



## Solvent extraction of rubidium and cesium from salt lake brine with t-BAMBP–kerosene solution

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**Abstract:** The residues of salt lake brine from which potassium had been removed were used to extract  $\text{Rb}^+$  and  $\text{Cs}^+$  together with a sulphonated kerosene (SK) solution of 1.0 mol/L 4-tert-butyl-2-( $\alpha$ -methylbenzyl) phenol (t-BAMBP).  $\text{Rb}^+$  and  $\text{Cs}^+$  were enriched and separated effectively by precipitating  $\text{Mg}^{2+}$  before extraction and by scrubbing out  $\text{K}^+$  and  $\text{Na}^+$  repeatedly before stripping. The effects of the volume ratio of organic phase to aqueous extraction phase (O/A), alkalinity of aqueous phase ( $c(\text{OH}^-)$ ), interference from  $\text{K}^+$  and  $\text{Mg}^{2+}$ , and ratio the volume of organic phase to aqueous scrubbing phase (O/A') were investigated. The experimental brine was extracted optimally by 5-stage extraction with 1.0 mol/L t-BAMBP in SK,  $c(\text{OH}^-)=1$  mol/L, and O/A=1:1. The scrubbing yield of rubidium was only about 10.5% when the extraction solvent was washed 3 times with  $1 \times 10^{-4}$  mol/L NaOH at O/A'=1:0.5. After 5-stage countercurrent extraction, the final extraction yields of  $\text{Rb}^+$  and  $\text{Cs}^+$  reached 95.04% and 99.80%, respectively.

**Key words:** salt lake brine; t-BAMBP; extraction; rubidium ion; cesium ion

### 1 Introduction

Rare metals rubidium and cesium are playing increasingly important roles in the fields of new energy, new materials, night-vision equipments, fiber optic telecommunication systems and metal-ion catalysts [1–3]. Due to the highly similar properties, rubidium and cesium usually coexist in minerals. There are many large hinterland salt lakes worldwide containing abundant rubidium and cesium but often at trace concentrations (about 0–20 mg/L) [4].  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Li}^+$  and other ions also coexist in salt lake brine, the concentrations of which are thousands or even hundreds of thousands times that of  $\text{Rb}^+$  and  $\text{Cs}^+$ . Moreover, these ions have very similar physical and chemical properties to those of  $\text{Rb}^+$  and  $\text{Cs}^+$  [5,6], so it remains hard to separate trace  $\text{Rb}^+$  and  $\text{Cs}^+$  economically from salt lake brine and to purify them.

Two methods, i.e., adsorption [7,8] and solvent extraction [9,10], have been widely used to enrich and separate low-concentration  $\text{Rb}^+$  and  $\text{Cs}^+$  hitherto. For the solvent extraction method, substituted phenols, such as 4-sec-butyl-2( $\alpha$ -methylbenzyl) phenol (BAMBP) and

4-tert-butyl-2 ( $\alpha$ -methylbenzyl) phenol (t-BAMBP), are highly practically valuable due to high selectivities toward  $\text{Rb}^+$  and  $\text{Cs}^+$  and easy stripping [11–13]. But up to now, phenolic extraction solvents have seldom been used to extract  $\text{Rb}^+$  and  $\text{Cs}^+$  from salt lake brine which contains high concentrations of other ions.

The samples of salt lake brine from Qinghai Province (China) and the intermediate liquids in potassium fertilizer or in lithium carbonate manufacturing process using salt lake brine as raw material were analyzed. The residual brine from potassium extracting system had  $\text{Rb}^+$  and  $\text{Cs}^+$  at the concentrations 8–10 times those in the original brine. Especially, the contents in the brine samples from East or West Taijnar Lake (Qinghai Citic Guoan Technology Development Co., Ltd.) were the highest. The concentrations of  $\text{Rb}^+$  and  $\text{Cs}^+$  rose to 20.0 mg/L and 2.0 mg/L, respectively, whereas they were only 2.3 mg/L and 0.35 mg/L respectively in the original brine. A new process was proposed in this work to extract  $\text{Rb}^+$  and  $\text{Cs}^+$  from salt lake brine with t-BAMBP. Magnesium was first precipitated before extraction. Meanwhile, potassium and sodium were washed before stripping. Finally,  $\text{Rb}^+$  and  $\text{Cs}^+$  were enriched, and they were then

separated easily by further extraction to give pure compounds.

