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COMPLEXES OF CYCLIC AMINOMETHYLPHOSPHINES WITH Pt(II), Pd(II), Cu(I), AND Ag(I) SALTS

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1,3,5-Diazaphosphorinanes and 1,5,3,7-diazadiphosphacyclooctanes form complexes with Pt(II), Pd(II), Cu(I), and Ag(I) salts. Platinum and palladium are coordinated through phosphorous atoms. In the case of 1,3-diphenyl-5-p-toluidenomethyl-1,3,5-diazaphosphorinane complex formation with platinum and palladium is accompanied by formation of a new polydentate ligand, p-tolylbis-(1,3-di-p-tolyl-1,3,5-diazaphosphorinane-5-yl)methylamine, where the metal is also bonded to phosphorus atoms.

Keywords: 1,3,5-diazaphosphorinanes, 1,5,3,7-diazadiphosphacyclooctanes, complexes, platinum, palladium, ring conformation.

Functionally substituted phosphines have several coordination centers and are able to undergo different conversions in the process of complex formation. Aminomethyl derivatives of phosphines containing a P—C—N moiety possess these qualities. Moreover, published data on their chemical properties and structure and the possibility of using a number of physicochemical methods for this class of compounds make it possible to draw conclusions regarding the structure of their complexes in solution and in the solid state. It has been shown previously [1, 2] that heterocycles with a P—C—N moiety, containing a $PhCH_2$ group at the nitrogen atoms, exist in a conformational equilibrium in which there is a form with axial orientation of the substituent at the nitrogen atom and equatorial at the phosphorus atom. In complexes of cyclic aminomethylphosphines with BH, the number of added borane

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*Deceased.

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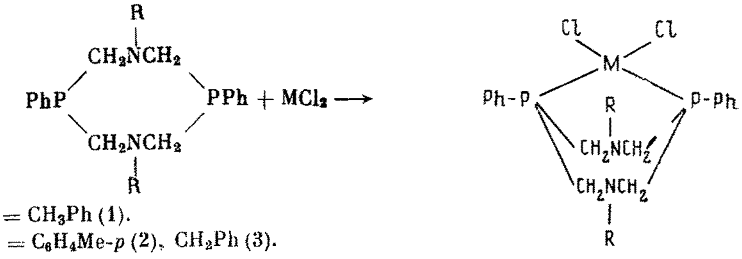
molecules depends on the conformational composition of the heterocycles [3]. We have described previously [4] a method for synthesis of the complex trans-$(PhP(CH_2NRCH_2)_2PPh)PtCl_2$, where R = C_6H_4Me-p. It appeared that replacing the tolyl groups by benzyl at the nitrogen atoms in 1,5,3,7-diazadiphosphacyclooctanes leads to a change in the configuration of the central atom in the complex with $PtCl_2$:



M = Pt, R = CH_2Ph (1).
M = Pd, R = C_6H_4Me-p (2), CH_2Ph (3).

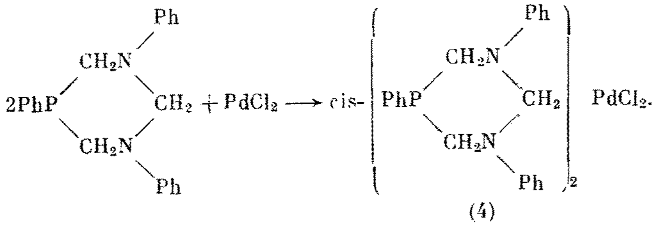
(1)-(3)

The ^{31}P NMR spectra of solutions of the cis-$(PhP(CH_2NRCH_2)_2PPh)PtCl_2$ complex (1) display one signal with a chemical shift (CS) of −26 ppm and SSCC with the platinum of 2929 Hz. The occurrence of one signal and its significant displacement compared with the CS of the original ligand (−50 ppm) indicate coordination of the platinum through a phosphorus atom and the SSCC $^1J_{P-Pt}$ value is characteristic of a cis complex (see [4], R = C_6H_4Me-p, δ = 13 ppm, J_{P-Pt} = 2297 Hz). The IR spectra of crystalline complex 1 (dark-brown crystals) also indicate a cis configuration of the Pt atom: ($\nu Pt-Cl_{cis}$) 315, 326 cm^{-1} (two bands), in contrast with $\nu Pt-Cl_{trans}$ 313 cm^{-1} (one band) [5]. No further Pt–P–Pt interaction is observed in the ^{31}P NMR spectra which makes it possible to rule out the dimeric structure of a binuclear cluster-type complex. Moreover, a binuclear cis,cis complex should experience strong steric interactions. The difference in structure of the 1,5,3,7-diazadiphosphacyclooctane complexes with $PtCl_2$ when R = C_6H_4Me-p and CH_2Ph may be explained either by a difference in the conformational composition of the heterocycles (the orientation of substituents) or by conformational features of the heterocycles [6].

