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Reactions of Acenaphthenediimine Aluminum Hydride with 1,3-Dicyclohexylcarbodiimide and 2,6-Di-*tert*-Butyl-4-Methylphenol

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Abstract—The reaction of [(Dpp-Bian)AlH(THF)] (**I**) (Dpp-Bian = 1,2-bis[(2,6-diisopropylphenyl)imino]acenaphthene) with 1,3-dicyclohexylcarbodiimide (DCC) is accompanied by reduction of the carbodiimide C=N bond giving the complex [(Dpp-Bian)Al(DCC(H))] (**II**). In the reaction of hydride **I** with 2,6-di-*tert*-butyl-4-methylphenol, the Al–H bond is retained to give the amino-amine complex [(Dpp-BianH)Al(H)(OC_6H_2-2,6-*tert*-Bu_2-4-Me)] (**III**). In compounds **II** and **III**, the diimine ligand is reduced to the dianion. New diamagnetic compounds **II** and **III** were characterized by IR and NMR spectroscopy, elemental analysis, and X-ray diffraction (CFA files CCDC nos. 1903665 (**II**) and 1903666 (**III**)).

Keywords: aluminum, hydrides, redox active ligands, diimine ligands, synthesis, molecular structure

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INTRODUCTION

Aluminum hydrides are actively used in industry as components of Ziegler–Natta catalysts for α-olefin polymerization and oligomerization and synthesis of stereoregular diene rubbers [1, 2]. In addition, they are effective reagents for the preparation of aluminum chalcogenides [3–8] and alumoxanes [7, 9] and are also used as reducing agents [10]. In particular, an important class of aluminum hydrides is represented by complexes in which the Al–H group is chelated by N,N-ligands, the reactivity of which has been studied in sufficient detail [11–17]. Reactions of these derivatives with compounds containing both an active hydrogen atom and multiple bonds receive particular attention, because of their similarity to the elementary steps of the catalytic cycles involved in functionalization of unsaturated organic molecules. For example, there are published data about reactions of β-diketiminate aluminum hydrides with water [7], phenols, alcohols [18–21], organic acids [5, 20, 22–25], amines, and thiols [19, 26–28]; stoichiometric reductive reactions of unsaturated compounds containing C=X (X = N, O, S), N=N, or C≡N groups [9, 29–35]; and transformation of amidinate [36] and β-diketiminate [37] hydrides to acetylenides. In addition, activation of O–H and N–H bonds by bis(imino)pyridine aluminum monohydride [($^{Ph}I_2P^{2-}$)AlH] ($^{Ph}I_2P$ = 2,6-(2,6-*iso*-$Pr_2C_6H_3N{=}CPh)_2C_5H_3N$) [38, 39] and formation of alumoxanes, [{($^{Ph}HI_2P^{-}$)AlH}$_2$(μ-O)] and [{($^{Ph}HI_2P^{-}$)-Al(OH)}$_2$(μ-O)], upon the reaction with one equivalent or an excess of water, respectively, were reported [40].

Recently, we prepared for the first time aluminum hydrides with radical anion and dianion acenaphthene-1,2-diimine ligands: [(Dpp-Bian)Al(H)$_2$-Li(THF)$_3$], [{Dpp-Bian(H_2)}Al(H)$_2$Li(Et_2O)$_2$], [(Dpp-Bian)AlH(THF)] (**I**), [(Dpp-Bian)AlH(Cl)], and [(Dpp-Bian)AlH(Me)] (Dpp-Bian = 1,2-bis[(2,6-diisopropylphenyl)imino]acenaphthene) [41, 42]. The complex [(Dpp-Bian)Al(H)$_2$Li(THF)$_3$] was found to react with benzophenone (the product [(Dpp-Bian)Al(H)($OCHPh_2$)Li(THF)$_2$] was isolated and characterized) and with 2,6-di-*tert*-butyl-4-methylphenol (this gave [(Dpp-Bian)Al(H)(OC_6H_2-2,6-*tert*-Bu_2-4-Me)][Li(THF)$_4$]) [41]. However, this hydride proved to be inert towards PhC≡CH, $Me_3SiC{\equiv}CH$, and *iso*-Pr_2NH [41].

