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ARTICLES

Anion-Exchange Extraction of Doubly Charged Anions by Higher Quaternary Ammonium Salts with Different Steric Accessibility of the Exchange Center

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Abstract—An increase in the steric accessibility of a quaternary ammonium salt (QAS) exchange center leads to a significant increase in the exchange constant for most of the studied doubly charged anions (sulfate, sulfite, molybdate, tungstate, hydrogen phosphate, and selenate), which reaches for "pure" anion-exchange systems of more than five orders of magnitude. The effect of minimizing of an anion-exchange affinity was detected when a solvating additive, heptyl *p*-trifluoroacetylbenzoate, was introduced into the organic phase. The effect of steric accessibility is not entirely lost due to the conservation of charge of the solvate. The introduction of a solvating additive improves the affinity of anions for the QAS phase significantly (up to 6.5 orders of magnitude). The effect of anion size on the effect of steric accessibility of the exchange center was found for sulfate, sulfite, and thiosulfate ions. An inverse relationship was found for the tetrathionate ion; that is, with a decrease in steric accessibility of the QAS exchange center, anion-exchange extraction is weakened due to the large size of the $S_4O_6^{2-}$ ion.

Keywords: anion-exchange extraction, quaternary ammonium salt, solvating additive

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Anion-exchange extraction with higher quaternary ammonium salts is widely used in the technology of nonferrous metals, rare, and trace elements and in other fields. Higher QASs have also proven themselves as promising analytical reagents for the extraction-photometric determination of hydrophobic anions, including metal complexes, and for the creation of anion-selective electrodes [1–4].

It is known that anion-exchange extraction of doubly charged anions with higher QASs proceeds insignificantly [5], and, therefore, extractants of this class are rarely used for their separation and determination. The search for the extractants for hydrophilic doubly charged anions that are difficult to preconcentrate and determine is an urgent task.

One of the ways to improve the extraction characteristics of anion exchange systems based on higher QASs is the use of neutral acidic carriers. It was known [6, 7] that trifluoroacetyl derivatives exhibit a high solvating ability with respect to carbonate and carboxylate ions; they are also used to study the possibility of extraction of other anions [1, 8, 9]. The effect of the concentration of hexyl *p*-trifluoroacetylbenzoate on the anion-exchange extraction of sulfate, acetate, benzoate, oxalate, carbonate, *o*-bromobenzoate, and *o*-benzoyl benzoate ions was studied in [5, 10, 11].

