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CHEMICAL EQUILIBRIUM OF TRIOCTYLMETHYLAMMONIUM CHLORIDE WITH AQUEOUS SOLUTION OF CR(VI)

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ABSTRACT

The distribution and dissociation equilibria of phase-transfer catalyst, trioctylmethyl-ammonium chloride (Q^+Cl^-), for the system of QCl–toluene–aqueous $HCrO_4^-$ solutions were measured at 298K for the systems of toluene–water or –aqueous electrolyte solutions.

The evaluated distribution coefficients of trioctylmethylammonium chloride and the complex formed between trioctylmethylammonium cation and $HCrO_4^-$ were correlated as a function of the ionic strength in the aqueous solutions. The dissociation constants, K_1 and K_2, were independent of ionic strength.

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Key Words: Distribution coefficient; Dissociation constant; Trioctylmethylammonium chloride; Ionic strength; Salting-out parameter

INTRODUCTION

Hexavalent chromium, Cr(VI), is a metal that has received considerable attention. It is used extensively in industrial applications such as electroplating, electrofinishing, steelmaking, leather tanning, and corrosion inhibition, and it has been recognized for long as a toxic substance due to its strong oxidizing potential and ease with which it can cross biological membranes (1). The removal of Cr(VI) from industrial wastewater is of great interest mostly due to the high toxicity of chromium(VI) compounds. During the recent years, the application of different solvent extraction technologies to chromium removal and concentration has been studied widely; conventional liquid–liquid extraction (2), emulsion liquid membranes (3), supported liquid membranes (4), and nondispersive extraction (5) are some of the new alternatives reported in the literature.

Quaternary ammonium salts, as a carrier, have been shown to be very effective for the removal of chromate ion from acid solution in the liquid membrane, i.e., trioctylamine (4), tri-dodecylamine (6), tri-*iso*-octylamine (7), and trioctylmethylammonium chloride (5,8–10). Hexavalent chromium may exist in the aqueous phase in different ionic forms with total chromate concentration and pH value dictating which particular chromate species will predominate (11). Because various chemical species of Cr(VI) in the aqueous solution are formed according to the total concentration of Cr(VI) and pH, several models are used to analyze the transport of Cr(VI) with a carrier. Teramoto et al. (4) presented the extraction model such as $HCrO_4^- + H^+ + \text{Aliquat 336} \leftrightharpoons$ complex, and studied the stability and performance of a spiral-type flowing liquid membrane module by using the measured flux of Cr(VI) and the feed-side mass transfer coefficient, but did not consider the transport mechanism of Cr(VI) with chemical reaction in the liquid membrane. Alonso et al. (9) studied the kinetic experiments on the extraction of Cr(VI) with Aliquat 336 performed in hollow fiber modules, whose model was $CrO_4^{2-} + 2\ \text{Aliquat 336} \leftrightharpoons \text{complex} + 2\,Cl^-$. Because the Cr(VI) ion exists mainly in the form of $HCrO_4^-$ in the pH range 2–5 and the Cr(VI) range 50–200 ppm (11), the equilibrium reaction of Cr(VI) with trioctylmethylammonium chloride can be described by the following model:

$$HCrO_4^- + \text{Aliquat 336} \leftrightharpoons \text{complex} + Cl^-$$

In this work, the aqueous dissociation constants of trioctylmethylammonium chloride (Aliquat 336) and its distribution coefficient between toluene and water or aqueous electrolyte solutions were measured in the pH range of 3 and the Cr(VI) range of 50–200 ppm.

