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Effect of structural variations of dibenzocrown ether resins and their acyclic analogues upon ion-pair sorption of alkali metal salts from aqueous and aqueous methanol solutions

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Ion-pair sorption of alkali metal salts from aqueous and aqueous methanol solutions by acyclic and cyclic dibenzopolyether resins possessing different side arm groups such as hydroxy, methoxy and carboxy has been investigated. The results reveal that both sorption selectivity and efficiency are influenced by: (1) the methanol content of the aqueous sample solution; (2) the acyclic or cyclic nature of the polyether unit; (3) the conformational positioning of the side arm group with respect to the crown ether cavity; and (4) the identity of the counteranion species of the alkali metal salt. For *sym*-(C₃H₇)(R')dibenzo-16-crown-5 resins, the sorption selectivity and efficiency increased as the R' group was varied: -OCH₃ < -OH < -OCH₂CO₂H. The highest sorption efficiency and Na⁺ selectivity was obtained for *sym*-(propyl)dibenzo-16-crown-5-oxyacetic acid resin (7) in which the pendent carboxylic acid group is oriented over the crown ether cavity. The use of a less hydrated anion in the alkali metal salt species enhances the ion-pair efficiency: SO₄²⁻ < NO₃⁻, Cl⁻, Br⁻ < I⁻ < SCN⁻. Monovalent metal selective sorption was noted for competitive ion-pair sorption of NaCl, KCl, MgCl₂ and CaCl₂ by resin 7.

INTRODUCTION

The development of chelating resins for the separation of selected metal species from aqueous solutions has received considerable attention.^{1,2} Recently, we reported the use of crown ether carboxylic acid resins for selective sorption and separation of alkali metal cations from aqueous solution.³⁻⁹ It was found that preorganization of the binding site by conformational positioning of a pendent carboxylic acid group over

the crown ether cavity provided excellent sorption efficiency and selectivity in alkali metal cation separation. The results demonstrated that by appropriate molecular design of the binding site in polyether resins, it is possible to produce novel resins with enhanced selectivities in metal ion separation processes.

Blasius and coworkers¹⁰⁻¹² synthesized a series of neutral crown ether resins by polycondensation of dibenzocrown ethers with formaldehyde. These resins exhibited a selective binding ability for alkali metal salts in solution. Compared with conventional ion-exchange resins, however, interaction between the neutral crown ether binding site and the metal cation was weak. To enhance the binding ability, additional neutral chelating groups should be introduced at the binding site of the crown ether resins.

For this purpose, acyclic and cyclic dibenzopolyether resins possessing different side arm groups such as hydroxy, methoxy, and carboxy have been prepared. Under acidic conditions, the carboxylic acid group can act as a neutral chelating group for metal salt separation. The structural variations within the polyether resins are shown in Figure 1. We have already found that the pendent side arm group, R', is conformationally mobile when R = H. In contrast, attachment of a propyl group at R orients the R' group over the crown ether cavity.³ Thus the influence of the side arm group identity as well as its conformational positioning relative to the binding site upon ion-pair sorption of alkali metal chlorides from aqueous methanol solutions may be evaluated.

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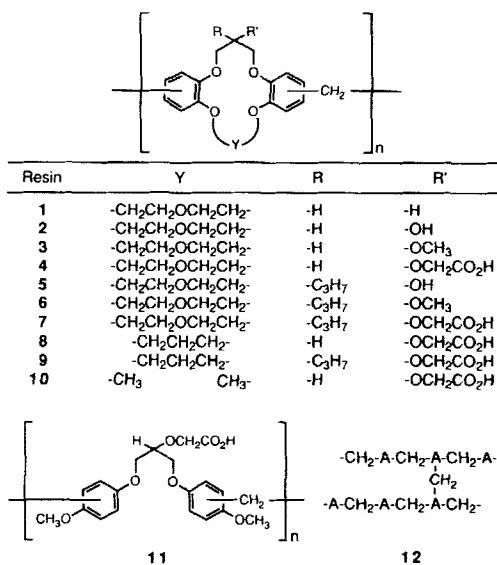


Figure 1 Structures of dibenzopolyether resins.

