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Notes

Calix[4]azacrown Ethers in Polymeric CTA Membrane

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Calixarenes, which are macrocyclic compounds available in a variety of ring sizes, are of particular interest as inclusion hosts for ions and specific molecules.[1-3] 1,3-Distal capping of calix[4]arene at the lower rim has been achieved with polyethers linkages such as calix-crown ethers,[4] and double calix-crowns.[5] In particular, calix[4]arene crown-6 in which a pentaethylene glycol unit connects with the 1,3-dialkyloxy calix[4]arene framework in the 1,3-alternate type was known to give high cesium ion selectivity over other alkali metal ions.[6-15] Calix-bis-crown having two crown loops can adopt 1 : 2 complexation, but it still had conflicting results by conductometric, spectroscopic, and X-ray crystallographic studies.[8,9] Although calix-bis-crowns have two cavities that can simultaneously capture two metal ions, they have shown to even worse extractability than calix-mono-crowns, probably due not only to electrostatic repulsion between the two metal ions, but also to an induced conformational change that does not favor binding of the second metal.[9]

Instead of calix[4]crown ethers, calix[4]azacrown ethers with a potential pendent arm in the nitrogen atom have been rarely reported because of synthetic difficulties.[17-19] The combination of azacrown ether and calix[4]arene would result in an optimized structure for metal ion encapsulation through (1) electrostatic interactions between the metal ion and both the oxygen atoms and a nitrogen atom as electron donors, (2) π-metal interactions between the metal ion and two rotated aromatic nuclei of the 1,3-alternate calixarene.

Simple two phase extraction, bulk liquid membrane (BLM), and supported liquid membrane (SLM) using calix-crown derivatives has been explored to investigate binding capabilities, transport rates, and transport permeability for the cesium ion.[20] As one of the membrane technologies, polymeric inclusion membrane (PIM) system has been recently investigated taking advantages of (i) easy setup, (ii) high selectivity, and (iii) high durability, has been developed.[21,22] PIMs with cellulose triacetate (CTA) polymer as a supportor, together with a hydrophobic non-volatile organic solvent (e.g., NPOE) as a plasticizer and an organic carrier were found to cover the several problems occurred in SLM

1   2   3
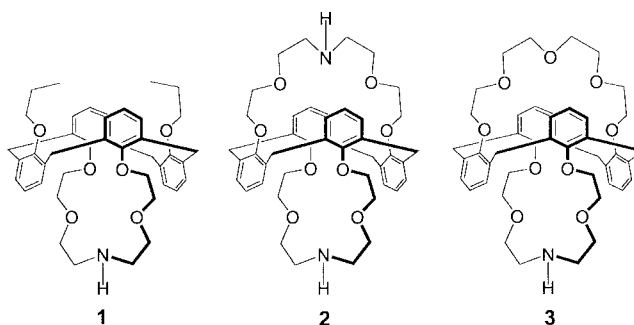
system such as loss of organic carrier with plasticizer into aqueous solution, slow transport rate, and low durability.[20-22] However, the measurement of the transport rate and permeability toward metal ions in PIMs system using the calixaza-crown ethers as an organic carrier has never been reported. We now report the experimental results of the transport flux values for the transition metal ions in PIMs system (single and competitive ion experiments) along with permeability coefficient (P) for silver ion.

Experimental Section

Unless specified otherwise, reagent-grade reactants and solvents were obtained from chemical suppliers and used as received. Deionized water was prepared by passing distilled water through an Organo G-10 cartridge. Calix[4]arene.[1] 1.[17] and 2[18] were prepared by the adaptation of the reported procedures. ^{1}H NMR and ^{13}C NMR spectra were obtained from the Korea Basic Science Research Institute, Daejon, Korea.

Syntheses.

25,27-Bis(5-chloro-3-oxapentoxy) calix[4]arene (4). Calix[4]arene (10.02 g, 23.6 mmol), tosylate of 2-(2-chloro-ethoxy)ethanol (13.87 g, 49.5 mmol), K_2CO_3 (3.31 g, 23.6 mmol) and acetonitrile (200 ml) were refluxed for 24 h under N_2. The crude residue was dissolved in CH_2Cl_2 and acidified (10% aqueous HCl). The organic layer was dried ($MgSO_4$). Recrystallization from Et_2O gave 2 as a solid in 90% yield, mp 174-175 °C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ

