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Iron Tricarbonyl Complexes Based on *N*,*N*'-Disubstituted Phenanthrenediimines

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Abstract—The oxidative addition of *N*,*N*'-disubstituted phenanthrenediimines to iron carbonyls was studied. The reactions of acceptor phenanthrenediimines with $Fe_2(CO)_9$ give iron(I) tricarbonyl complexes with anion-radical forms of the ligands. The synthesized compounds were characterized by NMR and IR spectroscopy. The structures of the complex based on *N*,*N*'-bis(3-trifluoromethylphenyl)phenanthrenediimine and imidazol-2-one ligand was established by X-ray diffraction (XRD) (CIF files CCDC nos. 2173471 and 2173472, respectively).

Keywords: phenanthrenediimine, iron(I), iron carbonyl, XRD

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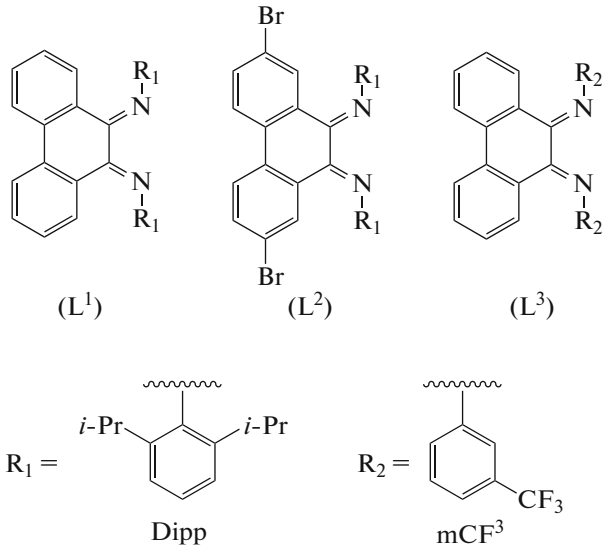
INTRODUCTION

Ligands of the α-diimine series are applied for the construction of a broad range of metal complexes of nontransition and transition elements. Researchers are interested in similar ligands due to their redox activity: they can reversibly accept one or two electrons being in the coordination sphere of the metal.

Phenanthrenediimines are poorly studied ligands in coordination chemistry mainly because of the relatively recent discovery of rational ways of the synthesis [1, 2]. A combination of the extended aromatic system and possibilities of controlling steric and electronic effects makes it possible to use these compounds for the stabilization of low-valent states of Group 14–15 elements [3–5]. The iron complexes with the α-diimine ligands [6–13] are interesting as catalysts for diverse chemical transformations [9, 14–21]. There are no reports on the synthesis iron complexes with phenanthrendiimines ligands in the literature.

The purpose of this work is to synthesize iron complexes based on *N*,*N*'-disubstituted 9,10-phenanthrenediimines. Diimines differed in steric hindrance and electron-withdrawing properties were chosen for the study: 2,6-di-*iso*-propylphenyl (L^1) with a high steric hindrance, 2,6-di-*iso*-propylphenyl-substituted 2,7-dibromophenanthrenediimine (L^2) with bulky substituents and a high acceptor ability, and 3-trifluoromethylphenyl (L^3) with the highest electron-withdrawing ability but a low steric hindrance (Scheme 1).



Scheme 1.

EXPERIMENTAL

Ligands L^1 and L^3 [2] and 2,7-dibromophenanthrenequinone [22] were synthesized according to literature procedures. All manipulations on the synthesis of the complexes were carried out in an evacuated system. Solvents were purified using standard procedures. IR spectra were recorded on an FSM-1201 spectrometer in Nujol in a range of 4000–400 cm^{-1}. NMR spectra were recorded on Bruker Avance III (400 MHz) and Avance Neo (300 MHz) spectrome-

ters. Elemental analysis was carried out on an Elementar Vario El Cube instrument.

Synthesis of *N*,*N*'-bis(2,6-di-*iso*-propylphenyl)-2,7-dibromophenanthrene-9,10-diimine (L^2). A sixfold excess of 2,6-di-*iso*-propylaniline (15.46 mL, 82 mmol) was added to stirred solution of 2,7-dibromo-9,10-phenanthrenequinone (5.0 g, 13.66 mmol) in toluene (50 mL), and $TiCl_4$ (2.99 mL, 5.18 g, 27.3 mmol) was added dropwise to the resulting solution. The reaction was carried out for 4 h. The organic layer was washed with water to the neutral reaction. After the solvent was removed, the product was isolated from acetonitrile. The yield was 6.9 g (73.8%).

