In the past decade, the role of complementary size, shape, and functionality in the molecular recognition of neutral molecules has been pointed out. Artificial receptor molecules have been designed with multiple recognition sites and a well-defined geometry, capable of strong and selective binding of the appropriate substrates by hydrogen bonding, ionic interaction, and/or hydrophobic interactions. Rebek et al. have used molecular clefts with acidic functions for the complexation of diamines, such as imidazole. Our group has shown that additional hydrogen-bond donors or electrophilic metal centers, incorporated in the cavities of macrocyclic polyethers, improve strongly the complexation of urea. This type of cocomplexation of a neutral guest by a host molecule either by hydrogen bonding or by coordination with a metal ion, is frequently observed in (metallo)enzymes.

Imidazole and guanidine are two biologically important organic bases. These base residues are part of the essential organic bases in natural polypeptides. Guanidine is one of the organic bases that can act as a proton donor or proton acceptor.

![Chart I. Structures of Carrier Ligands](image)

For the optimal complexation of polyfunctional cations such as uranium, and guanidinium salts, it has been shown that crown ethers with at least 27 ring atoms are needed for the formation of encapsulated complexes.

Table 1. Extraction Efficiency of Benzo and Dibenzo Crown Ethers for Imidazolium Perchlorate

<table>
<thead>
<tr>
<th>crown ether ring size</th>
<th>[CE]_{m}^{+}</th>
<th>[CE]_{m}^{-}</th>
<th>extraction efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>24</td>
<td>0.20</td>
<td>0.19</td>
</tr>
<tr>
<td>2</td>
<td>27</td>
<td>0.12</td>
<td>0.12</td>
</tr>
<tr>
<td>3</td>
<td>30</td>
<td>0.20</td>
<td>0.18</td>
</tr>
<tr>
<td>4</td>
<td>33</td>
<td>0.18</td>
<td>0.18</td>
</tr>
<tr>
<td>5</td>
<td>30</td>
<td>0.15</td>
<td>0.15</td>
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<tr>
<td>6</td>
<td>30</td>
<td>0.11</td>
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</tr>
<tr>
<td>7</td>
<td>33</td>
<td>0.20</td>
<td>0.20</td>
</tr>
</tbody>
</table>

*The initial crown ether concentration in the organic phase.

The total crown ether concentration in the organic phase at equilibrium. The ratio of the imidazolium perchlorate concentration and the total crown ether concentration in the organic phase at equilibrium; Standard deviation ±10%.

The crown ether carriers used in the extraction and transport experiments of guanidinium salts.

Results

The crown ether carriers used in the extraction and transport experiments are depicted in Chart I.

Liquid–Liquid Phase Transfer of Imidazolium Perchlorate. The complex formation of benzo (1–4) and...
bonds are indicated by dashed lines. The perchlorate anion is studied by means of two-phase liquid-liquid extraction dibenzo crown ethers (5-7) with imidazolium salts was studied by means of two-phase liquid-liquid extraction experiments. The amount of salt that is transferred to the organic phase was determined by 1H NMR spectroscopy, after separation of the two liquid phases. The solubility of the crown ethers in water was verified, with use of 1,2,4,5-tetramethylbenzene as an internal standard in the organic phase, by comparing the ratio of crown ether to 1,2,4,5-tetramethylbenzene in the chloroform solution before and after equilibration. Further details of the extraction experiments are described in the Experimental Section. The amount of imidazolium perchlorate transferred to the organic phase is expressed as the extraction efficiency, which represents the ratio between the complexed imidazolium perchlorate and the total crown ether concentration in the organic phase at equilibrium. The results of the extraction experiments with the crown ethers 1-7 are summarized in Table I. Firstly, the results indicate that for complexation of imidazolium a ring size of at least 30 ring atoms is needed; smaller rings do not extract any imidazolium salts. Secondly, the incorporation of a second aromatic ring reduces the extraction efficiency and renders the extraction more dependent on variation of the conformation of the crown ether ring (5-7).

