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EFFECTS ON THE MICELLAR SOLUBILIZATION OF ORGANIC COMPOUNDS
BY SURFACTANT MICELLES.   II. NATURE AND POSITION   OF
SUBSTITUENT GROUPS IN AROMATIC ACIDS.

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ABSTRACT

The semiequilibrium dialysis method has been used
to investigate the equilibrium solubilization of o-, m-
and p-toluic acids by 0.1N HCl aqueous solutions of the
cationic surfactant 1-hexadecylpyridinium   chloride
(cetylpyridinium chloride), throughout a range of
concentrations of the toluic acids and surfactant.
Values of the apparant solubilization constant, K, of
the neutral acids have been correlated with mole
fractions of the acid in the micelle X_A , where $K = X_A$
/[monomeric acid]. The activity   coefficients of both
acid and surfactant were obtained, consistent with the
Gibbs-Duhem equation.

Several conclusions can be drawn from the results:
(a) For each of the three toluic acid - cetylpyridinium
chloride systems, K is found to vary nearly linearly
with X_A , throughout the investigated range of acid
concentration. (b) The presence of both the methyl and
carboxyl groups as substituents in the benzene ring

enhances the solubilization much more than the additive effect of both groups, when present separately. (c) The position of the substituent groups with respect to each other, will affect the extent of solubilization of the investigated acids. (d) The difference in the values of K for the three acids show a contradiction to a group contribution model developed recently.

INTRODUCTION

The solubilization of organic compounds in surfactant micelles
has been studied utilizing various techniques such as vapor pressure
(1), gas chromatography (2), nmr-spin-echo technique (3), micellar
enhanced ultra filtration (MEUF) and the semiequilibrium dialysis'
method (SED) which has been recently developed and used extensively
(4-9). Some important factors are known to influence the extent of
solubilization as, for example, bulk solution effects (10,11),
possible Laplace pressure effects (12,13), and the effect caused by
the interaction between the electrical potential at the ionic micellar
surface and polar or polarizable nonelectrolyte molecules solubilized
within the micelle (14). In many applications of micellar- based
separation methods, the concentration of organic solutes in micelles
may be expected to be relatively small. Thus, it will be very helpful
to be able to predict the partition coefficient of organic molecules
in aqueous surfactant micelles (the solubilization constant).

As a contribution to the accumulating information about the
solubilization of a variety of organic compounds in different
surfactants, we investigated in Part I of this series (15) the effect
of inserting a $(CH2)_n.COO$ group between the hydrogen and the phenyl
group of benzene, on the extent of solubilization of benzene in the
micelles of hexadecylpyridinium chloride (CPC). The study enabled also
the comparison of the solubilization of the three acids. To add still
more to the accumulating information of this subject, we studied, in
the present work, the solubilization of o- , m- , and p- toluic acids
in CPC. Several conclusions could be drawn from the results of such a
study when compared with previous results obtained for toluene and for

benzoic acid, such as the effect of the nature and position of the substituent groups present on the extent of solubilization. Also, the results of the present study could be used to test the applicability of a new approach made recently (16) attempting to predict the value of the solubilization constant, based on a group contribution model depending only on the nature of the organic compound and on the groups present as substituents or branches.

EXPERIMENTAL

The SED technique has been described in detail previously (4). A 0.1M HCl solution containing known concentrations of the surfactant and solute was placed on one side of the membrane (retentate), and the other side (permeate) contained 0.1M HCL solution. The initial surfactant concentration is chosen to be much higher than the critical micelle concentration (cmc), to ensure that most of the surfactant micelles are present in micellar form. The HCl solution is used instead of an aqueous solution to reduce the concentration of free hexadecylpyridinium ion in the retentate solution, and also to supress the ionization of the organic acids used, hence, minimizing any complications in the final computations. Equilibrium is usually reached within 18-24 hours, at which time the activity of free organic solute is the same in the retentate and permeate.

Molalities were used to assure maximum accuracy in final calculations. The initial molality of the organic solute in the retentate varied from 0.0103 - 0.129 for o-toluic acid (OTA), 0.0102 - 0.1022 for m-toluic acid (MTA) and 0.0109 - 0.1135 for p-toluic acid (PTA). The initial CPC concentration was 0.05 to 0.2 molal. The cmc of CPC is 0.00080M (17).

All organic acids used (Aldrich 99.5%) and hexadecylpyridinium chloride monohydrate (Hexcel), were used as received.

The permeate solution was analyzed using UV spectroscopy. Extinction coefficients (absorptivities) were determined at 10 nm intervals across the major CPC, OTA, MTA, and PTA absorbance peaks, assuming that Beer's law applies to the very dilute solutions of the individual components. The concentrations of the organic solute and CPC in the permeate were calculated simultaneously, using a nonlinear least squares computer program (4). Retentate concentrations were obtained by correcting the original concentrations for the small amount of solute transferred into the permeate side.

