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Dedicated to the 115th anniversary of B.A. Arbuzov's birth

Synthesis and Structure of N-Pyridyl-Containing Cyclic Aminomethylphosphines

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Abstract—N-pyridyl-containing cyclic aminomethylphosphines have been prepared via condensation of the corresponding bis(hydroxymethyl)arylphosphines with *p*-aminopyridine and *m*-aminomethylpyridine.

Keywords: aminomethylphosphine, pyridylphosphine, 1,5-diaza-3,7-diphosphacyclooctanes

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Transition metal complexes with 1,5-diaza-3,7-diphosphacyclooctanes are mimetics of hydrogenases, effective and inexpensive catalytic systems for practically important processes of interconversion between electrical energy and chemical bonds energy which is essential for the development of alternative energetics based on hydrogen [1, 2]. In particular, high catalytic activity of bisligand nickel(II) complexes with P_2N_2-ligands has been shown in the processes of electrochemical hydrogen oxidation and proton reduction of protons to hydrogen [3].

According to [4, 5], the introduction of pyridyl group to the phosphorous or nitrogen atom of the ligand significantly increases the catalytic activity of the complexes. This fact has been caused by the possibility of secondary interactions, for example, the formation of hydrogen bonds or proton transfer in the coordination sphere, which is a background for the creation of fuel cells. The introduction of pyridyl substituent to the phosphorous atom requires the use of pyridylphosphines which are difficult to synthesize. The introduction of pyridyl substituents to the nitrogen atom is simpler because the corresponding amines are commercially available.

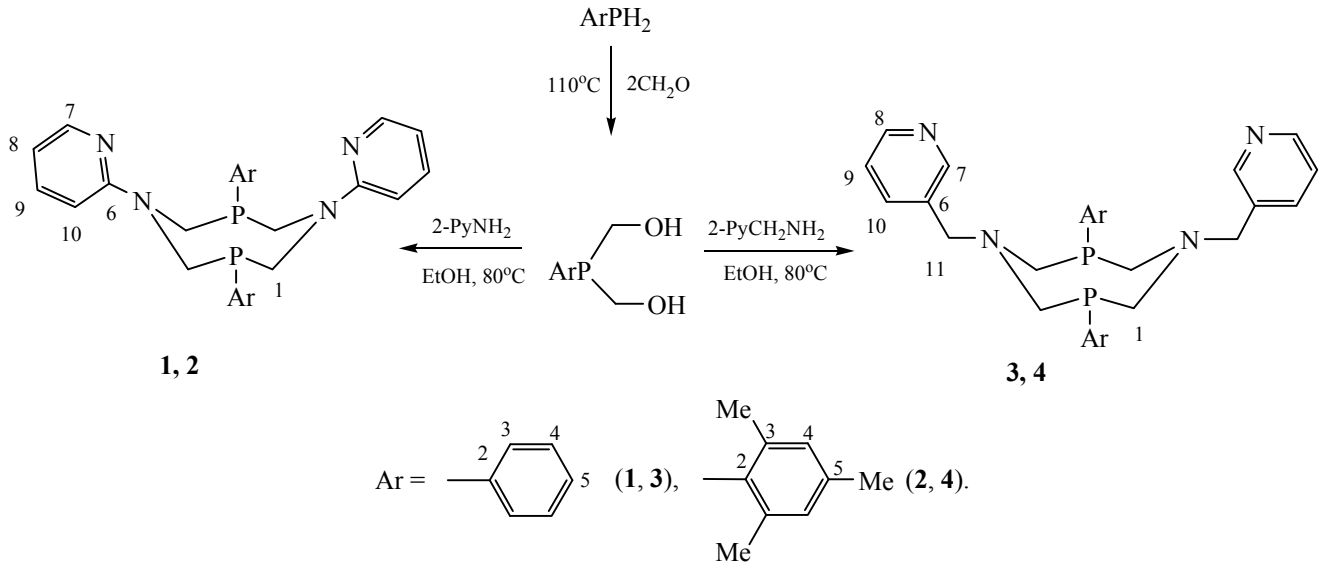
The first 1,5,3,7-diazadiphosphacyclooctane containing pyridyl substituents at nitrogen atoms [1,5-di(2-pyridyl)-3,7-phenyl-1,5,3,7-diazadiphosphacyclooctane] was synthesized in 1980, in 26% yield [6]. Despite the description of molecular structure of this compound in the crystal in 1999 [7], the study of its structure in the solution has not been performed, yet this is necessary for further investigation of structure and properties of the related complexes.

