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Synthesis and Crystal and Molecular Structures of 1,3-Di-*p*-tolyl-5-(5'-allyl 2'-ethoxybenzyl)-1,3,5-diazaphosphacyclohexane Complexes with Ni(II) and Pt(II) Salts

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Abstract—The reaction of heterocyclic phosphine ligand, 1,3-di-*p*-tolyl-5-(5'-allyl-2'-ethoxybenzyl)-1,3-diaza-5-diazaphosphacyclohexane (L), containing a functional substituent at phosphorus, with (1,5-cyclooctadiene)dichloroplatinum(II) and nickel(II) chloride gave complexes $PtCl_2L_2$ (**I**) and $NiCl_2L_2$ (**II**), respectively, with the *cis*- and *trans*-configurations of the central ion. The structure of the complexes was determined by X-ray diffraction (CIF files CCDC no. 2097704 (**I**), 2097705 (**II**)).

Keywords: cyclic phosphines, platinum complex, nickel complex, configuration, molecular structure

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INTRODUCTION

Cyclic aminomethylphosphines are unusual hybrid ligands for the coordination chemistry of transition metals due to specific conformational behavior of the heterocycle on the metal matrix, which restricts the possible mutual arrangements of the lone electron pair (LP) of the heteroatoms and due to the position of the hard donor atom (heterocyclic nitrogen) in the close vicinity of the central ion [1–4]. Currently, cyclic diphosphines, namely 1,5-diaza-3,7-diphosphacyclooctanes [5–8] and 1-aza-3,6-diphosphacycloheptanes [9, 10], are the most studied types of cyclic aminomethylphosphines. It was demonstrated that the spatial proximity of the nitrogen atoms (not involved in the coordination) and the metal (Ni, Co) accounts for the activity of P,P-chelate complexes in the key reactions of hydrogen energy production [7, 8, 11–17]. In addition, cyclic aminomethylphosphines exhibited unusual stereoisomeric transitions and unique ring fusion reactions to give larger rings [18–20] or splitting to give smaller rings upon complexation [21, 22].

The corresponding cyclic monophosphines, namely, 1,3-diaza-5-phosphacyclohexanes, have been much less studied than cyclic diphosphines [23–29]. According to published data, upon complexation, these ligands (with phenyl or aminomethyl substituents at the phosphorus atom) change the predominant chair conformation with the equatorial position of the P-substituent to a chair conformation with the axial position of this substituent and correspond to ligands with a small Tolman cone angle [24, 28]. It was of interest to elucidate their ability to stabilize various isomers of nickel(II) and platinum(II) square-planar complexes, evaluate the proneness of ligands to reactions accompanied by a change in the ring size, and describe the effect of coordination to the transition metal on the conformational behavior of the heterocycle.

EXPERIMENTAL

Commercially available chemicals were used in the study. Anhydrous nickel dichloride (Sigma-Aldrich) and (1,5-cyclooctadiene)dichloroplatinum(II) (Sigma-Aldrich) were used as received. Acetonitrile and dimethylformamide (DMF) were purified and dried prior to use by standard procedures. 1,3-Di-*p*-tolyl-5-(5'-allyl-2'-ethoxybenzyl)-1,3-diaza-5-phosphacyclohexane (**L**) was obtained by a reported procedure [30].

IR spectra were measured on a Specord M-80 spectrometer in the 600–4000 cm^{-1} range (mineral oil mulls). ^{31}P NMR spectra were recorded on a Bruker MSL-400 spectrometer (161 MHz), and ^{1}H NMR spectra were recorded on a Bruker WM-250 spectrometer (250 MHz).

Synthesis of bis(1,3-di-*p*-tolyl-5-(5'-allyl-2'-ethoxybenzyl)-1,3-diaza-5-phosphacyclohexane)dichloroplatinum(II) (I). A solution of ligand L (0.100 g, 2.17 mmol) in acetonitrile (1 mL) was added to a hot solution of (1,5-cyclooctadiene)dichloroplatinum(II) (0.041 g,

1.08 mmol) in acetonitrile (1 mL), and the mixture turned green. The reaction mixture was slowly cooled down to room temperature for 24 h. The transparent light yellow crystals thus formed were collected on a filter and recrystallized from acetonitrile. The yield of **I** was 0.09 g (69%), T_m = 128°C.

