# Distribution of Gibberellic Acid from the Aqueous Phase to the Organic Phase

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ABSTRACT: The reactive extraction of gibberellic acid (GA) using tricaprylmethylammonium chloride (Aliquat 336) diluted in three different alcohols, three different esters, and two different ketones were used to explore the effect of diluent on the gibberellic acid extraction. Experimental measurements have been carried out at $T/K = 298.15$. The loading ratio (Z), extraction efficiency (E), and distribution coefficients (K_D) have been computed. The greatest extraction efficiency has been a value of 79 % for isoamyl alcohol. The linear solvation energy relationship (LSER) model has been fitted for the experimental values of alcohols. The LSER model gives close results to experimental data; R^2 is 0.991.
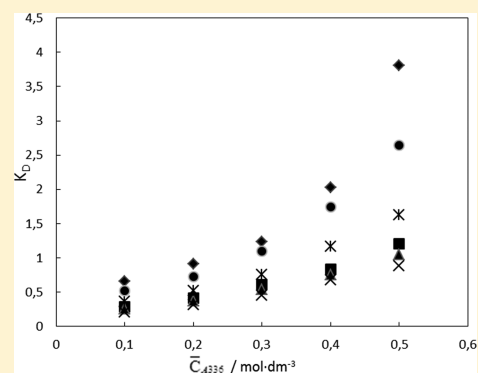
1. INTRODUCTION

Gibberellic acid is one of the gibberellins. It is used for growing plants if used in small amounts. Giberellic acid has an important effect on mRNA for the germination of the seeds. Gibberellic acid is a hormone that also affects the growth of plants in a controlled manner, but it may have an adverse impact on their growth. Gibberellic acid should be used in specific amounts. It is usually used in concentrations between (0.01 and 10) mg·L^{-1}.[1]

Carboxylic acids have been seen as attractive for their use in important medical and pharmaceutical applications in recent years.[2] Besides their known applications as cleaners for household, industrial, and electronic elements, they are also used as automotive oil additives, oil and water well flow enhancers, pH control, cosmetics, and chemical intermediate manufacturing.[3] The extractability of most organic acids by current solvents is very low, and reactive extraction must be considered. Reactive extraction is an important recovery technique which has been receiving increased attention, over the past decade in particular, for the recovery of carboxylic acids. Reactive extraction uses an extractant to remove the acid. Long-chain aliphatic tertiary amines (anion exchange extractants) with seven to nine carbon atoms in each alkyl group are the most effective and most-used extractants for carboxylic acids. When the aliphatic tertiary and quaternary amines are dissolved in different modifiers (solvents) they are powerful extractant reagents for carboxylic acids.[4]

The reactive extraction of some carboxylic acids with tertiary and quaternary amines as an extractant was investigated by Uslu, and equilibrium parameters and thermodynamic parameters have been presented previously.[5−7] Lactic acid extraction with alamine 336 was studied in terms of extraction kinetics by Wasewar et al.[8] Aliquat 336 is a reactant which removed metallic ions from aqueous solutions in previous works,[9,10] but

recently it has been used for the reactive extraction of acids.[11−14] The recovery of neuraminic acid from reaction solutions has been proposed by Zimmermann et al.[15] as a new method. Bora et al. have used it for extracting β-lactam antibiotics and kinetic studies and stripping process have been suggested. Keshav et al.[16] have explained the equilibrium model for extracting propionic acid from aqueous phase Aliquat 336 in various diluents.

This study examines the effect of diluents on the extraction of gibberellic acid by a quaternary amine (A336). The extraction of gibberellic acid from aqueous solutions by A336 in diluents has been studied in the range of amine concentration from (0.1 to 0.5) mol·L^{-1}. Batch extraction experiments have performed by using A336, which is dissolved in three type diluents—type 1 is ketones (MIBK and DIBK), type 2 is alcohols (isoamyl alcohol, octan-1-ol, decan-1-ol), and type 3 is esters (dimethyl phthalate, dimethyl adipate, dimethyl succinate). Active diluents (such as alcohols) give the higher distribution coefficient than inactive diluents (such as aliphatic solvents). Therefore, active diluents were chosen such as alcohols, esters, and ketones, and which types of active diluent more effective were investigated. The distribution coefficients (K_D), the loading factors (Z), and the extraction efficiencies (E) have been calculated from experimental data. There are no available data about the above-mentioned system (gibberellic acid + A336 + diluent) in the literature.
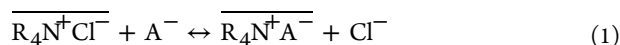
2. THEORY

The mechanism of reactive extraction between Aliquat 336 (A336) and gibberellic acid (GA) can be defined

$$\overline{R_4N^+Cl^-} + A^- \leftrightarrow \overline{R_4N^+A^-} + Cl^- \qquad (1)$$

In eq 1, $\overline{R_4N^+Cl^-}$ is the quaternary ammonium salt (A336), and A^- is the anion from the dissociation of GA.

