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Electrochemical Study of Structural Effects in Complexation of Nano-baskets: Calix[4]-1,2-crown-3, crown-4, -crown-5, -crown-6

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RETRAC ΈD 'HU) RE I 'R A (STUDY OF BINDING NANO-BASKETS BY VOLTAMMETRY RETRACTED RETRACT 1093 ED RETRAG ΕI 'R E \mathbf{RE} -Th R R REPR AT -71 A(A R HC OSOHO RET R HCO2CH2O ΕD RÉTRÀ **DORET** TIFI RE ГED F. ΓE. RETRA A RK R HCO2CH20 ó HCO2CH20 HCO2CH2O ò ó REACHO RECORD RE HCOROHAC RA HCO2CH2D-Tel Q **3**³RET RETRAC PEI RETRAC A2C' ΓFΙ 'R \mathbf{RE} ΈD TЕ FIG. 1 RThe chemical structure of eight calificrown seaffolds were synthesized and studied RETRA ₽IIRA RET Att **FK**E **KETR** ̈́R L'RA EI ЕI A(Diethylene glycol, TPP, DEAD Toluene, 7J. Maf. Ts(OCH;CH5)mOTS Weight State St NaH HE D R C2H (THF refus, 24 h) RCO,CH-0 dъ R n <u>Yield</u> 1 61% 2 81% 3 84% 4 92% R(6-9) Yield 88% 91% 97% 98% R10-13 1-20 RA THE DAY RETR 5 R ED Å 3 66% 42% Dietinylene Biycol, TPP, DEAP RFJ FD BrCH₂CO₂C₂H₅ TED RETR RE R TPP, DEAD toluene.r.t tor.n=1. m(m=3.5), DMF 10% Me4N^{*}OH^(aq) THF reflux, 24 h RAU Ш ΕI A нсосно \mathbb{R} CHICO, CH.O Ś **RETR**Å ⁽¹⁵⁻¹⁸⁾ ED 22% RE 98% 97% R⁽¹⁹⁼²⁰⁾ 98% 96% R 134 A(ED 569 639 cone *p-tert-*butyl-23,26-til(carboxymethoxy)calix[4]arene-1,2-ETRACTEI RETR Έ retra-n-butylammonium hexa-fluorolphosphate (TBAPF6) was RE'IN ΕL used as supporting electrolyte. ΈD R A RE RETAR R K KA(ΈD E. ⊣Ľ RESULTS AND DISCUSSION REFIRACTED RETRACTED The complexation of 0.1 mM compounds 10–13 and 23–26 **RET R**E' А TED EI ED RET toward various cations (including alkali, alkaline earth, transi-tion metals, and lanthanides) were investigated by cyclic voltam-RETR K⁄A(10-13 carbon electrode in 0.1 M PBAPF₆/actionitrite solution. Ac-RETR RET НI 'R ΗÌ same voltammetric behavior, which is no reduction peak and TED RETR two anodic peaks at 0.8 and 1.4 V in scaffolds 23-26, and 0.9 and 1.3 V in scaffold 10-13, respectively. etential vsr AgraditvR A RE 'R A(FIG. 3. Cyclic voltammograms of 0.1 mM **10–13** and **23–26** at glassy carbon This phenomenon is related to the redox behavior of car-Roxylic acids and intramolecular H-bonding (between two of) working electrode. ΗÏ carboxylic acid groups) in 10–13 and 23–26. RETRACTED RETRA Although the R Ε'I A ЕI RF -Ŭ I) RETRA **RA** RETRA RETRAC TEI Έ ΈD ΈI RETRACTED RETRACTED RETRACTED RETRACTED <u> ΣΕΥΡΑΟΤΈΝ ΣΕΥΡΑΟΤΈΝ ΣΕΥΡΑΟΤΈΝ ΣΕΥΡΑΟΤΈΝ</u>

RETR ED) 1094 **RE** B. MOKHTARI AND K. POURABDOLLAH R 10 µA R E F. - cs-: 12 Cs :10 -Os* -i C 13 -ì H Rb+ 13 Rb1:10 ٦Ì Ro+:12 <u>_</u>K*:10 -<u>k:12</u> F H.K-:11 H R E E E sr²⁺:10 Ba²⁺ RF Sr Ca2+:10 Ca2+:12 11 REAL Pb2+:12 Ř :11 Pb2+ R R 4 Co2+:12 :13 Co2+:11 Co2+ R R Zn2+:12 RE R Ni2+:12 R RE Nd3+:12 7 K -Tb :10 D E F) R 0.6 1.0 Epotentia 0.2 Epotentiat 1.0 1.0 R Eotenthal As. Ag/Ag T(M RFIG. 4. Differential pulse voltammograms of 0.1 mM 10-13 in the presence of D1 mM alkalicalkaline earth, transition metals and