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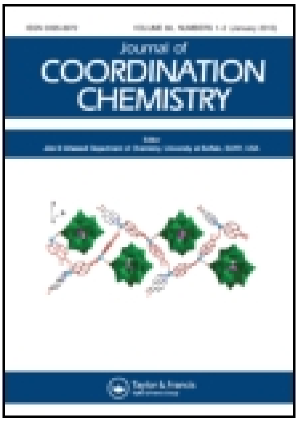

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Investigation of ionizable nano-baskets of calix[4]-1,2-crown-3 by differential pulse voltammetry

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Nano-baskets of proton di-ionizable *p-tert*-butylcalix[4]arene-1,2-crown-3 in cone and 1,2-alternate conformations and the *p-tert*-butylcalix[4]arene-1,2-thiacrown-3 in cone conformation were synthesized and their binding abilities towards alkali and alkaline earth metals as well as some lanthanides were studied using differential pulse voltammetry. The novelty of this study was investigation of the macrocyclic complexes by voltammetric behaviors of two acidic moieties in each scaffold. Their voltammetric behaviors were closely related to the complex formation by entrapment of cation into crown ether cavity and ion-dipole interaction between cation and acidic moieties in calixcrowns. The results revealed the selective changes in voltammetric behavior of synthesized scaffolds toward the cations. Moreover, the sulfur in the crown ring changed the binding ability of scaffold to more cations. Furthermore, the position of crown-3 ether in 1,2-alternate instead of cone enhanced the domain of binding ability to more cations.

Keywords: Nano-basket; Voltammetry; Calixcrown; Ionizable moieties

1. Introduction

Nano-baskets of calixarenes and calixcrowns are a versatile class of macrocycles, which have been subject to extensive research in development of many extractants, transporters and stationary phases (using gas chromatograph, Teif Gostar Faraz Co.) over the past four decades [1–5]. Functionalization of calix[4]arenes at both the upper rim and the lower rim has been extensively studied. Attaching donors to the lower rim of a calix[4]arene can improve the binding strength of the parent calixarene dramatically. Two main groups of lower-rim functionalized calix[4]arenes are calix[4]arene podands and calixarene-crown ethers [6, 7]. Distal hydroxyl groups can be connected to give 1,3-bridged calix[4]crowns, while connection between proximal hydroxyl groups leads to 1,2-bridged calix[4]crowns. The 1,3-bridged calix[4]crowns exhibit high binding affinity and selectivity toward alkali and alkaline earth metal cations [8], while research on 1,2-bridged calix[4]crowns lags far behind. Combining crown ethers with calix[4]arenes increases the cation binding ability of the parent calixarenes, and control of the selectivity is obtained through modulation of the crown ether size [9–12]. Attachment of

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Table 1. List of calixarenes used in the voltammetric techniques.

