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Sustainable Recovery of Gold (I) From Thiosulfate Solution Using Guanidium Ionic Liquid- Based Aqueous Two-Phase Systems

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Abstract: *Thiosulfate leaching has gained attention as a safer and more environmentally friendly alternative to the conventional cyanidation method for gold extraction. Despite its advantages, the efficient recovery of gold in its monovalent state (Au(I)) from thiosulfate solutions remains a significant challenge. In this study, a novel aqueous two-phase system (ATPS) was developed using eco-friendly guanidinium-based ionic liquids (GILs) combined with inorganic salts to extract gold from mildly alkaline thiosulfate solutions. Key parameters affecting extraction efficiency—such as salt type and concentration, GIL content, extraction time, pH, and initial gold concentration—were systematically investigated. Under optimal conditions, high extraction efficiencies were achieved: 95.8% with [*C_3*-Gun]Cl, 96.5% with [*C_4*-Gun]Cl, and 97.7% with [*C_5*-Gun]Cl. Spectroscopic analysis and density functional theory (DFT) simulations revealed that *$Au(S_2O_3)_2^{3-}$* anions replace *Cl^-* in the GILs and bind to guanidinium cations via electrostatic interactions. Potassium thiocyanate (KSCN) effectively stripped over 99% of the gold from the GIL-rich phase while regenerating the ionic liquid. The system maintained good performance over five extraction-stripping cycles, with only slight efficiency reductions. These results highlight the potential of GIL-based ATPS as a green, efficient, and reusable method for gold recovery from thiosulfate leachates.*

I. INTRODUCTION:

Cyanidation has long been the dominant method for gold extraction due to its simplicity and efficiency; however, the severe environmental risks associated with cyanide use have led to increased interest in alternative leaching agents [1–4]. Thiosulfate leaching has emerged as a promising substitute, offering high selectivity, low toxicity, and good adaptability to refractory ores [5–7]. Despite its advantages, the recovery of Au(I) from thiosulfate leachate remains challenging due to the poor affinity of common adsorbents for the gold-thiosulfate complex [8–10].

Among recovery methods, solvent extraction has shown high selectivity, but conventional extractants often rely on hazardous organic solvents, contradicting sustainability goals [11]. Ionic liquids (ILs), particularly guanidinium-based ILs (GILs), have gained attention for their tunability, low volatility, and biocompatibility, making them greener alternatives to traditional extractants [12]. However, their use in gold extraction from thiosulfate solutions remains largely unexplored.

Aqueous two-phase systems (ATPSs), formed without volatile organic solvents, present a cleaner alternative for liquid–liquid extraction and have shown promise in metal recovery applications [13-15]. This study combines the green potential of GILs with the sustainability of ATPSs to develop an efficient and environmentally friendly method for Au(I) extraction from thiosulfate leaching solutions. The effects of key extraction parameters were systematically investigated, and the extraction mechanism was explored using FT-IR, XPS, and DFT analysis.

Experimental Section (Short Version)

Chemicals: All chemicals used in this study were of analytical grade and used without further purification. The details of the reagents and their suppliers are listed in Table 1.

