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[ahmad alkhawaldeh](#) *

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Article

Cyclic Voltammogram Analysis of the 3,3' - (1,4-Phenylene) Perchlorates

Ahmad khalaf alkawaldeh ^{1,*}

¹ Department of Medical Allied Sciences, Zarqa University College, Al-Balqa Applied University, Jordan

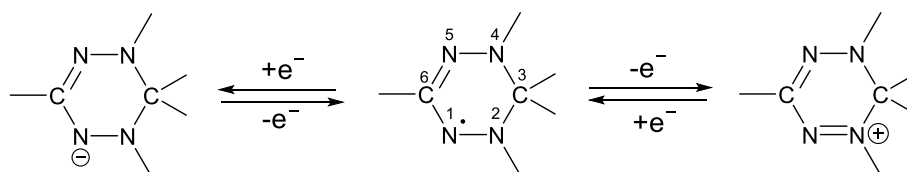
* Correspondence: ahmad.khawaldeh@bau.edu.jo

Abstract: As a consequence of a three-stage synthesis from terephthalic aldehyde, a series of 3,3' - (1,4-phenylene) perchlorates including methyl, hydrogen, acetyl, bromio, methoxy and nitro groups (R = Br, H, Me). The matching formazans were reacted with formalin in dioxane in the presence of perchloric acid to produce tetrazin ium salts. Formazans were synthesized by reacting terephthalic aldehyde phenylhydrazone with ar ene diazonium tosylates in a DMF/pyridine combination. The use of ar ene diazonium tosylates greatly simplified the separation and purification of formazans. The p henylhydrazone was produced in aqueous dioxane using the usual approach from terephthalic aldehyde and phenylhydrazine/hydrochloric acid. Individual f ormazans and tetrazinium perchlorates were isolated and characterized by elemental analysis, IR, NMR, UV spectroscopy. The electrochemical reduction of tetrazinium perchlorates was investigated using cyclic voltammetry (CV). Thus, all salts in the cathode region have two one-electron reduction peaks, which are connected to the sequential production of a radical cation and a biradical. It was discovered that donor substituents (R: OMe, Me) in the aromatic ring at position 1 accelerate tetrazinium cation reduction, whereas acceptor substituents (R: NO₂, COMe,) inhibit this process. On this premise, it was proposed that in the presence of donor substituents in the aromatic ring at position 1, the matching bis-verdazyl radicals should be easily generated. As a result, 3,3'-(1,4-phenylene) perchlorates are possible antecedents of symmetric biradical systems based on verdazyl radicals.

Keywords: cyclic voltammograms; chronoamperometric, radical, electron-withdrawing substituent; electrode potential

1. Introduction

Tetrazinyl radicals (verdazyls), first reported in 1964 by Kuhn and Trishman, are "electronamphoteric," that is, they can be oxidized and restored to form with stable anions cations (Scheme 1).



Scheme 1. Oxidation and reduction of verdazyl radicals.

Precursors in verdazilny x radicals are typically salts of 5,6-dihydro-1,2,4,5-tetrazine I, from which neutral leuco base is formed, which oxidizes tsya to radicals. Researchers are interested in verdazyl radicals because of their remarkable chemical stability, magnetic characteristics, structural diversity, and capacity to form complexes with metals. The electrochemical properties of these radicals have received far less attention; for instance, the electrochemical behavior of the triphenylverdazyl radical in DMF in the absence of carboxylic acids has been investigated (Alkhawaldeh, 2020).

The cyclic voltammetry method was used to investigate the redox properties of a series of triaryl verdazyl radicals with varied substituents in aromatic fragments, and a correlation between both the

electron spin distribution and redox properties was discovered. The researchers discovered that the boundary orbitals that contribute to the oxidation and recovery processes are localized in different parts of the molecule: the HOMO atom and nitrogen in position 2 contribute the most to the oxidation process, while the LUMO atom and carbon in position 6 contribute the most to the recovery process (Alkhawaldeh, 2022).

In this context, new organometallic (aliphatic, cyclic and heterocyclic) compounds are being searched and used with a variety of functional groups of most transition metals. Iron is necessary for the ecosystem and is non-toxic. Iron, as we all know, plays a critical role in the ecosystem. This element is also very important in the biosphere. Iron is required for photosynthesis (cytochromes, ferredoxins), as well as a variety of enzymes. In the absence of iron, the formation of chlorophyll is delayed. Chlorosis (yellowing) and loss of leaf color are possible, especially when young. With prolonged iron deficiency, dense grasses die off at the edges of the leaf blade, tree shoots die off, overall productivity decreases, and plant resistance to diseases decreases (Alkhawaldeh, 2021).