Concept of study	Type of calixarene	Analyte	References
Differential pulse anodic stripping voltammetry	*p*-isopropylcalix[6]arene	silver	[13]
Stripping voltammetry	Langmuir–Blodgett film of a calix[4]arene derivative	silver	[14]
Stripping voltammetry	Langmuir–Blodgett film of allylcalix[4]arene	cadmium	[15]
Anodic stripping voltammetry	Langmuir–Blodgett film of *p*-allylcalix[4]arene	cadmium	[16]
Anodic stripping voltammetry	Langmuir–Blodgett film of *p*-allylcalix[4]arene	mercury	[17]
Anodic stripping voltammetry	4-tert-butyl-1 -(ethoxycarbonyl-methoxy)thia-calix[4]arene	mercury	[18]
Differential pulse anodic stripping voltammetry	Langmuir–Blodgett film of allylcalix[4]arene	copper	[19]
Adsorptive square wave voltammetry	tetrabenzyl ether calix[4]arene	adduct formation of 2-furaldehyde	[20]
Cyclic voltammetry	calix[4]arene derivative	dopamine	[21]
Adsorptive stripping voltammetry	*p*-*tert*-butyl-calix[6]arene	folic acid	[22]
Voltammetric methods	calix[4]arene crown-4 ether	norepinephrine	[23]
Cyclic voltammetry	calix[4]arene crown-4 ether	dopamine	[24]
Cyclic and differential pulse voltammetry	*p*-tetra-butyl calix[6]arene-L-Histidine	epinephrine and serotonin	[25]
Voltammetric methods	calixcrownchips	antigens and antibodies	[26]
Voltammetric methods	c-methylcalix[4]resorcenarene and calix[8]arene	nicotinamide	[27]
Conductometric chemosensor	c-benzylresorcinolcalixarene	amines and amino acids	[28]
Electrochemical impedance spectroscopy	calix[4]arene crown-4 ether film	norepinephrine	[29]
Conductivity gas sensor	–	methanol, ethanol and propanol	[30]
Capacitive sensing sensor	–	amino acids	[31]
Electrochemical flow injection analysis	resorcin[4]arene	carbofuran	[32]
Faradic electrochemical impedance spectroscopy	carboxylic-calixarene, benzyl-calixarene, and long chain sulphonated-calixarene	arginine and lysine	[33]

proton-ionizable groups to calixcrowns can further improve their extraction properties because the ionized group not only participates in metal ion coordination, but also eliminates the need to transfer aqueous phase anions into the organic phase. Some voltammetric techniques using calixarenes are tabulated in table 1.

In this work, three proton-ionizable calixcrowns with different proton-ionizable moieties were synthesized including cone *p*-*tert*-butylcalix[4]arene-1,2-crown-3 diacid, 1,2-alternate *p*-*tert*-butylcalix[4]arene-1,2-crown-3 diacid, and cone *p*-*tert*-butylcalix[4]-arene-1,2-thiacrown-3 diacid. The voltammetric behavior of the synthesized scaffolds was determined using some alkali, alkaline earth, transition metals, and lanthanides.

2. Synthesis procedures

Reagents were obtained from commercial suppliers and used directly, unless otherwise noted. Acetonitrile (MeCN) was dried over CaH_2 and distilled immediately before use.

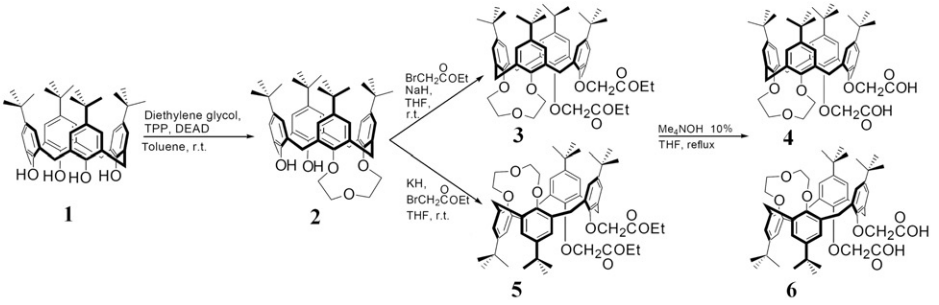

Figure 1. Synthesis of *p*-*tert*-butylcalix[4]arene-1,2-crown-3 di-carboxylic acids (**4** and **6**).

Tetrahydrofuran (THF) was dried over sodium with benzophenone as an indicator and distilled just before use. Cs_2CO_3 was activated by heating at 150°C overnight under high vacuum and stored in a desiccator. Melting points were determined with a Mel-Temp melting point apparatus. Infrared (IR) spectra were recorded with a Perkin-Elmer Model 1600 FT-IR spectrometer as deposits from CH_2Cl_2 solution on NaCl plates. The ^1H and ^{13}C NMR spectra were recorded with a Varian Unity INOVA 500 MHz FT-NMR (^1H 500 MHz and ^{13}C 126 MHz) spectrometer in $CDCl_3$ with Me_4Si as internal standard unless mentioned otherwise. Chemical shifts (δ) are given in ppm downfield from TMS and coupling constants (J) are in Hz.