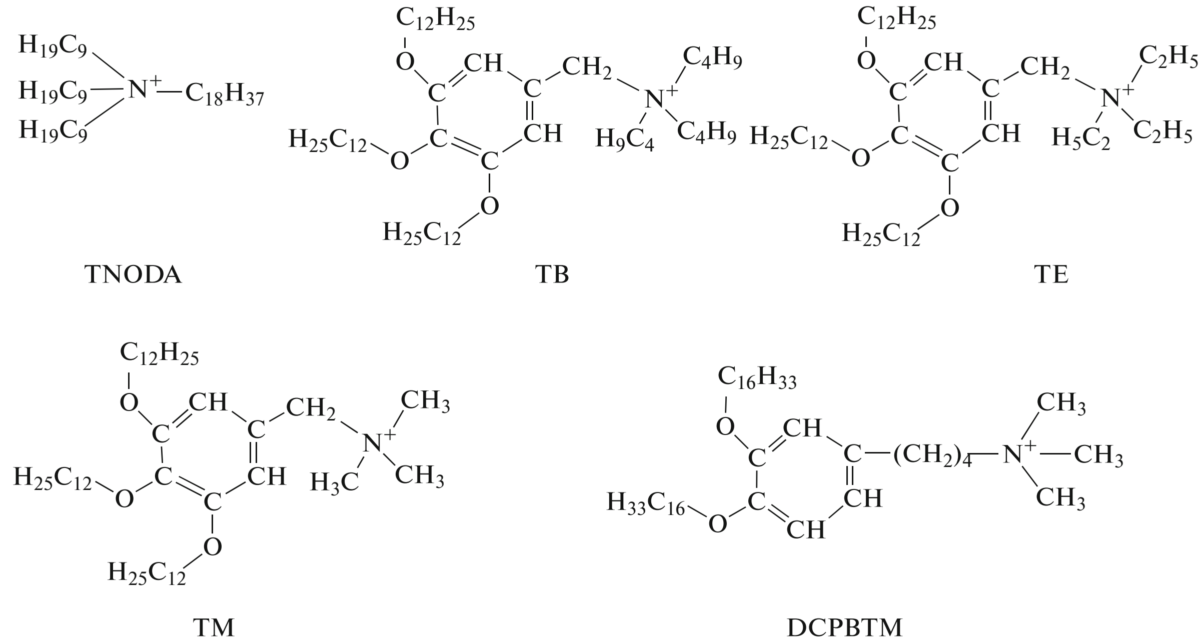
The targeted use of anion-exchange extraction systems for both analytical and applied purposes should be based on a series of anion-exchange affinity, in which the anions are arranged in accordance with the exchange constants for a standard anion, which is usually chloride. However, some doubly charged anions are absent in the Hofmeister series: $ClO_4^- > SCN^- > BF_4^- > I^- > NO_3^- > Br^- > Cl^- > Ac^- > SO_4^{2-} > F^- > H_2PO_4^- > C_2O_4^{2-} > HCO_3^- > HPO_4^{2-} > CO_3^{2-} > PO_4^{3-} > OH^-$ [12, 13].

To determine the exchange constants, the intermediate anion method was previously proposed [14, 15], using a suitable acid dye, for example, Methyl Red (MR) in the salt (sodium) form (MR^-Na^+).

The goal of this work was to study the effect of the steric accessibility of the exchange center of higher QASs on the anion-exchange affinity of doubly charged anions (sulfate, sulfite, thiosulfate, tetrathionate, molybdate, tungstate, hydrogen phosphate, and selenate ions) for the chloride ion by the intermediate ion method both in pure anion-exchange systems and in the presence of a solvating additive, heptyl *p*-trifluoroacetylbenzoate. The effect of the size of a doubly charged inorganic anion on the magnitude of the steric accessibility effect of the QAS exchange center was also examined.

EXPERIMENTAL

Extraction was studied at a temperature of 298 ± 1 K. The following higher QASs were used, the synthesis of which was described in [16, 17]: R-tributylammonium, R-triethylammonium, and R-trimethylammonium bromides, where R is 3,4,5-tris(dodecyloxy)benzyl, trinonyloctadecylammonium iodide, or 4-(3,4-dicetoxyphenyl)butyltrimethylammonium chloride. The chemical structures of these substances are presented in Scheme 1.



Scheme 1. Chemical structure of the cations of quaternary ammonium salts: trinonyloctadecylammonium (TNODA), 3,4,5-tris(dodecyloxy)benzyltributylammonium (TB), 3,4,5-tris(dodecyloxy)benzyltriethylammonium (TE), 3,4,5-tris(dodecyloxy)benzyltrimethylammonium (TM), and 4-(3,4-dicetoxyphenyl)butyltrimethylammonium (DCPBTM).

All higher QASs were purified in a hexane–acetonitrile/isopropyl alcohol extraction system. The selected QASs significantly differ in steric accessibility of the exchange center (the quaternary nitrogen atom), since they contain hydrocarbon radicals of various lengths from C_1 to C_9.

We used Methyl Red (analytical grade), toluene (analytical grade), NaCl (cp grade), NaOH (fixanal), $(NH_4)_2SO_4$ (cp grade), Na_2SO_4 (analytical grade), Na_2SO_3 (analytical grade), $Na_2S_2O_3 \cdot 5H_2O$ (analytical grade), $Na_2S_4O_6$ (synthesized by the procedure [18]), $Na_2MoO_4 \cdot 2H_2O$ (cp grade), $Na_2WO_4 \cdot 2H_2O$ (cp grade), $Na_2HPO_4 \cdot 12H_2O$ (analytical grade), Na_2SeO_4 (technical grade), and aqueous ammonia (high-purity grade).