DATA ANALYSIS AND RESULTS

The detailed methods of analysis of the data obtained were given previously (5, 7-9). The ionization of the acids has been neglected, since it was minimized by using 0.1M HCl, as mentioned before. The relationships leading to the calculation of the solubilization constants of the organic acids used in CPC, and the calculation of the activity coefficients of the organic acids and surfactant can be summarized in the following equations.

$$[A]_{tot} = \gamma_A X_A c_A^o + X_A/(1-X_A) [CPC]_{mic} \qquad (1)$$

$$[CPC]_{tot} = \gamma_{CPC}(1-X_A) c_{CPC}^o + [CPC]_{mic} \qquad (2)$$

$$K = X_A / c_A \qquad (3)$$

$$K = K_o (1-b X_A) \qquad (4)$$

$$\gamma_A = 1 / (K c_A^o) \qquad (5)$$

$$\gamma_A = a / (1 - b X_A) \qquad (6)$$

$$\ln \gamma_{CPC} = \left[1/(1-b) \right] \left\{ b \ln(1-X_A) - \ln(1-b X_A) \right\} \qquad (7)$$

$$K = (1-X_A) \left\{ [A]_{tot}^{ret} - [A]_{tot}^{per} \right\} / \left\{ [A]_{tot}^{per} [CPC]_{mic}^{ret} - [A]_{tot}^{ret} [CPC]_{mic}^{per} \right\} \qquad (8)$$

$[A]_{tot}$ and $[CPC]_{tot}$ denote the total concentration of the organic acid and surfactant, respectively. $[CPC]_{mic}$ is the molar concentration of the surfactant in the micelle, X_A is the intramicellar mole fraction of organic acid, and γ_A' and γ_{CPC} are activity coefficients based on the pure component standard state for the organic acid and the pure micelle standard state for the surfactant. c_A^o is selected to approximately equal to the concentration of monomeric organic acid at which the partial pressure or fugacity of acid is equal to that of pure acid at the same temperature. c_{CPC}^o is the concentration of monomeric surfactant in the bulk phase in the absence of added organic solute.

Table I lists all of the primary results for the o-, m- and p-toluic acid solutions in CPC. The first 2 columns list the experimental values for both acid and surfactant concentrations in the retentate solution. Columns 3 and 4 list the values of the acid and surfactant concentrations in the permeate solution. The last column lists the value of the concentration of the acid in the permeate solution, as predicted from the model.

Table II lists the values of the least-squares parameters (Ko and b) derived by fitting all of the $[A]_{tot}^{per}$ data for a given toluic acid with the above mentioned model. The table also includes values for c_A^o and c_{CPC}^o used in the analysis. The relative root-mean-square deviations tabulated are those in $[A]_{tot}^{per}$ for each system.

Figures 1-3 show the dependence of Y_A and Y_{CPC} on X_A for the three CPC-toluic acid systems. The 3 figures are quite similar in shape, Y_A increases with increasing the acid mole fraction into the micelle, whereas Y_{CPC} decreases in the same direction.

Figures 4-6 are plots of K against X_A, the straight lines corresponding to the values of K and b for each system. The points indicate values of K calculated point by point by a method similar to that described previously (5), making the assumption that the value of X_A in the permeate solution is equal to that in the retentate.

DISCUSSION AND CONCLUSIONS

The most important factors influencing the extent and region of solubilization of organic solutes into surfactant micelles, are refered to in the "Introduction", and can be summarized as:

1- Bulk solution effects

   2- Possible Laplace pressure effects

3- The effect caused by the interaction between the electrical potential at the ionic micellar surface and polar or polarizable nonelectrolyte molecules solubilized within the micelle.

The expected effects of these factors have been discussed previously (7, 15, 18), explaining the assumed structure of the ionic micelles, and the subsequent effect of this structure on the solubilization of different organic compounds. Accordingly, benzene, the parent compound of the substances investigated in the present study, is distributed, when present as the organic solute, almost uniformly throughout the surface and core regions of the CPC micelle (19).

In Part I of this series (15), it was found that K for benzoic acid is considerably larger than that for benzene (72 M^{-1} as compared with $40M^{-1}$), which was attributed to the fact that the -COOH group is intensily charged and capable of hydrogen bonding with water, together with considering the 3 factors mentioned above. Also, when a methyl group, -CH3, is introduced into benzene, in toluene, the aliphatic character in the molecule is increased, in addition to the already present aromatic character of the benzene molecule, resulting in an apparent alteration of the 3 factors, such that K for toluene is much higher than that for benzene (8), (125 M^{-1} as compared with 40 M^{-1}).

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In the present study, the compound used contains a carboxylic acid group and a methyl group, both introduced in the same benzene ring (toluic acid), where its o-, m- and p- substituents were investigated.

It is clear from Table II that the values of the apparent solubilization constant K_o for the 3 acids are all higher than that for either benzene or toluene. This indicates that the presence of both groups, as substituents into the benzene ring at the same time, not only does not contradict the effect of either of them on the factors influencing the solubilization, but actually enhances the solubilization to a larger extent than the algebric addition of the separate effect of both the -COOH and -CH3 groups with respect to the benzene molecule.