This study aimed to synthesize aminomethylphosphines containing pyridyl substituents at the nitrogen atoms, including new compounds, and elucidation of their structure in solution as well as in crystalline state.

The common method of the preparation of aminomethylphosphines is the Mannich-like reaction in the phosphine–formaldehyde–amine systems. This approach allows preparation of various functionalized derivaives using the corresponding functionalized amines [5–10]. The use of *p*-aminopyridine and *m*-aminomethylpyridine in the condensation reaction has yielded a series of N-pyridyl-containing cyclic aminomethylphosphines.

Bis(hydroxymethyl)phenyl and bis(hydroxymethyl)-mesityl phosphines were prepared via addition of paraformaldehyde at 100–110°C to the corresponding primary phosphines as described in [11, 12]. Further condensation with amines occurred under reflux in ethanol during 8–10 h and led to the formation of the target 1,5-diaza-3,7-diphosphacyclooctanes **1**–**4** (Scheme 1) with moderate yields (28–67%).

Scheme 1.



Compounds **1**–**4** are white crystalline powders soluble in chloroform, acetone, and DMF, and stable in air in solid state. Their structures were confirmed by ^{1}H, ^{31}P, and ^{13}C NMR as well as IR spectroscopy (see the table), their composition was confirmed by the elemental analysis data.

The IR spectra of the pyridylphosphines contained characteristic bands of ν(Py) vibrations at 1422–1481 and 1574–1602 cm^{-1} and did not contain the bands assignable to the vibrations of amino and hydroxy groups. That fact indicated cyclic structure of the investigated compounds. The NMR spectra evidenced symmetric structure of heterocycles **1**–**4** in solutions. Each ^{31}P NMR spectrum contained a single signal (see the table) in the range typical of P-phenyl- and P-mesityl-substituted diazadiphosphacyclooctanes [12]. The signals of heterocycles **3** and **4** containing methyl-pyridyl substituents at nitrogen atoms are upfield shifted compared with the pyridyl-substituted analogs **1** and **2**. In the ^{1}H NMR spectra of the studied heterocycles except compound **1**, methylene protons of the heterocycle H^{1} appeared as AB-part of the $(AB)_2X$-system (two doublets of axial and equatorial protons with spin-spin coupling constants $^{2}J_{HH}$ 13.9–14.7 Hz, see the table). Such system is typical of diazadiphosphacyclooctanes in the *chair–chair* conformation with equatorial substituents at the phosphorous atoms. It should be noted that such pattern of the signals of equatorial and axial protons of heterocycle is typical of diazadiphosphacyclooctanes [8, 13]. In the spectrum of *P*-phenyl substituted heterocycle **1** in $CDCl_3$, AB-part of the $(AB)_2X$-system was registered as two doublets of axial and equatorial protons with $^{2}J_{HH}$ = 14.5 Hz and $^{2}J_{PH}$ = 4.1 Hz for axial and $^{2}J_{PH}$ = 8.92 Hz for equatorial protons. The signals of aromatic proton of compounds **1**–**4** were observed in the characteristic region. In the spectra of compounds **3** and **4**, signals of

Yields and spectroscopic parameters of 1,5-diaza-3,7-diophosphacyclooctanes **1**–**4**

Comp. no.	Yield, %	δ_P, ppm[a]	H^{1}_{ax}			H^{1}_{eq}		
			δ_H, ppm[b]	$^{2}J_{HH}$	$^{2}J_{PH}$	δ_H, ppm[b]	$^{2}J_{HH}$	$^{2}J_{PH}$
1	41	–40.05	3.95	14.5	4.12	4.84	14.50	8.92
2	68	–33.55	4.35	14.67	14.67	4.82	14.67	14.67
3	28	–67.17	3.47	13.95	13.95	3.69	13.95	13.95
4	31	–78.14	3.55	14.30	14.30	4.17	14.30	14.30

[a] Solvent DMF. [b] Solvent $CDCl_3$.

