s	1589 m
$\nu(\text{CC}_{\text{aryl}})$	1476 s	1488 s	1485 s	1490 s	1480 s	1487 s	1490 s	1488 s
$\nu(\text{PN})$	1216–	1260–	1220–	1180–	1210–	1179–	1200–1120 vs	1180–1120 vs
	1112 vs	1177 vs	1120 vs	1120 vs	1120 vs	1164 vs		
$\nu(\text{PO}_{\text{aryl}})$	940 s	945 s	940 s	944 s	940 s	941 s	950 s	958 s
$\nu(\text{CN})$	1580	1612 m	1585	1652 m	1590	1605 m	1590	1589 m

group, forms complexes with Cu(II) and Pt(II) with a metal to ligand ratio of 1 : 2 (Cu^{2+}) or 1 : 1 (Pt^{2+}), with both nitrogen atoms of the pyridylmethylamino fragment of the phosphazene ligand participating in the complexation [4]. With Co(II), the complex is formed with one $\text{N}_3\text{P}_3(\text{OC}_6\text{H}_5)_5\text{R}$ molecule which is coordinated via nitrogen atom of the phosphazene ring and pyridyl nitrogen atom. In complexes of metals with L^2 and L^4 , the metal ion always coordinates exclusively with the pyridine group [8–10]. Preparation of the complex $[\text{Pt}L^1\text{Cl}_2]$ from PtCl_2 and L^1 was described previously in [4]. We expected that the yield of the complexes could be increased by taking the nitrile complexes $[\text{Pt}(\text{RCN})_2\text{Cl}_2]$ as starting compounds, because of high solubility of these complexes in organic solvents and lability of nitrile ligands in replacement reactions.

In this study we examined the reactions of Pt(II) aceto- and propionitrile complexes $[\text{Pt}(\text{MeCN})_2\text{Cl}_2]$ and $[\text{Pt}(\text{EtCN})_2\text{Cl}_2]$ with cyclotriphosphazenes of the general formula $\text{N}_3\text{P}_3(\text{OC}_6\text{H}_5)_5\text{R}$, where R is 2-pyridylmethylamino (L^1), 3-pyridylmethylamino (L^2), 2-(2-pyridyl)ethylamino (L^3), or 2-pyridylmethoxy (L^4) group and analyzed the crystal structures of the complexes obtained. This paper continues our previous studies [11, 12]. Platinum(II) complexes of the general formula $[\text{Pt}L^n\text{Cl}_2]$ were prepared by heating *cis*- $[\text{Pt}(\text{RCN})_2\text{Cl}_2]$ (R = Me, Et) with appropriate cyclotriphosphazene L^n in 1 : 1.1 ratio in acetonitrile or dichloromethane. The compounds were characterized by IR spectroscopy, ^1H and ^{31}P NMR spectroscopy, FAB mass spectrometry, and elemental analysis (C, H, N, Pt). Particular attention was given to X-ray diffraction study of the complexes $[\text{Pt}L_3\text{Cl}_2]$ and $[\text{Pt}L_4\text{Cl}_2]$.

In the IR spectra of the compounds $[\text{Pt}L^n\text{Cl}_2]$ ($L^n = L^1-L^4$, Table 1), the band of stretching vibrations of the C=N bonds of the pyridine fragment shifts from 1580–1590 (for free phosphazene) to 1600–1650 cm^{-1} (for the complex), which suggests, according to published data [7], participation of the pyridine nitrogen

atom in the coordination. The characteristic bands in the IR spectra of Pt(II) complexes with cyclotriphosphazenes (L^1-L^4) and of the free phosphazenes are given in Table 1.

