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Iminoquinones and Diimines, *N*-Hetero Analogues of 9,10-Phenanthrenequinone, in Coordination Chemistry

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Abstract—The synthesis of N-hetero analogs of 9,10-phenanthrenequinones is discussed. The features of their structure are considered. Information on the use of these compounds in coordination chemistry as neutral, mono-, and dianionic ligands is summarized.

Keywords: 9,10-phenanthrenequinone, α-diimines, iminoquinones, redox-active ligands

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INTRODUCTION

In the last decades, ligands of variable valence, so-called redox-active ligands, have attracted considerable attention in coordination chemistry. These ligands, being located in the metal coordination sphere, are able to reversibly add one or two electrons to give a radical anion or a dianion. The main representatives of this class of ligands are *o*-quinones, *o*-iminoquinones, α-dithiolenes, and α-diimines [1]. Ligands of this type incorporated in metal complexes can considerably affect their reactivity. 1,2-Bis(imino)acenaphthene (BIAN) is an actively studied redox-active ligand. Using this ligand, it was possible to obtain compounds of Main Group metals with metal–metal bonds able to reversibly add alkynes [2], similarly to transition metal complexes. In the case of magnesium complex with mono(imino)acenaphthenequinone, this addition is irreversible [3]. The activation of small molecules [4] and addition of heterocumulenes [5, 6] were observed for bis(imino)acenaphthene-based digallane.

The BIAN complexes of transition metals are well known and proved to be efficient catalysts for cycloisomerization [7], polymerization of alkenes [8, 9], copolymerization of CO and styrene [10], copolymerization of CO2 and methylenecyclopropene [11], and hydrogenation of alkynes [12]. 9,10-Phenanthrene mono- and diimines, which are close structural analogues of BIAN, combine the fused aromatic system of phenanthrene and controllable steric and electronic effects via variation of substituents at the nitrogen atoms or introduction of additional groups into peripheral parts of the molecule. This variability suggests that they are potentially able to form a broad range of compounds with various elements in which the diimine exists in neutral, radical anion, or dianionic state.

PHENANTHRENEQUINONEIMINES

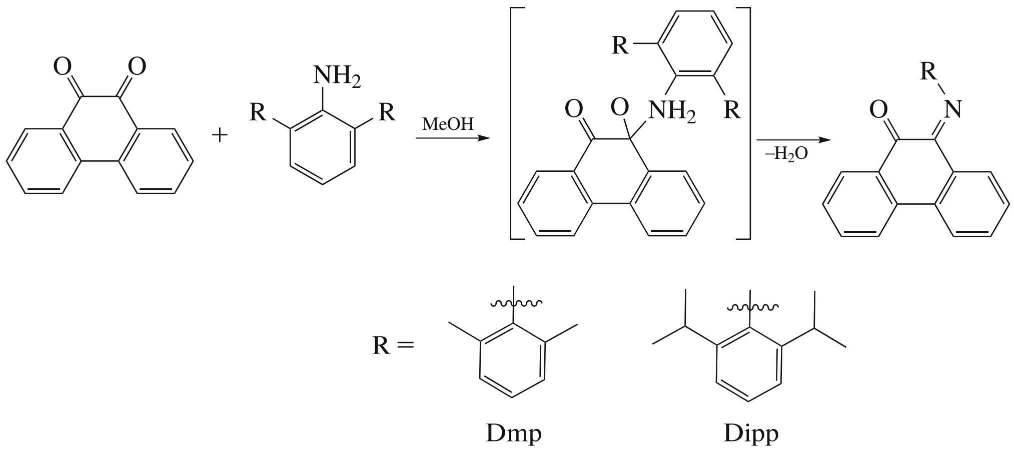
Phenanthrenequinone is employed in coordination chemistry as neutral, radical anion, and dianionic ligand [13, 14]. However, the preparation and use of 9,10-phenanthrenequinone mono- and diimines containing substituents at the nitrogen atom was not adequately studied until recently.

The first mention of phenanthrenequinoneimine dates back to the late 19th century [15]. More recently, it was utilized as a phenanthrenediamine precursor [16], while the first study devoted to copper(I) quinoneimine complexes and their oxidation with oxygen appeared only at the end of the 20th century [17].

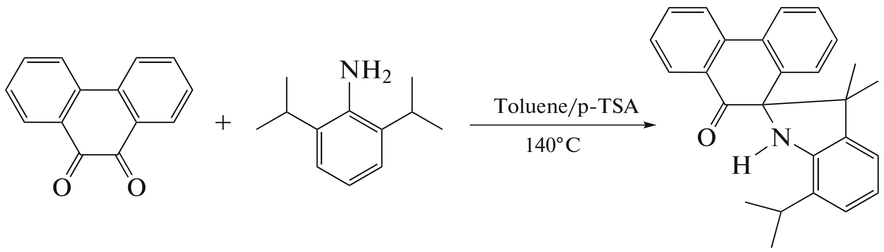
In the beginning of the 21st century, *N*-substituted phenanthrenequinoneimine was prepared; 1,2-nucleophilic addition of an amine to the carbonyl group of *o*-quinone gave substituted *o*-iminophenanthrenequinones [18]. The amine was represented by *o*-toluidine, 2,6-dimethylaniline, and 2,6-di-*iso*-propylaniline. The reactions were carried out in methanol on heating in the presence of catalytic amounts of formic acid. The products were isolated in preparative amounts only in the case of sterically crowded disubstituted anilines.

Later, a group of authors [19] reported the synthesis of phenanthrenequinoneimines containing a 2,6-di-*iso*-propylphenyl substituent with a *para*-halogen atom, which were obtained by the same procedure.

These compounds were chosen as models for studying non-covalent interactions of the lone pairs of the halogen atoms and hydrogen atoms in conjugated systems.



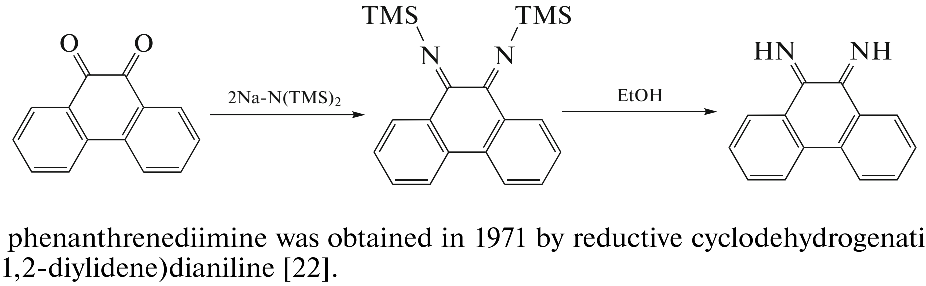
Attempts to replace the second oxygen atom by using more drastic reaction conditions [20] gave the product of reaction between the *iso*-propyl group in the N-substituent and the phenanthrene moiety, along with the formation of quinoneimine.