EXPERIMENTAL

Measurements of the dissociation and distribution equilibria of trioctylmethylammonium chloride, $Q^{+}Cl^{-}$ (Q^{+}; trioctylmethylammonium cation) were carried out for systems of toluene–water, aqueous electrolyte solutions, or aqueous Cr(VI) at 298K. Using a shaker, $2.5 \times 10^{-5}\,m^3$ of toluene solution of 1–30% (v/v) trioctylmethylammonium chloride was shaken vigorously for about 2 hr with the same volume of aqueous solution of 50–200 ppm Cr(VI). A modifier, 2-ethyl hexanol, was added to all organic solutions in order to avoid the segregation of a third (second organic) phase (9). Its concentration was determined by the orange II-chloroform method with an ultraviolet spectrophotometer (Hewlett-Packard, Model 8425A, USA) (13). The concentration of Cl^{-} in the aqueous phase was measured by an ion chromatograph (Waters, Model 600E, USA). The concentration of Cr(VI) in the aqueous solution was measured by the colorimetric diphenyl carbazide method (14) with an ultraviolet spectrophotometer. All chemicals used were of reagent grade. Trioctylmethylammonium chloride (Aliquat 336) was received from Aldrich Chemical Company, Inc. (USA).

ANALYTICAL PROCEDURE

Hexavalent chromium, Cr(VI), exists as several chromate species such as $HCrO_4^-$, CrO_4^{2-}, $Cr_2O_7^{2-}$, and H_2CrO_4 according to the acidity and concentration of Cr(VI) in the solution (11). For concentrations of Cr(VI) around 50–200 mg/L and pH values around 3, $HCrO_4^-$ ion is predominant (11). Hexavalent chromium is taken up from aqueous solutions through anion exchange of $HCrO_4^-$, and the species $QHCrO_4$ is formed in the organic solution (12). For an organic–aqueous two-phase system containing Aliquat 336 (QCl) and $HCrO_4^-$, the overall equilibrium reaction and its equilibrium constant are defined as follows:

$$HCrO_4^- + \overline{QCl} \leftrightharpoons \overline{QHCrO_4} + Cl^- \qquad (1)$$

$$K = \frac{[Cl^-][\overline{QHCrO_4}]}{[HCrO_4^-][\overline{QCl}]} \qquad (2)$$

Figure 1 shows the distribution and dissociation equilibria of QCl in an organic–aqueous two-phase system. It is assumed that the distribution of QCl, dissociation of QCl, formation of $QHCrO_4$, and distribution of $QHCrO_4$ occur

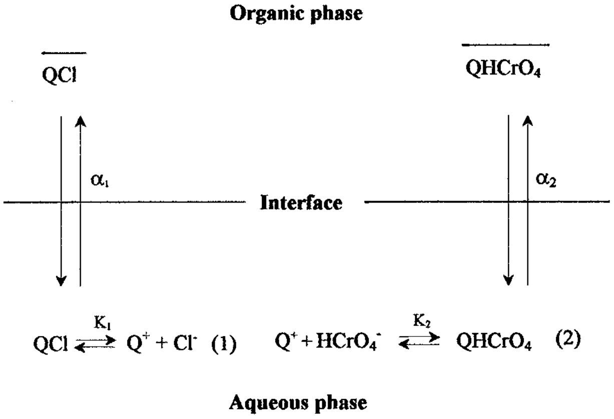

Figure 1. Equilibrium reaction model of Aliquat 336 with Cr(VI) aqueous solution.

along the reaction as in Eqs. (1) and (2). The overbar in Fig. 1 refers to the organic phase.

The dissociation constants in the aqueous phase and distribution constants are defined as follows:

$$K_1 = \frac{[Q^+][Cl^-]}{[QCl]} \tag{3}$$

$$K_2 = \frac{[QHCrO_4]}{[Q^+][HCrO_4^-]} \tag{4}$$

$$\alpha_1 = \frac{[QCl]}{[\overline{QCl}]} \tag{5}$$

$$\alpha_2 = \frac{[QHCrO_4]}{[\overline{QHCrO_4}]} \tag{6}$$

When the aqueous phase is water, $[Q^+] = [Cl^-]$ holds according to the requirement of electrical neutrality (15). And the mass balance of Q^+ results in

$$[QCl]_o = [\overline{QCl}] + [QCl] + [Q^+] \tag{7}$$

The observed concentration of Q^+ in aqueous phase results in

$$[Q^+]_{obs} = [QCl] + [Q^+] \tag{8}$$

It is assumed in Eq. (8) that the volumes of aqueous and organic phase are identical, in accordance with the present experimental conditions.

