## Reactions of pyrrole-2,5-dicarbaldehyde with 2-aminodiphenylamine in the presence of nickel and cobalt trimethylacetates

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The reactions of pyrrole-2,5-dicarbaldehyde (1) with 2-aminodiphenylamine (3) taken in a ratio of 1:2 in the presence of polinuclear nickel(II) and cobalt(II) trimethylacetate complexes were studied in MeCN. The reaction with  $Ni_9(HOOCCMe_3)_4(\mu_4$ - $OH_{3}(\mu_{3}-OH)_{3}(OOCCMe_{3})_{12}$ (2) afforded the mononuclear complex Ni(OOCCMe<sub>3</sub>)<sub>2</sub>[(PhHN)C<sub>6</sub>H<sub>4</sub>NHCHC<sub>4</sub>H<sub>2</sub>NCHNC<sub>6</sub>H<sub>4</sub>(NHPh)] (4) with a new ligand, which is the product of condensation of one molecule 1 with two molecules 3 and contains two NHPh groups. By contrast, the reaction with the polymeric complex  $[Co(OH)_n(OOCCMe_3)_{2-n}]_x$  (5, n = 0.05-0.1) gave rise to the tetranuclear complex  $Co_4(\mu_4-O)(\mu-OOCCMe_3)_4L_2$  (6), where L is, apparently, the product of further oxidation of Schiff's base to the benzimidazole derivative. The structures of compounds 4 and 6 were established by X-ray diffraction analysis.

**Key words:** nickel, cobalt, trimethylacetate complexes, pyrrole-2,5-dicarbaldehyde, 2-aminodiphenylamine, condensation, X-ray diffraction analysis, magnetic properties.

Recently,<sup>1</sup> we have demonstrated that the reaction of pyrrole-2,5-dicarbaldehyde (1) with *o*-phenylenediamine in the presence of the nonanuclear nickel(II) cluster Ni<sub>9</sub>(HOOCCMe<sub>3</sub>)<sub>4</sub>( $\mu_4$ -OH)<sub>3</sub>( $\mu_3$ -OH)<sub>3</sub>(OOCCMe<sub>3</sub>)<sub>12</sub> (2) afforded the condensation product of three diamine molecules and two dialdehyde molecules to form a complicated ligand "enveloping" the tetranuclear metal carboxylate fragment. In the present study, we found that the chemical properties of *o*-phenylenediamine in the reactions with nickel carboxylates<sup>2</sup> differ from those of its *N*-phenyl-substituted analog, *viz.*, 2-aminodiphenylamine (3), although the condensation of dicarbaldehyde 1 with *o*-phenylenediamine or diamine 3 without additional activation by transition metal complexes proceeds similarly to yield [1+1] Schiff's bases.<sup>3,4</sup>

## **Results and Discussion**

We found that the reactions of compounds 1 and 3 in MeCN in the presence of cluster 2 afforded the mononuclear nickel complex  $Ni(OOCCMe_3)_2[(PhHN)C_6H_4NHCHC_4H_2NCHNC_6H_4(NHPh)]$  (4) with the ligand, which is the product of condensation of one molecule 1 with two molecules 3 (Scheme 1). According to the X-ray diffraction analysis, only three of five N atoms capable of forming complexes are involved in interactions with the metal atom in complex 4 (Fig. 1, Table 1). The shortest Ni–N bond (2.009(3) Å) is that with the central N(2) atom, whereas the bond with the N(3) atom of the azomethine fragment (2.110(4) Å) is somewhat longer, and the longest distance (2.187(4) Å)is that between the Ni atom and the N(1) atom of the diphenylamine group. The coordination environment about the Ni<sup>II</sup> atom is completed to a distorted octahedron through binding with two O atoms of the chelate trimethylacetate group (Ni-O, 2.097 and 2.150(3) Å) and one O atom of the terminal trimethylacetate group (Ni-O, 1.997(3) Å). The tridentate coordination of the ligand gives rise to two adjacent five-membered Ni(1)N(1)C(17)C(18)N(2) and Ni(1)N(2)C(23)C(24)N(3) metallocycles. Both virtually planar fragments adopt a strongly flattened envelope conformation with the Ni atom deviating from the plane of the four other atoms of the metallocycles by

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Fig. 1. Molecular structure of complex 4.