It should also be highlighted that iron compounds are useful in the petrochemical industry. Many compounds have been investigated in automobiles to replace the environmentally toxic tetraethyl lead as a fuel additive. Although great effort has been done to improve the effectiveness of fuel additives in this area, more effective ways are still required, and development is now continuing (Alkhawaldeh, 2020).

It is well known that the reaction of aliphatic ketones with ferrocene is simpler and yields more than that of cyclic ketones. It is worth noting that there are numerous uncertainties around the manufacturing of ferrocenylcycloketones. Reactions between aliphatic ketones and ferrocene were carried out in the presence of a catalyst $\text{H}_2\text{SO}_4/\text{DEAN}$ (diethylammonium naphthenate) (2:1), and matching ferrocenylcycloketones were produced. The reaction between cyclic ketones and ferrocene has been studied as a follow-up to previous studies. (Alkhawaldeh, 2020).

The use of 3-phenyl-1,5-diaza-7-oxa-4-phospha-1,3,5,7-tetraazaphosphorin-2-ylidene in a symmetrically second nonaqueous second redox battery is discussed. Describes the synthesis of 6-oxoverdazyl radical polymers with customizable electrochemical characteristics. Verdazyl radicals also have photofluorescent characteristics. Thus, the chemistry of verdazyl radicals is fascinating and diversified, but the salts of 1,3,5-triaryl-5,6-dihydro-1,2,4,5-tetrazine derivatives of I, which are precursors Kuhn radicals, have received far less attention. Our research focused on the synthesis of a series of 3,3'-(1,4-phenylene) perchlorates as probable precursors of verdazyl biradicals (Alkhawaldeh, 2020).

The reactivity of hydrogen sulfide and various mercaptanes with aliphatic and aromatic compounds has been investigated. This method yielded noxious-smelling ferrocenylthioethers. These chemicals dissolve in almost all organic solvents, particularly benzene. This feature can be used to purify some oils that include mercaptane or its fractions. Given this, hazardous compounds from exhaust gases, particularly unburned residues, are currently the most significant and urgent environmental issues for diesel and non-standard motor fuels that contaminate air. It is widely known that the substitution of ferrocene for the aromatic core in organic compounds results in products with features that are not characteristic of or are less exhibited in the starting compounds.

2. Experimental

Before usage, the solvents were dried and distilled. All reagents were acquired from Sigma-Aldrich Rus LLC / Merck LLC and used in their original form. NMR spectra of ^1H and ^{13}C of compound solutions were recorded on an ECX400 spectrometer JNM (JEOL, Japan; 400.1 and 100.6 MHz, respectively) for the substance solutions in DMSO, and chemical shifts relative to SiMe_4 were determined. The constants of spin-spin coupling were measured in hertz. On an Infracum FT-02 Fourier spectrometer, IR spectra of KBr pellets were acquired (Russia). Elemental analysis was carried out using a Vario MICRO CHNS analyzer (Germany).

TLC conditions for analysis: adsorbent - Silufol UV-245, eluents - benzene; benzene - ethyl acetate (2: 1), development in an iodine chamber. Using an MP-50 melting point analyzer, the melting points of the compounds were evaluated in sealed glass capillaries (Mettler Toledo, Switzerland). Cyclic voltammetry was used to collect electrochemical data in a solution of acetonitrile (0, 1 M

supporting electrolyte Bu_4NBF_4) utilizing a Gamry (Canada) potentiostat in a 5 ml electrochemical cell. As a working electrode, a glassy carbon (CU) electrode $S^2 = 0, 125 \text{ cm}^2$ is utilized. Before taking measurements, the electrode was meticulously polished and rinsed. A platinum auxiliary electrode was used, as well as a typical silver chloride electrode as a reference electrode ($E^0 = 0.33 \text{ B, Me, CN}$ and Fc). By blowing, all solutions were totally deaerated.