Then, from Eqs. (3), (5), and (8), one obtains

$$\frac{[\mathrm{Q}^+]_{\mathrm{obs}}}{\sqrt{[\mathrm{QCl}]_{\mathrm{o}} - [\mathrm{Q}^+]_{\mathrm{obs}}}} = \alpha_1 \sqrt{[\mathrm{QCl}]_{\mathrm{o}} - [\mathrm{Q}^+]_{\mathrm{obs}}} + \sqrt{K_1 \alpha_1} \tag{9}$$

When the aqueous phase is aqueous $HCrO_4^-$ solution, the mass balance of Cl^- and $HCrO_4^-$ results in

$$[\mathrm{QCl}]_{\mathrm{o}} = [\overline{\mathrm{QCl}}] + [\mathrm{QCl}] + [\mathrm{Cl}^-] \tag{10}$$

$$[\mathrm{HCrO}_4^-]_{\mathrm{o}} = [\mathrm{HCrO}_4^-] + [\mathrm{QHCrO}_4] + [\overline{\mathrm{QHCrO}_4}] \tag{11}$$

The observed concentrations of Cl^- and $HCrO_4^-$ in aqueous phase results in

$$[\mathrm{Cl}^-]_{\mathrm{obs}} = [\mathrm{QCl}] + [\mathrm{Cl}^-] \tag{12}$$

$$[\mathrm{HCrO}_4^-]_{\mathrm{obs}} = [\mathrm{HCrO}_4^-] + [\mathrm{QHCrO}_4] \tag{13}$$

Using Eqs. (2), (4), (6), and (10)–(13), one obtains

$$\frac{[\mathrm{Cl}^-]_{\mathrm{obs}} - \alpha_1([\mathrm{QCl}]_{\mathrm{o}} - [\mathrm{Cl}^-]_{\mathrm{obs}})}{[\mathrm{QCl}]_{\mathrm{o}} - [\mathrm{Cl}^-]_{\mathrm{obs}}} = K \frac{[\mathrm{HCrO}_4^-]_{\mathrm{obs}}}{[\mathrm{HCrO}_4^-]_{\mathrm{o}} - [\mathrm{HCrO}_4^-]_{\mathrm{obs}}} - K\alpha_2 \tag{14}$$

Using Eqs. (3)–(6), the overall equilibrium constant, K, is expressed as follows:

$$K = K_1 K_2 \alpha_1 / \alpha_2 \tag{15}$$

Using the observed equilibrium concentrations of the relevant species for the various initial concentrations, the distribution coefficients, α_1 and α_2, and the dissociation constants, K_1 and K_2, can be evaluated by the simplex method from Eqs. (9) and (14), respectively.

RESULTS AND DISCUSSION

The experimental results of the distribution and dissociation equilibria of Q^+Cl^- for the system of toluene–water are shown in Fig. 2 in accordance with Eq. (9). The best straight-line fit was determined by the least-squares method with the correlation coefficient of 0.96. From the intercept and slope of the line, the values of distribution and dissociation constant α_{1w} and K_1 were evaluated as