Table	1.	Principal	bond	lengths	( <i>d</i> )	and	bond	angles	(œ)	in
compl	ex	4								

Bond	$d/\text{\AA}$	Angle	ω/deg
Ni(1)-O(1)	2.097(3)	- 0(1)-Ni(1)-O(2)	61.56(13)
Ni(1) - O(3)	1.997(3)	O(1) - Ni(1) - N(1)	101.1(2)
Ni(1) - N(2)	2.009(3)	O(1) - Ni(1) - N(3)	100.5(2)
O(1) - C(1)	1.278(6)	O(2) - Ni(1) - N(1)	93.5(2)
O(3) - C(6)	1.270(5)	O(2) - Ni(1) - N(3)	94.8(2)
N(1)-H(1N)	1.00(5)	O(3) - Ni(1) - N(2)	109.84(14)
N(1) - C(17)	1.453(5)	N(1) - Ni(1) - N(2)	78.8(2)
N(2) - C(23)	1.289(6)	N(2) - Ni(1) - N(3)	80.3(2)
N(3)-C(27)	1.355(6)	Ni(1) - O(2) - C(1)	89.0(3)
N(4) - C(28)	1.317(6)	Ni(1) - N(1) - C(11)	118.3(3)
N(5) - H(5N)	0.89(6)	C(11) - N(1) - C(17)	115.4(4)
N(5)-C(35)	1.415(6)	Ni(1) - N(2) - C(23)	115.9(4)
Ni(1) - O(2)	2.150(3)	Ni(1) - N(3) - C(24)	109.3(3)
Ni(1) - N(1)	2.187(4)	C(24) - N(3) - C(27)	104.9(4)
Ni(1) - N(3)	2.110(4)	C(34) - N(5) - C(35)	126.0(5)
O(2) - C(1)	1.247(5)	O(3) - C(6) - O(4)	125.0(5)
O(4) - C(6)	1.247(5)	O(1) - Ni(1) - O(3)	92.06(13)
N(1) - C(11)	1.450(5)	O(1) - Ni(1) - N(2)	158.3(2)
N(2)-C(22)	1.392(6)	O(2) - Ni(1) - O(3)	153.5(2)
N(3)–C(24)	1.356(5)	O(2) - Ni(1) - N(2)	96.7(2)
N(4)-H(4N)	0.81(4)	O(3) - Ni(1) - N(1)	94.4(2)
N(4)-C(29)	1.429(6)	O(3) - Ni(1) - N(3)	87.0(2)
N(5)-C(34)	1.402(6)	N(1) - Ni(1) - N(3)	158.3(2)
		Ni(1) - O(1) - C(1)	90.7(3)
		Ni(1) - O(3) - C(6)	132.2(3)
		Ni(1) - N(1) - C(17)	108.8(3)
		Ni(1) - N(2) - C(22)	117.8(3)
		C(22) - N(2) - C(23)	125.6(4)
		Ni(1)-N(3)-C(27)	145.6(4)
		C(28) - N(4) - C(29)	126.6(5)
		O(1) - C(1) - O(2)	118.6(5)

