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✻ Magnesium and calcium complexes with two diimine radical-anion ligands. Molecular structure of the Ca complex with 1,2-bis[(2,6-diisopropylphenyl)imino]acenaphthene

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Four- and five-coordinate magnesium and calcium complexes containing two diimine radical-anion ligands with compositions (dpp-BIAN)$_2$Mg (**1**), (dpp-BIAN)$_2$Ca (**2**), (dtb-BIAN)$_2$Mg (**3**), and (dtb-BIAN)$_2$Ca(THF) (**4**) (dpp-BIAN is 1,2-bis[(2,6-diisopropylphenyl)imino]acenaphthene and dtb-BIAN is 1,2-bis[(2,5-di-*tert*-butylphenyl)imino]acenaphthene) were synthesized. At 120 K, the ESR spectra of complexes **1**—**4** in a toluene matrix show signals characteristic of biradical derivatives. The molecular structure of compound **2** was established by X-ray diffraction analysis. At 293 K, the magnetic moments of compounds **1**, **2**, **3**, and **4** are 2.55, 2.57, 2.76, and 2.79 μ_B, respectively, which are indicative of the presence of two unpaired electrons localized on the ligands.

Key words: magnesium, calcium, acenaphthenediimine, structure, biradicals, ESR spectroscopy.

In recent years, diimines, including conformationally rigid 1,2-bis[(2,6-diisopropylphenyl)imino]acenaphthene (dpp-BIAN), have been widely used as chelating ligands in transition metal chemistry.[1] Complexes of Ni, Rh, Pt, Pd, Cu, and other metals with this ligand are among the most efficient olefin polymerization catalysts.[2—6] However, until recently main-group metal complexes with such ligands remained unknown. In 2003, we prepared first complexes of main-group metals (Na, Li, Mg, and Ca) with various reduced forms of the BIAN ligand.[7] We found that alkali metals successively reduce dpp-BIAN to mono-di, tri-, and tetraanions,[7,8] whereas exclusively the complexes with the dpp-BIAN dianion, (dpp-BIAN)$^{2-}$M^{2+}L$_n$ (M = Mg, Ca; L = THF, C$_5$H$_5$N), are formed in quantitative yields in the reactions with Mg or Ca in THF.[9] X-ray diffraction studies demonstrated that monomeric complexes, in which the metal atoms are chelated by the dpp-BIAN dianion, are formed in all cases. The number of solvent molecules coordinated to the central atom increases with increasing ionic radius of the metal atom. For example, the magnesium complex crystallizes with three coordinated solvent molecules from a THF solution, whereas the calcium complex crystallizes with four solvent molecules. Recently, we have synthesized two new acenaphthenediimine ligands, *viz.*, 1,2-bis[(2,5-di-*tert*-butylphenyl)imino]acenaphthene (dtb-BIAN) and 1,2-bis[(2-biphenyl)imino]acenaphthene (bph-BIAN), and prepared magnesium and calcium complexes with these ligands.[10]

Recent studies demonstrated that the (dpp-BIAN)Mg(THF)$_3$ complex, which is readily generated *in situ*, is highly reactive toward various organic compounds.[11—13] Some reactions of this complex are accompanied by reduction of organic compounds and oxidation of the coordinated dpp-BIAN dianion to the radical anion to give paramagnetic complexes. The latter were isolated in the crystalline state and characterized by ESR spectroscopy and X-ray diffraction analysis.[11—13]

As part of our continuing studies of Group II metal complexes with acenaphthene-1,2-diimine radical-anion ligands, we made an effort to prepare complexes with two paramagnetic radical-anion ligands. In the present study, we synthesized homoleptic magnesium and calcium complexes with two acenaphthene-1,2-diimine radical-anion ligands, which contain no coordinated solvent molecules, and characterized these complexes by ESR spectroscopy.

