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Dedicated to the 90th birthday of Academician I.I. Moiseev

Synthesis of New Bulky Bis(amidine) with the Conformationally Rigid *meta*-Phenylene Bridge and Its Dilithium Derivative $[1,3\text{-}C_6H_4\{NC(Ph)N(2,6\text{-}iso\text{-}Pr_2C_6H_3)\}_2]Li_2(TMEDA)_2$

A. O. Tolpygin[a], A. V. Cherkasov[a], G. K. Fukin[a], and A. A. Trifonov[a, b, *]

[a] *Razuvaev Institute of Organometallic Chemistry, Russian Academy of Sciences,*
Nizhny Novgorod, 603600 Russia

[b] *Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences,*
Moscow, 119991 Russia

**e-mail: trif@iomc.ras.ru*

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Abstract—The reaction of 2,6-diisopropylaniline with *m*-phenylenedibenzimidoyl chloride $1,3\text{-}C_6H_4\{N{=}C(Ph)Cl\}_2$ in the presence of triethylamine in a toluene solution affords bis(amidine) $1,3\text{-}C_6H_4\{NC(Ph)NH(2,6\text{-}iso\text{-}Pr_2C_6H_3)\}_2$ (H_2L) in which two amidinate fragments are bound by the conformationally rigid *m*-phenylene linker. The metallation of bis(amidine) by *n*-butyllithium in the presence of tetramethylethylenediamine (TMEDA) occurs readily in a tetrahydrofuran solution at −70°C to form the corresponding lithium amidinate complex $1,3\text{-}C_6H_4\{NC(Ph)N(2,6\text{-}iso\text{-}Pr_2C_6H_3)\}_2Li_2(TMEDA)_2$ (**I**). The X-ray structure analysis shows that complex **I** is monomeric (CIF file CCDC no. 1873201). Each amidinate fragment NCN is bound to one lithium atom via the κ^2-N,N chelating mode.

Keywords: ansa-bis(amidinate) ligand, alkaline metals, lithium bis(amidinate), synthesis, structure

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INTRODUCTION

Monoanionic amidinate ligands $[RC(NR')_2]^-$ have been widely used during the recent two decades for the synthesis of complexes of both metals of the main groups and early and late transition metals [1–6]. Amidinate ligands behave as four-electron donor systems providing a high electrophilicity of the metal center in the corresponding coordination compounds [7, 8]. Owing to a variety of coordination modes with metal ions and the easiness of tuning their electronic and steric properties by the variation of substituents at nitrogen and carbon atoms and the introduction of additional donor groups into the side chain [9–13] or binding two NCN fragments between each other through an appropriate bridging group [14–17], the amidinate and bis(amidinate) ligands are considered as a promising platform for the synthesis of a wide range of coordination compounds of metals of various nature. Lithium amidinates are widely used precursors for the synthesis of the corresponding complexes of diverse metals by exchange reactions.

In this work, we report the synthesis of new bis(amidine) $1,3\text{-}C_6H_4\{NC(Ph)NH(2,6\text{-}iso\text{-}Pr_2C_6H_3)\}_2$ (H_2L) in which the amidinate fragments are linked by the conformationally rigid bridging group $1,3\text{-}C_6H_4$ and the synthesis and structure of its dilithium derivative $1,3\text{-}C_6H_4\{NC(Ph)N(2,6\text{-}iso\text{-}Pr_2C_6H_3)\}_2Li_2(TMEDA)_2$ (**I**) (TMEDA is tetramethylethylenediamine).

EXPERIMENTAL

All procedures on the synthesis and isolation of the products were carried out in an argon atmosphere or in a vacuum apparatus using the standard Schlenk technique. Diethyl ether and tetrahydrofuran (THF) were dehydrated by potassium hydroxide and then distilled over sodium benzophenone ketyl. Hexane and toluene were dehydrated by reflux and distillation over metallic sodium. Benzene-d_6 was dehydrated over metallic sodium, degassed, and condensed in vacuo. Pyridine-d_5 was dehydrated over calcium hydride. *meta*-Phenylenebenzimidoyl chloride $1,3\text{-}C_6H_4\{N{=}C(Ph)Cl\}_2$ was prepared according to a published procedure [18]. Pyridine-d_5, benzene-d_6, $CDCl_3$, $1,3\text{-}C_6H_4(NH_2)_2$, and *iso*-$PrC_6H_3NH_2$ were commercial reagents (Acros).