7.34 (s. 2H, -O*H*), 7.08 (d, *J* = 7.5 Hz. 4H, Ar*H*$_m$-calix), 6.90 (d, *J* = 7.5 Hz. 4H. Ar*H*$_m$-calix). 6.77 (t. *J* = 7.5 Hz, 2 H. Ar*H*$_p$-calix). 6.70 (t, 2H, *J* = 7.5 Hz, Ar*H*$_p$-calix), 4.45 (d, *J* = 13.5 Hz, 4H. Ar*CH*$_2$Ar). 4.21-4.19 (m. 4H, ArOCH$_2$*CH*$_2$). 4.06-4.04 (m. 4H. O*CH*$_2$CH$_2$Cl), 3.99 (t. *J* = 6.5 Hz. 4H. ArO*CH*$_2$CH$_2$), 3.75 (t. *J* = 6.5 Hz. 4H. OCH$_2$*CH*$_2$Cl), 3.40 (d, *J* = 13.5 Hz. 4H. Ar*CH*$_2$Ar). FAB-MS *m/z* (M$^-$) calcd. 634.9, found 635.2. Anal. Calcd for C$_{36}$H$_{38}$Cl$_2$O$_6$: C, 68.03; H. 5.98. Found: C. 68.00; H, 5.95.

25,27-Bis(5-chloro-3-oxapentoxy) calix[4]arene crown-5 (5). Compound 4 (10.08 g, 16.5 mmol), tetraethyleneglycol ditosylate (9.12 g, 18.1 mmol), Cs$_2$CO$_3$ (16.15 g. 49.5 mmol) and acetonitrile (200 mL) were refluxed for 24 h under N$_2$. The crude mixture was dissolved in CH$_2$Cl$_2$ and acidified (10% aqueous HCl). The organic layer was dried over MgSO$_4$. Column chromatography on silica gel with 1 : 2 EtOAc : hexane as eluents gave 5 as an oil in 73% yield. ^{1}H-NMR (600 MHz. CDCl$_3$): δ 7.12-7.08 (m. 8H. Ar*H*$_m$-calix). 6.93-6.86 (m. 4H. Ar*H*$_p$-calix), 3.87 (s. 8H. Ar*CH*$_2$Ar). 3.61-3.41 (m, 24H. -*CH*$_2$-), 3.20-3.13 (m, 8H. -*CH*$_2$-). FAB-MS *m/z* (M$^+$) calcd. 793.2. found 793.8. Anal. Calcd for C$_{44}$H$_{52}$Cl$_2$O$_9$: C, 55.48; H, 6.55. Found: C, 55.45; H. 6.57.

N-(p-Toluenesulfonyl) calix[4]crown-5-azacrown-5 (6). *p*-Toluenesulfonamide (0.90 g. 5.26 mmol), Cs$_2$CO$_3$ (6.54 g. 20.1 mmol) and DMF (100 mL) were heated to 70 °C for 30 min. Then, compound 5 (4.04 g, 5.04 mmol) dissolved in DMF (20 mL) was added dropwise for 3 h. After refluxing for 24 h under N$_2$, the crude mixture was dissolved in CH$_2$Cl$_2$ and the treated with saturated NaHCO$_3$ aqueous solution. The organic layer was dried over MgSO$_4$. Column chromatography on silica gel with 1 : 2 (EtOAc : hexane) as eluents gave 6 as a solid in 50% yield. mp 125-127 °C. ^{1}H-NMR (400 MHz. CDCl$_3$): δ 7.74 (d. *J* = 8.1 Hz. 2H, Ar*H*-tosyl). 7.34 (d, *J* = 8.1 Hz, 2H, Ar*H*-tosyl). 7.12 (d. *J* = 7.5 Hz, 4H, Ar*H*$_m$-calix). 7.05 (d. *J* = 7.5 Hz, 4H. Ar*H*$_m$-calix). 6.91 (t, *J* = 8.7 Hz. 2H. Ar*H*$_p$-calix), 6.82 (t, *J* = 8.7 Hz. 2H. Ar*H*$_p$-calix), 3.87 (s, 8H, Ar-*CH*$_2$-Ar), 3.63 (s, 4H, O*CH*$_2$*CH*$_2$O), 3.57 (s. 4H. O*CH*$_2$CH$_2$NH), 3.49-3.46 (m. 8H. ArO*CH*$_2$*CH*$_2$O), 3.34-3.28 (m, 8H. ArO*CH*$_2$*CH*$_2$O). 3.16-3.07 (m, 8H. O*CH*$_2$*CH*$_2$O). 2.46 (s, 3H. Ar*CH*$_3$-tosyl). FAB-MS *m/z* (M$^+$) calcd. 893.1, found 894.0. Anal. Calcd for C$_{51}$H$_{59}$NO$_{11}$S: C. 68.53; H. 6.60. Found: C, 68.50; H. 6.63.