0.31 and 0.09 Å, respectively. At the same time, the Ni atom is at a distance of only 0.15 Å from the N(1)C(17)C(18)N(2)C(23)C(24)N(3) plane passing through all non-metal atoms of both metallocycles. The planar conformation of the ligand as a whole (except for the phenyl substituents of the amino groups) is a consequence of conjugation between the  $\pi$ -electron systems of the double bonds of the pyrrole ring and two phenylene fragments. Apparently, slight distortions can be considered as a result of steric nonbonded interactions between the atoms within the ligand and redistribution of the electron density within the organic rings through coordination of some N atoms to the metal atom. The geometry of the ligand can be characterized by the torsion angles in the C(11)N(1)C(17)C(22)N(2)C(23)C(24)N(3)C(27)C(28)N(4)C(29)C(34)N(5)C(35)chain (146.3, 2.9, 177.6, -177.0, 5.5, -177.9, 176.5, -3.8,176.2, 174.7, 1.5, and -158.3°, respectively). Analysis of these values shows that this ligand is nearly symmetrical with respect to the central N(3) atom of the pyrrole ring (*i.e.*, it possesses a twofold pseudoaxis passing through the N(3) atom and the midpoint of the C(25)–C(26) bond). The largest distortion of the symmetry associated with coordination of the metal atom is manifested in the difference between the first (C(11)N(1)C(17)C(22)) and the last (C(29)C(34)N(5)C(35)) fragments in the chain. The corresponding dihedral angles between the planes of the phenyl groups at the C(11) and C(17) atoms and at the C(29) and C(35) atoms are 67.4 and  $46.9^{\circ}$ , respectively. These Ph groups form angles of 69.3, 6.4, 12.9, and  $45.0^{\circ}$ , respectively, with the plane of the central pyrrole ring. The distribution of the bond lengths in the ligand (the N(2)-C(23), C(24)-N(3), C(25)-C(26), and C(28)—N(4) bond lengths are close to the double bond length;<sup>5</sup> see Table 1) is indicative of a particular contribution of the zwitterionic form bearing a positive charge on the N(4) and a negative charge on the C(27)atom of the pyrrole ring. It should be noted that the ligand is formally neutral (the positive charge of the divalent Ni atom is compensated by the negative charges of two trimethylacetate anions). Another perturbing factor responsible for a distortion of the planar structure of the ligand is associated with the presence of three active protons (at the N atoms) capable of hydrogen bonding. Two protons, viz., H(1N) and H(5N), form rather strong intramolecular N-H...O hydrogen bonds (N(1)-H(1N), 1.00(5) Å; H(1N)...O(4), 1.75(5) Å; N(5)-H(5N), 0.89(6) Å; H(5N)...O(1), 1.98(6) Å), whereas the third hydrogen atom, viz., H(4N), is involved in a short H...O contact to a much lesser extent (N(4)-H(4N), 0.81(4) Å;H(4N)...O(1), 2.31(5) Å).

The presence of a magnetic center, *viz.*, the Ni<sup>II</sup> atom in the 20-electron configuration, is responsible for the magnetic properties of complex **4**. The magnetic susceptibility measurements demonstrated that the effective magnetic moment of complex **4** varies from 0.77 to 2.89  $\mu_{\rm B}$  in the temperature range of 2–301 K (Fig. 2).

The reaction of compound **1** with **3** in MeCN in the presence of polymeric cobalt trimethylacetate  $[Co(OH)_n(OOCCMe_3)_{2-n}]_x$  (5, n = 0.05-0.1) afforded



Fig. 2. Temperature dependence of the effective magnetic moment  $(\mu_{eff})$  for complex 4.



Fig. 3. Molecular structure of complex 6.