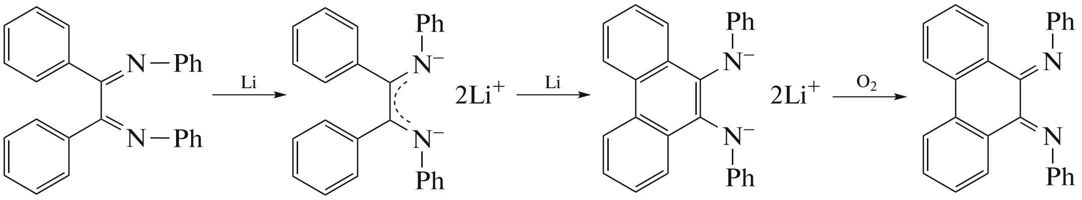
Thus, the preparation of phenanthrenediimines by mere condensation in the presence of Brønsted acids proved to be impossible.

PHENANTHRENEDIIMINES

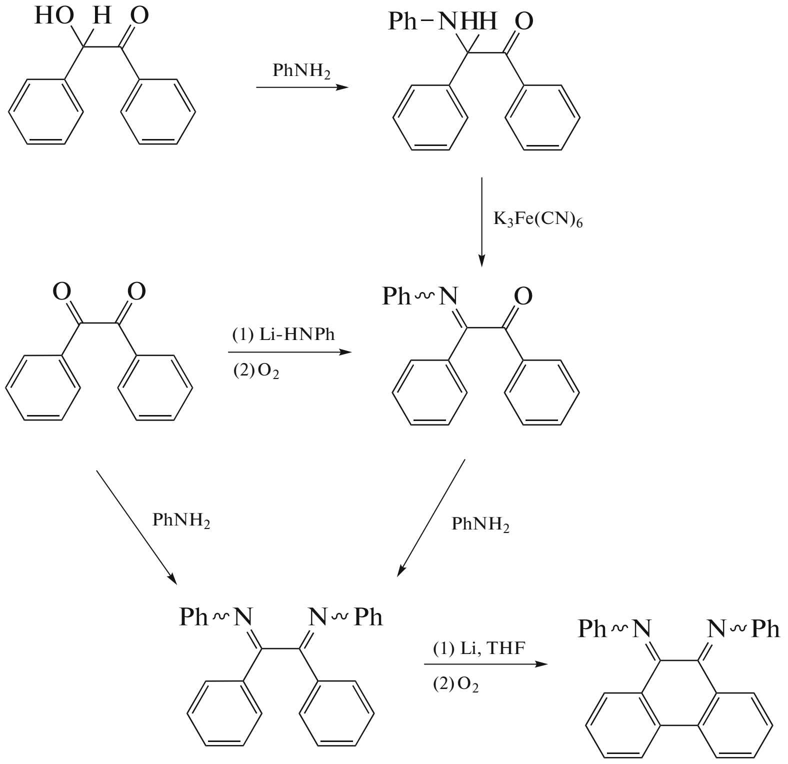
Unsubstituted 9,10-phenanthrenediimine was first obtained by G. Tuchtenhagen in 1968 by the reaction of sodium bis(trimethylsilyl)amide with 9,10-phenanthrenequinone [21]. However, the introduction of substituents to the nitrogen atoms is impossible by this method; this restricts the applicability of these diimines in coordination chemistry.



A substituted phenanthrenediimine was obtained in 1971 by reductive cyclodehydrogenation of *N*,*N*′-(1,2-diphenylethane-1,2-diylidene)dianiline [22].



Later [23], several new approaches were proposed for the preparation of phenanthrenequinone-α-diimine derivatives, starting from 2-hydroxy-1,2-diphenylethanone or dibenzoyl.

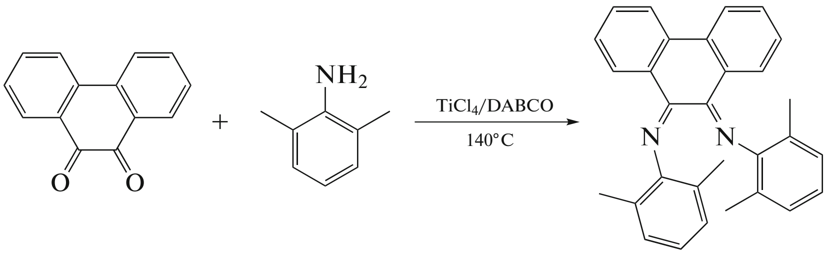


The reaction of imino ketone with another aniline provides the possibility to obtain 9,10-Phenanthrenediimines with various substituents at nitrogen atoms. The reaction of dibenzoyl with an excess of aniline gives diimine only for anilines with small substituents in the aromatic ring: tolyl-, methoxyphenyl-, or 2-*iso*-propylaniline. Phenanthrenediimines with alkyl substituents cannot at all be prepared by this procedure.

In addition, an unusual structure was found [23] for *N*,*N*'-diphenyl-9,10-phenanthrenediimine, namely, *Z*,*Z*-configuration both in the solid state and in solution, which is not typical of diimines with a rigid structure. Figure 1 shows its molecular structure in two projections.

In the case of more sterically hindered *o*-*iso*-propylphenyl substituent, *Z*,*E*-configuration is formed, as was indicated by the presence of NMR signals for nonequivalent protons of the *iso*-propyl groups.

The synthesis of new *N*,*N*'-disubstituted 9,10-phenanthrenediimine reported in [24] is based on the reaction of 9,10-phenanthrenequinone with 2,6-dimethylaniline in the presence of the $TiCl_4$/1,4-diazabicyclo[2.2.2]octane (DABCO) system at 140°C, which gives the product in 50% yield.



This reaction is temperature-sensitive and when conducted at elevated temperature (160°C) it gives the product of interaction between the methyl group of *E*-configured substituent and the phenanthrene moiety in up to 35% yield.