In comparing the values of K for the three acids, as can be seen from Table II, it can be clearly seen that the values of K vary in the order m->p->o- substituents. The lowest value for the o- substituent can be explained on the basis that the presence of the -COOH and -CH3 groups, ortho to each other, will impose some steric hinderence of one on the other, thus decreasing the expected effect for each group, when present separately, on the extent of solubilization. The fact that the value of K for the p-substituent is less than that for the m-substituent, although there is no steric hinderence in both, can be explained on the basis that when the two substituent groups are present 'para' to each other, there tends to be a competing effect between surface solubilization due to the -COOH group, and core solubilization due to the -CH3 group, resulting in the noticed lower value for K than that for the m-substituent, where there is neither the steric effect nor the competing effect of the substituent groups.

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The apparent difference in the value of K for the three acids is in clear contradiction to a group contribution model developed recently (16). According to this model, all 3 values of K should be, theoretically, the same since the model is based on the assumption that the value of K is dependent only on a certain contribution from the groups present, according to their nature and irrelevant of their position. A similar contradiction to the model was also shown in Part 1 of this series, where, according to the model, values of K were expected to differ because the substituents are different, but the results showed that the K values are almost the same, indicating that the extent of solubilizaton will definitely depend on several factors based on both nature and position of the substituents.

It can be generally seen, from Figs. 1-6, that, for all three toluic acids, the solubilization constants decrease, and the activity coefficients increase, as the mole fraction of the acid solubilized in the micelle increases. This is in accordance with previous results for highly polar and aliphatic solutes, solubilized by ionic surfactant micelles (5-9, 15).

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Table I. Experimental and Calculated Values of Toluic Acid Concentrations in the Permeate for Known Retentate Solutions Containing CPC and Acid.[a]

o-Toluic Acid

Retentate Solution		Permeate Solution		
[A]	[CPC]	[A]	[CPC]	$[A]_{pred.}$[b]
1.2876E-01	2.2185E-01	3.3736E-03	8.8270E-05	3.5368E-03
1.2887E-01	2.2185E-01	3.2479E-03	7.9830E-05	3.5407E-03
6.4434E-02	1.1349E-01	3.1397E-03	0.0000E+00	3.3475E-03
6.4590E-02	1.1349E-01	2.9836E-03	0.0000E+00	3.3568E-03
5.9827E-02	1.1097E-01	2.9743E-03	5.8945E-05	3.1522E-03
5.9854E-02	1.1098E-01	2.9483E-03	5.1155E-05	3.1535E-03
2.7838E-02	5.3831E-02	2.6304E-03	3.4645E-05	2.8308E-03
2.7835E-02	5.3836E-02	2.6334E-03	2.9494E-05	2.8392E-03
1.1015E-01	2.1708E-01	2.8257E-03	0.0000E+00	3.9006E-03
1.1021E-01	2.1905E-01	2.7683E-03	3.3370E-05	3.0029E-03
5.3449E-02	1.0864E-01	2.5774E-03	0.0000E+00	2.8396E-03
5.3445E-02	1.0863E-01	2.5012E-03	1.5236E-05	2.8398E-03
5.0312E-02	1.0594E-01	2.4549E-03	4.3225E-05	2.7293E-03
5.0320E-02	1.0589E-01	2.4475E-03	0.0000E+00	2.7275E-03
1.9210E-02	4.2857E-02	2.1492E-03	1.4885E-06	2.3509E-03
1.9226E-02	4.2860E-02	2.1329E-03	0.0000E+00	2.3530E-03
8.6024E-02	2.2079E-01	1.9654E-03	5.4336E-05	2.2587E-03
8.6033E-02	2.2080E-01	1.9563E-03	4.5223E-05	2.2590E-03
4.2273E-02	1.1100E-01	1.9396E-03	2.2083E-05	2.1435E-03
4.2308E-02	1.1099E-01	1.9235E-03	3.1197E-05	2.1446E-03
6.1099E-02	2.2298E-01	1.3986E-03	7.4127E-05	1.5445E-03
6.1109E-02	2.2288E-01	1.3895E-03	6.9170E-05	1.5447E-03
2.8082E-02	1.0476E-01	1.2916E-03	2.7510E-05	1.4636E-03
4.3658E-02	2.1674E-01	1.0603E-03	1.1233E-04	1.1139E-03
2.0106E-02	1.0209E-01	9.5785E-04	6.9573E-05	1.0563E-03
2.0106E-02	1.0210E-01	9.5734E-04	6.0634E-05	1.0563E-03
2.0510E-02	2.1033E-01	4.5237E-04	9.6693E-05	5.2558E-04
2.0492E-02	2.1032E-01	4.7047E-04	1.0724E-04	5.2514E-04
1.0444E-02	1.0918E-01	4.3899E-04	6.4735E-05	5.0314E-04
1.0391E-02	1.0919E-01	4.4391E-04	6.1587E-05	5.0051E-04

[a] All concentrations in $mol-l^{-1}$

[b] Calculated results obtained by fitting the activity coefficient with equations 5 & 7, using constants in Table II.