the tetranuclear complex  $Co_4(\mu_4-O)(\mu-OOCCMe_3)_4L_2$ (6), where L is the product of condensation of two molecules 3 with one molecule 1 (see Scheme 1). Unlike the analogous reactions involving nickel trimethylacetate derivatives, condensation in the reaction under consideration is accompanied by oxidation of Schiff's base to the benzimidazole derivative. X-ray diffraction study of brown single crystals of solvate  $6 \cdot 2 MeCN \cdot C_6 H_5 Me$ (Fig. 3, Table 2) showed that four Co atoms in molecule **6** are linked through the bridging O atom (Co(1)-O,1.949(3) Å; Co(2)–O, 1.928(4) Å) and are also linked in pairs through two bridging trimethylacetate groups (Co(1)-O(1), 2.121(6) Å; Co(1)-O(3), 2.008(6) Å; Co(2)-O(2) 1.945(6), Å; Co(2)-O(4), 1.989(5) Å). Although the newly formed ligands in compound 6 are formally built of two diamine molecules 3 and one dicarbaldehyde molecule 1 (like in complex 4), their geometric characteristics are substantially different. The involvement of the N-substituted amino groups in condensation giving rise to two benzimidazole fragments leads to an essential change in the coordinating ability of the L ligand and virtually prevents the N atoms of these groups from being involved in interactions with the metal atoms. As a result, unlike the "enveloping" mode of coordination of the ligands in nickel complex 4, the bridg-

Table 2. Principal bond lengths (d) and bond angles ( $\omega$ ) in complex 6

Bond	$d/{ m \AA}$	Angle	ω/deg
Co(1)-0	1.949(3)	O-Co(1)-O(3a)	101.9(2)
Co(1) - O(3)	2.008(6)	O(3a) - Co(1) - N(1)	140.7(2)
Co(1) - N(3)	2.168(6)	O(3a) - Co(1) - O(1a)	87.6(2)
Co(2) - O(2)	1.945(6)	O - Co(1) - N(3)	100.98(18
Co(2) - N(4)	1.998(6)	N(1) - Co(1) - N(3)	80.4(3)
O(2) - C(1)	1.279(10)	O - Co(2) - O(2)	116.9(3)
O(4) - C(6)	1.257(9)	O(2) - Co(2) - O(4)	100.6(3)
Co(1) - O(1)	2.121(6)	O(2) - Co(2) - N(4)	105.5(3)
Co(1) - N(1)	2.032(6)	Co(2) - O - Co(2a)	115.0(4)
Co(2) - O	1.928(4)	Co(2a) - O - Co(1)	100.09(5)
Co(2) - O(4)	1.989(5)	O - Co(1) - N(1)	117.4(3)
O(1) - C(1)	1.259(9)	O - Co(1) - O(1a)	100.06(14
O(3) - C(6)	1.264(9)	N(1) - Co(1) - O(1a)	87.7(2)
., .,		O(3a) - Co(1) - N(3)	90.6(2)
		O(1a) - Co(1) - N(3)	158.8(2)
		O - Co(2) - O(4)	104.3(2)
		O - Co(2) - N(4)	119.6(2)
		O(4) - Co(2) - N(4)	108.1(3)
		Co(2) - O - Co(1)	106.35(5)
		Co(1) - O - Co(1a)	129.7(4)

*Note.* The atoms denoted by the index "a" are generated from the basis atoms by the symmetry operation 1 - x, y, -z + 0.5.

ing mode is observed for each new ligand in complex **6**. In the latter complex, the ligands each are coordinated to two Co atoms, one metal atom being coordinated in a monodentate mode through the N atom of the benzimidazole ring (Co(2)—N(4), 1.998(6) Å), whereas another metal atom being coordinated in a chelating mode through the N atoms of the pyrrole (Co(1)—N(1), 2.032(6) Å) and benzimidazole rings (Co(1)—N(3), 2.168(6) Å). As a result, only the N atoms suitable for binding with the metal centers are involved in coordination.