RESULTS AND DISCUSSION

Preparation of dibenzocrown ether resins

The resins were prepared by condensation polymerization of the corresponding dibenzopolyether monomers with formaldehyde in formic acid. The preparation and characterization of resins 1, 4 and 7–11 have been reported elsewhere.^{3,8,9} By the same synthetic procedure, resins 2, 3, 5 and 6 were obtained in yields of 76–83%. The structural representations of 1–11 imply polymers with no crosslinking. This is most certainly an oversimplification since some level of crosslinking is anticipated. A possible structure for the partially crosslinked resins is given by 12 (Fig 1) in which A is the dibenzopolyether unit. The elemental analysis results obtained for resins 2, 3, 5 and 6, are consistent with the presence of methylol ($-\text{CH}_2\text{OH}$) groups on some of the aromatic rings in 12. For resins 3, 5 and 6, the elemental analyses results are consistent with those for structure 12 with 3.0, 5.3 and 4.5 methylol groups, respectively. For resin 2, the observed combustion analysis results do not fit well with the calculated values even when six methylol groups are introduced. This resin had been purified by washing with methanol and water several times, but a difference between the observed and the calculated analysis values remained. Also a second synthesis of 2 did not produce a resin for which satisfactory elemental analysis could be obtained. Although some contaminants may be present in resin 2, its sorption behaviour was investigated in the same manner as that used for the other resins. No unusual sorption behaviour was noted for resin 2.

Effect of side arm species variation of dibenzo-16-crown-5 resins 1–7 upon ion-pair sorption of alkali metal chlorides

To provide greater relevance to actual metal separation systems, competitive rather than single species alkali metal chloride sorption by dibenzo-16-crown-5 resins possessing different side arm groups such as hydroxy, methoxy and carboxy was investigated. Aqueous or aqueous methanol solutions (5.0 ml) of the five alkali metal cations (0.10 M in each) as the chlorides were shaken with the resin (0.040 g) for 3 h (even though sorption is probably complete within a matter of minutes).⁹ The resins were filtered and purged with air using an aspirator for 20 min to remove the sample solutions completely. A purging time of 10 min was found to be sufficient to obtain constant sorption results. The resins were dried at 80°C. Of the dried resins, weighed portions (0.020 g) were shaken with 5.0 ml of pure water to strip the alkali metal chlorides from the resins into an aqueous solution for analysis by ion chromatography. With this stripping procedure, only the ion-pair sorbed species were recovered in the stripping solution. Thus ion-exchange sorption of alkali metal cations by resins with carboxy side groups was eliminated by using this evaluation method. From the ion-chromatographic analysis data, the amounts of sorbed alkali metal chlorides per unit amount of dried resin (mmol/g) were calculated. The reproducibility of the alkali metal chloride sorption and stripping procedure has been demonstrated.⁹ For a given resin, reproducibility of alkali metal chloride sorption from 80% methanol–20% water was found to be reproducible within ± 0.07 times the stated value for lithium chloride and ± 0.05 times the stated values for other alkali metal chlorides.

Results for the competitive sorption of alkali metal chlorides by resins 2–4 as a function of methanol content in sample solutions are presented in Figures 2(b)–2(d). For comparison, data reported for dibenzo-16-crown-5 resin 1⁹ are included (Fig 2a). It is known that the metal ion–crown ether interaction is enhanced as the polarity of the solution is lowered.¹³ Thus the sorption of each alkali metal chloride increased as the methanol content in the sample solution was enhanced, with the exception of LiCl sorption. Since the cavity size of the 16-crown-5 ring (2.0–2.4 Å) fits well with the ion diameter of Na⁺ (1.9 Å), dibenzo-16-crown-5 resin 1 exhibits Na⁺ selectivity for ion-pair sorption of alkali metal chlorides. The sorption selectivity observed for resin 1, which has no side arm groups, is Na⁺ > K⁺, Rb⁺, Cs⁺ > Li⁺. It is seen, however, that the attachment of hydroxy, methoxy and carboxy functions to the dibenzo-16-crown-5 ring enhances K⁺ sorption. Thus observed selectivities for sorption from 80% methanol–20% water are: Na⁺, K⁺ > Rb⁺,