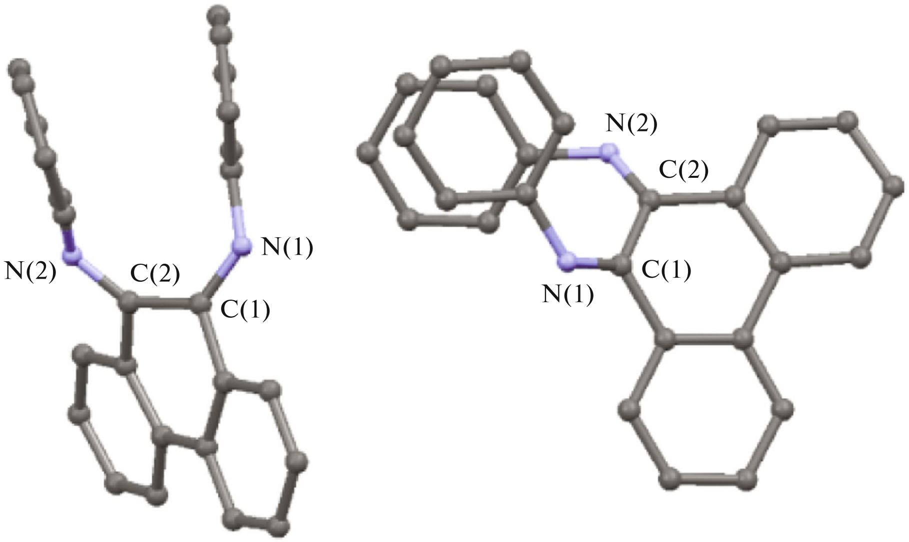
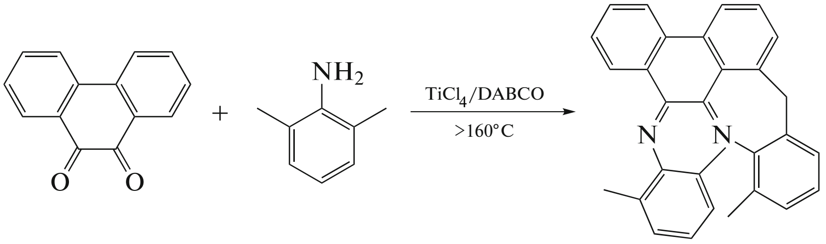
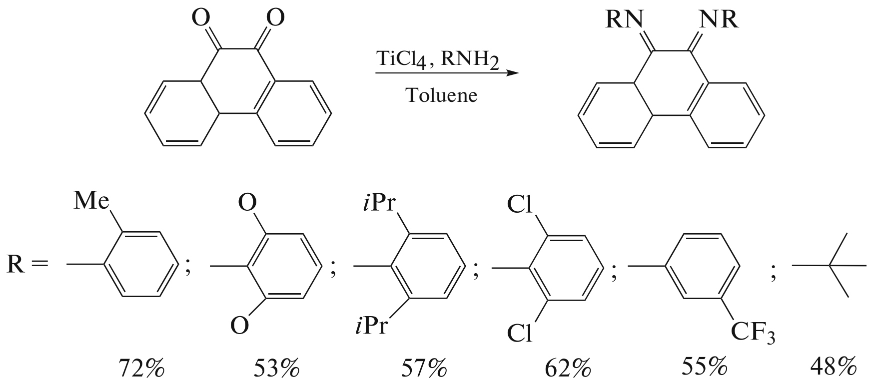


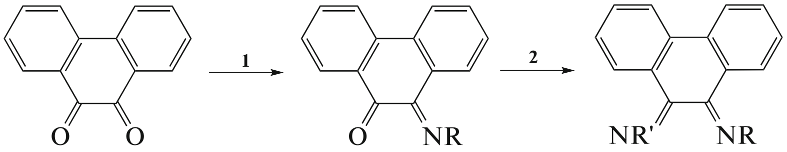
Fig. 1. Molecular structure of *N*,*N*'-diphenylphenanthrene-9,10-diimine.



In 2011, the procedure for the preparation of diimines in the presence of titanium tetrachloride was modified at the Laboratory of the Chemistry of Organoelement Compounds of the Razuvaev Institute of Organometallic Chemistry, Russian Academy of Sciences. The reaction with a sixfold excess of primary amine in the presence of 2 equiv. of $TiCl_4$ in toluene carried out under milder conditions (temperatures between room temperature and 70°C) resulted in the synthesis of phenanthrenediimines with *N*-substituents of different nature and different electronic properties [25].



In view of the fact that the reactions of 9,10-phenanthrenequinone with primary amines carried out in refluxing methanol in the presence of formic acid stop after the formation of phenanthrenequinoneimine, the authors proposed a synthesis of *N*,*N*'-disubstituted 9,10-phenanthrenediimines with various *N*-substituents comprising two steps: (1) reaction of 9,10-phenanthrenequinone with excess 2,6-di-*iso*-propylaniline in methanol in the presence of formic acid to give quinoneimine (85% yield); (2) reaction of 10-((2,6-di-*iso*-propylphenyl)imino)phenanthren-9(10H)-one with aromatic or aliphatic amine (e.g., *tert*-butylamine) and $TiCl_4$ in toluene at room temperature to give diimine (75% yield for t-$BuNH_2$ and 70% yield for 2,6-dimethylaniline).



According to X-ray diffraction data, crystalline diimines with Dmp, Dipp, and *t*-Bu substituents and the diimine with alkyl and aryl substituents exist in the *Z*,*E*-form, with the aryl-substituted imine group being in the *E*-form and the alkyl-substituted imine group being in the *Z*-form (Fig. 2).

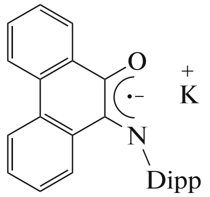
It was also found that *N*,*N*'-di-*tert*-butyl-9,10-phenanthrenediimine retains the *Z*,*E*-configuration in solution, as evidenced by the signals for non-equivalent *tert*-butyl groups in the NMR spectrum. Phenanthrenediimines with sterically hindered aryl substituents behave in a similar way.

There is only one example [26] of the synthesis of phenanthrenediimine without the use of titanium tetrachloride. The procedure includes refluxing of ethanol solutions of *p*-nitroaniline and phenanthrenequinone in the presence of a catalytic amount of glacial acetic acid. In parallel with the studies carried out at the Razuvaev Institute of Organometallic Chemistry, in 2012, a group of Chinese researchers [27] reported the synthesis of *N*-hetero analogues of 9,10-phenanthrenequinone, where phenanthrenediimines were also prepared from both phenanthrenequinone and phenanthrenequinoneimines using titanium tetrachloride. The key distinction of this procedure is the use of DABCO and higher temperature (140°C).

PROPERTIES OF *N*-HETERO ANALOGUES OF 9,10-PHENANTHRENEQUINONE

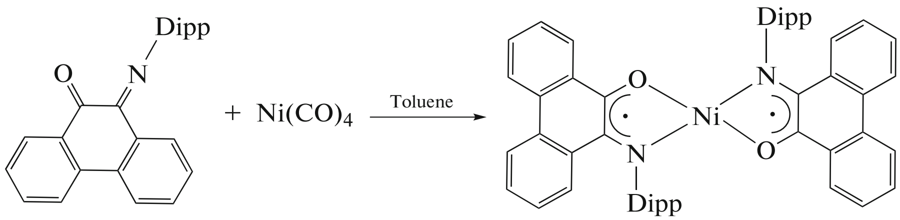
Properties of phenanthrenequinoneimines. Transition metal complexes with quinoneimine are of interest as catalysts of polymerization and various organic reactions. The role of phenanthrenequinoneimine in this case is to stabilize various states of metals owing to the ability to exist in several reduced states.

The reduction of *o*-iminobenzoquinones and benzoquinones with potassium metal is known to comprise two steps and to result in the dianionic form via the formation of a stable radical anion. It was found that [18] phenanthrenequinoneimines behave in a similar way when reduced with alkali metals.



The ESR spectrum of the phenanthrenequinoneimine radical anion exhibits a triplet (1 : 1 : 1) due to the hyperfine coupling of the unpaired electron with the nitrogen atom and the multiplet due to splitting on the phenanthrene protons ($a_N = 6.6$ G, $g_i = 2.0041$).

Phenanthrenequinoneimine-based complexes can be prepared by oxidative addition reactions to zerovalent transition metal complexes. The reaction of nickel tetracarbonyl with 2,6-di-*iso*-propyl-substituted phenanthrenequinoneimine gives a radical anion complex [18].



The use of $Ni(COD)_2$ and the neutral ligand made it possible to obtain dianionic complex [28].

