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SEPARATION STUDY OF STRONTIUM THROUGH A BULK LIQUID MEMBRANE CONTAINING DECYL-18-CROWN-6

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ABSTRACT

Decyl-18-crown-6 was used as an excellent ion carrier for the transport of Sr^{2+} ion through a chloroform bulk liquid membrane. In the presence of EDTA at pH 10 as a suitable stripping agent in the receiving phase, the amount of strontium transported across the liquid membrane after 4 hr is (96.5 ± 2.7)%. The selectivity of transport from the aqueous solutions containing other M^{n+} cations such as Na^+, K^+, Cs^+, Ag^+, Mg^{2+}, Ca^{2+}, Ba^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, Pb^{2+}, and Fe^{3+} ions was investigated.

Key Words: Decyl-18-crown-6; EDTA; Liquid membrane; Picric acid; Strontium transport

INTRODUCTION

Despite the absence of known biological roles for Sr^{2+} ion, separation and analysis of trace amounts of this cation in many environmental and industrial

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samples and, especially, in radioactive waste is of critical importance (1). Being chemically similar to calcium ion, ^{90}Sr may penetrate into the bone structure, where its long half-life (29 year) and penetrative radioactivity can cause severe hazards to health and life. Currently, simple and rapid methods for separation, concentration, and determination of trace levels of strontium are needed urgently to support the process development for vitally important nuclear waste management program (2–4). In many cases, it is necessary to preconcentrate and separate Sr^{2+} ion from large volumes of sample solutions containing predominant amounts of alkali and alkaline earth cations that may interfere the strontium determination (5). Among different methods used for the separation of strontium ion from the associated elements (1,6,7), solvent–solvent extraction using macrocyclic ligands has received considerable attention (8–12).

Use of liquid membranes for selective metal ion transport has received extensive attention during the past two decades (13–16). These are useful methods for assessing the partitioning of metal into and out of the organic phase and of considerable importance in medicine, water purification, and metallurgy (16,17). Macrocyclic crown ethers have been used widely to mimic biological membranes and to separate metal ions, particularly alkali and alkaline earth cations (14,18). Although several reports on the relative transport of alkaline earth cations by substituted crown ethers are available (19–25), selective membrane transport of Sr^{2+} ion has received much less attention (26,27). It should be noted that in both latter reports (26,27), di-2-ethylhexylphosphoric acid (D2EHPA) was used as strontium ion carrier in the membrane phase.

In recent years, we have used some crown ethers and their aza- and thia-substituted derivatives as suitable ion carriers for the efficient and selective transport of Zn^{2+} (28), Cu^{2+} (29,30), Pb^{2+} (31,32), Cd^{2+} (33), and Ag^{+} ions (34,35). We have also reported the successful use of some 9,10-anthraquinone and 9-anthrone derivatives for the selective transport of Ca^{2+} ion (36). In this paper, we describe a highly efficient and selective method for the transport of Sr^{2+} ions through a bulk liquid membrane containing decyl-18-crown-6 as an excellent ion carrier. The receiving phase contained EDTA at pH 10 which was found to play an important role in the transport process.

EXPERIMENTAL

Reagents

Decyl-18-crown-6 was purchased from Merck chemical company and used as received. Reagent chloroform (Merck) was used as the membrane organic solvent. Picric acid and the nitrate salts of all cations (all from Merck) were of the

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highest purity available and used without any further purification except for vacuum drying over P_2O_5. Doubly distilled deionized water was used throughout.

Apparatus

A bulk-type liquid membrane cell was used (33). The determination of strontium was carried out on a Perkin–Elmer 603 atomic absorption spectrometer (AAS) with a strontium hollow cathode lamp and a deuterium background corrector, at a wavelength of 230 nm (resonance line) using an adjusted air–acetylene flame. AAS determinations of all other cations were performed under the recommended conditions for each metal ion. pH measurements were made with a Corning ion analyzer 250 pH/mV meter equipped with a combined glass–calomel electrode.

