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DISTRIBUTION BEHAVIOR OF AMINOBENZOIC ACIDS BY EXTRACTION WITH DI(2-ETHYLHEXYL)PHOSPHORIC ACID AND TRIOCTYLAMINE

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DISTRIBUTION BEHAVIOR OF AMINOBENZOIC ACIDS BY EXTRACTION WITH DI(2-ETHYLHEXYL)PHOSPHORIC ACID AND TRIOCTYLAMINE

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ABSTRACT

Extraction based on reversible chemical complexation can be a high-capacity and high-selectivity technique for separating polar organic solutes from dilute solution. Aminobenzoic acid is a valuable chemical product. However, the downstream processing technology for the recovery of aminobenzoic acid presents a challenging separation problem. Aminobenzoic acid is resistant to biodegradation, so a method for treating wastewater that contains aminobenzoic acid needs to be developed. In this paper, we present a series of extraction equilibrium experiments for aminobenzoic acid with di(2-ethylhexyl)phosphoric acid (D2EHPA) dissolved in a mixture of *n*-octane and *n*-octanol and in trioctylamine dissolved in *n*-octanol. The effects of extractant concentration and pH on the distribution ratio are discussed in detail. Both (1:1) and (1:2) com-

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plexes are formed in the extraction of aminobenzoic acid with D2EHPA. Expressions for the equilibrium distribution ratio are proposed, and the experimental data are in good agreement to the proposed expressions.

INTRODUCTION

Extraction based on reversible chemical complexation can be a high capacity and high selectivity technique for separating polar organic solutes from dilute solution (1). In recent years, researchers have expressed a continuous interest in the extraction of carboxylic acids (2,3), phenols (4), and amines (5,6) based on reversible chemical complexation. Several years ago, the extraction of organic compounds with an acid group and an alkaline group on the molecule attracted much attention from researchers, and many interesting results were presented (7–9).

Aminobenzoic acid is a valuable chemical product. Its principal commercial applications are in the dye and pharmaceutical industries. Either in the process of production or application, wastewater that contains aminobenzoic acid is always generated. This kind of wastewater is resistant to biodegradation, and therefore a suitable technology to treat the wastewater and recover the valuable products must be developed.

We used *p*-aminobenzoic acid (PABC) and *o*-aminobenzoic acid (OABC) as model acids. Di(2-ethylhexyl)phosphoric acid (D2EHPA) and trioctylamine (TOA) were chosen as extractants and *n*-octanol was chosen as a diluent. The effects of extractant concentrations and pH value on equilibrium are discussed.

MATERIALS AND METHODS

Materials

D2EHPA was a product of the Chemical Reagent Factory of Beijing (P.R. China). It was further purified by recrystallization with copper hydroxide as described by Patridge and Jensen (10). TOA was of chemical grade. Other reagents, such as PABC, OABC, *n*-octanol, sulfuric acid (H_2SO_4), sodium sulfate (Na_2SO_4), and sodium hydroxide (NaOH), were all of analytical grade.

Extraction Experiments

All of the extraction experiments were carried out in a bath shaker at 298 ± 0.5 K and 200 ± 2 rpm. D2EHPA solutions, prepared by dissolving D2EHPA in a mixture of *n*-octane and *n*-octanol, contained approximately 0.373–0.894 mol/L D2EHPA. TOA solutions, prepared by dissolving TOA in *n*-octanol, con-



tained approximately 0.232–0.464 mol/L TOA. The aqueous ionic strength was fixed at 0.2 mol/L. The organic phase (10 mL) and the aqueous phase (10 mL) were stirred in the bath shaker for 1 hour. After equilibration, the phases were separated by centrifugation.

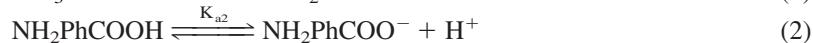
Analysis

The pH of the aqueous phase was measured with a digital pH meter (model SA 520, USA). The concentrations of PABC and OABC in the aqueous solution were measured through the use of an UV spectrophotometer (model Hp8452, USA) at a wavelength of 266 nm and 310 nm, respectively. The concentration of PABC and OABC in the organic phase was calculated according to the mass balance of each. Experiments proved that the error was less than 2%.