In the ^1H NMR spectrum of $[\text{Pt}L^3\text{Cl}_2]$ (Fig. 1), all the signals are shifted upfield, and the CH_2 protons of the phosphazene ligand become nonequivalent. This nonequivalence is attributable to closure of a nonplanar six-membered ring. In the ^{31}P NMR spectra of $[\text{Pt}L^3\text{Cl}_2]$, the signals (X_2B pattern) do not shift relative to the free ligand, which indicates that the nitrogen atoms of the phosphazene ring do not participate in the complexation [δ_{P} , ppm: 7.12 d (P_X , J_{XB} 69.8 Hz), 7.65 d (P_X , J_{XB} 82.8 Hz), and 21.33 d.d. (P_B , J_{XB} 74.1 Hz, J_{XB} 80.7 Hz)].

The structure of $[\text{Pt}L^3\text{Cl}_2]$ was proved by single crystal X-ray diffraction (Fig. 2). The structural data and measurement conditions are given in Table 2. The selected bond lengths and angles are given in Table 3. The nearest surrounding of the central Pt^{2+} ion in this compound is almost undistorted planar square, and all the bond lengths and angles in the coordination core are close to those reported previously for $[\text{Pt}L^1\text{Cl}_2]$ [4]. The bond lengths and angles in the P_3N_3 ring are close to those reported for noncoordinated pyridylalkylaminocyclotriphosphazenes [7].

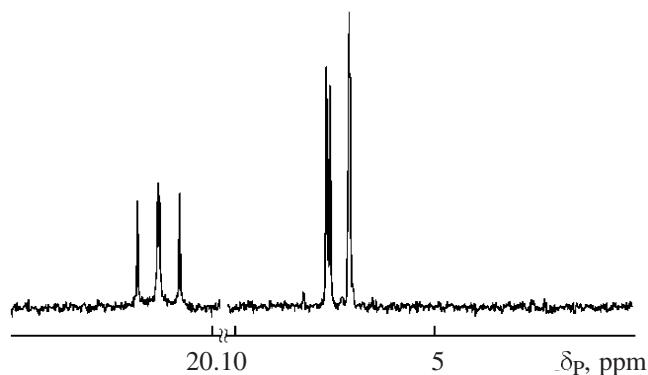
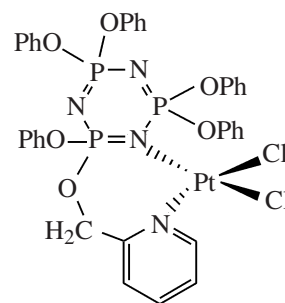
**Fig. 1.** ^{31}P NMR spectrum of $[\text{Pt}L^3\text{Cl}_2]$.

Table 2. Crystallographic data and unit cell parameters for [PtL³Cl₂] and [PtL⁴Cl₂]

Parameter	[PTL ³ Cl ₂]	[PTL ⁴ Cl ₂]
Formula	C ₃₇ H ₃₄ Cl ₂ N ₅ O ₅ P ₃ Pt	C ₃₆ H ₃₁ Cl ₂ N ₄ O ₆ P ₃ Pt
Molecular weight	987.59	974.55
Crystal size, mm	0.32 × 0.11 × 0.06	0.44 × 0.14 × 0.10
Temperature, K	293(2)	293(2)
Wavelength, Å	0.71073	0.71073
Crystal system	Triclinic	Triclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> $\bar{1}$
<i>a</i> , Å	10.385(2)	10.627(2)
<i>b</i> , Å	12.127(2)	12.304(2)
<i>c</i> , Å	15.413(3)	15.018(3)
α , deg	90.97(3)	92.09(3)
β , deg	95.87(3)	103.92(3)
γ , deg	90.37(3)	91.53(3)
Volume, Å ³	1930.6(6)	1903.5(6)
<i>Z</i>	2	2
Density, g cm ⁻³	1.699	1.700
μ , mm ⁻¹	3.946	4.002
Number of unique reflections	7177	6727
Number of reflections with <i>I</i> > 2 σ (<i>I</i>)	6765	6322
Final <i>R</i> 1	0.0227	0.0368
<i>wR</i> 2	0.0523	0.0867

The reaction of [Pt(EtCN)₂Cl₂] with phosphazene L⁴ containing pyridylmethoxy groups yields the complex [PtL⁴Cl₂] in which the ligand L⁴ is coordinated via nitrogen atoms of the pyridine and phosphazene rings to form a seven-membered ring (Scheme 2). This coordination mode could be expected, because of low affinity of methoxyl oxygen for Pt(II).