Differences in the configuration of the central atom have not been noted in the 1,5,3,7-diazadiphosphacyclooctane complexes with $PdCl_2$.

The IR spectra of the crystalline complexes 2 (pale-yellow crystals) and 3 (orange crystals) contain double ν(Pd–Cl) bands, 289, 303 cm^{-1} for 2 and 293, 316 cm^{-1} for 3, which indicates a cis configuration for the Pd–Cl bonds. The occurrence of one signal each in the ^{31}P NMR spectra and the size of the CS (2.37 ppm for 2 and −9.6 ppm for 3) suggest coordination of the Pd through phosphorus atoms. Data from elemental analysis, the IR spectra of the crystals, and also the ratio of integral intensities of all types of protons in the PMR spectra reveal no change in the structure of the ligand.

A cis complex with coordination of the metal through a phosphorus atom is formed by interaction of 1,3,5-triphenyl-1,3,5-diazaphosphorinane with $PdCl_2$. The metal is bonded to two ligand molecules:

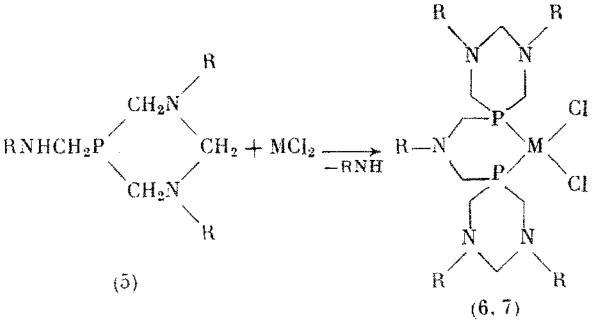


(4)

The IR spectra of the crystalline complex 4 (orange crystals) have ν(Pd–Cl) bands at 302 and 277 cm^{-1} and the ^{31}P NMR spectra have a signal with a CS of 1.37 ppm.

Of special interest are cases when changes occur during synthesis of the complexes which are uncharacteristic of the free ligands. For example, reactions are known for 1,3,5-diazaphosphorinanes with an aminomethyl radical at the P atom which leads to tris(aminomethyl)phosphines and 1,1'-diaza-3,3'-diphosphetidines [7, 8].

During formation of complexes of 1,3-di-p-tolyl-5-p-toluidinomethyl-1,3,5-diazaphosphorinane (5) with $PtCl_2$ and $PdCl_2$ yet another reaction path is followed which is not known for these compounds. "Dimerization" of the ligands occurs as a result of separation of p-toluidine with the formation of cis-$RN(CH_2P(CH_2NR)_2CH_2)_2PtCl_2$ (6) and cis-$RN(CH_2P(CH_2NR)_2CH_2)_2PdCl_2$ (7) complexes:



(5)

(6, 7)

R = p-MeC$_6$H$_4$, M = Pt (6), Pd (7).

This reaction may be considered as the initial stage in the synthesis of macrocyclic compounds taking place at a metal atom which fulfills the function of a matrix.

Complexes 6 and 7 are pale-grey and pale-yellow crystalline substances, respectively. Their IR spectra contain no absorption bands for N—H groups (the spectrum of the original ligand has the characteristic νN—H absorption at 3430 cm^{-1}). The long-wavelength region of the IR spectrum for 6 absorbs at 309 and 292 cm^{-1} and that for 7 has a composite band with a maximum at 286 cm^{-1}. The shape of the absorption spectra is typical of Pt and Pd cis complexes. The ^{31}P NMR spectroscopic data lead to the same conclusion. These spectra each have one signal with a CS of −23.2 ppm for 6 (J_{P-Pt} = 3210 Hz) and −3.4 ppm for 7. The CS values and also the occurrence of J_{P-Pt} for SSCC in complex 6 is a definite indication of coordination of the metals through phosphorus and the equivalence of the phosphorus atoms in the complex molecule. The value of J_{P-Pt} for SSCC corresponds to a cis complex. The PMR spectra of complexes 6 and 7 have identical groups of signals (Fig. 1): two quartets of phenylene protons and two singlets of methyl protons attributed to four equivalent tolyl groups in diazaphosphorinane rings and one tolyl of the chelate moiety and also multiplets of methylene protons with an integral intensity ratio of (16:4):(12:3):14, respectively.

