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## FURTHER STUDY ON THE ENRICHMENT OF HEAVY WATER IN CONTINUOUS-TYPE THERMAL-DIFFUSION COLUMNS

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### ABSTRACT

The separation equations for the enrichment of heavy water in the continuous-type thermal-diffusion columns have been derived by treating the enriching and stripping sections separately. The experimental works for the separation of  $\text{H}_2\text{O}-\text{HDO}-\text{D}_2\text{O}$  system have been also conducted. The present theoretical predictions are in better agreement with the experimental results than those predicted in previous works, in which the average value of concentration–product term was taken as one at the feed position, while in the present study it was made separately in enriching and stripping sections.

*Key Words:* Continuous-type; Separation; Thermal diffusion; Water isotopes

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## INTRODUCTION

Nuclear energy plays an important role in fulfilling society's energy requirements by fission reaction, and perhaps will be supplied by thermonuclear fusion in the distant future. It has been realized that heavy water,  $D_2O$ , is the most feasible moderator and coolant used in fission reactors to furnish excess neutrons, which may be absorbed by materials other than uranium, and may serve as an indirect nuclear fuel for fusion reactors. Between 1940 and 1945, four heavy water production plants were placed in operation by the U.S. government under the Manhattan District Program (1,2).

Thermal diffusion is an unconventional process for separating liquid or gas mixtures. It is a powerful purification technique used to concentrate desired highly valuable materials such as isotopes and rare gases, as well as to remove the undesired ones. The first application of thermal diffusion was to separate the isotope mixtures of uranium at Oak Ridge during World War II. This method is particularly attractive for separation of hydrogen isotopes because of large ratios of molecular weights. It has been shown that heavy water can be concentrated in a thermal-diffusion column (3-5). The well-known separation phenomenon of thermogravitational thermal-diffusion column was pointed out by Clusius and Dickel (3,4) and the complete theory of separation in a Clusius-Dickel column was first presented by Furry et al. (6,7).

Convective current is the dominant factor of the thermogravitational thermal diffusion to produce a cascading effect, analogous to the multistage effect of countercurrent extraction, and thus a relatively large separation is obtained. In fact, the design of a thermogravitational thermal-diffusion column will produce the desirable cascading effect and the undesirable effect of remixing with the diffusion along the column axis and across the column. Thus, it is concluded that any improvement in equilibrium separation must be associated with a suppression of remixing effect and an enhancement of the cascading effect. Consequently, many significant papers were devoted entirely or largely to improve the performance of thermal-diffusion columns with inclination (8,9), rotation (10), moving-wall (11), and packing (12), as well as by winding a wire helix (13). The enrichments obtained from these improved columns are somewhat better than that from the conventional Clusius-Dickel column.

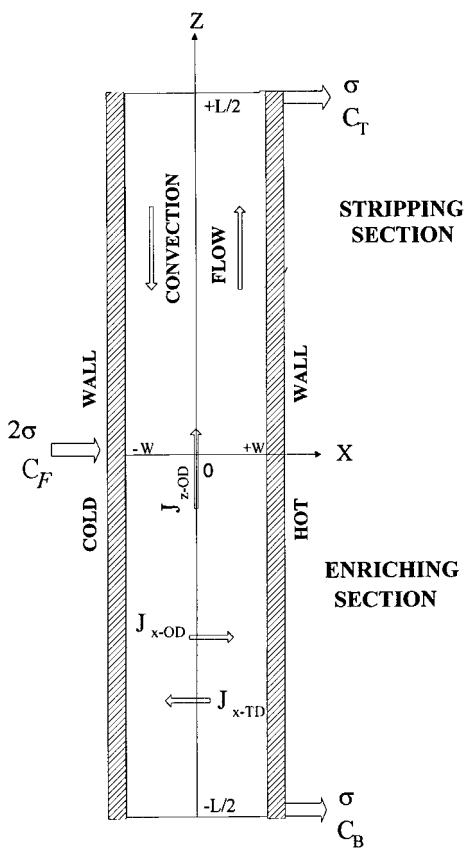
The separation theory of thermal diffusion for binary systems (3-5,14-18) have been well developed. Yeh et al. (19-21) studied the enrichments of heavy water in thermal-diffusion columns, however, the well-established separation theory for  $H_2O$ -HDO- $D_2O$  system is still not available. Most of the previous investigators took the concentration-product term,  $C\hat{C}$ , as that at the feed position in the column. For more precise analysis, we will consider its average values separately in enriching and stripping sections here. Accordingly, the



mathematical treatment may be cumbersome. Finally, the present theory will be confirmed with the experimental results.