The ³¹P NMR spectrum of [PtL⁴Cl₂] (Fig. 3) differs essentially from the ³¹P NMR spectra of complexes with ligands L¹–L³. In this case, we deal with a strongly coupled ABC system, which convincingly proves the nonequivalence of all the three phosphorus atoms of the phosphazene ring and hence coordination of Pt²⁺ via phosphazene nitrogen atom. This conclu-

Scheme 2.

sion is proved by a single crystal X-ray diffraction study of [PtL⁴Cl₂] (Fig. 4; Tables 2, 3).

The nearest surrounding of the Pt²⁺ ion in [PtL⁴Cl₂] is square-planar, as in [PtL³Cl₂]. Two P–N bonds in the phosphazene ring are somewhat longer [P¹–N¹ and P²–N¹ bond lengths are 1.623(5) and 1.605(5) Å] than in the noncoordinated phosphazene L⁴ [7]. This may be due to involvement of the lone electron pair of the phosphazene nitrogen atom in a donor–acceptor bond with the platinum ion and to possible distortion of the “quasi-aromaticity” of the phosphazene ring. Similar effect was found previously in the complex of Co(II) with L¹ in which the ligand L¹ is also coordinated via one of the nitrogen atoms of the phosphazene ring and pyridine nitrogen atom [4]. It is interesting that the reaction of L⁴ with copper(II) nitrate yields a complex of trans structure in which two ligands are coordinated with the Cu(II) ion in the mono-

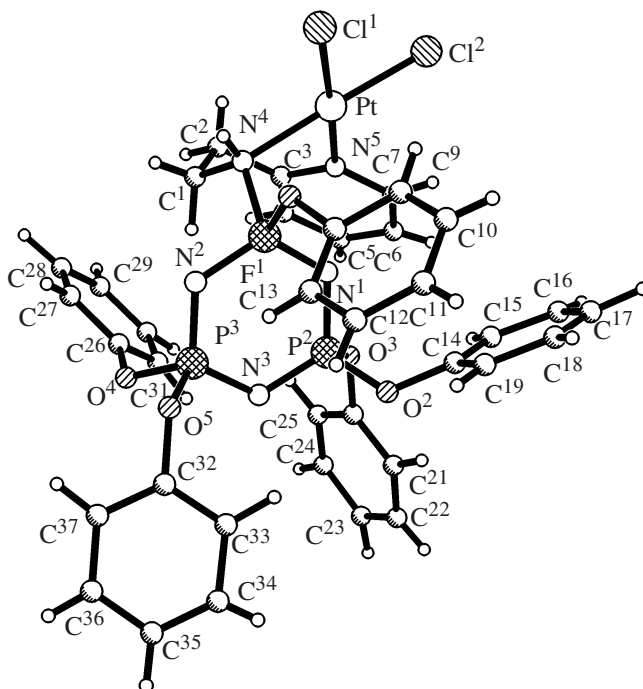
**Fig. 2.** Molecular structure of [PtL³Cl₂].