Here we report the molecular structure and spectral characteristics of the complexes [(Dpp-Bian)-Al(DCC(H))] (**II**) and [(Dpp-BianH)Al(H)(OC_6H_2-2,6-*tert*-Bu_2-4-Me)] (**III**), which are obtained by the reaction of compound **I** with 1,3-dicyclohexylcarbodiimide (DCC) and 2,6-di-*tert*-butyl-4-methylphenol, respectively.

EXPERIMENTAL

All of the synthesized compounds are sensitive to oxygen and water; therefore, all operations on their synthesis, isolation, and identification were carried out in a vacuum using Schlenk technique or in a nitrogen atmosphere (Glovebox M. Braun). Dpp-Bian was prepared by condensation of acenaphthenequinone and 2,6-di-isopropylaniline (both Aldrich) in acetonitrile. Diethyl ether was dried and stored over sodium benzophenone. The solvent was withdrawn by vacuum condensation immediately prior to use. Deuterobenzene (Aldrich) was dried over sodium benzophenone and withdrawn by vacuum condensation into NMR tubes containing samples for recording the NMR spectra. Compound **I** was prepared by a reported procedure [41].

Synthesis of [(Dpp-Bian)Al(DCC(H))] (II). DCC (0.253 g, 1.25 mmol) was added to a solution of compound **I** (0.75 g, 1.25 mmol) in diethyl ether (20 mL). The mixture was stirred for 15 min at 50°C. After cooling to room temperature, the resulting blue solution was concentrated (5 mL) and kept for 24 h at 10°C. The precipitated blue crystals of compound **II** were separated by decantation, washed with cold diethyl ether, and dried in a vacuum. The yield was 0.46 g (64%). $T_m > 193$ (dec.).

^{1}H NMR (200 MHz; C_6D_6; 298 K; δ, ppm (*J*, Hz)): 7.61 (s, 1H), 7.30 (s, 6H), 7.08 (d, 2H), 6.87 (m, 2H), 6.20 (d, 2H, *J* = 6.8), 3.95 (sept, 4H, *J* = 6.8), 2.78 (br.s, 2H), 1.60–1.42 (m, 9H), 1.33 (d, 12H, *J* = 6.8), 1.24 (d, 12H, *J* = 6.8), 0.97 (br.s, 10H).

IR (ν, cm^{-1}): 1913 w, 1856 w, 1787 w, 1655 m, 1616 w, 1591 w, 1564 w, 1539 s, 1513 s, 1438 w, 1321 s, 1261 m, 1220 s, 1193 w, 1179 w, 1135 w, 1119 m, 1079 w, 1056 w, 998 w, 961 w, 939 s, 925 s, 829 w, 844 w, 813 m, 801 w, 764 s, 723 m, 686 m, 667 s, 622 w, 603 w.

For $C_{49}H_{63}N_4Al$		
Anal. calcd., %	C, 80.07	H, 8.64
Found, %	C, 80.03	H, 8.58

Synthesis of [(Dpp-BianH)Al(H)(OC$_6$H$_2$-2,6-*tert*-Bu$_2$-4-Me)] (III). 2,6-Di-*tert*-butyl-4-methylphenol (0.18 g, 0.85 mmol) was added to a solution of compound **I** (0.51 g, 0.85 mmol) in diethyl ether (20 mL). The mixture was heated for 20 min at 60°C. The resulting violet solution was concentrated to a 5 mL volume and kept for 24 h at 10°C. The precipitated red-violet crystals of compound **III** were separated from the solution by decantation, washed with cold diethyl ether, and dried in vacuum. The yield was 0.6 g (74%). $T_m > 201$ (dec.).

^{1}H NMR (200 MHz; C_6D_6; 298 K; δ, ppm (*J*, Hz)): 7.40 (s, 1H), 7.35–7.18 (m, 4H), 7.15–7.07 (m, 4H), 7.07–6.97 (m, 2H), 6.92–6.74 (m, 2H), 6.28 (d, 1H, *J* = 6.8), 6.24 (d, 1H, *J* = 6.8), 3.99–3.69 (m, 3H), 3.50 (sept, 1H, *J* = 6.8), 2.30 (s, 3H), 1.49 (s, 18H), 1.48–1.39 (m, 9H), 1.16–1.05 (m, 6H), 0.91 (d, 3H, *J* = 6.8), 0.59 (d, 3H, *J* = 6.8), 0.52 (d, 3H, *J* = 6.8).