For $C_{38}H_{40}N_2Br_2$

Anal. calcd., %	C, 66.67	H, 5.89	N, 4.09
Found, %	C, 66.78	H, 5.92	N, 4.06

^{1}H NMR (300 MHz; C_6D_6; δ, ppm): 8.78 (s, 1H), 7.23–7.29 (m, 1H), 7.20 (s, 1H), 7.01–7.07 (m, 3H), 6.90–7.00 (m, 6H), 2.97 (sept, J = 6.8 Hz, 2H), 2.02 (sept, J = 6.8 Hz, 2H), 1.12 (d, J = 6.9 Hz, 6H), 1.09 (d, J = 6.9 Hz, 6H), 0.82 (d, J = 6.6 Hz, 6H), 0.75 (d, J = 6.7 Hz, 6H).

^{13}C NMR (75 MHz; C_6D_6; δ, ppm): 156.91, 146.18, 135.18, 135.08, 134.64, 133.84, 132.15, 131.74, 130.49, 130.16, 127.8, 127.55, 125.91, 125.14, 125.02, 124.01, 123.85, 122.59, 29.04, 27.56, 24.61, 23.29, 22.62, 22.25.

IR (ν, cm^{-1}): 1640 s, 1617 s, 1587 m, 1486 m, 1438 s, 1406 m, 1361 m, 1322 m, 1280 s, 1254 s, 1219 m, 1187 w, 1168 w, 1101 w, 1078 w, 1057 w, 1041 w, 1005 w, 962 w, 936 w, 892 m, 837 w, 812 s, 794 m, 765 s, 760 s, 732 m, 717 m, 701 w, 669 w, 652 w, 512 w, 459 w.

Synthesis of complex $L^2Fe(CO)_3$ (I). A solution of L^2 (0.2 g, 0.292 mmol) in toluene was added to a suspension of $Fe_2(CO)_9$ (0.106 g, 0.292 mmol) in toluene. The mixture was stirred for 12 h, and the color changed from red to intense vinous. Toluene and $Fe(CO)_5$ were removed under reduced pressure. The residue was dissolved in hexane. After concentrating to 2 mL, the complex was isolated as a dark red powder. The yield was 0.146 g (60.6%).

For $C_{41}H_{40}N_2O_3Br_2Fe$

Anal. calcd., %	C, 59.73	H, 4.89	N, 3.40
Found, %	C, 59.79	H, 4.90	N, 3.38

^{1}H NMR (300 MHz; C_6D_6; δ, ppm): 7.2–7.37 (m, 3H), 6.88–7.10 (m, 9H), 3.03–3.08 (m, 4H), 1.22 (br.s, 12H), 0.84 (br.s, 12H).

^{13}C NMR (75 MHz; C_6D_6; δ, ppm): 211.87, 157.53, 144.31, 141.67, 138.23, 137.81, 129.85, 129.44, 129.01, 128.63, 126.79, 126.33, 126.07, 125.33, 124.64, 124.21, 124.0, 123.56, 119.54, 118.56, 27.95, 23.64, 23.11.

IR (ν, cm^{-1}): 2044 s, 1979 s, 1967 s, 1588 w, 1487 m, 1462 s, 1456 s, 1377 s, 1333 s, 1303 w, 1277 w, 1257 w, 1180 m, 1167 m, 1127 m, 1090 m, 1068 m, 999 s, 898 m, 895 m, 846 m, 820 s, 793 m, 761 s, 725 m, 698 m, 667 m, 623 m, 609 m, 585 w, 570 w, 541 w, 521 w, 506 w.

Synthesis of complex $L^3Fe(CO)_3$ (II) and phenanthroimidazol-2-one (L^4). A solution of L^3 (0.3 g, 0.607 mmol) in toluene was added to a suspension of $Fe_2(CO)_9$ (0.110 g, 0.303 mmol) in toluene. The mixture was stirred for 12 h, and the color changed from red to intense vinous. Toluene and $Fe(CO)_5$ were removed under reduced pressure. The residue was dissolved in hexane and filtered from a colorless powder (L^4). After the solvent was replaced by diethyl ether and the solution was concentrated to 2 mL, complex **II** was isolated as dark red needle-like crystals. The yield of compound **II** was 0.135 g (35%), and the yield of L^4 was 0.137 g (43.1%) based on diimine.