In the case of benzo-30-crown-10, a crystalline compound was obtained from the CDCl3 layer upon addition of a small amount of diethyl ether. This was a 1:1 complex of benzo-30-crown-10 with imidazolium perchlorate as proven by single-crystal X-ray analysis (see Figure 1) and 1H NMR spectroscopy. In this complex the imidazolium cation is encapsulated in the macrocyclic cavity, with short nonbonding distances between NH and CH groups of the cation and ether oxygens of benzo-30-crown-10 (see Table II). These short contacts can be described as weak nonlinear hydrogen bonds. In the ORTEP view only the most linear hydrogen bond for every donating group is depicted; there are no hydrogen bonds between the imidazolium cation and perchlorate anion. Solid complexes of imidazolium perchlorate with the other investigated macrocycles could not be isolated. Extraction experiments have also been carried out with excess of a mixture of both guanidinium and imidazolium perchlorate (ratio 1:1) in the aqueous phase, and the crown ethers 3 and 7, respectively, in the organic phase. In the 1H NMR spectra only guanidinium protons were observed, indicating a selective complexation of guanidinium cations.

### Bulk Liquid Membrane Transport

The transport experiments have been carried out in a rectangular U-tube of well-defined shape and dimensions at a constant temperature of 25.0 ± 0.5 °C. A detailed description of the measurement set up and the dimensions has been published previously. Further experimental conditions are included in the Experimental Section. After completion of the transport experiments, the receiving phase was analyzed for imidazolium salt. In the case of the selectivity studies the receiving phase was analyzed for both imidazolium and guanidinium salts by a potentiometric titration. A comparison of the transport of imidazolium salts with the results of guanidinium thiocyanate transport requires that the same anion is used, in order to eliminate the anion effect. However, imidazolium thiocyanate is not commercially available, and preparation from imidazolium chloride by anion exchange resulted in an impure product. Therefore, imidazolium mesylate was prepared from imidazole and methanesulfonic acid. The transport of imidazolium mesylate with crown ether 3 as a carrier was investigated, but no transport was observed. Thiocyanate anions were introduced in the source phase by the addition of 1 equiv of lithium thiocyanate. For each of the crown ethers the flux of lithium thiocyanate has been determined depending on the pH of the source phase (eq 1). During these experiments the pH of the

<table>
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<tbody>
<tr>
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<td>2.40</td>
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<tr>
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<td>C39</td>
<td>O5</td>
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<td>2.51</td>
<td>127</td>
</tr>
<tr>
<td>C39</td>
<td>O8</td>
<td>3.15</td>
<td>2.71</td>
<td>108</td>
</tr>
</tbody>
</table>

*Estimated standard deviations less than 0.01 Å and 1° (no contribution from calculated H atom positions). Majority (A) and minority (B) position of disordered atom O23.

(26) ImH+ = imidazolium cation; Im = imidazole; A- = anion; CE = crown ether; m = membrane phase; w = aqueous phase. Equation 1 has a pK, value equal to 6.39.
Selective Transport of Polyfunctional Cations

\[ (\text{ImH}^+) = \frac{K_{ex}(1)}{(\text{Im}^+)(\text{H}^+)} (1) \]
\[ (\text{ImH}^+) + (\text{A}^-) + (\text{CE})_m \frac{K_{ex}(1)}{(\text{ImH}^+\text{CE}^-)_m} (2) \]
\[ (\text{Im}^+) + (\text{CE})_m \frac{K_{ex}(2)}{(\text{ImCE})_m} (3) \]
\[ (\text{H}^+) + (\text{A}^-) + (\text{CE})_m \frac{K_{ex}(3)}{(\text{H}^+\text{CE}^-)_m} (4) \]

