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Separation Study of Palladium through a Bulk Liquid Membrane Containing Thioridazine·HCl and Oleic Acid

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ABSTRACT

A chloroform membrane system containing a given mixture of thioridazine·HCl and oleic acid was applied for the uphill transport of Pd^{2+} ions. In an HCl medium of pH 2 the ligand can form a stable complex with Pd(II) which is readily extractable in the membrane phase. In the presence of nitrite ion as a suitable stripping agent in the receiving phase, the amount of palladium transported across the liquid membrane after 120 minutes is 96.3 ± 2.5%. The selectivity and efficiency of Pd^{2+} transport from aqueous solutions containing other cations such as K^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Pb^{2+}, Cd^{2+}, Fe^{3+}, Cr^{3+}, and Al^{3+} ions were investigated. It was found that none of these cations interfere with palladium transport, even at a M^{n+}/Pd^{2+} molar ratio of >100.

Key Words. Liquid membrane; Cooperative palladium transport; Thioridazine·HCl; Oleic acid; Nitrite

INTRODUCTION

Prior to 1930, palladium was used only in moderate amounts, principally in dental alloys and jewelry (1). With increased and reliable supplies available, it has found widespread use in different areas of science and technology, including coating agents (2), brazing alloys (3), petroleum (4), and electrical (5, 6) industries as well as in a wide variety of catalytic chemical

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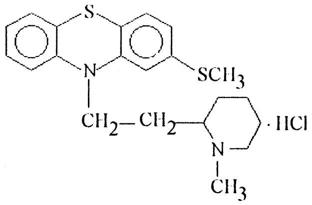
reactions (7). Thus, due to its increasing use on one hand, and the toxicity of Pd(II) compounds to mammals, microflora, fish, and higher plants on the other (8, 9), the separation, concentration, and determination of palladium is of special interest.

Solvent extraction has been widely used for separation of the platinum metals (10–13). However, increasing interest has been focused in recent years on liquid membrane methods for the selective separation and concentration of Pd(II) ions (14–28). These are useful methods for assessing the partitioning of metal into and out of the organic phase and are of considerable importance in medicine, water purification, and metallurgy (29, 30). The liquid membrane systems used for palladium separation are of the bulk (14), emulsion (15–22), and supported types (23–28).

In recent years we have been involved in the design and preparation of bulk liquid membrane systems containing some crown ether derivatives for the selective and efficient transport of a variety of metal ions such as Cu^{2+} (31, 32), Zn^{2+} (33, 34), Hg^{2+} (35), Pb^{2+} (36, 37), Cd^{2+} (38), and Ag^{+} (39). In this paper we report a highly selective liquid membrane system containing a mixture of thioridazine·HCl (TRHCl) and oleic acid for the uphill transport of the Pd(II) ion. The receiving phase contains the nitrite ion which was found to play an important role in the transport process.



TRHCl

EXPERIMENTAL

Reagents

Reagent-grade thioridazine·HCl (TRHCl) and other phenothiazine compounds (all from Aldrich) were used as received. HPLC-grade chloroform (Merck) was used as the membrane organic solvent. All other chemicals used were of the highest purity available from either Merck or Fluka and used without further purification except for vacuum drying over P_2O_5 to get rid of any adsorbed moisture. Doubly distilled deionized water was used throughout.

Apparatus

A bulk-type liquid membrane cell was used in this study (38). The atomic absorption spectrophotometer used for the measurement of metal concentration in the aqueous phases was a Shimadzu AA-670 instrument. The atomic absorption measurements were carried out under the recommended conditions for each metal ion. The pH measurements were made with a Metrohm 692 pH-meter using a combined glass electrode.

