

Research Article

The Diorganoselenium and Selenides Compounds Electrochemistry

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The electrochemical behavior of Ar_2SeCl_2 , and Ar_2Se_2 , ($\text{Ar}:\text{CH}_3\text{OC}_6\text{H}_5$; $\text{C}_2\text{H}_5\text{OC}_6\text{H}_5$) in acetonitrile (AN) containing tetrabutylammonium tetrafluoroborat (TBAFB) as supporting electrolyte was studied on a stationary electrode (spe). In order to elucidate the electrode reactions linear potential scan, cyclic voltammetry and controlled-potential coulometry were employed using a platinum electrode. It is shown that Ar_2SeCl_2 and Ar_2Se_2 are reduced and oxidized to Ar_2Se , $\text{Ar}_2\text{Se}_2\text{Ar}_2$, Se, and $\text{Ar}_2\text{Se}(\text{BF}_4)_2$. It is generally accepted that as final electrochemical reduction products, the corresponding Ar_2Se , Ar_4Se_2 , and Se were formed. The disappearance of the diorganoselenium and selenide in the course of the coulometric experiments was validated by measuring the limiting current of the voltammetric waves at spe and UV spectrometry.

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1. INTRODUCTION

Organoselenium and organotellurium compounds are well known for their antimicrobial [1–4], anti-inflammatory [5, 6], and biocidal activities [7]. Organoselenium compounds have found applications as oxygen transfer reagents in organic [1], and organometallic synthesis [2], and as oxygen-donor ligands in main and transition metal complexes.

Organoselenium and organotellurium compounds [8–11] the electrochemical behavior of diphenyl and bis(p-anisyl) tellurium dichloride, bis(p-anisyl) telluride [4], diphenyl ditelluride [5], and aromatic diselenides-ditellurides and diphenyl selenide have been studied on the rate of rotation electrode, the mercury electrode, and the platinum grids in aprotic solvents (CH_2Cl_2 , DMF, CH_3CN) [6, 7].

There are a lot of publications concerning the electrochemistry of organotellurium compounds [12–23]. Aromatic selenides are relatively stable toward reduction [24]. The electrochemical behavior of the diaryl selenium dichlorides and diaryl selenides has not been studied on a stationary electrode yet. However, a comparative study of the electroreduction of bis(p-anisyl) and bis(p-ethoxyphenyl) selenium

dichloride (Ar_2SeCl_2), bis(p-anisyl), and bis(p-ethoxyphenyl) selenide (Ar_2Se_2) was carried out in acetonitrile (AN).

2. EXPERIMENTAL

2.1. Materials

In this work, electrochemical experiments were carried out by a three-electrode system. The organometallic selenium compounds were prepared according to the procedure described in the literature [25, 26]. The compounds studied were given in the elemental analysis data in Table 1. The amount of Carbon and Hydrogen were determined by the usual combustion method. The selenium was estimated according to the known reported method [27].

2.2. Procedure

In this work, a stock solution of 0.5 mM Ar_2SeCl_2 and supporting electrolyte (tetrabutylammonium tetrafluoroborat; TBAFB) in AN was prepared. AN was obtained from Aldrich Chemical Co. (HPLC grade) and was transferred with a syringe from the original container into the electrochemical

TABLE 1: The comparison of the identification data of selenium compounds. (Ph: C₆H₅) with literature.

Compound	From this study			Literature			Ref.
	Mp°C	C%	H%	Mp°C	C%	H%	
(CH ₃ OPh) ₂ SeCl ₂	163	46.58	3.95	163	46.18	3.88	[15]
(C ₂ H ₅ OPh) ₂ SeCl ₂	139-140	50.87	4.60	139	49.0	4.63	[15]
(CH ₃ OPh) ₂ Se ₂	56-58	57.18	4.82	56-57	57.35	4.81	[16]
(C ₂ H ₅ OPh) ₂ Se ₂	61-62	59.73	5.75	60-62	59.82	5.65	[16]

cell. The temperature for all experiments was 20±1°C. Because of the diaryl diselenides are sensitive to light; the electrolysis solutions were protected by an aluminum foil. Electrolysis experiments were carried out under nitrogen atmosphere.