IR (ν, cm^{-1}): 808 (C–H$_{ar}$), 994 (P–Ar), 1612 (C=C$_{ar}$), 1636 (CH$_2$=CH–), 3028 (H–C$_{ar}$). ^{31}P NMR (DMF; δ, ppm, *J*, Hz): –3.63 ($^1J_{PtP}$ 3400). ^{1}H NMR (DMF-d$_7$; δ, ppm, *J*, Hz): 1.28 t (3H, $^3J_{HH}$ 6.0, O–CH$_2$–<u>CH$_3$</u>), 2.35 s (6H, <u>CH$_3$</u>C$_6$H$_4$), 3.07 d (2H, $^3J_{HH}$ 6.5, <u>CH$_2$</u>–CH=CH$_2$), 3.42 d (2H, $^2J_{PH}$ 12.5, P–<u>CH$_2$</u>–Ar), 3.93 dd ($^2J_{HH}$ 13.25, $^4J_{PH}$ 4.5, N–<u>CH$_e$</u>–N), 4.02 q ($^3J_{HH}$ 6, O–<u>CH$_2$</u>–CH$_3$) (totally 3H), 4.87–4.93 m (2H, CH$_2$–CH=<u>CH$_2$</u>), 5.19 d (1H, $^2J_{PH}$ 13.25, N–<u>CH$_a$</u>–N), 5.65–5.81 m (1H, CH$_2$–<u>CH</u>=CH$_2$), 7.06–7.17 m (7H, *o*-C$_6$H$_4$–N + C$_6$H$_3$), 7.26 d (4H, $^3J_{HH}$ 8, *m*-C$_6$H$_4$–N) (the signals of the P–CH$_2$–N groups are covered by the signal of water in DMF-d$_7$).

For $C_{60}H_{79}N_5O_4P_2Cl_2Pt$

Anal. calcd., %	C, 57.09	H, 6.26	N, 5.55	P, 4.92	Cl, 5.63
Found, %	C, 56.47	H, 6.20	N, 5.45	P, 5.10	Cl, 5.11

Synthesis of bis(1,3-di-*p*-tolyl-5-(5'-allyl-2'-ethoxybenzyl)-1,3-diaza-5-phosphacyclohexane)dichloronickel(II) (II). A solution of nickel(II) chloride (0.015 g, 0.11 mmol) in DMF (1 mL) was added to a hot solution of ligand L (0.100 g, 0.21 mmol) in acetonitrile (1 mL), and the resulting mixture was slowly cooled to room temperature in 24 h. The red crystals thus formed were collected on a filter and washed with acetonitrile. The yield of **II** was 0.04 g (35%), T_m = 162°C.

IR (ν, cm^{-1}): 808 (C–H$_{ar}$), 1404 (C–N), 1510 (C=C$_{ar}$), 1610 (C=C$_{ar}$), 1640 (CH$_2$=CH–). ^{31}P NMR (DMF; δ, ppm): –45.58 (broad signal).

For $C_{58}H_{76}N_4O_4P_2Cl_2Ni$

Anal. calcd., %	C, 64.27	H, 2.49	N, 5.17	P, 5.72	Cl, 6.55
Found, %	C, 64.47	H, 2.20	N, 5.45	P, 5.70	Cl, 6.21

The X-ray diffraction study of the crystals of **I** and **II** was carried out at the Department of X-ray Diffraction Studies of the Spectral Analytical Center for Collective Use on a CAD-4 Enraf-Nonius four-circle automated diffractometer at a temperature of 21°C (MoK_α radiation). Crystals of **I**, $C_{58}H_{70}N_4O_2P_2Cl_2Pt\cdot C_2H_3N$, triclinic; at 21°C, a = 11.534(2), b = 12.378(2), c = 21.086(2) Å, α = 93.54(6)°, β = 92.44(6)°, γ = 108.00(6)°, V = 2851.7(12) Å^3, Z = 2, ρ(calcd.) = 1.426 g/cm^3, space group $P\bar{1}$. Crystals of **II**, $C_{58}H_{70}N_4O_2P_2Cl_2Ni$, triclinic; at 21°C, a = 10.119(3), b = 10.580(4), c = 13.693(3) Å, α = 72.55(2)°, β = 89.66(2)°, γ = 85.54(3)°, V = 1393.9(8) Å^3, Z = 1, ρ(calcd.) = 1.247 g/cm^3, space group $P\bar{1}$ (molecule in the special position at the center of symmetry).

