

A SINGLE DROP LIQUID EXTRACTION EXPERIMENT

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This note describes an experiment designed to measure mass transfer coefficients for liquid extraction from single drops. This experiment is one of several laboratory experiments which are assigned as term projects in the chemical engineering undergraduate mass transfer course at the University of California, Santa Barbara. The course is ordinarily taken by juniors and proceeds the "transport processes" laboratory course taken during the senior year.

Students, working in groups of two, are assigned individual projects which can reasonably be completed in six to eight weeks. The experiment described here is one of the more successful experiments developed for this course. The apparatus is simply constructed, quite inexpensive and the experimental results obtained may be bracketed by calculations based on theoretical models for the mass transfer process.

THE APPARATUS FOR this experiment is shown schematically in Figure 1. The extraction column consists of a 1¼-inch diameter glass tube four feet in length and closed at each end with a rubber stopper. Toluene was the continuous phase and the discontinuous phase, water, was introduced into the extraction column through a glass tubing nozzle at the top of the column. The solute, benzoic acid, was extracted from toluene by water. The water drops fall through the column and coalesce in the funnel at the bottom of the column. The water level in the funnel could be maintained constant by adjusting the flow rate of water leaving the column to that of the water entering the column. Drop sizes could be conveniently varied by varying the diameter of this drop formation nozzle.

The falling velocities of the water drops were measured using a stopwatch and the drop diameters were determined by counting the drop formation rate and by measuring the volumetric flow rate. The students used three different drop sizes in the experiments. The measured drop

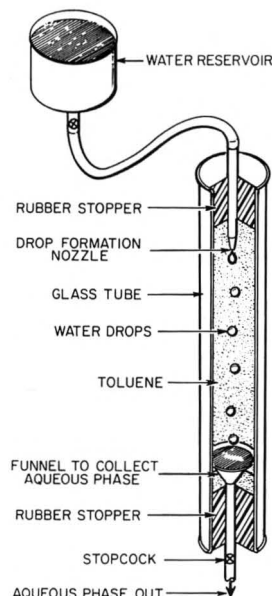


Figure 1.—Schematic of Experimental Apparatus.

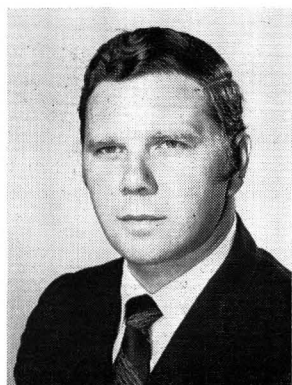
velocities were compared to velocities predicted from the correlation of Klee and Treybal (1956). The results of this comparison are shown in Table I. It was found that the measured drop velocities were about 9% lower than the predicted velocities. These differences are small and may be attributed to the effect of the solute on the physical properties of toluene.

One of the desirable features of the water-benzoic acid-toluene system is the relative simplicity of the concentration measurements. Benzoic acid concentrations in each phase were determined by titrating with sodium hydroxide solutions to a phenolphthalein end-point. For the toluene phase a sodium hydroxide in ethanol solution was used for the titrations.

Before running the extraction experiment equilibrium data were obtained in the usual man-

TABLE I.—Comparison of Predicted and Measured Drop Velocities

Drop Diameter cm	Terminal Velocity (cm/sec)	
	Predicted	Measured
0.356	10.5	9.5
0.448	12.4	11.5
0.720	11.5	10.7



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ner using a separatory funnel. It was found that the equilibrium curve was linear over a wide range in concentration (up to 0.36 molar in the toluene phase).

OVERALL MASS TRANSFER coefficients were calculated from the measured exit concentration of the aqueous phase. The overall mass transfer coefficient defined in terms of the average concentration difference is given by Equation 1.

$$K_w = \bar{N}_A / (\bar{x}^* - \bar{x}) \quad (1)$$

Because the distribution coefficient of benzoic acid strongly favors the organic phase, the concentration of benzoic acid in toluene did not change significantly during the course of the experiments.

To interpret the mass transfer data, students were asked to use three theoretical models which would represent probable limits for the individual phase mass transfer coefficients. The overall mass transfer coefficient can then be calculated using the additivity of resistances concept as stated by Equation. 2.

$$\frac{1}{K_w} = \frac{1}{k_w} + \frac{1}{mk_T} \quad (2)$$

The major resistance to mass transfer in this system is in the discontinuous phase; this is due mainly to the large value of m .