Table I. (continued)

m-Toluic Acid

Retentate Solution		Permeate Solution		
[A]	[CPC]	[A]	[CPC]	$[A]^b_{prod.}$
6.0572E-02	1.0600E-01	2.1714E-03	5.2337E-05	2.3042E-03
2.9406E-02	5.3030E-02	1.9730E-03	5.0245E-05	2.1324E-03
2.9453E-02	5.3031E-02	1.9252E-03	2.9902E-05	2.1363E-03
1.0217E-01	2.3602E-01	1.4580E-03	1.1923E-04	1.7225E-03
1.0214E-01	2.3600E-01	1.4858E-03	1.3877E-04	1.7221E-03
4.4931E-02	1.0338E-01	1.3711E-03	8.1008E-05	1.6522E-03
4.4918E-02	1.0339E-01	1.3840E-03	7.3743E-05	1.6515E-03
8.8854E-02	2.2120E-01	1.3055E-03	1.0734E-04	1.5836E-03
8.8921E-02	2.2116E-01	1.3397E-03	1.4185E-04	1.5832E-03
4.3217E-02	1.0915E-01	1.2773E-03	6.5946E-05	1.5280E-03
4.3219E-02	1.0914E-01	1.2759E-03	7.0792E-05	1.5281E-03
6.3776E-02	2.2023E-01	9.8033E-04	1.2429E-04	1.1133E-03
6.3983E-02	2.2024E-01	9.7732E-04	1.3620E-04	1.1135E-03
3.0951E-02	1.0841E-01	1.0329E-03	8.5278E-05	1.0722E-03
3.0985E-02	1.0842E-01	9.9073E-04	7.6905E-05	1.0734E-03
4.4084E-02	2.1958E-01	6.8983E-04	1.2254E-04	7.5253E-04
4.4092E-02	2.1958E-01	6.8125E-04	1.2232E-04	7.5269E-04
2.3382E-02	1.1792E-01	6.6701E-04	8.5341E-05	7.3151E-04
2.3397E-02	1.1793E-01	6.5237E-04	7.7548E-05	7.3194E-04
2.1171E-02	2.1639E-01	2.9095E-04	1.1593E-04	3.5797E-04
2.1189E-02	2.1639E-01	2.9362E-04	1.1797E-04	3.5773E-04
1.0201E-02	1.0555E-01	2.7970E-04	7.5614E-05	3.4687E-04
1.0201E-02	1.0554E-01	2.7931E-04	6.9182E-05	3.4686E-04

[a] All concentrations in mol-l^{-1}

[b] Calculated results obtained by fitting the activity coefficient with equations 5 & 7, using constants in Table II.

Table 1. (continued)

p-Toluic Acid

Retentate Solution		Permeate Solution		
[A]	[CPC]	[A]	[CPC]	$[A]^b_{pred.}$
5.9056E-02	1.0439E-01	2.3420E-03	9.7058E-04	2.0985E-03
5.9066E-02	1.0490E-01	2.3816E-03	9.8480E-04	2.0988E-03
2.8073E-02	5.1118E-02	2.0769E-03	9.3285E-04	1.9863E-03
2.8016E-02	5.1095E-02	2.1340E-03	8.5599E-04	1.9834E-03
1.1348E-01	2.2861E-01	2.1135E-03	9.4812E-04	1.9088E-03
1.1347E-01	2.2862E-01	2.1162E-03	9.3232E-04	1.9087E-03
5.1662E-02	1.0565E-01	2.0360E-03	9.8157E-04	1.8507E-03
5.1741E-02	1.0583E-01	1.9575E-03	8.1530E-04	1.8507E-03
8.7189E-02	2.2224E-01	1.5000E-03	6.7697E-04	1.5421E-03
8.7191E-02	2.2225E-01	1.4982E-03	6.6713E-04	1.5421E-03
4.3473E-02	1.1283E-01	1.4754E-03	6.1475E-04	1.4933E-03
4.3416E-02	1.1237E-01	1.5064E-03	6.2413E-04	1.4969E-03
6.2199E-02	2.1447E-01	1.1769E-03	5.9241E-04	1.1652E-03
6.2206E-02	2.1451E-01	1.1692E-03	5.5409E-04	1.1651E-03
3.0466E-02	1.0661E-01	1.1227E-03	5.0697E-04	1.1291E-03
3.0463E-02	1.0662E-01	1.1252E-03	4.9463E-04	1.1289E-03
4.2103E-02	2.1457E-01	7.4325E-04	4.1847E-04	8.0511E-04
4.2094E-02	2.1457E-01	7.5193E-04	4.1024E-04	8.0495E-04
2.1863E-02	1.1292E-01	7.1352E-04	3.5840E-04	7.8198E-04
2.1863E-02	1.1294E-01	7.1295E-04	3.3595E-04	7.8185E-04
2.1994E-02	2.1620E-01	3.4920E-04	2.4446E-04	4.2658E-04
2.1984E-02	2.1619E-01	3.5957E-04	2.6244E-04	4.2640E-04
1.0947E-02	1.0915E-01	3.4302E-04	2.2256E-04	4.1300E-04
1.0945E-02	1.0916E-01	3.4533E-04	2.1921E-04	4.1291E-04