The commonly employed method involves the reaction of *p*-*tert*-butylcalix[4]arene (**1**) and diethylene glycol ditosylate in the presence of strong base failed to give *p*-*tert*-butylcalix[4]arene-1,2-crown-3 (**2**). Variations of the base, as well as elevating reaction temperature, were performed, but no detectable **2** was found. Another entirely different method was utilized for the synthesis of **2** (figure 1). Under Mitsunobu reaction conditions, **1** was treated with triphenylphosphine (TPP) and diethylazodicarboxylate (DEAD) and reacted with diethylene glycol to form **2**. The reaction was quenched after 0.5 h and gave a 54% yield. Compared with the commonly used methods, the Mitsunobu reaction had a much shorter reaction time with a reasonable yield.

Moreover, conversion of the diethylene glycol into diethylene glycol ditosylate was not necessary. *p*-*Tert*-butylcalix[4]arene-1,2-crown-3 (**2**) was treated with sodium hydride in THF and reacted with ethyl bromoacetate at room temperature for 2 days to form *p*-*tert*-butylcalix[4]arene-1,2-crown-3 diester (**3**) in the cone conformation in 61% yield. The synthesis procedure is shown in figure 1, schematically. The resulting diester **3** was hydrolyzed with aqueous tetramethylammonium hydroxide in THF under reflux to give diacid **4** in 88% yield. *p*-*Tert*-butylcalix[4]arene-1,2-crown-3 (**2**) was treated with potassium hydride in THF and reacted with ethyl bromoacetate at room temperature for 3 days to form *p*-*tert*-butylcalix[4]arene-1,2-crown-3 diester **5** in the 1,2-alternate conformation in 48% yield. The synthesis is depicted in figure 1. The resulting diester **5** was hydrolyzed with aqueous tetramethylammonium hydroxide in THF under reflux to give diacid **6** in 96% yield.

The *p*-*tert*-butylcalix[4]arene-1,2-thiacrown-3 (**7**) was synthesized following the method used for making **2** (figure 2). Under Mitsunobu reaction conditions, *p*-*tert*-butylcalix[4]arene (**1**) was treated with triphenylphosphine and diethylazodicarboxylate and reacted with 2,2′-thiodiethanol to form **7** in 29% yield. Details of the

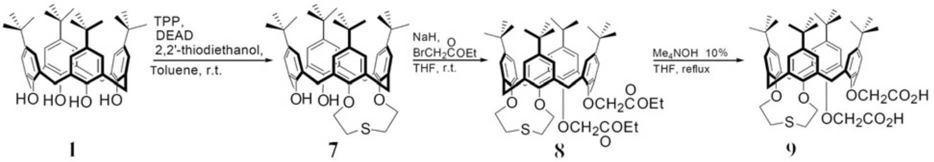

Figure 2. Synthesis of *p-tert*-butylcalix[4]arene-1,2-thiacrown-3 di-carboxylic acid (**9**).

preparation and characterization of all of the calixarenes are provided in "Supplementary material."

3. Experimental

Voltammetric measurements were obtained with a Model 660 electrochemical workstation (CH Instruments, Austin, TX, USA) with a three-electrode cell including a Pt wire counter electrode, a glassy carbon working electrode and a Ag/Ag$^+$ (0.1 mol L^{-1}) reference electrode, separated from the solution by a plug. The surface of the glassy carbon electrode was polished using 0.3 µm alumina (Buehler, Lake Bluff, MN) and residual alumina particles were thoroughly removed by placing the working electrode in an ultrasonic cleaner for 20 min and was dried and washed with pure acetonitrile. Calixcrowns 0.1 mmol L^{-1} and stock solution of metal perchlorate salts with various concentrations were prepared using acetonitrile. Tetra-*n*-butylammonium hexafluorohphosphate (TBAPF6 0.1 mol L^{-1}) was used as supporting electrolyte.