Table 3. Selected bond lengths (d) and bond angles (ω) in $[\text{PtL}^3\text{Cl}_2]$ and $[\text{PtL}^4\text{Cl}_2]$

Bond	d , Å	Bond	d , Å	Angle	ω , deg	Angle	ω , deg
Pt–N ⁵	2.016(3)	P ³ –O ⁴	1.579(3)	N ⁵ PtN ⁴	88.41(14)	O ¹ P ¹ N ¹	110.1(3)
Pt–N ⁴	2.092(3)	P ³ –N ²	1.584(3)	N ⁵ PtCl ²	89.83(10)	N ³ P ² O ³	110.3(3)
Pt–Cl ²	2.2821(12)	C ¹ –C ²	1.500(6)	N ⁴ PtCl ²	177.90(11)	N ³ P ² O ⁴	114.1(3)
Pt–Cl ¹	2.3012(13)	C ² –C ³	1.499(6)	N ⁵ PtCl ¹	176.55(10)	O ³ P ² O ⁴	100.7(3)
P ¹ –O ¹	1.573(3)	Pt–N ¹	2.075(5)	N ⁴ PtCl ¹	90.61(11)	N ³ P ² N ¹	115.5(3)
P ¹ –N ¹	1.574(3)	Pt–N ⁴	2.019(6)	Cl ² PtCl ¹	91.21(5)	N ² P ¹ N ⁴	109.40(16)
P ¹ –N ²	1.578(3)	Pt–Cl ²	2.267(2)	O ¹ P ¹ N ¹	112.22(17)	N ³ P ² O ³	110.75(17)
P ¹ –N ⁴	1.724(3)	Pt–Cl ¹	2.280(2)	O ¹ P ¹ N ²	111.06(17)	N ³ P ² N ¹	118.69(17)
P ² –N ³	1.567(3)	P ¹ –N ²	1.554(6)	N ¹ P ¹ N ²	117.51(17)	O ³ P ² N ¹	105.03(17)
P ² –O ³	1.581(3)	P ¹ –N ¹	1.623(5)	O ¹ P ¹ N ⁴	95.76(16)	N ³ P ² O ²	106.54(16)
P ² –N ¹	1.582(3)	P ² –N ³	1.559(6)	N ¹ P ¹ N ⁴	108.64(18)	O ³ P ² O ²	104.91(15)
P ² –O ²	1.592(3)	P ² –N ¹	1.605(5)	N ⁴ PtN ¹	89.7(2)	N ¹ P ² O ²	110.14(16)
P ³ –N ³	1.576(3)	P ³ –N ³	1.581(6)	N ⁴ PtCl ²	177.53(17)	N ³ P ³ O ⁵	111.38(16)
P ³ –O ⁵	1.578(3)			N ¹ PtCl ²	90.90(16)	N ³ P ³ O ⁴	110.29(17)
				N ⁴ PtCl ¹	89.01(16)	O ⁵ P ³ O ⁴	99.09(15)
				N ¹ PtCl ¹	177.36(16)	N ³ P ³ N ²	117.40(17)
				P ² N ¹ Pt	120.5(3)	O ⁵ P ³ N ²	106.19(17)
				Cl ² PtCl ¹	90.53(7)	O ⁴ P ³ N ²	110.89(16)
				P ¹ N ¹ Pt	116.8(3)	O ³ P ² N ¹	111.6(3)
				C ⁶ N ⁴ Pt	119.1(5)	O ⁴ P ² N ¹	103.5(3)
				C ² N ⁴ Pt	122.4(5)	O ⁶ P ³ N ²	109.0(3)
				N ² P ¹ O ²	113.1(3)	O ⁶ P ³ O ⁵	95.6(3)
				N ² P ¹ O ¹	105.7(3)	O ⁵ P ³ N ²	112.3(3)
				O ² P ¹ O ¹	106.4(3)	O ⁶ P ³ N ³	113.4(3)
				N ² P ¹ N ¹	116.3(3)	O ⁵ P ³ N ³	110.0(3)
				O ² P ¹ N ¹	104.8(3)	N ² P ³ N ³	115.0(3)
				P ² N ¹ P ¹	122.0(3)	P ¹ N ² P ³	123.8(4)
				P ² N ³ P ³	124.9(4)		

dentate fashion via pyridine nitrogen atoms [6]. The Pt–N and Pt–Cl bond lengths in $[\text{PtL}^4\text{Cl}_2]$ are close to those found in $[\text{PtL}^1\text{Cl}_2]$ and $[\text{PtL}^3\text{Cl}_2]$ (Table 3), and also in some other related compounds [4, 7, 13]. The steric effect of the 2-pyridylmethoxy group linked to the phosphazene ring additionally favors formation of a square-planar *cis* complex with the Pt(II) : ligand ratio of 1 : 1.