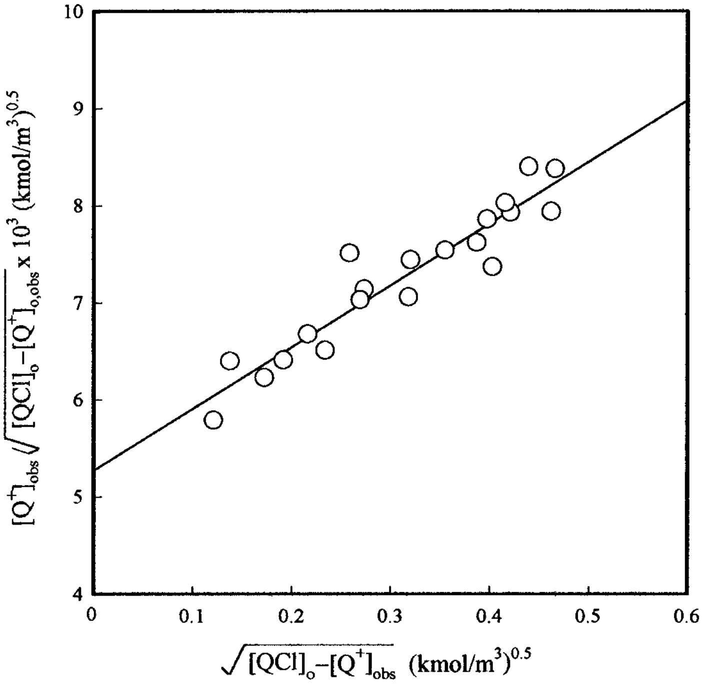

Figure 2. Plots of data according to Eq. (9) for system of QCl–toluene.

0.0065 and 0.0047 kmol/m^3, respectively. The dissociation constant, K_1, was independent of the ionic strength.

When the aqueous phase contains substantial amounts of common anion, Cl^-, the dissociation of QCl is suppressed, and $[Q^+] \cong 0$ holds. In this case, the following equation can be obtained from Eqs. (5), (7), and (8)

$$[Q^+]_{obs} = \alpha_1([QCl]_o - [Q^+]_{obs}) \tag{16}$$

Figure 3 shows the distribution equilibria of QCl for the system of toluene–aqueous NaCl solutions with ionic strength as the parameter. The dissociation of QCl in the aqueous NaCl solutions was suppressed by less than a few percent by adding NaCl. The experimental data for constant ionic strength are found to be linear in accordance with Eq. (16). The distribution coefficients, α_1, were evaluated for each ionic strength of 0.2, 0.5, 1.0, and 2.0 kmol/m^3 from the slopes of the respective straight lines, and their values were 0.00577, 0.00404, 0.00298, and 0.00128, respectively.

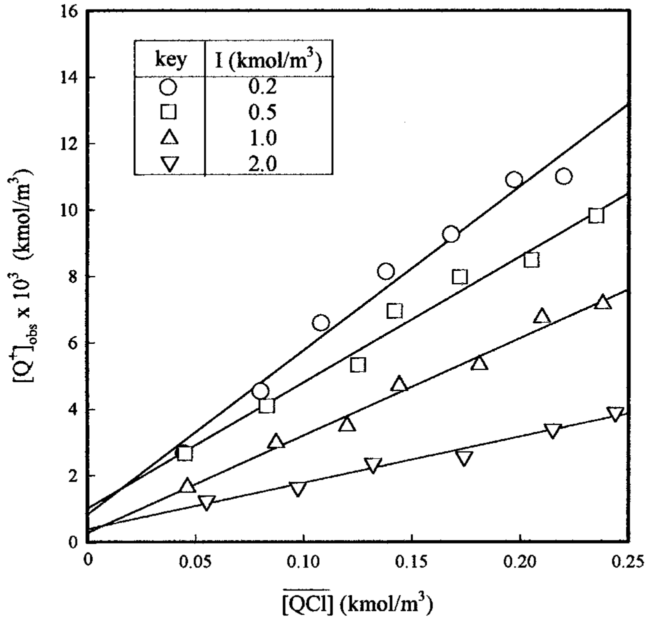

Figure 3. Plots of data according to Eq. (16) for system of QCl–toluene–aqueous NaCl solutions.

Figure 4 shows a plot of $\alpha_1/\alpha_{1\mathrm{w}}$ against the ionic strength. The value of $\alpha_{1\mathrm{w}}$ evaluated from Fig. 1 is also shown. The data obey a linear relationship in accordance with the formula of van Krevelen and Hoftijzer (16), which was originally proposed for the correlation of gas solubility in single-electrolyte solution and was applied successfully also to a liquid–liquid system (17). From the slope of the line, the resulting expression is

$$\log(\alpha_1/\alpha_{1\mathrm{w}}) = -0.296 I_{\mathrm{NaCl}} \qquad (17)$$

and 0.296 m³/kmol is the salting-out parameter, $K_{\mathrm{QCl,NaCl}}$, for the QCl–NaCl system. The salting-out parameter, $K_{\mathrm{QCl,HCrO_4^-}}$, for the QCl–$\mathrm{HCrO_4^-}$ system cannot be evaluated directly in this system, because QCl reacts with $\mathrm{HCrO_4^-}$ in the aqueous phase as shown in Fig. 1.

