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A kinetic study of mercury(II) transport through a membrane assisted by new transport reagent

Mehmet Karakus¹, Hamza Korkmaz Alpoguz^{1*}, Ahmet Kaya¹, Nurcan Acar², Ahmet Orhan Görgülü³ and Mustafa Arslan^{3,4}

Abstract

Background: A new organodithiophosphorus derivative, namely O-(1,3-Bispiperidino-2-propyl)-4-methoxy phenyldithiophosphonate, was synthesized and then the kinetic behavior of the transport process as a function of concentration, temperature, stirring rate and solvents was investigated.

Results: The compound **1** was characterized by elemental analysis, IR, ¹H and ³¹P NMR spectroscopies. The transport of mercury(II) ion by a zwitterionic dithiophosphonate **1** in the liquid membrane was studied and the kinetic behavior of the transport process as a function of concentration, temperature, stirring rate and solvents was investigated. The compound **1** is expected to serve as a model liquid membrane transport with mercury(II) ions.

Conclusion: A kinetic study of mercury(II) transport through a membrane assisted by O-(1,3-Bispiperidino-2-propyl)-4-methoxy phenyldithiophosphonate was performed. It can be concluded that the compound **1** can be provided a general and straightforward route to remove toxic metals ions such as mercury(II) ion from water or other solution.

Background

Dithiophosphorus Derivatives have been a subject of intensive study due to having an important role in medicine, agricultural and industrial application fields in the last decades [1-15]. They have been utilized as additives in lubricant oils, solvent-extraction reagents for metals, and flotation agents for mineral ores, insecticides and pesticides [1-5]. While many dithiodiphosphonates and their derivatives such as thiophosonyl disulfane have been synthesized by a ring opening reaction of Lawesson reagent's or its analogues and alcohols, zwitterionic dithiophosphonate and thiophosonyl disulfane type compounds are rare. Dithiophosphonates can be oxidized to bis(thiophosonyl)disulfane by Cu(II) ions or iodine [7]. Recently, a few researches have focused on the zwitterionic dithiophosphonates [6]. Dithiophosphonates can be oxidized to bis(thiophosonyl)disulfane by Cu(II) ions or iodine [7]

In this paper, we reported the synthesis of O-(1,3-Bispiperidino-2-propyl)-4-methoxy phenyldithiophosphonate and the kinetic behavior of the transport process as

a function of concentration, temperature, stirring rate and solvents was also investigated. The compound **1** was characterized by elemental analysis, IR and ¹H-NMR and ³¹P-NMR spectroscopies.

Results and discussion

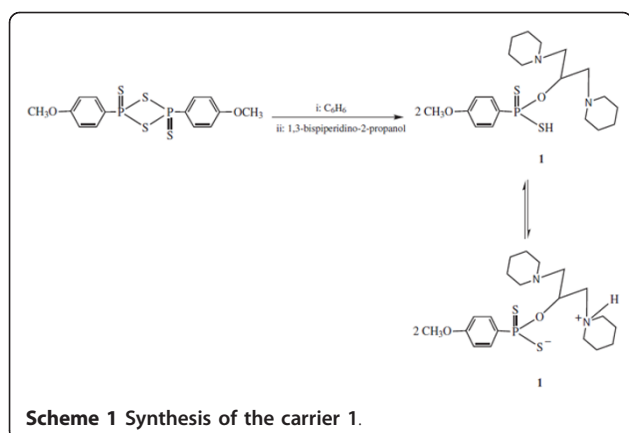
The reaction of 2,4-bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane-2,4-disulfide with 1,3-bispiperidino-2-propanol gave rise to the formation **1** which was previously synthesized (Scheme 1) [16]. The spectroscopic data of the compound **1** was remained. The compound was obtained in high yield and was characterized by elemental analysis, IR, ¹H-NMR and ³¹P-NMR spectroscopies. Although dithiophosphonates were usually obtained as liquid product, the compound **1** was obtained as solid product due to zwitterionic character in which the H atom of the initially formed P-SH group transferred to the piperidino group. The IR spectra of **1** showed its characteristic bands at 671 cm⁻¹ for n(PS)_{asym} and 548 cm⁻¹ for n(PS)_{sym} stretchings.