According to the Kyuchoukov et al.[17] suggestion, if we assume that the undissociated molecules are removed by chemical interaction according to
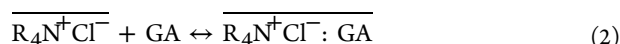
$$\overline{R_4N^+Cl^-} + GA \leftrightarrow \overline{R_4N^+Cl^- : GA} \qquad (2)$$

with the extraction equilibrium constant

$$K_E = \frac{C_{(R_4N^+Cl^- : GA)}}{C_{(GA)} \cdot \overline{C_{(R_4N^+Cl^-)}}} \qquad (3)$$

The concentration is expressed by C, and the overbar indicates the organic phase.

The distribution coefficient, K_D, is expressed as the ratio of the equilibrium concentration of gibberellic acid in both phases

$$K_D = \frac{\overline{C}_{GA}}{C_{GA,total}} \qquad (4)$$

The loading of the extractant, Z, is expressed as the ratio of the total concentration of acid in the organic phase, divided by the total concentration of extractant in the organic phase

$$Z = \frac{\overline{C}_{GA}}{C_{A336}} \qquad (5)$$

Since the both volumes are equal, the efficiency of extraction is described by the following equation,

$$E = 1 - \frac{C_{GA,aq}}{C_{GA,total}} \qquad (6)$$

3. CHEMICALS AND METHODS

3.1. Chemicals. Aliquat 336 (IUPAC name: tricaprylmethylammonium chloride) is a mixture of C_8 (octyl) and C_{10} (capryl) chains with C_8 predominating. Aliquat 336, which is a quaternary ammonium salt, is a clear reddish-brown liquid with the molecular mass of 0.404 kg·mol^{-1}. The density of Aliquat 336 is 0.88 kg·dm^{-3}. Gibberellic acid (Merck, purity in weight > 99 %) is a carboxylic acid and is shown in Figure 1. Alcohols
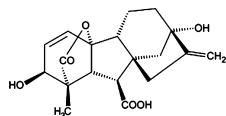

Figure 1. Chemical structure of gibberellic acid.

(isoamyl alcohol (Merck, purity in weight > 99 %), octan-1-ol (Merck, purity in weight > 99 %), nonan-1-ol (Merck, purity in weight > 99 %), decan-1-ol (Merck, purity in weight > 99 %)), ketones (methyl isobutyl ketone, MIBK (Merck, purity in weight > 99 %) and diisobutyl ketone, DIBK (Merck, purity in weight > 99 %)), esters (dimethyl phthalate (Merck, purity in weight > 99 %), dimethyl adipate (Merck, purity in weight >

99 %), dimethyl succinate (Merck, purity in weight > 99 %) have been used without further purification.

3.2. Methods. A sample of 0.65 g·L^{-1} of gibberellic acid was dissolved in distilled water to prepare the initial solution ($1.877 \cdot 10^{-3}$ mol·L^{-1}) according to the production of gibberellic acid by fungi *Gibberella fujikuroi*. The initial organic phases were prepared by the dissolution of trioctylamine (TOA) in the diluents to produce solutions with approximately at eight constant concentrations (0.1 mol·L^{-1}, 0.2 mol·L^{-1}, 0.3 mol·L^{-1}, 0.4 mol·L^{-1}, 0.5 mol·L^{-1}, 0.6 mol·L^{-1}, 0.7 mol·L^{-1}, 0.8 mol·L^{-1}). Amine extractants must always be used in the form of a solution in organic diluents due to its high viscous and corrosive properties. Equal volumes of an aqueous GA solution and an organic solution of A336 were stirred in an Erlenmeyer flask. The mixing of the two phases was carried out by a temperature-controlled Nuve Shaker ST402 at 50 rpm and 25 °C for 2 h. After equilibration, both phases were separated. Amount of GA in aqueous solutions was measured by spectrophotometry (at 254 nm) according to the method previously described by Holbrook et al.[18]

4. RESULTS AND DISCUSSION

4.1. Distribution Coefficient (K_D). The physical extraction of gibberellic acid was studied before reactive extraction. Results of physical extraction of gibberellic acid by pure solvents not containing Aliquat 336 in the organic phase were presented in Table 1. The low affinity of gibberellic acid toward these diluents, particularly in lower concentrations of acid, that is, its higher solubility in water than in organic solvents, is the cause of the low distribution coefficient. Pure isoamyl alcohol has the highest extraction degree of 6.393 % of gibberellic acid.