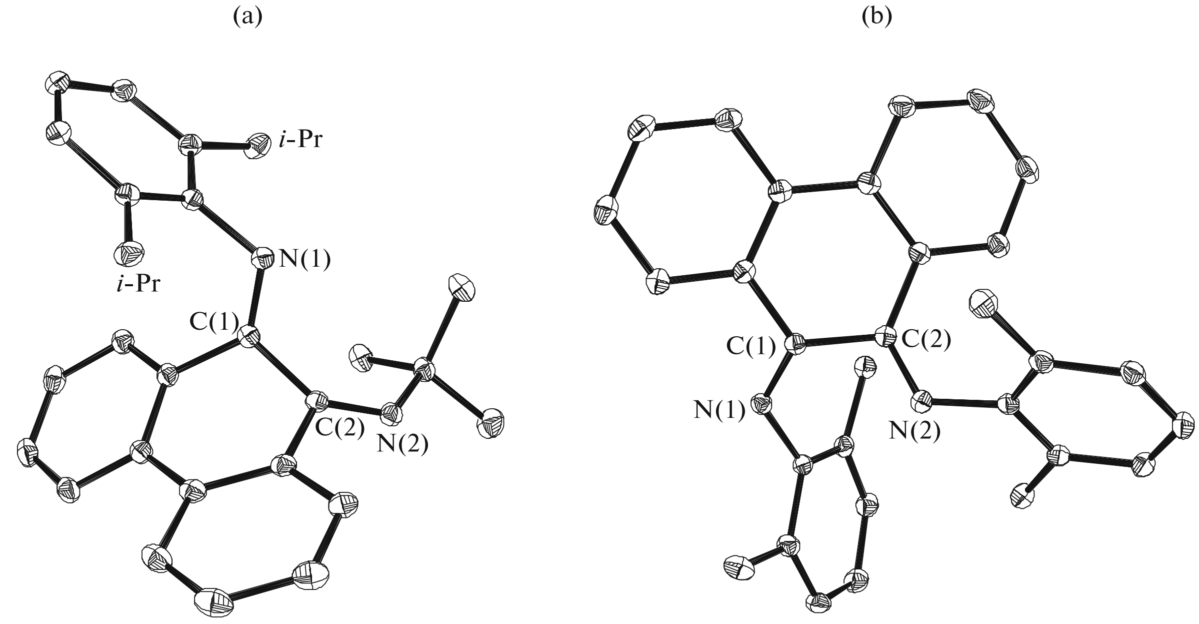
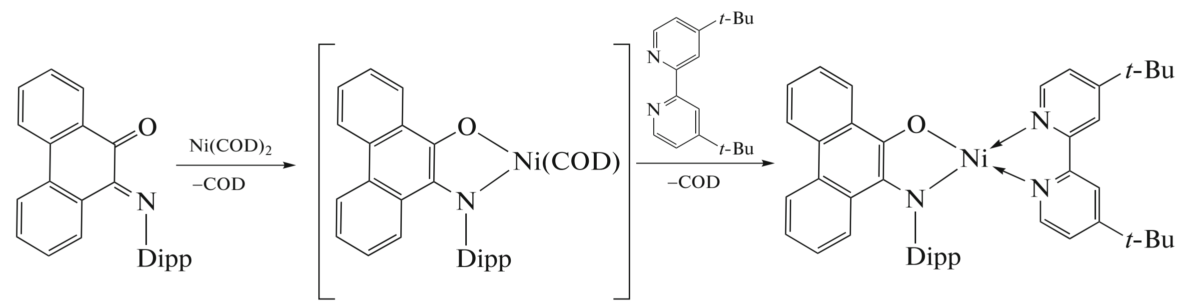
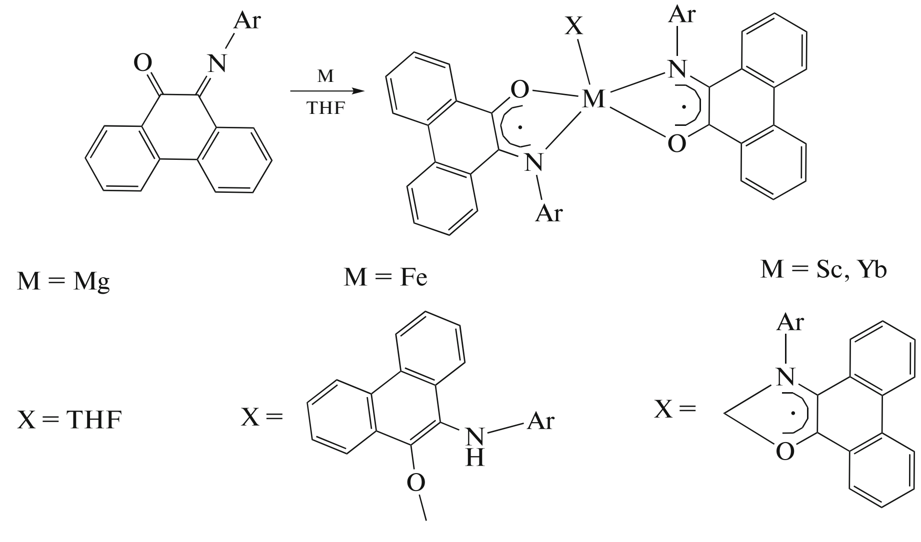


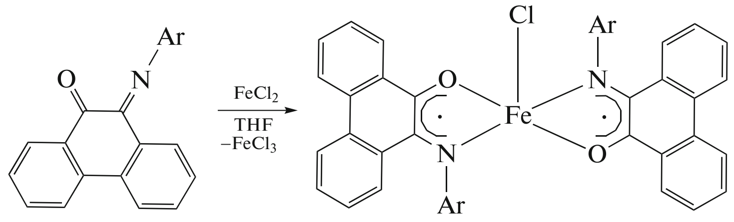
Fig. 2. Molecular structure of phenanthrenediimines with sterically hindered substituents: (a) *N*-*tert*-butyl-*N*'-(2,6-di-*iso*-propylphenyl)-, (b) *N*,*N*'-bis(2,6-dimethylphenyl)-substituted diimines.

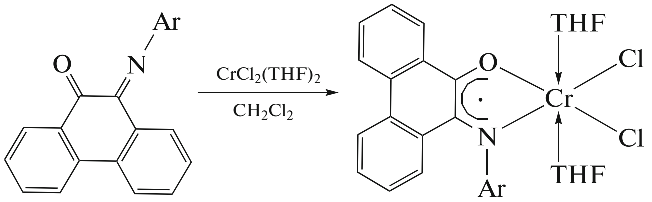


Metals such as iron [29], magnesium [30], scandium, and ytterbium [31] can reduce phenanthrenequinoneimines to radical anions.