IR spectra were recorded on a Bruker-Vertex 70 instrument. The samples were prepared under dry argon as suspensions in Nujol. ^{1}H, ^{13}C, and ^{7}Li NMR spectra were detected on Bruker Avance DPX-200 and Bruker Avance III instruments (400 MHz, 25°C, C_6D_6, pyridine-d$_5$, $CDCl_3$). Chemical shifts were presented in ppm relative to the known shifts of residual protons of deuterated solvents.

Synthesis of 1,3-phenylenobis{(2,6-diisopropylphenyl)phenyl}amidine (H_2L). Triethylamine (15.0 mL, 10.95 g, 108.16 mmol) and then benzoyl chloride (12.5 mL, 15.20 g, 108.16 mmol) were poured to a solution of *m*-phenylenediamine (5.85 g, 54.08 mmol) in dichloromethane (100 mL). The reaction mixture was stirred for 12 h at 40°C and washed with water. The organic phase was separated and dried over calcined calcium chloride. The solvents were removed on a rotary evaporator, and a solid residue was washed and dried in vacuo at 50°C to a constant weight. The yield was 15.57 g (91%).

Phosphorus pentachloride (20.50 g, 98.45 mmol) was added to a suspension of *N*,*N*'-(1,3-phenylene)dibenzamide (15.57 g, 49.22 mmol) in chlorobenzene (50 mL), and the mixture was stirred at 60°C. After the end of hydrogen chloride evolution, the solvent was removed in vacuo and a residue was dissolved in toluene. 2,6-Diisopropylaniline (17.45 g, 98.46 mmol) and Et_3N (9.96 g, 98.46 mmol) were added to the obtained solution of 1,3-C_6H_4{N=C(Ph)Cl}$_2$ (17.39 g, 49.22 mmol) in toluene (70 mL). The reaction mixture was stirred at 110°C for 72 h. The solvent was removed in vacuo, and a solid residue was dissolved in diethyl ether (100 mL) and washed with a 1% aqueous solution of Na_2CO_3 (3 × 100 mL). The ethereal layer was separated and dried over calcined $MgSO_4$. The solvent was removed in vacuo, and a solid residue was recrystallized from hexane. The yield of bis(amidine) H_2L as a white amorphous powder was 16.87 g (54%).

For $C_{44}H_{50}N_4$			
Anal. calcd., %	C, 83.07	H, 7.94	N, 8.82
Found, %	C, 83.38	H, 8.01	N, 8.64

Mass spectrum (EI; 70 eV; *m/z* (I_{rel}, %)): 634.3 $[M]^+$ (15). IR (ν, cm^{-1}): 3390 s, 1632 s, 1596 s, 1538 s, 1351 s, 1263 s, 1094 s, 1031 s, 777 s. ^{1}H NMR (200 MHz; 25°C; $CDCl_3$; δ, ppm (*J*, Hz)): 1.30 (d, 24H, $(CH_3)_2CH$, $^3J_{H,H}$ = 6.9 Hz); 2.96 (sept, 4H, $(CH_3)_2CH$, $^3J_{H,H}$ = 6.9 Hz); 6.08 (br.s, 2H, N*H*); 6.83 (t, 2H, *iso*-$Pr_2C_6H_3$, $^3J_{H,H}$ = 7.7 Hz); 7.07 (d, 4H, *iso*-$Pr_2C_6H_3$, $^3J_{H,H}$ = 7.7 Hz); 7.14 (br.s, 2H, C_6H_4, *iso*-$Pr_2C_6H_3$); 7.21 (d, 4H, C_6H_4, *iso*-$Pr_2C_6H_3$, $^3J_{H,H}$ = 6.1 Hz); 7.34 (s, 5H, C_6H_4, *iso*-$Pr_2C_6H_3$); 7.49 (s, 3H, C_6H_4, *iso*-$Pr_2C_6H_3$). ^{13}C NMR (100 MHz; 25°C; $CDCl_3$; δ, ppm): 22.5 ($(CH_3)_2CH$); 27.9 ($(CH_3)_2CH$); 118.6, 122.8, 123.6, 128.3, 128.8, 129.8, 132.5 (C_6H_4, *iso*-$Pr_2C_6H_3$); 140.3 (N*C*N).