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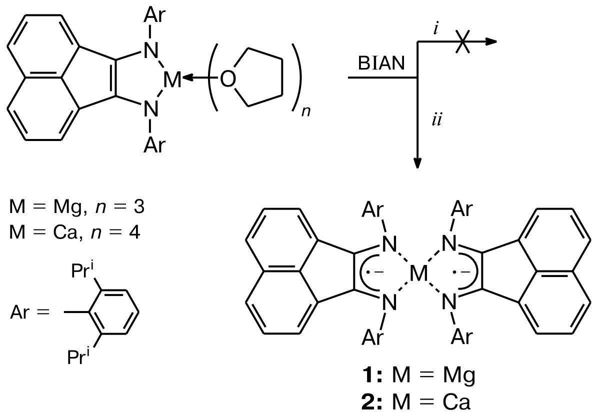
Results and Discussion

Synthesis of the (dpp-BIAN)$_2$Mg (1) and (dpp-BIAN)$_2$Ca (2) complexes. Direct reduction of dpp-BIAN with an excess of magnesium or calcium metal in a THF solution affords the complexes with the dianionic diimine ligand, (dpp-BIAN)$^{2-}$M^{2+}(THF)$_n$ (M = Mg, n = 3; M = Ca, n = 4).[9] However, the addition of an equimolar amount of dpp-BIAN to solutions of these complexes in THF (after separation from the metal) did not give rise to bis-ligand complexes. The formation of such complexes would proceed through the displacement of the coordinated THF molecules with neutral diimine accompanied by electron transfer from the (dpp-BIAN)$^{2-}$ dianion to the diimine ligand. The possibility of this process is determined by the balance between Coulomb, steric, and orbital interactions in the starting (dpp-BIAN)M(THF)$_n$ compounds (M = Mg, n = 3; M= Ca, n = 4) and, correspondingly, in the target complexes with two dpp-BIAN radical-anion ligands. In the case of the (dpp-BIAN)Mg(THF)$_3$ complex, we succeeded in performing this reaction simply by replacing THF as the solvent with toluene or 1,2-dimethoxyethane (DME). The process was accompanied by a change in the color of the reaction mixture from bright-green, which is characteristic of the (dpp-BIAN)$^{2-}$ dianion, to red-brown, which is characteristic of the radical anion, and gave the (dpp-BIAN)$_2$Mg complex (**1**) containing two radical-anion ligands (Scheme 1). In the case of the calcium derivative (dpp-BIAN)Ca(THF)$_4$, the reaction with neutral dpp-BIAN occurred only upon the replacement of THF with toluene to give (dpp-BIAN)$_2$Ca (**2**) (see Scheme 1).

Scheme 1



Reagents: *i*. THF; *ii*. toluene or DME for M = Mg, toluene for M = Ca.

Apparently, magnesium and calcium complexes with one dianionic ligand and coordinated THF molecules are energetically more favorable in a THF solution than the corresponding complexes with two radical anions. Complexes **1** and **2** were isolated in 69 and 76% yields, respectively, as pale-brown square-shaped crystals. Unlike the starting compounds, complexes **1** and **2** are paramagnetic both in solution and in the crystalline state. The effective magnetic moments of compounds **1** and **2** (at 293 K, 2.55 and 2.57 μ_B, respectively) are approximately equal to the calculated value for two noninteracting unpaired electrons (2.46 μ_B).

Study of solutions of compounds **1** and **2** in toluene by low-temperature (120 K) ESR spectroscopy confirmed that these compounds are biradical complexes with two unpaired electrons localized on the ligands. The anisotropic spectrum of compound **1** is shown in Fig. 1, *a*. The ESR spectra of compounds **1** and **2** have half-field signals (Δm_s = 2) characteristic of biradicals. The distances between the geometric centers of localization of the unpaired electrons in molecules **1** and **2**, which were calculated from the parameters of the ESR spectra, are 5.28 Å ($D_{\|}$ = 37.8 mT) and 5.74 Å ($D_{\|}$ = 29.4 mT), respectively. The latter distance is somewhat shorter than the distance between the midpoints of the C—C bonds in the diimine fragments of two dpp-BIAN ligands (6.08 Å) in the structure of **2** established by X-ray diffraction analysis.