Heptyl *p*-trifluoroacetylbenzoate (H*p*-TFAB) was synthesized at the Department of Analytical Chemistry of the Belorussian State University according to the procedure [16].

Extraction was carried out in test tubes with ground stoppers. The organic phase of the extraction system consisted of the QAS^+MR^- solutions in toluene (c = 0.001 M); the aqueous phase was the solutions of the corresponding salts with a concentration of 5×10^{-4} to 1.0 M. The pH in the solutions of all salts was maintained at 9.8 ± 0.1, because it was previously found that the distribution coefficient P for Methyl Red in the toluene–water system has a constant value of 2×10^3 at pH > 9.5.

After reaching the equilibrium concentration and layering, the phases of the extraction system were analyzed by photometry for the dye concentration (a Solar CM2203 spectrofluorimeter) at 410 and 435 nm for the organic and aqueous phases, respectively. The molar absorption coefficient for an aqueous solution of Methyl Red is 2×10^4, and for a toluene solution of QAS^+MR^-, it is $(6.2 \pm 0.2) \times 10^3$.

The conversion of QAS to the QAS^+MR^- form was carried out as follows: 100 mL of a 0.001 M QAS^+Cl^- toluene solution was treated several times with 100 mL of a 0.005 M MR^-Na^+ solution in a separatory funnel; the completeness of the conversion of QAS into the QAS^+MR^- form was monitored photometrically until the constant absorbance was reached. If QASs were in bromide or iodide forms, then 100 mL of their toluene solutions were pretreated four times with 20 mL of a

5 M NaCl solution, and then washed three times with 100 mL of distilled water.

The main reaction of anion-exchange extraction is described by the equation

$$2\mathrm{QAS}^+\mathrm{MR}^- + \mathrm{An}^{2-} \leftrightarrow \left(\mathrm{QAS}^+\right)_2\mathrm{An}^{2-} + 2\mathrm{MR}^-. \quad (1)$$

The exchange constants of anions for a standard chloride ion were calculated by the ratio

$$K_{2\mathrm{Cl}^-}^{\mathrm{An}^{2-}} = \frac{[(\mathrm{R_3R'N^+})_2\mathrm{An}^{2-}]_{\mathrm{org}}[\mathrm{Cl}^-]^2_{\mathrm{aq}}}{[\mathrm{R_3R'N^+Cl^-}]^2_{\mathrm{aq}}[\mathrm{An}^{2-}]_{\mathrm{aq}}}. \quad (2)$$

There is the possibility of complexation of sodium cations and doubly charged anions. The pK of the complex $\mathrm{NaSO_4^-}$ is 0.72, and for the complex $\mathrm{NaS_2O_3^-}$, it is 1.08 [19]. In this regard, preliminary studies were carried out in which the concentrations of doubly charged anions, especially sulfate and thiosulfate, were varied. In [20], to study the anion-exchange equilibria involving sulfate ions, their constant concentration of 0.9 M was used. It was found that at the same concentrations of sodium sulfate and ammonium sulfate (for example, 0.1 M), in the case of $\mathrm{(NH_4)_2SO_4}$, the sulfate ions displaced 1.5–2 times more MR^- ions from the organic phase compared with $\mathrm{Na_2SO_4}$. According to [3], the complex ion $\mathrm{NaSO_4}^-$ is practically not extracted by QAS. No data were found on the instability constants of the complexes of the ammonium cation with the studied anions.

The constant values of the exchange constants $K(\mathrm{An}^{2-}/2\mathrm{Cl}^-)$ are reached for the concentrations of An^{2-} ions less than 5×10^{-3} M, which is due to a decrease in the complexation effect and, accordingly, an increase in the activity of exchanging anions (it should be noted that no noticeable effect of ionic strength on exchange constants for singly charged ions is found [3]).