By blowing argon through all of the solutions, they were entirely deaerated. To a solution of 6.07 g (0.042 mole) phenylhydrazine hydrochloride and 2.88 g anhydrous sodium acetate in 70 ml of water, add tiny amounts of 2.68 g (0.02 mole) terephthalic aldehyde in 25 ml of dioxane. The reaction mixture was stirred at room temperature for 60 minutes after the addition was completed. The precipitate was filtered off and the filter was cleaned with water. Recrystallized from an ethanol/DMSO combination after being dried in the air.

$t\text{-BuONO}$ was added to the resultant mixture after it had been cooled to 0°C . The reaction mixture was agitated at 0°C for 30 minutes, then at room temperature for another 30 minutes. The arenediazonium tosylates produced were used without further purification. This reaction mixture turned a deep dark cherry color. The reaction mixture was kept at a temperature of around 0°C for 24 hours. The reaction mixture was then given 2 ml of water, and the precipitated precipitate was filtered off and washed on the filter with methanol, water, methanol, and diethyl ether in that order.

3. Results and Discussions

Starting from terephthalaldehyde, a straightforward three-stage synthesis of 3,3'-(1,4-phenylene) perchlorates was achieved. Their electrochemical characteristics were investigated using cyclic voltammetry. In three phases, symmetric tetrazinium perchlorates ($\text{R: MeO, H, CO, Me, Me, Br, NO}_2$) were synthesized from terephthalic aldehyde (Figure 1). Terephthalaldehyde phenylhydrazone was obtained in the first step, formazans in the second, and suitable perchlorates in the third. The method proposed by Katritzky was utilized to synthesize perchlorates.

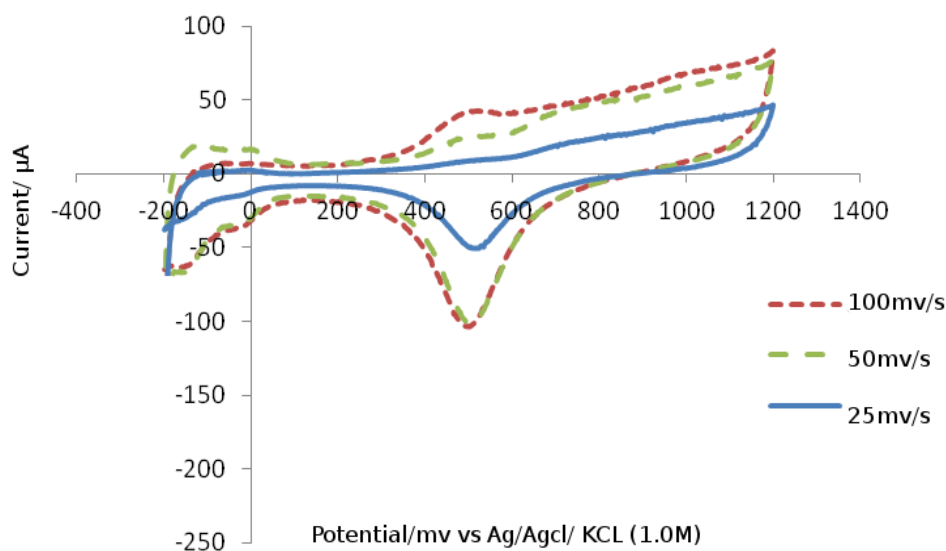


Figure 1. Cyclic voltammograms of 3,3'-(1,4-phenylene).

Formazans were created by combining phenylhydrazone and terephthalaldehyde 3 with tosylates arenediazonium, which were derived from steam ameschennyh anilines. The decision to employ tosylates arenediazonium was taken since getting arenediazonium chlorides in the case of some para-substituted anilines is challenging due to limited solubility in water and low reactivity. Because of the solubility of arenediazonium tosylates in organic solvents, we were able to streamline the purifying technique and employ arenediazonium chlorides. Almost all formazans are dark red crystalline compounds that are highly pigmented. However, unlike the remaining compounds,

compound f is orange in hue. Cyclization of formazans was carried out in dioxane solution using a 37 percent formalin solution in the presence of perchloric acid.