### THE EQUATION OF SEPARATION

Jones, Furry, and Onsager (6) gave a well-developed theory to present the separation equation for a thermal-diffusion column in continuous operations. Figure 1 shows the flows and fluxes prevailing in a continuous flow column. Yeh and Yang (19) developed the transport equation for the enrichment of heavy water in batch-type Clusius-Dickel column. For continuous operation, the



**Figure 1.** Flows and fluxes in a continuous-type thermal-diffusion column for separation of heavy water.



transport equations may be modified from the previous results as follows:

$$\tau_e = -\sigma C_B = -\sigma C_e + H C_e \hat{C}_e - K \frac{dC_e}{dz} \quad (1)$$

for the enriching section, and

$$\tau_s = \sigma C_T = \sigma C_s + H C_s \hat{C}_s - K \frac{dC_s}{dz} \quad (2)$$

for the stripping section. The transport constants in Eqs. (1) and (2) are defined by

$$H = \frac{\alpha \beta_T \bar{\rho} g (2\omega)^3 B (\Delta T)^2}{6! \mu \bar{T}} < 0 \quad \text{for } \alpha < 0 \quad (3)$$

and

$$K = \frac{\bar{\rho} g^2 \beta_T^2 (2\omega)^7 B (\Delta T)^2}{9! \mu^2 D} + 2\omega \bar{\rho} D B \quad (4)$$

while the pseudo-concentration products,  $C_e \hat{C}_e$  and  $C_s \hat{C}_s$ , are defined as

$$C \hat{C} = C \left\{ 0.05263 - (0.05263 - 0.0135 K_{eq}) C - 0.027 \left\{ \left[ 1 - \left( 1 - \frac{K_{eq}}{4} \right) C \right] C K_{eq} \right\}^{1/2} \right\} \quad (5)$$

in which the equilibrium constant  $K_{eq}$  for the following equilibrium relation



is

$$K_{eq} = \frac{C_{2F}^2}{C_{1F} C_{3F}} = \frac{[\text{HDO}]^2}{[\text{H}_2\text{O}][\text{D}_2\text{O}]} \cdot \frac{19 \times 19}{18 \times 20} \quad (7)$$

$K_{eq}$  does not vary sensitively within the operating temperature range. For instance, the values of the equilibrium constant are  $K_{eq} = 3.80$  and  $3.793$ , respectively, at  $T = 25$  and  $30.5^\circ\text{C}$  (22).

Equations (3) and (4) were obtained according to Jones and Furry theory with the physical properties considered as constants. The simultaneous solution of this set of equations is complicated and inconvenient to analyze due to the nonlinear forms of Eqs. (1) and (2). Usually, the degree of separation for isotope mixtures is very small; therefore, the simplest approximation for  $C \hat{C}$  in the whole



column was made in the previous works (21), such as

$$C\hat{C} = C_F \hat{C}_F \quad (8)$$

where  $C_F \hat{C}_F$  denotes the value of  $C\hat{C}$  at feed position.

In the present study, however, rather reasonable approximations for  $C_e \hat{C}_e$  ( $= F_e$ , an appropriate constant) and  $C_s \hat{C}_s$  ( $= F_s$ , another appropriate constant) will be made, and Eqs. (1) and (2) reduce to

$$H[F_e + (\sigma/H)(C_B - C_e)] = K \frac{dC_e}{dz} \quad (9)$$

and

$$H[F_s - (\sigma/H)(C_T - C_s)] = K \frac{dC_s}{dz} \quad (10)$$

Equations (9) and (10) could be rewritten as

$$-F_e + \sigma' C_B - \sigma' C_e = \frac{dC_e}{dz'} \quad (11)$$

and

$$-F_s - \sigma' C_T + \sigma' C_s = \frac{dC_s}{dz'} \quad (12)$$

in which

$$\sigma' = \frac{\sigma}{(-H)} \quad \text{and} \quad z' = \frac{z(-H)}{K}. \quad (13)$$

For solving Eqs. (11) and (12), the following boundary conditions are reached:

$$C_e = C_s = C_F \quad \text{at } z' = 0 \quad (14)$$

$$C_s = C_T \quad \text{at } z' = \frac{L'}{2} \quad (15)$$

$$C_e = C_B \quad \text{at } z' = -\frac{L'}{2} \quad (16)$$

where

$$L' = \frac{L(-H)}{K} \quad (17)$$



The degrees of separation,  $\Delta_e$  and  $\Delta_s$ , in the enriching and stripping sections, respectively, were obtained by solving Eqs. (11) and (12) with the use of Eqs. (14)–(16). The results are

$$\Delta_e = C_B - C_F = \frac{F_e(1 - e^{-\sigma' L/2})}{\sigma'} \quad (18)$$

$$\Delta_s = C_F - C_T = \frac{F_s(1 - e^{-\sigma' L/2})}{\sigma'} \quad (19)$$

Summing Eqs. (18) and (19) gives the degree of separation for the whole column

$$\Delta = C_B - C_T = \Delta_e + \Delta_s = \frac{(F_e + F_s)(1 - e^{-\sigma' L/2})}{\sigma'} \quad (20)$$