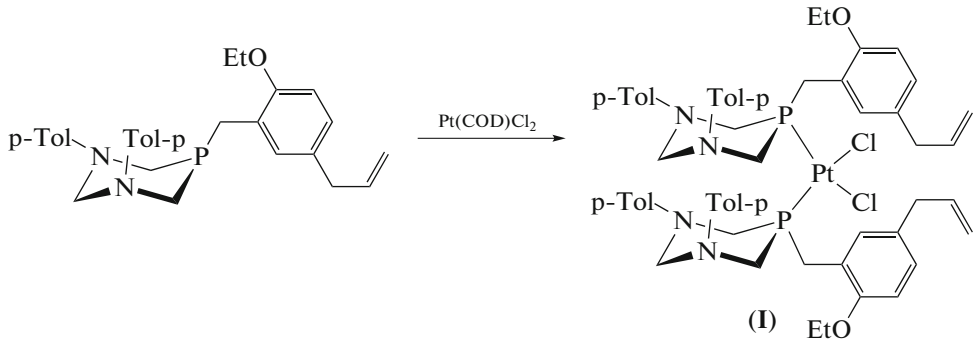
The unit cell parameters and intensities of 11 881 (**I**) and 4419 (**II**) reflections, of which 7270 and 3246 reflections had $I \geq 2\sigma(I)$, respectively, were measured on a CAD-4 Enraf-Nonius four-circle automated diffractometer at 21°C (MoK_α radiation). For the structure of **II**, absorption corrections were not applied (μMo 5.44 cm^{-1}); for the structure of **I**, absorption corrections were applied empirically (μMo 26.56 cm^{-1}). The structures were solved by direct methods and refined first in the isotropic and then in the anisotropic approximation (for all non-hydrogen atoms). The hydrogen atoms were identified from the Fourier difference maps and refined using the riding model. The final R-factors were R = 0.0827, R_w = 0.2133 over 7270 reflections on $F^2 \geq 2\sigma$ (for **I**) and R = 0.0408, R_w = 0.1057 over 3246 reflections on $F^2 \geq 2\sigma$ (for **II**). Preliminary data processing was performed on a DEC AlfaStation 200 computer using the MolEN package [31]; the structure solution and final refinement were carried out using the WinGX program package [32]. The drawing of structures and analysis of the geometry were carried out using PLATON [33] and MERCURY [34] software.

The crystallographic parameters of compounds **I** and **II** were deposited with the Cambridge CCDC/FIZ Karlsruhe joint database (CCDC nos. 2097704, 2097705, respectively) and can be received free of charge on request from www.ccdc.cam.ac.uk/data_request/cif.

RESULTS AND DISCUSSION

Since known data were limited to information about the coordinating ability of 3-phenyl-1,5-diaza-3-phosphacyclohexanes [29, 35], compounds with more electron-donating and sterically more demanding functionally substituted 5'-allyl-2'-ethoxybenzyl radical at the phosphorus atom were chosen as investigation objects. The starting ligand L was obtained by a previously reported procedure [30].

According to published data [27–29], cyclic aminomethylphosphine ligands with the 1,5-diaza-3-phosphacyclohexane core are expected to form the *cis*-isomer of the platinum(II) complex. Indeed, the reaction of one equivalent (1,5-cyclooctadiene)dichloroplatinum(II) with two equivalents of ligand L gives a complex with the *cis*-configuration of the central ion. This complex was isolated as large transparent yellowish crystals with the composition $L_2PtCl_2\cdot CH_3CN$, stable to oxidation and hydrolysis (Scheme 1).