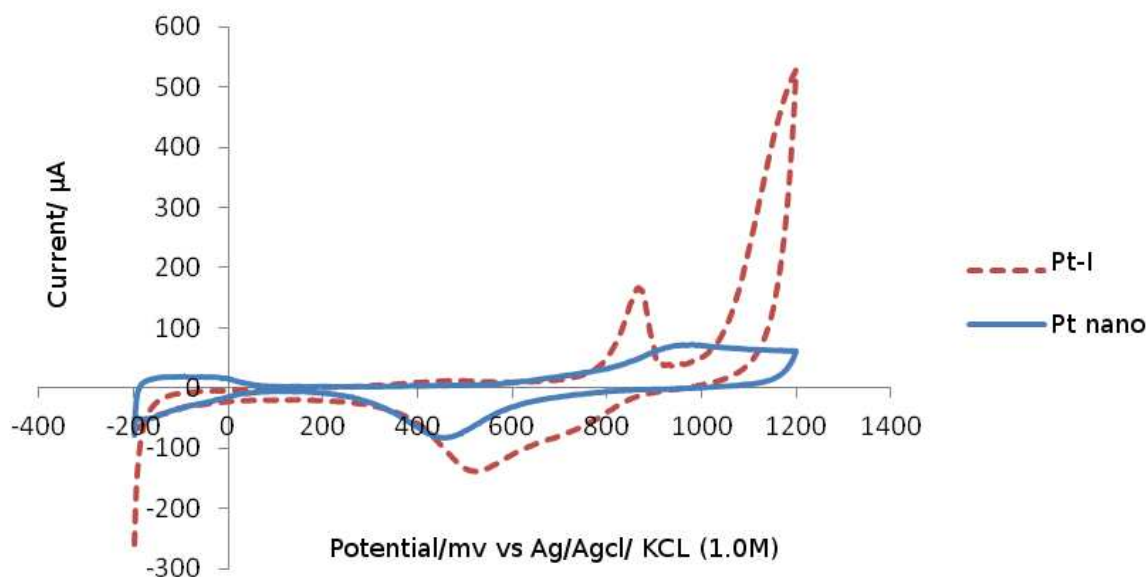


Figure 2. Cyclic voltammograms of platinum oxidation phenylhydrazine and terephthalaldehyde.

Formazans were synthesized by mixing phenylhydrazine and terephthalaldehyde 3 with tosylates ap ene diazonium obtained from steam-s ameschennyh anilines. Because arenediazonium chlorides are difficult to obtain in the case of some para-substituted anilines due to restricted solubility in water and low reactivity, the choice to use tosylates arenediazonium was made. We were able to expedite the purification procedure and use arenediazonium chlorides due to the solubility of arenediazonium tosylates in organic solvents. Almost all formazans are intensely colored dark red crystalline molecules. Compound f, on the other hand, is orange in color, unlike the other compounds. Formazans were cyclized in dioxane solution with a 37 percent formalin solution in the presence of perchloric acid (Figure 2).

According to the literature second technique, fenilgidrazon was generated in 90% yield by condensation of terephthalic aldehyde and phenylhydrazine; its constants correspond to the literature data. Tosylates ap ene diazonium were synthesized by adding claim -toluolsulfokisloty and tert -butyl nitrite to aniline solutions in a combination of THF - CH₃COOH. Without additional purification, oli diazonium was used to drive the reaction with the chemical. The reactions of tosylates arendiazo n and I with phenylhydrazine were carried out in a combination of DMF and pyridine in a 2:1 ratio. Formazan was acquired in the form of CV spectra that were independently analyzed.

Spectrum: Acquisition 6372

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
I	53	L-series	9.49	11.82	17.08	0.7
Pt	78	M-series	70.80	88.18	82.92	2.9
Total:			80.29	100.00	100.00	

Figure 3. Cyclic voltammograms of platinum and iodine.

Thus, there are three absorption maxima in the UV spectra of all formazans: 240-260 nm, 305-330 nm, and 380-420 nm. The absorption bands in the IR spectra are typical of the bonds C = N ($1400 - 1490 \text{ cm}^{-1}$), N-H ($3400 - 3300 \text{ cm}^{-1}$) and N = N ($1500-1480 \text{ cm}^{-1}$) formazan fragment. When working on formazans 37 percent formalin solution in the presence of perchloric acid, perchlorates triarylverdaziliya was produced in dioxane. UV spectra were used to confirm the structure of perchlorates. The UV spectra show absorption maxima at 250-260 nm and 370-420 nm, as well as a maloin intensity peak at 610-720 nm. The absorption bands in the IR spectra of salts are indicative of the bonds C = N ($1600-1590 \text{ cm}^{-1}$), N = N ($1480-1550 \text{ cm}^{-1}$) and the perchlorate anion ($1200 - 1250 \text{ cm}^{-1}$). Verdazil perchlorates are crystalline compounds that range in color from dark blue to dark brown, depending on the type of the substituent (Figure 3).