If the simplest approximation, Eq. (8), is taken,  $F_e = F_s = C_F \hat{C}_F$ , and Eq. (20) reduces to

$$\Delta = \frac{2C_F \hat{C}_F (1 - e^{-\sigma' L/2})}{\sigma'} \quad (20a)$$

The appropriate values of  $F_e$  and  $F_s$  may be determined by the method of least square. Since the separations efficiency obtained from the enriching and stripping sections are somewhat different, therefore, the calculation of the appropriate values of these constants must be conducted separately from  $C_F$  to  $C_B$  for  $F_e$  in the enriching section and from  $C_T$  to  $C_F$  for  $F_s$  in the stripping section. Accordingly, minimizing the following integration

$$R_e = \int_{C_F}^{C_B} (C_e \hat{C}_e - F_e)^2 dC_e \quad (21)$$

i.e.,

$$\frac{dR_e}{dF_e} = \int_{C_F}^{C_B} -2(C_e \hat{C}_e - F_e) dC_e = 0 \quad (22)$$

one obtains

$$F_e = \frac{1}{C_B - C_F} \int_{C_F}^{C_B} C_e \hat{C}_e dC_e \quad (23)$$



for the enriching section. Similarly, for the stripping section

$$F_s = \frac{1}{C_F - C_T} \int_{C_T}^{C_F} C_s \hat{C}_s dC_s \quad (24)$$

It is noted from Eqs. (23) and (24) that the appropriate values of  $F_e$  and  $F_s$ , thus obtained, are exactly the average values of  $C_e \hat{C}_e$  and  $C_s \hat{C}_s$ , respectively, in their concentration ranges.

Since the last term of Eq. (5) is hard to integrate, we will expand it in Taylor's series and only retain the first two terms, which may result in good approximation. That is

$$\begin{aligned} C\hat{C} = & 0.05263C - 0.05263C^2 + 0.0135C^2K_{eq} \\ & - 0.027C^{3/2}K_{eq}^{1/2} \left( 1 - \frac{1}{2}C + \frac{1}{8}CK_{eq} \right) \end{aligned} \quad (25)$$

Thus, from Eqs. (23) and (24) one may obtain

$$F_e = \frac{M(C_B^2 - C_F^2) - N(C_B^{5/2} - C_F^{5/2}) + P(C_B^3 - C_F^3) + Q(C_B^{7/2} - C_F^{7/2})}{2(C_B - C_F)} \quad (26)$$

$$F_s = \frac{M(C_F^2 - C_T^2) - N(C_F^{5/2} - C_T^{5/2}) + P(C_F^3 - C_T^3) + Q(C_F^{7/2} - C_T^{7/2})}{2(C_T - C_F)} \quad (27)$$

in which

$$M = \frac{5263}{100000} \quad (28)$$

$$N = \frac{27K_{eq}^{1/2}}{1250} \quad (29)$$

$$P = \frac{9K_{eq}}{1000} - \frac{5263}{150000} \quad (30)$$

$$Q = \frac{27}{3500}K_{eq}^{1/2} - \frac{27}{14000}K_{eq}^{3/2} \quad (31)$$

Also, from Eqs. (18) and (19) the concentrations at the bottom and top of the column,  $C_B$  and  $C_T$ , may be expressed as follows:

$$C_B = C_F + \Delta_e \quad (32)$$

$$C_T = C_F - \Delta_s \quad (33)$$



Substitution of Eqs. (32) and (33) into Eqs. (26) and (27) gives

$$F_e = \frac{\{M\Delta_e(2C_F + \Delta_e) - N[(C_F + \Delta_e)^{5/2} - C_F^{5/2}] + P[(C_F + \Delta_e)^3 - C_F] + Q[(C_F^3 + \Delta_e)^{7/2} - C_F^{7/2}]\}}{2\Delta_e} \quad (34)$$

$$F_s = \frac{\{M\Delta_s(2C_F - \Delta_s) - N[C_F^{5/2} - (C_F - \Delta_s)^{5/2}] + P[C_F^3 - (C_F - \Delta_s)^3] + Q[C_F^{7/2} - (C_F - \Delta_s)^{7/2}]\}}{2\Delta_s} \quad (35)$$

Once the appropriate values of  $F_e$  and  $F_s$ , as well as  $\Delta_e$  and  $\Delta_s$ , are calculated from Eqs. (18) and (34) and Eqs. (19) and (35), respectively, the degree of separation obtainable in the whole column may be evaluated by Eq. (20).