Calix[4]crown-5-azacrown-5 (3). To a solution of dioxane (100 mL) and methanol (20 mL) were carefully added 6 (6.09 g. 6.7 mmol), Na$_2$HPO$_4$ (2.23 g. 14.76 mmol) and 6% Na(Hg) (5.00 g). The mixture refluxed for 2 days at 80 °C under N$_2$. The crude mixture was dissolved in CH$_2$Cl$_2$ and the treated with saturated NaHCO$_3$ aqueous solution. The organic layer was dried (MgSO$_4$). Recrystallization from hexane gave 3 as a solid in 70% yield. mp 158-160 °C. ^{1}H-NMR (400 MHz. CDCl$_3$): δ 9.45 (broad s. 1H, N*H*), 7.18 (d. *J* = 7.5 Hz, 4H. Ar*H*$_m$-calix). 7.08 (s, 4H. Ar*H*$_m$-calix). 6.94 (t, *J* = 7.5 Hz, 4H. Ar*H*$_p$-calix), 3.90-3.84 (m. 8H, O*CH*$_2$*CH*$_2$O), 3.79 (s, 8H. Ar-*CH*$_2$-Ar). 3.65-3.63 (m. 12H, O*CH*$_2$CH$_2$NH. O*CH*$_2$*CH*$_2$O). 3.56 (t, *J* = 6.5 Hz. 4H. ArO*CH*$_2$ CH$_2$O-crown-5). 3.28 (t, *J* = 6.5 Hz. 4H, ArOCH$_2$*CH*$_2$O-crown-5).

2.98 (s. 4H, OCH$_2$*CH*$_2$NH). FAB-MS *m/z* (M$^+$) calcd. 739.1, found 739.5. Anal. Calcd for C$_{44}$H$_{53}$NO$_9$: C. 71.44; H, 7.17. Found: C, 71.49; H, 7.15.

Calorimetric titration. The calorimetric titration were carried out using TRONAC Model 1250 isothermal isoperibol titration calorimeter equipped with a 25-mL glass Dewar flask and a 10 mL-precision constant-rate burette for titrant delivery. The thermostat was maintained at 25 ± 0.02 °C with a TRONAC model 40 precision temperature controller. Procedures. calibrations, and data analyses including determinations of ΔH value by non-linear regression methods were adapted from earlier recommendation. The ligand solution (1.0 mM of calix[4]azacrown ether. 25 mL) was titrated with 10 mM of titrant in 90% (v/v) MeOH solution.

CTA membrane and transport experiment. CTA (0.062 g) in 5.0 mL of CH$_2$Cl$_2$ was mixed with 3.0 mL of 10 % (v/v) NPOE (2-nitrophenyloctyl ether) in CH$_2$Cl$_2$ and 0.50 mL of 10% (v/v) tris(2-butoxyethyl) phosphate (TBEP) in CH$_2$Cl$_2$, and 2.0 mL of the ligands 1-3 (1.0 mM) in CH$_2$Cl$_2$. The NPOE and TBEP were used as membrane plasticizers. The solvent of this mixed solution was allowed to slowly evaporate in a 9.0 cm diameter petri dish which was covered by a watch glass. After 18-hour evaporation. a few drops of water were swirled on top of the film to help loosen it from the glass.[21] The polymer film was then carefully peeled off the petri dish. The thickness of the membrane film was 30 μm. The membrane was placed between 200 mL of two cylindrical glass vessels. The area of the membrane was 19.6 cm^2. Both source and receiving aqueous phase were stirred with glass stirrers at about 600 rpm by synchronous motors at 25 °C. The initial concentration of AgNO$_3$. Cd(NO$_3$)$_2$, Co(NO$_3$)$_2$, Cu(NO$_3$)$_2$. Ni(NO$_3$)$_2$, Pb(NO$_3$)$_2$. and Zn(NO$_3$)$_2$ was 0.010 M, respectively. 5.0 mL from the receiving phase was periodically taken and the metal cations transported from source phase into receiving phase through PIMs were determined by atomic absorption spectrometer. Feed solution in competitive ion transport experiments was composed of 0.01 M of seven component metal ions in deionized water. Three independent experiments were employed in the determination of each cation transport rate and permeability constant. The standard deviations were less than ± 10%.