For $C_{31}H_{16}N_2O_3F_6Fe$

Anal. calcd., %	C, 58.84	H, 2.57	N, 4.39
Found, %	C, 58.70	H, 2.54	N, 4.42

Complex **II**: ^{1}H NMR (400 MHz; C_6D_6; δ, ppm): 8.03 (d, J = 8.1 Hz, 2H), 7.73 (d, J = 5.2 Hz, 2H), 7.27 (d, J = 7.4 Hz, 2H), 7.15–7.23 (m, 4H), 7.07 (t, J = 7.6 Hz, 2H), 6.90 (t, J = 7.9 Hz, 2H), 6.77 (t, J = 7.7 Hz, 2H).

^{13}C NMR (101 MHz; C_6D_6; δ, ppm): 209.83, 159.21, 148.30, 130.66, 129.95, 127.80, 127.56, 127.04, 126.68, 126.43, 126.00, 124.12, 122.47, 122.39, 122.35, 120.7, 120.67, 120.64.

IR (ν, cm^{-1}): 2042 s, 1980 s, 1973 s, 1750 w, 1638 m, 1618 m, 1587 m, 1460 s, 1406 w, 1378 s, 1280 m, 1254 w, 1215 w, 1168 w, 1100 w, 1078 w, 1056 w, 1004 w, 962 w, 936 m, 899 w, 891 m, 839 w, 811 s, 795 s, 760 s, 731 m, 723 s, 669 w, 664 w, 631 w, 587 w, 568 w.

Ligand L^4: ^{1}H NMR (300 MHz; $CDCl_3$; δ, ppm): 8.79 (s, J = 8.4 Hz, 2H), 7.90 (s, 2H), 7.7–7.87 (m, 6H), 7.54–7.62 (m, 2H), 7.33–7.42 (m, 2H), 7.24 (d, J = 8.0 Hz, 2H).

^{13}C NMR (75 MHz; $CDCl_3$; δ, ppm): 154.40, 137.16, 132.32 (q, J = 33.3 Hz), 131.88, 130.35, 128.31, 126.89, 125.71 (dq, J = 11.5, 3.7 Hz), 125.34, 124.03, 121.71, 121.26, 120.71, 120.61.

IR (ν, cm^{-1}): 1695 s, 1611 w, 1597 w, 1568 w, 1520 m, 1494 m, 1459 s, 1445 s, 1431 m, 1396 m, 1385 m, 1333 s, 1310 m, 1285 m, 1269 m, 1253 m, 1200 m, 1183 m, 1172 s, 1139 s, 1122 s, 1093 m, 1066 s, 1050 w, 1005 w, 991 w, 978 w, 951 w, 900 w, 887 w,

Table 1. Crystallographic data and XRD experimental parameters for compounds **II** and L^4

Parameter	Value	
	II	L^4
Empirical formula	$C_{31}H_{16}N_2O_3F_6Fe$	$C_{29}H_{16}N_2OF_6$
FW	634.31	522.44
T, K	100(2)	100(2)
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$C2/c$
a, Å	9.7494(6)	22.9831(10)
b, Å	12.2373(7)	10.2587(5)
c, Å	13.1216(7)	9.6320(4)
α, deg	111.594(2)	90
β, deg	111.236(2)	96.9224(16)
γ, deg	92.218(2)	90
V, Å^3	1329.78(13)	2254.45(17)
Z	2	4
ρ_{calc}, mg/m^3	1.584	1.539
μ, mm^{-1}	0.647	0.128
θ, deg	2.28–29.13	2.97–27.48
Number of collected reflections	10505	14581
Number of independent reflections	6942	2592
R_{int}	0.0240	0.0496
$S(F^2)$	1.023	1.053
R_1, wR_2 ($I > 2\sigma(I)$)	0.0468, 0.1134	0.0506, 0.1105
R_1, wR_2 (for all parameters)	0.0603, 0.1228	0.0758, 0.1210
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.653/–0.770	0.330/–0.350

856 m, 811 m, 793 w, 776 w, 756 m, 738 s, 717 m, 700 m, 667 w, 658 w, 650 m, 614 w, 550 w, 528 w.