The progress of electrolysis was monitored by recording UV-Vis spectral changes with applying a potential (E_{pc}) at peak values. For this purpose 20–25 ml of the electrolyzed solution was transferred into the quartz cell of the measurement container from the working compartment of the electrolysis cell and then corresponding spectra were recorded. The spectral changes were continuously monitored at different time intervals to detect the wave length region in which the UV-Vis absorption for intermediate products was generated. When the reduction peak (I_c) disappeared, electrolysis was stopped and the electrolyzed solution was transferred from cell into the measurement container. The products were further characterized by using ¹H NMR, FT-IR, and mass spectra.

The working and counter electrodes were made of platinum foil (4 cm²); the same electrode was used in cyclic voltammetry experiments. Controlled and the scanning potentials were supplied by potentiostat Wenking Model POS73 and recording was made by using a Rikadenk RW-11T X-Y recorder. Coulometric experiments were made in an improved three-compartment cell with high A/V ratio (electrode area/solution volume) and good geometry, enabling homogeneous potential distribution on the working electrode and thus fast and precise measurements of the products. The FT-IR spectra were recorded on a Matson-1000, FT-IR spectrophotometer using KBr pellets. Perkin-Elmer UV-1601PC spectrophotometer and mass spectra on JEOL JSM-6400 were used to record UV-Vis spectral changes during electro reduction process.

3. RESULTS AND DISCUSSION

The results of the voltammetric studies are shown in Figure 1. In concentrations up to 2 mM, three reduction waves appeared on the cyclic voltammograms, and these reactions which were linearly proportional to the Ar₂MCl₂ concentration were controlled by the diffusion rate of Ar₂MCl₂.

The first cathodic peak ($E_{pc} = -0.26$ V) and the first anodic peak ($E_{pa} = -0.32$ V) were found to be reversible with $\Delta E_p = 0.06$ V and the ratio of cathodic to anodic peak currents which corresponds to a single one-electron process ($I_{pc}/I_{pa} \approx 1$). The variation of the recorded values is reported

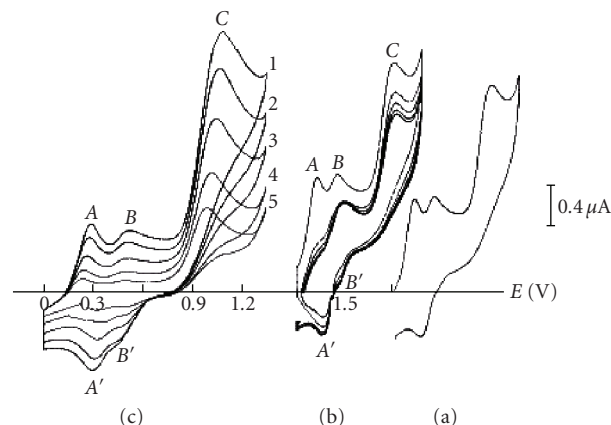


FIGURE 1: (a) Cyclic voltammogram of 0.004 M (C₂H₅OPh)₂SeCl₂; (b) cyclic voltammogram of 0.0035 M (CH₃OPh)₂SeCl₂; (c) rate of potential scans: (1) 50 mV/s, (2) 40 mV/s, (3) 30 mV/s, (4) 20 mV/s, and (5) 10 mV/s, conditions: 0.1 M TBABF₄ in CH₃CN.

TABLE 2: The first cathodic and anodic peak potential for the (CH₃OPh)₂SeCl₂ in AN solution containing 0.1 m Bu₄NBF₄ at 20°C.