4. Results and discussion

The complexation of 0.1 mmol L^{-1} **4**, **6**, and **9** towards various cations (including alkali, alkaline earth, transition metals, and lanthanides) were investigated by cyclic voltammetry (CV) and differential pulse voltammetry (DPV) at glassy carbon electrode in 0.1 mol L^{-1} TBAPF$_6$/acetonitrile solution. According to figure 3, CVs of host macrocycles show almost the same voltammetric behavior, which is no reduction peak and two anodic peaks at 0.8 and 1.4 V in scaffolds **4** and **6**, and 0.9 and 1.3 V in scaffold **9**.

This phenomenon is related to the redox behavior of carboxylic acids and intramolecular H-bonding (between two of carboxylic acid groups) in **4**, **6**, and **9**. Although oxidation of carboxylic acid exhibits one oxidation peak with two electron and proton transfer in organic solution, formation of intramolecular H-bonding between two carboxylic acids in **4**, **6**, and **9** makes one proton transfer easier and the other more difficult, leading to oxidation peaks at a more positive potential and a less positive potential, respectively. Oxidation behavior of two carboxylic acid groups in **4**, **6**, and **9** implies that H-bonding to carboxylic acid moiety is influenced by binding to cations because the carboxylic acids are located around the crown ether.

In the rest of the experiments, DPV was used instead of CV to get a better resolution of waves in the same condition. The voltammetric behaviors of carboxylic acids in host

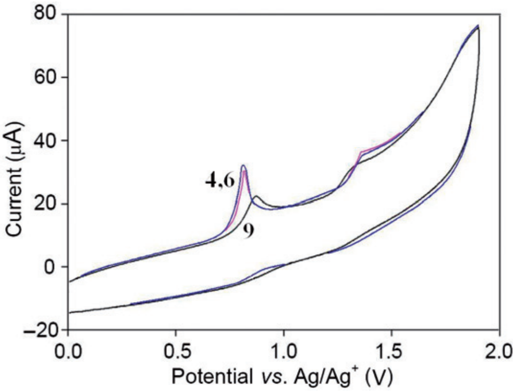

Figure 3. Cyclic voltammograms of 0.1 mmol L^{-1} **4**, **6**, and **9** at glassy carbon working electrode.

scaffolds were compared with different cations in DPV to investigate the binding properties of **4**, **6**, and **9**. A constant volume of 10 μL per injection of the cation in 0.1 mol L^{-1} $TBAPF_6$ was added into the cell to make 0.1–3.0 equivalent of cation in solution. DPVs were recorded after adding stoichiometric equivalent of cations successively to the respective electrochemical solution. DPVs of **4**, **6**, and **9** depict almost the same voltammetric behaviors with two anodic peaks, as indicated in CV. Figure 4 presents the differential pulse voltammograms of 0.1 mmol L^{-1} **4**, **6**, and **9** in the presence of 0.1 mmol L^{-1} alkali, alkaline earth, transition metals, and lanthanides in CH_3CN. Obviously, the wave-shape, current, and peak potentials of the traces are reproducible.

As depicted in the left column of figure 4, **4** in the presence of Na^+ and Pb^{2+} shows significant voltammetric changes of DPV in both the peak current and the potential. According to the middle column of figure 4, by addition of one equivalent of alkali, alkaline earth, transition metals, and lanthanides to a solution containing macrocycle **6**, no significant change was observed in the peak current or potential except for Pb^{2+}. Based upon the right column of figure 4, addition of one equivalent of K^+, Na^+, Ba^{2+}, and Pb^{2+} to **9** caused large decrease in anodic peak currents or even disappearance of the original second anodic peak. This is due to the electrostatic interaction between **9** and the cations, leading to electrostatic perturbation of intramolecular H-bonding by encapsulation of cations into crown ether and two carboxylic acid groups.