RESULTS AND DISCUSSION

Behavior of Aminobenzoic Acids in Aqueous Solution

Aminobenzoic acid has 1 carboxylic group, 1 amino group, and a benzene ring that connects with the carboxylic group and the amino group. Two dissociation equilibria exist in aqueous solutions



The dissociation constants for Eqs. (1 and 2) can be described as

$$K_{a1} = \frac{[\text{A}^+][\text{H}^+]}{[\text{A}^+]} \quad (3)$$

$$K_{a2} = \frac{[\text{A}^-][\text{H}^+]}{[\text{A}]} \quad (4)$$

where A^+ , A^- are the cation and anions of aminobenzoic acid. A is the neutral molecule of aminobenzoic acid. The pK_{a1} and pK_{a2} of the model acids in aqueous solutions are listed in Table 1.

Table 1. Dissociation Constants of Model Aminobenzoic Acids

Dissociation Constants	PABC	OABC
pK_{a1}	2.50	2.11
pK_{a2}	4.87	4.95

Data from (11).



Distribution Ratio Definition

The distribution ratio of aminobenzoic acids between the organic and the aqueous phases is defined as

$$D = \frac{[A]_{(o)}}{[A]_{(w)}} \quad (5)$$

where $[A]_{(o)}$ is the concentration of aminobenzoic acids in the organic phase, and $[A]_{(w)}$ is the total concentration of all species of aminobenzoic acids in the aqueous phase.

Physical Extraction Behavior of Aminobenzoic Acid with *n*-Octanol

In all the experiments, *n*-octanol was chosen as diluent, so its extraction characteristics play an important role in the extraction of aminobenzoic acid by D2EHPA or TOA.

As shown in Fig. 1, with an increase of pH value, the distribution ratio D of OABC and PABC between the organic and the aqueous phases is represented a peak, and this peak is at the point of $\text{pH} = (\text{p}K_{a1} + \text{p}K_{a2})/2$. The solid lines in Fig. 1 are as predicted by the following equation:

$$D = D_m \varphi = D_m (1 + 10^{\text{p}K_{a1}-\text{pH}} + 10^{\text{pH}-\text{p}K_{a2}})^{-1} \quad (6)$$

where $(1 + 10^{\text{p}K_{a1}-\text{pH}} + 10^{\text{pH}-\text{p}K_{a2}})^{-1}$ is the molar fraction of the neutral molecule at a specific pH, and D_m is the distribution ratio of aminobenzoic acid between the organic and the aqueous phases when the molar fraction of the neutral

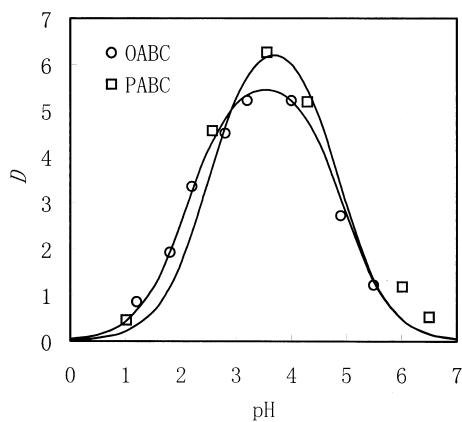


Figure 1. Physical extraction behavior of aminobenzoic acid with *n*-octanol.



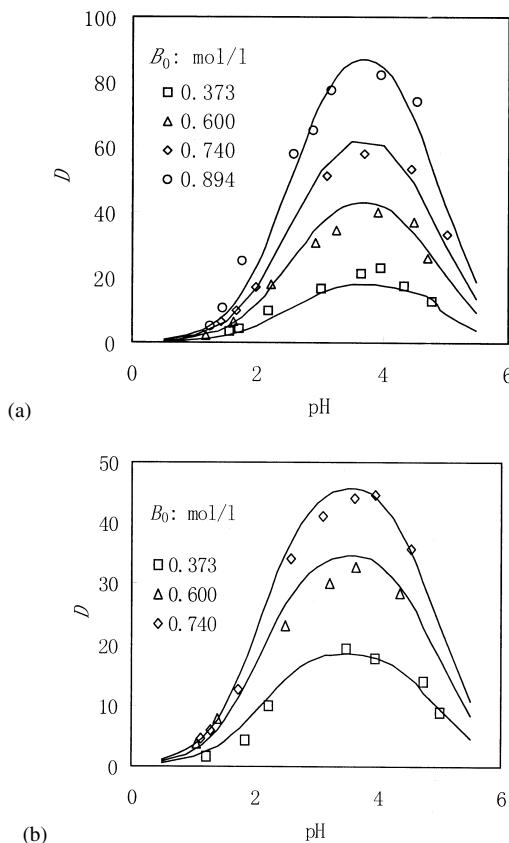


Figure 2. Effect of the initial concentration of D2EHPA (B_0) and pH value on the distribution ratio: (a) Initial concentration of PABC is 7.29 mmol/L, and (b) initial concentration of OABC is 7.29 mmol/L.

molecule in the aqueous phase is 1.0. The values of D_m are 7.12 and 5.86 for PABC and OABC, respectively.