Scan rate (mV/s)	E_{pc} (V)	E_{pa} (V)	ΔE_p (V)	I_{pc} (μA)	I_{pa} (μA)	I_{pc}/I_{pa}
10	-0.26	-0.32	0.06	0.15	0.16	0.94
20	-0.27	-0.33	0.06	0.25	0.27	0.93
30	-0.28	-0.34	0.06	0.45	0.49	0.92
40	-0.29	-0.35	0.06	0.65	0.71	0.92
50	-0.30	-0.36	0.06	0.85	0.93	0.91

in Table 2. Coulometric measurements displayed the number of electrons were shown in Figure 2. Involved in the electroreduction was determined by regression method of the current-time curve Figure 3. In fact, the approximation of time current is linear. R^2 is almost unit.

The working electrode potential was maintained about -1.1 V of the reduction peak. The value of n , calculated from the net charge passing through the solution was found to be 3.8 ± 0.2 in AN [28]. The ratio of currents corresponding to the first and second peaks, respectively, was in all cases close to 1 : 1, and the total amplitude of both waves always gave a current value corresponding to two-electron process.

The results of these experiments were interpreted according to the method of Nicholson and Shain [27]. It was found that the dependence of I_{pc}/C on the concentration (Figure 4) or potential scan rate (Figure 5) is similar to that suggested by Nicholson and Shain [29] and Liftman-Albeck for such ECEC reaction [30]. At high scan rates, the whole cathodic waves showed lower negative cathodic potential values (Table 2).

The chloride ions, formed during the reduction of the dichloride were adsorbed on the platinum electrode as reported [31]. Two electrons participate in the reduction of one molecule of dichloride and other two electrons in the reduction of the supporting electrolyte. The product of the two-electron reduction of selenium organometallic formed

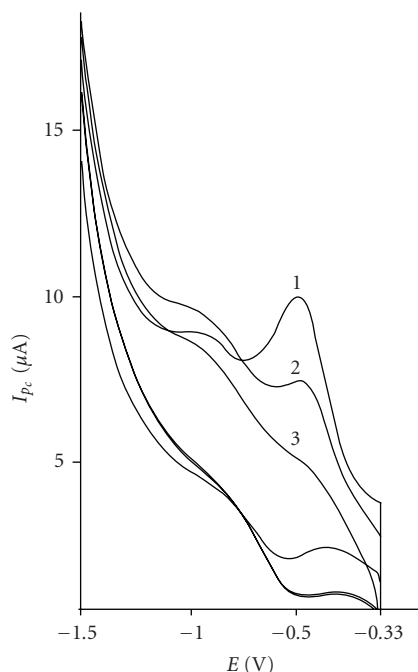


FIGURE 2: The cyclic voltammograms of AN containing 2 mM Ar_2SeCl_2 at stationary platinum electrode (2.5 cm^2): (1) at the beginning, (2) during, (3) and at the end of electrolysis. Supporting electrolyte: 0.1 M TBAFB, scan rate: 100 mV/s.

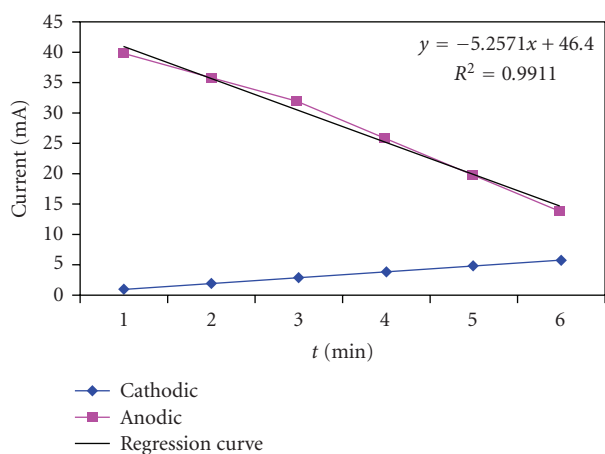
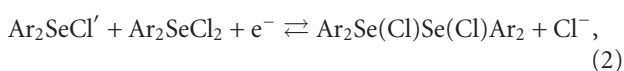
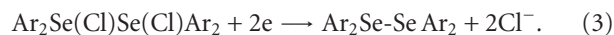