EXPERIMENTAL

Cyclotriphosphazenes $\text{L}^1\text{--L}^4$ were prepared by the procedure described in [1, 7]. The starting platinum(II) nitrile complexes *cis*- $[\text{Pt}(\text{RCN})_2\text{Cl}_2]$ (R = Me, Et) were synthesized as described in [14].

The IR spectra of the compounds were recorded on a 55XC Nicolet spectrometer. Samples were prepared as KBr pellets. The NMR spectra were recorded on the following devices: ³¹P, Jeol LA 400 (162 MHz, with external reference; ¹H and ³¹P, Bruker AM 250

and Bruker WM 360 (145 MHz for ³¹P, with external reference). The mass spectra were taken on the following mass spectrometers: EI, Varian MAT 711 and Varian MAT 112; FAB⁺, Varian MAT CH5. Elemental analysis (C, H, N) was performed with a Perkin–Elmer CHN Analyzer 240; the platinum content was determined by the standard procedure involving decomposition of samples with sulfuric acid. The crystal structures of the complexes were determined with Enraf–Nonius CAD-4 automatic four-circle diffractometers (Karpov Institute of Physical Chemistry, Moscow, Russia).

The complexes were prepared by the procedures described in [11, 12].

Dichloro[pentaphenoxy(2-pyridylmethylamino)-cyclotriphosphazene]platinum, $[\text{PtL}^1\text{Cl}_2]$. A solution of 85 mg of pentaphenoxy(2-pyridylmethylamino)-cyclotriphosphazene in 2 ml of dichloromethane was added to a solution of 40 mg of $[\text{Pt}(\text{EtCN})_2\text{Cl}_2]$ in 2 ml of dichloromethane. The mixture was refluxed at