One can conclude from Fig. 1 and Eq. (6) that the neutral molecules of aminobenzoic acid were extracted with *n*-octanol.

Effect of D2EHPA Concentration and Equilibrium pH on Distribution Ratio

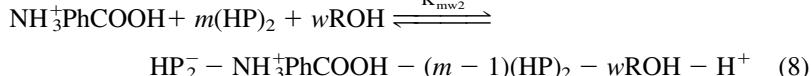
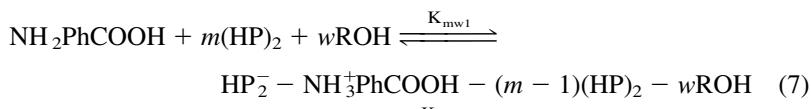
As shown in Fig. 2, at a constant extractant concentration, the distribution ratio of OABC and PABC between phases presents a peak with an increase of pH



value. An increase of the initial extractant concentration (B_0) increases the distribution ratio; this result is ascribed to enhancement of the extraction capacity of the organic phase.

Determination of the Extraction Model with D2EHPA

The reactions of D2EHPA with aminobenzoic acid can be expressed by Eqs. (7 and 8)



where Ph is C_6H_4 ; $(\text{HP})_2$ is dimeric D2EHPA; and ROH is *n*-octanol. K_{mw1} and K_{mw2} are equilibrium constants for Eqs. (7 and 8), respectively.

The extraction equilibrium constants (K_{mw1} , K_{mw2}) can be expressed as

$$K_{\text{mw1}} = \frac{[\overline{\text{PAR}}]}{[\text{A}](\overline{\text{HP}}_2)^m[\overline{\text{ROH}}]^w} \quad (9)$$

$$K_{\text{mw2}} = \frac{[\overline{\text{PAR}}][\text{H}^+]}{[\text{A}^+](\overline{\text{HP}}_2)^m[\overline{\text{ROH}}]^w} = K_{\text{mw1}}K_{\text{a1}} \quad (10)$$

where the organic-phase species are marked with an overbar. PAR is the complex; its structure is depicted in the right side of Eq. (7).

Equation (5) can be rewritten as

$$D = \frac{[\overline{\text{PAR}}] + [\overline{\text{A}}]}{[\text{A}^+] + [\text{A}] + [\text{A}^-]} = \frac{K_{\text{mw1}}[(\overline{\text{HP}}_2)^m[\overline{\text{ROH}}]^w + \phi D_m]}{1 + 10^{\text{p}K_{\text{a1}} - \text{pH}} + 10^{\text{pH} - \text{p}K_{\text{a2}}}} \quad (11)$$

where $[\overline{\text{A}}]$ is the neutral molecule concentration in the organic phase; this part of the neutral molecule is chemically extracted by *n*-octanol. ϕ is the volume fraction of *n*-octanol in the bulk solvent.

By rewriting Eq. (11) and taking the logarithm of both sides, we obtained the following equation:

$$\begin{aligned} \log[D(1 + 10^{\text{p}K_{\text{a1}} - \text{pH}} + 10^{\text{pH} - \text{p}K_{\text{a2}}}) - \Phi D_m] \\ = \log K_{\text{mw1}} + m \log[(\overline{\text{HP}}_2)^m] + w \log[\overline{\text{ROH}}] \end{aligned} \quad (12)$$

For convenient expression, the left side of Eq. (12) was abbreviated to $\log D'$. The plots of $\log D'$ vs. $\log[(\overline{\text{HP}}_2)^m]$ and $\log[\overline{\text{ROH}}]$ are shown in Figs. 3 and 4.