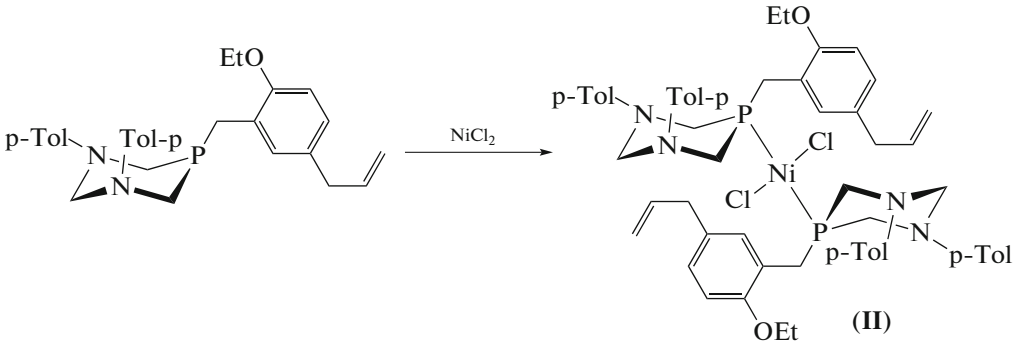


Scheme 1.

The IR spectrum of complex **I** in the 600–4000 cm^{-1} range is very similar to the spectrum of the initial heterocyclic ligand. The positions and integral intensity ratios of the signals of various groups of protons in the ^1H NMR spectrum indicate that the ligand structure is retained upon complex formation. The downfield shift of the ^{31}P NMR signal of compound **I** (δ_P = –3.63 ppm) with respect to the signal of the initial 1,3-diaza-5-phosphacyclohexane (δ_P = –39.94 ppm) and the stereospecific spin–spin coupling constant $^1J_{PtP}$ (3400 Hz) attest to the formation of a square-planar *cis*-complex in which the metal atom is coordinated to phosphorus atoms, because NMR data are similar to the data observed previously for the platinum *cis*-complexes of 5-phenyl-1,3-diaza-5-phosphacyclohexanes ($\Delta\delta_p$ 35–39 ppm, $^1J_{PtP}$ 2900–3400 Hz) [27, 29]. The ^1H NMR spectra indicate that both ligands in the complex are equivalent and have a chair conformation, since the methylene protons of the N–CH_2–N moiety appear as a doublet at 5.19 ppm ($^2J_{HH}$ 13.2 Hz) and a doublet of doublets at 3.93 ppm ($^2J_{HH}$ 13.2, $^4J_{PH}$ 4.5 Hz), which is typical of this conformation. An X-ray diffraction study identified the exact structure of *cis*-complex **I** (Fig. 1). The crystal of complex **I** is an acetonitrile solvate, but no specific interactions of the complex with the solvent molecule are observed (Fig. 1).

The platinum atom in the molecule has a square-planar coordination (the sum of the bond angles at platinum is 360°), but the angles between the ligands considerably differ: the Cl(2)Pt(1)P(5b) angle has decreased to 83.5(1)°, while the P(5a)Pt(1)P(5b) angle has increased to 102.1(1)°. These distortions of the platinum bond angles are much more pronounced than those in the case of *cis*-bis(1,3,5-triphenyl-1,3-diaza-5-phosphacyclohexane)dichloroplatinum(II) [36] and are due to steric factors: repulsion of the bulky substituents at the phosphorus atoms of the *cis*-phosphine ligands. The cyclic phosphine ligands in **I** occur in the chair conformation, the *para*-tolyl substituents at the N(3) atoms of both ligands occupy equatorial positions, and the substituents at P(5) and N(1) occupy axial and pseudoaxial positions, respectively. The endocyclic nitrogen atoms in the ligands of complex **I** have different hybridizations: N(1) atoms have a flattened trigonal coordination (the sums of bond angles at N(1) are 351.8(8)° and 353.2(8)°), while N(3) atoms have a trigonal pyramidal geometry (the sums of bond angles are 336.6(8)° and 349.6(8)°). Crystallization of the asymmetric conformer in which one of the ligands has axial P–M bond does not take place. Among isolated crystals, no crystals of different type were observed, indicating the formation of only one complex. Mention should be made of the difference from the previously described formation of the platinum dichloride complex with 1,3,5-triphenyl-1,3-diaza-5-phosphacyclohexane, where the reaction conducted in acetonitrile gave a complex with different conformations of the ligands [36].

Nickel(II) phosphine complexes are more labile than platinum analogues, and their spin state depends on the central ion configuration and the number of attached ligand molecules. The phosphine complexes of Ni(II) tend to form square-planar or tetrahedral four-coordinate configurations of the central ion. Reactions of phosphines with nickel chloride in acetonitrile give, most often, bright red-colored square-planar crystalline complexes [35]. The reaction of anhydrous Ni(II) chloride with ligand L afforded complex L_2NiCl_2 (**II**) as intensely colored red crystals, which is indicative of a square-planar configuration of the nickel atom (Scheme 2).



