

SOLVENT EXTRACTION OF SUCCINIC ACID

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Abstract. Distribution studies for the system water–succinic acid–organic solvent have been made at $25 \pm 2^\circ$. The phase equilibria of the systems were measured at concentrations varying between 0.05 M to 0.2 M. Organic solvents used were amyl alcohol, amyl acetate, chloroform, ether, carbon tetrachloride, benzene, toluene, bromobenzene, chlorobenzene, nitrobenzene and aniline. Dielectric constant and structure of the solvents appear to be the decisive factors in fixing the extraction efficiency.

Liquid–Liquid extractions based on a knowledge of the simple distribution law¹ have been widely used in purification and separation of compounds because of their simplicity, convenience and rapidity to perform. The technique is industrially important. For this purpose, a knowledge of distribution ratio is essential. Various equations have been developed and improved upon^{2,3,4} in order to obtain expressions for the distribution law which could explain the experimental results. The technique has been employed extensively for the extraction of various organic materials.⁵

Organic compounds are generally extracted by organic solvents in the uncharged form. Brown and Mathieson⁶ undertook the distribution studies of acetic acid and its chloro derivatives between water and various organic solvents in order to derive a suitable expression for the distribution ratio. Murty *et al.*⁷ have reported similar results for adipic acid. Succinic acid as such has not been investigated much. Zaheeruddin *et al.*^{8,9} have reported the distribution of succinic acid between water and amyl alcohol in the presence of neutral salts. They found that the distribution ratio in the absence of salts favours the aqueous phase. The presence of salts, however, shifts the equilibrium in favour of the organic layer. It was desired in the present work to study the distribution behaviour of succinic acid between water and various other solvents and to determine the suitability of these for the purpose of extracting the solute from its aqueous solutions.

Experimental

Reagents. All the solvents used in the investigation were provided by E. Merck (Germany). They were twice distilled before used and the first and the last few ml of the distillate were rejected. They were tested refractometrically for the purpose of purity which was found to be in the range of 99.08 – 100%. Succinic acid used as solute for various experiments was BDH analytical

reagent.

Method. Zaheeruddin and A. Aziz⁸ have reported that the distribution law is obeyed in its simple form within a concentration range of 0.05 M to 0.2 M succinic acid. Therefore, a series of five concentrations varying between the said range were prepared for each equilibrating system which consisted of 50 ml of the aqueous solutions and 50 ml of the organic solvents. The mixtures were prepared in clean and dry separatory funnels with their stems chopped off near their stop cocks. The mixtures were shaken thoroughly and were equilibrated in a water bath. The temperature of the bath was maintained at $25 \pm 2^\circ$ with a thermoregulator. Agitation of the mixtures was continued at 5 min intervals. Agitation for longer periods did not produce any further change. After an hour when the phase separation had occurred, the mixtures were removed from the bath and phases were separated without disturbing the equilibrium. Ten ml. of each layer were titrated in triplicate against freshly prepared standard, carbonate free sodium hydroxide using phenolphthalein as indicator.

The partition coefficient was calculated using the formula, $K = C_w/C_o$ in which C_w and C_o represent solute concentrations in g moles/litre in the aqueous and organic layers respectively.

The experiments were repeated for each of the solvents reported in the results.

The partition coefficient reported is the average value of five determinations for each solvent at the temperature of the experiment. K values also indicate the average deviation of the measurements.

Percent extraction has been calculated from the average value of the reported partition coefficients.

Results and Discussion

Values of the partition coefficient for the distribution of succinic acid between water and various solvents

TABLE 1. PARTITION COEFFICIENT AND PERCENT EXTRACTION OF SUCCINIC ACID.

Organic extractant	Partition coefficient	Percent extraction	Dielectric constant ¹¹
Amyl alcohol	1.517 ± 0.002	39.72	13.9
Amyl acetate	16.055 ± 0.010	5.86	4.75
Ether (diethyl)	7.523 ± 0.050	11.73	4.355
Chloroform	208 ± 3.2	0.48	4.806
Carbon tetrachloride	598 ± 8	0.17	2.228
Benzene	108.1 ± 4.6	0.92	2.274
Bromobenzene	416 ± 6	0.24	5.40
Chlorobenzene	65.6 ± 3.4	1.50	5.621
Nitrobenzene	151.04 ± 3.9	0.66	34.82
Aniline	3.127 ± 0.015	24.23	6.89
Toluene	296 ± 6	0.34	2.379

at 25+2° indicate that the simple distribution law is obeyed for all the equilibrating systems under study. This is in confirmation with the results reported¹⁰ earlier for water-succinic acid-hexanol system. Conformity with the simple distribution law is also indicative of the fact that dimerisation in the organic phases is positively excluded. However, there does occur some dissociation¹² in the aqueous phase, but this dissociation is not appreciable enough to influence the equilibrium or to affect the validity of the simple distribution law.

The temperature control within ±2° appears to be not very efficient. However, an earlier investigation¹³ revealed that an increase in temperature shifts the equilibrium in favour of the organic phase which in turn increases the percent extraction of the solute.

It is observed from the K values that the partition coefficient greatly favours the aqueous layer in all the equilibrating systems under study. Percentage of the solute extracted by the various solvents indicate clearly that in general aromatic solvents are poor extractants for succinic acid. This is obvious due to the structural differences between the solute and the aromatic solvents under report. The efficiency of aliphatic solvents varies with their nature. It appears that within this class, dielectric constant of the solvent is the decisive factor in determining its extraction efficiency. This is confirmed when the percent extraction values of different solvents are compared against their corresponding dielectric constants¹¹. A gradual increase in percent extraction is observable with a corresponding increase in the dielectric constant values. Carbon tetrachloride evidently is seen to be the least effective whereas amyl alcohol has been found to be the most efficient. Nitro-

benzene, though highly polar (Dielectric constant = 34.82) yet it is a poor extractant because of its aromatic nature. The remaining aromatic solvents except aniline, also show some degree of regularity with reference to their extracting capacity for succinic acid which can be related. With their dielectric constants. Aniline being basic in character does not fit in this pattern because the solute itself is acidic in nature. Thus the percent extraction is relatively very high.

Another solvent which does not fit into the proposed pattern, is ether. It is observed that the % extraction value corresponding to ether is not compatible with its dielectric constant. This may be explained on the basis of the high solubility of ether (7.5 g/100 of water at 20°), which results in a relatively high concentration of ether in the aqueous layer near the interface. Due to this reason, the solubility of the organic solute improves in the aqueous phase which results in lower percent extraction than expected of the high dielectric constant of the solvent.

The over all increasing effectiveness with reference to the extraction efficiency of various solvents studied, falls in the following order:

- (a) Aliphatic solvent: Amyl alcohol < chloroform < amyl acetate < ether < carbon tetrachloride.
 (b) Aromatic solvents: Aniline < chlorobenzene < benzene < nitrobenzene < toluene < bromobenzene.

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