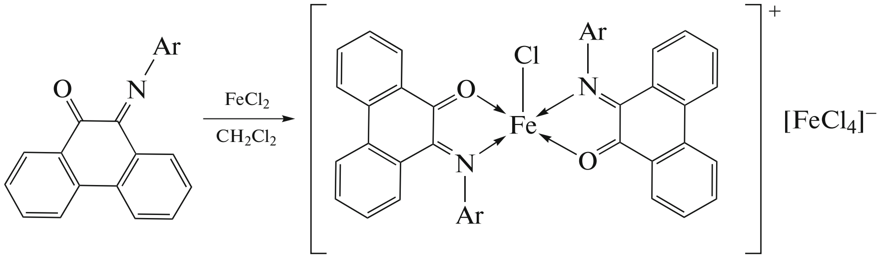


In the case of iron [29] and chromium [27] salts, complexes can be obtained by oxidation of M(II) to M(III).

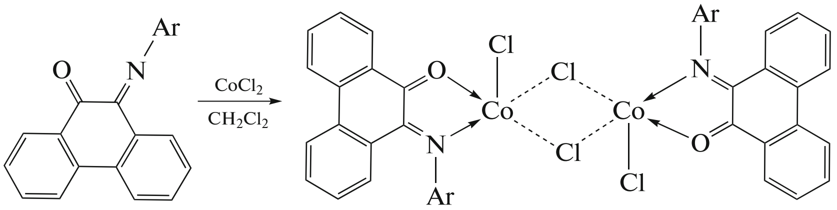




However, when the dichloromethane is used as the solvent, iron oxidation does not take place; instead, a complex with the neutral phenanthrenequinoneimine is formed [29].

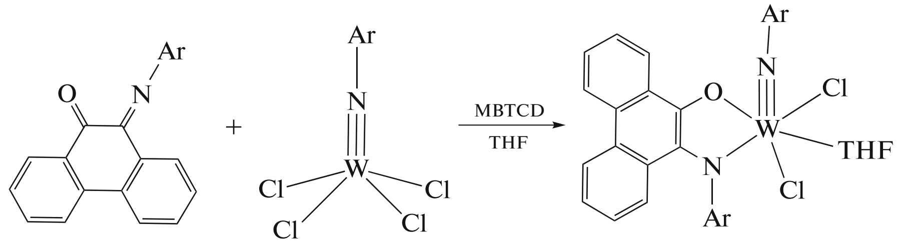


Pphenanthrenequinoneimines react with cobalt(II) in a similar way [32].

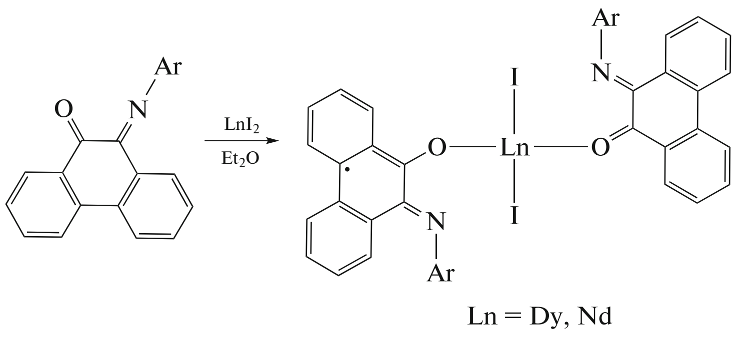


The radical anion chromium complexes and cobalt complexes with neutral phenanthrenequinoneimine show good selectivity in diene polymerization giving *cis*-1,4-products.

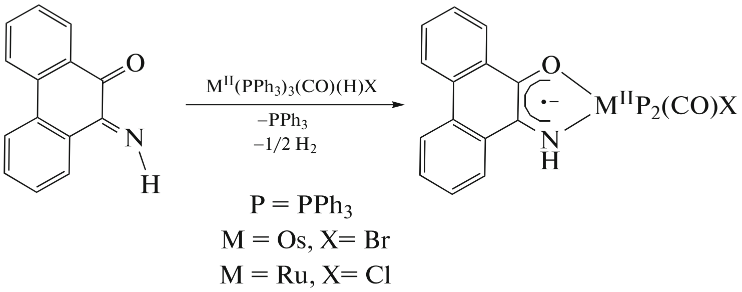
The reduction of high-valence tungsten chloride in the presence of phenanthrenequinoneimine on treatment with 1-methyl-3,6-bis(trimethylsilyl)-1,4-cyclohexadiene (MBTCD) gives a dianionic complex. If chlorine atoms are replaced by alkyl groups, the reaction affords a precursor of the catalyst for ring opening metathesis polymerization of norbornene; however, the catalyst activity is moderate in comparison with that of the diazadiene analogue because of high thermal stability [33].



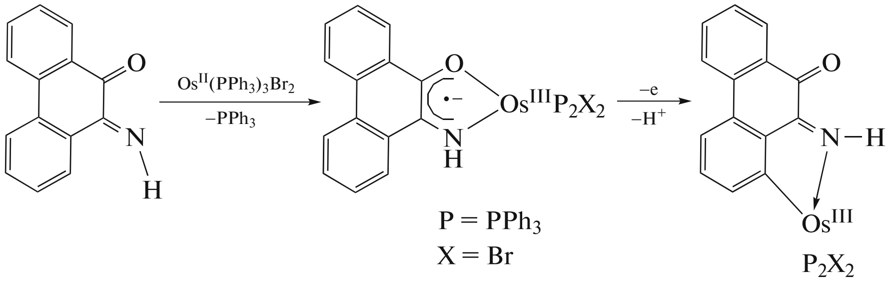
The reduction of quinoneimines with dysprosium(II) and neodymium(II) iodides gives radical anion complexes of an unusual structure. They contain two ligands, neutral and singly reduced ones, bound to the metal via oxygen atoms rather than in the chelating mode [31].



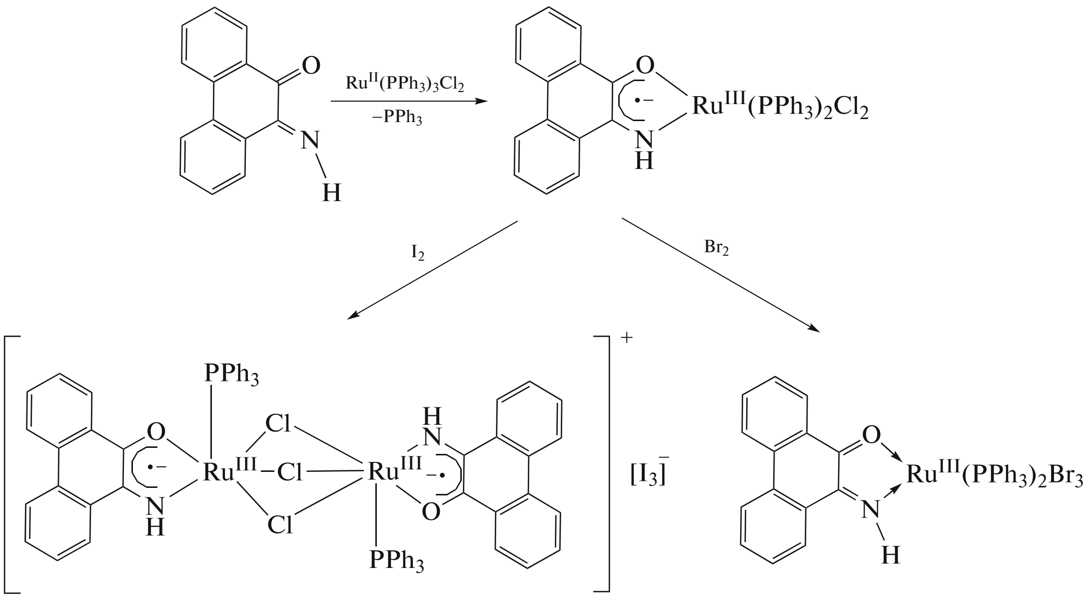
The reduction of phenanthrenequinoneimines with osmium(II) [34] and ruthenium(II) [35] hydrides can be used to prepare radical anion complexes in which the metal retains the initial oxidation state.