Synthesis of dilithium bis(tetramethylethylenediaminate)1,3-phenylenobis{(2,6-diisopropylphenyl)phenyl}amidinate (I). A 1.14 M solution of *n*-BuLi in hexane (11.2 mL, 12.75 mmol) was added at –70°C to a solution of compound **I** (4.05 g, 6.33 mmol) in THF (5 mL). The temperature of the reaction mixture was slowly brought to room temperature, and the mixture was stirred for 2 h. The addition of TMEDA (5 mL) to the reaction mixture resulted in the precipitation of a yellow powder of compound **I**. The yield was 3.37 g (82%). Yellow crystals of complex **I** were obtained by the recrystallization of the obtained powder from a THF–hexane (1 : 5) system.

For $C_{56}H_{80}N_8Li_2$			
Anal. calcd., %	C, 76.50	H, 9.17	N, 12.75
Found, %	C, 76.72	H, 9.21	N, 12.81

IR (ν, cm^{-1}): 1640 s, 1617 s, 1575 s, 1315 s, 1254 s, 1221 s, 1103 s, 1037 s, 946 s, 913 s, 865 s, 783 s, 741 s. ^{1}H NMR (400 MHz; 25°C, pyridine-d$_5$; δ, ppm (*J*, Hz)): 0.85 (d, 12H, $(CH_3)_2CH$, $^3J_{H,H}$ = 6.2 Hz); 0.99 (d, 12H, $(CH_3)_2CH$, $^3J_{H,H}$ = 6.2 Hz); 2.11 (s, 24H, CH_3, TMEDA); 2.33 (s, 8H, CH_2, TMEDA); 3.62 (sept, 4H, $(CH_3)_2CH$, $^3J_{H,H}$ = 6.2 Hz); 6.22–7.28 (m, 20H, C_6H_4, *iso*-$Pr_2C_6H_3$). ^{1}H NMR (400 MHz; 25°C, benzene-d$_6$; δ, ppm (*J*, Hz)): 1.13, 1.22 (d, 12H, $(CH_3)_2CH$, $^3J_{H,H}$ = 6.8 Hz); 1.97 (br.s, 32H, TMEDA); 3.60 (sept, 4H, $(CH_3)_2CH$, $^3J_{H,H}$ = 6.8 Hz); 6.78–7.11 (m, 16H, C_6H_4, *iso*-$Pr_2C_6H_3$); 7.34–7.36 (m, 4H, C_6H_4, *iso*-$Pr_2C_6H_3$). ^{13}C NMR (50 MHz; 2°C, pyridine-d$_5$; δ, ppm): 22.8, 24.7 ($(CH_3)_2CH$); 27.9 ($(CH_3)_2CH$); 45.7 (CH_3, TMEDA); 57.9 (CH_2, TMEDA); 114.2, 120.6, 122.5, 123.6, 126.9, 130.6, 141.2, 154.9 (C_6H_4, *iso*-$Pr_2C_6H_3$); 167.6 (N*C*N). ^{7}Li NMR (155.5 MHz; 25°C, pyridine-d$_5$; δ, ppm): 3.3.

The X-ray structure analysis of complex **I** was carried out on a Bruker Smart Apex diffractometer (ω scan, MoK_α radiation, λ = 0.71073 Å, *T* = 150 K). Experimental sets of intensities were integrated, absorption corrections were applied, and the structure was refined using the SMART [19], TWINABS [20], and SHELX [21] program packages. The structure was solved by a direct method and refined by full-matrix least squares for F^2_{hkl} in the anisotropic approximation for non-hydrogen atoms. Hydrogen atoms were placed in geometrically calculated positions and refined isotropically with fixed thermal parameters $U(H)_{iso} = 1.2U(C)_{eq}$ ($U(H)_{iso} = 1.5U(C)_{eq}$ for methyl fragments).