The dependence of the exchange constant (for sulfate ions) on the salt concentration (ionic strength of the solution, μ) is described by the equation [3]

$$\log K'^{\mathrm{SO_4^{2-}}}_{2\mathrm{Cl}^-} = \log K^{\mathrm{SO_4^{2-}}}_{2\mathrm{Cl}^-} + 2.27\frac{\sqrt{\mu}}{1+\sqrt{\mu}}, \quad (3)$$

where $K^{\mathrm{SO_4^{2-}}}_{2\mathrm{Cl}^-}$ is the ion exchange constant close to thermodynamic.

In this work, we present concentration exchange constants that do not take into account the change in the concentration of doubly charged ions due to the formation of Na^+ complexes with An^{2-}, because the values of the instability constants of Na^+ with An^{2-} may not be reliable (or are absent). Apparently, these constants were calculated by changing the total activity of particles in solutions of the corresponding sulfates or thiosulfates [19]. In addition, it is difficult to take into account the activity coefficients of Na^+, $\mathrm{SO_4^{2-}}$, $\mathrm{S_2O_3^{2-}}$, and $\mathrm{NaSO_4^-}$, and others against the background of complexation.

RESULTS AND DISCUSSION

Exchange constants of doubly charged anions for chloride ion in pure anion-exchange systems. The values of the exchange constants $K(\mathrm{Cl}^-/\mathrm{MR}^-)$ for different QASs are presented below (with $c(\mathrm{Cl}^-)$ = 0.001 M).

QAS	TNODA	TB	TE	TM	DCPBTM
$K(\mathrm{Cl}^-/\mathrm{MR}^-)$	6.8×10^{-4}	4.5×10^{-4}	1.8×10^{-4}	7.6×10^{-5}	5.2×10^{-5}

The exchange constants $K(\mathrm{An}^{2-}/2\mathrm{Cl}^-)$ depending on the nature of the higher QAS are compared in Table 1. It is evident that the exchange constants strongly depend on the nature of the QAS used as an anion exchanger. All QASs have radicals of sufficient length to prevent their dissolution in water. In this regard, the dependence of $K(\mathrm{An}^{2-}/2\mathrm{Cl}^-)$ on the nature of the QAS cannot be explained by a change in the concentration of extractant or extraction products.

These effects can be explained by the features of the ionic association of QAS cations with exchanged anions [4, 20–22]. For singly charged anions, the association process proceeds in accordance with the equation

$$\mathrm{R_4N^+} + \mathrm{X^-} \xleftrightarrow{(K_{\mathrm{ass}})_{\mathrm{X^-}}} \mathrm{R_4N^+X^-}. \quad (4)$$

For doubly charged anions, the association goes in two stages, that is,

$$\mathrm{R_4N^+} + \mathrm{Y^{2-}} \xleftrightarrow{(K'_{\mathrm{ass}})_{\mathrm{Y^{2-}}}} (\mathrm{R_4N^+Y^{2-}})^-, \quad (5)$$

$$(\mathrm{R_4N^+Y^{2-}})^- + \mathrm{R_4N^+} \xleftrightarrow{(K''_{\mathrm{ass}})_{\mathrm{Y^{2-}}}} (\mathrm{R_4N})_2\mathrm{Y}^{2-}, \quad (6)$$

where $(K'_{\mathrm{ass}})_{\mathrm{Y^{2-}}}$, and $(K''_{\mathrm{ass}})_{\mathrm{Y^{2-}}}$ are the constants of the formation of associates according to Eqs. (5) and (6), respectively.