Naturally, higher distribution coefficients were obtained in reactive extraction than physical extraction. Tables 2 to 4 show the results of the equilibrium data on the reactive extraction of gibberellic acid from the aqueous phase to the organic phase. The prepared constant concentrations of Aliquat 336 in various solvents are in the range of 0.1 mol·L^{-1} to 0.5 mol·L^{-1}. The initial gibberellic acid concentration in aqueous phase was $1.877 \cdot 10^{-3}$ mol·L^{-1}. The concentration of gibberellic acid in organic phase after the extraction at the equilibrium decreases from $1.487 \cdot 10^{-3}$ to $0.324 \cdot 10^{-3}$ with an increasing amount of A336 from 0.1 mol·L^{-1} to 0.5 mol·L^{-1}. The distribution coefficient increases from 0.26 to 3.81 with an increasing initial A336 concentration among the all diluents used in this study. It can be seen that the extraction power of (Aliquat 336 + modifier) mixture changes with the increasing initial concentration of Aliquat 336 in the organic phase.

According to results which come from Tables 2 to 4, the efficiency of diluents is listed as follows: alcohol: isoamyl alcohol > octan-1-ol > decan-1-ol; ester: dimethyl succinate > dimethyl adipate > dimethyl phthalate; ketone: methyl isobutyl ketone (MIBK) > diisobutyl ketone (DIBK).

Results presented in the tables and the above-mentioned ranking can be explained by solvent polarity, which is a function of the transition energy (E_T). Furthermore, E_T was defined for the charge-transfer absorption band of 1-ethyl-4-(methoxycarbonyl) pyridinium iodide in the appropriate solvent by Kosower.[19,20] A high E_T value corresponds to high solvent polarity. Dimroth and Reichardt[21] have proposed a solvent polarity parameter, $E_T(30)$, which is based on the transition energy (E_T) for the longest-wavelength solvatochromic absorption band of the pyridinium *N*-phenolate betaine dye. Brooker et al.[22] suggested an indicator called "merocyanine" for

Table 1. Results[a] for Physical Extraction of Gibberellic Acid with Pure Solvents[13,14]

	solvent	$\overline{C}_{GA}$ $(mol \cdot L^{-1}) \cdot 10^3$	C_{aq} $(mol \cdot L^{-1}) \cdot 10^3$	K_D	$E/\%$
esters	dimethylsuccinate	1.835	0.042	0.023	6.095
	dimethyladipate	1.839	0.038	0.021	5.515
	dimethylglutarate	1.847	0.03	0.016	4.354
ketones	diisobutylketone	1.773	0.104	0.058	15.094
	methylisobutylketone	1.801	0.076	0.042	11.030
alcohols	isoamylalcohol	0.12	1.757	0.068	6.393
	octan-1-ol	0.087	1.79	0.048	4.635
	decan-1-ol	0.059	1.818	0.032	3.143

[a]$\overline{C}_{GA}/mol \cdot L^{-1}$ is the concentration of gibberellic acid in the organic phase. K_D is the distribution coefficient. $E/\%$ is the extraction efficiency.

Table 2. Extraction Equilibrium Results for the System (Gibberellic Acid + Aliquat 336 + Ester)[a]

solvent (ester)	$\overline{C}_{A336}$ $(mol \cdot dm^{-3}) \cdot 10^3$	$\overline{C}_{GA}$ $(mol \cdot dm^{-3}) \cdot 10^3$	K_D	Z	$E/\%$
dimethylsuccinate	0.1	0.384	0.26	3.84	20.46
	0.2	0.518	0.38	2.59	27.59
	0.3	0.659	0.54	2.19	35.11
	0.4	0.814	0.76	2.03	43.36
	0.5	0.962	1.05	1.92	51.25
dimethyladipate	0.1	0.366	0.24	3.66	19.49
	0.2	0.473	0.33	2.36	25.19
	0.3	0.615	0.48	2.05	32.76
	0.4	0.784	0.71	1.96	41.76
	0.5	0.911	0.94	1.82	48.53
dimethylphthalate	0.1	0.324	0.21	3.24	17.26
	0.2	0.46	0.32	2.30	24.50
	0.3	0.583	0.45	1.94	31.06
	0.4	0.766	0.68	1.91	40.80
	0.5	0.879	0.88	1.75	46.83

[a]$\overline{C}_{A336}/mol \cdot L^{-1}$ is the concentration of Aliquat 336 in organic phase. $\overline{C}_{GA}/mol \cdot L^{-1}$ is the concentration of gibberellic acid in the organic phase. K_D is the distribution coefficient. Z is the loading factor, and $E/\%$ is the extraction efficiency.