Procedure

All transport measurements were carried out at ambient temperature (23–25°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 palladium chloride (5 mL, 5.0×10^{-4} M). The outer aqueous phase (receiving phase) contained sodium nitrite (10 mL, 3.0×10^{-2} M). The pH of the source phase was adjusted to 2.0 with hydrochloric acid. The chloroform solution (20 mL) containing 5.0×10^{-4} M TRHCl and 5.0×10^{-2} M oleic acid (OA) laid below these aqueous phases, and bridged the two aqueous phases. The organic layer was slowly stirred by a Teflon-coated magnetic bar (2 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 cooperative carriers for reference. Detailed conditions are included in the graphs and tables of the text.

RESULTS AND DISCUSSION

It is well known that phenothiazine compounds can form stable colored complexes with Pd(II) in hydrochloric acid media (40–42) which can be successfully used for the spectrophotometric determination of palladium (43–45). The interaction between palladium(II) (as $PdCl_4^{2-}$) and TRHCl is believed to involve protonation of the drug followed by H-bonding between the protonated nitrogen and one of the chlorine atoms on the palladium complex anion.

Since most of the phenothiazine derivative–palladium complexes are readily extractable in lipophilic liquids (46–48), we decided to use TRHCl as a potential ion carrier for the selective transport of Pd(II) ions through a chloroform liquid membrane. The preliminary experiments showed that a serious problem associated with the use of TRHCl as carrier in the organic membrane is its substantial bleeding from the membrane phase into the aqueous phases. However, as has been noticed elsewhere (31, 33, 38, 39, 48), the addition of a long-chain fatty acid such as oleic acid not only significantly reduces the degree of carrier loss but also could have a cooperative effect in the uphill transport of Pd^{2+} ions through the liquid membrane. It is interesting to note that oleic acid and other fatty acids have already been used for the concentration and determination of phenothiazine derivatives (47, 49, 50) and their palladium complexes (47). The main advantages of this system with respect to more common amine extractant carriers (17, 27, 51) include the increased selectivity of the system as well as the reduced time of transport of palladium as $PdCl_4^{2-}$.

The membrane system operated here is illustrated schematically in Fig. 1. After complexation of the carrier with $PdCl_4^{2-}$ on the left side of the membrane, the complex formed 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 stable $[Pd(NO_2)_2Cl_2]^{2-}$ complexed ion. At this stage the carrier diffuses back across the membrane. The net result is the transport of palladium from the aqueous source phase to the aqueous receiving phase across the bulk of the organic phase (the membrane). Actually, the transport process of palladium through the liquid membrane is illustrated by the following equations:

1. TRHCl in the membrane phase reacts with $PdCl_4^{2-}$ to form a ML_2X_4 complex (43):

$$2TRHCl_{(MP)} + PdCl_4^{2-}{}_{(SP)} = [Pd(TRH)_2Cl_4]_{(MP)} + 2Cl^-_{(SP)} \tag{1}$$

2. Nitrite ion in the stripping solution reacts with $Pd(TRH)_2Cl_4$ to strip palladium into the receiving phase:

$$Pd(TRH)_2Cl_{4(MP)} + 2NO^-_{2(RP)} = Pd(NO_2)_2Cl^{2-}_{2(RP)} + 2TRHCl_{(MP)} \tag{2}$$

The membrane system was first optimized with respect to the relative concentrations of TRHCl/OA by keeping the amount of one of the constituents constant and varying the concentration of the other one. The results are shown in Fig. 2. As seen, while TRHCl and OA alone can transport about 47 and 1% of palladium after 120 minutes, respectively, a given mixture of them (about 5.0×10^{-4} M TRHCl and 5.0×10^{-2} M OA) is much more effective in the efficient transport of palladium. A possible reason for this cooperative behavior could be the existence of some proton donor–proton acceptor interactions between the fatty acid (as donor) and the nitrogen atoms of the phenothiazine derivative used (as acceptor) (33). Another possibility is that the OA forms an

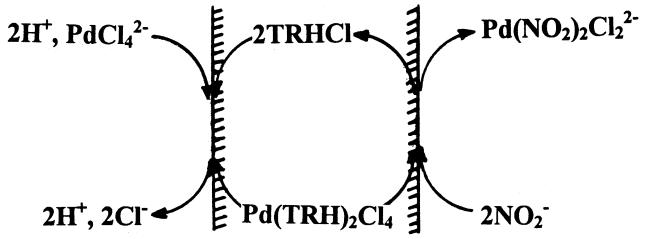


FIG. 1 Liquid membrane system for transport of Pd^{2+} ions.