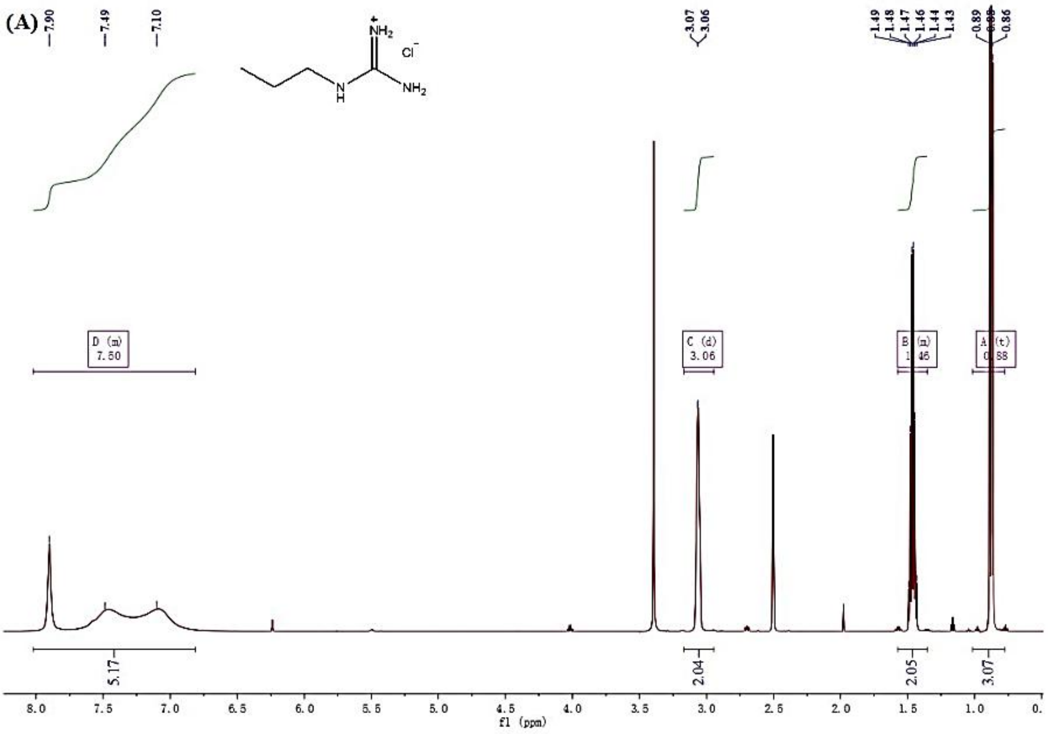
Table 1: Chemicals

Reagent	Purity	Manufacturer
Guanidine Hydrochloride	99.5%	Adamas Chemical Reagent
1-Propanamine	99%	Adamas Chemical Reagent

1-Butylamine	99%	Adamas Chemical Reagent
Amylamine	99%	Adamas Chemical Reagent
Potassium Dihydrogen Phosphate	99%	Adamas Chemical Reagent
Dimethyl Sulfoxide-d_6 (DMSO-d_6)	99.8%	Adamas Chemical Reagent
Gold	99.99%	Sino-Platinum Metals Co., Ltd.

Synthesis of GILs: Guanidinium-based ionic liquids (GILs) were synthesized by reacting guanidine hydrochloride with an excess of alkylamine (molar ratio 1:1.2) in a three-neck flask fitted with a reflux condenser. The mixture was stirred at 80 °C for 12 hours until ammonia evolution ceased. Excess amine was removed under reduced pressure, yielding transparent yellow viscous liquids soluble in water. The resulting monoalkyl GILs—1-propylguanidinium chloride ([C_3-Gun]Cl), 1-butylguanidinium chloride ([C_4-Gun]Cl), and 1-pentylguanidinium chloride ([C_5-Gun]Cl)—were obtained in good yields (e.g., [C_5-Gun]Cl: 83%).

Characterization: The GILs were characterized by ^{1}H NMR spectroscopy using DMSO-d_6 as solvent. The chemical shifts confirmed the successful formation of GILs (see Figure 1 for spectra).



