

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

Synthesis and Crystal Structures of the Reaction Products of the Magnesium Acenaphthenediimine Complex with Tosyl Azide

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Abstract—The reaction of [(Dpp-Bian)Mg(THF)₃] (**I**) (Dpp-Bian is 1,2-bis[(2,6-diisopropylphenyl)imino]acenaphthene) with tosyl azide (TosN₃) affords the binuclear amide complex [(Dpp-Bian)Mg(THF)(NTos)(THF)Mg(Dpp-Bian)] (**II**) in which the magnesium atoms are linked with the nitrogen atom by the bridging *p*-tolylsulfamide group. In the presence of pyridine, the reaction leads to the compound [(Dpp-Bian)Mg(Py)(NTos)(Py)Mg(Dpp-Bian)] (**III**) in which NTos has different coordination modes with the metal atoms. The diimine ligand is in the radical anion state in both compounds. New paramagnetic compounds **II** and **III** are characterized by IR spectroscopy, EPR spectroscopy, elemental analysis, and X-ray diffraction analysis (CFA files CCDC no. 1878433 (**II** · 1.5C₅H₁₂) and 1878432 (**III** · 2C₇H₈ · 0.5OC₄H₈)).

Keywords: magnesium, redox-active ligands, acenaphthene-1,2-diimines, azides, synthesis, molecular structure

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INTRODUCTION

Diimines of the Bian (bis(imino)acenaphthenes) family can easily be reduced to dianions by activated magnesium in polar solvents. Numerous magnesium derivatives, [(Dpp-Bian)Mg(THF)₃] (**I**) (Dpp is 2,6-diisopropylphenyl), [(Dpp-Bian)Mg(THF)₂], [(Dpp-Bian)Mg(Py)₃] [1], [(Dtb-Bian)Mg(THF)₂] (Dtb is 2,5-di-*tert*-butylphenyl) [2], and [(Bph-Bian)Mg(THF)₂] (Bph is 2-biphenyl) [3], in which the Bian ligand is in the dianionic state, were synthesized using this method, under different reaction conditions, and structurally characterized. The reactivity of [(Dpp-Bian)Mg(THF)₃] was studied in most detail. This complex was shown to exhibit the reduction properties due to the electron transfer from the diimine ligand to metal atom upon substrate binding and to be involved in the addition reactions of organic compounds containing a mobile hydrogen atom, such as alkynes, enolized ketones, nitriles, and others. For example, compound **I** reduced diphenyl ketone to form the pinacolate complex [(Dpp-Bian)Mg(THF)₂]{O₂C₂Ph₄}, and its reaction with 9(10*H*)-anthracenone gave the 9-anthracenolate radical anion complex [4]. The [(Dpp-Bian(*H*))-Mg(C≡CPh)(THF)₂] complex was obtained by the reaction of compound **I** with a

PhC≡CH excess. The reaction proceeded via the oxidative addition of the C–H bond [5]. The subsequent treatment of this complex with benzophenone afforded paramagnetic carbinolate [(Dpp-Bian)Mg{OC(Ph)₂C≡CPh}(THF)₂] [5]. Compound [(Dpp-Bian)Mg(THF)₃] is readily oxidized by diverse halogen-containing reagents (for example, Ph₃SnCl, CuCl, HgCl₂, or SiCl₄) to form compounds resembling the Grignard reagents of the general formula [(Dpp-Bian)MgX(S)]_{*n*} (S = THF: X = Cl and Br, *n* = 2; S = DME: X = I, *n* = 1) with the simultaneous formation of Ph₆Sn₂, Cu⁰, Hg⁰, or Si₂Cl₆, respectively [6]. The addition of dibenzyl ketone, 1,1-diphenyl ketone, or camphor to compound **I** affords the corresponding enolate derivatives [7]. The treatment of compound **I** with benzoyl peroxide or perbenzoic acid gives complexes [(Dpp-Bian)MgO₂CPh(THF)]₂ and [(Dpp-Bian)(H)-MgO₂CPh(THF)]₂, respectively [8]. Magnesium complex **I** reacts with ethyl halides to form the products of ligand-centered oxidative addition [(Dpp-Bian)(Et)MgX(THF)_{*n*}] (X = Cl, *n* = 0; X = Br, *n* = 2; X = I, *n* = 1) [9].