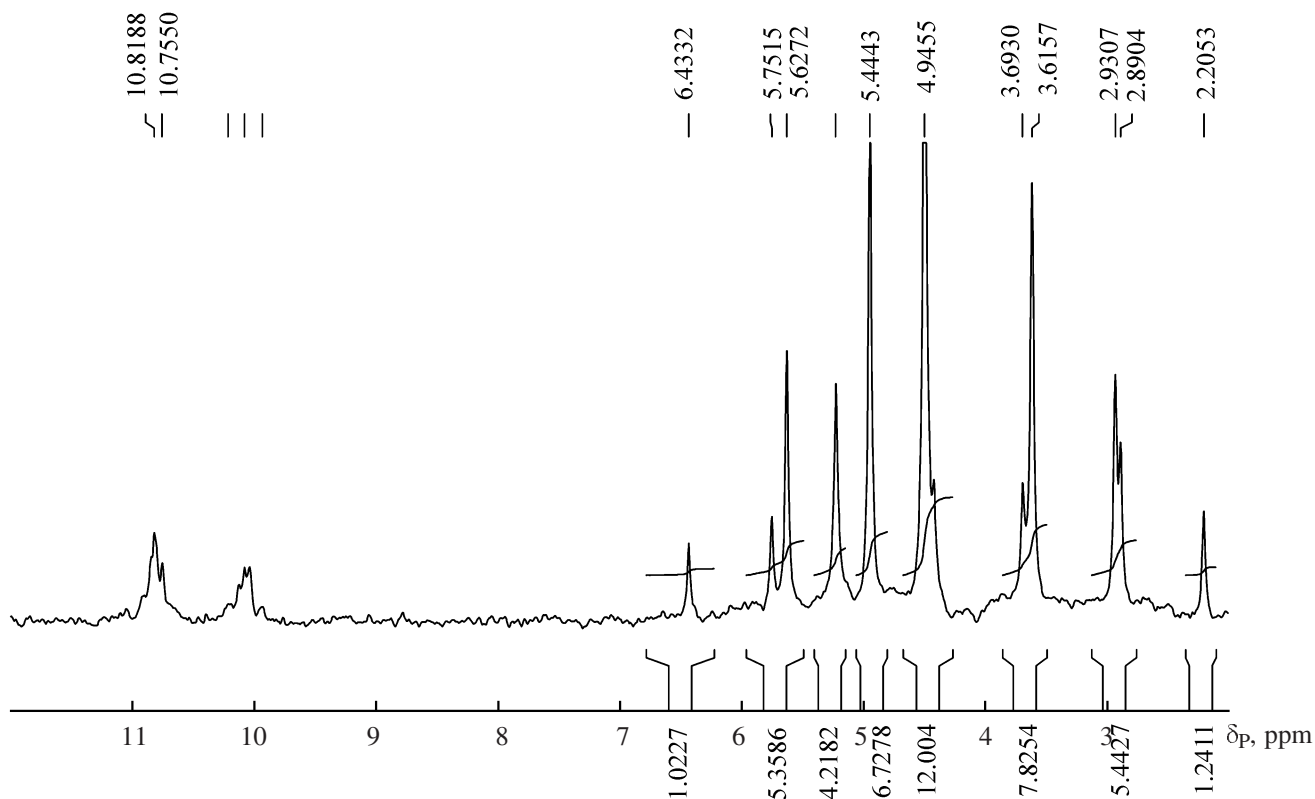


Fig. 3. ^{31}P NMR spectrum of $[\text{PtL}^4\text{Cl}_2]$.

50°C for 4 h. After cooling, the solution was filtered and the solvent was removed. The resulting orange oil was recrystallized from dichloromethane–diethyl ether to obtain yellow transparent crystals. Yield 39.2 mg

(37%), mp 190°C. Mass spectrum [M 973.6]; m/z : 974 [$M + \text{H}$], 938 [$M - \text{HCl}$], 902 [$M - 2\text{HCl}$], 708, 600, 507, 383, 307, 217, 154, 107, 77. Found, %: C 42.27; H 3.25; N 7.13; Pt 20.98. $\text{C}_{36}\text{H}_{32}\text{Cl}_2\text{N}_5\text{O}_5\text{P}_3\text{Pt}$

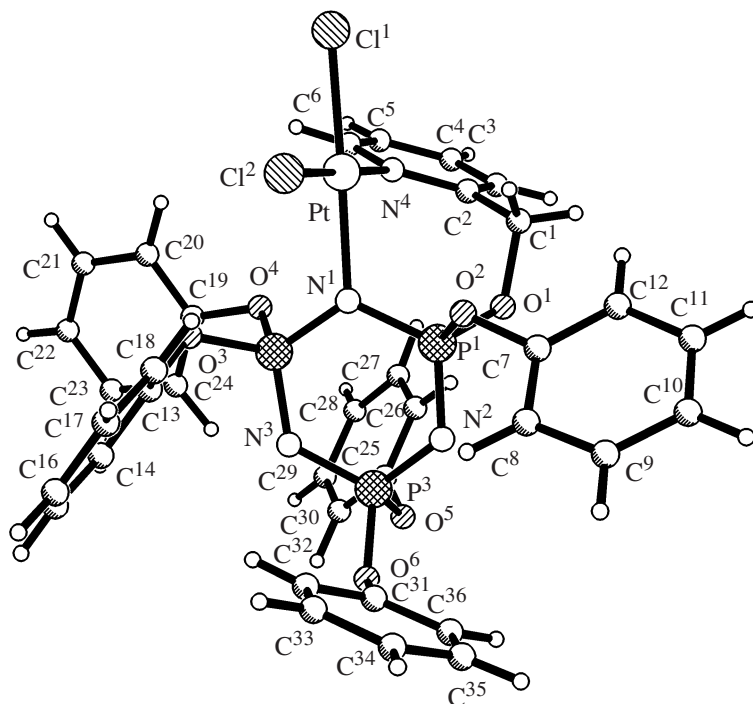


Fig. 4. Molecular structure of $[\text{PtL}^4\text{Cl}_2]$.

