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Biphasic Water Systems Based on Alkylbenzyltrimethylammonium Chloride and Inorganic Salting-out Agents

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Abstract

The development of new two-phase aqueous systems based on surfactants for extraction requires the identification of patterns of delamination depending on the nature of the surfactant and salting-out agent. The phase equilibria in ternary aqueous delaminating systems containing a cationic surfactant – alkylbenzyltrimethylammonium chloride (catamine AB) and an inorganic salting-out agent were studied. Catamine AB forms stratifying systems with chlorides (KCl, NaCl, NH₄Cl, LiCl, CaCl₂), sulphates (K₂SO₄, Na₂SO₄, (NH₄)₂SO₄), nitrates (KNO₃, NH₄NO₃, NaNO₃, Mg(NO₃)₂, LiNO₃, Al(NO₃)₃, Ca(NO₃)₂), as well as with Na₂CO₃, K₂CO₃, KHCO₃, NaBr, KSCN, NH₄F, (NH₄)₂HPO₄ and HNO₃. The influence of the nature of the salting-out agent on the process of system delamination is discussed. The best salting-out agents for catamine AB are nitrates, which have relatively low hydration energy in the series of studied anions. The phase diagram of the water – catamine AB – sodium chloride system was built up. The stratification region exists in a rather wide acidity range up to 2.4 mol/L H₂SO₄ (or HCl), and up to 2.8 mol/L NH₃ (or 4.7 mol/L NaOH). The phases remain transparent at all studied concentrations of inorganic acids and bases. The extraction ability of the water – catamine AB – sodium chloride system was evaluated. Thallium (III) is quantitatively recovered in the range of sulphuric acid concentrations from 0.5 to 2 mol/L. The range of recoverable metals may be expanded by using organic complexing agents. Quantitative extraction of lanthanum (100 µg) is observed in the system containing 2.2 g of surfactant, 2.0 g of the salting-out agent, 1 mL of 0.01 mol/L Arsenazo III solution with a total volume of 15 mL and pH ~ 8.

Keywords: phase equilibria; catamine AB; two-phase water systems

INTRODUCTION

Organic solvents may be excluded from liquid extraction by using aqueous systems that stratify as a result of chemical interactions between the components of the aqueous solution [1], and also stratifying aqueous solutions of surface-active substances (SAS) after the addition of salting-out reagents. Among anionic SAS, the compounds possessing this ability are alkyl sul-

phates, alkyl sulphonates [2, 3], oxyphos B (potassium bis(alkylpolyoxoethylene)phosphate) [4]; among non-ionic substances – synthanols (alkyl ethers of polyethylene glycol) [5], synthamides (polyethylene glycol esters of monoethanolamides of synthetic fatty acids) [6], neonols (oxyethylated nonylphenols) [7]; among cationic substances – cetylpyridinium chloride [8], catamine AB (alkyldimethylbenzylammonium chloride) [9, 10]. Mixtures of anionic and cationic SAS

are able to get stratified without the addition of a salting-out agent [11, 12]. A specific feature of these systems is a high concentration of water in the extract, which provides hydration of the extracted hydrophilic compounds.

The development of new extraction systems "water – SAS – salting-out agent" requires determination of the regularities of layering depending on the nature of SAS and the salting-out agent. For polyethylene glycol as an example, it is demonstrated that the salting-out capacity of the salts is dependent first of all on the energy of anion hydration [13]. The same regularity is observed for oxyethylated SAS: synthamides [14], neonols [15] and oxyphos B [16].

The goal of the present work was to reveal the effect of the nature of salting-out agent on phase equilibria of the aqueous solutions of a cationic SAS containing no oxyethyl groups, namely catamine AB, and to demonstrate the possibility to use it for extraction.

EXPERIMENTAL

Alkylbenzyltrimethylammonium chloride, $[C_nH_{2n+1}N^+(CH_3)_3CH_2C_6H_5]Cl$, where $n = 10-18$, 50 % aqueous solution (catamine AB), TU 9392-003-48482528-99. It may contain up to 0.5 % ternary amines and up to 1.7 % salts of ternary amines as impurities.