Important progress in the chemistry of coordination magnesium derivatives was achieved when β-diketimate ligands were used [10, 11]. The

compounds with the magnesium–magnesium bond $\{[(\text{ArNacnac})\text{Mg}]_2\}$ (ArNacnac is $[(\text{ArNCMe})_2\text{CH}]$), where Ar is Dpp (2,6-diisopropylphenyl) [12], Ar is Dep (2,6-diethylphenyl) [13], Ar is Mes (2,4,6-trimethylphenyl) [14], or Ar is Xyl (2,6-dimethylphenyl) have been obtained for the first time [13]. These compounds manifested themselves as universal reducing agents in organic and inorganic chemistry [15]. For example, the two-electron reduction of dicyclohexylcarbodiimide [12], azobenzene [16], cyclooctatetraene [16], and anthracene [17] results in the insertion of a substrate into the Mg–Mg bond. The magnesium β -diketoiminate complexes give the reductive cross-coupling products with 1-adamantanyl azide and *tert*-butyl isocyanate, the hexazene and oxamide complexes, respectively [16], with the formation of new N–N and C–C bonds. The magnesium β -diketiminates derivatives with the Mg–Mg bond act as reducing agents for small molecules, for example, CO_2 , inducing the disproportionation of the latter to CO and carbonate ligand [18]. In addition, the controlled activation of CO_2 , Ph_2CCO , N_2O [19], and CO [20] can be carried out using these derivatives.

In the recent time, we concentrated our attention on studying the reactivity of the gallium derivative $[(\text{Dpp-Bian})\text{Ga-Ga}(\text{Dpp-Bian})]$ containing the metal–metal bond [21]. The processes of reduction by alkaline [22] and alkaline-earth metals [23] and lanthanides [24], oxidative addition [25], solvate-induced intramolecular electron transfer [26], and reversible addition of alkynes [27, 28] were discovered. The results of studying the reactivity of digallane toward the compounds containing cumulated and conjugated carbon–carbon and carbon–element bonds [29–31] turned out to be especially interesting. Digallane $[(\text{Dpp-Bian})\text{Ga-Ga}(\text{Dpp-Bian})]$ is capable of binding sulfur(IV) oxide to form the dithionite complexes [30] and reversibly adding isothiocyanates [29].

Azides containing cumulated multiple bonds can easily be involved in cycloaddition reactions and further can be modified with the formation of valuable organic products. However, the reactivity of the Dpp-Bian derivatives of metals of the main subgroups toward azides remains unstudied to the present time.

In this work we report the study of the reaction of the magnesium complex $[(\text{Dpp-Bian})\text{Mg}(\text{THF})_3]$ with tosyl azide containing both the azide and sulfone groups and the corresponding reaction products, complexes $[(\text{Dpp-Bian})\text{Mg}(\text{THF})(\text{NTos})(\text{THF})\text{Mg}(\text{Dpp-Bian})]$ (**II** · $1.5\text{C}_5\text{H}_{12}$) and $[(\text{Dpp-Bian})\text{-Mg}(\text{Py})(\text{NTos})(\text{Py})\text{Mg}(\text{Dpp-Bian})]$ (**III** · $2\text{C}_7\text{H}_8$ · $0.5\text{OC}_4\text{H}_8$), and their molecular structures and spectral characteristics (hereinafter the complexes are designated as **II** and **III** for simplicity).

EXPERIMENTAL

The initial compound **I** and the newly obtained complexes **II** and **III** are sensitive to air oxygen and moisture and, hence, all manipulations on their synthesis, isolation, and identification were conducted in vacuo using the Schlenk technique. Toluene, THF, and pentane were dried and stored over sodium benzophenone or a sodium–potassium alloy. Pyridine was dried and stored over sodium.

IR spectra were recorded on a Bruker TENSOR 37 spectrometer in a form of Nujol mull. EPR spectra were obtained on a Bruker ER 200 D-SRC spectrometer equipped with an ER 4105 DR double resonator (working frequency 9.5 GHz) and an ER 4111 VT temperature controller. Diphenylpicrylhydrazyl (DPPH, $g = 2.0037$) was used as a standard for g factor determination. Compounds Dpp-Bian [32], $[(\text{Dpp-Bian})\text{Mg}(\text{THF})_3]$ [1], and TosN_3 [33] were synthesized using published procedures. C, H, N, and S elemental analyses were carried out on an Elementar Variomircocube instrument. The yields of the products were calculated on the amount of the initial Dpp-Bian used for the synthesis (0.50 g, 1.0 mmol).