FIGURE 3: Changes with respect to time in the oxidation and reduction peaks of bis(pethoxyphenyl) selenium dichloride (Ar_2SeCl_2) electrolyzed at constant potential -1.1 V , cathodic and anodic current curves.

a dirtily-yellow solution in color, its state of aggregation in the solution has established and its formulation is $\text{Ar}_2\text{M}-\text{M}\text{Ar}_2$ [32]. Therefore, the electron reduction reactions of bis(p-anisylphenyl) or bis(p-ethoxyphenyl)selenium dichloride can be formulated by the following equations:



while at higher cathodic potentials:



Under the same conditions, three reduction peaks (A, B, and C) which are corresponding to (1), (2), and (3), respectively, and also two oxidation peaks (A' , B') were observed. This reaction is base-promoted (Hofmann elimination). The above mechanism is supported by the following experimental results.

- (i) In the far-IR region the reduction compounds exhibited $\mathbf{n}(\text{C}-\text{Se})$ $460\text{--}500 \text{ cm}^{-1}$, $\mathbf{n}(\text{Ar}-\text{O}-\text{CH}_3)$ $1150\text{--}1060 \text{ cm}^{-1}$, $\mathbf{n} \text{C}=\text{C}(\text{aromatic})$ $1660\text{--}1450 \text{ cm}^{-1}$, respectively. No signal were recorded at $\mathbf{n}_s(\text{Se}-\text{Cl})$ $275\text{--}245 \text{ cm}^{-1}$ and $\mathbf{n}_{as}(\text{Se}-\text{Cl})$ $245\text{--}255 \text{ cm}^{-1}$. ^1H NMR spectra of the reduction compounds showed various signals at wave lengths of δ 6.7–6.9 ppm (aromatic hydrogen) and at δ 3.6–3.8 (alkyl hydrogen). Mass spectrum; showed m/e 386 [$(\text{C}_2\text{H}_5\text{O})_2\text{C}_6\text{H}_4\text{SeCl}_2+$]; 149, 148, 147 (SeCl_2+); 280–281 [$(\text{C}_2\text{H}_5\text{O})_2\text{C}_6\text{H}_4\text{SeCl}+$]; 166–168 ($\text{C}_2\text{H}_5\text{OC}_6\text{H}_4+$); 77 (C_6H_4+).
- (ii) The obtained radicals (Ar_2SeCl ; Ar_2Se) (in the first and second step of the reduction of diaryl selenium dichloride) were short lived. We are not able to study.
- (iii) The chloride ions formed during the reduction of the bis aryl selenium dichloride were adsorbed on the electrode as shown in Figure 1(c) [22]. The slope of $\log I_p$ versus $\log \nu$ for the first value of the reduction peak of Ar_2SeCl_2 (a in Figure 5) was obtained as 0.8. However, this value was decreased because of adsorption.
- (iv) It was found that the dependence of the value I_{pc}/I_{pa} both for the peaks of the selenium dichloride (a and b in Figure 5) on the rate of potential scan (V) and the dependence of I_{pc} on $\sqrt{\nu}$ are similar to those suggested by Nicholson and Shain for such reactions.

The cyclic voltammograms of $(\text{C}_2\text{H}_5\text{OPh})_2\text{Se}_2$ are shown in Figure 6. The initial positive scans show a single anodic peak C at approximately -0.52 V and three cathodic peaks (A, A' , and B) are also observed at potentials of -0.15 V , 0.30 V , and 0.80 V (Figure 8). Peak A is most probably due to the reduction of selenium deposited on the working electrode and peak B (0.80 V in Figure 6 and 1.5 V in Figure 8) is most probably due to the reduction of Bu_4N^+ ion.