The boundaries of the regions of stratification in the systems "water – catamine AB – salting-out agent" were determined by means of isothermal titration, phase transitions were recorded visually. The system "water – catamine AB – sodium chloride" was studied using the cross sections method [17]. The coordinates of the critical point were determined using Alekseev's rule. The system is conventionally three-component because catamine AB is a mixture of a number of homologues with technological impurities. In our studies, 100 % catamine AB in phase diagrams is a commercially available preparation – 50 % aqueous solution of alkyltrimethylbenzylammonium chloride (Catamine AB, TU 9392-003-48482528-99).

The distribution of $1 \cdot 10^{-4}$ moles of metal ions in the system "water – catamine AB – NaCl – H_2SO_4 " was studied placing the solution containing 2.25 g NaCl, 1 mL of 0.1 mol/L metal salt, 3 mL 50 % solution of catamine AB into a separating funnel; the solution of sulphuric acid was used to make the necessary acidity, and the volume was increased to 15 mL by adding distilled

water. The mixture was stirred for 1 min. After stratification, the extract was quantitatively transferred into a conical flask for titration, 50 mL of distilled water was added, and metal content was determined by means of chelatometry. This method was used also to determine the residual content of metal ions in the raffinate. Extraction extent was calculated using the obtained results.

RESULTS AND DISCUSSION

Catamine AB forms stratifying systems with chlorides (KCl , $NaCl$, NH_4Cl , $LiCl$, $CaCl_2$), sulphates (K_2SO_4 , Na_2SO_4 , $(NH_4)_2SO_4$), nitrates (KNO_3 , NH_4NO_3 , $NaNO_3$, $Mg(NO_3)_2$, $LiNO_3$, $Al(NO_3)_3$, $Ca(NO_3)_2$), as well as with Na_2CO_3 , K_2CO_3 , $KHCO_3$, $KSCN$, $NaBr$, NH_4F , $(NH_4)_2HPO_4$ and HNO_3 . Both phases are mobile transparent liquids (except the systems with carbonates), in which the upper layer is a turbid gel-like phase.

The boundaries of stratification regions formed after the introduction of various nitrates into the aqueous solution of catamine AB are presented in Fig. 1. The regions of two-phase equilibrium are situated close to the water apex (up to 99 mass % of water) and exist within a rather broad acidity range.

The area of the stratification region depends on the nature of the salt and increases as a sequence: $KNO_3 < Mg(NO_3)_2 < NaNO_3 < NH_4NO_3 < Ca(NO_3)_2 < LiNO_3 < Al(NO_3)_3$.

In the systems with chlorides (Fig. 2), stratification regions are substantially more narrow and are situated at a longer distance from the water apex of the composition triangle, water concentration in the stratifying mixtures does not exceed 85 mass %. On the basis of the value of stratifying region, the salts may be ranged as $CaCl_2 > LiCl > NH_4Cl > NaCl > KCl$.

In addition to the mentioned salts, stratification of the aqueous solutions of catamine AB was observed in the presence of carbonates (Fig. 3). These systems may be used for extraction from alkaline solutions. Stratification boundaries in the systems with sulphates (Fig. 4), ammonium fluoride, ammonium hydrophosphate are rather narrow, and water concentration in stratifying mixtures is lower than in nitrate and chloride systems. In the system with sodium bromide, stratification is conserved within a broad concentration range (Fig. 5).

A phase diagram of the system "water – catamine AB – sodium chloride" was plotted (Fig. 6). The phase regions established in the diagram are: L – homogenous, $L_1 + L_2$ – strati-