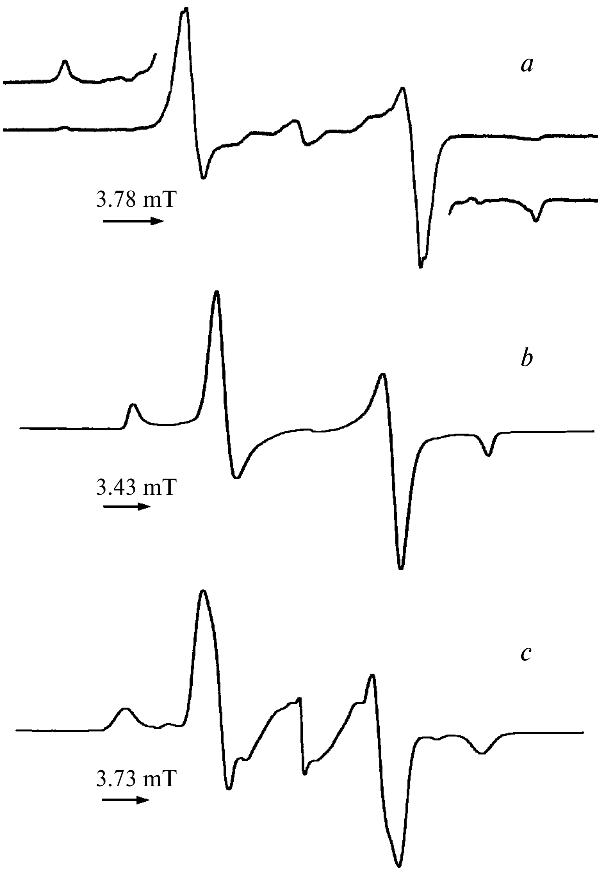
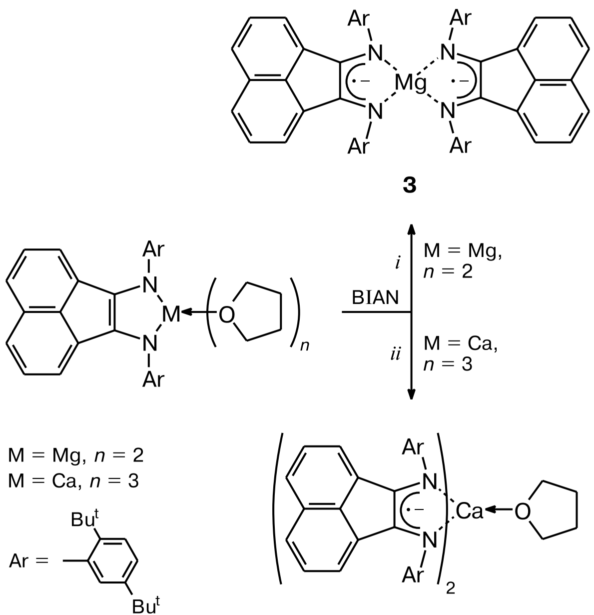


Fig. 1. ESR spectra of complexes **1** (*a*), **3** (*b*), and **4** (*c*) in toluene at 120 K. Half-field signals are omitted.

The IR spectrum of compound **1** is identical to that of **2**. In these spectra, the C=N stretching vibration bands characteristic of the free ligand (at 1671, 1652, and 1642 cm^{-1}) and the C—N stretching bands of the dianion (at 1315—1330 cm^{-1})[9] are absent; instead, the spectra show bands at 1500—1600 cm^{-1} corresponding to C—N stretching vibrations of the radical-anion ligands.[11—13]

Synthesis of the (dtb-BIAN)$_2$Mg (3) and (dtb-BIAN)$_2$Ca(THF) (4) complexes. Unlike the complexes with the (dpp-BIAN)$^{2-}$ dianion, the magnesium and calcium derivatives of the dtb-BIAN ligand react with neutral dtb-BIAN even in THF to form (dtb-BIAN)$_2$Mg (**3**) and (dtb-BIAN)$_2$Ca(THF) (**4**), respectively (Scheme 2).

Scheme 2



Reagents: *i.* 1) THF, 2) benzene; *ii.* 1) THF, 2) hexane.