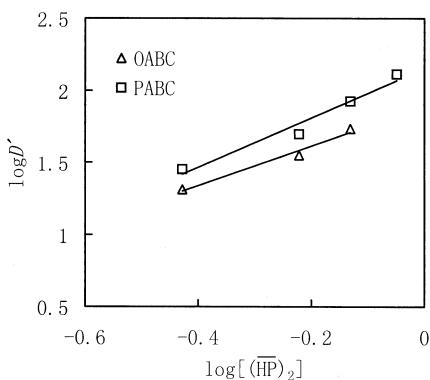


Figure 3. Plots of $\log D'$ vs. $\log[(HP)_2]$.

By fitting the least-square data, we obtain the slopes, i.e., the values of m and w , in Eq. (12). The values of m were 1.72 and 1.27 for PABC and OABC, respectively. The values of w were 0.06 and 0.01 for PABC and OABC, respectively.

Because the values of w for PABC and OABC were very close to zero, one can conclude that *n*-octanol makes little contribution to the formation of the complex in the organic phase, and it acts as a polar solvent to and physically extracts a part of neutral molecules of aminobenzoic acid.

The values of m for PABC and OABC were $1 < m < 2$, which suggests both (1:1) and (1:2) complexes formed with D2EHPA in the extraction.

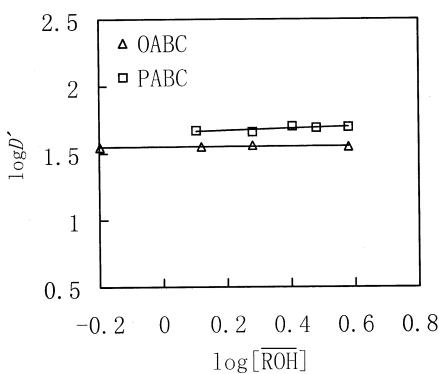
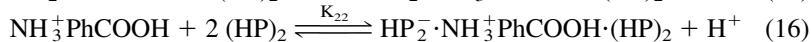
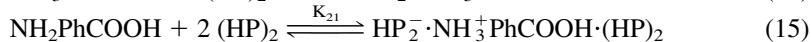
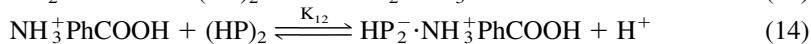
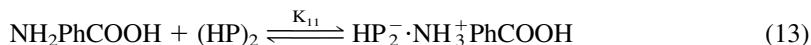


Figure 4. Plots of $\log D'$ vs. $\log[ROH]$.



According to the Eqs. (7 and 8), the stoichiometry of the extraction can be expressed as



where $\text{HP}_2^-\cdot\text{NH}_3^+\text{PhCOOH}$ is the (1:1) complex, expressed by PA_{11} in subsequent equations. $\text{HP}_2^-\cdot\text{NH}_3^+\text{PhCOOH}\cdot(\text{HP})_2$ is the (1:2) complex, expressed by PA_{12} in subsequent equations. K_{11} , K_{12} , K_{21} , and K_{22} are the respective extraction equilibrium constants, and they can be described as

$$K_{11} = \frac{\overline{[\text{PA}_{11}]}}{[\text{A}]\overline{[(\text{HP})_2]}} \quad (17)$$

$$K_{21} = \frac{\overline{[\text{PA}_{12}]}}{[\text{A}]\overline{[(\text{HP})_2]^2}} \quad (18)$$

$$K_{12} = K_{11}K_{a1} \quad (19)$$

$$K_{22} = K_{21}K_{a1} \quad (20)$$

Therefore, Eq. (11) can be rewritten as

$$D = \frac{\overline{[\text{PA}_{11}]} + \overline{[\text{PA}_{12}]} + \overline{[\text{A}]} }{[\text{A}^+] + [\text{A}] + [\text{A}^-]} = \frac{K_{11}\overline{[(\text{HP})_2]} + K_{21}\overline{[(\text{HP})_2]^2} + \phi D_m}{1 + 10^{pK_{a1}-pH} + 10^{pH-pK_{a2}}} \quad (21)$$

We used computer programming to optimize the following object function so that $E(K_{11}, K_{21})$ is at a minimum

$$E(K_{11}, K_{21}) = \sum_{i=1}^{i=n} (D_{\text{exp},i} - D_{\text{cal},i})^2 \quad (22)$$

where $D_{\text{exp},i}$ is the experimental value, and $D_{\text{cal},i}$ is the calculated value from Eq. (21). K_{11} and K_{21} was 22.93 L/mol and 103.55 L²/mol² for PABC, and it was 45.33 L/mol and 31.50 L²/mol² for OABC, respectively.