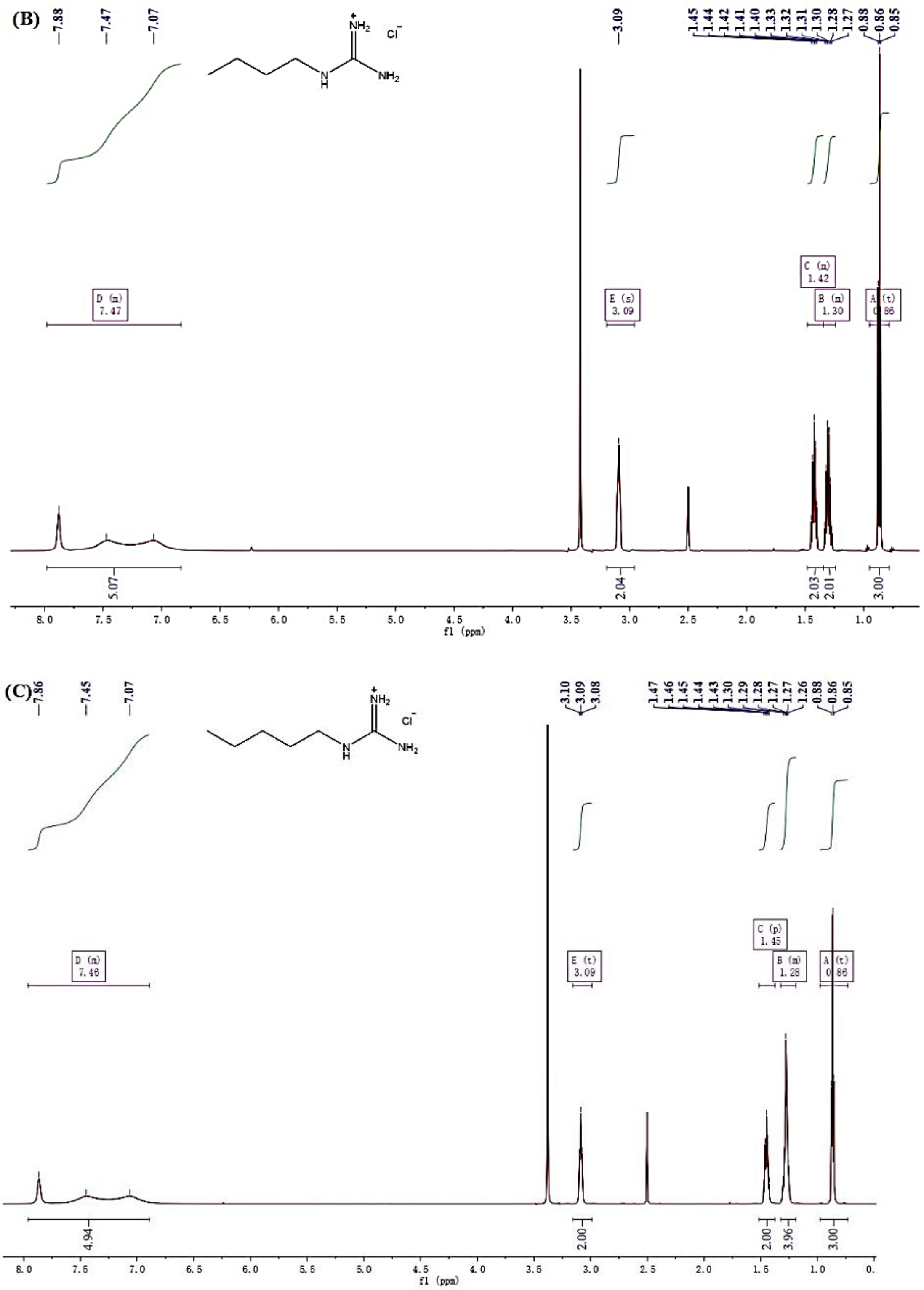


Figure 1:^{1}H NMR spectra of [C_3-Gun]Cl, [C_4-Gun]Cl and [C_5-Gun]Cl

Phase Diagram Determination

The phase behavior of GIL-based aqueous two-phase systems (ATPSs) was determined via cloud point titration at 298.15 K. A prepared solution of K_2HPO_4 was incrementally added to a known concentration of GIL until turbidity appeared, indicating phase separation. The total mass was measured using a Mettler Toledo analytical balance (±0.0001 g). Ultrapure water was then added dropwise until the solution became clear again, and the final mass was recorded. This procedure was repeated to obtain sufficient data to construct the binodal curve of the ATPS.

The experimental data were fitted using the Merchuk equation (Eq. 1) [16]:

$$w_{IL} = a\ exp(bw_K^{0.5} - cw_K^3) \quad (1)$$

Where w_{IL} and $w_K$are the mass fractions of GIL and K_2HPO_4($wt\%$)respectively, and a, b, and c are fitting parameters.

A selected point within the two-phase region was analyzed to determine phase compositions. Phosphate concentrations were measured using a conductivity meter, and GIL content was calculated using the fitted binodal equation. The total length of the tie line (TLL) and its slope (S) were calculated using Eqs. 2 and 3 [17].

$$TLL = [(w_S^T - w_S^B)^2 + (w_G^T - w_G^B)^2]^{0.5} \quad (2)$$

$$S = (w_G^T - w_G^B)/(w_S^T - w_S^B) \quad (3)$$

Effective Excluded Volume (EEV) Analysis

To assess the salt's phase separation capability, EEV theory was applied. According to the statistical geometry model, the binodal data were analyzed using:

- For large differences in molecular weights (neglecting f_{213}) [18]

$$ln\left[V_{213}^* \frac{w_S}{M_S}\right] + V_{213}^* \frac{w_G}{M_G} = 0\,(4)$$

- For similar molecular weights (includingf_{213}):

$$ln\left[V_{213}^* \frac{w_S}{M_S} + f_{213}\right] + V_{213}^* \frac{w_G}{M_G} = 0 \quad (5)$$

Here, w_G and $w_S$are the mass fractions of GIL and salt, M_G and M_S their respective molecular weights, V_{213}^* the effective excluded volume, and f_{213} the volume correction factor accounting for incomplete packing of the salt in the solvent phase.

In this study, due to comparable molecular weights of GIL and salt, Eq. 5 was used for EEV calculations.

Gold(I) Extraction Procedure

The total mass of each aqueous two-phase system (ATPS) was adjusted to 5.0000 g using deionized water. A known quantity of guanidinium-based ionic liquid (GIL), a pre-prepared K_2HPO_4 solution, and 1.0 mL of Au(I) solution (200 mg/L) were accurately added into a separation funnel. The mixture was incubated in a constant-temperature oscillator at 25 °C until equilibrium was reached.

Once phase separation occurred, the top (GIL-rich) and bottom (salt-rich) phases were carefully collected, and their volumes recorded. The pH of the salt-rich phase was measured using a Mettler pH meter. The Au(I) concentration in the salt phase was determined using an atomic absorption spectrophotometer (Z2000, Hitachi, Japan). The Au(I) content in the GIL-rich phase was calculated by mass balance.

Each experiment was performed in triplicate to ensure reproducibility, maintaining a deviation below 2.0%.