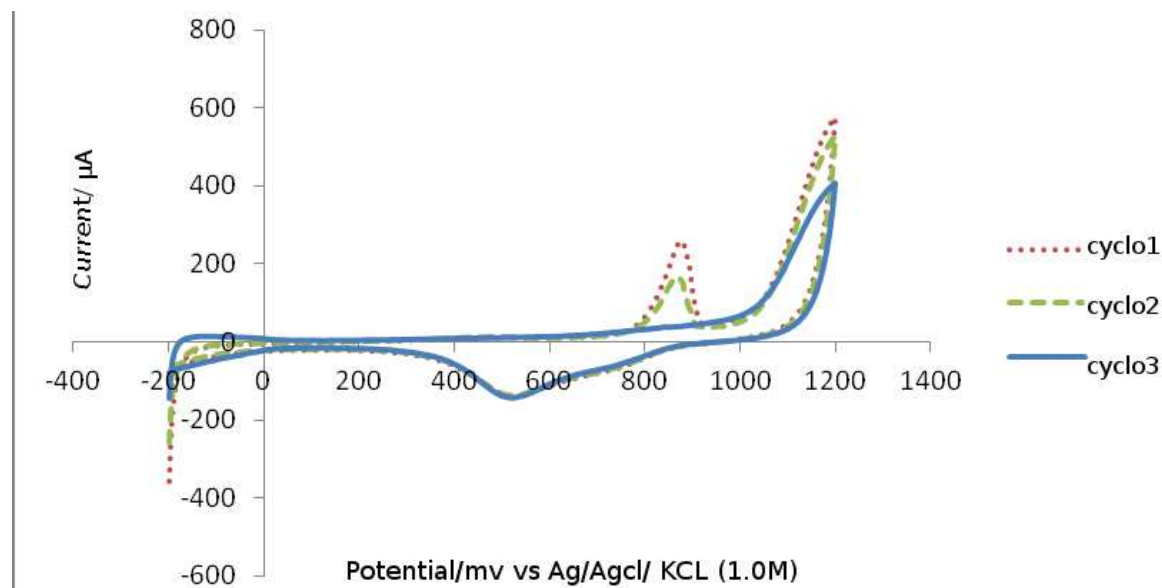


Figure 4. Cyclic voltammograms of 3,3'-(1,4-phenylene) perchlorate.

The absorption bands in the IR spectra of salts are indicative of the bonds C = N ($1700- 1800 \text{ cm}^{-1}$), N = N ($1800-1950 \text{ cm}^{-1}$) and the perchlorate anion ($1300- 1400 \text{ cm}^{-1}$). Verdazil perchlorates are crystalline compounds that range in color from dark blue to dark brown, depending on the type of the substituent. Previously, using triarylverdazyl radicals as an example, the DFT approach was utilized to investigate the effect of various substituents on the electron density distribution for border orbitals. The presence of donor substituents facilitates the oxidation of radicals to cations, whereas acceptor substituents inhibit the oxidation of the radical (Figure 4).

Without a doubt, the nature of the substituent will affect the electron density distribution and, therefore, the ability of verdazylum cations to be reduced to create the corresponding verdazyl radicals in the case of verdazylum perchlorates. We previously studied the electrochemical properties of triarylverdazilnyh radicals using the method of cyclic voltammetry (CV) and found that substituents in the 2-position tetrazinilnogo fragment affect the value of the radical oxidation potential as substituents and in the 6-position affect the radical reduction potentials values.