For $C_{29}H_{16}N_2OF_6$

Anal. calcd., %	C, 66.67	H, 3.09	N, 5.36
Found, %	C, 66.75	H, 3.12	N, 5.31

XRD of compounds **II** and L^4 was carried out on a Bruker D8 Quest single-crystal diffractometer (MoK_α radiation, λ = 0.71073 Å, φ and ω scan modes). Diffraction data were collected, initial indexing of reflections was performed, and unit cell parameters were refined using the APEX3 program [23]. Experimental sets of intensities were integrated using the SAINT program [24, 25]. The structures of compounds **II** and L^4 were solved by direct methods using the dual-space algorithm in the SHELXT program [26] 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. The structures were calculated using the SHELXTL software [27, 28]. An absorption correction was applied in the SADABS program [29]. The crystallographic data and structure refinement parameters for compounds **II** and L^4 are given in Table 1. Selected bond lengths and bond angles of compounds **II** and L^4 are listed in Table 2.

The structures were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 2173471 (**II**) and 2173472 (L^4); https://www.ccdc.cam.ac.uk/structures/).

RESULTS AND DISCUSSION

The oxidative addition of diimines to iron carbonyls was chosen as a convenient method for the synthesis of metal complexes. A similar approach makes it

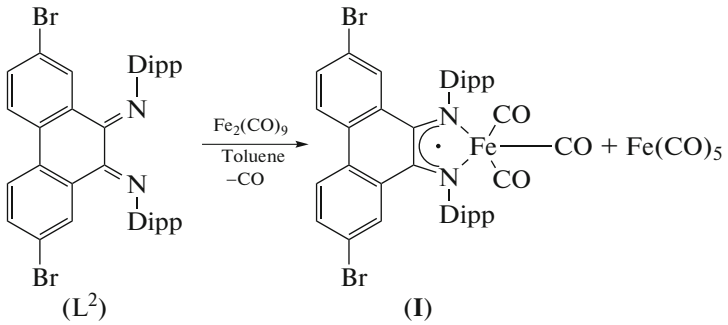
Table 2. Selected bond lengths (Å) and bond angles (deg) in compounds **II** and L^4

Bond	*d*, Å	Bond	*d*, Å
		II	
Fe(1)–C(29)	1.803(2)	Fe(1)–N(2)	1.909(2)
Fe(1)–C(30)	1.808(2)	N(1)–C(1)	1.347(3)
Fe(1)–C(31)	1.801(2)	N(2)–C(2)	1.350(3)
Fe(1)–N(1)	1.906(2)	C(1)–C(2)	1.430(3)
		L^4	
C(15)–O(1)	1.222(3)	N(1)–C(1)	1.405(2)
C(15)–N(1)	1.383(2)	C(1)–C(1A)	1.367(3)
Angle	ω, deg	Angle	ω, deg
		II	
N(1)Fe(1)N(2)	79.44(7)	N(2)Fe(1)C(30)	159.52(10)
N(1)Fe(1)C(30)	92.54(9)	C(31)Fe(1)N(1)	111.17(9)
N(2)Fe(1)C(29)	92.43(9)	C(31)Fe(1)N(2)	104.36(9)
C(29)Fe(1)C(30)	86.35(10)	C(31)Fe(1)C(29)	95.50(10)
N(1)Fe(1)C(29)	153.26(9)	C(31)Fe(1)C(30)	96.10(11)
		L^4	
N(1)C(15)N(1A)	105.5(2)	N(1)C(15)O(1)	127.25(10)

possible to prepare the target complexes with a minimum amount of by-products. It is found that phenanthrenediimines do not react with iron pentacarbonyl even on heating regardless of their steric or acceptor characteristics.

When $Fe_2(CO)_9$ is used, only diimines L^2 and L^3 with a high acceptor ability enter into the reaction. The reaction occurs with stirring for 12 h in toluene. After the solvent was removed, a pale yellow volatile product was found in the evacuated system. The volatile was determined by mass spectrometry as $Fe(CO)_5$. Similar reactions of diazadienes with $Fe_2(CO)_9$ to form iron pentacarbonyl were exemplified [6, 30].

The interaction of two equivalents of ligand L^2 with one equivalent of $Fe_2(CO)_9$ (Scheme 2) resulted in a change in the solution color from red to violet, and gas evolution was observed. After volatile components were separated, the residue contained 50% diimine taken in the reaction and complex **I**, which was isolated in the individual state as a dark red powder.