The distribution coefficient (D) and extraction efficiency ($E\%$) were calculated using the following equations [19]:

$$D = \frac{C_T}{C_B} \quad (6)$$

$$E\% = \frac{C_T V_T}{C_T V_T + C_B V_B} \times 100\% \quad (7)$$

Where$C_T$and C_B are the Au(I) concentrations (mg/L) in the GIL and salt phases, respectively, and $V_T$and V_B are the corresponding volumes (mL) of the two phases.

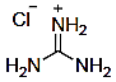
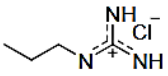
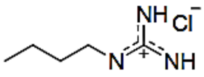
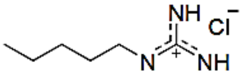
Recycling Experiments

To assess the reusability of the GIL-based ATPS, five extraction/stripping cycles were conducted. After each gold(I) extraction, the salt-rich phase was reserved for reuse. The gold-loaded GIL-rich phase was treated with 6.0 mol/L potassium thiocyanate (KSCN) for 10 minutes to strip the gold, then washed with saturated KCl solution. The regenerated GIL and salt phases were recombined to form a new ATPS. A small amount of K_2HPO_4 was added in each cycle to compensate for salt loss due to the gold solution addition.

Characterization

GILs were characterized using ^{1}H NMR. FT-IR and XPS analyses were performed on the GIL phase before and after gold loading in Table 2.

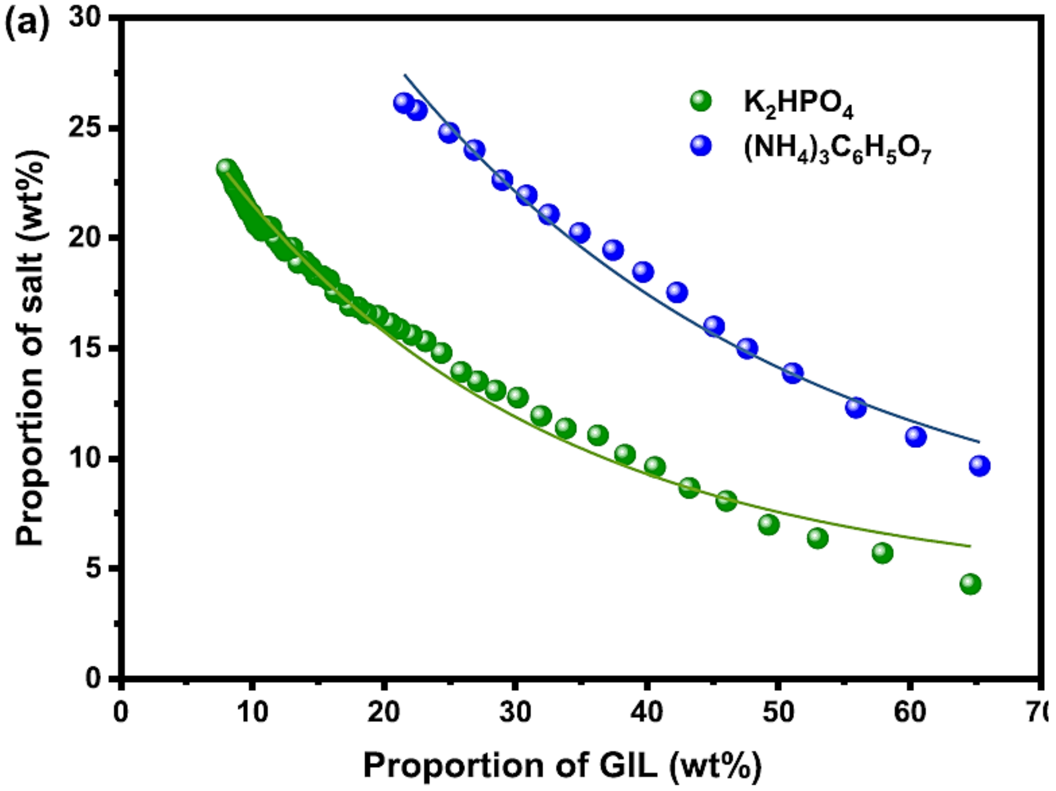
Table 2: Preparation of GILs

Guanidine hydrochloride	Alkylamine	GILs	Acronyms
			[C$_3$-Gun]Cl
			[C$_4$-Gun]Cl
			[C$_5$-Gun]Cl

II. Results and Discussion

Screening of GILs: Monoalkyl guanidinium-based ionic liquids (GILs) with short alkyl chains were synthesized using primary amines of different chain lengths. Among them, [C$_3$-Gun]Cl, [C$_4$-Gun]Cl, and [C$_5$-Gun]Cl successfully formed stable aqueous two-phase systems (ATPSs) with K_2HPO_4 at 298.15 K. GILs with longer chains (C6–C8) resulted in precipitation and failed to produce clear phase separation. Thus, only the short-chain GILs were selected for further gold(I) extraction studies.