[a] All concentrations in $mol-l^{-1}$

[b] Calculated results obtained by fitting the activity coefficient with
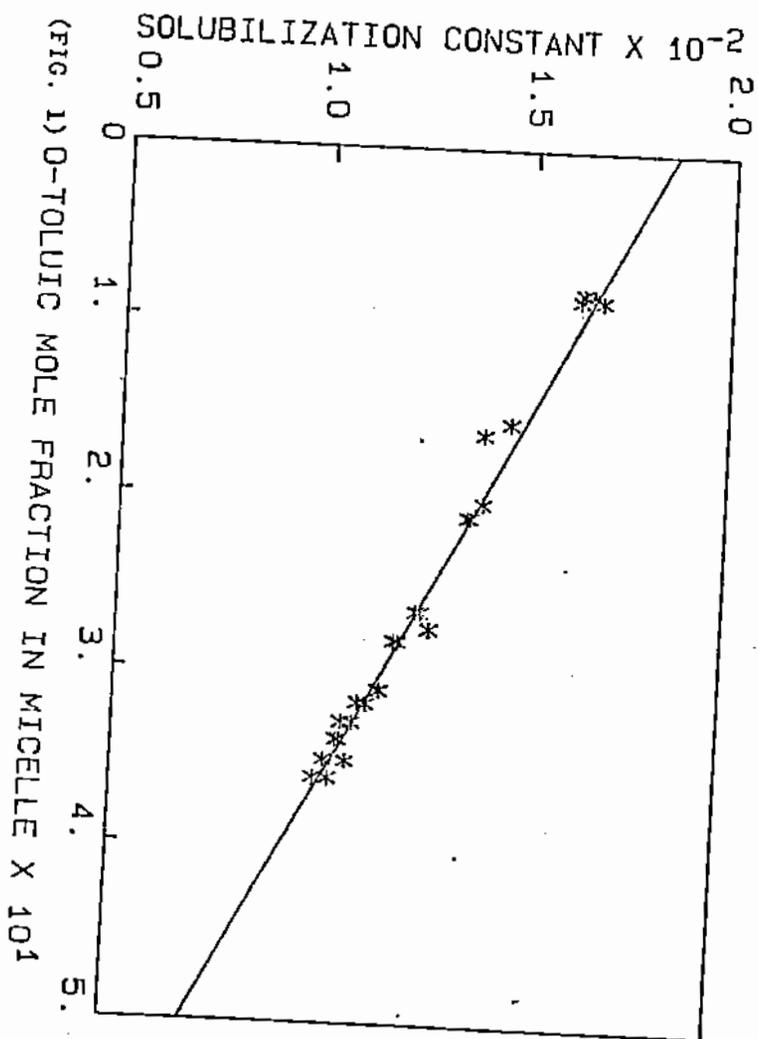equations 5 & 7, using constants in Table II.

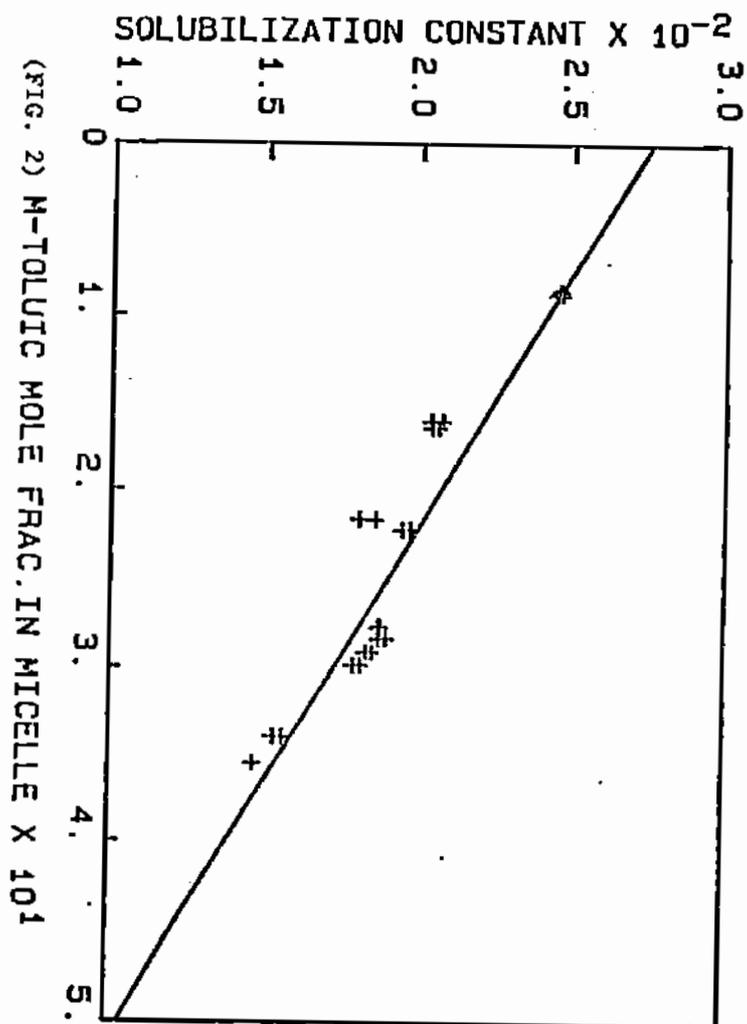Table II: Least Squares Parameters for Toluic Acids in 1-Hexadecylpyridinium Chloride at 25°C.