lanthanides in CH1CNoxidation of carboxylic acid exhibits one oxidation peak with RIn the rest of the experiments, DRV was used instead of CV two electron and proton transfer in organic solution, the for-mation of intranolecular H-bonding between two carboxylic to get a better resolution of waves in the same condition. The voltammetric behaviors of carboxytic acids in each host scafacids in 10-13 and 23-26 causes one proton transfer easier and folds were compared with different cations in DPV to investigate the binding properties of 10-13 and 23-26. A constant volume the other more difficult, leading to oxidation peaks at a more positive potential and a less positive potential, respectively. Oxof $10 \,\mu\text{L}$ per injection of the cation in 0.1 M TBAPF₆ was added idation behavior of two earboxylic acid groups in 10-13 and 23-26 implies that H-bonding to carboxylic acid moiety is ininto the cell to make 0.1-3.0 equivalent of cation in the solution. DPVs were recorded after adding stoichiometric equivalent of fluenced by binding to cations because the carboxytic acids are located around the crown ether molety. cations successively to the respective electrochemical solution. DPVs of **10–13** and **23–26** depict almost same voltammetric 'R RETRA R E E. RE'I 'R R ΕI REI DETD A TED DI TD TTT DI T Α TTTT DI

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R The changes of current ratio in the first anodic peak for complexation of 0. In M scaffolds 10-13 and 23-26 with the 0.1 mM D
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$\begin{array}{c} R_{26}^{5} = TR \stackrel{(0.89)}{_{-0.89}} \stackrel{(0.93)}{_{-0.88}} \stackrel{(0.91)}{_{-0.93}} \stackrel{(0.39)}{_{-0.91}} \stackrel{(0.89)}{_{-0.91}} \stackrel{(0.89)}{_{-0.90}} \stackrel{(0.98)}{_{-0.58}} \stackrel{(0.98)}{_{-0.97}} \stackrel{(0.89)}{_{-0.49}} \stackrel{(0.94)}{_{-0.89}} \stackrel{(0.97)}{_{-0.95}} \stackrel{(0.97)}{_{-0.95}} \stackrel{(0.97)}{_{-0.98}} \stackrel{(0.97)}{_{-0.98}} \stackrel{(0.97)}{_{-0.98}} \stackrel{(0.97)}{_{-0.99}} ($
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Rehaviors with two anodic peaks Ras Findicated and W Figure 4 Reserved in the peak current and the peak porentral sexcept for
shows the differential pulse voltammograms of 0.1 mM 10–13 in Rhe presence of 0.1 mM alkalia alkaline earth transition metals.) Regarding to the role of binding site in the complex, the and lanthanidae in CH CN
R As depicted in the first column of Figure 4, the macrocycle 10 in the presence of Na ⁺ shows significant voltammetric changes
of DPV in the peak current and the potential This is due to the electrostatic interaction between 10 and the cations lead- (and hydroxyl groups of carboxylic acids) may cause deproto-
ing to electrostatic perturbation of intramolecular H-bonding by encapsulation of Na ⁺ into crown ether and two carboxylic acid