## 2 Experimental

### 2.1 Reagents

t-BAMBP (98% GC purity) was obtained from Beijing Realkan Septrtech Co., Ltd. Sulphonated kerosene (SK) was prepared with concentrated sulfuric acid. All other reagents were analytical reagents and used as received.

The residual liquid with potassium products eliminated was obtained from East Taijnar Plant (Citic Guoan Technology Development Co., Ltd., China) and used as the study object. The concentrations of cations are:  $\rho(\text{Rb}^+)=20.0$  mg/L,  $\rho(\text{Cs}^+)=2.0$  mg/L,  $\rho(\text{Na}^+)=19.39$  g/L,  $\rho(\text{Mg}^{2+})=28.38$  g/L,  $\rho(\text{K}^+)=2.772$  g/L and  $\rho(\text{Li}^+)=0.685$  g/L; pH: 6.

### 2.2 Experimental design

The structure of extractant (t-BAMBP) is shown in Fig. 1.

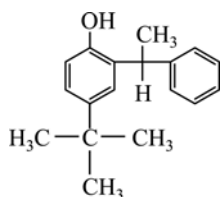
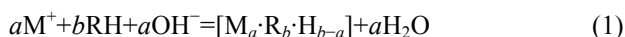


Fig. 1 Structure of t-BAMBP

In alkaline solution, phenols extract alkali metal ions probably by cation exchange through the acidic hydrogen ions. Then the resulting phenolic salt with strong hydrophobicity (dissoluble in organic phase and insoluble in water phase) can be easily stripped by inorganic acid. The extraction reaction can be described as follows (where RH refers to phenol and  $\text{M}^+$  refers to  $\text{Rb}^+$  or  $\text{Cs}^+$ ) [14,15]:



Phenols are excellent extractants for alkali metals and have high selectivities for  $\text{Rb}^+$  and  $\text{Cs}^+$  over other elements in the same group. The extractabilities of alkali metals follow the descending order of  $\text{Cs}^+ > \text{Rb}^+ > \text{K}^+ > \text{Na}^+ > \text{Li}^+$ .  $\text{Rb}^+$  and  $\text{Cs}^+$  extracted into the organic phase can be easily stripped by inorganic acid following the reaction below. At the same time, the extractant is recovered.



### 2.3 Analytical method

$\text{Rb}^+$  and  $\text{Cs}^+$  were examined by flame atomic absorption spectrometry with PerkinElmer 700/800

atomic absorption spectrometer.

### 2.4 Experimental process

$\text{Mg}^{2+}$  in the experimental brine was firstly removed with sodium hydroxide to eliminate its influence on extraction, and magnesium hydroxide precipitate was then separated by centrifugation as fire retardant or rubber stuffing. Then the alkalinity was adjusted, and the brine was extracted with t-BAMBP solution. Most of  $\text{Rb}^+$  and  $\text{Cs}^+$ , a small amount of  $\text{K}^+$  and trace  $\text{Na}^+$  in the brine were extracted into the organic phase.  $\text{RbCl}$  and  $\text{CsCl}$  were readily prepared by washing the organic phase with  $1.0 \times 10^{-4}$  mol/L NaOH and by stripping with 2.0 mol/L HCl, and the stripping liquid was evaporated for crystallization. The technological process is described in Fig. 2.