Salt Type and EEV Analysis: Salt type significantly affects ATPS formation. Compared to $(NH_4)_3C_6H_5O_7$, K_2HPO_4 showed a higher salting-out capacity and a larger effective excluded volume (EEV), confirming its stronger ability to induce phase separation with GILs shown in figure 2 (a). Hence, K_2HPO_4 was chosen as the phase-forming salt in all subsequent experiments [20].



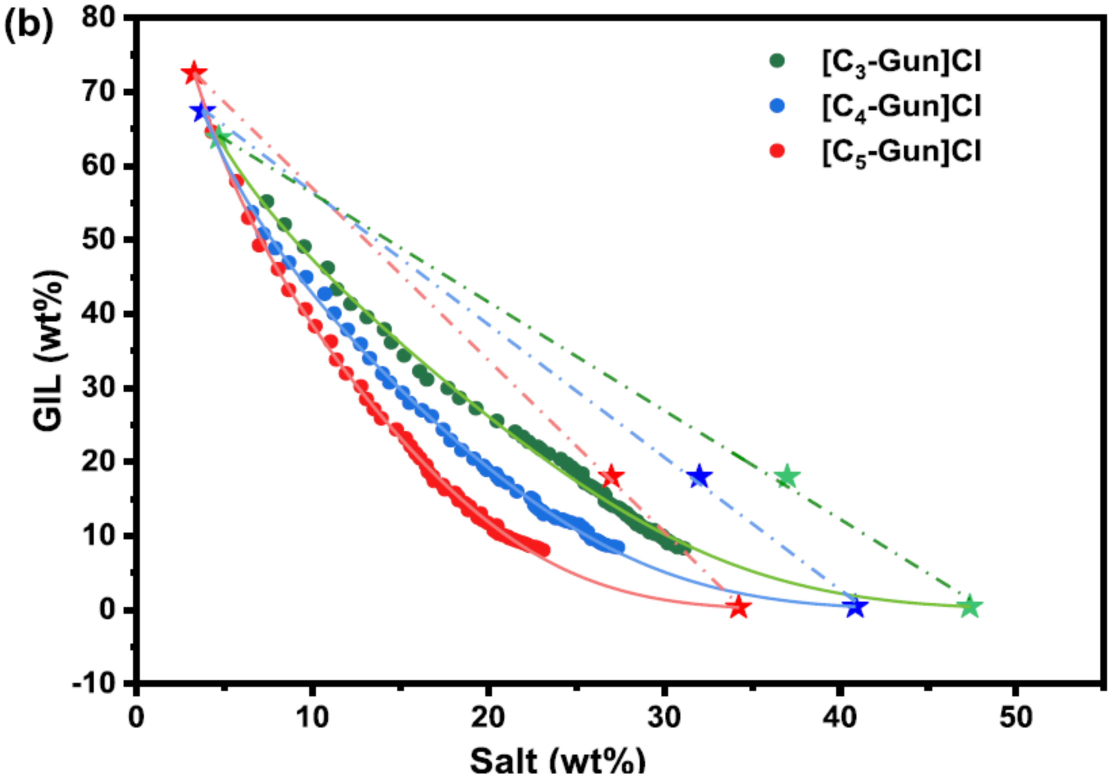
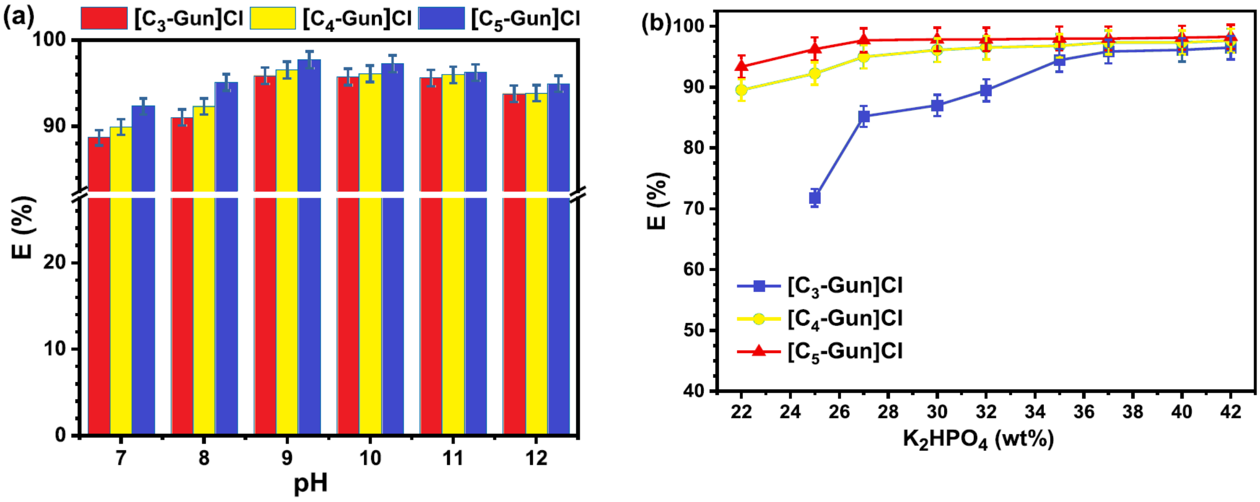