The ¹H-NMR spectra of **1** indicated that the phenyl protons displayed doublet at 8.13-8.08 ppm (³J_{PH} = 14.19 Hz, J_{HH} = 8.81 Hz) and 6.90-6.87 ppm (⁴J_{PH} = 2.45 Hz, J_{H, H} = 8.81 Hz), respectively. The spectra showed the expected signals for piperidino and

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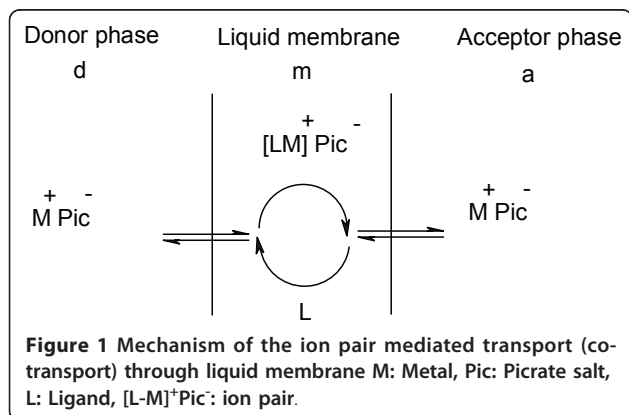


methylene protons. However, the proton on N atom was not observed in the spectra of ^1H NMR. The ^{31}P -NMR spectra of **1** displayed a quartet at 112.73 ppm because of the coupling with hydrogen nuclei and the presence of two isomer in solution (see scheme 1).

Transport studies

In our previous report [16], the transport of Cu(II) ions from aqueous phase was carried out by using the compound **1** as the carrier. In this work, the transport of mercury(II) ion by the zwitterionic dithiophosphonate **1** in the liquid membrane was studied and the kinetic behavior of the transport process as a function of concentration, temperature, stirring rate and solvents was investigated.

The mechanism of the ion pair mediated transport (co-transport) is given in Figure 1. L represents the carrier **1**. At the interface between donor and membrane, metal picrate ion pair forms complex with ligand, then the $[\text{LM}]^+\text{Pic}^-$ complex diffuses through the membrane. At the interface between membrane and acceptor, the carrier ion pairs are decomplexed and M^+Pic^- is liberated into the acceptor phase. Finally, the ligand carrier diffuses back across the membrane aqueous boundary



layers. The variation of the metal picrate concentration with time was directly measured in both the donor (C_d) and acceptor (C_a) phases. In the experiments, the variation of picrate ion concentration with time was directly measured in both donor (C_d) and acceptor phases (C_a). The corresponding change of picrate ion concentration in the membrane phase was determined from the material balance between the phases.

For practical reasons, the dimensionless reduced concentrations were used:

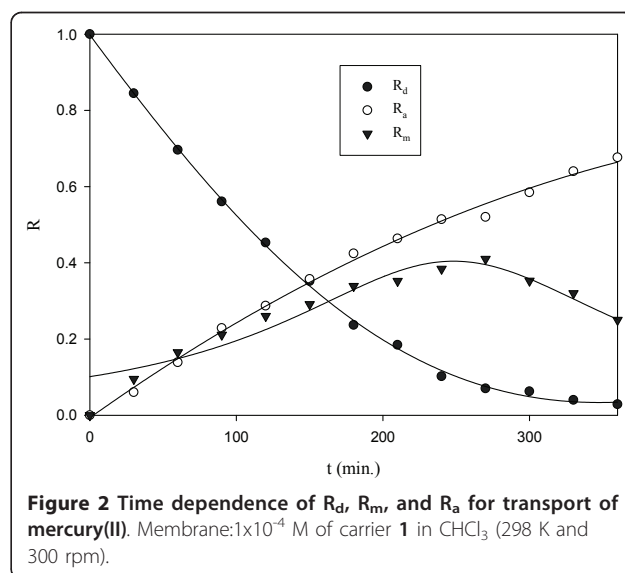
$$R_d = \frac{C_d}{C_{d0}} \quad R_m = \frac{C_m}{C_{d0}} \quad R_a = \frac{C_a}{C_{d0}} \quad (1)$$

where C_{d0} is the initial mercury(II) ion concentration in the donor phase, while C_d , C_m and C_a represents the mercury(II) ion concentration in donor, membrane and acceptor phases, respectively. The material balance with respect to the reduced concentrations can be expressed as $R_d + R_m + R_a = 1$. From this expression, the kinetic behavior of the consecutive irreversible first order reactions can be described as follows;



where k_1 and k_2 are the apparent membrane entrance and exit rate constants, respectively. The kinetic scheme for consecutive reaction systems and the kinetic parameters of k_1 and k_2 from the obtained data were calculated by fitting equations as described in the previous studies [17-24].