Certain conclusions may be drawn regarding the three-dimensional structure of the compounds in solution from analysis of the PMR spectra of the original ligand and complexes 6 and 7, using 1,3,5-diazaphosphorinanes and their derivatives as model compounds together with Erastov and Nikonov's approach for determination of the three-dimensional structure of heterocycles [9].

The low inversion barriers at the nitrogen atoms and the lack of substituents at the C atoms in the heterocycles give rise to 1,3,5-diazaphosphorinanes and their analogs existing in solution as an equilibrium mixture of conformers which differ in the orientation of substituents on the heteroatoms. This also holds for complexes 6 and 7. The form with an axial substituent at the N atom has a low probability owing to syn-axial interactions with one of the substituents at the P atom. The PMR spectra of complexes 6 and 7 represent an $(AB)_2CDX$ system typical of diazaphosphorinanes [9]. The equivalence of the methylene groups of the P—CH_2—N moieties in 1,3,2,5-diazaphosphorinane rings in pairs and between themselves and the equivalence of the CH_2 groups of P—CH_2—N moieties in the chelate ring indicate identical conformational behavior of the diazaphosphorinane rings and the symmetrical structure of the chelate ring.

The sum total of the spectroscopic characteristics and comparison of the SSCC of the original ligand 5 and complexes 6 and 7 with the SSCC for 1,3,5-diazaphosphorinanes and their oxides show that in solution ligand 5 occurs predominantly in a chair conformation with equatorial orientation of the aminomethyl grouping at the P atom. In the complexes the diazaphosphorinane rings are in a chair conformation, as follows from the equivalence of the methylene groups in the P—CH_2—N moiety and observation of "rule W" for the methylene protons in the N—CH_2—N moiety. Here the P—Pt bonds, owing to ready inversion of the rings, may be both axial and equatorial relative to the plane of diazaphosphorinane ring. From

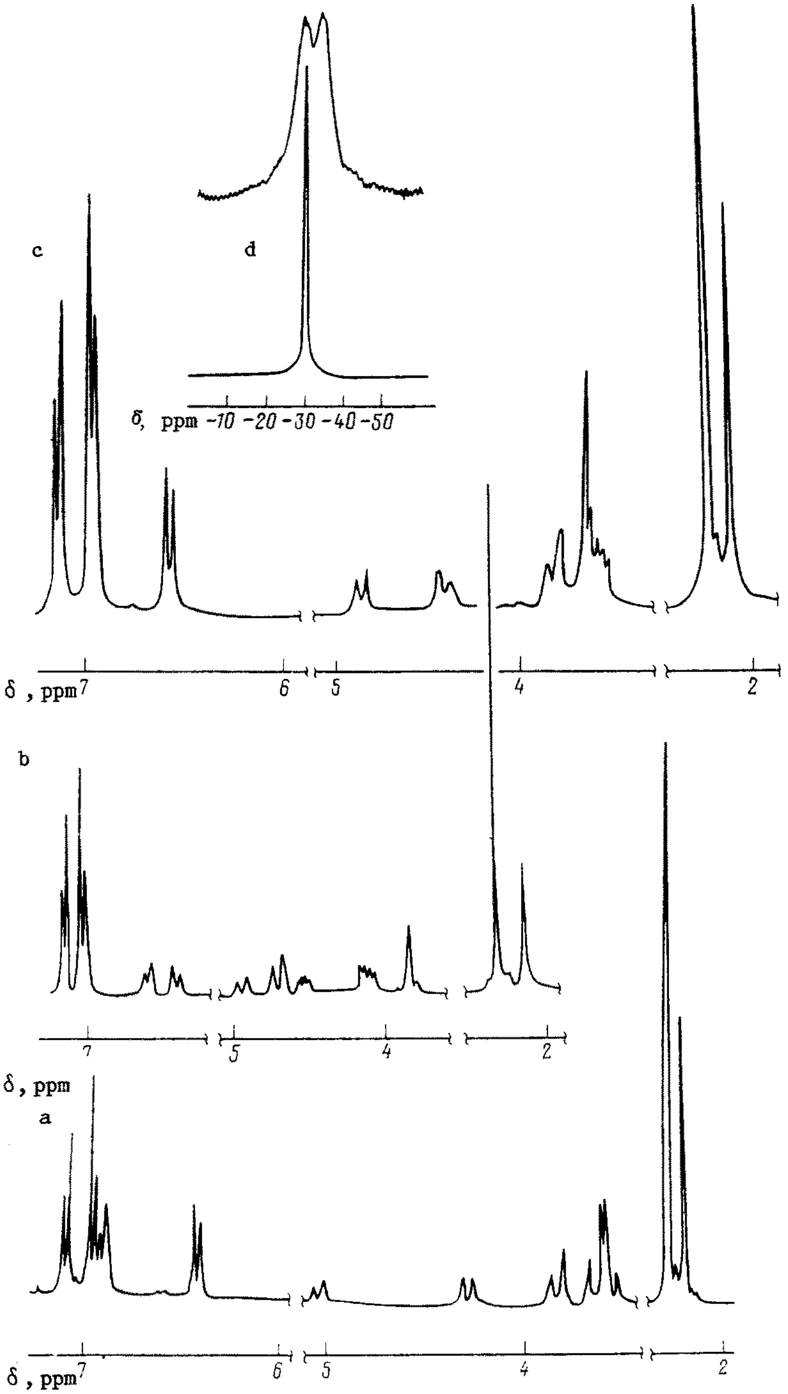
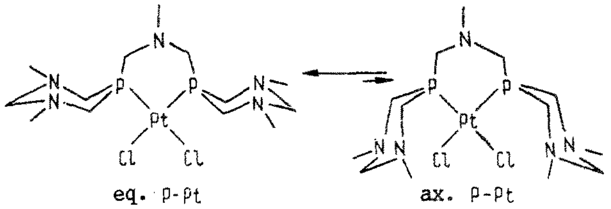