Then, taking into account the processes of association, the exchange constant of singly charged anions to doubly charged can be written in the form of

Table 1. Logarithms of the exchange constants $K(\mathrm{An}^{2-}/2\mathrm{Cl}^-)$ for various higher quaternary ammonium salts ($c_{\mathrm{An}^{2-}} = 0.001$ M)

Anion	TNODA	TB	TE	TM	DCPBTM
SO_4^{2-}	−2.4	−2.0	−0.3	1.2	2.0
SO_3^{2-}	−3.5	−3.0	−0.8	1.0	1.9
$S_2O_3^{2-}$	−2.1	−1.9	−0.9	0.2	0.6
$S_4O_6^{2-}$	−1.9	−2.2	−2.9	−3.3	−3.6
MoO_4^{2-}	−3.9	−3.7	−2.7	−1.4	−1.0
WO_4^{2-}	−5.0	−4.8	−4.0	−3.1	−2.6
HPO_4^{2-}	−3.0	−2.7	−1.1	0.4	0.8
SeO_4^{2-}	−2.9	−2.5	−0.1	1.6	2.2

$$\log K_{2X^-}^{Y^{2-}} = \log K_{2X^-}^{Y^{2-}}(\text{ion}) + \log \frac{\left(K'_{\text{ass}}\right)_{Y^{2-}}\left(K''_{\text{ass}}\right)_{Y^{2-}}}{(K_{\text{ass}})^2_{X^{2-}}}. \quad (7)$$

According to the Fuoss theory, the values of the ionic association constants depend on the charges of associating ions (z), their effective sizes (a), and the dielectric constant of the solvent (ε) as follows:

$$\log K_{\text{ass}} = 2.6 + 243\left|z_{\mathrm{An}^{2-}} z_{\mathrm{R_4N}^+}\right|/\varepsilon a + 3\log a. \quad (8)$$

An analysis of Eq. (8) enables one to explain the effect of the nature of the QAS cation on the relative efficiency of interaction with single- and double-charged anions. According to [4], in the case of contact ion pairs, when there are no solvent molecules between associated ions, the value a of the closest approach between the QAS cation and the anion is usually within 4–8 Å. Thus, in solvents with low ε (2.4 for toluene), the second term of Eq. (8) makes a decisive contribution to the value of the ion association constant. An improvement in the steric accessibility of the QAS exchange center, accompanied by a decrease in the parameter a of the closest approach, should result in an increase in the ion association constants. It also follows from Eq. (8) that, with comparable sizes of singly and doubly charged anions, the effect of the steric accessibility of the QAS exchange center on the value of the first association constant of the QAS cation with a doubly charged anion, $\left(K'_{\text{ass}}\right)_Y^{2-}$, should be much stronger than the value of the association constant with a singly charged anion, $(K_{\text{ass}})_X^-$, because of the more significant the magnitude of the charge $z_{Y^{2-}}$.

In using a QAS with four long-chain substituents, the ionic association with a doubly charged anion in the second stage (Eq. (6)) is significantly hindered by steric obstacles. An improvement in the steric accessibility of the exchange center should be accompanied by a sharp decrease in the parameter of the closest approach of the second QAS cation to the negatively charged associate $[\mathrm{QAS}^+\mathrm{Y}^{2-}]^-$. As a result, an increase in the second association constant $(K''_{\text{ass}})_Y^{2-}$ may turn out to be more significant than an increase in $(K_{\text{ass}})_X^-$ for singly charged anions, for which the steric accessibility of the QAS exchange center affects the value of a to a lesser extent.

Since the parameter a of the closest approach is, to a first approximation, an additive function of the radii of the anion and cation [22], it is evident that the effect of the steric accessibility of the QAS exchange center on the values of association constants is determined by the sizes of associating anions [23]. In particular, it was demonstrated using the example of singly charged anions [4] that more pronounced effects of changes in the values of the ion association constants are observed in the case of smaller anions [23], which causes the minimizing of the exchange constants.