IR (ν, cm^{-1}): 3244 s, 1900 s, 1593 m, 1534 s, 1351 w, 1321 w, 1296 w, 1256 s, 1213 w, 1190 w, 1157 w, 1126 m, 1109 w, 1070 m, 1056 w, 1039 w, 1028 m, 979 w, 948 w, 932 m, 910 m, 885 s, 859 w, 847 w, 821 m, 804 s, 780 w, 773 w, 758 s, 688 w, 665 w, 638 w, 623 w, 610 m, 601 w, 578 s, 549 m, 512 w.

For $C_{51}H_{65}N_2OAl$		
Anal. calcd., %	C, 81.78	H, 8.75
Found, %	C, 81.31	H, 8.68

IR spectra were measured on an FSM-1201 spectrometer in the 450–4000 cm^{-1} range (mineral oil mulls). The ^{1}H NMR spectra were measured on a Bruker DPX-200 spectrometer (200 MHz). Melting points were determined in sealed evacuated tubes. Elemental analysis was performed by combustion of the sample in oxygen according to Pregl's method.

X-ray diffraction analysis of II and III. The crystals of compounds **II** and **III** suitable for X-ray diffraction were prepared by crystallization from diethyl ether. The crystals chosen for analysis were coated by mineral oil (Aldrich), attached to a glass capillary, and placed into a cold nitrogen flow of Agilent Xcalibur Eos (**II**) and Super Nova Atlas (**III**) diffractometers. In these diffractometers, the experimental sets of reflection intensities (MoK_α (**II**) and CuK_α (**III**) radiation, ω-scan mode) were integrated using CrysAlisPro software [43]. The SCALE3 ABSPACK software [44] was used to apply absorption corrections. The structures were solved by the dual-space method using the SHELXT program package [45]. The non-hydrogen atoms were refined by the full-matrix least-squares method on F^2_{hkl} in the anisotropic approximation using SHELXTL software [46] (for **II**) and OLEX2 software [47] (for **III**). The hydrogen atoms were placed into geometrically calculated positions and refined in the riding model, exept for the hydrogen atoms of the carbodiimide moieties in **II** and of N(2) and Al(2) in **III**, which were found from difference Fourier maps and refined isotropically. In the asymmetric part of the crystal cell of **II**, two diethyl ether solvate molecules were found in general positions, apart from two [(Dpp-Bian)Al(DCC(H))] molecules. One Et_2O molecule is disordered over two positions. The key crystallographic characteristics and X-ray experiment details for **II** and **III** are summarized in Table 1 and some bond lengths and bond angles are in Table 2.

The structures of **II** and **III** are deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1903665 (**II**) and 1903666 (**III**); www.ccdc.cam.ac.uk/structures).

RESULTS AND DISCUSSION

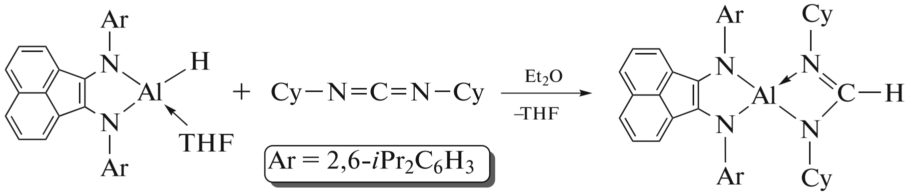
We have shown that aluminum hydrides can be stabilized by both the dianion and radical anion of Dpp-Bian [41, 42]. Preliminary investigations of the reactivity of the resulting complexes indicate that, despite their chemical inertness in comparison with other aluminum hydrides, including those containing redox inactive amide ligands,

Table 1. Crystallographic data, X-ray experiment and structure refinement details for **II** and **III**