Scheme 2.

The IR spectrum of the obtained complex **II** in the region of 700–4000 cm^{-1} is rather similar to the spectra observed for the initial ligand and complex **I**, which means that the structure of the heterocyclic phosphine ligand is retained. The complex is poorly soluble in most organic solvents. On heating in DMF, complex **II** decomposes, and the ^{31}P NMR spectrum exhibits only a signal for the free ligand at –45.58 ppm.

The structure of complex **II** was determined by X-ray diffraction. This complex is *trans*-bis[1,3-di-*p*-tolyl-5-(5'-allyl-2'-ethoxybenzyl)-1,3-diaza-5-phosphacyclohexane]dichloronickel(II) with a square-planar configuration (Fig. 2). It is noteworthy that in the previously synthesized bis(1,3,5-triphenyl-1,3-diaza-5-phosphacyclohexane)dichloronickel(II), the central nickel atom has *cis*-configuration [35, 37], which is apparently attributable to smaller size of the phenyl substituent at the phosphorus atom (Scheme 2).

In the crystal of **II**, the complex is located in a special position at a center of symmetry; therefore, in Fig. 2, the symmetrically dependent atoms have the same numbering. The bond angles at the nickel atom are somewhat distorted: Cl(2)NiP(5), 87.10(4)°; Cl(2)'NiP(5), 92.90(4)°. The Ni(1)–Cl(2) distances (2.165(1) Å) are somewhat shorter than Ni(1)–P(5) (2.2120(9) Å). The heterocyclic ligands of complex **II** have a chair conformation, the *para*-tolyl substituent at N(3) is in the equatorial position, while substituents at N(1) and P(5) are in the pseudoaxial positions. It is noteworthy that, like in complex **I**, the nitrogen atoms of the heterocycle have different coordinations: the N(1) atom has a highly flattened pyramidal geometry (the sum of bond angles at this atom is 354.4°), while the N(3) atom has a trigonal pyramidal geometry (the sum of bond angles is 335.4°). The bond lengths at N(1) are markedly shorter than those at N(3), which is

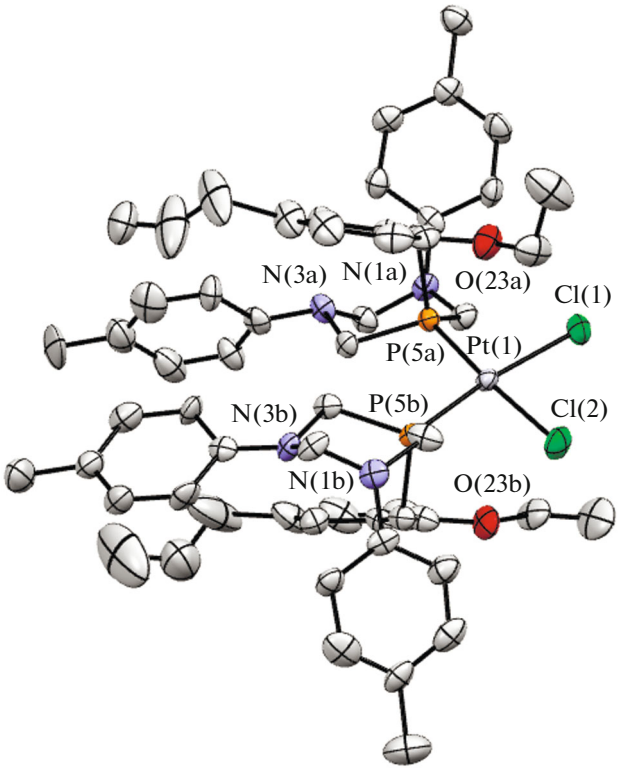


Fig. 1. Geometry of complex **I** in the crystal. Hydrogen atoms are omitted.