Fig. 1. ^1H (a-c) and ^{31}P (d) NMR spectra of compounds 5 (a), 6 (b), and 9 (c, d): (a-c) $CDCl_3$, (d) CH_2Cl_2 at 35°C (1) and at −90°C (2).

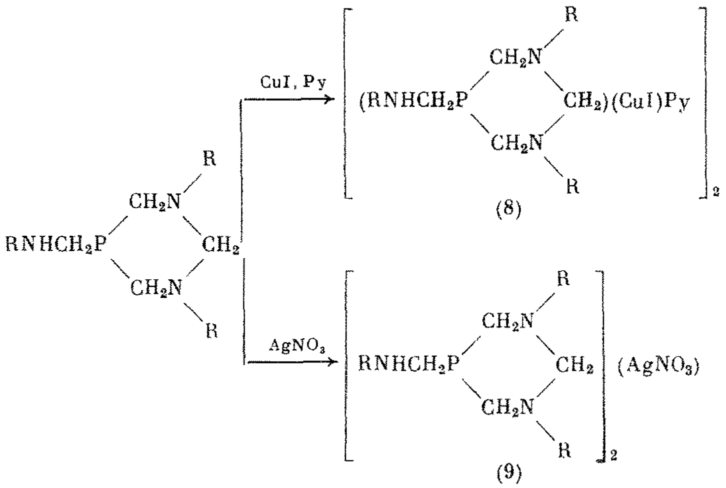
the SSCC $^2J_{PH}$ values for 6 and 7 it may be concluded that forms with equatorially directed P–Pt bonds predominate in the equilibrium. The chair conformation of the diazaphosphorinane rings and the presence of bridging P–C–N–C–P groupings give rise to a noncoplanar disposition of the planes of the P–Pt–P bonds and the diazaphosphorinane rings. Thus, formation of an M → P coordination bond leads to the same change in conformational behavior of heterocycles as in the transition from P^{III} to P=O [9] (see scheme on following page).

The low molar electrical conductance of the complexes makes it possible to rule out ionic structures.