Parameter	Value	
	II	**III**
Molecular formula	$C_{49}H_{63}N_4Al \cdot (C_2H_5)_2O$	$C_{51}H_{65}N_2OAl$
M	809.13	749.03
System	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$
Temperature, K	110(2)	150(2)
Wavelength, Å	0.71073	1.54184
a, Å	19.4515(2)	10.6712(2)
b, Å	22.1172(3)	24.1470(4)
c, Å	22.1641(3)	17.3371(3)
β, deg	95.298(1)	96.479(2)
V, $Å^3$	9494.5(2)	4438.85(14)
Z	8	4
ρ(calcd.), g/cm^3	1.132	1.121
μ, mm^{-1}	0.084	0.676
F(000)	3520	1624
Crystal size, mm	0.69 × 0.46 × 0.34	0.26 × 0.18 × 0.15
θ Range of measurements, deg	2.96–28.00	3.15–73.74
Ranges of indices	$-25 \le h \le 25$, $-29 \le k \le 29$, $-29 \le l \le 29$	$-10 \le h \le 13$, $-29 \le k \le 29$, $-21 \le l \le 18$
Number of observed reflections	167442	18187
Number of unique reflections (R_{int})	22886 (0.0539)	8683 (0.0287)
Number of reflections with $I > 2\sigma(I)$	16724	6925
Absorption corrections (max/min)	0.978/0.959	1.000/0.303
Constraints/parameters	68/1139	6/541
GOOF	1.060	1.025
R_1/wR_2 ($I > 2\sigma(I)$)	0.0505/0.1283	0.0441/0.1096
R_1/wR_2 (for all reflections)	0.0781/0.1412	0.0588/0.1211
$\Delta\rho_{max}/\Delta\rho_{min}$, e $Å^{-3}$	0.443/–0.485	0.260/–0.234

they can serve as reducing agents for unsaturated substrates and activate the O–H bonds in phenols.

The reaction of compound **I** with 1,3-dicyclohexylcarbodiimide involves addition of the latter to the Al–H bond, which proceeds in diethyl ether at 50°C for 15 min. The reaction product **II** was isolated as blue crystals by concentrating the solution (Scheme 1):



(I) **(II)**

Scheme 1.

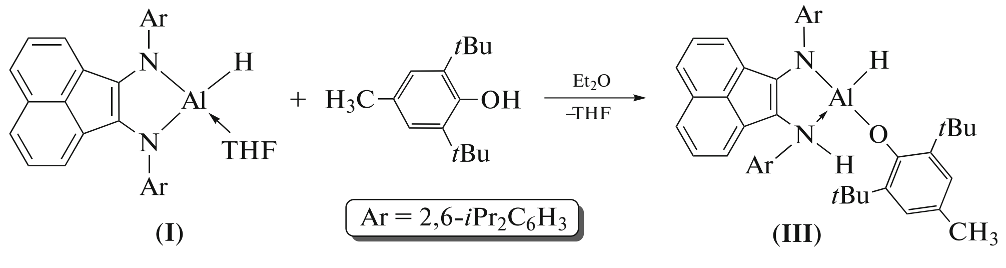
The ^1H NMR spectrum of compound **II** (Fig. 1a) in deuterobenzene exhibits signals for the methyl protons of 2,6-*io*-$Pr_2C_6H_3$: two doublets at 1.24 (12H) and 1.33 (12H) ppm and a septet for the methine protons (3.95 (4H) ppm). The doublets at 6.20 (2H) and 7.08 (2H) ppm and also signals at 6.87 (2H) and 7.30 (6H)

Table 2. Selected bond lengths (Å) and bond angles (deg) in compounds **II** and **III**