The variation of the reduced concentration of mercury (II) ion through the liquid membrane with 1×10^{-4} M of carrier **1** in CHCl_3 at 300 rpm and 25°C is presented in Figure 2. The observed experimental results reveal that R_d decreases exponentially with time, accompanied by a



simultaneous increase of R_a , whereas R_m presents a maximum at intermediate times.

Effect of Carrier Concentration in Membrane on Transport of Mercury(II) Ions

The transport experiments were carried out at three different initial carrier **1** concentrations 1×10^{-6} , 1×10^{-5} , and 1×10^{-4} M in CHCl_3 at 298 K and 300 rpm. The obtained kinetic parameters for the effect of concentration of carrier **1** are presented in Table 1. It was found that the initial carrier concentration influences the kinetic constants, as well as flux values and the results are in full agreement with previously obtained results [17-24]. It can be seen that both kinetic constants k_1 and k_2 or fluxes are dependent on the carrier concentration and increases steadily with the initial carrier concentration as shown in Figure 3. In addition, a blank experiment was performed with no present carrier in the membrane. There was no evidence of the movement of the mercury(II) ions through the liquid membrane in the blank experiment. When the carrier was utilized, the transport of mercury(II) ions through the liquid membrane was performed.

Effect of Temperature on Transport of Mercury(II) Ions

The effect of temperature on the transport of mercury (II) ions through the liquid membrane containing 1×10^{-4} M of carrier **1** in CHCl_3 was examined at 293, 298, 303, and 308 K (300 rpm). The experimental results are collected in Table 2. It is quite obvious that k_1 and k_2 increases with an increase in the temperature. Table 2 also shows that t_{\max} and R_m^{\max} decreases with an increase of temperature.

The activation energy was calculated from plot of the maximum membrane exit flux (J_a^{\max}) versus ($1/T$) at 300 rpm (Equation 3), as presented in Figure 4.

$$\ln(J) = \ln(A) - \frac{E_a}{R} \left(\frac{1}{T} \right) \quad (3)$$

The activation energy value for carrier **1** in the liquid membrane was found to be 1.36 kcal/mol by using the equation 3. As known, activation energy values are quite low for diffusion-controlled processes, whose rate constants are strongly affected by temperature [25]. It was pointed out that the activation energies of diffusion-controlled processes are lower than 10 kcal/mol [25]. The

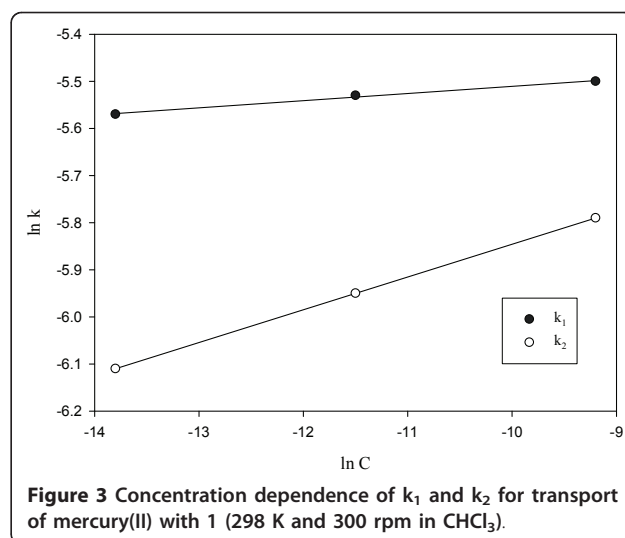


Figure 3 Concentration dependence of k_1 and k_2 for transport of mercury(II) with **1** (298 K and 300 rpm in CHCl_3).

calculated activation energy for carrier **1** shows that the transport of mercury(II) ion is diffusion-controlled processes.

Effect of Stirring Rate on Transport of Mercury(II) Ions

To achieve effective mercury(II) transport, it is necessary to explore the effect of stirring speed on the transport process. In the present investigation, the stirring rate of the membrane phase was carried out at three different stirring rate, 200, 300, and 400 rpm at 298 K when the carrier **1** concentration was 1×10^{-4} M in CHCl_3 . The results are given in Table 3 and indicate that the stirring rate affects the transport rate of mercury(II) through the liquid membrane. According to these results, the flux increases with increasing stirring rate due to decrease of the thickness of the diffusion boundary layers at both interfaces of the membrane.