Table 3. Extraction Equilibrium Results for the System (Gibberellic Acid + Aliquat 336 + Alcohol)[a]

solvent (alcohol)	$\overline{C}_{A336}$ $(mol \cdot L^{-1}) \cdot 10^3$	$\overline{C}_{GA}$ $(mol \cdot L^{-1}) \cdot 10^3$	K_D	K_D^{LSER}	Z	$E/\%$
isoamyl alcohol	0.1	0.745	0.66	0.625	7.45	39.69
	0.2	0.894	0.91	0.928	4.47	47.63
	0.3	1.042	1.24	1.124	3.47	55.51
	0.4	1.259	2.03	2.110	3.14	67.07
	0.5	1.487	3.81	3.516	2.97	79.22
octan-1-ol	0.1	0.530	0.39	0.368	5.30	28.24
	0.2	0.682	0.57	0.517	3.41	36.33
	0.3	0.848	0.82	0.932	2.83	45.18
	0.4	1.051	1.27	1.331	2.62	55.99
	0.5	1.202	1.78	1.710	2.40	64.04
decan-1-ol	0.1	0.427	0.29	0.317	4.27	22.75
	0.2	0.554	0.41	0.433	2.77	29.51
	0.3	0.715	0.61	0.634	2.38	38.09
	0.4	0.856	0.83	0.924	2.14	45.60
	0.5	1.029	1.21	1.280	2.05	54.82

[a]$\overline{C}_{A336}/mol \cdot L^{-1}$ is the concentration of Aliquat 336 in organic phase. $\overline{C}_{GA}/mol \cdot L^{-1}$ is the concentration of gibberellic acid in the organic phase. K_D is the distribution coefficient. K_D^{LSER} is the distribution coefficient obtained from LSER model. Z is the loading factor, and $E/\%$ is the extraction efficiency.

the polarity of solvents. Especially between DIBK and MIBK, polarity is an important factor that affects the extraction. It has been found that the extraction power of Aliquat 336 is more effective with MIBK than DIBK, because the MIBK polarity is higher than DIBK. The polarities of MIBK and DIBK are 0.315 and 0.123, respectively. Ketones and alcohols give high distribution values because they have π-electrons in their structures. This situation provides good solvating media.

Table 4. Extraction Equilibrium Results for the System (Gibberellic Acid + Aliquat 336 + Ketone)[a]

solvent (ketone)	$\overline{C}_{A336}$ (mol·L^{-1})·10^3	$\overline{C}_{GA}$ (mol·L^{-1})·10^3	K_D	Z	E/%
MIBK	0.1	0.643	0.52	6.43	34.25
	0.2	0.796	0.73	3.98	42.40
	0.3	0.985	1.10	3.28	52.47
	0.4	1.193	1.74	2.98	63.55
	0.5	1.362	2.64	2.72	72.56
DIBK	0.1	0.511	0.37	5.11	27.22
	0.2	0.642	0.52	3.21	34.20
	0.3	0.811	0.76	2.70	43.20
	0.4	1.015	1.17	2.53	54.07
	0.5	1.164	1.63	2.32	62.01

[a]$\overline{C}_{A336}$/mol·L^{-1} is the concentration of Aliquat 336 in organic phase. $\overline{C}_{GA}$/mol·L^{-1} is the concentration of gibberellic acid in the organic phase. K_D is the distribution coefficient. Z is the loading factor, and E/% is the extraction efficiency.

Table 5. Solvatochromic Parameters for Alcohols[a]

solvent	π^*	δ	β	α
isoamylalcohol	0.40	0	0.84	0.84
octan-1-ol	0.40	0	0.81	0.77
decan-1-ol	0.40	0	0.81	0.72

[a]π^*, δ, β, and α are the solvatochromic parameters.