II		**III**	
Bond	*d*, Å	Bond	*d*, Å
Al–N(1)	1.8281(12)	Al–H(1)	1.47(2)
Al–N(2)	1.8345(12)	Al–O(1)	1.716(2)
Al–N(3)	1.9311(13)	Al–N(1)	1.883(2)
Al–N(4)	1.8886(12)	Al–N(2)	2.071(2)
N(1)–C(1)	1.400(2)	N(1)–C(1)	1.367(2)
N(2)–C(2)	1.408(2)	N(2)–C(2)	1.469(2)
C(1)–C(2)	1.379(2)	C(1)–C(2)	1.373(2)
N(3)–C(37)	1.321(2)	N(2)–H(2)	0.79(2)
N(4)–C(37)	1.325(2)		
Angle	ω, deg	Angle	ω, deg
N(1)AlN(2)	93.83(5)	H(1)AlO(1)	112.1(7)
N(3)AlN(4)	70.31(5)	N(1)AlN(2)	88.69(5)
N(1)AlN(3)	124.01(6)	N(1)AlH(1)	120.9(7)
N(1)AlN(4)	118.49(6)	N(2)AlH(1)	105.2(8)
N(2)AlN(3)	120.64(6)	N(1)AlO(1)	116.24(6)
N(2)AlN(4)	132.75(6)	N(2)AlO(1)	109.85(5)
N(3)C(37)N(4)	112.46(13)		

ppm refer to the aromatic protons of the naphthalene moiety and phenyl groups at nitrogen atoms. The proton at the central carbodiimide carbon atom gives a singlet at 7.61 (1H) ppm and signals for the N-cyclohexyl substituents: a multiplet at 1.42–1.60 (9H) ppm and broadened singlets at 0.97 (10H) and 2.78 (2H) ppm. The quartet at 3.26 ppm and the triplet at 1.11 ppm refer to protons of diethyl ether used to grow the crystals.

The reaction of hydride **I** with 2,6-di-*tert*-butyl-4-methylphenol (1 : 1) involves the Al–N bond, similarly to reactions of the allyl derivative [(Dpp-Bian)AlEt(Et_2O)] [48] and bis(imino)pyridine aluminum hydrides [38, 39]. This attests to higher basicity of the Al–N bond compared with the A–H bond. The reaction is accompanied by a change in the solution color from blue to violet and affords complex **III**, which was isolated from diethyl ether as red-violet crystals in 74% yield (Scheme 2):



Scheme 2.

In the IR spectrum of **III**, Al–H vibrations appear as an intense band at 1900 cm^{-1}, which is close to that of the initial hydride **I** (1864 cm^{-1}) [41]. The band at 3244 cm^{-1} corresponds to N–H vibrations. For comparison, the N–H stretching band of [{Dpp-Bian(H)}AlEt{O(2,6-*tert*-Bu_2-4-MeC_6H_2)}] occurs at 3238 cm^{-1} [48].

The ^1H NMR spectrum of compound **III** (Fig. 1b) exhibits signals for the unsymmetrical Dpp-Bian ligand: doublets at 0.52 (3H), 0.59 (3H), and 0.91 (3H) ppm; two multiplets at 1.05–1.16 (6H) and 1.39–1.48 (9H) ppm; a septet at 3.50 (1H); and two overlapping signals at 3.69–3.99 (3H) ppm for the isopropyl groups of 2,6-*iso*-$Pr_2C_6H_3$. Also, two doublets (6.24

(1H) and 6.28 (1H) ppm) and two multiplets (6.74–6.92 (2H) and 6.97–7.07 (2H) ppm) correspond to protons of the naphthalene moiety. The signals of the aromatic protons of 2,6-*iso*-$Pr_2C_6H_3$ and the 2,6-*tert*-Bu_2-4-MeC_6H_2O aroxyl ligand are in the ranges of 7.07–7.15 (4H) and 7.18–7.35 (4H) ppm. The singlets at 1.49 (18H) and 2.30 (3H) ppm correspond to *tert*-butyl and methyl groups. The singlet at 7.40 (1H) corresponds to the proton at the nitrogen atom.

According to X-ray diffraction data, **II** and **III** are monomeric four-coordinate aluminum complexes (Fig. 2). Analysis of the bond lengths and the metallacycles of **II** and **III** attest to the dianionic state of the Dpp-Bian ligands in them (C(1)–C(2), 1.379(2) Å (**II**) and 1.373(2) Å (**III**)). In **II**, the N(1)–C(1) and N(2)–C(2) bond lengths differ little, being 1.400(2) and 1.408(2) Å, respectively. Similar values were found in the complex of methylaluminum with the Dpp-Bian dianion, [(Dpp-Bian)AlMe(Et_2O)], in which the N(1)–C(1) and N(2)–C(2) distances were 1.407(4) and 1.409(4) Å, respectively [49]. In complex **III**, the N(1)–C(1) and N(2)–C(2) bonds are considerably different (1.367(2) and 1.469(2) Å), which is caused by the presence of a proton at N(2). The Al–N^{BIAN} length distributions in the two complexes are also different: these bonds are equalized in **II** (1.8281(12) and 1.8345(12) Å) and differentiated in **III** (1.883(2); and Al–N(2), 2.071(2) Å).