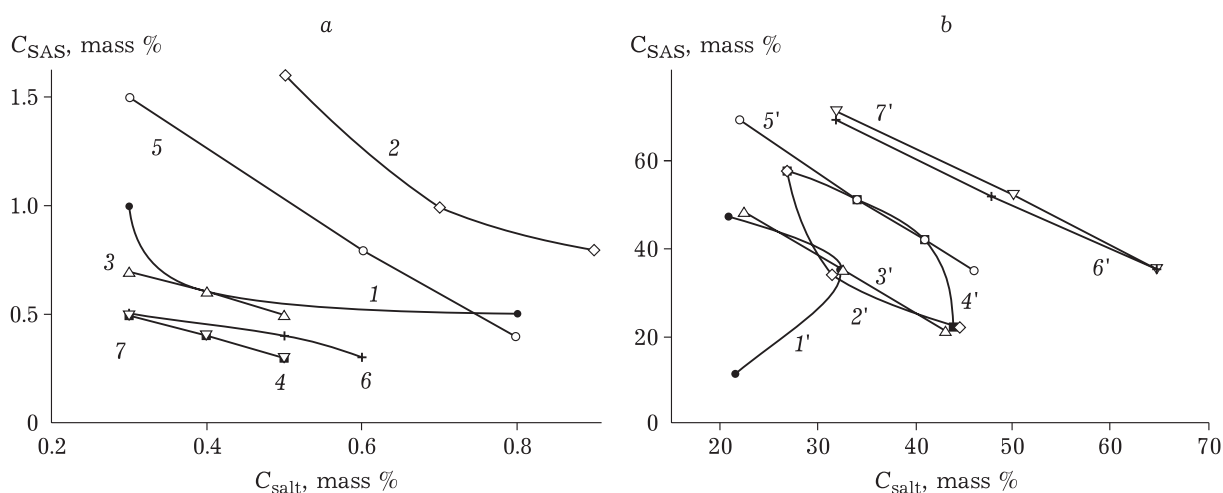


Fig. 1. Upper (a) and lower (b) boundaries of the stratification region in the systems "water - catamine AB - metal nitrate": 1, 1' - KNO_3 ; 2, 2' - $\text{Mg}(\text{NO}_3)_2$; 3, 3' - NaNO_3 ; 4, 4' - NH_4NO_3 ; 5, 5' - $\text{Ca}(\text{NO}_3)_2$; 6, 6' - LiNO_3 ; 7, 7' - $\text{Al}(\text{NO}_3)_3$. Here and in Fig. 2-5: C_{SAS} , C_{salt} are the concentrations of catamine AB and the salt, respectively. Non-primed digits designate the upper boundaries, primed digits mark the lower boundaries of the stratification.

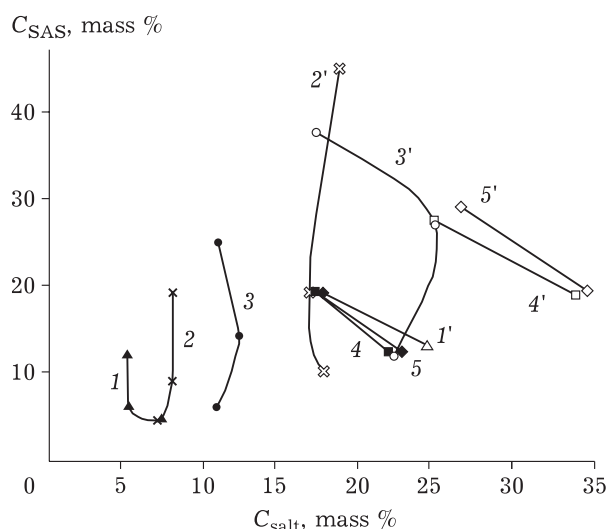


Fig. 2. Boundaries of the stratification region in the systems "water - catamine AB - metal chloride": 1, 1' - NaCl ; 2, 2' - KCl ; 3, 3' - NH_4Cl ; 4, 4' - CaCl_2 ; 5, 5' - LiCl . For designations, see Fig. 1.

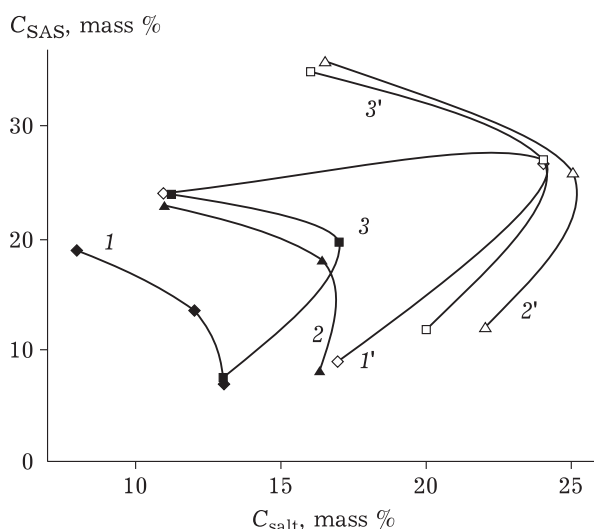


Fig. 3. Boundaries of the stratification region in the systems "water - catamine AB - metal carbonate": 1, 1' - Na_2CO_3 ; 2, 2' - KHCO_3 ; 3, 3' - K_2CO_3 . For designations, see Fig. 1.