$\text{Cs}^+ > \text{Li}^+$ for resins 2 and 3 and $\text{K}^+ > \text{Na}^+$, $\text{Rb}^+ > \text{Cs}^+ \gg \text{Li}^+$ for resin 4.

In Figures 3(a)–3(c) are shown data for the ion-pair sorption of alkali metal chlorides by resins 5–7 in which a propyl group is attached to the same polyether

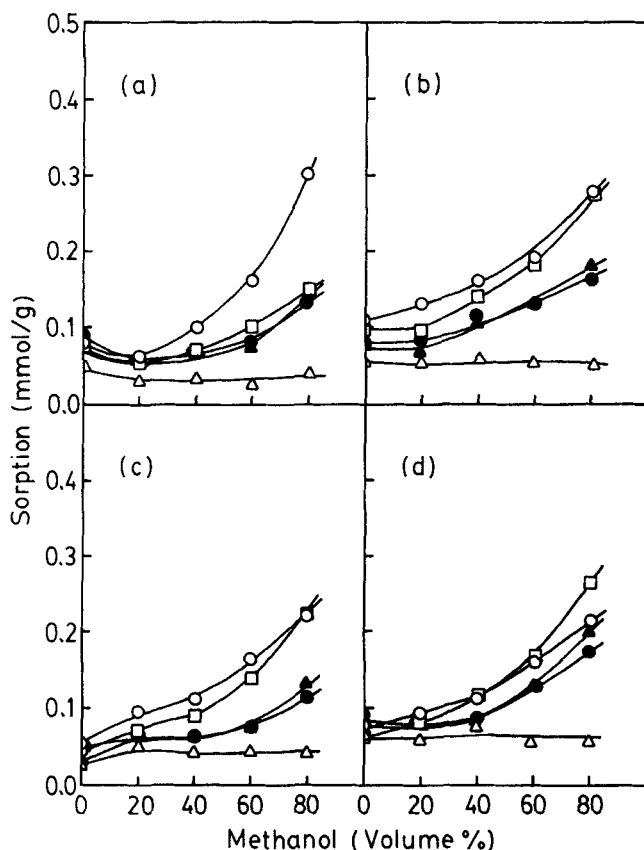


Figure 2 Sorption of alkali metal chlorides by dibenzocrown ether resins vs. methanol content of the aqueous methanol sample solution for resins (a) 1, (b) 2, (c) 3 and (d) 4: (Δ) Li^+ , (\circ) Na^+ , (\square) K^+ , (\blacktriangle) Rb^+ , (\bullet) Cs^+ .

ring carbon that bears the side arm group. The observed selectivities for sorption from 80% methanol–20% water are: $\text{Na}^+ > \text{K}^+ > \text{Rb}^+$, $\text{Cs}^+ > \text{Li}^+$ for 5, $\text{Na}^+ > \text{K}^+ > \text{Cs}^+ > \text{Rb}^+ > \text{Li}^+$ for 6; and $\text{Na}^+ \gg \text{K}^+ > \text{Rb}^+$, $\text{Cs}^+ > \text{Li}^+$ for 7. Clearly the Na^+ sorption selectivity is improved by introduction of the propyl chain on the crown ether ring. As we reported previously,³ the pendent side arm group is flexible when $\text{R} = \text{H}$. Thus a complex in which the K^+ ion perches on the crown ether oxygen while associating with the oxygen atom in the side arm may be formed for resins 2–4. In contrast, attachment of a propyl group to the same ring carbon that bears the side arm group is expected to position the side arm group over the crown ether cavity. Thus the enhancement of NaCl sorption relative to the sorption of other alkali metal chlorides is attributed to preorganization of the binding site in resins 5–7 which increases recognition of the metal ion that should be best accommodated within the crown ether cavity.