Effect of Solvent on Transport of Mercury(II) Ions

The present work was to investigate the physicochemical approach to co-transport of mercury(II) transport through a liquid membrane containing carrier **1**. Therefore, the effect of solvents on the transport process was studied under the same conditions, and the results obtained with CH_2Cl_2 and CCl_4 are presented in Table 4, along with analogous results for CHCl_3 . It has been observed that the membrane entrance and exit rate constants are found to vary in the order

Table 1 The kinetic parameters for mercury(II) ions at different carrier **1** concentrations in CHCl_3 (298 K and 300 rpm)

| Concentration (M) | $k_1 \times 10^3$ (min^{-1}) | $k_2 \times 10^3$ (min^{-1}) | R_m^{\max} | t_{\max} (min) | $J_d^{\max} \times 10^3$ (min^{-1}) | $J_a^{\max} \times 10^3$ (min^{-1}) | % Transport |
|--------------------|---|---|--------------|------------------|--|--|-------------|
| 1×10^{-6} | 3.81 | 2.21 | 0.47 | 340.17 | -1.04 | 1.04 | 57.56 |
| 1×10^{-5} | 3.95 | 2.61 | 0.45 | 309.39 | -1.16 | 1.16 | 58.96 |
| 1×10^{-4} | 4.10 | 3.07 | 0.42 | 280.79 | -1.29 | 1.29 | 67.64 |

Table 2 The kinetic parameters of mercury(II) transport using carrier 1 at different temperatures (Stirring rate is 300 rpm; solvent is CHCl₃)

| Temperature (K) | $k_1 \times 10^3$ (min ⁻¹) | $k_2 \times 10^3$ (min ⁻¹) | R_m^{\max} | t_{\max} (min) | $J_d^{\max} \times 10^3$ (min ⁻¹) | $J_a^{\max} \times 10^3$ (min ⁻¹) | % Transport |
|-----------------|--|--|--------------|------------------|---|---|-------------|
| 293 | 4.03 | 2.02 | 0.50 | 343.45 | -1.26 | 1.26 | 52.80 |
| 298 | 4.10 | 3.07 | 0.42 | 280.79 | -1.29 | 1.29 | 67.64 |
| 303 | 4.18 | 3.20 | 0.41 | 272.88 | -1.34 | 1.34 | 70.16 |
| 308 | 4.25 | 3.47 | 0.40 | 260.01 | -1.41 | 1.41 | 73.52 |

CH₂Cl₂ > CHCl₃ > CCl₄, and the variation of R_a values is illustrated in Figure 5. This shows that the R_a values are strongly affected by the membrane solvent system, and the higher transport efficiency was observed with CH₂Cl₂ solvent. The efficiency of CH₂Cl₂ with respect to the R_a values was higher than of CHCl₃ and CCl₄, because their viscosity values were in the reverse order.

The physicochemical properties of the solvents are given in Table 5. These observations suggest that viscosity is playing a major role in ion transport as well as the polarity. Thus, we have shown that the nature of the membrane solvent is one of the main factors in establishing transport efficiency.

Conclusion

A kinetic study of mercury(II) transport through a membrane assisted by O-(1,3-Bispiperidino-2-propyl)-4-methoxy phenyldithiophosphonate was performed. The kinetic behavior of the transport process as a function of concentration, temperature, stirring rate and solvents was investigated. It can be concluded that dithiophosphorus derivatives can be provided a general and straightforward route to remove toxic metals ions such as mercury(II) ion from water or other solution.

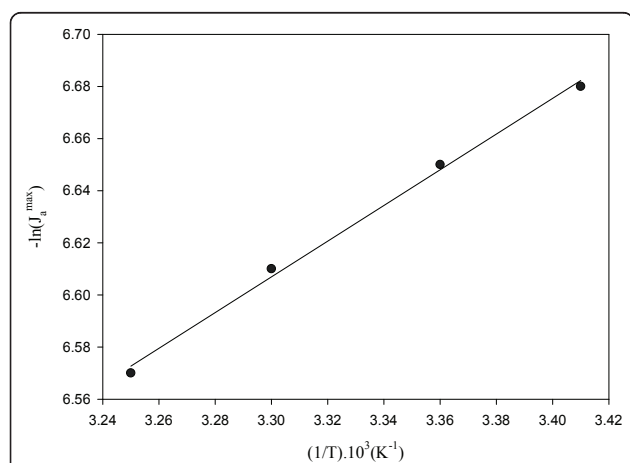


Figure 4 Arrhenius plots for transport of mercury(II) in liquid membrane. Membrane: 1×10^{-4} M of carrier 1 in CHCl₃ at 300 rpm.