"Dimerization" of ligand 5 occurs only in the presence of Pt or Pd. Coordination of the metal through the phosphorus probably expedites this process. When the reaction was



carried out in the presence of Cu or Ag salts the structure of the ligand did not alter. The IR spectra of the crystalline complexes with CuI (8) and with $AgNO_3$ (9) retain the characteristic absorption of the N–H bond and the main bands of the ligand skeletal vibrations. The ^{31}P NMR spectra of complex 8 contain one signal with a CS of −36 ppm (DMF) and −28.9 ppm (CH_2Cl_2). The slight displacements of the signals in the complexes relative to the signals of the original ligand do not make it possible to determine the coordination site of the metal reliably. However, it is known that in the case of Ag coordination through phosphorus SSCC $^1J_{P-Ag}$ may occur, which is observed when the spectra are measured at low temperatures. In fact, at −60°C the ^{31}P NMR spectra show a broadening of the signal and at −90°C a doublet appears (Fig. 1). Recording the spectra in similar conditions on a Bruker MSL-400 spectrometer with a ν^{31}P frequency of 162 MHz makes it possible to isolate the SSCC ~550 Hz, indicating coordination through the phosphorus.



The reaction with CuI was carried out in pyridine. Complexes 8 and 9 are white crystalline substances giving qualitative reactions for Cu(I), I, and Ag. The molecular weight of complex 8 determined ebullioscopically in benzene is 1300 (theoretical: 1345).

Thus, cyclic aminomethyl derivatives of phosphines may form complexes with Pt(II), Pd(II), Cu(I), and Ag(I) salts, those with Pt(II) and Pd(II) salts forming complexes with coordination through phosphorus. "Dimerization" of the ligand may occur when the ligand contains a functional group with a mobile hydrogen atom.

EXPERIMENTAL

The ^{1}H and ^{31}P NMR spectra were recorded on a Bruker WM-250 spectrometer with operating frequencies of ν^1H = 250 MHz and ν^{31}P = 101 MHz. The PMR spectra were recorded on a Varian T60 machine (60 MHz) at 34.5°C. Infrared spectra were measured on a UR-20 spectrometer. Spectra of the compounds under consideration in the far region of the spectrum were produced on a Bruker IFS-113v IR Fourier spectrometer over the frequency range 700-100 cm^{-1} at ~20°C. Samples were prepared by compressing the test substance with polyethylene. The number of spectrum accumulations was 256.

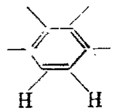
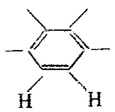
cis-[1,5-Dibenzyl-3,7-diphenyl-1,5,3,7-diazadiphosphacyclooctane]dichloroplatinum(II) (1). To 1 g (2.1 mmoles) of the corresponding diazadiphosphacyclooctane in 20 ml acetonitrile was added 0.55 g (2.1 mmoles) $PtCl_2$. The mixture was stirred until the platinum chloride had dissolved. The solution was filtered and solvent removed under vacuum. The resulting crystals were filtered and washed with acetone. Yield of 1: 0.25 g (16%), mp >350°C. ^{31}P NMR spectrum (δ, ppm): −26.4 (MeCN), J_{P-Pt} = 2929 Hz. Infrared spectrum (ν, cm^{-1}): oil 1650 (Ph), polyethylene 326, 315 (Pt–Cl). Found: C 48.12; H 4.84; N 4.24; P 7.77%. Calculated for $C_{30}H_{32}N_2P_2PtCl_2$: C 48.12; H 4.28; N 3.74; P 8.28%.

cis[(1,5-Di-p-tolyl-3,7-diphenyl-1,5,3,7-diazadiphosphacyclooctane]dichloropalladium (II) (2). To 1 g (2.1 mmoles) of diazadiphosphacyclooctane in 20 ml acetonitrile was added 0.37 g (2.1 mmoles) $PdCl_2$ and the mixture boiled for 5 h. The crystals formed on cooling were filtered and washed with acetonitrile. Yield of 2: 0.88 g (65%), mp 234-235°C. ^{31}P NMR spectra (δ, ppm): 2.4 (MeCN). Ratio of proton integral intensities in PMR spectra: $(C_6H_4 + C_6H_5):CH_2:CH_3 = 18:8:6$. Infrared spectrum (ν, cm^{-1}): oil 1615 (Ph), polyethylene 289, 303 (Pd–Cl), Found: C 54.57; H 4.93; N 4.51; P 9.41; Cl 10.27%. Calculated for $C_{30}H_{32}N_2P_2PdCl_2$: C 54.60; H 4.85; N 4.25; P 9.41; Cl 10.77%.