Synthesis of complex II. Tosyl azide TosN_3 (0.99 g, 0.5 mmol) was added to a solution of complex **I** in THF (25 mL). The color of the solution immediately changed from green to red. The mixture was stirred for 24 h, then all volatiles were removed in vacuo, and the residue was dissolved in pentane (15 mL). The solution was slowly concentrated to form red crystals. The yield was 0.44 g (60%).

IR (ν , cm^{-1}): 407 m, 430 m, 516 vw, 554 w, 581 m, 606 m, 670 s, 682 m, 761 vs, 768 s, 802 s, 818 m, 855 vs, 874 m, 920 m, 935 vs, 982 w, 1023 m, 1081 m, 1110 w, 1130 w, 1184 m, 1222 w, 1254 m, 1316 m, 1358 w, 1421 m, 1429 w, 1517 m, 1528 m.

For $\text{C}_{94.5}\text{H}_{121}\text{N}_5\text{O}_4\text{SMg}_2$

Anal. calcd., %	C, 77.12	H, 8.29	N, 4.76	S, 2.18
Found, %	C, 76.78	H, 8.11	N, 4.74	S, 1.77

Synthesis of complex III. Tosyl azide TosN_3 (0.99 g, 0.5 mmol) was added to a solution of complex **I** in THF (25 mL). The color of the solution immediately changed from green to red. Then pyridine (0.08 g, 1.0 mmol) was added. The solution was stirred for 24 h. All volatiles were removed in vacuo, and the residue was dissolved in toluene. The solution was slowly concentrated to form red crystals. The yield was 0.32 g (40%).

IR (ν , cm^{-1}): 465 w, 519 w, 554 m, 575 m, 637 w, 669 m, 683 m, 708 m, 752 s, 762 s, 787 m, 804 m, 818 m, 835 m, 858 w, 926 m, 941 m, 949 m, 990 m, 1042 m, 1067 w, 1105 m, 1184 m, 1225 m, 1254 s,

1275 w, 1318 m, 1329 m, 1362 m, 1495 m, 1526 s, 1593 m, 1603 m, 1642 m, 1653 m, 1671 m.

For $C_{105}H_{117}N_7O_{2.5}SMg_2$

Anal. calcd., % C, 78.93 H, 7.38 N, 6.14 S, 2.01

Found, % C, 78.46 H, 7.36 N, 6.33 S, 1.96

X-ray diffraction analyses of compounds **II** and **III**.

The crystals of compounds **II** and **III** suitable for X-ray diffraction analysis were obtained by crystallization from pentane and toluene, respectively. The crystals chosen for analysis were covered with a mineral oil (Aldrich), fixed on a glass capillary, and placed in a cold nitrogen flow of Stoe IPDS 2 or Stadi Vari diffractometers. The structures were solved using the SHELXS/T program [34]. The remained non-hydrogen atoms were localized by a direct method using consecutive calculations of the difference Fourier maps. The positions of atoms were refined by full-matrix least squares for F_{hkl}^2 in the anisotropic approximation for all nonhydrogen atoms using SHELXL [34]. The contributions of hydrogen atoms were taken into account in the calculations but were not refined. In all cases, the positions of the highest peaks and the values of the residual electron density in the final difference Fourier maps were chemically insignificant.