Source phase is maintained at 5.1, which means that the imidazolium concentration is about 80 times larger than the imidazole concentration. The extraction equilibrium constant \( K_{ex}(1) \) will be much larger than \( K_{ex}(2) \) because the complexes formed with charged molecules are more stable than complexes of hosts with neutral molecules. Consequently, the concentration of \((\text{ImH}^+\text{CE}^-)_m\) will be much larger than the concentration of \((\text{ImCE})_m\). The effect of proton transport (eq 4) on the overall flux is dependent on the ratio between \( [K_{ex}(3)][(\text{H}^+]_m \text{ and } K_{ex}^*(1)]/[\text{ImH}^+)_m\]. At a pH of 5.1 the \([\text{H}^+]_m\) is much smaller than \([\text{ImH}^+)_m\) and \( K_{ex}(3) \) will be much smaller than \( K_{ex}(1) \) because no measurable \( [\text{H}_2\text{O}^+] \) could be detected after completion of the transport experiments. Therefore eq 4 is of minor importance, and this means that the complex concentration in the membrane phase is mainly determined by eq 2. On the basis of these arguments we can conclude that in our experiments the transport mechanism and corresponding equations are comparable with the crown ether mediated transport of guanidinium thiocyanate.

The values of transport of imidazolium thiocyanate as given in Table III are the average of at least two independent experiments with a maximum standard deviation of \( \pm 10\% \). In analogy with the extraction experiments, leakage of the carrier from the membrane is negligible. When no carrier is present in the membrane phase, the amount of imidazolium thiocyanate transported through the membrane during 24 h was below the detection limit \( \langle 2 \times 10^{-8} \text{ mol cm}^{-2} \text{ h}^{-1} \rangle \). Crown ether 3 shows the highest flux of imidazolium thiocyanate, but the flux is significantly lower than in the guanidinium thiocyanate transport (Table III). When the rates of transport of imidazolium thiocyanate are compared with the extraction efficiencies of the corresponding crown ethers given in Table I, the same tendency is observed. The highest extraction efficiencies correspond to the highest fluxes in the case of the carriers 3-5. Because of the lower limit of detection of imidazolium \( \langle 5 \times 10^{-8} \text{ M} \rangle \) a quantitative analysis of the receiving phase for the carriers 2, 6, and 7 was not possible.

The selectivity of the crown ethers was investigated in competition experiments with both guanidinium and imidazolium cations present in the source phase. These experiments are only interesting for the crown ethers 3-5 because these carriers are able to transport both cations, as was shown in independent experiments. The amounts of imidazolium transported to the receiving phase are nearly the same as in the single transport experiments, but in addition guanidinium thiocyanate is transported. At the source phase interface there will be a competition between the crown ether molecules and both the guanidinium and imidazolium salts. When the concentrations of the salts in the membrane phase are similar, the difference in transport of guanidinium and imidazolium salts is attributed to the difference in complexation constant. On the basis of the complexation equilibrium it is obvious that the complex concentration of imidazolium salts has to decrease in the presence of guanidinium salts, because these carriers are able to transport both cations, while the same explanation is valid for the transport of guanidinium salts in the attendance of imidazolium salts. It should be noted that the values of the flux for a single experiment are calculated with eq 5,

\[ J = \frac{D_m}{l} \text{[complex]}_m = \frac{D_m[\text{CE}]_m}{l} \left[ K_{ex}[\text{salt}]_w^{0.2} \right] \frac{K_{ex}[\text{salt}]_w^{0.2}}{1 + K_{ex}[\text{salt}]_w^{0.2}} \]  

of the salts in the membrane phase from 1.0 to 0.5 M. Since an enlargement of the flux in a competition experiment is not likely (only when due to a common-ion effect), the calculated values are probably too low. An explanation is presumably a saturation of the organic phase with salt at high salt concentrations in the aqueous phase (1.0 M). In that case the experimentally determined partition coefficient does not have the real value.