cis-[1,5-Dibenzyl-3,7-diphenyl-1,5,3,7-diazadiphosphacyclooctane]dichloropalladium(II) (3). Produced similarly to 2. Yield: 63%, mp 222-224°C. ^{31}P NMR spectrum (δ, ppm): −9.6 (DMF). Ratio of proton integral intensities in PMR spectra: $C_6H_5:CH_2 = 20:12$. Infrared spectrum (ν, cm^{-1}): oil 1630 (Ph), polyethylene 293, 316 (Pd–Cl). 154 (Pd–P). Found: C 54.48; H 4.95; N 4.24; P 9.44; Cl 10.71%. Calculated for $C_{30}H_{32}N_2P_2PdCl_2$: C 54.60; H 4.86; N 4.25; P 9.41; Cl 10.77%.

cis-[Bis(1,3,5-triphenyl-1,3,5-diazaphosphorinane)]dichloropalladium(II) (4). To a solution of 1.5 g (4.5 mmoles) of 1,3,5-diazaphosphorinane in 15 ml acetonitrile was added 0.41 g (2.25 mmoles) $PdCl_2$ and stirred until the $PdCl_2$ had dissolved. After 14 h the

TABLE 1. Parameters of the PMR Spectra of Compounds 5-9 [δ, ppm and J, Hz (H)*]

Grouping	Parameter	5	6	7	8	9	1,3,5-Tri-Ph-5-X-1,3,5-diazamphos phorinanes	
							X = UEP [1]	X=O [2]
1,3,5-Diazaphosphorinane ring								
	δ	7.08	7.08	7.09	7.00	7.05		
	δ	6.95	6.96	6.97	6.88	8.5		
	$^3J_{HH}$	8.5	8.5	8.5	8.5	(8H)		
	(H)	(8H)	(16H)	(16H)	(8H)	4.80	4.95	5.18
N-CH^a-N	δ	5.0	4.87	4.88	4.9	13.2	12	12
	$^2J_{PH}$	12.9	12.9	13.5	12.7	0	–	–
	$^4J_{PH}$	0	0	0	0	(1H)		
	(H)	(1H)	(2H)	(2H)	(1H)	4.39	4.55	4.53
N-CH^e-N	δ	4.28	4.26	4.54	4.26	13.2	12	12
	$^2J_{HH}$	12.9	12.9	13.5	12.7	0 **	3	3
	$^4J_{PH}$	2.8	4	0 **	0 **	(1H)		
	(H)	(1H)	(2H)	(2H)	(1H)	3.82	4.18	4.25
P-CH^a-N	δ	3.81	4.63	4.81	3.86	15.4	14	14
	$^2J_{HH}$	15.5	14.8	15.0	14.8	0 *	3.1	11.2
	$^2J_{PH}$	0	0	0	0	(2H)		
	(H)	(2H)	(4H)	(4H)	(2H)	3.59	3.91	3.87
P-CH^e-N	δ	3.58	4.05	4.16	3.65	15.4	14	14
	$^2J_{HH}$	15.4	14.8	15.0	14.8	9.0	18.1	8.8
	$^2J_{PH}$	15.7	4.5	0	9.2	(2H)		
	(H)	(2H)	(4H)	(4H)	(2H)	2.25		
Ar-CH$_3$	δ	2.27	2.25	2.26	2.25	(6H)		
	(H)	(6H)	(12H)	(12H)	(6H)			
Chelate-ring moieties								
	δ	6.89	6.52	6.44	6.80	6.87		
	δ	6.42	6.35	6.35	6.40	6.49		
	$^3J_{HH}$	8.3	7.4	8.6	8.3	8.3		
	(H)	(4H)	(4H)	(4H)	(4H)	(4H)		
P-CH$_2$-N	δ	3.59	3.77	3.73	3.55	3.66		
	$^2J_{HH}$	0	0	0	0	0		
	$^2J_{Ph}$	6.3	0	0	0	0		
	(H)	(2H)	(4H)	(4H)	(2H)	(2H)		
Ar-CH$_3$	δ	2.19	2.06	2.09	2.16	2.15		
		(3H)	(3H)	(3H)	(3H)	(3H)		

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*H = intensity.