Metallic selenium had poor adherence on the electrode surface. This conclusion was supported by the fact, as shown in (Figure 6), the continuous cycling between -0.80 V and 1.28 V leads to a resulting increase in mass of the product indicating that most of the deposited $\text{Se}(0)$ is not reoxidized. The small amplitude of peak values (A, A' in Figure 6) confirms this indication. Because of this reason, the electrode reactions of diselenides are followed by catalytic reactions of supporting electrolyte and chemical reactions in bulk of the solutions. After the first cyclic voltammogram, the amplitude of the peaks (A, A') were decreased and then remained stable.

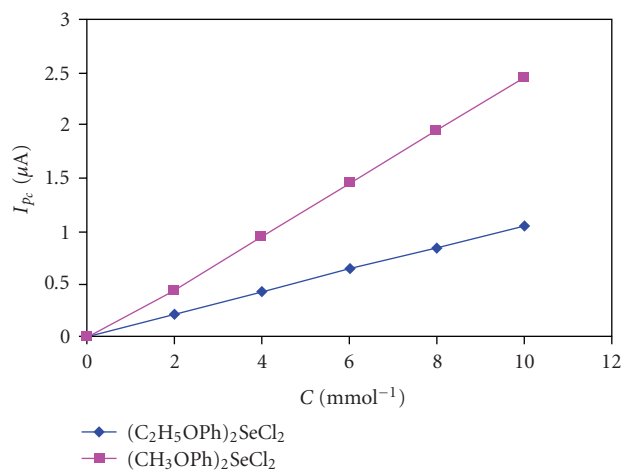


FIGURE 4: The dependence of the peak currents of the concentrations, conditions: 0.1 M TBABF₄ in CH₃CN.

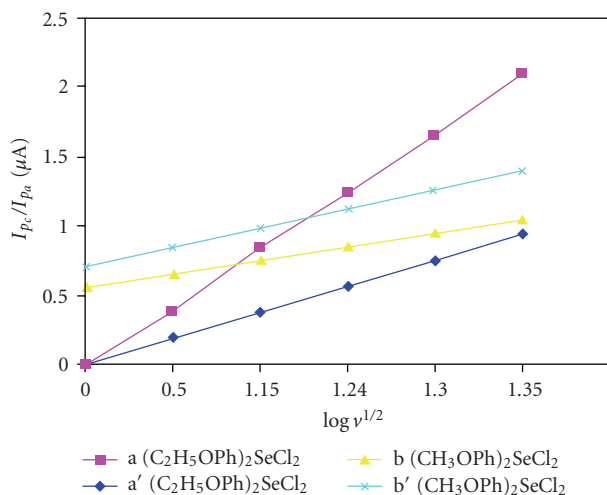


FIGURE 5: The dependence of I_{pc} and I_{pc}/I_{pa} in CV, conditions: 0.1 M TBABF₄ in CH₃CN.

This result shows that electrode-solution interface reactions are slow.

The dependence of the peak currents of the waves on the scan rate is shown in Figure 7. The limiting currents of the waves of $(\text{C}_2\text{H}_5\text{OPh})_2\text{Se}_2$ (a in Figure 7) and the waves of $(\text{C}_2\text{H}_5\text{OPh})_2\text{Se}_2$ at spe are linearly proportional to the concentration of diselenides (b in Figure 7). The I_c of the reduction peak Ar_2Se_2 is also linear with respect to the scan rate $v^{1/2}$ ($\text{mV}^{1/2}\text{s}^{-1/2}$) (c and d in Figure 7), since the reduction process is diffusion controlled. During constant potential electrolysis at plateau potentials of the cathodic wave of $(\text{C}_2\text{H}_5\text{OPh})_2\text{Se}_2$ the limit current of the wave takes place as shown e in Figure 7). This is due to the formation of selenide in the reduction of the diselenide which catalyses the reduction of the tetrabutylammonium cation of the supporting electrolyte. The metallic selenium produced as a result of this reaction is not readily oxidized.