Figure 2: Phase Diagram under different salts (a) and ILs (b), T=298.15

Phase Diagram and Tie Line Data: Phase diagrams for [C$_3$-Gun]Cl, [C$_4$-Gun]Cl, and [C$_5$-Gun]Cl with K$_2$HPO$_4$ were constructed and fitted using the Merchuk equation ($R^2 > 0.99$). The ability to form ATPS increased with longer alkyl chains: [C$_5$-Gun]Cl> [C$_4$-Gun]Cl> [C$_3$-Gun]Cl. This is attributed to increased hydrophobicity, which reduces water affinity and enhances phase separation [21]. Based on tie-line length (TLL) analysis, optimal salt contents were determined as 27.0 wt% for [C$_5$-Gun]Cl, 32.0 wt% for [C$_4$-Gun]Cl, and 37.0 wt% for [C$_3$-Gun]Cl shown in Figure 2(b).

Effect of pH: Gold(I) thiosulfate exists primarily as Au(S$_2$O$_3$)$_2^{3-}$ in the pH range 7–12. Extraction efficiency increased with pH, peaking at pH 9.00: 95.8% for [C3-Gun]Cl, 96.5% for [C4-Gun]Cl, and 97.7% for [C5-Gun]Cl. Beyond pH 9.00, efficiency declined slightly shown in Figure 3(a). The enhanced extraction near pH 9.00 correlates with the dominance of HPO$_4^{2-}$ (stronger salting agent) over H$_2$PO$_4^-$. Therefore, pH 9.00 was selected as optimal for further tests[22].



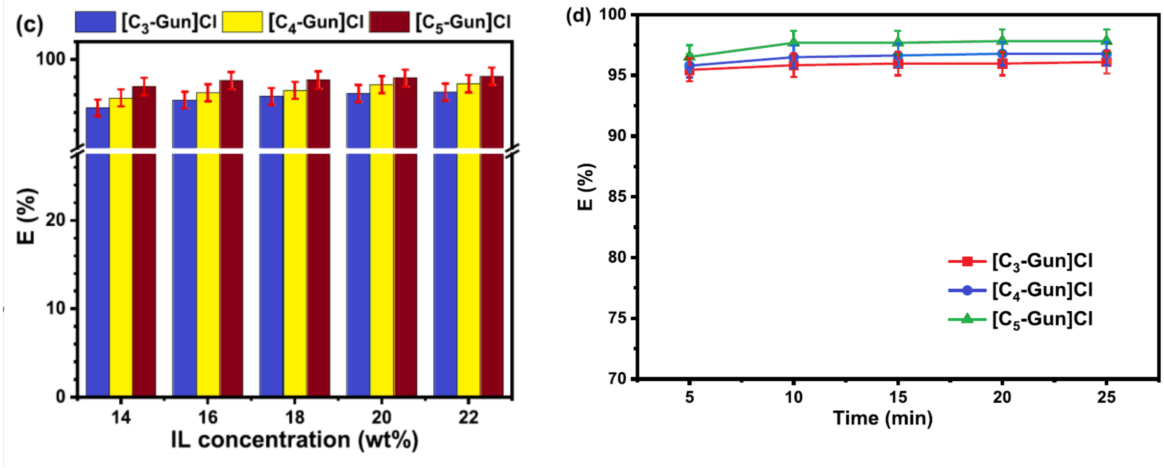


Figure 3: (a) Influence of equilibrium pH, (b) Inorganic salt Concentration, (c) GIL concentration, and (D) time on Au(I) extraction.

Effect of Salt Content: Raising K_2HPO_4 concentration from 22.0 to 42.0 wt% improved extraction efficiencies due to stronger phase separation driven by the kosmotropic effect of salts. Maximum extraction reached 96.5% for [C_3-Gun]Cl, 97.6% for [C_4-Gun]Cl, and 98.3% for [C_5-Gun]Cl. Optimal salt contents for practical application were maintained as previously determined in figure 3(b) [23].

Effect of GIL Concentration: Increasing GIL concentration enhanced extraction efficiency due to stronger electrostatic interactions with $Au(S_2O_3)_2^{3-}$. When GIL content increased from 14.0 to 22.0 wt%, gold extraction efficiency rose to 96.3% ([C_3-Gun]Cl), 97.3% ([C_4-Gun]Cl), and 98.1% ([C_5-Gun]Cl) shown in figure 3 (c). Considering cost and efficiency, 18.0 wt% was chosen as the standard concentration, achieving 95.8–97.7% extraction across the GILs.