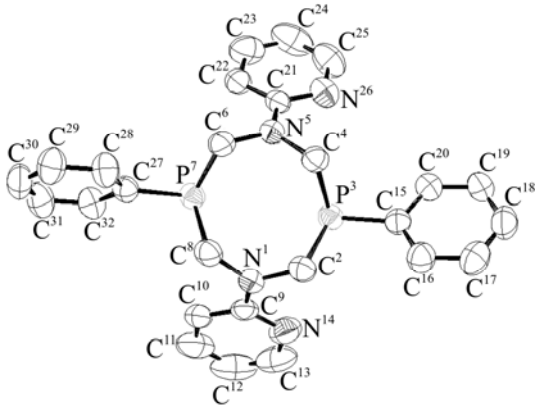


Fig. 1. The general view of a molecule of compound **1** in the crystal.

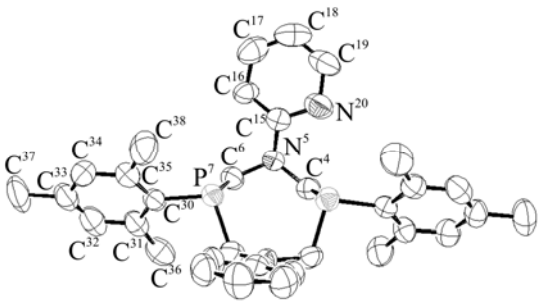


Fig. 2. The general view of a molecule of compound **2** in the crystal.

pyridyl protons are found at 7.18 and 7.13 (C^9H), 7.66 and 7.59 ($C^{10}H$), 8.45 and 8.40 (C^8H), 8.59 and 8.50 ppm (C^7H), respectively. The signals of P-phenyl substituents in the spectrum of compound **3** are registered as multiplet signal at about 7.27 ppm and are partially overlapped with the solvent signal ($CDCl_3$). The signals of the H^4 atoms of mesityl fragments are registered as singlet at 6.81 ppm in the spectrum of compound **4**. Methylene protons of the NCH_2Py fragment are registered as singlet at 4.21 (**3**) and 4.27 ppm (**4**).

In the case of 2-pyridyl substituted compound **1**, the signals of the H^3 and H^5 atoms of phenyl substituents at the phosphorous atoms and of the proton at the C^9 atom overlapped, forming a multiplet signal at 7.41 ppm. The signals of the protons at the C^8 and C^{10} atoms are also present as multiplet signal at 6.55 ppm. The H^4 atoms of P-phenyl substituents and the proton at the C^7 atom are registered as a doublet of doublets at 7.66 and doublet of doublets of doublets at 8.18 ppm. In the spectra of compound **2**, the signals of pyridyl protons at the C^{10}, C^8, C^9, and C^7 atoms are registered at 6.25, 6.49, 7.33, and 8.09 ppm, respectively. The signal of the H^4 atom of mesityl substituent was observed as a singlet at 6.94 ppm. Moreover, in the spectra of P-mesityl-substituted compounds **2** and **4**, the narrow signals of methyl groups in the *meta*-position of the mesityl moieties are observed at 2.31–2.54 and 2.21–2.51 ppm, respectively. The equivalence of the methyl groups indicated free rotation about the exocyclic P–C bonds at room temperature.

