

# Selective $\omega$ -thiocaprolactam-based recovery of Au(III) from chloride media in solvent extraction and polymer inclusion membrane systems

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## Abstract

The selective transport of Au(III) over Cu(II) and Hg(II) using  $\omega$ -thiocaprolactam as extractant and carrier in solvent extraction and polymer inclusion membrane transport experiments is discussed. Solvent extraction experiments were carried out using the extractant dissolved in chloroform to establish the chemical equilibria involved in the Au(III)- $\omega$ -thiocaprolactam separation system. Au(III) is extracted into the organic phase quantitatively ( $E$  (%) > 99%) at low extractant concentrations from aqueous HCl solutions. Cu(II) extraction kinetics is slow compared to Au(III) extraction, allowing for the efficient separation of both metals. Polymer inclusion membranes (PIMs) are designed as alternatives to the use of high amounts of hazardous and expensive reagents. Several stripping phases were tested. Up to 70% of Au(III) can be recovered from 1 M HCl feed aqueous phase solutions. Interference from Cu(II) or Hg(II) is negligible at short times (160 min) in competitive ( $[M(\text{II}, \text{III})]_{0,\text{feed}} = 0.1 \text{ mM}$ ) membrane transport experiments using a cellulose triacetate/NPOE/ $\omega$ -thiocaprolactam PIM and a potassium iodide–hydrochloric acid aqueous stripping solution. Apparent diffusion coefficients in the membrane for Au(III) and Hg(II) in the system were estimated to be  $2.2 \times 10^{-11}$  and  $3.4 \times 10^{-15} \text{ m}^2 \text{ s}^{-1}$ , respectively.

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## 1. Introduction

The processing of electronic wastes containing considerable amounts of valuable components for their recovery or separation is of great interest. Circuit boards, particularly, printed wire boards (PWBs) [1,2], contain a great variety of metals in widely varying concentrations. However, recycling operations of PWBs make most emphasis in the recovery of precious metals since they are by far the most valuable components of these materials. Precious metals in PWBs can be recovered utilizing several conventional and non-conventional techniques [3–5]. A currently used approach for the recovery of precious metals is copper smelting, although the long time and energy consumption it requires may discourage its use. Hydrometallurgy is an interesting alternative since it offers a more direct method of extraction. Methods comprising solvent extraction (SX) of gold

and other precious metal bearing chloride leach solutions have been reported [6–8].

It is well known that SX methods usually consume large amounts of reagents, solvents and extractants, which in many cases are considered as environmental and health hazards. In order to reduce the amounts of reactants and energy needed for separation and to decrease the environmental and economic impact of SX separations, several membrane-based separation techniques have been proposed in the past years. Supported liquid membranes (SLM) have been extensively studied since they offer high transport rates, good selectivities and automatization possibilities, although lack of stability hinders their use in practical applications. As an alternative to SLMs, polymer inclusion membranes (PIMs) have been developed. In these membranes, the carrier is efficiently confined within a polymeric matrix consisting of a support (cellulose triacetate, CTA, or polyvinyl chloride, PVC) and a plasticizer, improving membrane stability.

Alguacil [9], in a recently published work cites several of the most frequently solvent extraction reagents used for Au(III),

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### Nomenclature

$A$	effective membrane area ( $\text{m}^2$ )
$D_{\text{calc}}$	calculated distribution coefficient
$D_{\text{exp}}$	experimental distribution coefficient
$D_0$	apparent diffusion coefficient in the membrane phase ( $\text{m}^2 \text{s}^{-1}$ )
$J$	flux of metal ion ( $\text{mole m}^{-2} \text{s}^{-1}$ )
$K_E$	Cu(II) extraction equilibrium constant
$K_{Ei}$	Au(III) extraction equilibrium constants ( $i = 1, 2$ )
$k_f$	Pseudo-first-order rate constant of extraction at the feed aqueous phase solution–membrane interface ( $\text{min}^{-1}$ )
$l$	membrane thickness (m)
$P$	membrane permeability ( $\text{m s}^{-1}$ )
$t$	time
$t_{\text{lag}}$	lag-time
$V_f$	volume of the feed aqueous phase solution ( $\text{m}^3$ )
$V_0$	volume of the membrane ( $\text{m}^3$ )
$V_s$	volume of the stripping aqueous phase solution ( $\text{m}^3$ )
$x_R$	mole fraction of $\omega$ -thiocaprolactam
$x_f$	gold mole fraction in the feed aqueous phase solution
$x_m$	gold mole fraction in the membrane
<i>Greek letters</i>	
$\beta_4$	overall formation constant for $\text{AuCl}_4^-$
$\beta_{Ei}$	overall extraction constants for Au(III) ( $i = 1, 2$ )
$\theta$	membrane porosity