Apparently, the observed differences in the transformations of diamine 3 within the coordination spheres about the Co and Ni atoms in the trimethylacetate complexes are associated with the difference in the electronic characteristics of the metal centers (the Co<sup>II</sup> atom has one less electron than the Ni<sup>II</sup> atom) and, correspondingly, with the difference in coordinating ability of the atoms. Generally, the Ni<sup>II</sup> atom in the trimethylacetate complexes (except for the binuclear lantern-like dimers) has an octahedral ligand environment,<sup>6</sup> unlike the Co atom, which can have different coordination numbers (from 4 to 6).<sup>7,8</sup> In addition, the Co<sup>II</sup> atom in these systems is readily oxidized to form Co<sup>III</sup> derivatives,<sup>9,10</sup> which can serve as intermediates in the process under study. Probably, it is these intermediates that stimulate oxidation of Schiff's base to the benzimidazole derivative accompanied by reduction of the metal center to the starting Co<sup>II</sup> state. The results of the present study demonstrate that the chemical assembly of complicated organic molecules can be controlled by choosing metal centers at which condensation of pyrrole-2,5-dicarbaldehyde with 2-aminodiphenylamine proceeds under similar conditions.

## **Experimental**

Pyrrole-2,5-dicarbaldehyde (1),<sup>11</sup> nonanuclear nickel trimethylacetate Ni<sub>9</sub>(HOOCMe<sub>3</sub>)<sub>4</sub>( $\mu_4$ -OH)<sub>3</sub>( $\mu_3$ -OH)<sub>3</sub>(OOCCMe<sub>3</sub>)<sub>12</sub> (2),<sup>12</sup> and polymeric cobalt trimethylacetate [Co(OH)<sub>n</sub>(OOCCMe<sub>3</sub>)<sub>2-n</sub>]<sub>x</sub> (5)<sup>7</sup> (n = 0.05-0.1) were prepared according to known procedures. All reactions were carried out with the use of anhydrous solvents. The IR spectra were recorded on a Specord M-80 spectrophotometer in KBr pellets in the frequency range of 392–4000 cm<sup>-1</sup>. The static magnetic susceptibility was measured on a SQUID MPMS-59 magnetometer in the temperature range of 2–301 K.

SynthesisoftheNi(OOCCMe\_3)\_2[(PhHN)C\_6H\_4NHCHC\_4H\_2NCHNC\_6H\_4(NHPh)]complex (4). Diamine 3 (130 mg, 0.0575 mmol) and dicarb-aldehyde 1 (62 mg, 0.5 mmol) were successively dissolved inMeCN (70 mL) and then complex 2 (184 mg, 1 mmol) wasadded. The reaction solution was stirred at ~20 °C for 30 min,concentrated to 15 mL at 50 °C, and kept at ~20 °C for 6 h.The large prismatic dark-brown (almost black) crystals thatformed were separated from the mother liquor by decantation,

washed with cold toluene, and dried *in vacuo*. The yield was 243.5 mg (68%). IR, v/cm<sup>-1</sup>: 3648 w, 3056 w, 2960 w, 2920 w, 2856 w, 1619 m, 1600 m, 1568 m, 1520 m, 1504 m, 1448 m, 1408 s, 1352 m, 1336 m, 1320 s, 1224 w, 1160 w, 1048 m, 1016 m, 928 w, 774 s, 696 s. Found (%): C, 67.06; H, 6.05; N, 9.84. C<sub>40</sub>H<sub>43</sub>N<sub>5</sub>NiO<sub>4</sub>. Calculated (%): C, 67.04; H, 6.01; N, 9.78.

Synthesis of the Co<sub>4</sub>( $\mu_4$ -O)( $\mu$ -OOCCMe<sub>3</sub>)<sub>4</sub>·L<sub>2</sub> complex (6). Solid compound 1 (62 mg, 0.5 mmol) and solid compound 3 (184 mg, 1 mmol) were successively added to a solution of polymer 5 (130 mg) in MeCN (80 mL). The resulting solution was heated to 70 °C for 10 min. The dark-brown solution was concentrated to 15 mL, toluene (10 mL) was added, and the reaction mixture was kept at 5 °C for 2 days. The dark-brown crystals that formed were separated from the solution by decantation, washed with cold MeCN, and dried *in vacuo* (25 °C, 0.1 Torr). The yield was 121 mg (58%). IR, v/cm<sup>-1</sup>: 2960 w, 2928 w, 2872 w, 1576 s, 1480 m, 1450 m, 1416 m, 1384 m, 1328 w, 1224 m, 1152 w, 1032 w, 896 w, 792 w, 768 m, 744 m, 688 m, 632 w, 464 w. C<sub>80</sub>H<sub>76</sub>Co<sub>4</sub>N<sub>10</sub>O<sub>9</sub>•C<sub>7</sub>H<sub>8</sub>. Found (%): C, 63.31; H, 5.07; N, 8.55. Calculated (%): C, 63.36; H, 5.10; N, 8.50.