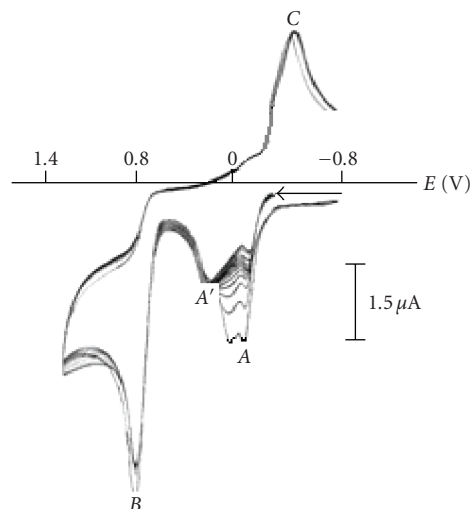


FIGURE 6: Multisweep-cyclic voltammograms of 0.0025 M $(\text{C}_2\text{H}_5\text{OPh})_2\text{Se}_2$, conditions: 0.1 M TBABF₄ in CH₃CN. Scan rate 100 mV/s.

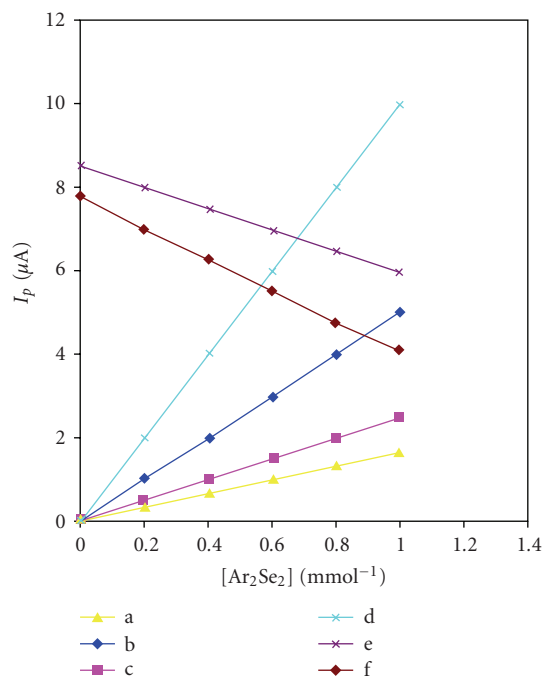


FIGURE 7: The dependence of peak currents (a), (b) on the concentration of $(\text{C}_2\text{H}_5\text{OPh})_2\text{Se}_2$; (c), (d) on the rate of potential scan; (e) on the amount of cathodic charge passed at (-0.52 V); (f) of anodic charge passed at (1.15 V). Concentration of Ar_2Se_2 0.0035 M (f), 0.003 M, 0.1 M TBABF₄.

After reaction, the electrode was cleaned from the metallic selenium, the first cyclic voltammogram is given in Figure 8. The role of electrode material in the reduction pathway appeared to be important, especially in the case of the stationary electrode. These results point to a different reduction mechanism or to a remarkable shift of the

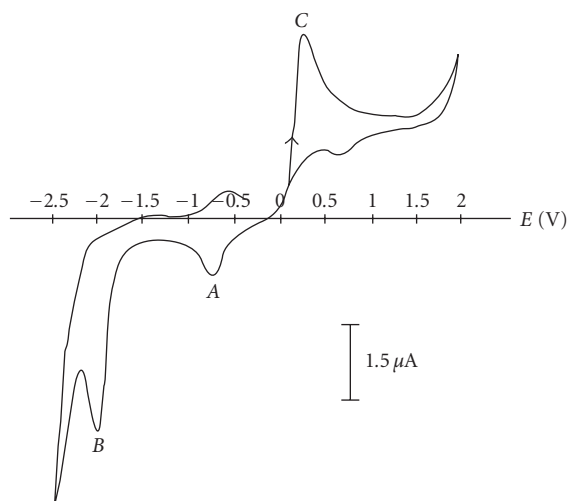


FIGURE 8: Cyclic voltammogram of 0.004 M $(\text{C}_2\text{H}_5\text{OPh})_2\text{Se}_2$, conditions: 0.1 M TBAFB in CH_3CN . Potential scan rate: 100 mV/s.