Especially isoamyl alcohol give unusually maximum distribution constants, higher than would be expected from polarity arguments alone.[4]

4.2. Loading Factor (Z) and Extraction Efficiency (E). The overall loading factor of Aliquat 336 (Z) is the ratio of the total amount of acid extracted to a total amount of Aliquat 336 in the organic phase. Numerical data of loading factors have been presented in Tables 2 to 4. A decrease in the loading factor has been observed, and overloadings have also been observed in all of the diluents in this study ($Z > 1$). Loading factors have been changed between 7.45 and 2.05 with decreasing A336 concentrations from 0.1 to 0.5 at all concentrations. High loading values show the aggregation and formation of salts between many acid molecules and A336 molecules. The most important factor is the solvation of amine salts (A336). Interactions between the acid–amine complex and the solvent provide information about solvation media. Therefore, inert solvents such as aliphatic hydrocarbons give a low distribution coefficient.[4]

The extraction degree (E) is defined as the ratio of gibberellic acid concentration in the organic phase to the initial acid concentration. The effect of the degree of extraction of gibberellic acid on the distribution between water and Aliquat 336 is presented in Tables 2 to 4 as the E column. A regular increase in extraction efficiency was observed with the increase in concentration of A336. In the area of A336 concentration of 0.6 mol·L^{-1}, the highest values of 79 % and 71 % of the

gibberellic acid are extracted with isoamyl alcohol and MIBK, respectively.

4.3. LSER Model. The hydrogen-bond formation properties of an acid + A336 system can be assessed by theoretically based models of the mass action law, including the physical interaction relations, as well as by assigning a generalized solvatochromic approach with a linear solvation energy relationship (LSER). Bizek et al.[4] suggested a modified version of the LSER model for estimating the extraction equilibria of an Aliquat 336–diluent–acid system.

In this study, the LSER model has been used for predicting a distribution coefficient for the (gibberellic acid + Aliquat 336 + alcohol) systems. The calculation of distribution coefficients of solvents in amine extractants using the LSER model equation was offered in Uslu's earlier work.[23] Briefly, the following equation can be used to describe the effect of diluents on the values of distribution coefficients (K_D) values:

$$\ln K_D = \ln K_D^0 + s(\pi^* + d\delta) + b\beta + a\alpha \tag{7}$$

In eq 7, π^* is the dipole–dipole interaction, and δ is dipole–induced dipole interaction. The solvatochromic parameter α term is of solvent HBD (hydrogen-bond donor) acidities. This explains the ability of the solvent to donate a proton from a solvent–solute hydrogen bond. The β term is of solvent HBA (hydrogen-bond acceptor) basicities. The β term is a measure of the solvent's ability for accepting a proton from a solute–solvent hydrogen bond. The coefficients s, d, a, and b include the solute properties that come from regression.[24]

The distribution coefficient values can be regressed with the solvatochromic parameters of the solvents from Table 5[25,26] according to eq 7. Experimental data have been regressed using a computer program SPSS v14.0. The results with regard to K_D^{LSER} have been presented in Table 3. The LSER model values show acceptable regression to the experimental data. The estimated values of parameters of the model are been presented in Table 6. It has been determined that, using this model, distribution coefficients of gibberellic acid between water and the A336 + diluent system can be defined. Consequently, the regression equation (eq 7) has been created to estimate the distribution coefficients:

$$\ln K_D = (-0.143) + (-1.643) \cdot (\pi^* + 0.\delta) + 1.535(\beta)$$
$$+ 2.192(\alpha) \tag{8}$$

5. CONCLUSION

Gibberellic acid extraction with a reactive extractant + organic diluent has been studied. Equilibrium parameters of reactive extraction of gibberellic acid were calculated in different conditions. It is concluded that diluents without amine extractants are unsuitable for the extraction of gibberellic acid because they have low distribution coefficients ($K_D \ll 1$). However, these solvents using Aliquat 336 are more effective since $K_d > 1$. The isoamylalcohol is the most effective diluent among diluents used in this study. It has a 79 % extraction efficiency. The results of the liquid–liquid equilibrium for

Table 6. Results of the Regression Coefficient for the LSER Equation[a]

coefficient	log K_D^0	s	d	b	a	R^2	χ
	−0.143	−1.643	0	1.535	2.192	0.991	0.011

[a]log K_D^0 is the intercept, and s, d, b, and a are coefficients come from regression. χ is the standard error.

alcohols have been regressed by a LSER model which takes into account physical interactions. Very close values to the experiment were reached by the LSER model; the R^2 is 0.99.

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Notes

The authors declare no competing financial interest.

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