The relationship between the salting-out parameter and contribution to the salting-out parameter are expressed as follows (16):

$$K_{\mathrm{QCl,NaCl}} = i_{\mathrm{QCl}} + i_{\mathrm{Na^+}} + i_{\mathrm{Cl^-}} \qquad (18)$$

$$K_{\mathrm{MIBK,NaCl}} = i_{\mathrm{MIBK}} + i_{\mathrm{Na^+}} + i_{\mathrm{Cl^-}} \qquad (19)$$

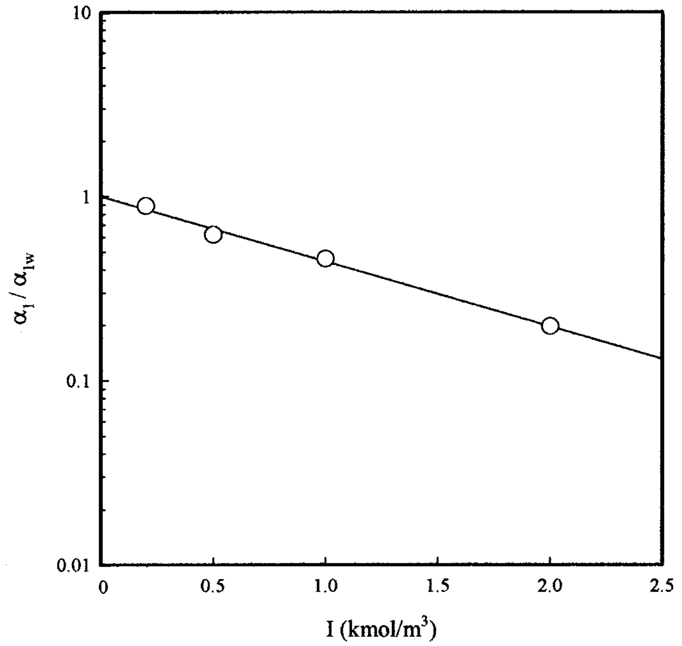

Figure 4. Correlation of distribution coefficient of QCl between toluene–aqueous NaCl solutions.

$$K_{\mathrm{MIBK,HCrO_4^-}} = i_{\mathrm{MIBK}} + i_{\mathrm{HCrO_4^-}} \tag{20}$$

$$K_{\mathrm{QCl,HCrO_4^-}} = i_{\mathrm{QCl}} + i_{\mathrm{HCrO_4^-}} \tag{21}$$

Using these equations, the salting-out parameter, $K_{\mathrm{QCl,HCrO_4^-}}$, for the QCl–$\mathrm{HCrO_4^-}$ system is expressed with the salting-out parameter for other systems as follows:

$$K_{\mathrm{QCl,HCrO_4^-}} = K_{\mathrm{QCl,NaCl}} + K_{\mathrm{MIBK,HCrO_4^-}} - K_{\mathrm{MIBK,NaCl}} \tag{22}$$

From the experimental equilibria for the system of MIBK (methylisobutylketone)–toluene–aqueous NaCl solutions, the contributions of MIBK in the aqueous solution were measured in range of NaCl concentrations, 0–2.0 kmol/m^3.

Figure 5 shows a plot of ratio of solubility of MIBK in aqueous NaCl solution to that in water against the ionic strength. The data obey a linear relationship in accordance with the formula of van Krevelen and Hoftijzer (16).