Extraction Mechanism and Performance Summary

Effect of Extraction Time: Gold(I) extraction reached equilibrium within 10 minutes for all GIL systems, demonstrating fast kinetics and high efficiencies: 95.8% ([C_3-Gun]Cl), 96.5% ([C_4-Gun]Cl), and 97.7% ([C_5-Gun]Cl) shown in Figure 3 (d). Hence, 10 minutes was chosen for further experiments.

Effect of Initial Gold Concentration: At low initial concentrations (≤50 mg/L), gold(I) was extracted with 100% efficiency. Even at high concentrations (up to 1000 mg/L), extraction remained excellent—above 93% in all systems—proving strong gold-binding capacity of GIL-ATPSs. Shown in figure 4(a). A standard 200 mg/L was selected for further studies.

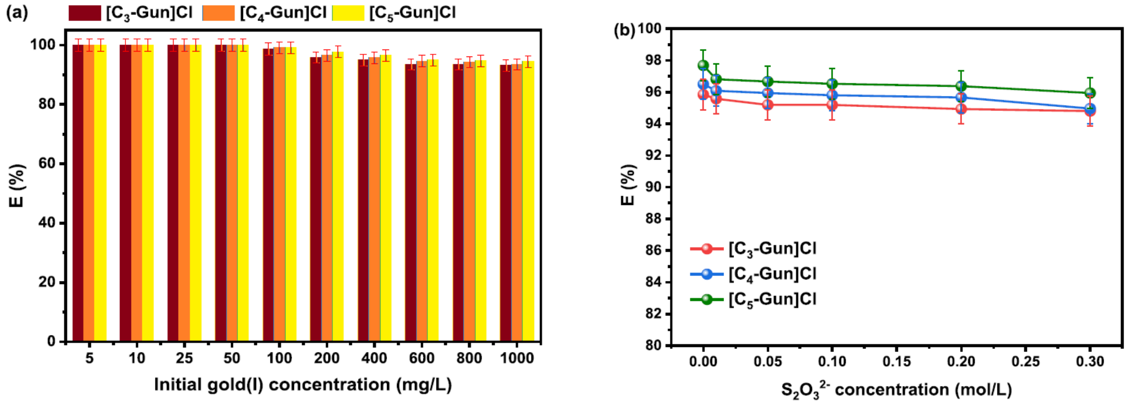


Figure 4. (a) Effect of initial gold (I) concentration and (b) $S_2O_3^{2-+}$ content on extraction of Au (I).

Effect of Thiosulfate ($S_2O_3^{2-}$) Concentration: Although higher thiosulfate levels slightly reduced extraction efficiency due to competition with $Au(S_2O_3)_2^{3-}$ for GIL binding, all systems maintained over 94% extraction even at 0.30 mol/L $S_2O_3^{2-}$, which aligns with practical leaching conditions shown in figure 4(b).

Selectivity for Gold(I): The GIL-ATPS systems showed excellent selectivity for Au(I) over Cu(II), Co(III), and Ni(II). Gold extraction remained high (93–97%), while co-extraction of other metals stayed below 13%. High separation coefficients confirmed the system's strong selectivity for gold.

Application in Real Leachate: Using oxidized ore leachate from Xinjiang, the [C5-Gun]Cl ATPS achieved nearly 100% Au(I) extraction, with minimal interference from other metals. Separation factors were exceptionally high—33,146 (Cu), 6,835 (Co), and 9,131 (Ni)—demonstrating excellent real-world applicability.

FT-IR Analysis:FT-IR spectra confirmed $Au(S_2O_3)_2^{3-}$ was successfully transferred to the GIL-rich phase. New S–O bond vibrations and shifts in C=N and C–N peaks upon gold loading suggest strong electrostatic interactions and anion exchange with guanidinium cations.

XPS Analysis:XPS data further confirmed the presence of Au(I) in the GIL phase as $Au(S_2O_3)_2^{3-}$. Shifts in binding energies of Au 4f, S 2p, and C 1s after extraction validated the occurrence of anion exchange between Cl^- in GIL and $Au(S_2O_3)_2^{3-}$. Higher Cl^- concentrations in solution reduced gold extraction, confirming the anion-exchange mechanism.