Table 1. Crystallographic data and structure refinement parameters for compound **I**

Parameter	Value
FW	879.16
Space group	$C2/c$
a, Å	20.8453(18)
b, Å	15.7829(15)
c, Å	17.4531(15)
β, deg	99.539(3)
V, Å^3	5662.7(9)
Z	4
ρ_{calcd}, mg/m^3	1.031
μ, mm^{-1}	0.060
Scan range over θ, deg	1.98–26.02
Number of measured reflections	12351
Number of reflections with $I > 2\sigma(I)$	4226
R_{int}	0.0515
Number of refined parameters	309
GOOF (F^2)	1.050
R_1 ($I > 2\sigma(I)$)	0.0805
wR_2 (for all data)	0.1992
Residual electron density (min/max), e/Å^3	–0.35/0.55

Table 2. Selected bond lengths (d) and bond angles (ω) in complex **I**

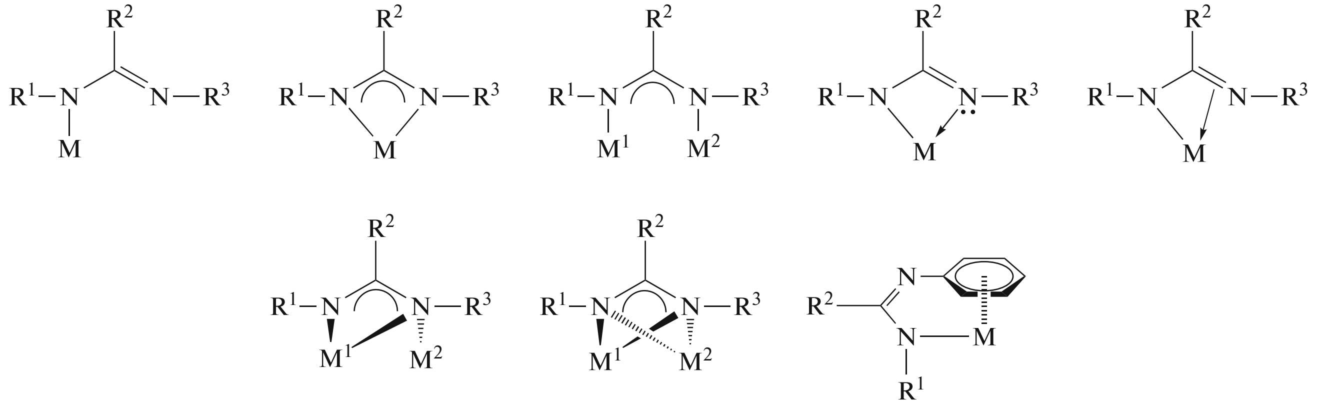
Bond	d, Å	Angle	ω, deg
Li(1)–N(2)	1.994(7)	N(2)Li(1)N(3)	123.9(4)
Li(1)–N(3)	2.035(9)	N(2)Li(1)N(1)	68.0(2)
Li(1)–N(1)	2.036(8)	N(3)Li(1)N(1)	131.4(4)
Li(1)–N(4)	2.073(8)	N(2)Li(1)N(4)	114.8(4)
N(1)–C(1)	1.331(4)	N(3)Li(1)N(4)	91.3(3)
N(2)–C(1)	1.330(4)	N(1)Li(1)N(4)	129.0(4)
N(1)–C(8)	1.423(4)	N(2)Li(1)C(1)	34.0(2)
N(2)–C(20)	1.393(4)	N(3)Li(1)C(1)	137.9(4)

The crystallographic data and structure refinement parameters for complex **I** are presented in Table 1. Selected bond lengths and bond angles are listed in Table 2.

The coordinates of atoms and other parameters for the structure of complex **I** were deposited with the Cambridge Crystallographic Data Centre (CIF file CCDC no. 1873201; ccdc.cam.ac.uk/getstructures).