among which are amines, phosphine oxides, and reagents containing sulfur or oxygen as donor atoms. Also several reports concerning applications of solid-supported liquid membranes are mentioned. Although some PVC-based membranes for Au(III) extraction from chloride media have been reported [10–12], to the best of our knowledge, only one report on Au(III) transport using CTA-based plasticized membranes has been published so far, where the extractants used were oxygen, nitrogen and sulfur donor crown ethers. These extractants showed the following order of selectivity:  $\text{Ag(I)} > \text{Cu(II)} > \text{Au(III)}$  [13].

In the present work, continuing our research on the extractive properties of  $\omega$ -thiocaprolactam [14], which is known to be a soft base that can react with soft acids such as Au(III), solvent extraction experiments were carried out in 1–3 M chloride media in order to establish the chemical equilibria involved in the Au(III)- $\omega$ -thiocaprolactam separation system. Furthermore, with the aim of reducing the organic inventory of the extraction procedure, the performance of  $\omega$ -thiocaprolactam as carrier in a polymer inclusion membrane prepared using cellulose triacetate (CTA) as the support and nitrophenyl octyl ether (NPOE) as the plasticizer is reported. Selectivity over Hg(II) and Cu(II) is studied and discussed as well.

## 2. Experimental

### 2.1. Reagents and instrumentation

$\omega$ -Thiocaprolactam (Aldrich) 98% was recrystallized twice from water. Stock solutions of the reagent were prepared by dissolving the appropriate weighted amounts in a given volume of chloroform previously washed successively with 0.01 M NaOH and deionized water until neutral pH was obtained. For most experiments Au(III) solutions were prepared from a  $1025 \text{ mg L}^{-1}$  in 5% HCl standard solution (Aldrich). All other chemicals were of analytical grade (Aldrich).

### 2.2. Solvent extraction experiments

Equal volumes of 5 mL of organic solutions of  $\omega$ -thiocaprolactam dissolved in chloroform and the aqueous metal solutions (0.1 mM) were shaken in separatory funnels at room temperature ( $20 \pm 2^\circ \text{C}$ ) until equilibrium was attained, using a Burrel-75 mechanical shaker. Ionic strength was fixed using HCl (1–3 M). The pH of the aqueous phases was measured using an Orion Research 701A pH-meter with a combined glass electrode Cole-Palmer 648, or determined by acid–base titration when necessary. In all cases, Au(III) extraction equilibrium is reached in less than 5 min. A Perkin-Elmer 3100 atomic absorption spectrometer was used for the analysis of Au(III) in the aqueous phases. An Agilent Technologies 7500 ICP-MS spectrometer was also used in some experiments. For the determination of Hg(II), the computer-controlled AAS spectrometer was coupled with a Perkin-Elmer FIAS-100 system. Mass balance was checked in some experiments, back extracting the organic phases with 0.1 M thiourea aqueous solutions. At least duplicate experiments were performed. The deviation observed was within  $\pm 5\%$ .

In order to evaluate the Au: $\omega$ -thiocaprolactam stoichiometric ratio, the Job's method of continuous variations was used. The sum of the initial concentration of Au(III) in the aqueous phase and the total concentration of  $\omega$ -thiocaprolactam was maintained constant at 0.1 mM, while the ratio between the total concentrations of Au(III) and the extractant was varied. Experiments were carried out using 1 M HCl as the aqueous phase, and equal volumes of organic and aqueous phases (10 mL) were shaken for 10 min. After phase separation, Au(III) concentration at equilibrium was determined in the aqueous phase by ICP-MS. Au(III) concentrations in the organic phase were determined from the difference between the initial and the final Au(III) concentrations in the aqueous phase.

### 2.3. Membrane preparation and transport experiments

PIMs were prepared according to the phase inversion procedure described by Sugiura [15], i.e., weighted amounts of CTA, plasticizer (NPOE) and carrier were dissolved in 10 mL of  $\text{CH}_2\text{Cl}_2$  (casting solution) and poured into a glass dish (5 cm diameter); the solvent was allowed to evaporate overnight and the membrane formed was peeled off after adding cold water. The exposed membrane area in membrane transport

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