An interesting feature is inherent in osmium(III) radical anion complexes. The reaction of these compounds with halogens (I_2, Cl_2, Br_2) is accompanied by replacement of N,O-chelation by N,C-chelation and removal of the radical anion state of the ligand [34].

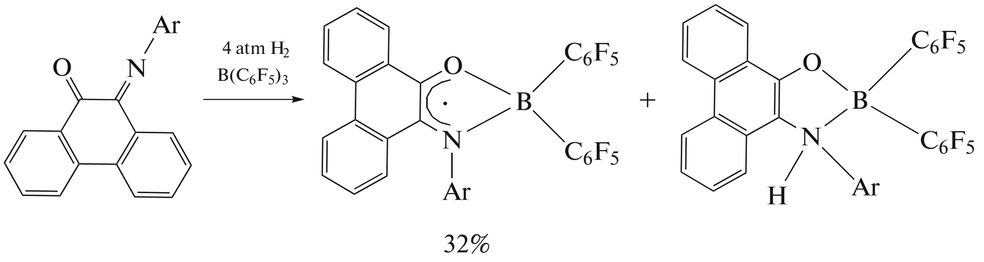


Similar ruthenium complexes do not show this type of behavior; they are oxidized to give a binuclear complex in the case of iodine or a complex with the neutral ligand in the case of bromine [35].



Ruthenium acetylacetonate can serve as yet another precursor for the preparation of radical anion complexes of ruthenium [36].

All of the above-listed compounds are highly sensitie to traces of moisture and air. However, phenanthrenequinoneimine can be used to obtain air-stable radical anion derivatives, which was demonstrated in relation to boracyclic radicals [37].



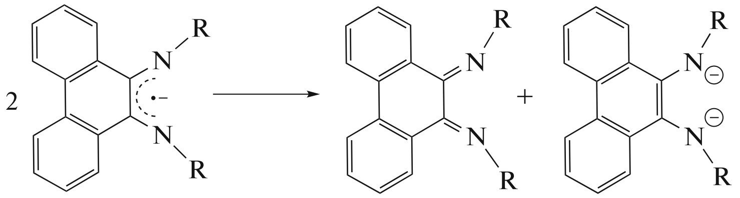
There are scattered publications dealing with the phenanthrenequinoneimine complexes with transition metals (Ni, Cr, Co, Fe, Cu, Ru, Os), magnesium, and boron in which the ligand occurs in the neutral, radical anion, or dianionic form. The singly reduced (radical anion) form can be regarded as the major phenanthrenequinoneimine species of the complexes. Analysis of the structural data indicates that the bond lengths in the chelate ring are in a narrow range for all forms of the ligand (Table 1).

Properties of phenanthrenediimines. Structural analogues of 9,10-phenanthrenediimines such as *o*-quinones, iminoquinones, 1,4-diazadienes (DAD), and BIAN react with alkali metals to give stable radical anion complexes, which can be easily detected by ESR. However, although phenanthrenediimines are structurally related to these compounds, in this case, the radical anion form of the ligand cannot be detected by ESR upon reduction with potassium. In order to evaluate the ability of phenanthrenediimine ligands to participate in redox reactions, their electrochemical properties were studied by cyclic voltammetry [38]. The reduction of a number of phenanthrenediimines, which occurs in this case as one irreversible two-electron step to give the dianion, was carried out.

No waves corresponding to single-electron reduction of neutral diimines to their radical anions were observed in the CV curve, which is indicative of low stability of the free radical anion form in solution. It was found that the radical anion form rapidly disproportionates to give the dianion and the initial diimine.

Table 1. Bond lengths in the chelate ring for phenanthrenequinoneimine-based complexes

Form	Bond, Å		
	C–C	C–N	C–O
Neutral	1.46–1.48	1.27–1.30	1.25–1.27
Radical anion	1.42–1.45	1.30–1.35	1.27–1.29
Dianionic	1.38–1.39	1.40–1.42	1.30–1.34

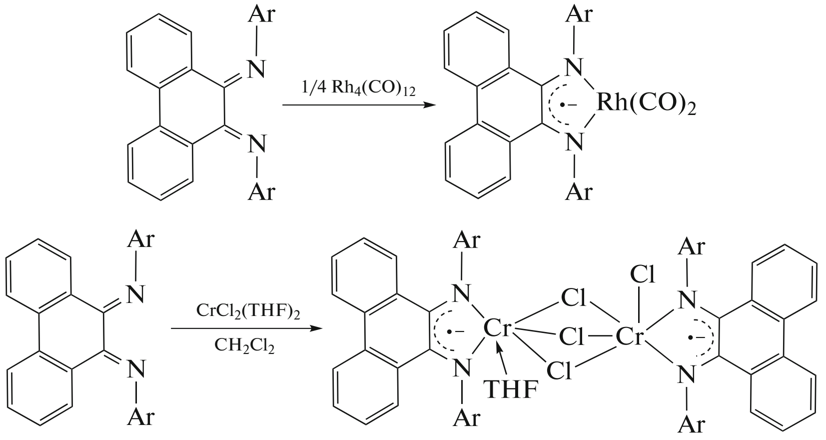


Despite the instability of radical anions of most phenanthrenediimines, the single-electron reduced form was obtained using lithium as the reducing agent. In this case, the ESR spectra show signal acquisition for the phenanthrenediimine radical anion, with the signal intensity decreasing subsequently down to zero (Fig. 3).

In the case of alkyl–alkyl and alkyl–aryl phenanthrenediimines, the single-electron reduced form cannot be detected by ESR even if the reduction is carried out by lithium.

This behavior of phenanthrenediimines was attributed to the fact that their reduction on treatment with potassium yields a solvent-separated electron pair and the resulting radical anion disproportionates. In the reaction with lithium, a contact ion pair is formed, which stabilizes the radical anion.

On going from the ionic species to the complex in which the metal forms a chelate ring with the radical anion, stabilization of the radical anion increases, as was demonstrated in [38] for the reaction with rhodium carbonyl and chromium dichloride [27].