The selectivity of the crown ethers with regard to the guanidinium and imidazolium salts appears from the difference between the fluxes of these salts in competition experiments. The selectivity of a ligand is in no case directly related to the relative fluxes, \( J(\text{guanidinium})/J(\text{imidazolium}) \).
(imidazolium), measured in single experiments.\textsuperscript{28} The thermodynamic extraction selectivity, $K_{\text{ex}}$(guanidinium)/$K_{\text{ex}}$(imidazolium), of a ligand is a better reflection of selectivity coefficients equal to 6, 12, and 11, respectively. These calculated values of $K_{\text{ex}}$ will be a better host for imidazolium than the aliphatic ether oxygens, and therefore, the hydrogen bonds will have a smaller contribution to the free energy of complexation. Because of this and with the difference in rigidity, 5 will be a better host for imidazolium than 6. A larger reduction of the extraction efficiency is observed for the 33-membered crown ethers 4 and 7. Although the catecholic oxygens are not needed in the hydrogen bonding, the rigidity of 7 will be too large for adopting the optimal conformation needed for complexation.

Complexation Constants. The large difference in the fluxes of imidazolium and guanidinium salts through the liquid membrane can be attributed to the difference in extraction equilibrium constant $K_{\text{ex}}$. This is the product of the partition coefficient of the salt and the complexation constant. The complexation constant for imidazolium can be calculated from $K_{\text{ex}}$ when the partition coefficient of the imidazolium salt is known, assuming complexation in the membrane phase. However, the amounts of imidazolium thiocyanate in the organic phase are too low for accurate analysis.\textsuperscript{30} Since both guanidinium and imidazolium are monovalent polyfunctional organic cations, and since there is a common anion, the partition coefficients of the salts can be expected to be roughly the same. Therefore the complexation constants are the deciding factor in determining selectivity. A qualitative value of the value of the complexation constant is possible from the X-ray studies. Only on the basis of the number of strong NH--O bridges it is likely that $K_{\text{ex}}$(imidazolium) is smaller than $K_{\text{ex}}$(guanidinium). In the literature only the complexation constants of the complexes of both guanidinium and imidazolium salts with 27-crown-9-hexa-carboxylate have been reported (in water: 9000 vs 350 M$^{-1}$, respectively).\textsuperscript{15} These data are in line with the statement of a lower complexation constant of the imidazolium complex compared with the guanidinium complex.

Selectivity Coefficient. The selectivity coefficient of compound 2 is high compared with the other values due to the ring size, which discriminates imidazolium cations very well, while it possesses a good fitting cavity for guanidinium complexation. The compounds 6 and 7 also have good selectivity coefficients, but the absolute amount of transported guanidinium will be lower. Preferential transport of imidazolium in the presence of guanidinium under similar conditions probably will never take place as long as the complexation occurs by encapsulation together with hydrogen bonding.

Conclusions

The results described in this paper reveal that for encapsulation of imidazolium salts macrocyclic crown ethers are necessary with at least 30 ring atoms. The first solid complex of an imidazolium salt, encapsulated by a crown ether, is isolated. The X-ray structure shows that two NH--O hydrogen bonds and three CH--O hydrogen bonds of imidazolium are monovalent polyfunctional organic cations, and since there is a common anion, the partition coefficients of the salts can be expected to be roughly the same. Therefore the complexation constants are the deciding factor in determining selectivity. A qualitative value of the value of the complexation constant is possible from the X-ray studies. Only on the basis of the number of strong NH--O bridges it is likely that $K_{\text{ex}}$(imidazolium) is smaller than $K_{\text{ex}}$(guanidinium). In the literature only the complexation constants of the complexes of both guanidinium and imidazolium salts with 27-crown-9-hexa-carboxylate have been reported (in water: 9000 vs 350 M$^{-1}$, respectively).\textsuperscript{15} These data are in line with the statement of a lower complexation constant of the imidazolium complex compared with the guanidinium complex.

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Selective Transport of Polyfunctional Cations

Experimental Section

Melting points were determined with a Reichert melting point apparatus and are uncorrected. The $^1$H NMR spectra were recorded with a Bruker WP-80, in CDCl$_3$ with Me$_3$Si as an internal standard. Mass spectra were obtained with a Varian Mat 311A.