**Band, width at half-height ≈3 Hz.

resulting crystals were filtered off and crystallized from acetonitrile. Yield of 4: 1.4 g (73%), mp 142-143°C. ^{31}P NMR spectrum (δ, ppm): 1.4 (DMF). Ratio of proton integral intensities in PMR spectra: $C_6H_5:CH_2 = 15:6$. Infrared spectrum (ν, cm^{-1}): oil 1595 (Ph), polyethylene 302, 277 (Pd—Cl), 175 (Pd—P). Found: C 60.19; H 5.02; N 6.88; P 7.49; Cl 8.69%. Calculated for $C_{42}H_{42}N_4P_2PdCl_2$: C 59.93; H 4.99; N 6.66; P 7.37; Cl 8.44%. The synthesis of ligand 5 is described in [8].

p-Tolylbis(1,3-di-p-tolyl-1,3,5-diazaphosphorinane-5-yl-methyl)aminedichloroplatinum-(II) (6). Produced similarly to 2 from 5 and $PtCl_2$, then crystallized from benzene. Yield: 80%, mp >350°C. ^{31}P NMR spectrum (δ, ppm): −23.2 (DMF), $J_{P-Pt} = 3210$ Hz. The PMR spectrum is given in Fig. 1 and Table 1. Infrared spectrum (ν, cm^{-1}): oil 1610 (Ph), polyethylene 309, 292 (Pt—Cl), 181 (Pt—P). Found: C 53.09; H 5.30; N 7.57; P 6.41; Cl 7.73%. Calculated for $C_{43}H_{51}N_5P_2PtCl_2$: C 53.47; H 5.28; N 7.25; P 6.42; Cl 7.35%.

p-Tolylbis(1,3-di-p-tolyl-1,3,5-diazaphosphorinane-5-yl-methyl)aminedichloropalladium-(II) (7). To 1.25 g (3.1 mmoles) of 5 in 20 ml chloroform was added 0.22 g (1.55 mmoles) $PdCl_2$ and stirred until the palladium salt had dissolved. The next day the solvent was removed under vacuum and the crystals were filtered off and washed with ether. Yield of 7: 1.14 g (78%), mp 228°C. ^{31}P NMR spectrum (δ, ppm): −3.4 (DMF). PMR spectrum (see Fig. 1 and Table 1). Infrared spectrum (ν, cm^{-1}): oil 1580 (Ph), polyethylene 301, 286 (Pd—Cl). Found: C 58.50; H 5.82; N 7.39; P 7.07%. Calculated for $C_{43}H_{51}N_5P_2PdCl_2$: C 58.90; H 5.82; N 7.99; P 7.07%.

Bis[(1,3-di-p-tolyl-5-toluidinomethyl-1,3,5-diazaphosphorinane)pyridineiodo]copper-(I) (8). To 3 g (7.4 mmoles) of 5 in 15 ml pyridine we added 1.42 g (7.4 mmoles) CuI. On completion of the exothermic reaction the solvent was removed under vacuum. The residue was crystallized from a 2:1 ether—acetone mixture. Yield of 8: 4.3 g (91%), mp 120-122°C. ^{31}P NMR spectrum (δ, ppm): −39.5 (DMF). PMR spectrum (δ, ppm): 8.72 d (2H, Py), 7.69 t (1H, Py), 7.30 m (2H, Py), then see Table 1. Infrared spectrum (ν, cm^{-1}): oil 1610 (Ph), 3290 (N—H). Found: C 53.19; H 5.45; N 0.08; P 4.80%. Calculated for $[C_{30}H_{35}N_4PCuI]_2$: C 53.49; H 5.20; N 8.32; P 4.60%.

Bis(1,3-di-p-tolyl-5-p-toluidinomethyl-1,3,5-diazaphosphorinane)nitratesilver(I) (9). To 1 g (2.5 mmoles) of 5 in 20 ml chloroform was added 0.21 g (1.3 mmoles) $AgNO_3$. After dissolution of the $AgNO_3$ the solvent was removed under vacuum. The residue was crystallized from a 3:1 ether—acetone mixture. Yield of 9: 1 g (83%), mp 140-142°C. ^{31}P NMR spectrum (δ, ppm): −36.0 (DMF), −28.9 (CH_2Cl_2). PMR spectrum (see Table 1). Infrared spectrum (ν, cm^{-1}): oil 1600 (Ph), 3370 (N—H). Found: C 61.53; H 6.19; N 10.21; P 6.85%. Calculated for $C_{50}H_{60}N_7P_2AgO_3$: C 61.54; H 6.15; N 10.05; P 6.35%.

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