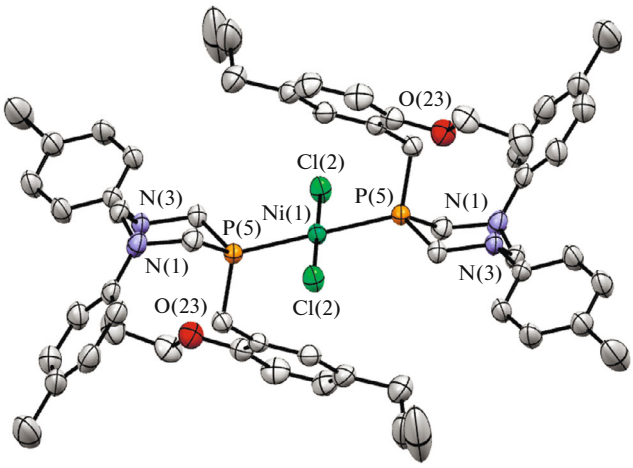


Fig. 2. Geometry of complex **II** in the crystal. The allyl group is disordered in the crystal over two positions with relative occupancies of 0.83 : 0.17 and is shown in the position with higher occupancy.

due to the conjugation of its lone pair with the π-system of the *para*-toluidine moiety.

It is noteworthy that in less sterically strained 8-membered heterocyclic phosphines such as 1,5-diaza-3,7-diphosphacyclooctanes and in metal complexes with these ligands, the nitrogen atoms have a trigonal-planar coordination and axial orientation of the substituent if the substituent is aromatic and a trigonal pyramidal coordination and equatorial orientation of the substituent if the nitrogen atom is bound to an sp^3-hybridized carbon atom. In the complexes with 1,3-diaza-5-phosphacyclohexane ligands with a trigonal-planar coordination of nitrogen atoms, the aromatic N-substituents should have occupied the axial position, which would have caused pronounced steric hindrance with the axial substituent at the phosphorus atom (1–3 *syn*-axial interactions); these complications are resolved via pyramidalization of nitrogen atoms and "squeezing-out" of one substituent to the equatorial position. The heterocycle remains in the chair conformation.

Thus, it was shown that the complex formation reactions of 1,3-diaza-5-phosphacyclohexanes with platinum and nickel chlorides are not accompanied by any changes in the ring size; the coordination bond is formed at the phosphorus atom, and the coordination is accompanied by a change in the predominant conformation of the ligand to the sterically less demanding one. Despite the conformational changes of the heterocyclic ligand, the replacement of the phenyl substituent at the phosphorus atom of 1,3-diaza-5-phosphacyclohexane by 5-allyl-2-ethoxybenzyl substituent results in the formation of a nickel complex with the *trans*-configuration of the central ion, unlike the platinum complex, which has the *cis*-configuration owing to the larger size of the central ion.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