Compounds **3** and **4** were isolated as red-brown crystals in 81 and 79% yields, respectively, by recrystallization from benzene and hexane and were characterized by ESR and IR spectroscopy. The magnetic moments (293 K) of complexes **3** and **4** (2.76 and 2.79 μ_B, respectively) are somewhat higher than the value expected for a system containing two unpaired electrons (2.46 μ_B). The ESR spectrum of a solution of magnesium complex **3** (see Fig. 1, *b*) in toluene at 120 K is similar to the spectra of complexes **1** and **2**. The distance between the centers of localization of the unpaired electrons calculated from this spectrum is 5.45 Å ($D_{\|}$ = 34.3 mT), which is 0.17 Å longer than the corresponding distance in magnesium complex **1**. This fact is consistent with the assumption that steric hindrance at the metal atom caused by the dtb-BIAN ligand is larger than that caused by the dpp-BIAN ligand. This is also confirmed by the fact that crystallization from a THF solution affords the (dtb-BIAN)Mg(THF)$_2$ complex with two coordinated solvent molecules,[10] whereas the (dpp-BIAN)Mg(THF)$_3$ complex crystallizes with three solvent molecules.[9] The ESR spectrum of complex **4** (see Fig. 1, *c*, 120 K, toluene) shows two signals belonging to biradical species ($D_{\|}$ = 37.3 mT, r_1 = 5.30 Å and $D_{\|}$ = 28.0 mT, r_2 = 5.83 Å). The presence of two biradical forms in the spectrum of (dtb-BIAN)$_2$Ca(THF) (**4**) is attributable to the fact that the solution contains both the five- and four-coordinate calcium compounds, *viz*., (dtb-BIAN)$_2$Ca(THF) and (dtb-BIAN)$_2$Ca. The spectra of complexes **3** and **4** have low-intensity half-field signals (Δm_s = 2) characteristic of biradicals. The structures of complexes **3** and **4** were established by X-ray diffraction analysis. Although these experiments allowed us to determine the overall geometry of the molecules, the *R* factors obtained for the refinement of the crystal structures of complexes **3** and **4** were high (0.1990 and 0.1833, respectively). Hence, it is unreasonable to discuss the geometric parameters of the molecules.

Molecular structure of compound 2. The molecular structure of complex **2** (Fig. 2) was established by X-ray diffraction analysis. Selected bond lengths and bond angles for compound **2** are given in Table 1.

The molecule of complex **2** is located on a crystallographic twofold rotation axis passing through the calcium atom, due to which both diimine ligands are geometrically equivalent. The dihedral angle between the planes of the diimine fragments is 28°. For four-coordinate calcium compounds, a tetrahedral geometry is more favorable than a square-planar geometry. In compound **2**, the dihedral angle between the planes of two ligands is 28°, which reduces steric hindrances caused by the isopropyl substituents of two ligands. This can clearly be seen from the projection shown in Fig. 2, *b*. In going from the com-

Table 1. Selected bond lengths (*d*) and bond angles (ω) in molecule **2**

Distance	*d*/Å	Angle	ω/deg
Ca—N(1)	2.371(3)	N(1)—Ca—N(2)	73.58(9)
Ca—N(2)	2.378(3)	C(1)—N(1)—C(13)	115.0(3)
Ca—C(1)	3.120(3)	C(1)—N(1)—Ca	111.6(2)
Ca—C(2)	3.131(3)	C(13)—N(1)—Ca	133.4(2)
C(1)—N(1)	1.334(4)	C(2)—N(2)—Ca	111.6(2)
C(2)—N(2)	1.342(4)	C(25)—N(2)—Ca	132.7(2)
C(1)—C(2)	1.442(5)	C(2)—N(2)—C(25)	115.8(3)
N(1)...N(1´)	4.600(4)	N(1)—C(1)—C(2)	122.1(3)
N(2)...N(2´)	4.632(4)	N(1)—C(1)—C(5)	130.4(3)
C(1)...C(1´)	6.236(5)		
C(2)...C(2´)	6.211(5)		

Note. Primed atoms are related to unprimed atoms by the symmetry operation $1 - x + 1, y, -z + 3/2$.