X-ray diffraction study of complexes 4 and 6. The X-ray diffraction data sets for complex 4 and solvate  $6 \cdot 2 MeCN \cdot C_6 H_5 Me$ were collected in the Center of X-ray Diffraction Studies (A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences) on a Bruker AXS SMART-1000 diffractometer equipped with a CCD detector ( $\lambda$ (Mo), graphite monochromator,  $\omega$  scanning technique, scan step was 0.3°, frames were exposed for 30 s,  $2\theta_{max} = 60^{\circ}$ ) according to a standard procedure.<sup>13</sup> The semiempirical absorption correction was applied.<sup>14</sup> The structure was solved by direct methods using the SHELXS97 program package<sup>15</sup> and refined by the full-matrix least-squares method with anisotropic thermal parameters for all nonhydrogen atoms using the SHELXL97 program package.<sup>16</sup> The positions of the H atoms of the amino groups were revealed from difference electron density maps and refined isotropically. The positions of the remaining H atoms were calculated geometrically and refined using the riding model.

Table 3. Crystallographic parameters of complexes 4 and  $6 \cdot 2 \text{MeCN} \cdot \text{C}_6 \text{H}_5 \text{Me}$ 

Compound	4	$6 \cdot 2 MeCN \cdot C_6 H_5 Me$
Compound	C40H43N5O4Ni	C <sub>91</sub> H <sub>90</sub> N <sub>12</sub> O <sub>9</sub> Co <sub>4</sub>
Space group	C2/c	Cc
a/Å	28.789(9)	18.554(3)
b/Å	17.276(4)	19.482(3)
c/Å	19.949(5)	25.971(4)
β/deg	133.493(13)	103.099(4)
$V/Å^3$	7198(3)	9143(2)
Z	8	4
$d_{\rm calc}/{\rm g}~{\rm cm}^{-3}$	1.322	1.258
$\mu/\text{mm}^{-1}$	0.587	0.773
$\theta$ range/deg	1.56-30.25	1.54-30.01
Number of independe	ent	
reflections	6176	6632
$R_1$	0.0798	0.0819
$wR_2$	0.2021	0.1975

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The principal crystallographic parameters of compounds 4 and 6 are given in Table 3. The selected bond lengths and bond angles for compounds 4 and 6 are listed in Tables 1 and 2, respectively.