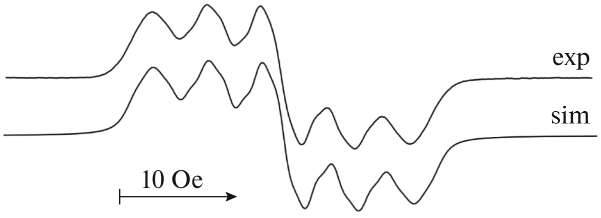
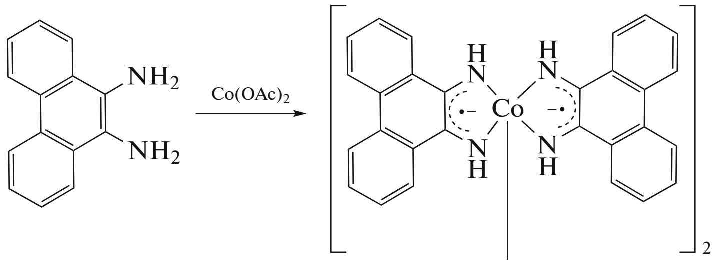


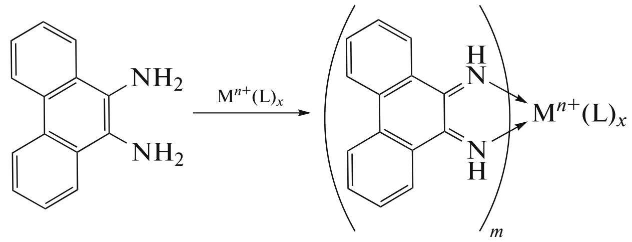
Fig. 3. ESR spectrum of the lithium complex of *N*,*N*-bis(2,6-di-*iso*-propylphenyl)-9,10-phenanthrenediimine, $g_i = 2.0030$, $a_{N1} = a_{N2} = 4.5$ G, $a_{H1} = a_{H2} = 1.45$ G, $a_{H3} = a_{H4} = 0.85$ G, $a_{Li} = 1.05$ G.

Worth noting are examples of radical anion complexes with unsubstituted phenanthrenediimine, which are prepared from phenanthrenediamine. The preparation of double-decker cobalt complexes [39] and multidecker rhodium complexes [40] was reported in the literature.

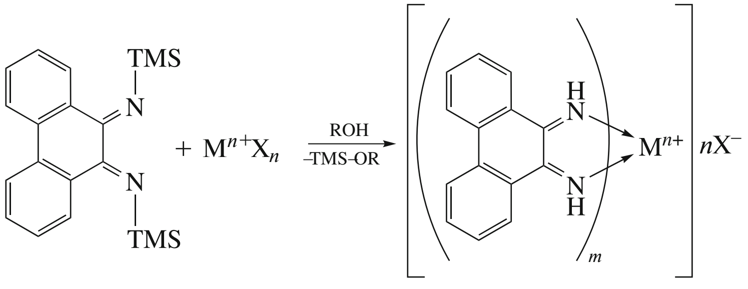


Despite the instability of the radical anion in solution, metal coordination can stabilize this state of the ligand. Until now, only the above-mentioned compounds have been described in the literature.

The use of phenanthrenediimines as neutral ligands for the preparation of coordination compounds can be divided into two periods, studies using *N*-unsubstituted phenanthrenediimine and studies performed after preparation methods for the substituted diimines have been developed. Transition metal complexes [41] and noble metal complexes [36, 42–45] based on pristine phenanthrenediimine were prepared. The synthesis was performed using diaminophenanthrene or bis(trimethylsilyl)phenanthrenediimine as the starting compound.



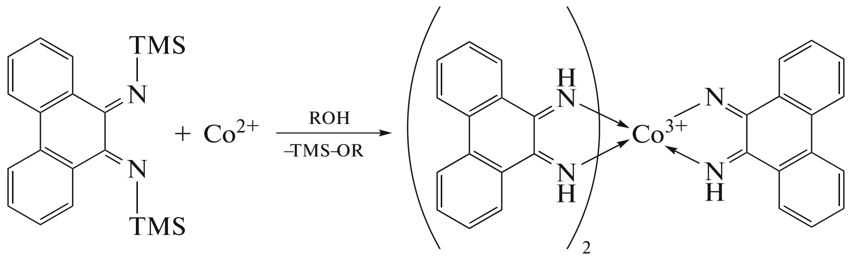
M = Ir, $n = 2$, $m = 1$
M = Ru, $n = 2$, $m = 1, 3$
M = Rh, $n = 3$, $m = 1, 2$



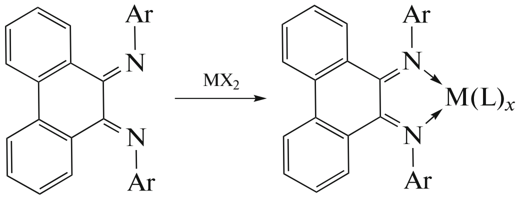
M = Fe, Ni, $n = 2$, $m = 3$
M = Cu, $n = m = 2$
M = Pd, $n = 2$, $m = 1$
M = Rh, $n = 3$, $m = 1$

X = Cl, ClO$_4$

However, in the case of cobalt, an unusual complex is formed, in which one ligand forms the amide bond with the metal and the oxidation state of the metal changes.



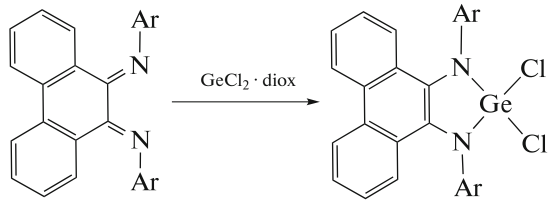
Metal complexes based on substituted phenanthrenediimines are constructed by mere reaction between the metal salt and the original diimine. Several derivatives of transition metals were reported (Zn [38, 46], Co [46], Cu [46], Ni [24, 46, 47]).



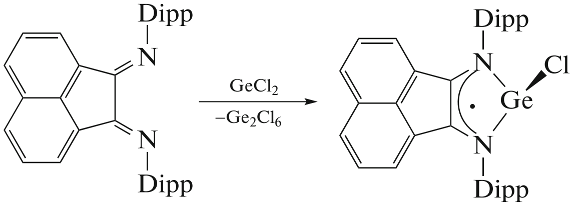
Complexes of $NiBr_2$ with phenanthrenediimines in the neutral form proved to be good catalysts for alkene polymerization [24, 48–50].

Complexes with noble metals are much less studied, with only a few reports of one research group being available. [51, 52]. It was found that palladacyclopentadiene complexes containing neutral phenanthrenediimines ligand are able to catalyze the synthesis of conjugated dienes from alkynes.

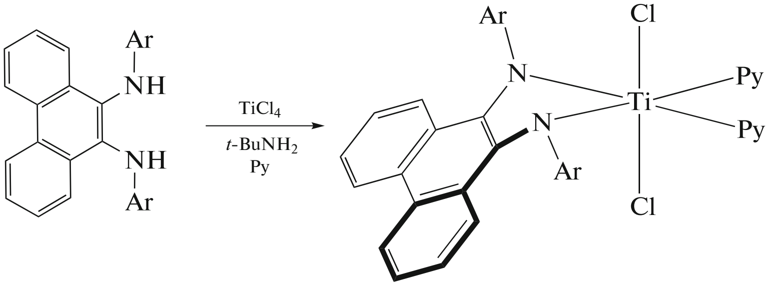
Phenanthrenediimines are readily reduced to the dianionic form with magnesium [30] or via oxidative addition to germanium(II) chloride dioxane complex [38]. It was found that the magnesium complex can catalyze stereoselective ring opening polymerization of a racemic mixture of lactides [53].