Regarding the role of binding site in the complex, the crown ether groups of **4** and **9** act mainly as landing sites for cations, while the carboxylic acid groups of **4**, **6**, and **9** constitute redox active centers as well as landing sites for binding. Figure 5 demonstrates metal ligation by either the crown ether groups or the carboxylic acid moieties of **4**, **6**, and **9**. The inclusion of divalent metal ions by the crown loop and hydroxyl groups of carboxylic acids may cause deprotonation of carboxylic acids [34, 35], which makes it difficult to oxidize carboxylic ion to carboxy radical in the oxidation process. The reason is attributed to the repulsion between the carboxy radical and the positive cations.

The peak height changes of the first oxidation potential of one equivalent of **4**, **6**, and **9** in the presence of one equivalent of different cations are tabulated in table 2. The bold entries depict the most binding abilities. The orders of current decreasing in the first

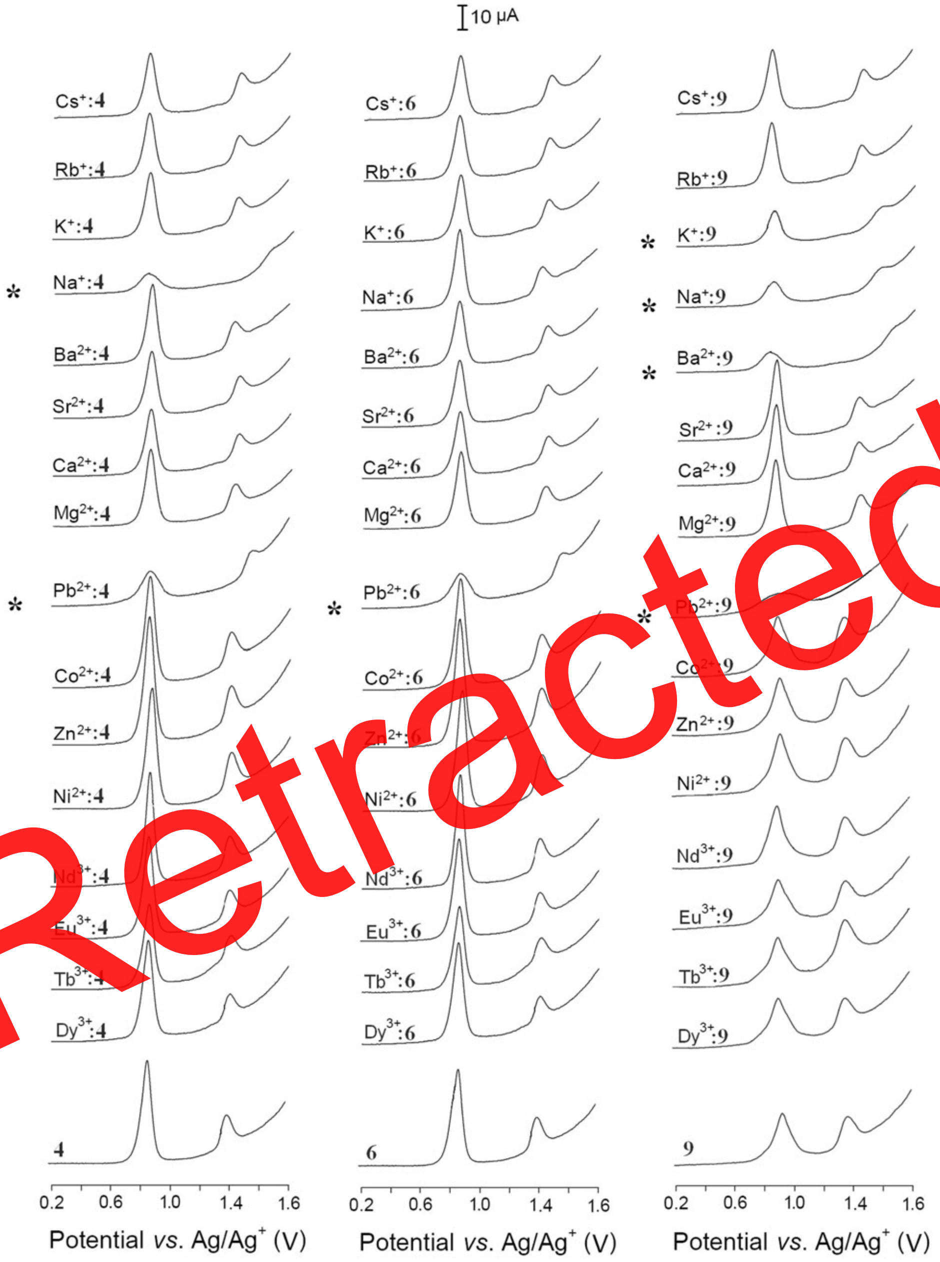

Figure 4. Differential pulse voltammograms of 0.1 mmol L^{-1} **4**, **6**, and **9** (left to right) in the presence of 0.1 mmol L^{-1} alkali, alkaline earth, transition metals, and lanthanides in CH_3CN.