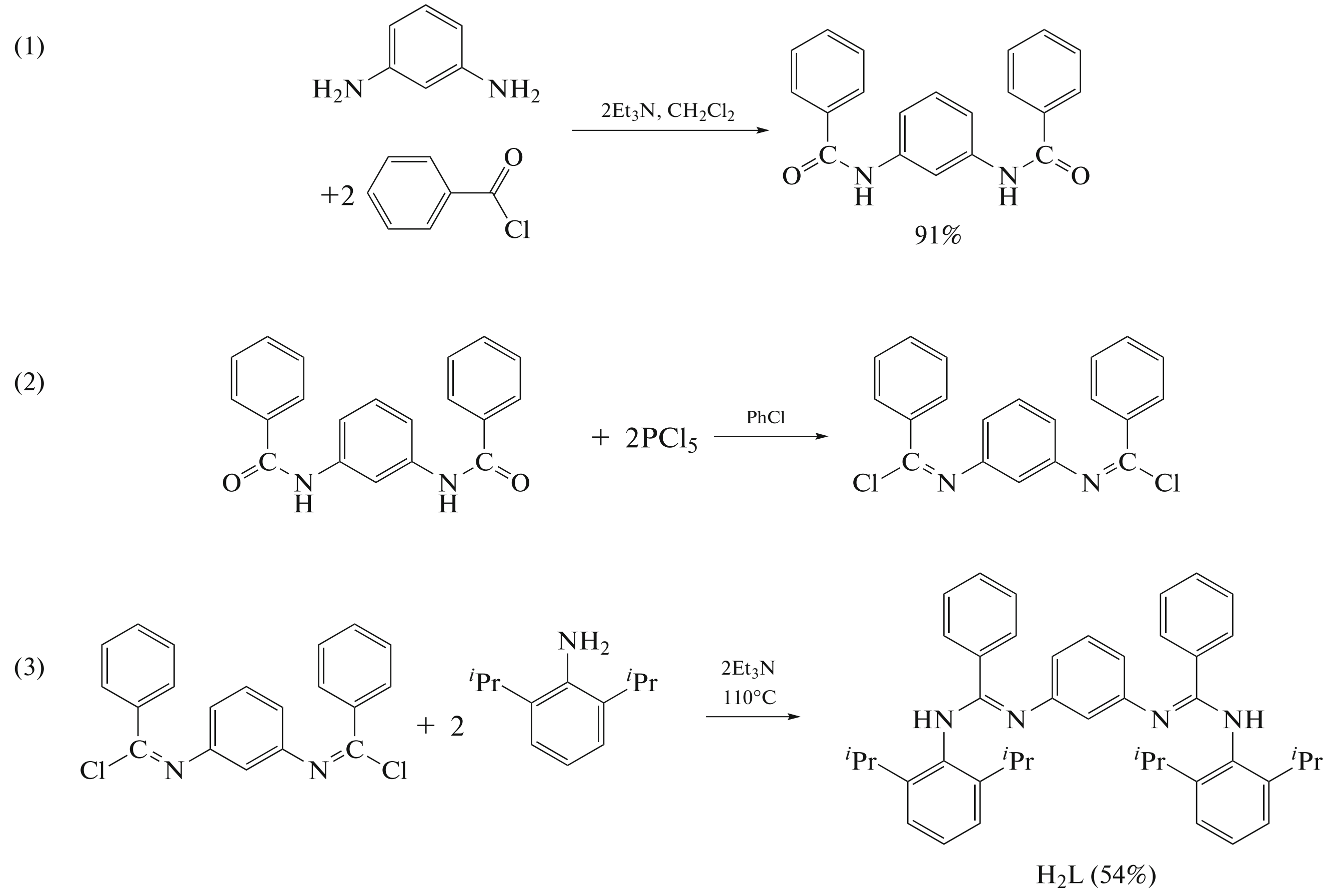
RESULTS AND DISCUSSION

Several types of binding the amidinate ligands with the metal ions (Scheme 1) are known [6]. The bis(amidinate) ligands demonstrate a wide variety of coordination possibilities. Depending on the nature and extension of the group linking two amidinate fragments, they can act as tri- and tetradentate dianionic ligands providing the synthesis of various monomeric, dimeric, and trimeric structures [14–17, 22, 23].



Scheme 1.

Bis(amidine) H_2L was obtained by the procedure developed by J. Arnold [24] for the synthesis of other representatives of this class of compounds. *N*,*N*'-(1,3-Phenylene)dibenzamide was obtained by the reaction of *m*-phenylenediamine with benzoyl chloride. Then the reaction of the corresponding benzoic acid amide with phosphorus pentachloride affords *N*,*N*'-(1,3-phenylene)dibenzimidoyl chloride ($1,3\text{-}C_6H_4\{N{=}C(Ph)Cl\}_2$) [18]. The final stage of the synthesis occurs as the reaction of *m*-phenylenedibenzimidoyl chloride with 2,6-diisopropylaniline (2 equiv mol) in toluene in the presence of triethylamine (Scheme 2). Bis(amidine) H_2L was isolated as a white amorphous powder in a yield of 54% and characterized by elemental analysis, mass spectrometry, IR spectroscopy, and NMR spectroscopy.