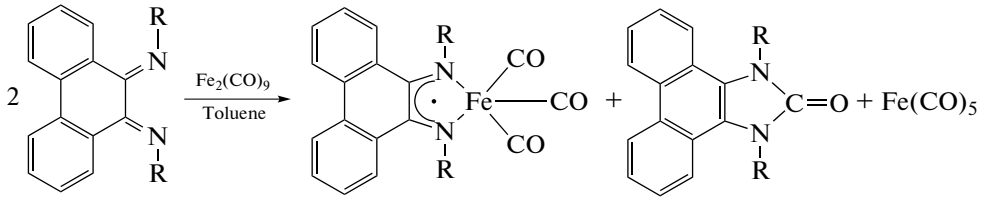


(L^2) (**I**)

Scheme 2.

The ^{1}H NMR spectrum of complex **I** exhibits signals from the hydrogen atoms of the equivalent alkyl groups of the aryl substituents at the nitrogen atoms, indicating that the organic fragment is symmetric (*E,E* configuration), whereas free diimine has a nonsymmetric *E,Z* configuration [2]. The ^{13}C NMR spectrum contains a peak at 211 ppm, which indicates the presence of the carbonyl ligand. The IR spectrum exhibits absorption bands of the organic ligand and intense absorption bands in ranges of 1975 and 2040 cm^{-1} in the terminal carbonyl stretching region.

In the case of 3-trifluoromethylphenyl-substituted diimine, the reaction with the same reactant ratio has some distinctions: no gas evolution is detected. The reaction mixture remained after $Fe(CO)_5$ removal contains two products isolated in the pure form: intensely colored metal complex **II** and colorless crystals of L^4 (Scheme 3).



(L^3) (**II**) (L^4)

R = mCF^3

Scheme 3.

A similarity of the NMR and IR spectra of compounds **I** and **II** suggests the same structures of these compounds.

The crystals of compound **II** suitable for XRD were obtained from diethyl ether. A molecule of compound **II** is a pentacoordinate iron complex (Fig. 1a). The Fe(1) atom has a distorted tetragonal pyramidal environment. The value of τ for the coordination sphere of Fe(1) is 0.10, which is close to a similar value for an ideal tetragonal pyramid ($\tau = 0$) [31]. The diimine ligand and two carbonyl substituents with the C(29) and C(30) atoms lie in the base of the pyramid, and the third C≡O ligand with the C(31) atom occupies the axial position. A similar structure is characteristic of the α-diimine complexes with iron tricarbonyl [7, 8]. The C–N and C–C bond lengths, which are equal to 1.347(3), 1.350(3), and 1.430(3) Å, respectively, are characteristic of the anion-radical form of the ligand [32]. The phenanthrene fragment of the diimine ligand is nonplanar with an average deviation of the carbon atoms of 0.14 Å. Complex **I** regardless of the conditions crystallizes as thin needle-like crystals, do not suitable for single-crystal x-ray diffraction study.

In spite of the radical nature of the organic ligand, complexes **I** and **II** are diamagnetic, which is associated with the antiferromagnetic interaction of electrons of the radical anion and low-spin iron(I) atom. Attempts to replace the CO groups in the iron(I) tricarbonyl complexes and to prepare the iron compounds in a higher oxidation state by the oxidation with a diimine excess were unsuccessful.

Colorless crystals of L^4 turned out to be poorly soluble in aromatic and saturated hydrocarbons. The IR spectrum of the compound contains an intense absorption band of the carbonyl group at 1695 cm^{-1}. The ^{1}H NMR spectrum exhibits signals from the protons of the phenanthrenediamide fragment in the symmetric configuration. The ^{13}C NMR spectrum contains a peak at 154 ppm along with the signals from the phenanthrene moiety. The structure of compound L^4 was solved by XRD (Fig. 1b). A molecule of L^4 is symmetric, and the rotation twofold axis passes through the center of the C(1)–C(1A) bond and further along the O(1)–C(15) bond. Unlike complex **II**, the phenanthrene fragment is planar. The average deviation of the carbon atoms from the phenanthrene plane is 0.03 Å. In molecules of compounds **II** and L^4, the CF_3 groups of the phenyl substituents are directed to different sides from the phenanthrenediimine ligand plane. A similar configuration of the 3-trifluoromethylphenyl substituents was found in the phenanthrenediimine complex of ZnI_2 [33]. The single mentioning of similar products formed due to the reactions of diimines with transition metal carbonyls is known only for the reaction of diazadiene with iron carbonyl [34].