fying, $L_1 + L_2 + S$ - monotectic equilibrium, $L + S$ - crystallization of sodium chloride. The nodes of the stratification area are almost parallel to the limiting node. The concentration ratio catamine AB/water/ NaCl in the critical point is equal to 8.0 : 84.0 : 8.0 (mass %). Stratification region is located as a narrow band along the catamine AB - water side and occupies 12.3 % of the area of the concentration triangle. Water concentration in the stratifying mixtures does not exceed 85.6 mass %, sodium chloride - 4.5 to 22.0 mass %. Both phases are transparent mobile liquids. The SAS phase is slightly yellowish, which is characteristic of catamine AB.

The composition of equilibrium liquid phases corresponding to the nodes of the stratification region is presented in Table 1. The phase enriched with SAS contains a substantial amount of salt and water, which provides hydration of extractable hydrophilic compounds unlike in the case of traditional extraction.

For the application of this system in extraction, an essential characteristic is the acidity range (pH) within which stratification is conserved. Investigation of the relations between the volumes of phases for different amounts of inorganic acids and alkalis was carried out in measuring tubes with tight caps. The total volume of the

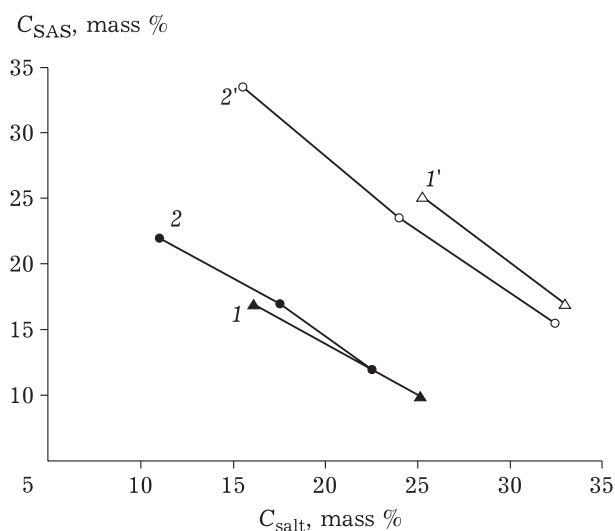


Fig. 4. Boundaries of the stratification region in the systems "water - catamine AB - metal sulphate": 1, 1' - $\text{Al}_2(\text{SO}_4)_3$; 2, 2' - $(\text{NH}_4)_2\text{SO}_4$. For designations, see Fig. 1.

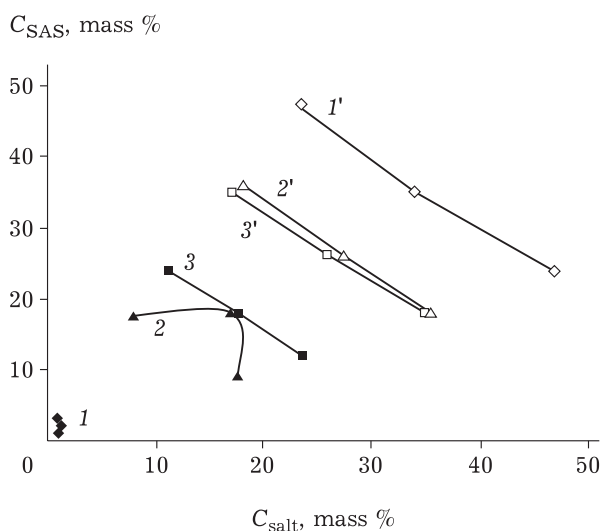


Fig. 5. Boundaries of the stratification region in the systems "water - catamine AB - metal salts": 1, 1' - NaBr; 2, 2' - NH_4F ; 3, 3' - $(\text{NH}_4)_2\text{HPO}_4$. For designations, see Fig. 1.