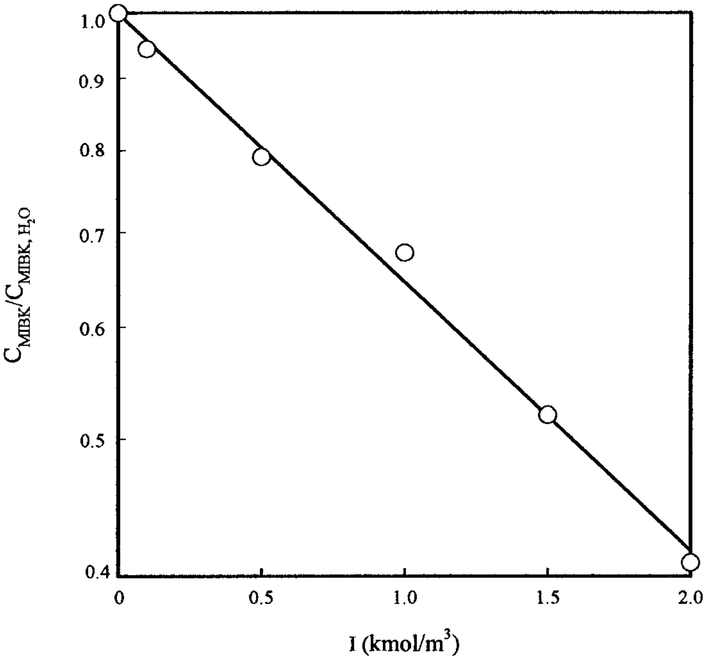

Figure 5. Solubility of MIBK in aqueous NaCl solutions.

The salting-out parameter, $K_{MIBK,NaCl}$, was obtained from the slope of this line and its value was 0.1891 m^3/kmol.

From experimental equilibria for the system of MIBK–toluene–aqueous $HCrO_4^-$ solutions, the measured concentration of MIBK in the aqueous solution was plotted against the ionic strength in Fig. 6, for which the data show a linear relationship.

The salting-out parameter, $K_{MIBK,HCrO_4^-}$, was obtained from the slope of the line and its value was 0.308 m^3/kmol. The salting-out parameter for the QCl–$HCrO_4^-$ system, $K_{QCl,HCrO_4^-}$, is evaluated using Eq. (22) and each salting-out parameter, and its value was 0.415 m^3/kmol.

The distribution coefficient QCl for the system of MIBK–toluene–aqueous NaCl–$HCrO_4^-$ solution is expressed as follows:

$$\log(\alpha_1/\alpha_{1w}) = -(0.296[\mathrm{NaCl}] + 0.415[\mathrm{HCrO_4^-}]) \tag{23}$$

where $\alpha_{1w} = 0.0065$.

In order to obtain the dissociation constant, K_2 and the distribution coefficient, α_2 in Fig. 1, the concentration of Cl^- and $HCrO_4^-$ in the aqueous solution for the system of QCl–toluene–aqueous $HCrO_4^-$ solutions were

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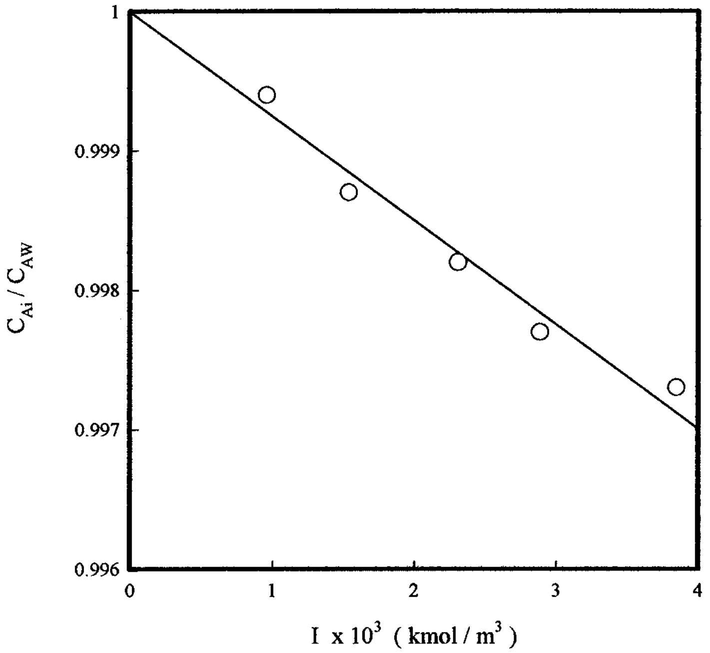

Figure 6. Solubility of MIBK in aqueous $HCrO_4^-$ solutions.

measured by varying QCl concentration in the range 0.02–0.65 kmol/m^3 for each concentration of $HCrO_4^-$ in the range of 50–200 ppm as parameter, and the experimental results are shown in Fig. 7 in accordance with Eq. (14). The best straight line fit was determined by the least-squares method. From the intercept and slopes of their values, K and α_2 were evaluated.