The geometries of the AlN(1,2)C(1,2) metallacycles in **II** and **III** are different. In complex **II**, this metallacycle is non-planar; the folding angle along the N(1)···N(2) line is ~14°. The aluminum atom deviates from the N(1,2)C(1,2) plane by ~0.3 Å. In complex **III**, the similar metallacycle is virtually planar, with the folding angle along the N(1)···N(2) line being 3.5° and the Al deviation from the N(1,2)C(1,2) plane being only 0.086 Å.

In **II**, the atoms of the ring formed by carbodiimide are located in one plane, which is virtually perpendicular to the Dpp-Bian plane (the dihedral angle is ~82°). The Al–N(3) (1.9311(13) Å) and Al–N(4) (1.8886(12) Å) bond lengths are comparable, so are the N(3)–C(37) (1.321(2) Å) and N(4)–C(37) (1.325(2) Å) distances.

In the aroxy aluminum hydride **III**, the Al–H bond (1.47(2) Å) is shorter than those in the starting complex **I** (1.53(2) Å) or the phenoxide [(Dpp-Bian)Al(H)(OC_6H_2-2,6-*tert*-Bu_2-4-Me)][Li($THF)_4$] (1.537(16) Å) [41], but differs little from the Al–H distance in the β-diketiminate derivative [Dpp-Nac-Nac)Al(H)(OC_6H_2-2,4,6-Me)] (1.50(2) Å) [21]. The Al–O distance in compound **III** (1.716(1) Å) is similar to that for [(Dpp-Bian)Al(H)(OC_6H_2-2,6-*tert*-Bu_2-4-Me)][Li($THF)_4$] (1.7495(10) Å) [41] and [(Dpp-Nac-Nac)Al(H)(OC_6H_2-2,4,6-Me)] (1.696(2) Å) [21], but markedly shorter than that in the aryloxy derivative [(2,6-*tert*-Bu_2-4-Me-C_6H_2O)AlH_2 · NMe_3] (1.762(4) Å) [50]. The N(2)–H(2) distance (0.79(2) Å) falls in the range typical of aluminum complexes with Dpp-Bian (0.78–0.91 Å) [48].

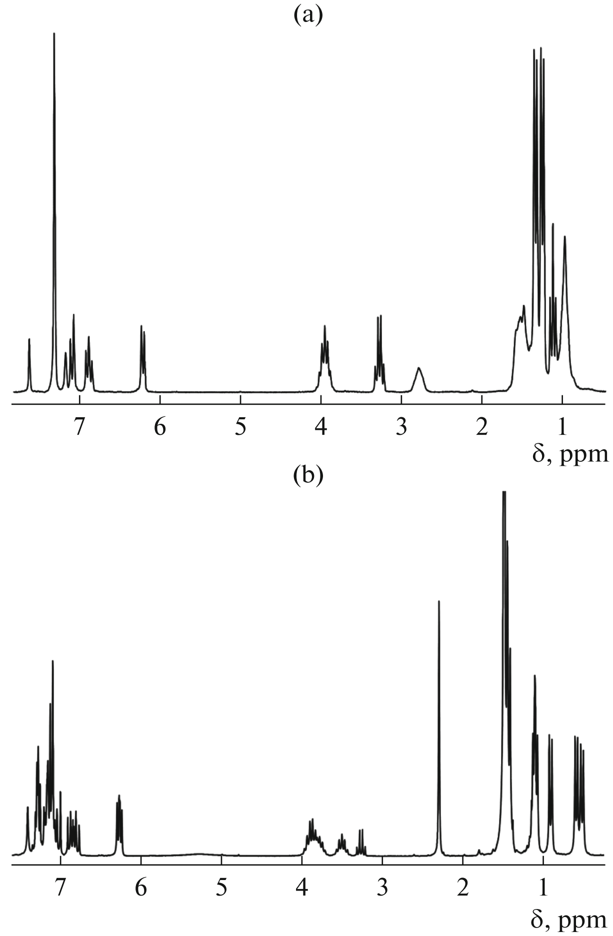


Fig. 1. ^1H NMR spectra of compounds (a) **II** and (b) **III** (200 MHz, C_6D_6, 298 K).