The alkali metal chloride sorption and loadings for resins 1–7 are presented in Table 1. The loading is

Table 1 Ion-exchange capacity and total sorption of alkali metal chlorides by dibenzo-16-crown-5 resins

Resin	Ion-exchange capacity (mmol/g)*	Total sorption (mmol/g)**	Loading (%)
1	2.92	0.76	26
2	2.48	0.96	39
3	2.54	0.74	29
4	2.39	0.93	39
5	2.36	0.71	30
6	2.26	0.58	26
7	2.17	0.87	40

* Calculated from elemental analysis data. ** Data from the 80% methanol–20% water system.

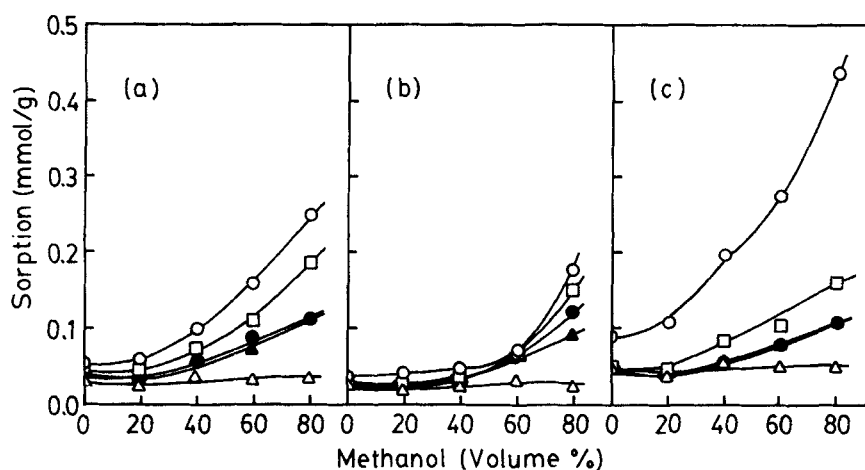


Figure 3 Sorption of alkali metal chlorides by dibenzocrown ether resins vs. methanol content of the aqueous methanol sample solution for resins (a) 5, (b) 6 and (c) 7: (Δ) Li^+ , (\circ) Na^+ , (\square) K^+ , (\blacktriangle) Rb^+ , (\bullet) Cs^+ .

defined as the total alkali metal chloride sorption (calculated from the total alkali metal cation concentration in the stripping solution) divided by the ion-exchange capacities (calculated from the elemental analysis data). The loadings vary from 40% for 7 to 26% for 6. It is noted that the methylation of a hydroxy group side arm reduces the sorption efficiency. When $R = C_3H_7$ for the dibenzo-16-crown-5 resins, the sorption selectivity and efficiency increases in the following R' group order: $-OCH_3 < -OH < -OCH_2CO_2H$. The highest sorption efficiency and Na^+ selectivity were obtained for resin 7 in which the pendent carboxy group is oriented over the crown ether cavity.

Ion-pair sorption of alkali metal chlorides from aqueous methanol solution by dibenzo-14-crown-4 carboxylic acid resins 8 and 9 and their acyclic analogues 10 and 11 Data for competitive ion-pair sorption of alkali metal chlorides by resins 8–11 as a function of the methanol content of the medium are presented in Figures 4(a)–4(d), respectively. Dibenzo-14-crown-4 carboxylic