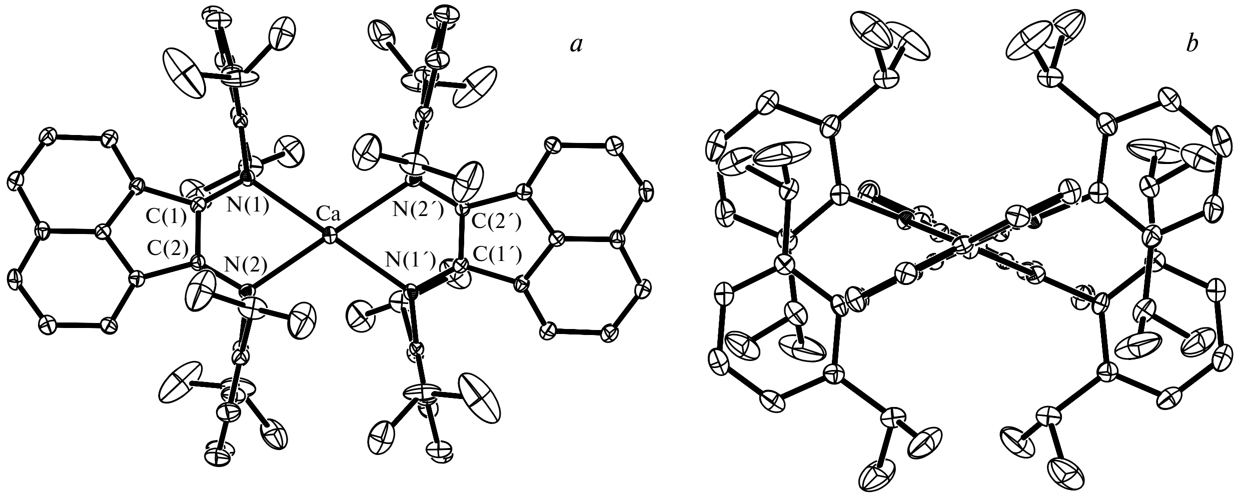


Fig. 2. Molecular structure of (dpp-BIAN)$_2$Ca (**2**). Thermal ellipsoids are drawn at the 30% probability level. The hydrogen atoms are omitted.

plexes with the BIAN dianion to the complexes with radical anions, one would expect elongation of the metal—ligand bonds due to weakening of the interaction between the cation and anion. Earlier,[11—13] we have observed this effect in mixed magnesium complexes with the dpp-BIAN radical-anion ligands. However, it appeared that the Ca—N bond lengths in compound **2** (2.371(3) and 2.378(3) Å) are even slightly shorter than those in the (dpp-BIAN)Ca(THF)$_4$ molecule (2.396(2) and 2.382(2) Å).[9] This apparent contradiction is attributable to shortening of the bonds involving the metal atom as its coordination number decreases. In complex **2** and (dpp-BIAN)Ca(THF)$_4$, the coordination number of the metal atom is four and six, respectively. Earlier,[9] we have already observed shortening of the metal—ligand bonds in going from (dpp-BIAN)Ca(THF)$_4$ to (dpp-BIAN)Ca(THF)$_3$. Along with the ESR data, the bond lengths in the diimine fragment of molecule **2** are also indicative of the radical-anion character of the ligands in this complex. In the diimine fragment of neutral dpp-BIAN, the N—C distances are short (both bond lengths are 1.282(4) Å) and the C(1)—C(2) bond is long (1.534(6) Å).[14] According to the symmetry of LUMO of neutral BIAN, two-electron reduction of the latter to the dianion should lead to elongation of the N—C bonds and shortening of the C—C bond. This situation is observed in the starting (dpp-BIAN)Ca(THF)$_4$ complex (N(1)—C(1), 1.391 Å; N(2)—C(2), 1.402 Å; C(1)—C(2), 1.409 Å; the atomic numbering scheme corresponds to that shown in Fig. 2).[9] One-electron oxidation of the dpp-BIAN dianion would result in the bond lengths intermediate between those in (dpp-BIAN)$^{2-}$ and (dpp-BIAN)^0. This situation is, in fact, observed in complex **2** (N(1)—C(1), 1.334(4) Å; N(2)—C(2), 1.342(4) Å; C(1)—C(2), 1.442(5) Å). The bond lengths in the BIAN radical anions are similar to those observed[11—13] in the diimine fragments of the complexes, which we have prepared earlier by the reactions of magnesium complex **5** with various organic reagents. For example, the bond lengths in the [(dpp-BIAN)Mg(THF)]$_2$[μ-O$_2$C$_2$Ph$_4$]$_2$ complex are as follows: N(1)—C(1), 1.324 Å; N(2)—C(2), 1.331 Å; C(1)—C(2), 1.457 Å. In the (dpp-BIAN)Mg(OC$_{14}$H$_9$)(THF) compound, the bond lengths are as follows: N(1)—C(1), 1.334 Å; N(2)—C(2), 1.339 Å; C(1)—C(2), 1.432 Å.[11]