It is known that BIAN, structurally similar to 9,10-phenanthrenediimines, behaves in a different way under these conditions, that is, forms a radical anion derivative [54]:

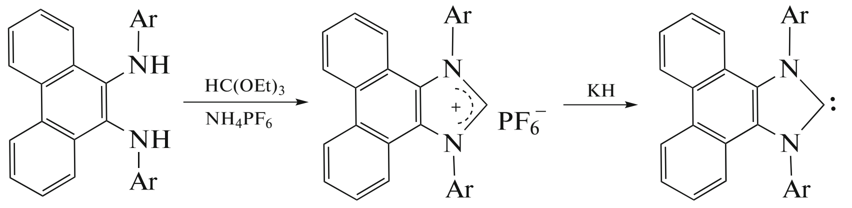


Titanium(IV) diamide complex can be prepared by the reaction of diamine with titanium tetrachloride in the presence of *tert*-butylamine [55].



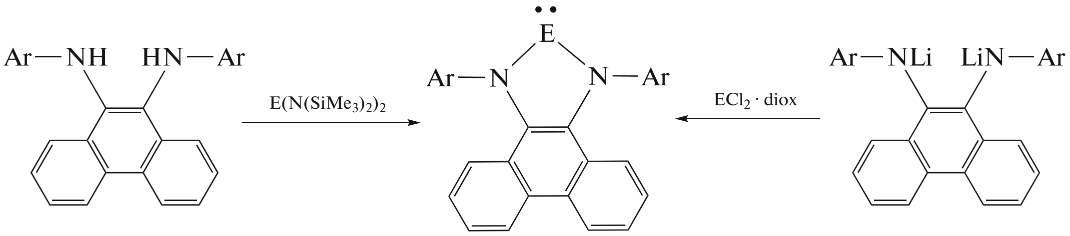
The distortion of the coordination unit geometry was attributed to the overlap of the lone pairs of nitrogen with the vacant d_{xy} orbital of titanium.

Owing to combination of the extended π-system of phenanthrene and the possibility of controlling the steric crowding of the coordination unit, phenanthrenediimines may be useful for stabilization of low-valence compounds of Group 13–15 elements, so-called Arduengo type carbene analogues. Only few publications addressing this issue are available from the literature. *N*-Heterocyclic carbene was obtained by treatment of the imidazolium salt with potassium hydride [56].



If the *N*-substituent is not bulky (*p*-Tol), carbene is formed as a dimer; however, even in the case of *o*-Tol, the monomeric product can be isolated.

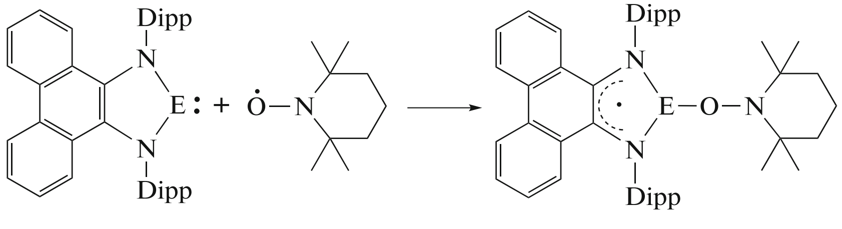
N-Heterocyclic germylenes [56, 57] and stannylenes [57] were prepared in two ways, by the reaction of dilithium diamide salt with metal dichloride or by the reaction of diamine with low-oxidation-state metal silylamide.



E: Sn, Ge

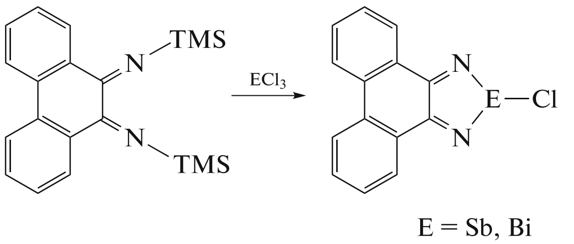
Attempted preparation of plumbylene by the reaction of the lithium salt of diimine with lead diiodide was unsuccessful and resulted in the liberation of the metal.

An interesting feature of *N*-heterocyclic carbene analogues is the ability to undergo redox transformations with variation of the oxidation state of the ligand. It is known that on treatment with stable radicals, metal complexes containing *o*-quinone- and α-diimine-based dianionic ligands are converted to radical anion derivatives, which are readily detected by ESR [58]. In order to prepare and study the stability of paramagnetic germylene and stannylene derivatives based on *N*,*N*'-bis(2,6-di-*iso*-propylphenyl)-9,10-phenanthrenediimine, reactions with oxygen-centered organic radicals, (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO) and 3,6-di-*tert*-butyl-2-ethoxyphenoxyl radical, were carried out [57].

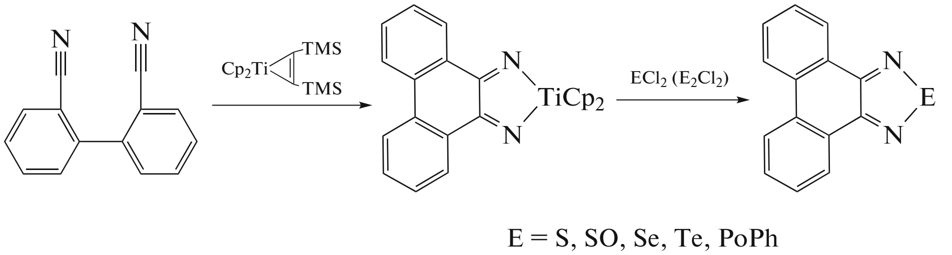


E = Sn, Ge

The first known 1,3,2-diazastiboles and 1,3,2-diazabismoles were synthesized using phenanthrenediimine. In the case of antimony, stabilization of the heterocycle can also be attained using benzyldiimine; however, in the case of bismuth, compound would be stable only if phenanthrenediimine is used [59].



Of certain interest for the preparation of *N*-heterocyclic compounds annulated to phenanthrene are reactions with diazatitanacyclopentadiene. The key features of this process include the absence of phenanthrenequinone in the reaction pathway and the absence of *N*-substituents; this can affect the heterocycle stability [60].



Thus, analysis of known literature sources demonstrates that *N*-hetero analogues of phenanthrenequinone can form complexes in all three redox forms: neutral, radical anion, and dianionic ones. Notably, despite the structural similarity of phenanthrenediimines and BIAN, the properties and behavior of complexes based on these ligands are different. In the case of BIAN, remarkable discoveries were made in relation to complexes of Group 13 elements; however, no publications on this topic are available for phenanthrenediimines. Studying phenanthrenediimines-based compounds of Group 14 elements showed their ability to add free radicals while retaining the valence state of the metal by means of participation of the redox-active ligand in the reaction. This behavior of Main Group metal complexes may be promising as regards their use in catalysis. However, the chemistry of *N*-hetero analogues of phenanthrenequinone is still at its infancy. The development of this research area was mainly held up by the lack of rational and convenient methods for the synthesis of 9,10-phenanthrene mono- and diimines. These methods appeared only in the beginning of the 21st century and thus opened up the scope for further research of metal complexes based on these molecules.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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