Extraction Equilibrium Summary

The extraction mechanism involves an anion exchange where the $Au(S_2O_3)_2^{3-}$ complex in the salt-rich phase binds electrostatically with guanidinium cations (Gun^+) in the GIL-rich phase, displacing Cl^- into the salt phase. This interaction is expressed by the equilibrium reaction:Equation (10),To quantify this, the equilibrium constant (K_eq) and distribution coefficient (D) are defined as:

$$n[C_5 - Gun^+.Cl^-]_{top} + [Au(S_2O_3)_2^{3-}]_{bottom} = [nC_5 - Gun^+.Au(S_2O_3)_2^{3-}]_{top} + [Cl^-]_{bottom} \tag{10}$$

$$K_{eq} = \frac{[nC_5 - Gun^+.Au(S_2O_3)_2^{3-}]_{top} \times [Cl^-]_{bottom}^n}{[C_5 - Gun^+.Cl^-]_{top}^n \times [Au(S_2O_3)_2^{3-}]_{bottom}} \tag{11}$$

$$D = K_{eq}[C_5 - Gun^+.Cl^-]_{top}^n / [Cl^-]_{bottom}^n \tag{12}$$

$$logD = logK_{eq} + nlog[C_5 - Gun^+.Cl^-]_{top} - nlog[Cl^-]_{bottom} \tag{13}$$

Since [C_5-Gun]Cl is in large excess, Cl^- concentration is negligible, and initial GIL concentration approximates the equilibrium concentration. By plotting log D vs. log [C_5-Gun]Cl, a slope of approximately 3.0 is observed, confirming a 1:3 stoichiometric ratio between $Au(S_2O_3)_2^{3-}$ and [C_5-Gun]Cl.

Thus, the final extraction reaction is:

$$3[C_5 - Gun^+.Cl^-]_{top} + [Au(S_2O_3)_2^{3-}]_{bottom} = [3C_5 - Gun^+.Au(S_2O_3)_2^{3-}]_{top} + 3[Cl^-]_{bottom} \tag{14}$$

This confirms that three GIL molecules participate in extracting one gold complex ion via an anion-exchange mechanism.

DFT Calculaions:

To validate the gold(I) extraction mechanism, Density Functional Theory (DFT) calculations were conducted using the Gaussian 09 software. The electrostatic potential (ESP) maps generated through Multiwfn and visualized using VMD revealed a strong electrostatic attraction between the guanidinium cation $C_5 - Gun^+$and the gold thiosulfate anion $Au(S_2O_3)_2^{3-}$. The high ESP of the guanidine group and the low ESP of the gold complex facilitated this interaction [24-26]. The reaction was thermodynamically favorable, with a free energy decrease of 1.33 eV upon complex formation, indicating a spontaneous and stable binding. Furthermore, HOMO-LUMO orbital analysis showed that the front orbitals in the complex were primarily determined by the gold thiosulfate ion, and no significant charge transfer occurred—further supporting the conclusion that the interaction was dominated by electrostatic forces rather than electron sharing.

The recovery of gold from the loaded GIL-rich phase and the recycling ability of the GIL system were also evaluated. Back-extraction was achieved using potassium thiocyanate (KSCN), with optimal stripping efficiency (up to 99.7%) observed at a concentration of 6.0 mol/L [27]. The high efficiency is attributed to the small charge density of SCN^- ions, which allows them to replace gold thiosulfate anions through competitive binding with guanidinium cations. Subsequently, a series of five extraction-stripping cycles were performed to test the system's recyclability [28-30]. The extraction and stripping efficiencies in the first cycle were 98.0% and 99.7%, respectively. After five cycles, only a slight decline was observed, with efficiencies still reaching 90.4% for extraction and 92.3% for stripping. These results demonstrate the excellent reusability and stability of the GIL-based ATPS, confirming its strong potential for industrial applications in gold(I) recovery from thiosulfate media.

III. Conclusions

In this study, three environmentally benign guanidinium-based ionic liquids (GILs) were successfully synthesized and employed to form green aqueous two-phase systems (ATPSs) with inorganic salts for the efficient extraction of Au(I) from thiosulfate solutions. The phase behavior of the GIL + K_2HPO_4 + H_2O systems was mapped using the cloud point titration method. Key parameters influencing extraction performance—such as the type and concentration of GIL and salt, extraction time, solution pH, and initial gold concentration—were systematically optimized. Among the tested GILs, [C_5-Gun]Cl exhibited the highest extraction efficiency, with performance improving alongside increasing alkyl chain length due to enhanced hydrophobicity and phase separation. Mechanistic insights obtained from FT-IR, XPS, and DFT analyses confirmed that the extraction of Au(I) involves an anion-exchange mechanism, where $Au(S_2O_3)_2^{3-}$ displaces Cl^- and forms a stable ion pair with guanidinium cations via electrostatic interaction. Moreover, the loaded GIL phase could be effectively regenerated using 6.0 mol/L KSCN, achieving nearly complete gold stripping. After five reuse cycles, extraction and stripping efficiencies remained above 90%, highlighting the robustness and recyclability of the system. The GIL-ATPS also demonstrated excellent selectivity for Au(I) in complex leachate containing Cu^{2+}, Co^{3+}, and Ni^{2+}. Overall, this work underscores the potential of green GIL-based ATPSs as a sustainable and efficient approach for gold recovery from thiosulfate leaching systems

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