The dissociation constant of reaction (2) is shown in Fig. 1, and K_2 is obtained from Eq. (15). The evaluated values of α_1, α_2, K, and K_2 for each

Table 1. Evaluated Values of α_1, α_2, K, and K_2

$[HCrO_4^-]_o$ (ppm)	$\alpha_1 \times 10^3$	$\alpha_2 \times 10^5$	$K \times 10^{-3}$	$K_2 \times 10^{-3}$ (m^3/kmol)
50	6.4940	5.3409	1.1445	2.003
80	6.4905	5.4250	1.1491	2.044
120	6.4857	5.3758	1.1306	1.994
150	6.4821	5.1785	1.1892	2.021
200	6.4762	5.1076	1.1485	1.927

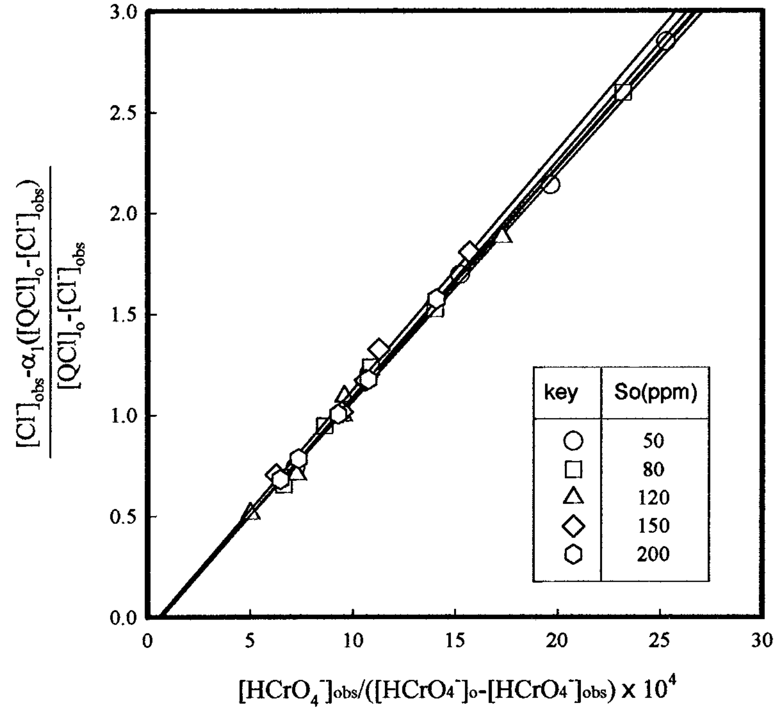

Figure 7. Plots of data according to Eq. (14) for system of QCl–toluene–aqueous $HCrO_4^-$ solutions.

concentration of $HCrO_4^-$ are listed in Table 1. The average value of K_2 was $1.998 \times 10^3\ m^3/kmol$, which was independent of the ionic strength substantially.

The values of α_2 evaluated from Fig. 7 are plotted against the feed concentration of $HCrO_4^-$ in Fig. 8. The best straight-line fit was determined by the least-squares method. From the slopes and intercepts of this line, the distribution of $QHCrO_4$ at water, α_{2w}, and the salting-out parameter for the system of QCl–toluene–aqueous $HCrO_4^-$ solution, $K_{QHCrO_4,HCrO_4^-}$, were evaluated, and their values are 5.53×10^{-5} and $8.385\ m^3/kmol$, respectively. The resulting expression is

$$\log(\alpha_2/\alpha_{2w}) = -8.385[HCrO_4^-]_o \qquad (24)$$

where $\alpha_{2w} = 5.53 \times 10^{-5}$.