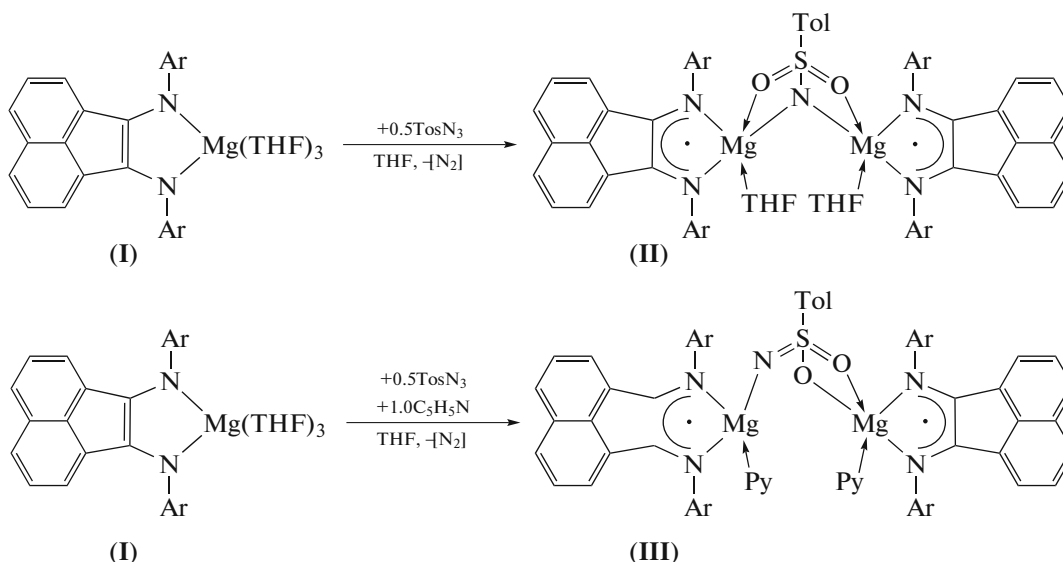
The crystal cell of compound **II** contains two solvate molecules of pentane in the general position disordered to a significant extent and, hence, refined in the isotropic approximation. The tolylsulfamide ligand is disordered over two positions with a ratio of 0.894 : 0.106. The crystals of compound **III** contain four solvate molecules of toluene in the general posi-

tion, one of which is disordered with the THF molecule in a ratio of 1 : 1. The main crystallographic characteristics and the X-ray diffraction experimental parameters for compounds **II** and **III** are presented in Table 1. Selected bond lengths and bond angles are given in Table 2.

The coordinates of atoms and other parameters of the structures were deposited with the Cambridge Crystallographic Data Centre (CFA files CCDC no. 1878433 (**II** · 1.5C₅H₁₂) and 1878432 (**III** · 2C₇H₈ · 0.5OC₄H₈); <http://www.ccdc.cam.ac.uk/structures>).

RESULTS AND DISCUSSION

The addition of tosyl azide (0.5 equiv mol) to a solution of compound **I** obtained by in situ reflux of diimine Dpp-Bian in THF results in an immediate change in the color of the solution from green to red indicating a change in the dianionic state of the Dpp-Bian ligand to the radical anion state. Red crystals of binuclear magnesium complex **II** were isolated from a pentane solution in a yield of 60% (Scheme 1). If pyridine (1 equiv mol) is added to the reaction mixture, the molecules of coordination THF in complex **II** are completely substituted by pyridine molecules accompanied by a change in the coordination mode of the *p*-tolylsulfamide ligand (Scheme 1). The formed compound **III** was isolated by recrystallization from toluene as red crystals in a yield of 40%. The IR spectra of compounds **II** and **III** exhibit intense bands at 1316 and 1184 cm⁻¹ (for **II**) and at 1318 and 1184 cm⁻¹ (for **III**) corresponding to symmetric and asymmetric stretching vibrations of the O₂SN groups.



Scheme 1.

Table 1. Crystallographic data and the experimental and structure refinement parameters for compounds **II** · 1.5C₅H₁₂ and **III** · 2C₇H₈ · 0.5OC₄H₈