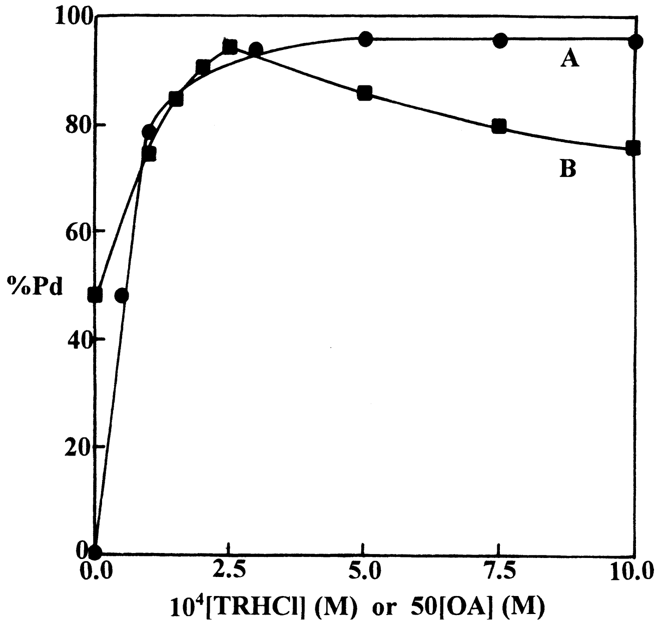


FIG. 2 Effect of TRHCl (A) and OA (B) concentration on palladium transport. Conditions: source phase, 5 mL of 5.0 × 10^{-4} M $PdCl_2$ at pH 2; membrane phase, 20 mL of 5.0 × 10^{-2} M of OA and varying concentration of TRHCl (A) or of 5.0 × 10^{-4} M TRHCl and varying concentration of OA (B) in chloroform; receiving phase, 10 mL of 3.0 × 10^{-2} M $NaNO_2$; time of transport, 120 minutes.

inverse micelle (52, 53), inside of which the carrier molecules may be trapped. The TRHCl may then be easily transported across the membrane inside the inverse micelle. It should be noted that the uphill cooperative transport of $PdCl_4^{2-}$ with TRHCl can also occur in the presence of other fatty acids such as palmitic acid and stearic acid, but to a somewhat lower extent.

Permeability of the membrane system for Pd^{2+} ion with respect to different stripping agents in the receiving phase was investigated, and the results are summarized in Table 1. 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 palladium transport. The use of NO_2^- ion as the stripping ligand in the receiving phase caused a large enhancement in the efficiency and selectivity of Pd^{2+} ions, while the presence of other receiving agents such as thiourea, SCN^-, $S_2O_3^{2-}$, and EDTA resulted in a pronounced decrease in the efficiency of palladium transport. The maximum concentration of sodium nitrite in the receiving phase was investigated (Fig. 3, Curve A) and found to be in the 3.0 × 10^{-2} to 5.0 × 10^{-2} M range. It is interesting to note that the absence of a metal ion receptor in the receiving phase resulted in no measurable palladium transport even for much longer time periods.