Thus, we demonstrated that the coordination environment of the aluminum atom composed of the redox active diimine ligand has a considerable effect on the nucleophilicity of the hydride ion in [(Dpp-Bian)AlH(THF)] (**I**). On the other hand, compound **I** is able to reduce multiple bonds, similarly to the hydride [(Dpp-Bian)Al$(H)_2$Li$(THF)_3$], described in our previous publication [42]. This was demonstrated by the reaction of **I** with 1,3-dicyclohexylcarbodiimide. On the other hand, the reaction of **I** with 2,6-di-*tert*-butyl-4-methylphenol shows that the basicity of the metal–nitrogen bonds in hydride **I** is higher than the basicity of the metal–hydrogen bonds, as opposed to [(Dpp-Bian)Al$(H)_2$Li$(THF)_3$], which reacts with 2,6-di-*tert*-butyl-4-methylphenol with protonolysis of the Al–H bond. A similar reactivity towards acidic substrates was also observed for [(Dpp-Bian)AlEt(Et_2O)] [48] and bis(imino)pyridine aluminum hydrides [38, 39].

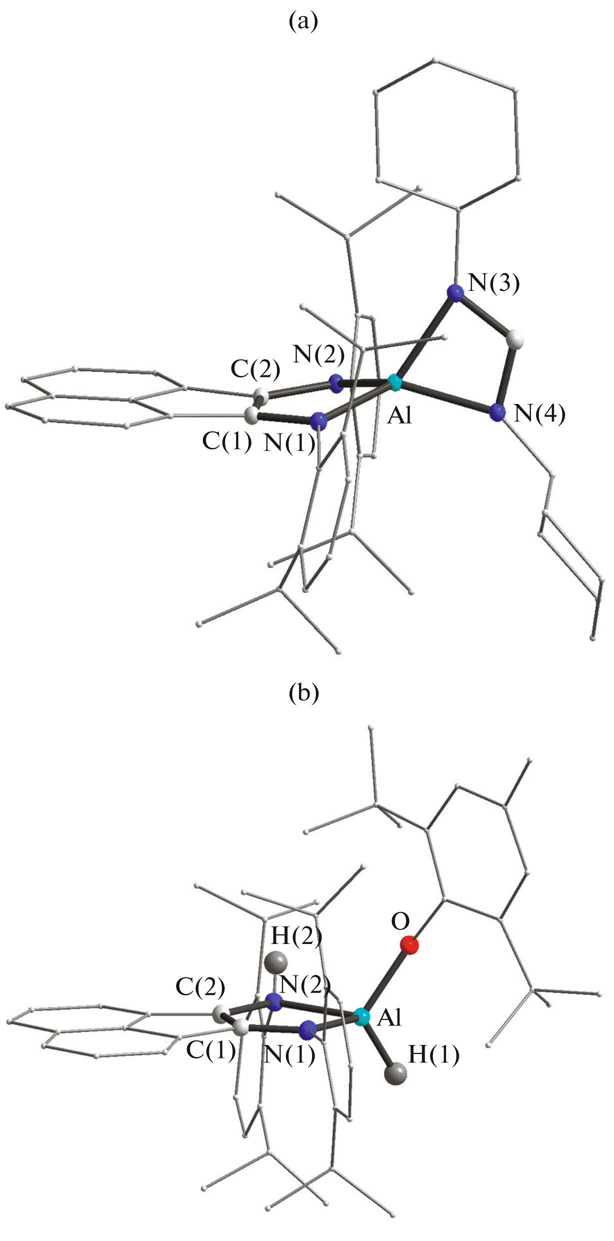


Fig. 2. Molecular structure of (a) **II** and (b) **III**. The thermal ellipsoids are drawn at the 30% probability level. The hydrogen atoms are omitted (except for H(1) and H(2) in **III**).

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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