Constants of exchange of doubly charged anions for chloride ion in anion-exchange systems containing a solvating additive, heptyl *p*-trifluoroacetylbenzoate. The values of the exchange constants $K(\mathrm{Cl}^-/\mathrm{MR}^-)$ for various higher QASs (the solvent is toluene, $c(\mathrm{H}p\text{-TFAB}) = 1 \times 10^{-2}$ M) are presented below:

QAS	TNODA	TB	TE	TM	DCPBTM
$K(\mathrm{Cl}^-/\mathrm{MR}^-)$	2.9×10^{-4}	1.0×10^{-4}	8.2×10^{-5}	5.4×10^{-5}	3.0×10^{-5}

Table 2. Logarithms of the exchange constants $K(\mathrm{An}^{2-}/2\mathrm{Cl}^-)$ for various higher quaternary ammonium salts ($c_{\mathrm{H}p\text{-TFAB}}$ = 0.001 M)

Anion	TNODA	TB	TE	TM	DCPBTM
SO_4^{2-}	2.6	2.7	3.2	3.6	3.9
SO_3^{2-}	2.0	2.3	3.1	3.4	3.6
$S_2O_3^{2-}$	1.7	1.9	2.4	2.6	2.6
$S_4O_6^{2-}$	–0.5	–0.7	–1.0	–1.3	–1.3
MoO_4^{2-}	2.0	2.0	2.4	2.6	2.6
WO_4^{2-}	1.5	1.5	1.8	2.0	2.1
HPO_4^{2-}	2.2	2.2	2.5	2.8	2.9
SeO_4^{2-}	2.8	2.9	3.4	3.8	4.2

The exchange constants $K(\mathrm{An}^{2-}/2\mathrm{Cl}^-)$ depending on the nature of the higher QAS are presented in Table 2. The introduction of a solvating additive leads, on the one hand, to a significant increase in the exchange constants, and, on the other hand, to their active reduction. For example, for sulfite, molybdate, and tungstate ions, an increase in the exchange constants reaches 5.5, 5.9, and 6.5 orders of magnitude, respectively (for the TNODA ion exchanger).

For example, for sulfite ions, the exchange constant increases by 1.4 orders of magnitude in the series of TNODA–TB–TE–TM–DCPBTM (Table 2), whereas in the absence of H*p*-TFAB in the organic phase, the steric effect reaches 5.4 orders of magnitude (Table 1). Apparently, the formation of a solvate shell around each of the studied anions leads to the reduction of the steric accessibility effect of QASs. At the same time, the effect manifests itself, which is due to the conservation of the solvate charge at –2.

Effect of the size of a doubly charged inorganic anion on the magnitude of the steric accessibility effect of higher quaternary ammonium salts. The data in Tables 1 and 2 also reflect the effect of the size of a doubly charged anion on the effect of steric accessibility of the QAS exchange center. Below are the radii of sulfur-containing doubly charged anions [23–25]:

Ion	SO_3^{2-}	SO_4^{2-}	$S_2O_3^{2-}$	$S_4O_6^{2-}$
Radius, nm	0.204	0.218	0.251	0.325

In the series of anions SO_3^{2-}–SO_4^{2-}–$S_2O_3^{2-}$, the effects associated with an increase in the steric accessibility of the exchange center both for purely anion-exchange systems (Table 1) and in the presence of a solvating additive in the organic phase (Table 2) level. For example, in the series of TNODA–TB–TE–TM–DCPBTM for the sulfite ion, the exchange constant increases by 5.4 orders of magnitude; for the sulfate ion, it increases by 4.4 orders of magnitude; for the thiosulfate ion, it grows by 2.7 orders of magnitude (Table 1).

An inverse relationship was found for the $S_4O_6^{2-}$ ion; that is, in the transition from TNODA to DCPBTM, the anion-exchange extraction is weakened. This is due to the large size of the ion, which, according to the Fuoss theory, should exhibit a higher anion-exchange affinity for sterically difficult QASs. The manifestation of such anion exchange affinity by tetrathionate ions is similar to the behavior of large metal complex anions [25–27].

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