TABLE 1
Effect of the Nature of Stripping Agent in the Receiving Phase on Palladium Transport[a]

Stripping agent	Percentage transported into receiving phase	Percentage remaining in source phase
$NaNO_2$	95	0
Thiourea	41	17
NaSCN	28	27
$Na_2S_2O_3$	12	24
EDTA	6	6
Sodium tartrate	0	3

[a] Conditions: source phase, 5 mL of 5.0×10^{-4} M of $PdCl_2$; membrane phase, 20 mL of 5.0×10^{-4} M TRHCl and 5.0×10^{-2} M OA in chloroform; receiving phase, 10 mL of 3.0×10^{-2} M of stripping agent; time of transport, 120 minutes.

The influence of the pH of the source phase on the transport efficiency of Pd(II) was studied in the 0.5 to 4.0 pH range, adjusted by hydrochloric acid, and the results are shown in Fig. 3 (Curve B). Although the complex $Pd(TRH)_2Cl_4$ is reported to be very stable in strongly acidic media (42, 43),

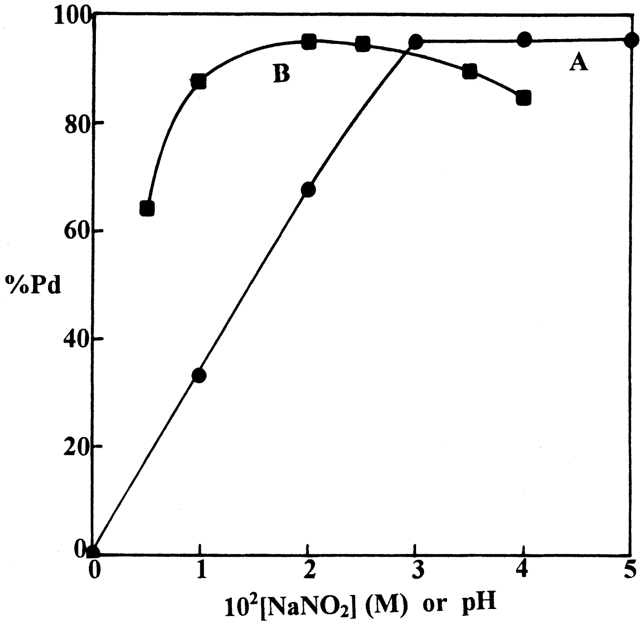


FIG. 3 Effect of $NaNO_2$ concentration in the receiving phase (A) and pH of source phase (B) on palladium transport. Conditions: source phase, 5 mL of 5.0×10^{-4} M $PdCl_2$ and pH 2 (A) or/and varying pH (B); membrane phase, 20 mL of 5.0×10^{-4} M of TRHCl and 5.0×10^{-2} M of OA in chloroform; receiving phase, 10 mL of varying concentration of $NaNO_2$ (A) or of 3.0×10^{-2} M $NaNO_2$ (B); time of transport, 120 minutes.

the increased concentration of Cl^- ion will result in diminished extraction of the complex into the membrane phase (see Eq. 1). Thus, maximum palladium transport occurs at a pH of about 2 (Fig. 3 Curve B), while at lower or higher pH values some decreased transport efficiency is observed.

Figure 4 shows the time dependence of Pd(II) transport through the liquid membrane under the optimum experimental conditions. It is quite obvious that the extraction of palladium from the aqueous source phase into the organic membrane occurs very rapidly, so that extraction seems to be almost complete after approximately 40 minutes. However, the release of Pd(II) into the aqueous receiving phase occurs at a slower rate. It was confirmed that about 95% of palladium ion was transported from the source phase into the receiving phase after 120 minutes under optimal conditions. The reproducibility of palladium transport was investigated, and the percent of metal ion transported after 120 minutes as obtained from 10 replicate measurements was found to be 96.3 ± 2.5.

The percentage transport of Pd^{2+} and M^{n+} cations, which were initially present with palladium in a M^{n+}/Pd^{2+} ratio of 100 in the source phase, into the receiving phase are listed in Table 2. As can be seen, none of the alkali, alkaline earth, transition, and heavy metal ions tried has a measurable interfering effect on the uphill transport of palladium ion. It is noteworthy that the M^{n+} cations used revealed no measurable interfering effect even when the excess amount of $M^{n+}/Pd^{2+} \geq 1000$ was employed.