Results and Discussion

The synthetic scheme for 3 is shown in Scheme 1. Compound 4 was prepared from the reaction of calix[4]arene and tosylate of 2-chloroethoxyethanol in the presence of K$_2$CO$_3$. Glycolic-cyclization of the calixarene 4 with penta(ethyleneglycol) *para*-ditosylate with 3 equivs of Cs$_2$CO$_3$ was carried out in refluxing acetonitrile for 24 h to give 5 in the 1,3-alternate conformation with 73% yield. This 1,3-alternate conformer of 5 was confirmed by ^{1}H (singlet peak of Ar*CH*$_2$Ar appeared at δ 3.87) and ^{13}C (Ar*CH*$_2$Ar at 38.7 ppm) NMR spectral assignments. Ring-closure of 5 was performed with 1 equiv of *p*-toluenesulfonamide in the presence of Cs$_2$CO$_3$ by heating at 70 °C in DMF for 24 h. The crude residue was chromatographed on silica gel to afford *N*-tosyl

Scheme 1. Synthetic routes for compound **3**.

Table 1. Enthalpy changes for complexation of metal cation by various organic ligands

Com-pound	ΔH (Kcal/mole)					
	Ag^-	Pb^{2+}	Cd^{2-}	Cu^{2-}	Co^{2-}	Ni^{2-}
1	-10.02	-8.91	-3.18	-8.71	-5.86	-4.81
2	-8.79	-8.88	-2.63	-7.65	-4.44	-2.47
3	-7.67	-7.73	-2.96	-2.99	-2.18	-0.22

Titrate: 0.001 M ligands in 90% (v/v) methanol. Titrant: 0.01 M metal cations in 90% (v/v) methanol.

Table 2. Single ion transport values of transition metal cations through PIM using various ligands

Compound	Flux values ($\times 10^{-5}$ mol · h^{-1} · m^{-2})^a		
	Ag^-	Pb^{2+}	Cd^{2-}
1	8.63	0	0
2	2.99	0	0
3	6.38	1.03	0

^{a}Transport condition: source phase (aqueous solution of metal nitrate 10 mM); membrane phase (CTA membrane) [carrier] = 1 mM; receiving phase: deionized water.

(1) provided one of the most exothermic reactions in cation interactions.

Polymeric inclusion membranes (PIMs), modeled after the diketone-containing membranes developed by Sugiura and are formed by the polymerization of cellulose triacetate (CTA) to give a thin film.[22] The membrane is then placed between an aqueous source and a receiving phase. While PIMs can effectively separate two aqueous phases, they are independent of the organic solvents in maintaining phase separation and in allowing continuous transport. Transport rates of the silver and transition metal ions through PIMs when ligands **1-3** were used are listed in Table 2. We selected Ag^+, Pb^{2+}, and Cd^{2-} as experimental target cations because they gave high exothermic data in calorimetric titration as shown previously. In a control experiment, no transport rate was detected in the absence of the ligands during more than 60-hour continuous run. Silver ion was rapidly transported compared to lead and cadmium ions. Ligand **1** gave much fast transport of silver ion consistent with those of calorimetric titration experiments. This trend is in good agreement with the results of thermodynamic titration.

Permeability coefficient (P, m · s^{-1}) was determined by plotting the logarithm of Cs/Co vs time according to Eq. (1)

$$\ln(Cs/Co) = -(P \, S/V) \, t \qquad (1)$$

where Co is the initial concentration of the cation in the source phase, Cs denotes the metal ion concentration in the source phase as a function of time. V and S are for volume of source phase and for membrane area, respectively.[22] To obtain the permeability coefficient of the silver ion, several different experimental sets were performed. The results are listed in Table 3 and depicted in Figure 1. The slope for $\ln(Cs/C0)/(S/V)$ vs time (h) indicates the permeability coefficient (P). Ligand **1** provided the largest permeability coefficient (−4.0) among ligand **1-3**. Law permeabilities of

calix[4]crown-6-azacrown-5 (**6**) in 50% yield. The use of K_2CO_3 as a base gave similar pattern in TLC but needed longer reaction time (72 h) than in the case of using Cs_2CO_3. The singlet peak observed at δ 3.87 for $ArCH_2Ar$ in ^1H NMR and single peak appeared at 38.7 ppm for $ArCH_2Ar$ in ^{13}C NMR spectra indicate that **6** exists in the 1,3-alternate conformation. Detosylation of **6** by the use of Na(Hg)-amalgam in the presence of Na_2HPO_4 in co-solvents of MeOH and dioxane gave the final product **3** (60% yield). The structure of **3** was also confirmed to be 1,3-alternate conformation by ^1H and ^{13}C NMR spectrometer as mentioned above.