Parameter	Value	
	II · 1.5C ₅ H ₁₂	III · 2C ₇ H ₈ · 0.5OC ₄ H ₈
F _w	1471.64	1597.73
Crystal system	Triclinic	Triclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> $\bar{1}$
Temperature, K	100(2)	100(2)
<i>a</i> , Å	13.8840(5)	13.1542(7)
<i>b</i> , Å	14.4214(5)	15.3969(7)
<i>c</i> , Å	22.6686(9)	24.9121(12)
α , deg	98.840(3)	83.328(4)
β , deg	99.585(3)	87.487(4)
γ , deg	102.968(3)	65.022(4)
<i>V</i> , Å ³	4274.3(3)	4542.6(4)
<i>Z</i>	2	2
ρ_{calcd} , g/cm ³	1.143	1.168
μ , mm ⁻¹	0.11	0.10
<i>F</i> (000)	1590	1712
Crystal size, mm	0.26 × 0.18 × 0.112	0.174 × 0.128 × 0.046
θ range, deg	1.478/26.123	1.603/26.181
Range indices	−17 ≤ <i>h</i> ≤ 11, −17 ≤ <i>k</i> ≤ 17, −27 ≤ <i>l</i> ≤ 27	−16 ≤ <i>h</i> ≤ 16, −19 ≤ <i>k</i> ≤ 18, −30 ≤ <i>l</i> ≤ 29
Observed reflections	36444	38474
Independent reflections (<i>R</i> _{int})	16594 (0.0716)	17691 (0.0556)
Reflections θ range <i>I</i> > 2 σ (<i>I</i>)	8071	10627
Absorption correction	STOE X-RED, LANA (max/min 0.9919/0.9774)	STOE X-RED, LANA (max/min 0.9954/0.9841)
Data/restraints/parameters	16594/33/976	17691/193/1233
GOOF	0.869	1.009
<i>R</i> ₁ / <i>wR</i> ₂ (<i>I</i> > 2 σ (<i>I</i>))	0.0679/0.1716	0.0652/0.1408
<i>R</i> ₁ / <i>wR</i> ₂ (all data)	0.1299/0.1923	0.1250/0.1686
$\Delta\rho_{\text{max}}/\Delta\rho_{\text{min}}$, e Å ⁻³	0.704/−0.613	0.494/−0.397

It should be mentioned that the reaction of complex **I** with tosyl azide in a molar ratio of 1 : 1 and the reaction in pyridine as a solvent result in the decomposition of the metal complexes and formation of free Dpp-Bian.

The EPR study of solutions of compounds **II** and **III** in toluene at low temperatures (150 K) confirm that they are biradical complexes with two lone electrons localized on the ligands. The anisotropic spectra of compounds **II** and **III** are presented in Fig. 1. The EPR spectra of compounds **II** and **III** exhibit half-field signals ($\Delta m_s = 2$) characteristic of biradicals.

According to the X-ray diffraction data, compound **II** (Fig. 2a) is the binuclear pentacoordinated magnesium complex with the bridging *p*-tolylsulfamide ligand characterized by the double S(1)–O(1) (1.476(3) Å) and S(1)–O(2) (1.484(3) Å) bonds and the ordinary S(1)–N(5) bond (1.502(3) Å) as in the [(^{RR}TBSC)₂Li₄(THF)₄] complex (^{RR}TBSC is (*R,R*)-*trans*-1,2-bis(2,4,6-triisopropylbenzenesulfonamido)cyclohexane) (S–O 1.463 and 1.469 Å, S–N 1.551 Å) [35]. The additional Mg···O contact leads to the situation where the S–O bonds are slightly elongated and the S–N bond is shortened, for example, compared to similar bonds in [In(N₂^{Tos}N^L)-

Table 2. Selected bond lengths (Å) and bond angles (deg) in compounds **II** · 1.5C₅H₁₂ and **III** · 2C₇H₈ · 0.5OC₄H₈

II · 1.5C ₅ H ₁₂		III · 2C ₇ H ₈ · 0.5OC ₄ H ₈	
Bond	<i>d</i> , Å	Bond	<i>d</i> , Å
S(1)–O(1)	1.475(3)	S(1)–O(2)	1.505(2)
S(1)–O(2)	1.484(3)	S(1)–O(1)	1.527(2)
S(1)–N(5)	1.503(3)	S(1)–N(5)	1.460(2)
Mg(1)–N(2)	2.106(3)	Mg(2)–O(2)	2.062(2)
Mg(1)–N(1)	2.138(3)	Mg(2)–O(1)	2.034(2)
Mg(1)–N(5)	2.088(3)	Mg(2)–N(4)	2.089(2)
Mg(2)–O(2)	2.175(3)	Mg(2)–N(3)	2.129(2)
Mg(2)–N(4)	2.131(3)	Mg(2)–N(7)	2.143(3)
Mg(2)–N(5)	2.116(3)	Mg(1)–N(2)	2.090(2)
N(4)–C(4)	1.339(4)	Mg(1)–N(1)	2.074(2)
N(2)–C(2)	1.331(4)	Mg(1)–N(6)	2.147(3)
N(1)–C(1)	1.336(4)	Mg(1)–N(5)	1.927(3)
N(3)–C(3)	1.336(5)	N(4)–C(4)	1.339(3)
C(2)–C(1)	1.446(4)	N(1)–C(1)	1.338(3)
		N(3)–C(3)	1.327(3)
Angle	ω, deg	Angle	ω, deg
O(1)S(1)O(2)	111.6(3)	O(2)S(1)O(1)	100.73(10)
O(1)S(1)N(5)	111.1(4)	N(5)S(1)O(2)	117.25(13)
O(2)S(1)N(5)	110.65(16)	N(5)S(1)O(1)	118.17(13)
N(2)Mg(1)O(1)	87.71(12)	O(2)Mg(2)N(4)	106.20(9)
N(2)Mg(1)N(1)	81.38(11)	O(2)Mg(2)N(3)	161.41(10)
N(1)Mg(1)O(1)	168.62(14)	O(1)Mg(2)O(2)	69.49(8)
N(5)Mg(1)O(1)	69.97(11)	N(4)Mg(2)N(3)	81.89(9)
		N(1)Mg(1)N(2)	84.05(9)
		N(5)Mg(1)N(2)	125.96(11)
		N(5)Mg(1)N(1)	119.30(11)
		S(1)O(2)Mg(2)	93.99(10)
		S(1)O(1)Mg(2)	94.45(10)