Procedure

All transport experiments were carried out at $25 \pm 2°C$. A cylindrical glass cell (inside diameter 4.0 cm) holding a glass tube (inside diameter 2.0 cm), thus separating the two aqueous phases, was used. The inner aqueous phase (source phase) contained metal nitrate and picric acid (5 mL). The outer aqueous phase (receiving phase) contained EDTA at pH 10 (10 mL). The chloroform solution (30 mL) containing decyl-18-crown-6 was laid below these aqueous phases, and bridged the two aqueous phases. The organic layer was slowly stirred (150 rpm) by a Teflon-coated magnetic bar (3 cm × 5 mm diameter). Determination of the metal ion concentration in both aqueous phases was carried out by AAS. Reproducibility was confirmed as ± 3% or better. A similar transport experiment was carried out in the absence of the carrier for reference. Detailed conditions are included in the tables of the text.

RESULTS AND DISCUSSION

The liquid membrane operated here is shown schematically in Fig. 1. The Sr^{2+} ion is transported from the source phase to the receiving phase via a chloroform membrane. Movement of the charged species through the hydrophobic membrane is accomplished by the presence of the host carrier decyl-18-crown-6, ion paired with picrate as a suitable counter anion. After complexation of the cation with carrier on the left side of the membrane, the complex diffuses down its concentration gradient. On the right side of the membrane, the metal ion is released into the receiving phase via formation of a

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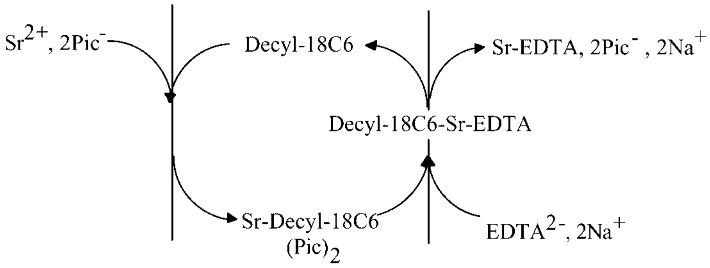

Figure 1. Liquid membrane system for transport of Sr^{2+} ions.

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ternary complex (carrier–metal ion–EDTA). The free carrier diffuses back across the liquid membrane and the cycle starts again. The net result is the transport of Sr^{2+} ion from the aqueous source phase to the aqueous receiving phase across the bulk of the organic phase (the membrane).

Macrocyclic crown ethers are well known as effective organic ligands for alkali and alkaline earth cations (37–39). Strontium ion with an ionic diameter of 2.4 Å (40) possesses a proper size to fit inside the cavity of 18-crown-6 with a diameter of 2.9 ± 0.3 Å (41) and form a fairly stable 1:1 complex (38,39). The substoichiometric extraction of Sr^{2+} ion with 18-crown-6 in the presence of picrate as a suitable counter anion is reported in the literature (10,42), despite the fact that cations such as Ba^{2+} and K^{+} ions possess a high tendency for complex formation with 18-crown derivatives (38,39). Thus, in this work we used decyl-18-crown-6 as a potential ion carrier for the liquid membrane transport of Sr^{2+} ion in the presence of picric acid. The substitution of a decyl group on the 18-crown-6 ring provided the macrocycle with enough lipophilicity to minimize its bleeding into the two aqueous phases. It is noteworthy that the picrate ion is well known to act as an excellent counter anion for the solvent–solvent extraction of a variety of metal ions with macrocyclic ligands (42–45).

In preliminary experiments, we measured the transport rates of different alkali and alkaline earth cations through a chloroform membrane containing decyl-18-crown-6 as carrier. The highest transport rate was obtained with Sr^{2+} ion. Moreover, the Sr^{2+} ion transport rate was found to increase tremendously while using some picric acid in the source phase and, especially, EDTA at pH 10 as a stripping agent, in the receiving phase. Thus, in the next steps, the experimental variables such as concentration of picric acid in the source phase, concentration of decyl-18-crown-6 in the organic membrane, concentration of EDTA at pH 10 in the receiving phase, and the time of transport were optimized, in order to achieve the highest efficiency in the transport of a $5.7 \times 10^{-5}\,M$ solution of Sr^{2+} across the membrane system used.