Enthalpy changes and permeability upon metal ion complexation. The enthalpy changes (H) of the ligand (**1-3**) for metal ion complexation was determined by titration calorimetry in 90% (v/v) methanol. Complexation of the cations with the ligand is exothermic as shown in Table 1, implying that the interaction of the ligand with cations is enthalpy driven. Carrier **1** forms the strongest complex with Ag^+ although **2** and **3** have two cavities able to simultaneously complex with cations. It was reported that although calix-*bis*-crowns have two cavities that can simultaneously capture two metal ions, they have been shown to have even worse extractability than calix-*mono*-crowns, probably due not only to electrostatic repulsion between the two metal ions, but also to an induced conformation change that does not favor binding of the second metal.[16] Based upon this thermodynamic study, we found that calixazacrown ether interact with silver ion more strongly than other cations probably due to a high electrostatic attraction between silver ion and nitrogen atoms. Furthermore, calix-*mono*-azacrown

Table 3. Logarithm of permeability coefficients[a] of silver ion in different concentration of ligands

	1		2		3	
	Conc. (mM)	Log P (m · s⁻¹)	Conc. (mM)	Log P (m · s⁻¹)	Conc. (mM)	Log P (m · s⁻¹)
PIM	1	-5.10	1	-5.52	1	-5.22
	10	-4.0	10	-4.30	10	-4.52

[a]Transport condition: source phase [AgNO₃] = 10 mM: membrane phase [carrier] = 1 mM and 10 mM: receiving phase: deionized water.
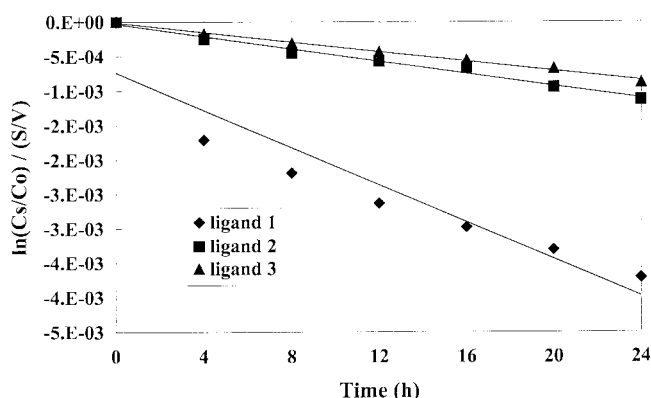

Figure 1. Kinetic plot of silver ion permeability as a function of time (h) through a PIM. Source phase: [AgNO₃] = 10 mM; membrane phase [Ligands] = 10 mM; receiving phase: deionized water.

ligand **2** and **3** are ascribed not only to an electrostatic repulsion between the two metal ions but also to an induced conformation change as mentioned previously. The Log *P* values increased with carrier concentration and reach a maximum at 10 mM of **1**. This relationship also complies with the diffusion-based mathematical model for organic ligand across a liquid membrane described by McBride.[23] To examine the long-term stability of the PIMs. same membrane containing **1** was repeatedly reloaded in successive experiments. Measurement of the transport flux every 4-hour gave constant values up to 15 days. After 15 days we observed some rust in the membrane, then observed small decline of permeability. Unlike SLMs. PIMs system retained a transparent solution without any loss of the organic materials into aqueous solutions during 20-days of stirring. indicating its excellent durability.

In conclusion, a series of calix[4]azacrown ethers for which the monoaza. and unsymmetrical crowned-azacrown. and bis-azacrown ether are examined in the lower rim of the 1,3-alternate calix[4]arene were examined for the thermodynamic parameter, transport abilities, and permeability toward silver and transition metal ions. These ligands were incorporated into polymer inclusion membranes (PIMs) composed of cellulose triacetate (CTA) as a support and 2-nitrophenyl octyl ether (NPOE) and tris(2-butoxyethyl) phosphate (TBEP) as a plasticizer. Calix[4]-*mono*-azacrown ether (**1**) showed the best selectivity toward a silver ion over other transition metal ions. In addition, among ligand **1-3**. ligand **1** (10 mM) gave the best permeability (-4.0) for silver

ion in CTA membrane.

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