Experimental

Solvents were purchased from Merck and distilled before use. 2,4-bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane-2,4-disulfide (Lawesson's reagent) were obtained from Aldrich. 1,3-Bispiperidino-2-propanol was synthesized by the methods given in the literature [26]. ¹H-NMR spectra were obtained in chloroform with a Bruker-DPX 400 FTNMR spectrometer. IR spectra was recorded on a Mattson 1000 FTIR spectrometer using KBr pellets in the range of 4000-400 cm⁻¹. Melting points were determined with a Gallenkamp apparatus without uncorrected.

Liquid Membrane Transport Experiments

The chemical reagents used in these experiments were mercury(II) nitrate, dichloromethane, chloroform, carbon tetrachloride and picric acid obtained from Merck. Mercury(II) picrate solution was prepared by the addition of a 1×10^{-2} M mercury(II) nitrate to a 2.5×10^{-5} M aqueous picric acid solution and shaken at 25°C for 1 hour. The aqueous solutions were prepared using demineralised water.

Kinetic Procedure

Mercury(II) ion transport experiments were conducted using a thermostated (Grand mark, model W14, Grants Instruments, Cambridge, England) apparatus. Transport experiments were carried out in a U-type cell (Figure 6). An organic solution (20 mL) containing the carrier 1 was placed in the bottom of the cell and two portions of aqueous donor and acceptor solutions (10 mL) were carefully added on top of them. Both surface areas were 2.5 cm². The organic phase was stirred at variable speeds magnetically (Chiltern mark, model HS 31). The initial phases consisted of the donor phase, an aqueous mercury(II) picrate (2.5×10^{-5} M) solution, while the membrane phase was made up by dissolving carrier 1 ($C_{\text{carrier}} = 10^{-4}$ M) in the organic phase. The acceptor phase consisted of doubly distilled water. Samples were taken from both water phases (acceptor and donor phases) at various intervals of time and the picrate ion concentration was analyzed by a spectrophotometric method [27]. The spectrophotometric measurements were performed by an UV-Vis Spectrometer Shimadzu

Table 3 The kinetic parameters of mercury(II) transport using carrier 1 at different stirring rates (T = 298 K; solvent is CHCl₃)

| Stirring Rate (rpm) | $k_1 \times 10^3$ (min ⁻¹) | $k_2 \times 10^3$ (min ⁻¹) | R_m^{\max} | t_{ax} (min) | $J_d^{\max} \times 10^3$ (min ⁻¹) | $J_a^{\max} \times 10^3$ (min ⁻¹) | % Transport |
|---------------------|--|--|--------------|----------------|---|---|-------------|
| 200 | 4.03 | 1.08 | 0.62 | 446.37 | -0.67 | 0.67 | 30.12 |
| 300 | 4.10 | 3.07 | 0.42 | 280.79 | -1.29 | 1.29 | 67.64 |
| 400 | 4.33 | 4.91 | 0.35 | 220.98 | -1.69 | 1.69 | 70.72 |

Table 4 The kinetic parameters for mercury(II) transport using carrier 1 when different solvents are used (298 K and 300 rpm)

| Solvent | $k_1 \times 10^3$ (min ⁻¹) | $k_2 \times 10^3$ (min ⁻¹) | R_m^{\max} | t_{\max} (min) | $J_d^{\max} \times 10^3$ (min ⁻¹) | $J_a^{\max} \times 10^3$ (min ⁻¹) | % Transport |
|---------------------------------|--|--|--------------|------------------|---|---|-------------|
| CH ₂ Cl ₂ | 4.51 | 6.12 | 0.31 | 189.61 | -1.92 | 1.92 | 89.72 |
| CHCl ₃ | 4.10 | 3.07 | 0.42 | 280.79 | -1.29 | 1.29 | 67.64 |
| CCl ₄ | 3.49 | 0.41 | 0.75 | 694.86 | -0.31 | 0.31 | 16.64 |

160A. Each experimental result reported is the arithmetic mean of two independent measurements.