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Table 1. Effect of Picric Acid Concentration in the Source Phase on Strontium Transport

Concentration of Picric Acid (*M*)	Percentage Transport into Receiving Phase	Percentage Remaining in Source Phase
0	51	25
1.0×10^{-4}	68	14
2.0×10^{-4}	73	11
2.5×10^{-4}	78	9
5.0×10^{-4}	81	5
1.0×10^{-3}	85	5
2.0×10^{-3}	92	4
2.5×10^{-3}	96	2
5.0×10^{-3}	96	2

Conditions: Source phase, 5 mL of $5.7\times10^{-5}\,M$ Sr^{2+} and varying concentration of picric acid; membrane phase 30 mL of $5.9\times10^{-3}\,M$ decyl-18-crown-6 in chloroform; receiving phase, 10 mL of $5\times10^{-2}\,M$ EDTA at pH = 10; time of transport, 4 hr.

Table 2. Effect of Decyl-18-crown-6 Concentration in the Membrane Phase on Strontium Transport

Concentration of Decyl-18-crown-6	Percentage Transport into Receiving Phase	Percentage Remaining in Source Phase
0	<2	95
$2.2\times10^{-4}\,M$	48	15
$5.9\times10^{-4}\,M$	52	9
$6.7\times10^{-4}\,M$	59	8
$1.2\times10^{-3}\,M$	76	6
$2.4\times10^{-3}\,M$	92	3
$5.9\times10^{-3}\,M$	96	1
$1.2\times10^{-2}\,M$	96	1

Conditions: Source phase, 5 mL of $5.7\times10^{-5}\,M$ Sr^{2+} and $5.0\times10^{-3}\,M$ picric acid; membrane phase, 30 mL of varying concentration of decyl-18-crown-6 in chloroform; receiving phase, 10 mL of $5.0\times10^{-2}\,M$ EDTA at pH = 10; time of transport, 4 hr.

The influence of picric acid concentration in the source phase on the transport of strontium was studied and the results are shown in Table 1. It is clear that the percentage of transported strontium increases with increasing concentration of picric acid. Maximum transport occurs when the picric acid

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Table 3. Effect of EDTA Concentration in the Receiving Phase on Strontium Transport

Concentration of EDTA	Percentage Transport into Receiving Phase	Percentage Remaining in Source Phase
0	7	48
1×10^{-3}	35	28
5×10^{-3}	63	12
1×10^{-2}	79	7
2×10^{-2}	86	3
3×10^{-2}	96	2
5×10^{-2}	97	1

Conditions: Source phase, 5 mL of $5.7\times10^{-5}\,M$ Sr^{2+} and $5.0\times10^{-3}\,M$ picric acid; membrane phase, 30 mL of $5.9\times10^{-3}\,M$ decyl-18-crown-6 in chloroform; receiving phase, 10 mL of varying concentration of EDTA at pH = 10; time of transport, 4 hr.

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concentration is about $2.5\times10^{-3}\,M$. Further increase in the concentration of picric acid has no measurable effect on the transport efficiency. It is interesting to note that the uphill transport of Sr^{2+} cannot be afforded in the absence of picric acid.

As it was mentioned before, the permeability of the membrane system is expected to be critically dependent on the nature of macrocycle used as carrier in the organic membrane phase. Due to the increased lipophilicity and/or Sr^{2+} complex stability of decyl-18-crown-6 with respect to other macrocyclic ligands such as 18-crown-6, dicyclohexyl-18-crown-6, dibenzo-18-crown-6, aza-18-crown-6, 15-crown-5, and benzo-15-crown-5, its use as Sr^{2+} ion carrier was found to result in the highest transport efficiency in the series.

The effect of the concentration of decyl-18-crown-6 on the transport efficiency of strontium was also studied and the results are summarized in Table 2. As seen, the percentage transport of Sr^{2+} increases with an increase in carrier concentration in chloroform. Maximum transport occurs at a concentration of about $6.0\times10^{-3}\,M$. A further excess of the ligand has no considerable effect on the transport efficiency.

As expected, it was found that the nature and composition of the ligand used as scavenger for the transported metal ion in the receiving phase could have a significant effect on the efficiency and selectivity of membrane transport (28–33). The use of EDTA at pH 10 as a stripping agent in the receiving phase was found to cause a rather large enhancement in the efficiency and selectivity of strontium transport. This is because of the strong interaction between Sr^{2+} and EDTA which can easily strip the cation from its complex with the carrier in the membrane phase. The optimum concentration of EDTA in the receiving phase