Thus, the reactions of *N,N'*-diaryl-substituted phenanthrenediimines with iron carbonyls were studied. It was found that $Fe_2(CO)_9$ reacted only with acceptor phenanthrenediimines affording the Fe(I) tricarbonyl complexes with the anion-radical ligand. One more reaction product, phenanthroimidazol-2-one, is formed when phenanthrenediimines with small substituents at the nitrogen atoms are used.

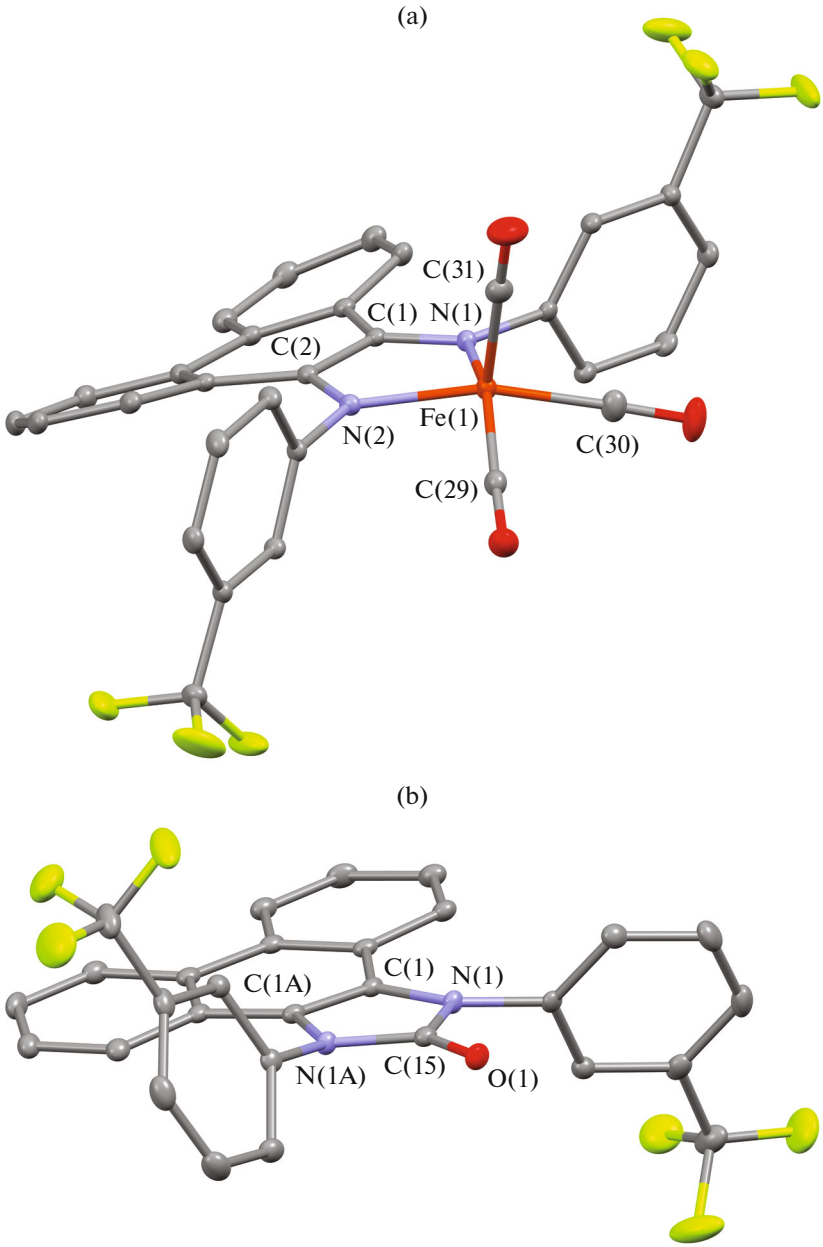


Fig 1. Molecular structures of (a) anion-radical complex **II** and (b) ligand L^4. Thermal ellipsoids are given with 30% probability. Hydrogen atoms are omitted.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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