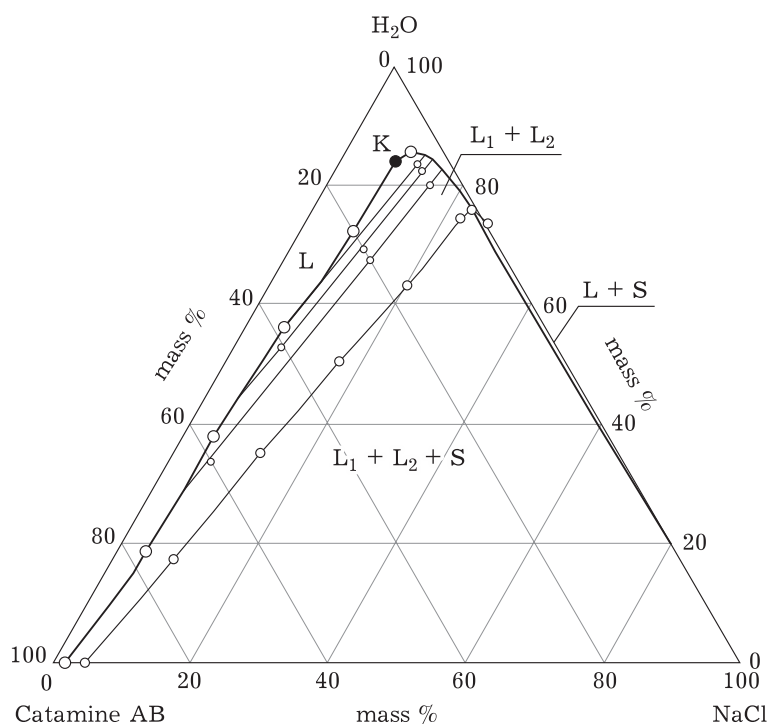


Fig. 6. Phase diagram of the system "water - catamine AB - NaCl" at 25 °C.

system was maintained to be equal to 15 mL, with catamine AB and NaCl content equal to 1.5 and 2.25 g, respectively. Under these conditions, the volume of the SAS phase is 1.2 mL, which is sufficient for practical purposes. The solutions of catamine AB in the absence of salting-out agents, inorganic acids and alkalis have pH ~ 6. The SAS phase is a transparent mobile pale-yellowish liquid above the aqueous phase. The region

of system stratification exists within a rather broad acidity range up to 2.4 mol/L H_2SO_4 (HCl). The phases of the system remain transparent for the whole studied concentration range of inorganic acids. It should be stressed that the volume of the SAS phase remains practically unchanged with an increase in the concentration of H_2SO_4 . In the presence of HCl, a slight decrease in the volume of the SAS

TABLE 1

Composition of equilibrium liquid phases (mass %) in the system "water – catamine AB – sodium chloride"

No.	Bottom phase (aqueous)			Top phase (SAS phase)		
	Catamine AB*	NaCl	Water	Catamine AB*	NaCl	Water
1	1.8	11.4	86.8	18.4	6.0	75.6
2	1.3	12.6	86.1	25.3	4.8	69.9
3	0.9	14.4	84.7	33.1	4.8	62.1

* Calculated for 100 % substances.

phase (to 0.8 mL) is observed for acid concentration higher than 1.6 mol/L.

Layering is conserved in the alkaline medium. After the introduction of NH_3 , the volume of the SAS phase remains almost unchanged, both phases remain transparent. The system becomes homogeneous when NH_3 concentration reaches 2.8 mol/L. With the addition of an alkali, the stratification region is conserved even in a broader pH range. Some decrease in the volume of the SAS phase from 1.2 to 0.8 mL is observed. It is established that the introduction of NaOH to the concentration of 4.7 mol/L does not disturb two-phase liquid equilibrium. Media with higher alkalinity were not studied.

A possible area of the application of the studied stratifying systems, namely liquid extraction, poses a number of special requirements:

- minimal possible content of SAS and salt;
- sufficient volume of the extract for practical purposes;
- transparent phases (when the systems are used for extraction-photometric methods of analysis);
- possibility to vary water concentration with the conservation of stratification;
- a broad range of acidity for the existence of the stratification region.

On the basis of these requirements, specific components ratios were determined for the systems of the highest interest for extraction (Table 2).