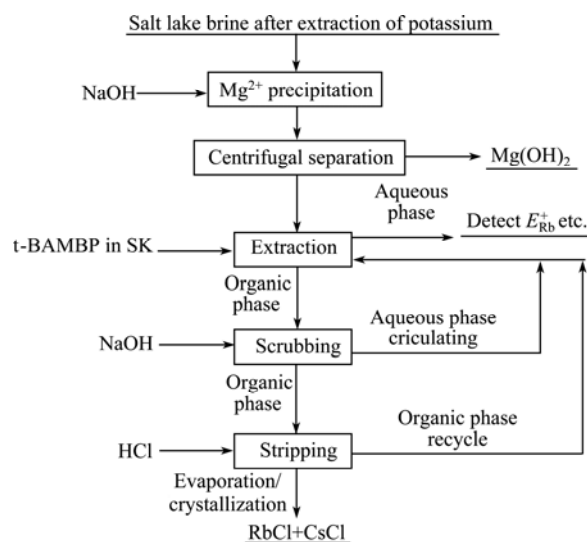


Fig. 2 Technological process of extraction

## 3 Results and discussion

### 3.1 Extraction ability of t-BAMBP and effect of phase ratio

The extraction ability of t-BAMBP towards the simulated brine which only contained  $\text{Rb}^+$  and  $\text{Cs}^+$  ( $\rho(\text{Rb}^+)=20.0$  mg/L,  $\rho(\text{Cs}^+)=2.0$  mg/L) and the effect of phase ratio on extraction were investigated firstly (Fig. 3). The extraction efficiency ( $E$ ) can be calculated from the following formula:

$$E = [M_1 / (M_1 + M_2)] \times 100\% \quad (3)$$

where  $M_1$  is the amount of extracted ion in organic phase;  $M_2$  is the amount of the residue ion in aqueous phase.

As shown in Fig. 3, when SK was used as extractant alone (without t-BAMBP in it), no  $\text{Rb}^+$  or  $\text{Cs}^+$  was extracted. So SK was only a diluent. t-BAMBP showed high extraction capacity towards the simulated brine which only contained  $\text{Rb}^+$  and  $\text{Cs}^+$ . The extraction yield of  $\text{Rb}^+$  was elevated with increasing the volume ratio of

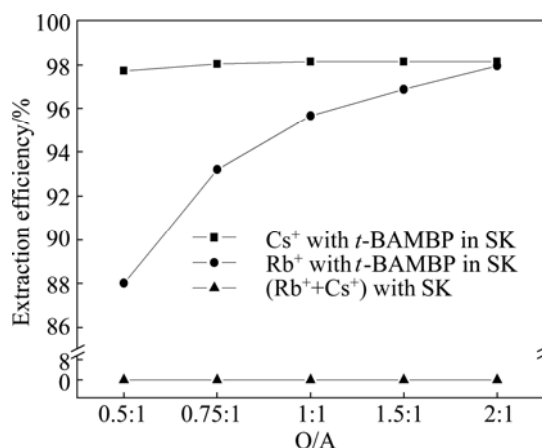


Fig. 3 Effect of phase ratio (O/A) on extraction efficiency

organic phase to aqueous phase (O/A), and that of Cs<sup>+</sup> changed slightly because it had almost been completely extracted at low phase ratio. When O/A=2:1, the extraction efficiencies of Rb<sup>+</sup> and Cs<sup>+</sup> were 97.95% and nearly 100%, respectively. The original concentration of Rb<sup>+</sup> was 10 times that of Cs<sup>+</sup>, so the extraction of Rb<sup>+</sup> was crucial because Cs<sup>+</sup> should be extracted first. Taking the cost of extraction into consideration simultaneously, O/A=1:1 was selected as the optimum phase ratio. The raw material of salt lake brine which contained other ions (e.g., alkali ions) at high concentrations may substantially reduce the extraction selectivities for Rb<sup>+</sup> and Cs<sup>+</sup>.

### 3.2 Effect of extractant concentration

The effect of t-BAMBP on extraction was investigated by changing its concentration (0.5 mol/L, 1 mol/L) and the extraction phase ratio (O/A=0.25:1, 0.5:1 and 1:1). The results are given in Table 1.