proups: According to the second column of Figure 4, by addition of one equivalent of atkali, atkaline earth, transition metals, and ical and the positive cations in scaffolds 10–13 and 23–26. The
hanthanides to a solution containing macrocycle/11, no changes were observed in the peak current or potentials except for Na ⁺ alent of 10-13 and 23-26 in the presence of one equivalent of
and Ba ²⁺ Based on the third column of Figure 4, the addition of one equivalent of K ⁺ , Na ⁺ , Pb ²⁺ to 12 caused large decrease ing orders of 10-13 and 23-26 of the first peak are Na ⁺ for
in anodic peak currents or even disappearance of the original scaffold 10, $Na^+ < Ba^{2+}$ for scaffold 11, $Na^+ < K^+ < Pb^{2+}$ for scaffold 13, Na^+ for scaffold 23, Ba^{2+}
perturbation of intramolecular H-bonding by binding of cations for scaffold 24, $K^+ < Ba^{2+}$ for scaffold 25, and $Ca^{2+} < Pb^{2+}$ for scaffold 26 that are in accordance with the order of po-
in the fourth column of Figure 4, by addition of one equiv- ratent of selected cations to macrocycle 13, no changes were rable 3. RACTED RETRACTED
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RETRACTED RETRACTED Between free and complexed macrocycles 10-13, and 23-26, which were determined by RETRACTED
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Dong, H.; Zheng, H.; Lin, L.; Ye, B. Sens. Actuators B 2006, 115, 303. Figure 5 depicted the effect of concentration of corresponded cations to scaffolds 10 and 23. With increasing the amounts of peak current at 0.8-V decreased quantitatively by increasing the Raoof, J.B. Ofani R., Alinezhad, A. Rezaie, S. RMonatshefte Chem. D concentration of Na⁺ and gradually reached to the minimum Nalue at around one equivalent. KEIRACIED 16. Wang, L.; Zhao, B.T.; Ye, B.X. Electroanalysis 2007, 19, 923. 17. Dong, H., Kin, A.; Zheng, H.; Zhao, G.; Ye, B. Electroanalysis 2006, 78, 1202. RETRAC R Vang, F. Diu, A; Wu, YJ. Gao, M. Rhuang, K. R. Chin Chem. Foc. 12009, 16, 778. A. W. T. Edd, M. Rhuang, K. R. REFICIUSIONS ТЕІ ľŀE 19. Canpolat, E.C.; Sar, F.; Coskun, N.Y.; Cankurtaran, H. *Electroanalysis* **2007**, 19, H09ACTED RETRACTE 20. Shamsipur, M.; Miranbeigi, A.A.; Teymouri, M.; Rasoolipour, S.; Asfari, 23-26 and their binding abilities toward alkali, alkaline earth, transition metals, and lanthanides were examined by differential 2RZ Anat. Chem. 2009, 871 6789. Dy RETTRACTERIA CITERIA pulse voltammetry. Compounds 12 and 25 showed their voltam-K.; Hianik, T. Bioelectrochem. 2010, 80, 55. 22 Vaze, V.D.; Srivastava, A.K. Electrochim. Actu 2007, 53, 1718. 23. Zhang, H.L.; Liu, Y.; Lai, G.S.; Yu, A.M.; Huang, Y.M.; Jin, C.M. Analyst metric behavior toward K⁺, 10–12 and 23 towards Na⁺; 11, 24 and 25 toward Ba+; 26 toward Ca2; and 12, 13, and 26 toward Ph²⁺ in CH₃CN. This was mainly related to the presence 242009, 1347314), CTTEDec Bureirs 1200, 19,496 of crown other group between two proximal carboxylic acid Liu, H.; Zhao, G.; Wen, L.; Ye, B. J. Anal. Chem. 2006, 61, 1104.
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