The salting-out ability of salts was determined from the area of stratification region: $\text{LiNO}_3 > \text{KSCN} > \text{NH}_4\text{NO}_3 > \text{Al}(\text{NO}_3)_3 > \text{Ca}(\text{NO}_3)_2 > \text{NaNO}_3 > \text{HNO}_3 > \text{CaCl}_2 > \text{LiCl} > \text{KCl} > \text{NH}_4\text{Cl} > \text{NaCl}$. The best salting-out agents for catamine AB are nitrates, as they provide the minimal value of hydration energy in the series of the studied anions. The observed dependence may be explained by the formation of the ionic associate of a positively charged SAS micelle with the anion of the salting-out agent. The

lower is the absolute value of the energy of anion hydration, the less is its hydration extent. The formed associate has higher hydrophobicity and forms its own phase more easily.

Catamine AB exhibits cation-active nature, therefore, there is the possibility of the extraction of metals able to form acidocomplexes, according to the anion exchange mechanism. The distribution of 0.01 mol/L metal ions depending on the concentration of sulphuric acid was studied in the system "water – catamine SA – sodium chloride" (Fig. 7). The recovery degree (R) of the microamounts of elements was determined from the content of these elements in the aqueous phase and in the extract. An advantage of the systems of this type is in the ability of the extract to be dissolved in water. Because of this, to determine metal ion content in the extract, it was transferred quantitatively into the flask for titration, 50–70 mL of distilled water were added, the necessary pH level was adjusted, the corresponding indicator was added, and the amount of the recovered metal was determined by means of chelatometry.

In the absence of an inorganic acid in this system, the extraction of metal ions is not quantitative. The introduction of H_2SO_4 leads to an increase in the degree of recovery of the majority of metal ions except for Cu, Zn, Cd. Of the highest interest in this system is the recovery of Tl (III). For the quantitative extraction of very stable tetrachlorothallate, the sufficient H_2SO_4 concentration is 0.5 mol/L. For the concentration of sulphuric acid 2 mol/L, the maximal degree of metal ion extraction is observed, %: Fe (III) – 80, Mo (VI) – 76, Ga – 51, Sc – 18. Further increase in acid concentration is impossible because homogenization of the system occurs. Metal ions are extracted according to the ion exchange mechanism in the form of chloride acidocomplexes. The proposed mechanism of Tl^{3+} extraction may be represented by the equation:

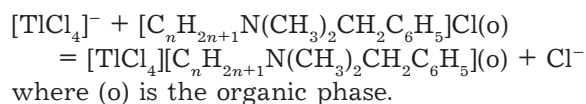
TABLE 2

Characterization of the stratifying region for the systems "water – catamine AB – salting-out agent"

Salting-out agent	m_{SAS} , g	m_{salt} , g	V_{SAS} , mL	pH_{eq}	C_{min} , %	S_{str} , %	Range of acidity for the existence of the stratification region
NaCl	1.25	2.5	1.0	6.3	10.9	12.3	from 2.4 mol/L H_2SO_4 (HCl) to 2.8 mol/L NH_3 (up to > 4.7 mol/L NaOH)
KCl [9]	1.5	1.3	3.0	6.0	9.4	21.6	from 3.5 mol/L HCl (3.0 mol/L H_2SO_4) to 4.0 mol/L NH_3 (0.47 mol/L NaOH)
LiCl [9]	–	–	–	–	13.6	26.5	–
$CaCl_2$ [9]	–	–	–	–	17.5	27.4	–
NH_4Cl [9]	0.95	2.45	2.4	5.0	12.8	20.6	from 2.8 mol/L HCl (1.4 mol/L H_2SO_4) to 0.4 mol/L NaOH
NH_4NO_3 [10]	0.9	1.0	1.0	5.6	1.5	76.3	from 8 mol/L HCl (11.5 mol/L H_2SO_4) to 7.9 mol/L NaOH (4.5 mol/L NH_3)
HNO_3 [10]	–	–	–	–	2.0	32.5	–
$LiNO_3$ [10]	–	–	–	–	1.5	81.2	–
$NaNO_3$ [10]	–	–	–	–	1.7	57.7	–
$Ca(NO_3)_2$ [21]	–	–	–	–	0.6	64.5	–
$Al(NO_3)_3$ [22]	–	–	–	–	1.3	73.3	–
KSCN [22]	0.9	0.6	–	–	3.5	76.4	to 6.0 mol/L H_2SO_4 (HCl)

Note. 1. m_{SAS} , V_{SAS} are the mass and volume of the SAS phase (catamine AB); m_{salt} is the mass of the salting-out agent; C_{min} is the minimal concentration of the salting-out agent at which the stratification is observed; pH_{eq} is the equilibrium pH value for the indicated amounts of components in the system; S_{str} is the area of the stratification region, in % of the area of the composition triangle. 2. Dash means the absence of data.