Experimental

The compounds under study are sensitive to atmospheric moisture and oxygen. Hence, all operations associated with the synthesis, isolation, and identification were carried out *in vacuo* or under dry argon using the Schlenk technique. 1,2-Bis[(2,6-diisopropylphenyl)imino]acenaphthene (dpp-BIAN) and 1,2-bis[(2,5-di-*tert*-butylphenyl)imino]acenaphthene (dtb-BIAN) were prepared by condensation of acenaphthenequinone and the corresponding aniline derivatives (Aldrich) in acetonitrile according to known procedures.[10,15] The dianionic complexes (dpp-BIAN)Mg(THF)$_3$, (dpp-BIAN)Ca(THF)$_4$, (dtb-BIAN)Mg(THF)$_2$, and (dtb-BIAN)Ca(THF)$_3$ were synthesized according to procedures described earlier.[9,10] Solutions of these complexes in THF were prepared from dpp-BIAN (0.50 g, 1.0 mmol) and dtb-BIAN (0.56 g, 1.0 mmol) by the reactions with an excess of Mg or Ca metal. In subsequent reactions, these solutions were used *in situ*. Tetrahydrofuran, 1,2-dimethoxyethane, toluene, benzene, and hexane were dried and stored over sodium benzophenone. The yields of products **1—4** given below were calculated with the respect to the total amount of the starting dpp-BIAN or dtb-BIAN compounds (2.0 mmol) used in the reactions. The IR spectra were recorded as Nujol mulls on a Specord M-80 spectrometer. The ESR spectra were measured on a Bruker ER 200D-SRC spectrometer equipped with an ER 4105DR dual-mode resonator (operating at ~9.5 GHz) and an ER 4111 VT temperature-controlling unit. The g factors were determined with the use of diphenylpicrylhydrazyl (DPPH) as the standard. The magnetic moments of the complexes were calculated from the magnetic susceptibilities of complexes **1—4**, which were determined using a known procedure[16] and are given without considering the Weiss constant.

Bis[1,2-bis{(2,6-diisopropylphenyl)imino}acenaphthene]magnesium (1). An equimolar amount of dpp-BIAN was added to a solution of the (dpp-BIAN)Mg(THF)$_3$ complex, which was prepared from dpp-BIAN (0.50 g, 1.0 mmol) in THF. The solvent was removed *in vacuo* and then DME (30 mL) was added to the residue, which was accompanied by a change in the color of the reaction mixture from green to red-brown. Compound **1** was isolated from a concentrated solution in DME as pale-brown square-shaped crystals. The yield was 0.71 g, (69%), m.p. >264 °C (with decomp.), $\mu = 2.55\ \mu_B$. Found (%): C, 84.12; H, 7.61. $C_{72}H_{80}MgN_4$ (1025.74 g mol^{-1}). Calculated (%): C, 84.31; H, 7.86. IR (Nujol mulls), ν/cm^{-1}: 1660 w, 1590 w, 1510 m, 1305 m, 1250 m, 1185 m, 1080 w, 1035 w, 930 s, 880 w, 860 m, 815 w, 790 m, 760 v.s, 685 w, 605 w. ESR (toluene, 120 K): $D_{\|} = 37.8$ mT ($r = 5.28$ Å).

Bis[1,2-bis{(2,6-diisopropylphenyl)imino}acenaphthene]calcium (2). The dpp-BIAN compound (0.50 g, 1.0 mmol) was added to a solution of the (dpp-BIAN)Ca(THF)$_4$ complex, which was prepared from dpp-BIAN (0.50 g, 1.0 mmol) in THF. The solvent was removed *in vacuo* and then toluene (30 mL) was added to the residue. Compound **2** was isolated from a concentrated solution in toluene as pale-brown rhombic platelet crystals in 76% yield (0.79 g), m.p. >264 °C (with decomp.), $\mu = 2.57\ \mu_B$. Found (%): C, 82.92; H, 7.55. $C_{72}H_{80}CaN_4$ (1041.51 g mol^{-1}). Calculated (%): C, 83.03; H, 7.74. IR (Nujol mulls), ν/cм$^{-1}$: 3060 w, 1650 w, 1590 w, 1510 m, 1310 m, 1250 w, 1190 m, 1135 w, 1100 w, 1040 w, 939 m, 890 w, 855 m, 820 m, 795 m, 765 v.s, 690 w, 605 w. ESR (toluene, 120 K): $D_{\|} =$ 29.4 mT ($r = 5.74$ Å).