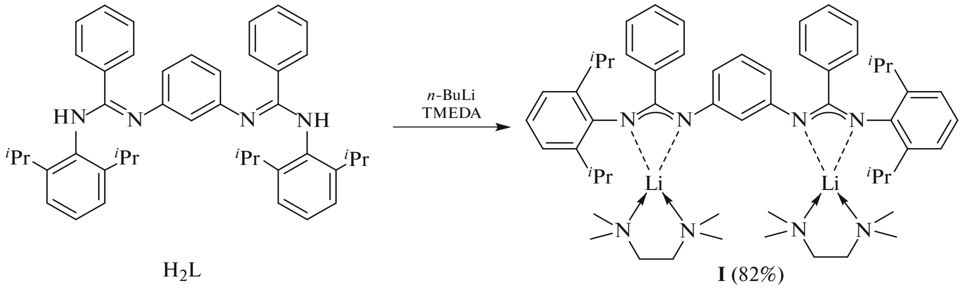


Scheme 2.

The methyl protons of the isopropyl groups appear as a doublet at 1.30 ppm with the spin-spin coupling constant equal to 6.9 Hz in the ^{1}H NMR spectrum of compound H_2L. The methine protons of the isopropyl groups give a septet with a chemical shift of 2.96 ppm and the spin–spin coupling constant $^3J_{H,H} = 6.9$ Hz. The broadened singlet at 6.08 ppm corresponds to two protons of the NH amidine fragments. Aromatic protons are observed in the characteristic range of the low field (6.83–7.49 ppm).

The IR spectrum of compound H_2L exhibits an intense absorption band at 1632 cm^{-1} corresponding to asymmetric vibrations of the C–N multiple bonds in the ligand, and the broad absorption band at 3390 cm^{-1} corresponds to vibrations of the N–H bonds.

Bis(amidine) H_2L is readily deprotonated by *n*-butyllithium in a THF solution at –70°C (Scheme 3). The treatment of the reaction mixture with TMEDA and the recrystallization of the reaction products from a THF–hexane (volume ratio 1 : 5) system gave lithium bis(amidinate) **I** as yellow crystals in a yield of 82%.



H_2L

I (82%)

Scheme 3.

The methyl protons of the isopropyl groups appear as two doublets ($^3J_{H,H}$ = 6.8 Hz) at 1.13 and 1.22 ppm in the ^{1}H NMR spectrum (benzene-d$_6$) of compound **I**. The methine protons of the isopropyl groups give a septet with a chemical shift of 3.60 ppm and a spin-spin coupling constant of 6.8 Hz. A set of signals in the characteristic range of the low field at 6.78–7.36 ppm corresponds to the aromatic protons. The methyl and methylene protons of the coordinated TMEDA molecules appear as a broadened singlet at 1.97 ppm. According to the data of ^{1}H NMR spectroscopy, complex **I** dissociates in a pyridine-d$_5$ solution, which is indicated by singlets at 2.11 and 2.33 ppm with the ratio of integral intensities of the signals equal to 12 : 4 in the ^{1}H NMR spectrum. The singlets are assigned to the methyl and methylene protons of free TMEDA. The lithium cores appear as a single signal at 3.3 ppm in the ^{7}Li NMR spectrum of complex **I** recorded in pyridine-d$_5$. The IR spectrum of compound **I** exhibits an intense absorption band at 1640 cm^{-1} corresponding to asymmetric vibrations of the CN multiple bonds of the amidinate fragments.

The crystals of complex **I** suitable for X-ray structure analysis were obtained by the slow condensation of hexane to a saturated solution of compound **I** in THF at room temperature. The molecular structure of bis(amidinate)lithium derivative **I** is presented in Fig. 1. The independent part of the crystal cell of complex **I** contains one molecule of the complex in the partial position on the 2-fold symmetry axis passing through the C(21) and C(23) atoms. The lithium atom in complex **I** was coordinated by two nitrogen atoms of the *ansa*-bis(amidinate) ligand $[1,3\text{-}C_6H_4\{NC(Ph)\text{-}N(2,6\text{-}Pr^i_2C_6H_3)\}_2]^{2-}$ and two nitrogen atoms of the TMEDA molecule. The formal coordination number of lithium in compound **I** is four. The amidinate fragments are unfolded relative to each other, and the lithium atoms are located at different sides of the plane in which the *m*-phenylene group lies. The Li–N bond lengths in complex **I** (1.994(7), 2.036(8) Å) are comparable with similar distances in the known lithium amidinate complexes [12, 25–32]. The C–N bond lengths in the amidinate fragments (C(1)–N(1) and C(1)–N(2) (1.330(4), 1.331(2) Å)) are close and indicate the delocalization of negative charges inside the NCN groups. The dihedral angle between the NCN planes of the amidinate fragments is 55.7(3)°.