* Total volume of the system is 15 mL.



The data on the extraction of metal ions from solutions with HCl have been published previously [18].

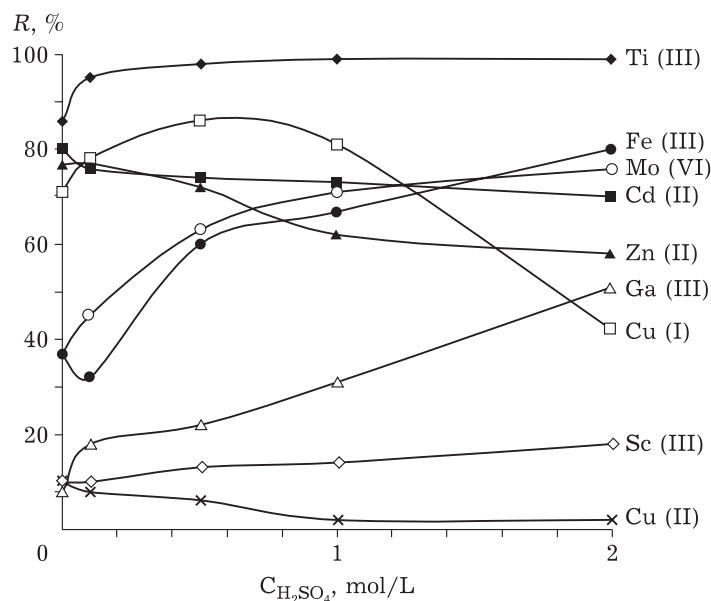


Fig. 7. Extraction of metal ions ($1 \cdot 10^{-4}$ mol) in the system "water – catamine AB – NaCl – H_2SO_4 ". R , % is the degree of metal ion recovery; $C_{H_2SO_4}$ is the concentration of sulphuric acid, mol/L; the mass of catamine AB $m_{catamine\ AB} = 1.5$ g; $m_{NaCl} = 2.25$ g; total volume $V = 15$ mL.

It is possible to broaden the range of recoverable metals by using organic complex-forming reagents. For example, a known photometric reagent arsenazo I in the system “water – catamine AB – sodium chloride” is extracted with the distribution coefficient equal to 470 [19]. The conditions of the quantitative recovery of lanthanum ions in the form of intracomplex compound with this reagent were determined. Lanthanum extraction was carried out in separating funnels in the presence of catamine AB 2.2 g, sodium chloride 2.0 g, 1 mL of 0.01 mol/L arsenazo I solution, and the solution of lanthanum nitrate containing 100 µg of the metal, to which ammonia solution was added to reach pH 8 (optimal conditions for the formation of the complex compound). Then the total volume of the system was brought to 15 mL by adding distilled water. The volume of the SAS phase was 2.8 mL. Lanthanum content in the extract was determined by means of photometry [20]. For this purpose, the extract was transferred quantitatively into the measuring flask 25 mL in volume, distilled water was added to make up the volume, and the optical density of the resulting solution was measured with an SF-2000 spectrophotometer (Russia) at 540 nm using the extract of the blank experiment as the reference solution for optical background.

CONCLUSION

The studies showed that the aqueous solutions of catamine AB are able to get stratified into two liquid phases under the action of inorganic salts. The highest salting-out capacity is characteristic of the anions with relatively low hydration energy. The obtained two-phase aqueous systems are suitable for the extraction of metal ions. Extraction of acidocomplexes proceeds in the form of ion associates according to anion-exchange mechanism, with catamine AB supplying the cation part. In the presence of additional organic complex-forming reagents, extraction of intracomplex compound is possible.

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