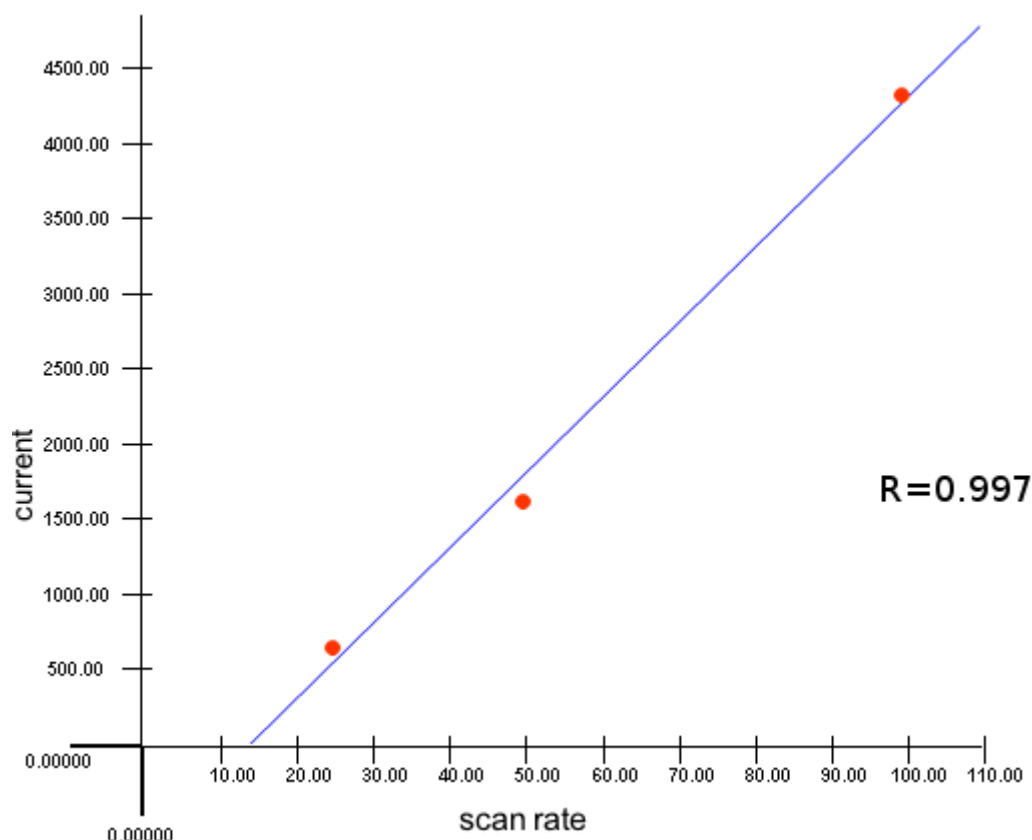


Figure 5. Linear relation ship of 3,3'- (1,4-phenylene) perchlorate.

As a result, the type of the substituents in tetrazine Eve's salts is thought to alter the ability to renew cation radicals. To test this idea and investigate the possibilityY and preparation of salts verdazilnyh. platinum was employed as a working electrode, platinum as an auxiliary electrode, and standard as a reference electrode. In desalt IME, whether vinyl CVA two -electron reduction peak in the range of -0.30 V to -1.00 V (Ag/AgCl/KCl), which we suppose is related to the recovery processes dictating first to the radical cation and then to the biradial. The type of the substituent in the vapor-position and an aromatic ring has an effect on the ability of verbals cations to recover, as shown in the Figure 5.

Cyclic voltammograms (CV) of chemicals (R: H, OMe and NO_2) are shown in Figure 5. CVs were recorded at a scanning rate of 200 mV/s for solutions in MeCN in the presence of a supporting electrolyte of 0.1 M Bu 4 NBF 4 (CVs are displayed with an offset relative to the Y axis). Cyclic voltammograms (CV) of chemicals (R: H, OMe and NO_2) are shown in Figure 5. CVs for solutions in MeCN in the presence of 0.1M Bu₄NBF background electrolyte were obtained at a scan rate of 200 mV/s.

As shown in the figure, under the influence of the methoxy group, the value of the first reduction potential decreased by 0.15 V and the second by 0.18 V, whereas under the influence of the nitro group, the first reduction potential increased by 0.18 V and the second by 0.10 V in comparison to unsubstituted derivatives.

4. Conclusions

Starting from ter eftalevogo aldehyde, a straightforward three-stage synthesis of 3,3'- (1,4-phenylene) perchlorate s was achieved. Their electrochemical characteristics were investigated using cyclic voltammetry. Curves in the cathode area are present on the two-electron reduction peak 1 in cyclic voltammetry, linked to sequential reduction th dication in a radical cation and further in radical. It was discovered that donor substituents R = Me, OMe accelerate verdazyl cation reduction, but acceptor substituents R: NO_2 , and COMe, hinder the process. Based on these findings, it was

hypothesized that the corresponding bis-verdazyl radicals would develop easily in the presence of donor substituents in the aromatic ring. As a result, verdazylum perchlorates can function as precursors of the equivalent bis-verdazyl radicals.

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