(973.6). Calculated, %: C 44.39; H 3.28; N 7.19; Pt 20.04.

Dichloro[pentaphenoxy(3-pyridylmethylamino)cyclotriphosphazene]platinum, [PtL²Cl₂]. A preheated solution of 0.743 g of pentaphenoxy(3-pyridylmethylamino)cyclotriphosphazene in 15 ml of acetonitrile was added to a solution of 0.360 g of [Pt(MeCN)₂Cl₂] in 10 ml of acetonitrile. The mixture was heated for 4 h, after which it was evaporated and the resulting oil was recrystallized from acetone–toluene, 1 : 1. Yield 56 mg (17%). Found, %: C 44.01; H 3.45; N 7.64. C₃₆H₃₂Cl₂N₅O₅P₃Pt. Calculated, %: C 44.39; H 3.28; N 7.19.

Dichloro{pentaphenoxy[2-(2-pyridyl)ethylamino]cyclotriphosphazene}platinum, [PtL³Cl₂]. An 0.300-g portion of [Pt(MeCN)₂Cl₂] was dissolved with slight heating (~30°C) in 10 ml of acetonitrile, and a solution of 0.762 g of pentaphenoxy[2-(2-pyridyl)ethylamino]cyclotriphosphazene in 20 ml of acetonitrile, heated to 40°C, was added dropwise. The mixture was heated for approximately 0.5 h at 60°C; in so doing, the color of the reaction mixture changed from pale yellow to bright yellow. After that, the solvent was evaporated to a minimal volume. The mixture was separated on a column packed with silica gel L 40/100 μm (Chemapol), eluent acetone–chloroform, 1 : 5. The crystals were grown from the middle fraction, R_f 0.8, acetone–chloroform, 1 : 5. 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. C₃₇H₃₄Cl₂N₅O₅P₃Pt (973.6). Calculated, %: C 45.00; H 3.47; N 7.09; Pt 19.75.

Dichloro[pentaphenoxy(2-pyridylmethoxy)cyclotriphosphazene]platinum, [PtL⁴Cl₂]. Weighed portions of [Pt(EtCN)₂Cl₄] (0.1 g) and pentaphenoxy(2-pyridylmethoxy)cyclotriphosphazene (L⁴, 0.189 g) were mixed in a chloroform solution (3 ml). The resulting mixture was thoroughly stirred until the components dissolved completely, after which it was heated at 50°C for 1.5 h. Then the solvent was removed, and the oily residue was crystallized from dichloromethane–acetone–chloroform–toluene–CCl₄, 1 : 1 : 1 : 1 : 1. R_f 0.75, acetone–chloroform, 1 : 3. Yield 48 mg (18.5%), mp 161°C. Mass spectrum (EI/70 eV) [M 974.6]; m/z: 1969 [2M – 2H + Na, 9%], 1911 [2M – HCl, 27%], 1875 [2M – 2HCl, 4%], 1801 [2M – 4HCl, 2%], 1459 [2M – 4 HCl – L⁴, 15%], 1097 [2M – 4HCl – 2L⁴, 4%], 996 [M – H + Na, 21%], 974 [M, 8%], 938 [M – HCl, 39%], 902 [M – 2HCl, 100%], 808 [25%]. Found, %: C 44.28; H 3.23;

N 5.67. C₃₆H₃₁Cl₂N₄O₆P₃Pt (974.56). Calculated, %: C 44.37; H 3.21; N 5.75.

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