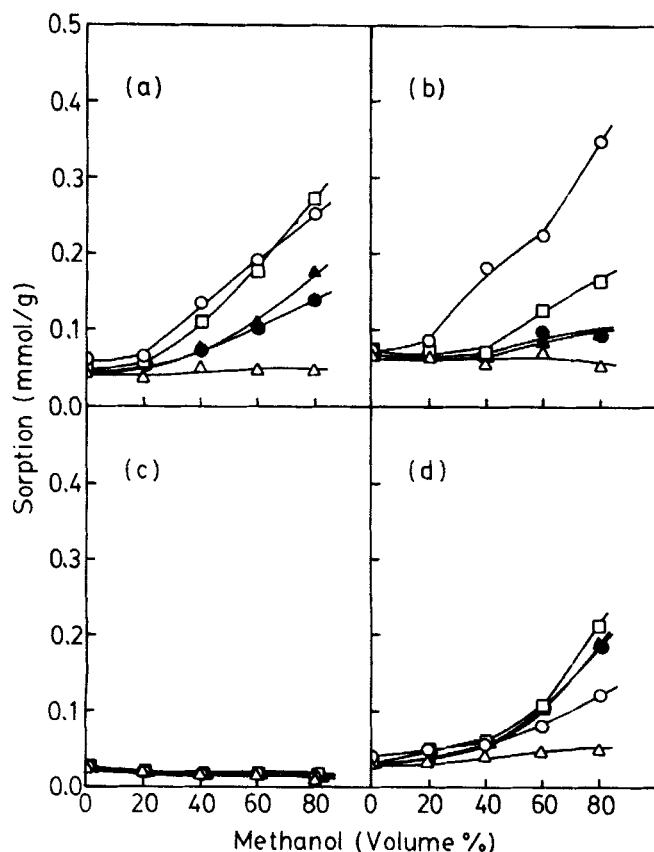


Figure 4 Sorption of alkali metal chlorides by dibenzopolyether resins vs. methanol content of the aqueous methanol sample solution for resins (a) 8, (b) 9, (c) 11 and (d) 10: (Δ) Li^+ , (\circ) Na^+ , (\square) K^+ , (\blacktriangle) Rb^+ , (\bullet) Cs^+ .

Table 2 Ion-exchange capacity and total sorption of alkali metal chlorides by dibenzo-14-crown-4 resins and their acyclic analogues

Resin	Ion-exchange capacity (mmol/g)*	Total sorption (mmol/g)**	Loading (%)
8	2.57	0.88	34
9	2.32	0.75	32
10	2.66	0.77	29
11	2.66	0.11	4

* Calculated from elemental analysis data. ** Data from the 80% methanol–20% water system.

acid resins 8 and 9 show similar sorption profiles to those observed for the corresponding dibenzo-16-crown-5 carboxylic acid resins 4 and 7. Thus resin 8 exhibits selective sorption for both Na^+ and K^+ (Fig 4a). By introducing a propyl group into crown ether ring, the Na^+ sorption selectivity is markedly improved (Fig 4b).

For a structural change to the acyclic polyether carboxylic acid resin 10 (Fig 4d), the sorption selectivity and efficiency decreased markedly. For acyclic polyether carboxylic acid resin 11 (Fig 4b), negligible sorption of the alkali metal chlorides for the entire range of methanol contents was noted. This demonstrates that interaction between the crown ether and the metal cation is a controlling factor for ion-pair sorption.

In Table 2 are recorded the alkali metal chloride sorption and loading data for resins 8–11 in 80% methanol–20% water. An increase in the number of donor oxygens generally enhances the dipole interaction with a metal cation as well as the hydrophilicity at the binding site of the resin. Compared with the loadings obtained from resins 8 and 9 (Table 2), resins 4 and 7 showed better loading (Table 1). The loadings observed for *p*-acyclic polyether resin 11 are less than 15% of that found for *o*-acyclic polyether resin 10. The pseudo crown ether structure of resin 10 apparently enhances the sorption efficiency and selectivity relative to resin 11. This result again demonstrates that a dipole interaction of the crown ether with the metal cation is an important factor for the ion-pair sorption of alkali metal chlorides from solution.