The lower limit for mass transfer rates should be given by assuming that the drops are completely stagnant. Since the drop contact times are short (on the order of 10 seconds), diffusion

into a stagnant drop is given to good approximation by the Higbie (1935) penetration theory:

$$k_w = 2C_w \sqrt{\frac{D_w}{\pi t}} \quad (3)$$

For stagnant drops the continuous phase mass transfer coefficient was obtained from the correlation of Ranz and Marshall (1952) for transport from rigid spheres:

$$\frac{k_w d}{C_T D_T} = 2.0 + 0.60 \text{Re}^{1/2} \text{Sc}^{1/3} \quad (4)$$

The second approach taken to model the mass transfer process was to use the circulating drop theory of Kronig and Brink (1950):

$$k_w = \frac{C_w d}{6\theta} \left\{ 1 - \frac{3}{8} \sum_{n=1}^{\infty} A_n^2 \exp\left(-\frac{64 B_n D_w \theta}{d^2}\right) \right\} \quad (5)$$

Following the suggestion of Treybal (1963) for the case of circulating drops, the continuous phase mass transfer coefficient was assumed to be given by penetration theory. The contact time in this case is taken as the drop diameter divided by the falling velocity.

An upper limit for mass transfer rates was assumed to be given by the fully turbulent drop model considered by Handlos and Baron (1957). The drop mass transfer coefficient for this case is given by.

$$k_w = \frac{0.00375 C_w v}{1 + \mu_w / \mu_T} \quad (6)$$

The continuous phase mass transfer coefficient for fully turbulent drops was assumed to be given by penetration theory as for the circulating drop case.

Table II shows the results of the theoretical calculations as compared to the experimental results. It is seen that the experimental results are within the limiting behavior as predicted by the stagnant drop and the fully turbulent drop theories and show closest agreement with the circulating drop theory (average deviation = 33%). □

TABLE II.—Comparison of Predicted and Measured Mass Transfer Coefficients

Drop Diameter cm	Overall Mass Transfer Coefficient, $K_E \times 10^5$, g moles/cm ² sec			
	Experiment	Theory		
		Stagnant	Circulating	Turbulent
0.356	12.6	4.40	6.70	73.0
0.448	10.7	4.88	8.34	88.2
0.720	7.86	4.65	10.32	81.2

(Continued on p. 35.)

analogous to that for the energy functions. Transforms of the entropy are termed Massieu functions which yield relations upon cross differentiation analogous to those of Maxwell.

SUMMARY

The theoretical basis, use, and construction of a mnemonic octahedron for the representation of the energy functions and their derivatives together with the Maxwell equations, has been presented. The device, which is easily constructed to any size—desk top or pocket version, should be valuable to both students and teachers. Although classic thermodynamics requires a minimum of memorization, we hope the THERMODORM will aid the student in understanding and unifying the energy functions together with their variables and prove to be more than an optimal crib-sheet for closedbook exams. □

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ChE book reviews

Processes and Systems in Industrial Chemistry, H. P. Meissner, Prentice-Hall, Englewood Cliffs, N.J. (1971), 386pp., \$14.95.

This book is primarily an introductory discussion of reaction kinetics and associated thermodynamics from an industrial chemical point of view. The author, however, includes chapters on liquid-solid equilibria, material balances and energy balances. There is also a chapter on the frequently neglected topic of electrochemical operations. Roughly one-third of the book is devoted to problems. The book is noteworthy in relating the discussion and the problems to specific chemical processes.

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Ed. Note. The author has classified processes "by (1) types of equilibria, (2) types of energy management problems, (3) types of rate problems (including catalysis), and (4) types of flow sheet patterns and materials handling problems." The book contains eleven chapters titled: Equilibrium in homogeneous systems; Heterogeneous reaction equilibria; Liquid-solid equilibria involving ions; Management of materials; Management of heat energy; Homogeneous reaction rates; Catalytic reactions and reactors; Reactions between gases and solids; Chemical reactors; Reactions having an unfavorable ΔF ; and Industrial electrochemical operations. There are 125 realistic case-type problems for effective illustration of the material.

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Nomenclature

A_n, B_n	Eigenvalues
C	Molar concentration
d	Drop diameter
D	Diffusion coefficient
k	Individual phase mass transfer coefficient
K	Overall mass transfer coefficient
m	Slope of equilibrium curve
\bar{N}_A	Average mass transfer rate
Re	Reynolds number
Sc	Schmidt number
v	Drop velocity
\bar{x}	Average mole fraction benzoic acid in water phase
x^*	Mole fraction benzoic acid that would be in equilibrium with the bulk toluene phase concentration
θ	Contact time
μ	Absolute viscosity

Subscripts:

T	Toluene phase
w	Water phase

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