Thus, we synthesized new bis(amidine) $1,3\text{-}C_6H_4\{NC(Ph)NH(2,6\text{-}iso\text{-}Pr_2C_6H_3)\}_2$ containing the conformationally rigid $1,3\text{-}C_6H_4$ linker capable of coordinating to lithium ions via the κ^2-N,N chelating mode. The use of the bidentate-chelating Lewis base (TMEDA) made it possible to isolate the lithium bis(amidinate) complex $1,3\text{-}C_6H_4\{NC(Ph)N(2,6\text{-}iso\text{-}Pr_2C_6H_3)\}_2Li_2(TMEDA)_2$ (**I**) as a monomer.

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This work was carried out in terms of the state task (theme no. 44.4, registration no. AAAA-A16-

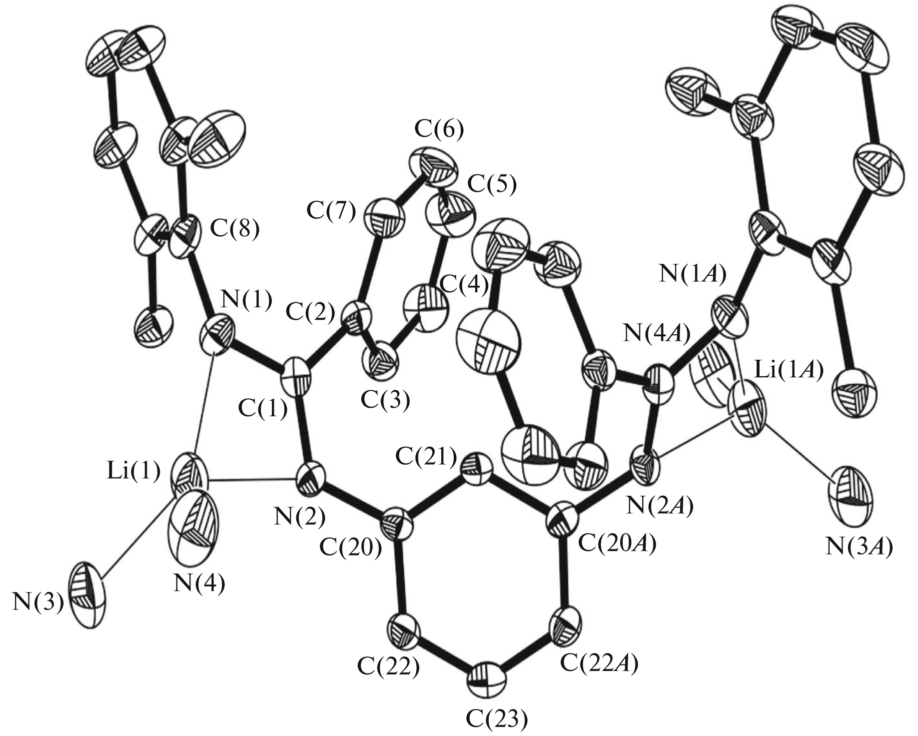

Fig. 1. Molecular structure of compound **I**. Thermal ellipsoids are presented with 30% probability. Hydrogen atoms and carbon atoms of the TMEDA molecules are omitted for clarity. The molecule lies on the 2-fold symmetry axis passing through the C(21) and C(23) atoms (*–x, y,* 1/2 *– z*).

116122110054-8) using the scientific equipment of the Center for Collective Use “Analytical Center of the Razuvaev Institute of Organometallic Chemistry of the Russian Academy of Sciences.” The X-ray structure studies were conducted in terms of the state task (theme no. 44.2, registration no. AAAA-A16-116122110053-1).

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