O - (1,3-Bispiperidino-2-propyl) - 4-methoxyphenyldithiophosphonate (1)

2,4-Bis (methoxyphenyl-1,3,2,4- dithiadiphosphetane-2,4-disulfide (Lawesson's reagent) (0.89 g, 2.21 mmol) was reacted with 1,3-bispiperidino-2-propanol (1 g, 4.42 mmol) in benzene (5 mL). The mixture was refluxed for 20-30 minutes. The product was obtained as orange solid. The orange solid was filtered, dried in air and recrystallised from CHCl₃. The yield is 1.35 g (72%) and mp:178-180°C. Elemental Analysis Calc. for C₂₀H₃₃N₂O₂PS₂: C, 56.04; H, 7.76; N, 6.53; S, 14.96. Found: C, 55.80; H, 7.52; N, 6.0; S, 14.58. IR (cm⁻¹): 1035 n(P-O-C), 671 n(PS), 548 n(PS). ¹H-NMR (CDCl₃),

d: 8.13-8.08 (dd, 2 H, ³J_{PH} = 14.19 Hz, J_{HH} = 8.81 Hz), 6.90-6.87 (dd, 2 H, ⁴J_{PH} = 2.45 Hz, J_{HH} = 8.81 Hz), 5.36-5.30 (m, 1 H, OCH), 3.81 (s, 3 H, OCH₃), 3.03-2.99 (m, 8 H, orto protons to N on the piperidino ring), 2.91-2.86 (m, 4 H, bridge methylene H), 1.82-1.77 (m, 8 H, meta protons to N on the piperidino ring), 1.56-1.53 (m, 4 H, para protons to N on the piperidino ring). ³¹P-NMR (CDCl₃), d: 112.73 (q, due to the coupling with hydrogen nuclei and the presence of two isomer in solution)(see scheme 1).

Table 5 Physicochemical characteristic of solvents used

| Physicochemical Properties | CH ₂ Cl ₂ | CHCl ₃ | CCl ₄ |
|----------------------------|---------------------------------|-------------------|------------------|
| ϵ_0 | 9.08 | 4.81 | 2.24 |
| η_D | 1.424 | 1.446 | 1.466 |
| μ | 1.959 | 1.354 | 0 |
| η | 0.437 | 0.58 | 0.969 |
| V_m | 64.2 | 96.5 | 96.5 |

ϵ_0 : dielectric constant (20°C); η_D : refractive index (20°C); μ : dipole moment (D); η : viscosity (cP); V_m : molar volume (M⁻¹).

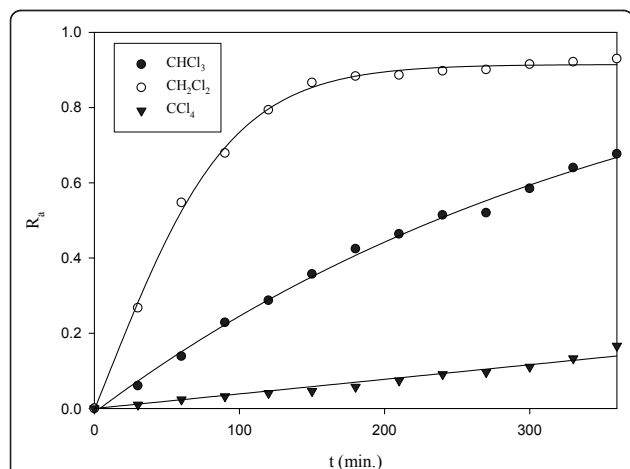


Figure 5 Time variation of reduced concentrations of mercury (II) in the acceptor phase during co-transport through liquid membrane using of carrier 1 in different solvents at a stirring rate of 300 rpm.

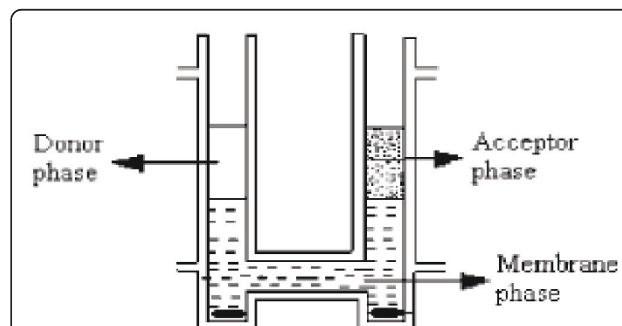


Figure 6 Bulk liquid membrane apparatus for transport of mercury(II) ions; d, donor phase; a, acceptor phase; m, membrane phase.

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Authors' contributions

MK has coordinated the study and characterization of the compound **1**. NA carried out the synthesis of the compound **1**. HKA and AK carried out the kinetic studies and participated in the design of the study. AOG and MA carried out the synthesis of the starting materials. All authors have read and approved the final manuscript.

Competing interests

The authors declare that they have no competing interests.

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