Table 1 Effects of t-BAMBP concentration and phase ratio on extraction efficiency

Concentration of t-BAMBP/ (mol·L <sup>-1</sup> )	Phase ratio (O/A)	Extraction efficiency/%				
		Li <sup>+</sup>	Na <sup>+</sup>	K <sup>+</sup>	Mg <sup>2+</sup>	Rb <sup>+</sup>
1.0	0.25:1	5.10	16.00	21.70	13.60	20.30
1.0	0.5:1	8.54	6.85	49.70	18.70	54.40
1.0	1:1	6.88	3.60	8.40	10.80	83.60
0.5	1:1	7.80	3.00	12.40	17.60	12.00

1) Oscillation at 20°C for 5 min; 2) The content of Cs<sup>+</sup> in aqueous phase after extraction was too low to be tested and can thus be considered to be entirely extracted.

As given in Table 1, K<sup>+</sup> and Mg<sup>2+</sup> interfered with the extraction of Rb<sup>+</sup> and Cs<sup>+</sup> significantly, with high extraction yields and maximum extractants. Hence, it is vital to evaluate the effects of the concentrations of K<sup>+</sup> and Mg<sup>2+</sup> on extraction. With rising phase ratio (O/A), the extraction yield of Rb<sup>+</sup> increased, and the extraction

rates of other ions reduced. With increasing the concentration of t-BAMBP, the extraction yield of Rb<sup>+</sup> rose obviously, whereas the viscosity of organic phase and the cost of extraction were augmented simultaneously. Accordingly, 1.0 mol/L phenol and O/A=1:1 were optimum for the process in most cases.

### 3.3 Effect of Mg<sup>2+</sup> and K<sup>+</sup>

The extraction efficiencies of Li<sup>+</sup> and Na<sup>+</sup> are much lower than those for Rb<sup>+</sup> and Cs<sup>+</sup> because their ionic radii are much smaller. However, K<sup>+</sup> and Mg<sup>2+</sup>, which have similar ionic radii to those of Rb<sup>+</sup> and Cs<sup>+</sup>, were bound to affect the extraction most significantly. After Mg<sup>2+</sup> was precipitated with NaOH or K<sup>+</sup> was removed with tartaric acid, the experimental brine was extracted with 1.0 mol/L t-BAMBP in SK when O/A=1:1.

A very small portion of Rb<sup>+</sup> (3.6%) was precipitated together with Mg<sup>2+</sup>, after which the single-stage extraction yield of Rb<sup>+</sup> reached 63.8%. On the contrary, a large amount of Rb<sup>+</sup> (65.7%) was precipitated with potassium hydrogen tartrate during K<sup>+</sup> elimination (without Mg<sup>2+</sup> precipitation). Probably, Rb<sup>+</sup> was impacted into the crystal lattice of K<sup>+</sup> precipitate because of their similar physicochemical properties. Furthermore, the extraction yield of remaining Rb<sup>+</sup> was very low after K<sup>+</sup> precipitation, revealing that Mg<sup>2+</sup> actually reduced the Rb<sup>+</sup> extraction yield. Therefore, it is necessary to remove Mg<sup>2+</sup> and to raise pH value of the brine before Rb<sup>+</sup>/Cs<sup>+</sup> extraction. The by-product Mg(OH)<sub>2</sub> can be used as fire resistant or rubber filler to reduce the costs remarkably.

### 3.4 Effect of c(OH<sup>-</sup>)

Figure 4 shows the extraction efficiencies of various alkali metals in the experimental brine with 1.0 mol/L t-BAMBP by adjusting c(OH<sup>-</sup>) in the aqueous phase from 0 to 1.0 mol/L. The extraction efficiencies of Rb<sup>+</sup> and Cs<sup>+</sup> increased with rising c(OH<sup>-</sup>), and the extraction yield of Rb<sup>+</sup> increased from 40.9% to 90.8% when c(OH<sup>-</sup>) rose to 1.0 mol/L.