Imidazolium mesylate was prepared from imidazole (Janssen Chimica) and methanesulfonic acid (Janssen Chimica). Imidazole (0.44 mol) was dissolved in 100 mL of water, and 1 equiv of methanesulfonic acid was added dropwise under stirring. The solution was extracted with chloroform, and the phases were separated. The water was evaporated in vacuo. The solid was treated with ether and dried under vacuum; after filtration: yield 80%; $^1$H NMR (D$_2$O) $\delta$ 8.7 (br s, 1 H, CH-N), 7.5 (br s, 2 H, CH=N), 7.0 (d, J = 1.2 Hz, CH=CH). Anal. Calcd for C$_{8}$H$_{10}$N$_{2}$O$_3$: C, 56.3; H, 7.4; N, 13.0. Found: C, 56.8; H, 7.4; N, 12.8.

Guanidinium thiocyanate was obtained from Fluka and used without further purification. Lithium thiocyanate (ICN Biomedicals) was used without purification and contained 25.2% $^{13}$C. The imidazolium and guanidinium concentrations in the receiving phase were determined by a potentiometric titration (vide infra).

Solutions. Chloroform was purified by fractional distillation. All solutions were prepared with water of reagent grade. The solutions were stored in Pyrex glass. Tetrabutylammonium perchlorate (0.44 mol) was dissolved in 100 mL of water, and 1 equiv of perchloric acid (11.6 M). Perchlorate in the chloroform phase was calculated from the intensities in the $^1$H NMR spectra.

Potentiometric Titration. The imidazolium and guanidinium concentrations in the receiving phase were determined by a potentiometric acid/base titration with tetrabutylammonium hydroxide (0.01 M) as a titrant. First, the water was evaporated, and the residue was dissolved in 5 mL of dimethylformamide (solvent for imidazolium thiocyanate) with an ultrasonic bath at 50 °C. Subsequently, 35 mL of acetonitrile was added to dissolve guanidium thiocyanate, and the solution was titrated with a solution of tetrabutylammonium hydroxide in a mixture of 2-propanol and methanol (3/1 (v/v)). In the titrations, carried out at room temperature with an automatic titrator (Metrohm titrprocessor E 636; electrode, glass/calomel Metrohm 6.0203.100), three inflection points were found. The first equivalence point corresponds to imidazolium, the second to guanidinium and the blank (small traces of acid in dimethylformamide and acetonitrile), and the last equivalence point is attributed to the deprototated ion of imidazole. The guanidinium salt concentration was corrected for the blank.

Membrane Transport Experiment.$^{20}$ The membrane phase, chloroform containing 1.0 mL of carrier, was stirred magnetically at 200 rpm. The source phase consisted of a 0.5 M aqueous solution of lithium thiocyanate, imidazolium mesylate, and lithium thiocyanate or imidazolium mesylate and guanidium thiocyanate, respectively. The receiving phase of distilled deionized water was analyzed for the amount of imidazolium and/or guanidinium at the end of 24 h by means of a potentiometric titration.

Acknowledgment. We thank Mr. W. Lengton (Laboratory of Analytical Chemistry) for performing the potentiometric titrations.

Registry No. 1, 72216-45-6; 2, 63144-76-3; 3, 77963-50-9; 4, 104946-62-5; 5, 104946-52-3; 6, 104946-54-5; 7, 87586-46-7; imidazolium perchlorate, 61385-48-6; guanidinium thiocyanate, 589-84-0; benzo-30-crown-10-imidazolium perchlorate, 118725-01-2; imidazolium thiocyanate, 118725-02-9; imidazolium mesylate, 82200-44-8.

Supplementary Material Available. Lists of positional parameters for all atoms, anisotropic thermal parameters for heavy atoms, isotropic thermal parameters for hydrogen atoms, and lists of bond lengths and bond angles for the benzo-30-crown-10-imidazolium perchlorate (1:1) complex, as determined by X-ray diffraction (8 pages). Ordering information is given on any current masthead page.

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$^{31}$ Talam, A. G.; van Vossen, H.; Soucholte, E. J. R.; van Beilen, J.; Reinhardt, D. N. Synthesis 1986, 8, 690.


$^{33}$ Structure Determination Package; Frenz, B. A. and Associates Inc., College Station, TX, Enraf Nonius, Delft, 1983.