The structure of pyridylphosphines **1** and **2** in crystalline state was investigated using X-ray diffraction analysis (Figs. 1 and 2). The conformation of the heterocycles in those molecules was the same: the eight-membered cycles in the crystal as well as in solution have the *chair–chair* conformation typical of diazadiphosphacyclooctanes [8, 10, 12]. Aromatic substituents at the phosphorous atoms are in equatorial positions, pyridyl substituents at the nitrogen atoms are in axial positions. The observed geometry parameters of the eight-membered cycles are typical of diazadiphosphacyclooctane: the P···P distances are 3.60–4.05 Å, the N···N distances are 3.75–4.11 Å. The nitrogen atoms of the heterocycles in both cases have practically undistorted planar triangle coordination (sum of bond angles in both cases was 359°) because of the conjugation with aromatic substituents. The pyridyl rings are in the eclipsed conformation with the bonds of nitrogen in the heterocycle. As a result, they could not coordinate metal atoms, and molecules of compounds **1** and **2** could act only as chelate ligands involving two phosphorous atoms simultaneously, which is typical of 1,5-diaza-3,7-diphosphacyclooctanes [4–8, 10–12]. Phenyl- and mesityl-substituted heterocycles **1** and **2** are differ from each other by position of aromatic rings of the substituents at phosphorous atoms relative to heterocycle: in the case of compound **2** these rings are located near its bisector plane (going through phosphorous atoms), in contrast, phenyl cycles in the molecule of compound **1** are practically eclipsed by one of the endocyclic P–C bonds, which is typical of P-mesityl- and P-phenyl-substituted diazadiphosphacyclooctanes [12].

In summary, new N-pyridyl-containing ligands, 1,5,3,7-diazadiphosphacyclooctanes, are prepared and their structure was elucidated. The obtained ligands are

of interest as precursors in the synthesis of Ni(II)-complexes for testing in electrocatalytic systems of hydrogen production and as promising precursors in the synthesis of various coordination compounds with other catalytically active metals [Fe(II), Ru(II)] and metals of copper group [Cu(I), Au(I), Ag(I)] for constructing photoluminescence systems.

EXPERIMENTAL

The ^{31}P NMR spectra were recorded using a Bruker CXP-100 spectrometer (161 MHz, 85% H_3PO_4). The ^{1}H and ^{13}C NMR were recorded using a Bruker Avance-400 instrument (400 and 100 MHz, respectively). The IR spectra were registered using a Bruker Vector-22 spectrometer in the 4000–400 cm^{-1} range in KBr pellets. Melting points were measured using a Boetius apparatus in sealed capillaries without calibration.

X-ray diffraction data for crystals of compounds **1** and **2** were collected using a Bruker Smart APEX II CCD automatic diffractometer [graphite monochromator, $\lambda(MoK_\alpha) = 0.71073$ Å, ω-scanning, 293(2) K]. Extinction was accounted for semiempirically using SADABS software [14]. Structures were solved via the direct methods using SIR software [15] and refined first under isotropic and then under anisotropic approximation using SHELXL-97 software [16]. Positions of H atoms were found geometrically and included in the refinement using the *rider* model. The calculations were performed using WinGX software [17] and APEX2 software [18]. Plotting and analysis of intermolecular interactions were performed using PLATON [19] and ORTEP software [20]. Investigation of the crystals was carried out at Federal Spectral Analytical Center for Joint Usage, Arbuzov Institute of Organic and Physical Chemistry (Laboratory of Diffraction Methods).

Crystals of compound **1** were colorless, prismatic, monoclinic, $C_{26}H_{26}N_4P_2$, $M = 456.45$, $a = 26.834(4)$ Å, $b = 10.5282(16)$ Å, $c = 19.562(3)$ Å, $\beta = 121.782(2)°$, $V = 4697.9(12)$ $Å^3$, $d_{calc} = 1.291$ g/cm^3, $Z = 8$, space group $C2/c$. Scanning over $2.1° < \theta < 26.00°$, $\mu(Mo) = 0.207$ mm^{-1}. 11543 reflections were collected, 4578 independent reflections were collected, including 3085 with $I \geq 2\sigma$. Final values of R factors: $R = 0.0435$ and $R_w = 0.1348$ over reflections with $F > 2\sigma(F^2)$, $R = 0.0732$ and $R_w = 0.1747$ over all 4578 reflections, goodness of fit (Good) 0.725.