When the ratio of the volume of organic phase to that of the aqueous phase is unity, the distribution ratio (D) of aminobenzoic acid between the organic phase and the aqueous phase can be calculated through the use of the following equations:

$$C_0 = (1 + D)[\text{A}](1 + 10^{pK_{a1}-pH} + 10^{pH-pK_{a2}}) \quad (23)$$

$$B_0 = [\overline{[(\text{HP})_2]} + K_{11}\overline{[(\text{HP})_2]}[\text{A}] + K_{21}\overline{[(\text{HP})_2]^2}[\text{A}] \quad (24)$$

$$D = \frac{K_{11}\overline{[(\text{HP})_2]} + K_{21}\overline{[(\text{HP})_2]^2} + \phi D_m}{1 + 10^{pK_{a1}-pH} + 10^{pH-pK_{a2}}} \quad (25)$$



where C_0 is the initial concentration of aminobenzoic acid in the aqueous. B_0 is the initial concentration of D2EHPA in the organic phase. The values of K_{11} and K_{21} for PABC and OABC are different in these equations. The solid lines in Fig. 2 are as predicted by this model.

Effect of TOA Concentration and Equilibrium pH on Distribution Ratio

As illustrated in Fig. 5, at a constant extractant concentration (B_0), the distribution ratio presents a peak with the increased pH. An increase of the initial extractant concentration (B_0) corresponds to an increase in the distribution ratio.

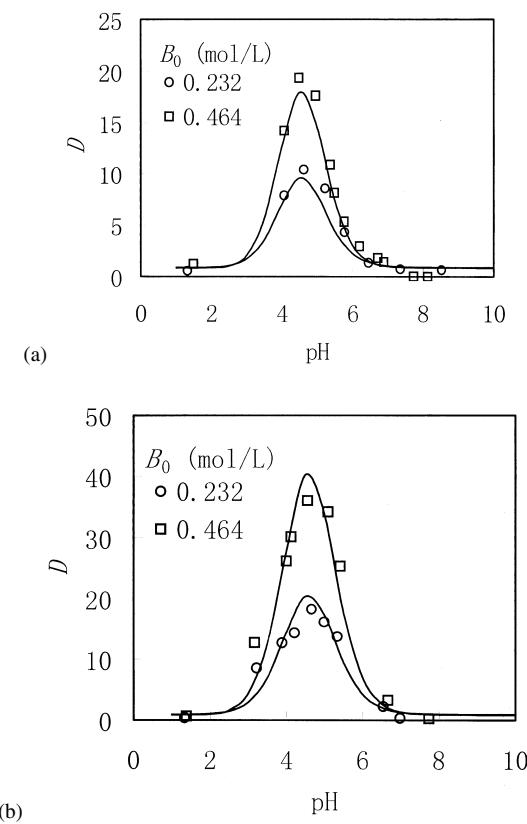


Figure 5. Effect of the initial concentration of TOA (B_0) and pH value on the distribution ratio: (a) Initial concentration of PABC is 7.29 mmol/L, and (b) initial concentration of OABC is 7.29 mmol/L.



These extraction phenomena are similar to those shown in Fig. 2, but the peak shifts toward the higher pH value in Fig. 5 because the basicity of TOA is higher than that of D2EHPA.

Determination of the Extraction Model with TOA as Extractant

The extraction equilibria with TOA can be described as



where R_3N is TOA. Aside from the 2 equilibria equations, 2 dissociation equilibria of aminobenzoic acid, Eqs. (1 and 2), and a physical extraction equilibrium can be determined.

The equilibrium constants in Eqs. (26 and 27) can be expressed as

$$K_1 = \frac{[R_3NH^+]}{[H^+] \cdot [R_3N]} \quad (28)$$

$$K_2 = \frac{[(R_3NH^+)COO^- phNH_2]}{[R_3N] \cdot [NH_2phCOOH]} \quad (29)$$

According to the mass balance of extractant and solute, the following equation, along with Eq. (23) can be obtained:

$$B_0 = [R_3N] + [R_3NH^+] + [(R_3NH^+)COO^- PhNH_2] \quad (30)$$

After a series of mathematical calculations for an equation group that consists of Eqs. (3,4,23,28,29, and 30), the expression for D can be obtained