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## References

- A. Yu. Chernyadyev, Yu. A. Ustynyuk, G. G. Aleksandrov, A. A. Sidorov, V. M. Novotortsev, V. N. Ikorskii, S. E. Nefedov, I. L. Eremenko, and I. I. Moiseev, *Izv. Akad. Nauk, Ser. Khim.*, 2001, 1271 [*Russ. Chem. Bull., Int. Ed.*, 2001, **50**, 1336 (Engl. Transl.)].
- A. E. Malkov, G. G. Aleksandrov, V. N. Ikorskii, A. A. Sidorov, I. G. Fomina, S. E. Nefedov, V. M. Novotortsev, I. L. Eremenko, and I. I. Moiseev, *Koord. Khim.*, 2001, 27, 677 [*Russ. J. Coord. Chem.*, 2001, 27 (Engl. Transl.)].
- 3. I. G. Fomina, A. A. Sidorov, and A. Yu. Chernyadyev, *Abstr. XVIIth International Chernyaev Meeting on Chemistry, Analysis and Technology of Platinum Metals*, Moscow, 2001, 125.
- A. Yu. Chernyadyev, Yu. A. Ustynyuk, O. V. Yazev, E. A. Kataev, M. D. Reshetova, A. A. Sidorov, G. G. Aleksandrov, V. N. Ikorskii, V. M. Novotortsev, S. E. Nefedov, I. L. Eremenko, and I. I. Moiseev, *Izv. Akad. Nauk, Ser. Khim.*, 2001, 2334 [*Russ. Chem. Bull.*, *Int. Ed.*, 2001, **50**, 2445].
- 5. H. A. Frank, O. Kennard, D. G. Watson, L. Brammer, A. G. Orpen, and R. Taylor, *J. Chem. Soc.*, *Perkin Trans. 2*, 1987, S1.
- I. L. Eremenko, S. E. Nefedov, A. A. Sidorov, and I. I. Moiseev, *Izv. Akad. Nauk, Ser. Khim.*, 1999, 409 [*Russ. Chem. Bull.*, 1999, 48, 405 (Engl. Transl.)].

- M. A. Golubnichaya, A. A. Sidorov, I. G. Fomina, M. O. Ponina, S. M. Deomidov, S. E. Nefedov, I. L. Eremenko, and I. I. Moiseev, *Izv. Akad. Nauk, Ser. Khim.*, 1999, 1773 [*Russ. Chem. Bull.*, 1999, **48**, 1751 (Engl. Transl.)].
- M. A. Golubnichaya, A. A. Sidorov, I. G. Fomina, L. T. Eremenko, S. E. Nefedov, I. L. Eremenko, and I. I. Moiseev, *Zh. Neorg. Khim.*, 1999, 44, 1479 [*Russ. J. Inorg. Chem.*, 1999, 44 (Engl. Transl.)].
- A. A. Sidorov, M. O. Ponina, S. M. Deomidov, V. M. Novotortsev, A. Demonceau, S. E. Nefedov, I. L. Eremenko, and I. I. Moiseev, *J. Chem. Soc., Chem. Commun.*, 2000, 1383.
- A. A. Sidorov, M. O. Talismanova, I. G. Fomina, G. G. Aleksandrov, V. M. Novotortsev, A. Demonceau, S. E. Nefedov, I. L. Eremenko, and I. I. Moiseev, *Izv. Akad. Nauk, Ser. Khim.*, 2001, 2106 [*Russ. Chem. Bull., Int. Ed.*, 2001, **50**, 2206].
- 11. R. Miller and K. Olsson, Acta Chem. Scand., 1981, B35, 303.
- I. L. Eremenko, M. A. Golubnichaya, S. E. Nefedov, A. A. Sidorov, I. F. Golovaneva, V. I. Burkov, O. G. Ellert, V. M. Novotortsev, L. T. Eremenko, A. Sousa, and M. R. Bermejo, *Izv. Akad. Nauk, Ser. Khim.*, 1998, 725 [*Russ. Chem. Bull.*, 1998, 47, 704 (Engl. Transl.)].
- 13. SMART (Control) and SAINT (Integration) Software, Version 5.0, Bruker AXS Inc., Madison, WI, 1997.
- 14. G. M. Sheldrick, SADABS, Program for Scaling and Correction of Area Detector Data, Göttingen University, Göttingen (Germany), 1997 (based on the method of R. H. Blessing, Acta Crystallogr., A, 1995, 51, 33).
- G. M. Sheldrick, SHELXS97, Program for the Solution of Crystal Structures, Göttingen University, Göttingen (Germany), 1997.
- G. M. Sheldrick, SHELXL97, Program for the Refinement of Crystal Structures, Göttingen University, Göttingen (Germany), 1997.

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