(CH₂SiMe₃)] (H₂N₂^{Tos}N^L is (2-NC₅H₄)CH₂N(CH₂-CH₂NHTos)₂) in which the oxygen atoms are not involved in coordination (S–O 1.443 and 1.452 Å, S–N 1.576 Å) [36]. The angle between the ligand planes in compound **II** is 80.09°.

The substitution of THF molecules by pyridine molecules results in the change of the coordination mode of the TosN ligand in complex **III**: the N(5) atom forms only one bond with the metal atom instead of two bonds as in complex **II** (Fig. 2b). This is accompanied by the stronger elongation of the sesquialteral S–O bonds in compound **III** (S–O 1.505(2) and 1.527(2) Å in **III** versus S–O 1.476(3) and 1.484(3) Å in **II**), and the S–N bond shortens by 0.04 Å and takes the character of a double bond (S–N 1.460(2) Å in **III** versus S–N 1.502(3) Å in **II**). A similar structure of the

TosN ligand is observed in [ArN(SO₂-*p*-Tol)Li(THF)₂]₂ (S–O 1.463(2) and 1.464(2), S–N 1.520(3) Å) [37]. The Mg–O distances also become noticeably shorter, which indicates that they are partially covalent (average Mg(2)–O(2) 2.062(2), Mg(2)–O(1) 2.034(2) Å in **III**; Mg(1)–O(1) 2.193(7), Mg(2)–O(2) 2.176(3) Å in **II**).

Thus, when reacting with TosN₃, the magnesium complex [(Dpp-Bian)Mg(THF)₃] shows the pronounced reduction properties in spite of the high affinity of azides and the Dpp-Bian dianionic complexes to cycloaddition. The influence of the donor ability of the solvent on the coordination mode of the sulfamide ligand was demonstrated. The formed derivatives are stable compounds and, hence, the subsequent modification of the bound azide substrates