$$D = \frac{\phi D_m \chi + K_2 B_0 - K_2 C_0 - \chi}{2\chi} + \frac{\sqrt{\alpha^2 + 4\beta}}{2\chi} \quad (31)$$

where

$$\alpha = K_2 C_2 - K_2 B_0 + \chi - \phi D_m \chi \quad (32)$$

$$\beta = (K_2 B_0 + K_2 \phi D_m C_0 + \phi D_m \chi) \chi \quad (33)$$

$$\chi = (1 + K_1 10^{-pH})(1 + 10^{pK_{a1}-pH} + 10^{pH-pK_{a2}}) \quad (34)$$

By using the computer programming to optimize the following object function, we kept $E(K_1, K_2)$ at a minimum

$$E(K_1, K_2) = \sum_{i=1}^{i=n} (D_{exp,i} - D_{cal,i})^2 \quad (35)$$

where $D_{exp,i}$ is the experimental value, and $D_{cal,i}$ is the value calculated from Eq. (31). K_1 and K_2 can be found to be 17 000 L/mol and 81.0 L/mol for PABC and 17 000 L/mol and 154.0 L/mol for OABC, respectively.



When the volume ratio of the organic phase to the aqueous phase is unity, the distribution ratio (D) of aminobenzoic acid between the organic phase and the aqueous phase can be calculated by using the following equations and Eq. (23):

$$D = \frac{\phi D_m \chi + K_2 B_0 - K_2 C_0 - \chi}{2\chi} + \frac{\sqrt{\alpha^2 + 4\beta}}{2\chi} \quad (36)$$

$$B_0 = [\overline{R_3N}] + K_1 \cdot 10^{-pH} \cdot [\overline{R_3N}] + K_2 [\overline{R_3N}] \cdot [A] \quad (37)$$

where the expressions of α , β , and χ are shown in Eqs. (32, 33, and 34). The solid lines in Fig. 5 are as predicted by the model.

CONCLUSIONS

In this paper, the extraction equilibrium of aminobenzoic acid with D2EHPA dissolved in the mixture of *n*-octane and *n*-octanol and TOA dissolved in *n*-octanol was presented in detail. D2EHPA and TOA were chosen as extractants, and similar extraction phenomenon was obtained for both compounds. At a constant extractant concentration (B_0), the distribution ratio presents a peak with an increased pH value. An increase of the initial extractant concentration (B_0) is associated with an increase in the distribution ratio. The peak of D shifts toward the higher pH value when TOA acts as the extractant because the basicity of TOA is higher than that of D2EHPA. Both (1:1) and (1:2) complexes formed in the extraction of aminobenzoic acid with D2EHPA. The extraction equilibrium constants were obtained. Expressions for the equilibrium distribution ratio with D2EHPA and TOA were proposed. The experimental data present good agreement to the obtained models, as can be seen from Figs. 2 and 5.

NOMENCLATURE

A^+	cation of aminobenzoic acid
A^-	anion of aminobenzoic acid
A	neutral molecule of aminobenzoic acid
$[A]_{(o)}$	concentration of aminobenzoic acid in the organic phase (L/mol)
$[A]_{(w)}$	concentration of aminobenzoic acid in the aqueous phase (L/mol)
B_0	initial extractant (D2EHPA or TOA) concentration in the organic
C_0	initial aqueous concentration of aminobenzoic acid (mol/L)
D	distribution ratio (dimensionless)
K_1	equilibrium constant in Eq. (26) (L/mol)
K_2	equilibrium constant in Eq. (27) (L/mol)
K_{11}	equilibrium constant in Eq. (13) (L/mol)
K_{12}	equilibrium constant in Eq. (14) (dimensionless)



K_{21}	equilibrium constant in Eq. (15) (L ² /mol ²)
K_{22}	equilibrium constant in Eq. (16) (L/mol)
K_{a1}, K_{a2}	dissociation constant of aminobenzoic acid (L/mol)
(HP) ₂	D2EHPA dimer
PA ₁₁	(1:1) complex of D2EHPA combined with aminobenzoic acid
PA ₁₂	(1:2) complex of D2EHPA combined with aminobenzoic acid
ϕ	volume fraction of <i>n</i> -octanol in bulk solvent
φ	molar fraction of neutral molecule in Eq. (6)
α, β, χ	symbols equivalent to the right-hand terms of Eqs. (32–34)

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BEHAVIOR OF EXTRACTED AMINOBENZOIC ACIDS

3485

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