Crystals of compound **2** were colorless, prismatic, triclinic, $C_{32}H_{38}N_4P_2$, $M = 540.60$, $a = 8.6122(11)$ Å, $b = 13.2875(17)$ Å, $c = 14.4152(18)$ Å, $\alpha = 108.247(2)°$, $\beta = 96.890(2)°$, $\gamma = 104.003(2)°$, $V = 1485.4(3)$ $Å^3$, $d_{calc} = 1.209$ g/cm^3, $Z = 2$, space group P-1. Scanning over $2.1° < \theta < 27.00°$, $\mu(Mo) = 0.174$ mm^{-1}. 11918 reflections were collected, 5447 independent reflections were collected, including 1790 with $I \geq 2\sigma$. Final values of R factors: $R = 0.0420$ and $R_w = 0.0449$ over reflections with $F > 2\sigma(F^2)$, $R = 0.1668$ and $R_w = 0.0638$ over all 5447 reflections, goodness of fit (Good) 0.638.

The crystallographic data were deposited at the Cambridge Crystallographic Data Center [CCDC 1867232 (**1**) and 1867236 (**2**)].

All reactions and manipulations were conducted under a Ar atmosphere using standard Schlenk techniques, and all solvents were degassed with argon. $PhPH_2$ and $MesPH_2$ were prepared as described in [11, 12]. *o*-Aminopyridine and *m*-aminopyridine were commercially available.

General procedure of synthesis of compounds 1–4. A mixture of 4.45 mmol of phenylphosphine and 9 mmol of paraformaldehyde was heated to 90°C until complete dissolution of paraformaldehyde. The obtained bis(hydroxymethyl)phenylphosphine was dissolved in 20 mL of degassed ethanol. Then 4.44 mmol of the corresponding amine was added. The reaction mixture was stirred at 100°C for 10 h. The precipitate was filtered off, washed with degassed ethanol, and dried at a reduced pressure for 3 h.

1,5-Di-(2-pyridyl)-3,7-phenyl-1,5,3,7-diazadiphosphacyclooctane (1). Yield 41%, mp 206–208°C. IR spectrum, ν, cm^{-1}: 1481 (Py), 1591 (Py). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.95 d. d (4H, H_B, $^{2}J_{HH} = 14.5$, $^{2}J_{PH} = 4.1$ Hz), 4.84 d. d (4H, H_A, $^{2}J_{HH} = 14.5$, $^{2}J_{PH} = 8.9$ Hz), 6.55–6.60 m (4H, $H^{8,9}$), 7.41 m (9H, H^3, H^5, H^{10}), 7.66 d. d (4H, $H^{4,4'}$, $^{3}J_{HH} = 6.3$, $^{4}J_{PH} = 1.5$ Hz), 8.18 d. d. d (2H, H^7, $^{3}J_{H4H3} = 4.9$, $^{4}J_{H4H2} = 1.9$, $^{5}J_{H4H1} = 0.2$ Hz). ^{31}P NMR spectrum (DMF): δ_P –40.05 ppm. Found, %: C 67.73; H 5.72; N 13.00; P 13.12. $C_{26}H_{26}N_4P_2$. Calculated, %: C 68.42; H 5.70; N 12.28; P 13.60.

1,5-Di-(2-pyridyl)-3,7-dimesityl-1,5,3,7-diazadiphosphacyclooctane (2). Yield 68%, mp 204–206°C. IR spectrum, ν, cm^{-1}: 1481 (Py), 1591 (Py). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.31 s (6H, Me^5), 2.54 s

(12H, Me^3), 4.35 d (4H, H^1, $^2J_{HH}$ = 14.7 Hz), 4.82 d (4H, H^1, $^2J_{HH}$ = 14.7 Hz), 6.25 d (2H, H^{10}, $^3J_{H1H2}$ = 8.6 Hz), 6.49 d. d (2H, H^8, $^3J_{H3H2}$ = 7.1, $^3J_{H3H4}$ = 4.9 Hz), 6.94 s (2H, H^4), 7.33 d. d. d (2H, H^9, $^3J_{H2H3}$ = 7.1, $^3J_{H2H1}$ = 8.6, $^4J_{H2H4}$ = 1.8 Hz), 8.09 d. d (2H, H^7, $^3J_{H4H2}$ = 4.9, $^4J_{H4H2}$ = 1.8 Hz). ^{13}C NMR spectrum (DMF-d_7), δ_C, ppm: 20.34 (Me^5), 23.20 (Me^5), 52.23 d (C^1, $^1J_{PC}$ = 18.3 Hz), 107.56 (C^{10}), 111.59 (C^8), 129.43 (C^4), 131.69 d (C^2, $^1J_{PC}$ = 25.9 Hz), 136.52 (C^9), 139.18 (C^3), 144.22 (C^5), 147.75 (C^7), 156.45 (C^6). ^{31}P NMR spectrum (DMF): d_P –33.55 ppm. Found, %: C 70.39; H 7.69; N 10.09; P 14.33. $C_{32}H_{42}N_4P_2$. Calculated, %: C 71.11; H 7.04; N 10.37; P 14.48.