peak of **4** and **9** are $Pb^{2+} < Na^{+}$ and $K^{+} < Na^{+} < Ba^{2+} < Pb^{2+}$, respectively, in accord with the order of potential shifts ($\Delta Ep_2 = Ep_2{*} - Ep_2$), which are tabulated in table 3. These indicate that the binding tendencies of **4** and **9** towards the above-mentioned cations follow the order $Pb^{2+} < Na^{+}$ and $K^{+} < Na^{+} < Ba^{2+} < Pb^{2+}$, respectively.

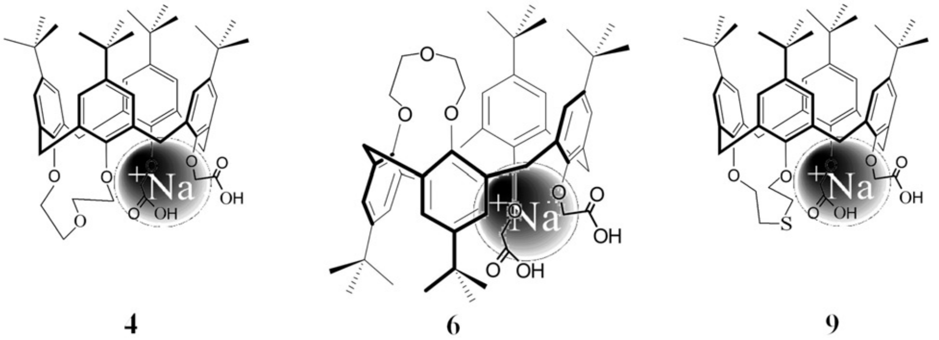

Figure 5. Graphical illustration of the metal ligation by either the crown ether groups or the carboxylic acid moieties of **4**, **6**, and **9**.

Table 2. Changes of current ratio in the first anodic peak for complexation of 0.1 mmol L^{-1} scaffolds **4**, **6**, and **9** with 0.1 mmol L^{-1} solutions of different metal perchlorate salts in CH_3CN.

Macrocycle	Cs^+	Rb^+	K^+	Na^+	Ba^{2+}	Sr^{2+}	Ca^{2+}	Mg^{2+}	Pb^{2+}	Co^{2+}	Zn^{2+}	Ni^{2+}	Nd^{3+}	Eu^{3+}	Tb^{3+}	Dy^{3+}
4	0.89	0.92	0.94	**0.44**	0.98	0.96	0.95	0.99	**0.48**	0.96	0.99	0.94	0.98	0.98	0.99	0.99
6	0.88	0.90	0.88	0.87	0.84	0.94	0.96	0.99	**0.50**	0.98	0.99	0.98	0.99	0.98	0.99	0.99
9	0.92	0.92	**0.64**	**0.46**	**0.38**	0.89	0.90	0.96	**0.32**	0.88	0.86	0.92	0.94	0.95	0.98	0.98

Table 3. Differences of peak potentials (mV) between free and complexed macrocycle, which were determined by differential pulse voltammetry.