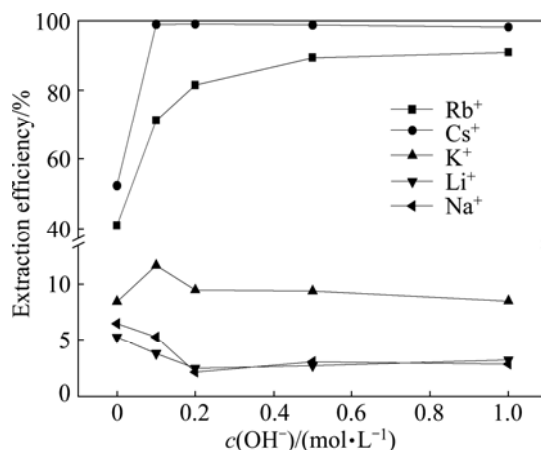


Fig. 4 Effect of c(OH<sup>-</sup>) on extraction efficiency

The extraction selectivity of t-BAMBP solution (a weak acidic solvent) in alkaline condition is much higher than that in neutral or acidic condition, so alkalinity is critical for the extraction system. Extra  $\text{Na}^+$  or  $\text{K}^+$  may be introduced into the experimental brine because  $c(\text{OH}^-)$  is adjusted by sodium hydroxide and potassium hydroxide. Comparably, high concentration of  $\text{Na}^+$  is tolerable because it exerts less effect on  $\text{Rb}^+/\text{Cs}^+$  extraction. In this case, NaOH was chosen to precipitate  $\text{Mg}^{2+}$  and to adjust  $c(\text{OH}^-)$  of the aqueous phase. According to the curves in Fig. 4,  $c(\text{OH}^-)=0.5$  mol/L was the most suitable.

### 3.5 Effect of scrubbing phase

As a weak acid, t-BAMBP can only extract  $\text{Rb}^+$  and  $\text{Cs}^+$  efficiently at high pH values, and it can be easily stripped with diluted acid [16]. To enrich  $\text{Rb}^+$  and  $\text{Cs}^+$  in the extractant and to scrub out  $\text{Na}^+$ ,  $\text{K}^+$  and other impurities as much as possible before stripping, the scrubbed extractant was stripped with HCl. Therefore, the effects of scrubbing phase and the volume ratio of organic phase to aqueous scrubbing phase ( $O/A'$ ) were investigated. The results are given in Table 2.

**Table 2** Effects of scrubbing phase and  $O/A'$  ratio on  $\text{Rb}^+$  scrubbing yield

Scrubbing phase	Phase ratio ( $O/A'$ )	Scrubbing yield, $E'/\%$		
		$\text{Rb}^+$	$\text{K}^+$	$\text{Na}^+$
$\text{H}_2\text{O}$	1:1	33.0	63.0	64.0
$1.0 \times 10^{-4}$ mol/L NaOH	1:2	29.3	40.5	77.1
$1.0 \times 10^{-4}$ mol/L NaOH	1:1	16.4	43.6	76.9
$1.0 \times 10^{-4}$ mol/L NaOH	1:0.5	10.5	57.7	69.4

Oscillation at 20 °C for 5 min.

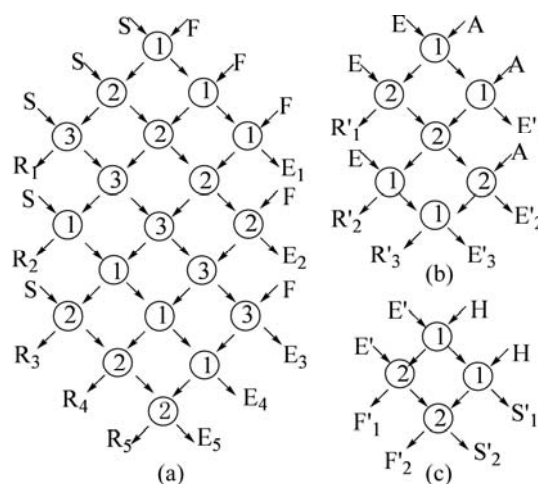
As shown in Table 2, the scrubbing yield of  $\text{Rb}^+$  decreased with increasing pH and decreasing  $O/A'$ . When  $c(\text{NaOH})=1 \times 10^{-4}$  mol/L and  $O/A'=1:0.5$ , the scrubbing yield of  $\text{Rb}^+$  was very low ( $E'(\text{Rb}^+)=10.5\%$ ), and 57.7% of  $\text{K}^+$  and 69.4% of  $\text{Na}^+$  could be scrubbed from the organic phase. As a result,  $\text{Rb}^+$  and  $\text{Cs}^+$  were further enriched in the organic phase. In the presence of considerable  $\text{Na}^+$  and  $\text{K}^+$  in the extractant, the actual concentrations of  $\text{Rb}^+$  and  $\text{Cs}^+$  after scrubbing remained low and were further enriched by reusing the extractant and scrubbing the extractant repeatedly through multi-stage extraction (Fig. 2).