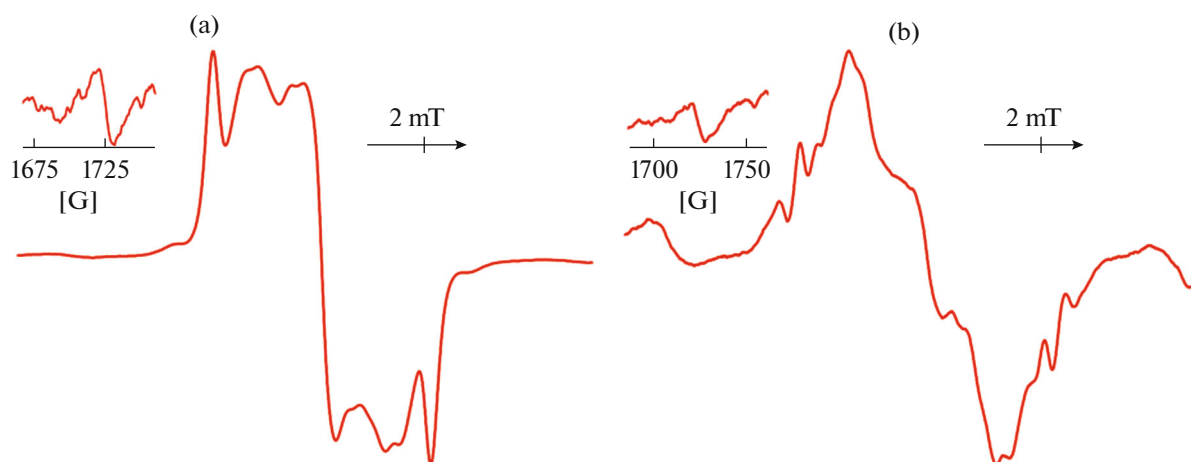


Fig. 1. EPR spectra (toluene, 150 K) of compounds (a) II and (b) III.

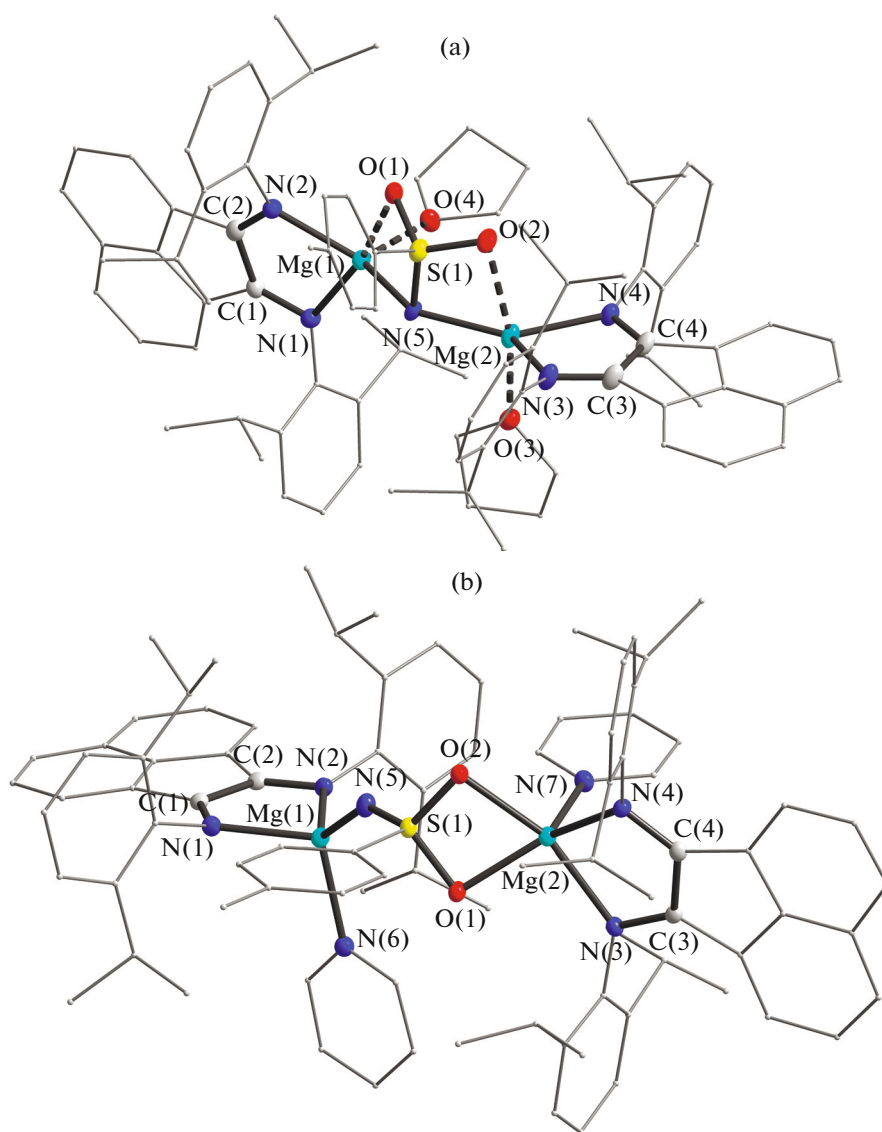


Fig. 2. Molecular structures of compounds (a) II and (b) III. Thermal ellipsoids of 30% probability. Hydrogen atoms are omitted.

aimed at preparing valuable organic products is poorly probable.

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