1,5-Di-(3-methylpyridine)-3,7-diphenyl-1,5,3,7-diazadiphosphacyclooctane (3). Yield 28%, mp 166–168°C. IR spectrum, ν, cm^{-1}: 1574 (Py), 1422 (Py). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 3.47 d (4H, H^1, $^2J_{HH}$ = 13.9 Hz), 3.69 d (4H, H^1, $^2J_{HH}$ = 13.9 Hz), 4.21 s (4H, H^{11}), 7.18 d. d (2H, H^9, $^3J_{H3H4}$ = 7.7, $^3J_{H3H2}$ = 4.9 Hz), 7.27 m (10H, H^{3-5}), 7.66 d (2H, H^{10}, $^3J_{H3H4}$ = 7.7 Hz), 8.45 d (2H, H^8, $^3J_{H3H2}$ = 4.9 Hz), 8.59 s (2H, H^7). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 56.86 (C^{11}), 61.48 (C^1), 123.37 (C^9), 128.76 d (C^4, $^1J_{PC}$ = 3.6 Hz), 128.86 (C^5), 132.57 d (C^3, $^1J_{PC}$ = 10.2 Hz), 133.89 (C^6), 136.81 (C^{10}), 138.84 d (C^2, $^1J_{PC}$ = 5.6 Hz), 148.77 (C^8), 150.64 (C^7). ^{31}P NMR spectrum (DMF): δ_P –67.17 ppm. Found, %: C 68.89; H 6.21; N 11.85; P 12.46. $C_{28}H_{36}N_4P_2$. Calculated, %: C 69.42; H 6.20; N 11.57; P 12.81.

1,5-Di-(3-methylpyridine)-3,7-dimesityl-1,5,3,7-diazadiphosphacyclooctane (4). Yield 31%, mp 132–134°C. IR spectrum, ν, cm^{-1}: 1422 (Py), 1602 (Py). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.21 s (6H, Me^5), 2.51 s (12H, Me^3), 3.55 d (4H, H^1, $^2J_{HH}$ = 14.3 Hz), 4.17 d (4H, H^1, $^2J_{HH}$ = 14.3 Hz), 4.27 s (4H, H^{11}), 6.81 s (2H, H^4), 7.13 d. d (2H, H^9, $^3J_{H3H2}$ = 7.8, $^3J_{H2H1}$ = 4.8 Hz), 7.60 d (2H, H^{10}, $^3J_{H3H2}$ = 7.6 Hz), 8.40 d. d (2H, H^8, $^3J_{H2H1}$ = 4.8, $^4J_{H4H1}$ = 1.7 Hz), 8.50 d (2H, H^7, $^4J_{H4H1}$ = 1.6 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 20.86 (Me^5), 23.90 (Me^3), 54.65 (C^1), 59.88 (C^{11}), 123.45 (C^9), 129.62 (C^4), 133.85 (C^2), 136.91 (C^{10}), 139.3 (C^3), 143.93 (C^2, C^6), 148.64 (C^8), 150.30 (C^7). ^{31}P NMR spectrum (DMF): δ_P –78.14 ppm. Found, %: C 71.64; H 8.14; N 9.83; P 11.02. $C_{30}H_{40}N_4P_2$. Calculated, %: C 71.83; H 7.39; N 9.86; P 10.91.

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CONFLICT OF INTERESTS

No conflict of interests was declared by the authors.

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