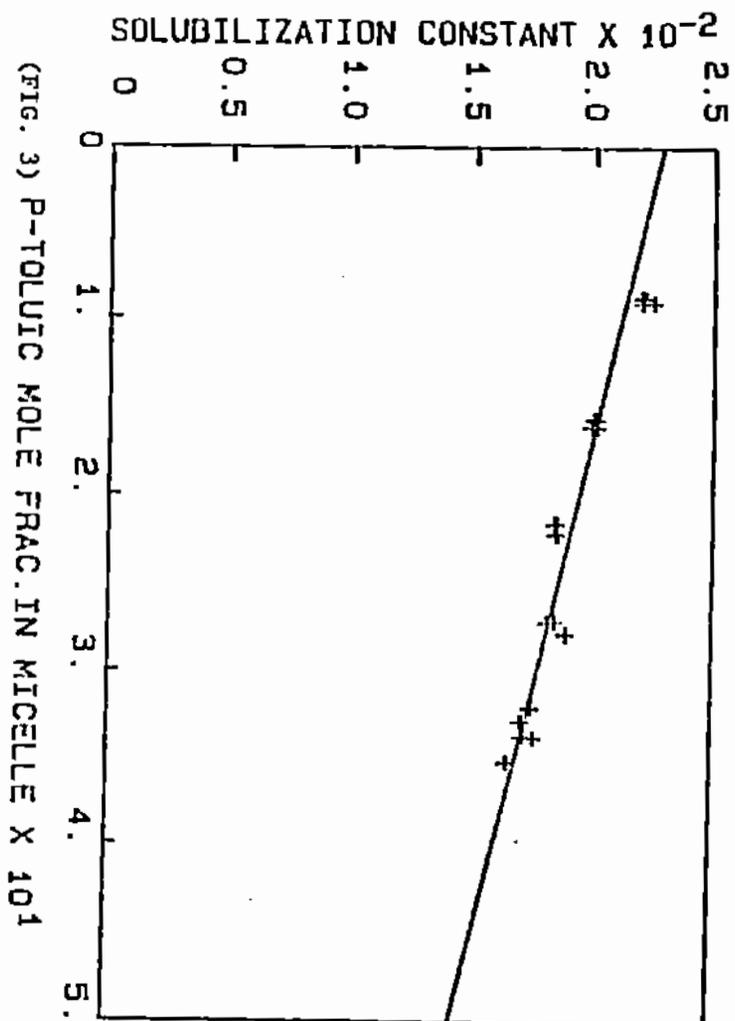| Solute	$K_0 (M^{-1})^a$	b^b	c_A^o	c_{CPC}^o	10^{-5} RMSD^c
o- Toluic Acid	185	1.244	0.0475	0.88 mm	4.768 mol-1-1
m- Toluic Acid	275	1.241	0.0261	0.88 mm	6.542 mol-1-1
p- Toluic Acid	229	0.754	0.0573	0.88 mm	3.298 mol-1-1