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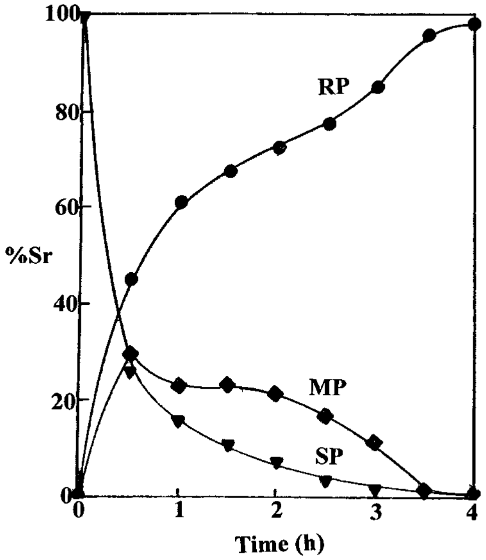

Figure 2. Time dependence of silver transport. Conditions similar to those mentioned in Table 4: (SP) source phase, (MP) membrane phase, (RP) receiving phase.

was investigated (Table 3) and found to be about $3 \times 10^{-2}\,M$. It is noteworthy that the absence of a metal ion acceptor in the receiving phase resulted in no promising transport of strontium ion even in much longer periods of time.

Figure 2 shows the time dependence of Sr^{2+} ion transport through the liquid membrane designed under optimal experimental conditions. It is obvious that the extraction of strontium ion from the aqueous source phase into the organic membrane occurs very rapidly, so that some 90% extraction of Sr^{2+} is obtained after approximately one and half hour. However, the release of strontium ion into the aqueous receiving phase occurs at a much slower rate. Thus, it seems reasonable to assume that the release of the Sr^{2+} ion from the crown ether complex is the rate determining step of the membrane transport. It was found, under the optimum experimental conditions, that >96% of total strontium was transported after 4 hr. The reproducibility of strontium transport was investigated and the per cent metal ion transported after 4 hr obtained from 10 replicate measurements was found to be 96.5 ± 2.7.

In Table 4 are listed the per cent of transported Sr^{2+} and M^{n-1} cations, which were present with strontium in equimolar concentrations, into the receiving phase. As it is seen, with the exception of potassium and barium ions, the interfering effect of all other cations used on the strontium transport is quite low. However, some 20% co-transport of K^+ and Ba^{2+} is presumably due to more

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Table 4. Amount of Cation Transported from Various Cation Mixtures Through the Membrane

Cation	Percentage Transport into Receiving Phase	Percentage Remaining in Source Phase
Mixture 1		
Sr^{2+}	94	3
Na^{+}	2	89
K^{+}	21	67
Mixture 2		
Sr^{2+}	96	3
Cs^{+}	8	79
Mg^{2+}	1	91
Mixture 3		
Sr^{2+}	94	4
Ca^{2+}	1	87
Ba^{2+}	18	71
Mixture 4		
Sr^{2+}	96	3
Ni^{2+}	2	92
Cd^{2+}	1	2
Mixture 5		
Sr^{2+}	94	4
Co^{2+}	2	88
Cu^{2+}	2	94
Mixture 6		
Sr^{2+}	95	3
Pb^{2+}	4	91
Zn^{2+}	6	92
Mixture 7		
Sr^{2+}	97	2
Ag^{+}	2	89
Fe^{3+}	4	83

Conditions: Source phase, 5 mL of $5.7 \times 10^{-5} M$ Sr^{2+} and $5.0 \times 10^{-3} M$ picric acid; membrane phase, 30 mL of decyl-18-crown-6 in chloroform; receiving phase, 10 mL of $5.0 \times 10^{-2} M$ EDTA at pH = 10; time of transport, 4 hr.

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or less convenient fit of these cations into the cavity of decyl-18-crown-6 and, consequently, the formation of relatively stable complexes in the membrane phase (38,39). However, it is noteworthy that, in the membrane systems, the balance of the rates between uptake and release steps often plays the most important role for the ion-selectivity.

CONCLUSIONS

The results show that, by using picric acid in the source phase and EDTA at pH 10 in the receiving phase, strontium ions can be transported through the bulk liquid membrane containing decyl-18-crown-6 as carrier, via formation of a strontium–crown–picrate complexed ion pair. The simplicity, low cost, short time, excellent efficiency, and high degree of selectivity for Sr^{2+} ion transport obtained by the membrane system studied, demonstrate its potential applicability to selective removal, concentration, and purification of strontium from its mixture.

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