### 3.6 Multi-stage continuous countercurrent extraction

Based on the above results, NaOH solution was added in the experimental brine (250 mL) from which potassium compounds had been separated in the fertilizer plant, aiming to eliminate  $\text{Mg}^{2+}$ . Then  $c(\text{OH}^-)$  was adjusted to 0.5 mol/L after precipitates were removed by centrifugation, and the contents of alkali metal ions were

analyzed by atomic absorption spectrometry. According to the distribution coefficients of these alkali metal ions in the experimental brine, 5-stage extraction was suitable for separating  $\text{Rb}^+$  and  $\text{Cs}^+$  from the brine. Therefore, 200 mL of brine in each bottle from which  $\text{Mg}^{2+}$  had been eliminated was extracted in 5-stage with 1.0 mol/L t-BAMBP+SK ( $O/A=1:1$ ) by shaking the bottle. The extraction procedure is shown in Fig. 5(a) and the results are shown in Table 3. The organic phase that was mixed with  $E_1$  to  $E_5$  after extraction was scrubbed in 3-stage with  $1 \times 10^{-4}$  mol/L NaOH ( $O/A'=1:0.5$ , Fig. 5(b)). The organic phase that was mixed with  $E'_1$  to  $E'_3$  after scrubbing was stripped in 2-stage with 2 mol/L HCl ( $O/A''=1:1$ ). The stripping procedure is shown in Fig. 5(c) and the results are summarized in Table 4.

The extracted organic phase was scrubbed in 3-stage with  $1.0 \times 10^{-4}$  mol/L NaOH. Most of  $\text{K}^+$  ions were eluted with  $E'(\text{K}^+)=97.81\%$ , and  $\text{Rb}^+$  ions were



**Fig. 5** Schemes of multi-stage continuous countercurrent extraction: (a) 5-stage countercurrent extraction; (b) 3-stage countercurrent scrubbing; (c) 2-stage countercurrent stripping (The diagram circle stands for single-stage extraction, and the figure in circle represents serial number. F: aqueous phase after  $\text{Mg}^{2+}$  precipitation; S: fresh extractant; E: organic phase loading alkaline metal ions after extraction; R: raffinate; A:  $1 \times 10^{-4}$  mol/L NaOH; E': organic phase after scrubbing; R': aqueous phase after scrubbing; H: 2.0 mol/L HCl; F': aqueous phase after stripping; S': organic phase after stripping. Temperature 20 °C, time 5 min)

**Table 3** Results of 5-stage continuous countercurrent extraction

Stage	$E(\text{Rb}^+)/\%$	$E(\text{K}^+)/\%$
$R_1$	95.04	21.54
$R_2$	94.42	19.41
$R_3$	93.72	13.45
$R_4$	88.96	10.87
$R_5$	80.77	11.80