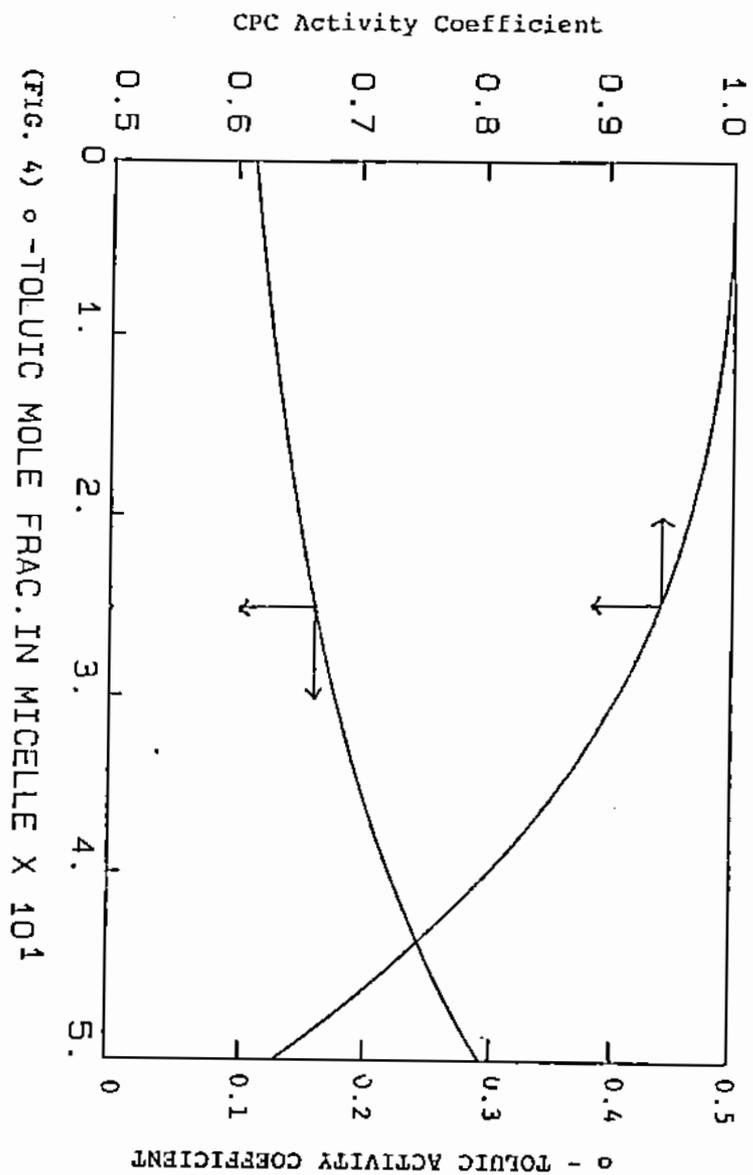^{a} Intercept of a plot of the solubilization constant, K, vs. the mole fraction of acid in the micelle.
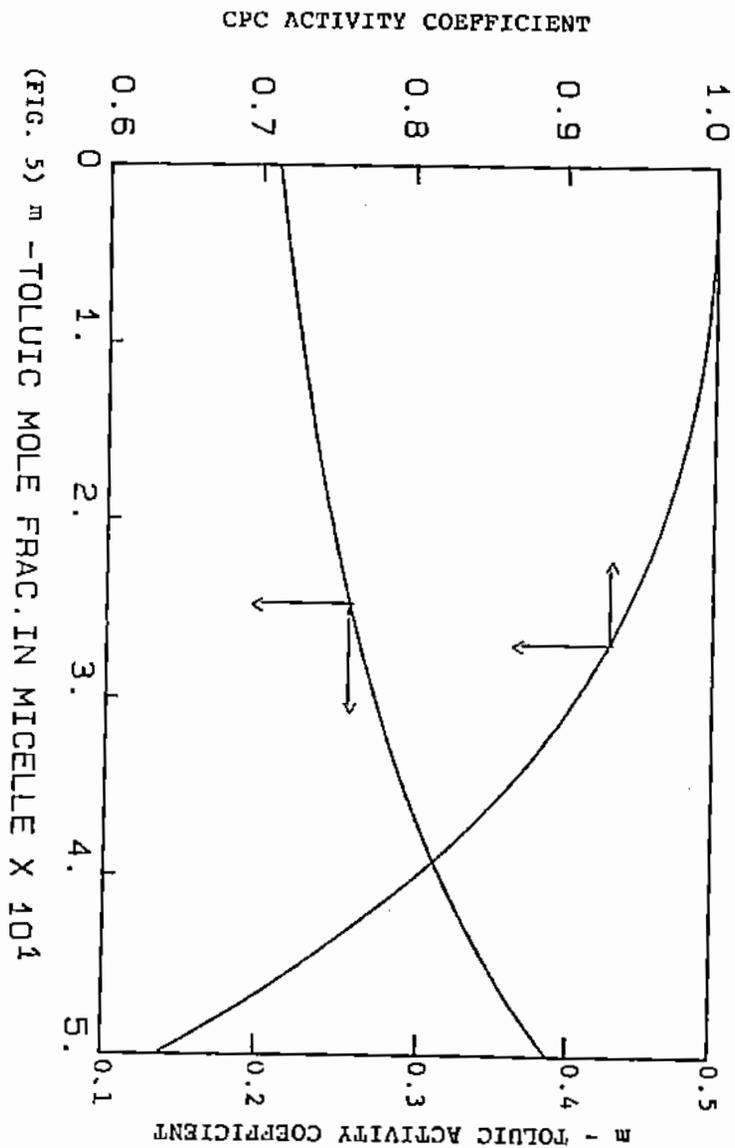
^{b} Parameter in equation 4.