REFERENCES

1. Karasik A.A. and Nikonov G.N., *Zh. Obshch. Khim.*, 1993, vol. 63, no. 12, p. 2775.
2. Karasik, A.A., Musina, E.I., Balueva, A.S., et al., *Pure Appl. Chem.*, 2017, vol. 89, no. 3, p. 293.
3. Latypov, S.K., Strelnik, A.G., Ignatieva, S.N., et al., *J. Phys. Chem. A*, 2012, vol. 116, no. 12, p. 3182.
4. Latypov, S.K., Strelnik, A., Balueva, A., et al., *Eur. J. Inorg. Chem.*, 2016, vol. 2016, no. 7, p. 1068.
5. Karasik, A.A., Bobrov, S.V., Nikonov, G.N., et al., *Russ. J. Coord. Chem.*, 1995, vol. 21, no. 7, p. 574.
6. Ignatieva, S.N., Balueva, A.S., Karasik, A.A., et al., *Inorg. Chem.*, 2010, vol. 49, no. 12, p. 5407.
7. Karasik, A.A., Balueva, A.S., Musina, E.I., et al., *Mendeleev Commun.*, 2013, vol. 23, p. 237.
8. Musina, E.I., Khrizanforova, V.V., Strelnik, I.D., et al., *Chem.-Eur. J.*, 2014, vol. 20, no. 11, p. 3169.
9. Musina, E.I., Karasik, A.A., Balueva, A.S., et al., *Eur. J. Inorg. Chem.,* 2012, p. 1857.
10. Karasik, A.A., Balueva, A.S., Moussina, E.I., et al., *Heteroat. Chem.,* 2008, vol. 19, no. 2, p. 125.
11. Galimullina, R.M., Valitov, M.I., Spiridonova, Yu.S., et al., *Russ. J. Phys. Chem. A*, 2011, vol. 85, no. 12, p. 2214.
12. Khrizanforova, V.V., Morozov, V.I., Strelnik, A.G., et al., *Electrochim. Acta*, 2017, vol. 225, p. 467.
13. Khrizanforova, V.V., Musina, E.I., Khrizanforov, M.N., et al., *J. Organomet. Chem.*, 2015, vols. 789–790, p. 14.
14. Kadirov, M.K., Karasik, A.A., Nizameev, I.R., et al., *Energy Technol.*, 2018, vol. 6, p. 1088.
15. Burrows, A.D., Dodds, D., Kirk, A.S., et al., *Dalton Trans.*, 2007, vol. 36, pp. 570–580.
16. Reback, M.L., Ginovska-Pangovska, B., Ho, M.-H., et al., *Chem.-Eur. J.*, 2013, vol. 19, p. 1928.
17. Smith, S.E., Yang, J.Y., DuBois, D.L., et al., *Angew. Chem., Int. Ed.* Engl., 2012, vol. 51, p. 3152.
18. Mock, M.T., Chen, S., O'Hagan, M., et al., *J. Am. Chem. Soc.*, 2013, vol. 135, p. 11493.
19. Kendall, A.J., Johnson, S.I., Bullock, R.M., et al., *J. Am. Chem. Soc.*, 2018, vol. 140, p. 2528.
20. Strelnik, I.D., Dayanova, I.R., Poryvaev, T.M., et al., *Mendeleev Commun.*, 2020, vol. 30, p. 40.
21. Musina, E.I., Fesenko, T.I., Strelnik, I.D., et al., *Dalton Trans.*, 2015, vol. 44, p. 13565.
22. Musina, E.I., Wittmann, T., Latypov, S., et al., *Eur. J. Inorg. Chem.,* 2019, p. 3053.
23. Musina, E.I., Karasik, A.A., Sinyashin, O.G., et al., *Adv. Heterocycl. Chem.*, 2015, vol. 117, p. 83.
24. Liu, C., Huang, J., Zhu, J., et al., *J. Appl. Polym. Sci.*, 2018, vol. 135, no. 9, p. 45721.
25. Karasik, A.A., Naumov, R.N., Sommer, R., et al., *Polyhedron*, 2002, vol. 21, p. 2251.
26. Karasik, A.A., Georgiev, I.O., Musina, E.I., et al., *Polyhedron*, 2001, vol. 20, p. 3321.
27. Jain, A., Helm, M.L., Linehan, J.C., et al., *Inorg. Chem. Commun.*, 2012, vol. 22, p. 65.
28. Nikonov, G.N., Karasik, A.A., Malova, E.V., et al., *Heteroatom. Chem.*, 1992, vol. 3, no. 4, p. 439.
29. Arbuzov, B.A., Karasik, A.A., Nikonov, G.N., et al., *Russ. Chem. Bull.*, 1992, vol. 41, p. 253.

30. Baymuhametov, F.Z., Zheltukhin, V.F., Nikonov, G.N., et al., *Russ. J. Gen. Chem.,* 2002, vol. 72, no. 11, p. 1754.

31. Straver, L.H. and Schierbeek, A.J., *MolEN. Structure Determination System. Program Description*, Nonius, 1994, vol. 1, p. 180.

32. Farrugia, L.J., *J. Appl. Crystallogr.*, 1999, vol. 32, no. 4, p. 837.

33. Spek, A.L., *J. Appl. Crystallogr.*, 2003, vol. 36, no. 1, p. 7.

34. Macrae, C.F., Edgington, P.R., McCabe, P., et al., *J. Appl. Crystallogr.*, 2006, vol. 39, no. 3, p. 453.

35. Arbuzov, B.A., Nikonov, G.N., Karasik, A.A., et al., *Russ. Chem. Bull.*, 1991, vol. 40, p. 191.

36. Karasik, A.A., Bobrov, S.V., Nikonov, G.N., et al., *Russ. Chem. Bull.*, 1993, vol. 42, p. 1587.

37. Khadiullin, R.Sh., Karasik, A.A., Plyamovatyi, A.Kh., et al., *J. Mol. Struct.*, 1993, vol. 293, p. 85.

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