**Table 4** Results of 2-stage continuous countercurrent stripping with HCl

Stage	$\rho(\text{Rb}^+)/$ ( $\text{mg}\cdot\text{L}^{-1}$ )	$\rho(\text{K}^+)/$ ( $\text{mg}\cdot\text{L}^{-1}$ )	$\rho(\text{Na}^+)/$ ( $\text{mg}\cdot\text{L}^{-1}$ )	$\rho(\text{Cs}^+)/$ ( $\text{mg}\cdot\text{L}^{-1}$ )	$P(\text{RbCl})/$ %	Single recovery efficiency/%
$S_1'$	50.70	3.45	7.65	<1.26	80.40	68.25
$S_2'$	14.80	1.00	6.16	<1.03	64.38	

hardly removed with  $E'(\text{Rb}^+)=14.81\%$ . The scrubbed liquid phase should be extracted repeatedly to increase the concentrations of  $\text{Rb}^+$  and  $\text{Cs}^+$ . When  $c(\text{NaOH})=0.5$  mol/L, the highest extraction efficiency of  $\text{Rb}^+$  reached 95.04% after 5-stage extraction, and the other cations (e.g.,  $\text{K}^+$  and  $\text{Na}^+$ ) had lower extraction yields, while the extraction yield of  $\text{Li}^+$  was negligible and almost all of  $\text{Cs}^+$  ions were extracted. After the organic phase was stripped with 2.0 mol/L HCl, the stripping yields of  $\text{Rb}^+$  and  $\text{Cs}^+$  were 95.2% and 99.5%, respectively. After the aqueous stripping solution was evaporated, the contents of RbCl and CsCl in the crystals were 85.40% and 9.25%, respectively. This mixed compound can be further separated easily as pure RbCl and CsCl by extraction, and relevant studies are still ongoing in our group.

## 4 Conclusions

1) The conditions under which trace  $\text{Rb}^+$  and  $\text{Cs}^+$  were extracted from the salt brine with potassium removed previously in plant were optimized. NaOH solution was added in the experimental brine to eliminate  $\text{Mg}^{2+}$  firstly. Then  $c(\text{OH}^-)$  was adjusted to 1.0 mol/L after  $\text{Mg}(\text{OH})_2$  was removed by centrifugation. The liquid phase was extracted with 1.0 mol/L t-BAMBP+SK at O/A=1:1. By using single-stage extraction, the extraction efficiency of  $\text{Rb}^+$  reached up to 90.8%, and the stripping efficiency was up to 84.6%.

2) After 5-stage continuous countercurrent extraction, the final extraction efficiency of rubidium reached 95.04%, and the purity of rubidium chloride reached 85.40% after 3-stage scrubbing by using  $1.0\times 10^{-4}$  mol/L NaOH and 2-stage stripping by using 2.0 mol/L HCl.

3) The extraction process proposed in this work can readily and economically be applied to extract trace  $\text{Rb}^+$  and  $\text{Cs}^+$  simultaneously from the brine of salt lakes in China or the brine containing copious other cations with potassium products removed. Hence, the method is highly valuable in practice.

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## t-BAMBP-煤油溶液萃取盐湖卤水中铷和铯离子

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**摘 要:** 将工厂提钾后的盐湖卤水作为提取  $\text{Rb}^+$  和  $\text{Cs}^+$  的实验用卤水, 将萃取剂 t-BAMBP 的磺化煤油溶液作为有机相进行萃取。在萃取之前预先沉淀出镁并作为一种产品, 在反萃前再多次洗涤分离出大部分的  $\text{K}^+$  和  $\text{Na}^+$ , 最终使  $\text{Rb}^+$  和  $\text{Cs}^+$  得到有效富集和分离。研究油水相比(O/A)、水相的碱性( $c(\text{OH}^-)$ )、 $\text{K}^+$  和  $\text{Mg}^{2+}$  的含量及洗涤油水相比(O/A)对萃取过程的影响。最佳工艺条件为: 1.0 mol/L 的 t-BAMBP 磺化煤油溶液, 水相碱性  $c(\text{OH}^-)=1$  mol/L, 油水相比 O/A=1:1。当用  $1 \times 10^{-4}$  mol/L NaOH 溶液洗涤萃取油相 3 次, 洗涤油水相比 O/A'=1:0.5 时, 铷和铯的洗脱率仅为 10.5%。经过 5 级逆流萃取, 最终铷和铯的萃取率分别达到了 95.04% 与 99.80%。

**关键词:** 盐湖卤水; t-BAMBP; 萃取; 铷离子; 铯离子

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