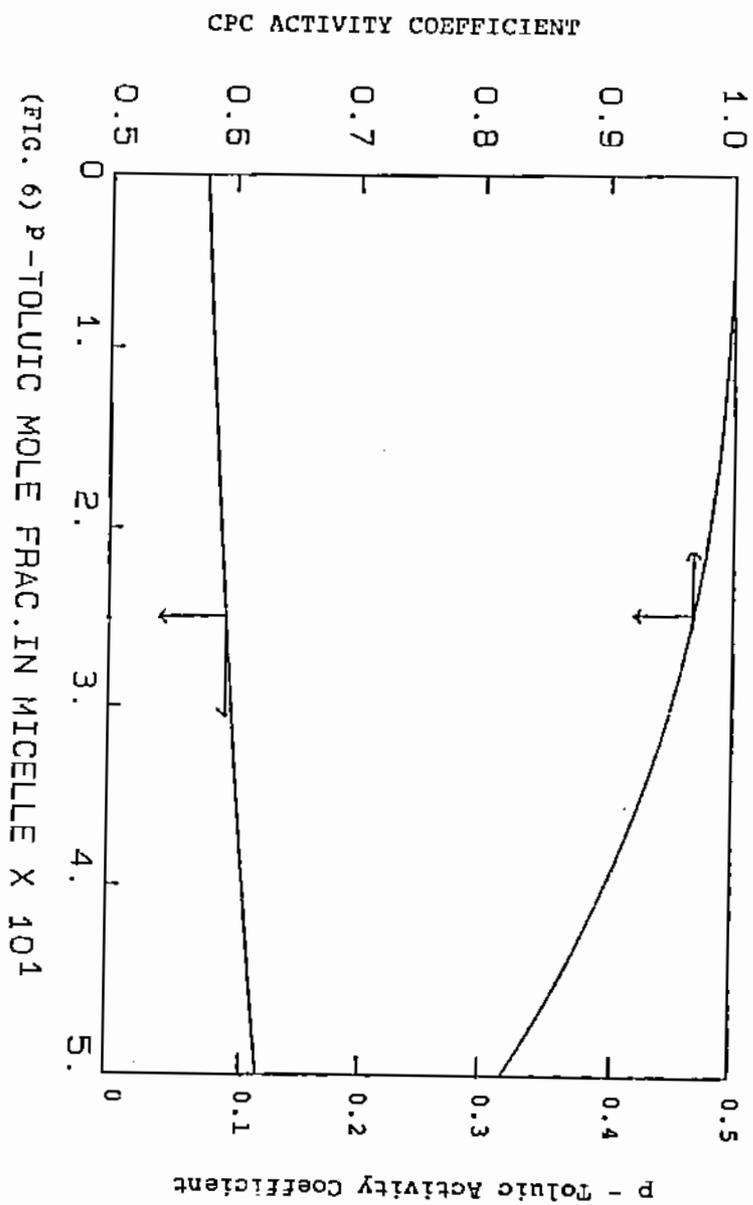^{c} Root mean square deviation in acid concentration in the permeate solution, fitted with model described.

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(FIG. 1) O-TOLUIC MOLE FRACTION IN MICELLE X 10¹

(FIG. 2) M-TOLUIC MOLE FRAC.IN MICELLE X 10¹

(FIG. 3) P-TOLUIC MOLE FRAC.IN MICELLE X 10¹

(FIG. 4) o -TOLUIC MOLE FRAC.IN MICELLE X 10¹

(FIG. 5) m –TOLUIC MOLE FRAC. IN MICELLE X 10¹

CPC ACTIVITY COEFFICIENT

m – TOLUIC ACTIVITY COEFFICIENT

(FIG. 6) P-TOLUIC MOLE FRAC. IN MICELLE X 10^1

CPC ACTIVITY COEFFICIENT

P - Toluic Activity Coefficient

بسم الله الرحمن الرحيم

العوامل المؤثرة على اذابة المركبات العضوية بواسطة المبلات

٢ ـ تأثير طبيعة ومكان المجموعات المستبدلة فى الاحماض العضوية

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يتناول هذا البحث استخدام طريقة المعايرة شبه المتزنة لتعيين ثوابت الاذابة لاحماض " الاورثو ، والمياوالبارا تولويك " فى المحاليل المائية المحضة للمنشط السطحى كلوريد الوكاديكل بريدينم .

وقد أجريت هذه الدراسة على تركزات مختلفة من الاحماض وأمكن استنتاج علاقة بين ثوابت الاذابة للاحماض المختلفة وبين تركيزاتها فى الميلات المتكوسه وهذه العلاقة تكاد تكون خطية بالنسبة امدى التركيزات المستخدمة .

كما أمكن ايضا الوصول الى النتائج التالية :

١ ـ أن وجود مجموعة المثيل والكربوكسيل معا على حلقة البنزين فى هذه الاحماض يزيد اذابتها بدرجة اكبر من مجموع تأثيرهما منفردين .

٢ ـ مكان تواجد كل من المجموعتين المذكورتين بالنسبة للاخرى يؤثر على درجة الاذابه للاحماض المعنيـة .

٣ ـ الفرق بين قيم ثوابت الاذابة للاحماض الثلاثة تظهر بعض التعارض مع الاتجاه المتوقع تبعا لنموذج نظرى حديث بنى على اساس مساهمة المجموعات الموجودة فى الجزى، بدون اجرا، تجارب عملية .