Effect of anion species upon ion-pair sorption of alkali metal salts by resin 7

The sorption of metal cations by neutral crown ethers resins requires simultaneous transfer of an aqueous phase anion. Thus the physical nature, such as hydration degree, of the anion species affects the sorption behaviour. To elucidate the effect of anion species upon ion-pair sorption of alkali metal cations,

Table 3 Effect of anionic species on ion-pair sorption of alkali metal salts from aqueous methanol solution by dibenzo-16-crown-5 resin 7

Anion	Methanol (%)	Sorption (mmol/g)					Total sorption (mmol/g)
		Li	Na	K	Rb	Cs	
SO ₄ ²⁻	40	0.04	0.09	0.04	0.05	0.06	0.28
NO ₃ ⁻	40	0.04	0.21	0.09	0.07	0.06	0.47
Cl ⁻	40	0.06	0.20	0.09	0.07	0.07	0.49
Cl ⁻	80	0.05	0.43	0.16	0.12	0.11	0.87
Br ⁻	80	0.04	0.40	0.16	0.13	0.12	0.85
I ⁻	80	0.04	0.42	0.16	0.17	0.17	0.96
SCN ⁻	80	0.05	0.52	0.27	0.20	0.19	1.23

the anions of the alkali metal salts were varied from chloride to bromide to iodide to thiocyanate to nitrate to sulphate and their sorption behaviour was evaluated.

Data for sorption of the five alkali metal cations as a function of the anion species by resin 7 are presented in Table 3. The sorption was investigated in 80% methanol–20% water. For nitrate and sulphate, however, 40% methanol–60% water was used due to their insolubility in solvent mixtures of lower polarity. The data in Table 3 show that the sorption efficiency increases in the following order: SO₄²⁻ < NO₃⁻, Cl⁻, Br⁻ < I⁻ < SCN⁻. Since the crown ether resin is relatively hydrophobic near the binding site, less hydrated anions are preferred as the concentration for the alkali metal cation sorption. A similar selectivity ordering has been reported by Blasius *et al.*¹² The divalent anion SO₄²⁻ is heavily hydrated and must form a 2:1 ion-pair with the alkali metal cations at the crown ether binding site. Thus the alkali metal sulphates showed very poor sorption in comparison with the sorption of alkali metal cations with monovalent anions.

Competitive ion-pair sorption of NaCl, KCl, MgCl₂ and CaCl₂ by resin 7

To evaluate the competitive ion-pair sorption of alkali metal and alkaline earth chlorides, competitive sorption of NaCl, KCl, MgCl₂, and CaCl₂ by resin 7 was investigated. Results of competitive sorption of these metal chlorides as a function of the methanol content of the medium are shown in Figure 5. Both NaCl and KCl sorption increase as the methanol content in the sample solution is enhanced. This sorption profile is consistent with that observed for competitive alkali metal chloride sorption in this resin (Fig 2c). The divalent metal cations are heavily hydrated and must be accompanied by two hydrated anions. Therefore, the alkaline earth chlorides exhibit very poor sorption. In this system, the monovalent alkali metal salts are selectively recovered from a mixed solution of monovalent and divalent metal cations.

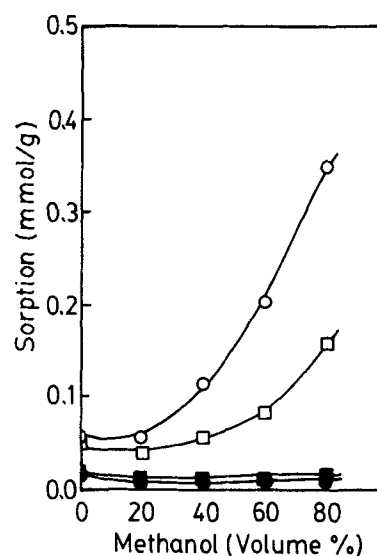


Figure 5 Sorption of alkali metal chlorides by dibenzocrown ether resin 7 vs. methanol content of the aqueous methanol sample solution: (○) Na⁺, (□) K⁺, (■) Mg²⁺, (●) Ca²⁺.