Bis[1,2-bis{(2,5-di-*tert*-butylphenyl)imino}acenaphthene]magnesium (3). The dtb-BIAN compound (0.56 g, 1.0 mmol) was added to a solution of the (dtb-BIAN)Mg(THF)$_2$ complex, which was prepared from dtb-BIAN (0.56 g, 1.0 mmol) in THF. The reaction was accompanied by a rapid change in the color of the mixture from emerald-green to gold-cherry. The solvent was removed *in vacuo* and then benzene (50 mL) was added to the residue. Compound **3** was isolated from a concentrated solution in benzene as red-brown crystals in 81% yield (1.05 g), m.p. 189 °C (with decomp.), $\mu = 2.76\ \mu_B$. Found (%): C, 85.25; H, 8.30. $C_{80}H_{96}MgN_4 \cdot 2C_6H_6$ (1294.20 g mol^{-1}). Calculated (%): C, 85.38; H, 8.41. IR (Nujol mulls), ν/cm^{-1}: 3040 w, 1605 w, 1522 s, 1295 w, 1270 w, 1220 w, 1190 m, 1130 w, 1065 m, 1035 w, 935 m, 895 w, 820 s, 770 m, 725 w, 679 v.s, 515 w. ESR (toluene, 120 K): $D_{\|} = 34.3$ mT ($r = 5.45$ Å).

Bis[1,2-bis{(2,5-di-*tert*-butylphenyl)imino}acenaphthene]calcium (4). The addition of dtb-BIAN (0.56 g, 1.0 mmol) to a solution of the (dtb-BIAN)Ca(THF)$_3$ complex, which was prepared from dtb-BIAN (0.56 g, 1.0 mmol) in THF, led to a rapid change in the color of the reaction mixture from emerald-green to gold-cherry. The solvent was removed *in vacuo* and then hexane (50 mL) was added to the residue. Compound **4** was crystallized as red-brown crystals from a concentrated hexane solution. The yield was 0.98 g (79%), m.p. 235 °C (with decomp.), $\mu = 2.79\ \mu_B$. Found (%): C, 81.97; H, 8.34. $C_{84}H_{104}CaN_4O$ (1225.83 g mol^{-1}). Calculated (%): C, 82.30; H, 8.55. IR (Nujol mulls), ν/cm^{-1}: 3070 w, 3030 w, 1665 m, 1640 m, 1600 s, 1550 w, 1510 s, 1190 m, 1130 w, 1060 m, 1025 m, 955 m, 930 m, 879 m, 820 s, 790 m, 765 s, 720 w, 670 m, 610 m, 560 w. ESR (toluene, 120 K): $D_{\|} = 37.3$ mT ($r_1 = 5.30$ Å), $D_{\|} = 28.0$ mT ($r_2 = 5.83$ Å).

X-ray diffraction study of compound 2. X-ray diffraction data were collected on a Siemens SMART CCD diffractometer (ω scanning technique, Mo-Kα radiation, $\lambda = 0.71073$ Å, graphite monochromator) at 173 K. The absorption correction was applied using the SADABS program.[17] The structure was solved by direct methods using the SHELXS90 program package[18] and refined by the full-matrix least-squares method against F^2 using the SHELXL97 program package.[19] All nonhydrogen atoms were refined anisotropically. The H atoms were placed in idealized positions ($U_{\rm iso} = 0.08$ Å^3). The geometry of the structure was analyzed using the PLATON program.[20] The crystallographic data and details of X-ray diffraction study and refinement for **2** are as follows: the crystal dimensions are 0.52×0.14×0.48 mm^3, the orthorhombic crystal system (*P*bcn), $a = 18.2769(1)$, $b = 15.2721(2)$, $c = 21.9171(3)$ Å, $\alpha = \beta = \gamma = 90°$, $V = 6117.65(12)$ Å^3, $Z = 4$, $d_{\rm calc} = 1.131 \cdot 10^3$ kg m^{-3}, $\mu = 0.147$ mm^{-1}, $F(000) = 2240$, $1.74 \le \theta \le 25.00°$, $-21 \le h \le 20$, $-18 \le k \le 16$, $-24 \le l \le 26$, a total of 28747 independent reflections were collected, 5383 independent reflections ($R_{\rm int} = 0.1609$), 5099 reflections with $I > 2\sigma(I)$, 356 parameters were refined, GOOF(F^2) = 1.023, the final *R* factors ($R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$), $R_1 = 0.0784$, $wR_2 = 0.1575$, the maximum and minimum residual densities were 0.278 and −0.338 e Å^{-3}, respectively.

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