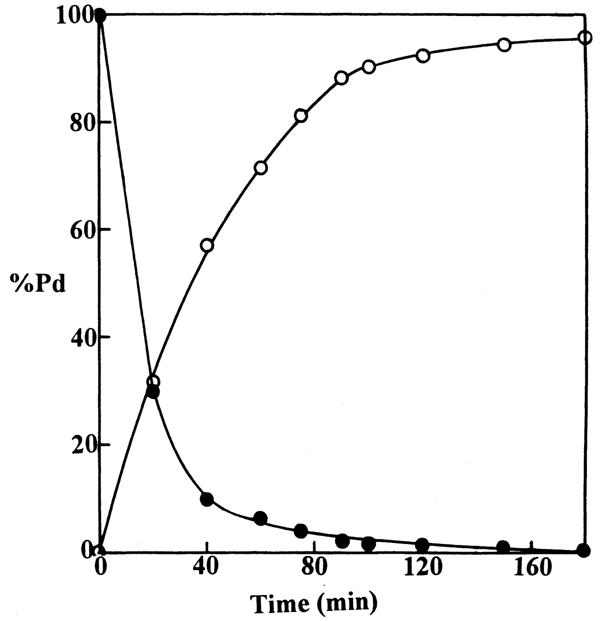


FIG. 4 Time dependence of palladium transport: (○) transported; (●) remaining. Conditions similar to those mentioned in Table 2.

TABLE 2

Amount of Cation Transported from Different Cation Mixtures Through the Membrane[a]

Cation	Percentage transported into receiving phase	Percentage remaining in source phase
Mixture 1:		
Pd^{2+}	95	0
K^+	0	94
Mg^{2+}	0	99
Ca^{2+}	0	99
Sr^{2+}	0	100
Ba^{2+}	0	99
Pb^{2+}	0	98
Mixture 2:		
Pd^{2+}	93	0
Co^{2+}	0	98
Ni^{2+}	0	97
Cu^{2+}	0	95
Zn^{2+}	3	97
Cd^{2+}	1	97
Fe^{3+}	1	98
Cr^{3+}	0	90
Al^{3+}	0	93

[a] Conditions: source phase, 5 mL of 5.0×10^{-4} M of Pd^{2+} and 5.0×10^{-2} of each cation at pH 2; membrane phase, 20 mL of 5.0×10^{-4} M of TRHCl and 5.0×10^{-2} M OA in chloroform; receiving phase, 10 mL of 3.0×10^{-2} M $NaNO_2$; time of transport, 120 minutes.

The interfering effect of alkali and alkaline earth cations was checked because of previous reports on the use of phenothiazine compounds as carrier ions in the transport of such metal ions as calcium (54) and sodium (55) in biological media. Moreover, it is interesting to note that although $PtCl_6^{2-}$ can form a strong ion pair with TRHCl, the resulting aggregate is not extractable in chloroform as the membrane phase. On the other hand, the low stability of a $SnCl_4^{2-}$ complex (56) and the electroneutrality or reduced charge of the chloride complexes of Sb (57) will result in increased selectivity of the system used for Pd over these cations.

CONCLUSION

Transport of palladium through a chloroform–TRHCl–OA bulk liquid membrane was studied. The mechanism of transport was discussed. The optimum conditions of transport were found at pH 2 in the feed solution, 5.0×10^{-4} M TRHCl and 5.0×10^{-2} M OA in the liquid membrane, and 3.0×10^{-2} M $NaNO_2$ in the stripping solution. The simplicity, excellent efficiency, and high degree of selectivity for Pd^{2+} ion transport shown by the membrane system demonstrate its potential applicability to the selective separation, concentration, or purification of palladium from mixtures.

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