CONCLUSIONS

This study demonstrates that the molecular design of the binding site in neutral crown ether resins is very important for enhancing their sorption selectivity and efficiency for use in metal salt separations from solution. Different sorption selectivities and efficiencies were obtained by varying the side arm groups as well as their conformational positioning near the binding site within the dibenzopolyether resin. When R = C₃H₇ for the dibenzo-16-crown-5 resins, the sorption selectivity and efficiency increased as the R' group was varied: -OCH₃ < -OH < -OCH₂CO₂H. The highest sorption efficiency and Na⁺ selectivity were obtained for *sym*-(propyl)dibenzo-16-crown-5-oxyacetic acid resin 7 in which the pendent carboxylic acid group is oriented over the crown ether cavity. Acyclic polyether carboxylic acid resin 11 exhibited very poor sorption for alkali metal chlorides which shows that the

metal-crown ether interaction is a dominant factor for the ion-pair sorption. Sorption efficiency was found to increase when less hydrated anions were utilized as the counterion for alkali metal cation sorption. Monovalent selective sorption from an alkali metal and alkaline earth salt mixture was achieved with resin 7.

EXPERIMENTAL SECTION

Apparatus and reagents

The apparatus and the sources of reagents were the same as those utilized in previous studies.³⁻⁹ To prevent metal contamination, all glassware was soaked in 5% HNO₃ solution for 24 h and rinsed with distilled, deionized water before use. Distilled water was purified by passage through three Barnstead D8922 combination cartridges in series. The separation and characterization of resins 1, 4 and 7-11 have been reported elsewhere.^{3,8,9} Synthetic procedure for preparation of *sym*-(hydroxy)-dibenzo-16-crown-5, *sym*-(hydroxy)(propyl)dibenzo-16-crown-5 and *sym*-dibenzo-16-crown-5 methyl ether are reported elsewhere.^{3,14}

Preparation of crown ether resins 2, 3, 5 and 6

The crown ether resins were synthesized by condensation polymerization of the corresponding monomers with formaldehyde in formic acid as described in detail previously.³ The resins were ground and used in powder form (finer than 100 mesh).

Resin 2, 78% yield. Anal. calcd. for 12·6.0CH₂OH: C, 65.11; H, 6.20. Found: C, 64.32; H, 5.35.

Resin 3, 77% yield. Anal. calcd. for 12·3.0CH₂OH: C, 66.83; H, 6.47. Found: C, 66.51; H, 6.23.

Resin 5, 83% yield. Anal. calcd. for 12·5.3CH₂OH: C, 67.30; H, 6.99. Found: C, 67.68; H, 6.58.

Resin 6, 76% yield. Anal. calcd. for 12·4.5CH₂OH: C, 68.17; H, 7.12. Found: C, 68.00; H, 6.86.

Ion-pair sorption of metal chlorides by resins 1-11

An aqueous or aqueous methanol solution (5.0 ml) of the five alkali metal chlorides (0.100 M in each) or the alkali metal and alkaline earth chlorides (0.100 M for Na⁺ and K⁺, and 0.050 M for Mg²⁺ and Ca²⁺) was shaken with 0.040 g of resins 1-11 for 3.0 h in a 25 ml pear-shaped flask with a 14/20 joint and a polypropylene stopper at room temperature (21-23°C) with a Burrel

wrist-action shaker. The mixture was filtered with a sintered glass funnel. To remove the sample solution, filtration was continued for 20 min under vacuum using an aspirator (the air was purged through the resin). The resin was dried at 80°C for 12 h. Of the dried resin, a 0.020 g sample was shaken with 5.0 ml of pure water for 1 h to strip the metal chlorides from the resin into an aqueous solution for analysis by ion chromatography with a Dionex Model 2000i ion chromatograph. The concentrations in the alkali metal and alkaline earth metal ion mixture were determined by atomic absorption spectrophotometry (Perkin Elmer Model 5000).

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