Macrocycle	Cs^+	Rb^+	K^+	Na^+	Ba^{2+}	Sr^{2+}	Ca^{2+}	Mg^{2+}	Pb^{2+}	Co^{2+}	Zn^{2+}	Ni^{2+}	Nd^{3+}	Eu^{3+}	Tb^{3+}	Dy^{3+}
4	15	13	10	**186**	9	6	8	2	**174**	5	1	11	2	5	3	2
6	12	16	7	4	23	5	12	2	**168**	6	3	4	0	3	2	2
9	5	9	**147**	**178**	**200**	18	12	6	**218**	14	15	9	3	1	0	4

Complexion behavior of **4**, **6**, and **9** scaffolds with increasing amounts of corresponding cations was continued. Figure 6 depicts the effect of concentration of corresponding cations to **4**, **6**, and **9** scaffolds. With increasing the amounts of cations, the anodic peaks of **4** at 0.8 V and 1.4 V decreased. The peak current at 0.8 V decreased quantitatively by increasing the concentration of Pb^{2+} and Na^+, and gradually reached the minimum value at one equivalent.

5. Conclusions

The voltammetric behavior of calix[4]crowns **4** and **6** and calix[4]thiacrown **9** and their binding abilities toward alkali, alkaline earth, transition metals, and lanthanides were examined by differential pulse voltammetry. Those compounds showed a selective

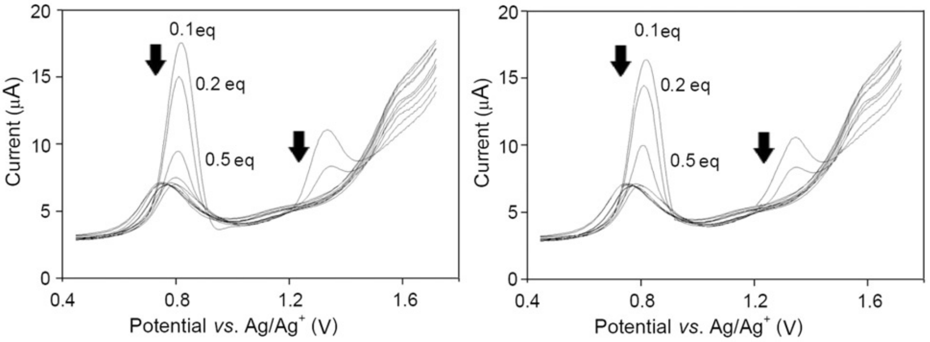

Figure 6. Differential pulse voltammograms of 0.1 mmol L^{-1} **4** with Na$^+$ (left) and Pb^{2+} (right) in CH$_3$CN.

change in voltammetric behavior toward the Na$^+$ and Pb^{2+} (for **4**), Pb^{2+} (for **6**), and K$^+$, Na$^+$, Ba$^+$, Pb^{2+} (for **9**) in CH$_3$CN. This is mainly due to the presence of crown (thia-) ether group and two proximal carboxylic acid moieties in the calix[4]arene framework. Comparing scaffolds **4** and **6** (containing crown-3 ether groups) with scaffold **9** (containing thiacrown-3 ether groups) revealed that sulfur in the crown ring changes the binding ability of scaffold to other cations. Comparing scaffold **4** (crown-3 on cone conformation) with **6** (crown-3 on 1,2-alternate conformation) showed that the domain of binding ability of **4** is more than **6**.

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