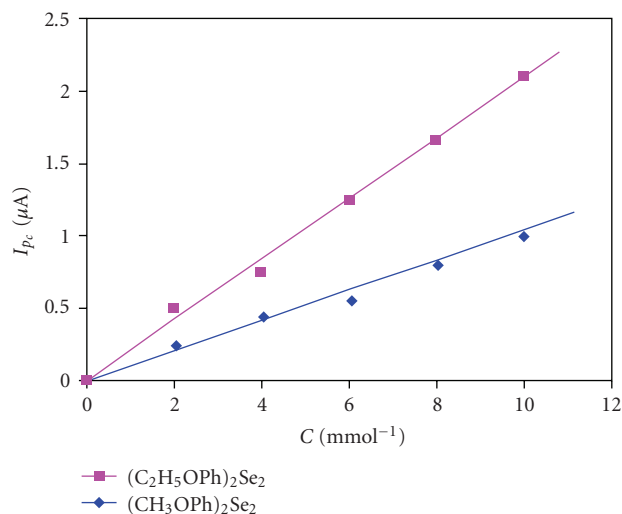
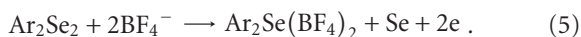
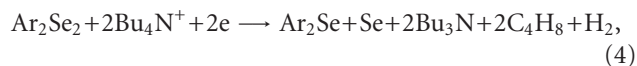


FIGURE 9: The dependence of the limiting currents of the waves on the concentrations.

redox potential of the intermediate compounds Ar_2Se and Ar_2SeBF_4 , to lower negative values (*B* in Figure 8).

The reaction mechanism is as follows:



The above mechanisms were supported by the following experimental results:

- (i) in the oxidation and reduction of Ar_2Se_2 , always metallic selenium separated at the electrode surface (*A* in Figure 6);
- (ii) the first anodic peak of $\text{Ar}_2\text{Se BF}_4^-$ (0.33 V in CH_3CN) (*A* in Figure 6) and the second anodic peak

of $\text{Ar}_2\text{Se}(\text{BF}_4)_2$ (0.80 V in CH_3CN) were observed only in the presence of BF_4^- ions in the solution (*B* in Figure 8);

- (iii) the peak currents of the oxidation peak of the Ar_2Se_2 and that of the reduction peak of the selenides were controlled by diffusion rate of the corresponding organoselenium compound (Figure 9);
- (iv) support for the assumption that anodic and cathodic reactions are charge-transfer reactions followed by irreversible equation (4) electrochemical reactions and equation (5) chemical reactions is given by the results of the cyclic voltammetry experiments but we were not able to analyze these reactions;
- (v) the ratio between the diffusion currents of the two peaks is 1 : 2 with a total consumption of two electrons;
- (vi) experiments have shown that the catalytic effect of the products Se , Ar_2Se , and $\text{Ar}_2\text{Se}(\text{BF}_4)_2$ that were formed on the interface between the Pt electrode and the electrolyte. As the thickness of metallic Se increases, mass transfer to the electrode interface decreases.

4. CONCLUSIONS

The electrochemical behavior of Ar_2SeCl_2 and Ar_2Se_2 on the stationary platinum electrode was investigated by cyclic voltammetry and controlled-potential coulometry measurements. During the reduction of Ar_2SeCl_2 at 0.52 V, a small amount of solid was precipitated at a total Faradic efficiency of a few percent and was identified as a mixture of $\text{Ar}_2\text{Se}(\text{BF}_4)_2$ and $\text{Ar}_2\text{Se}(\text{Cl})\text{Se}(\text{Cl})\text{Ar}_2$. The solutions contain $\text{Ar}_2\text{Se}-\text{SeAr}_2$ in measurable amounts at higher cathodic potentials such as 1.15 V.

Metallic selenium was not separated at the electrode surface. Metallic selenium separated at the electrode surface while using Ar_2Se_2 in the nonaqueous solution during both reduction and oxidation reactions.

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