The determined values of the salting-out parameters in Eqs. (23) and (24) were independent largely of the ionic strength. On other hand, the distribution coefficients, α_1 and α_2, were dependent on the ionic strength.

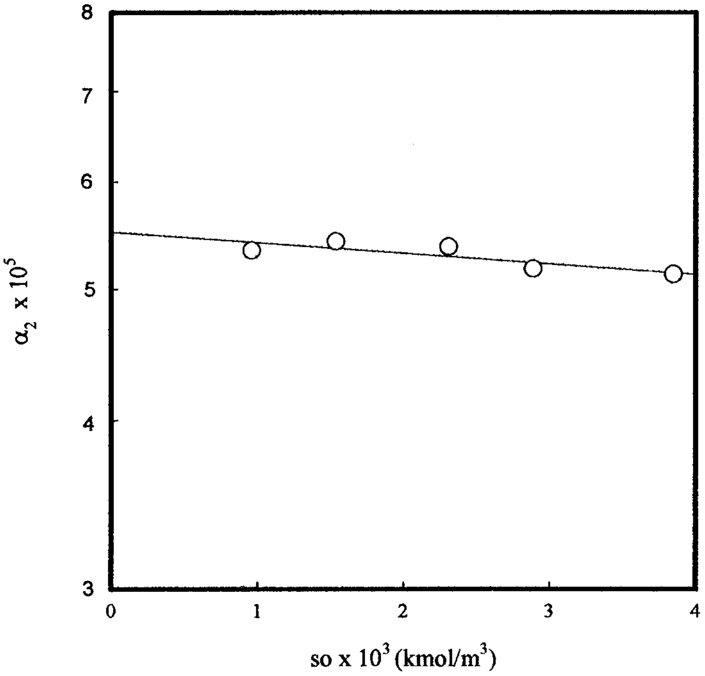

Figure 8. Correlation distribution coefficient of $QHCrO_4$ between toluene and aqueous $HCrO_4^-$ solutions.

CONCLUSION

The distribution and dissociation equilibria of phase-transfer catalyst, trioctylmethylammonium chloride (Q^+Cl^-), for the system of QCl^-–toluene–aqueous $HCrO_4^-$ solutions were measured at 298K for the systems of toluene–water and aqueous electrolyte solutions.

The evaluated distribution coefficients of trioctylmethylammonium chloride and the complex formed between trioctylmethylammonium cation and $HCrO_4^-$ were correlated as a function of the ionic strength in the aqueous solutions as follows:

$$\log(\alpha_1/\alpha_{1w}) = -(0.296[\mathrm{NaCl}] + 0.415[\mathrm{HCrO_4^-}])$$

$$\log(\alpha_2/\alpha_{2w}) = -8.385[\mathrm{HCrO_4^-}]_o$$

The dissociation constants, K_1 and K_2, were independent of the ionic strength.

NOMENCLATURE

I	ionic strength in aqueous solution (m^3/kmol)
i_j	contribution of species, j (m^3/kmol)
K	overall equilibrium constant defined as Eq. (2)
K_1	dissociation constant defined as Eq. (3) (kmol/m^3)
K_2	dissociation constant defined as Eq. (4) (m^3/kmol)
$K_{m,n}$	salting-out parameter for the system of species; m, toluene–aqueous species; n, solution (m^3/kmol)
Q^+	trioctylmethylammonium cation
QCl	trioctylmethylammonium chloride
so	feed concentration of $HCrO_4^-$ (kmol/m^3)

Greek symbols

α_1	distribution coefficient defined as Eq. (5)
α_2	distribution coefficient defined as Eq. (6)
α_{1w}	distribution coefficient of QCl for the toluene–water system
α_{2w}	distribution coefficient of $QHCrO_4$ for the toluene–water system

Superscript

—	organic phase

Subscripts

o	feed concentration
obs	observed value

ACKNOWLEDGMENTS

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