### GRAPHICAL REPRESENTATION

Combination of Eqs. (18) and (34) results in

$$\frac{(1 - e^{-\sigma'L/2})}{\sigma'} = \frac{\Delta_e}{F_e} = \frac{2\Delta_e^2}{\{M\Delta_e(2C_F + \Delta_e) - N[(C_F + \Delta_e)^{5/2} - C_F^{5/2}] + P[(C_F + \Delta_e)^3 - C_F^3] + Q[(C_F + \Delta_e)^{7/2} - C_F^{7/2}]\}} \quad (36)$$

$$\frac{(1 - e^{-\sigma'L/2})}{\sigma'} = \frac{\Delta_s}{F_s} = \frac{2\Delta_s^2}{\{M\Delta_s(2C_F - \Delta_s) - N[C_F^{5/2} - (C_F - \Delta_s)^{5/2}] + P[C_F^3 - (C_F - \Delta_s)^3] + Q[C_F^{7/2} - (C_F - \Delta_s)^{7/2}]\}} \quad (37)$$

Similarly, from Eqs. (19) and (35)

$$\frac{(1 - e^{-\sigma'L/2})}{\sigma'} = \frac{\Delta_s}{F_s} = \frac{2\Delta_s^2}{\{M\Delta_s(2C_F - \Delta_s) - N[C_F^{5/2} - (C_F - \Delta_s)^{5/2}] + P[C_F^3 - (C_F - \Delta_s)^3] + Q[C_F^{7/2} - (C_F - \Delta_s)^{7/2}]\}} \quad (37)$$

Accordingly, some graphical representations for  $(1 - e^{-\sigma'L/2})/\sigma'$  vs.  $\Delta_e$ , and  $(1 - e^{-\sigma'L/2})/\sigma'$  vs.  $\Delta_s$ , respectively, are plotted in Figs. 2 and 3, with  $C_F$  as a parameter. Finally, combination of Figs. 2 and 3 yields Fig. 4, which is the graphical representation of Eq. (20).



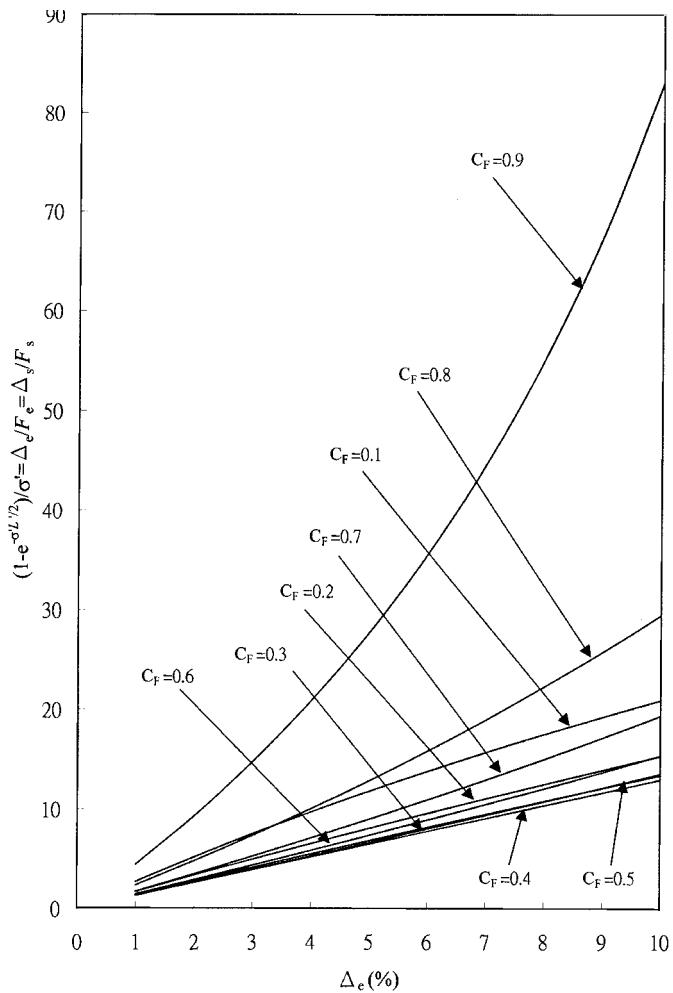


Figure 2.  $\Delta_e/F_e$  vs.  $\Delta_e$  with  $C_F$  as a parameter.

### EFFECT OF OPERATING PARAMETERS ON PERFORMANCE

There are many parameters that affect the performance in the thermal diffusion columns. Among them, column length ( $L$ ), flow rate ( $\sigma$ ), and feed concentration ( $C_F$ ) may be the most important factors.



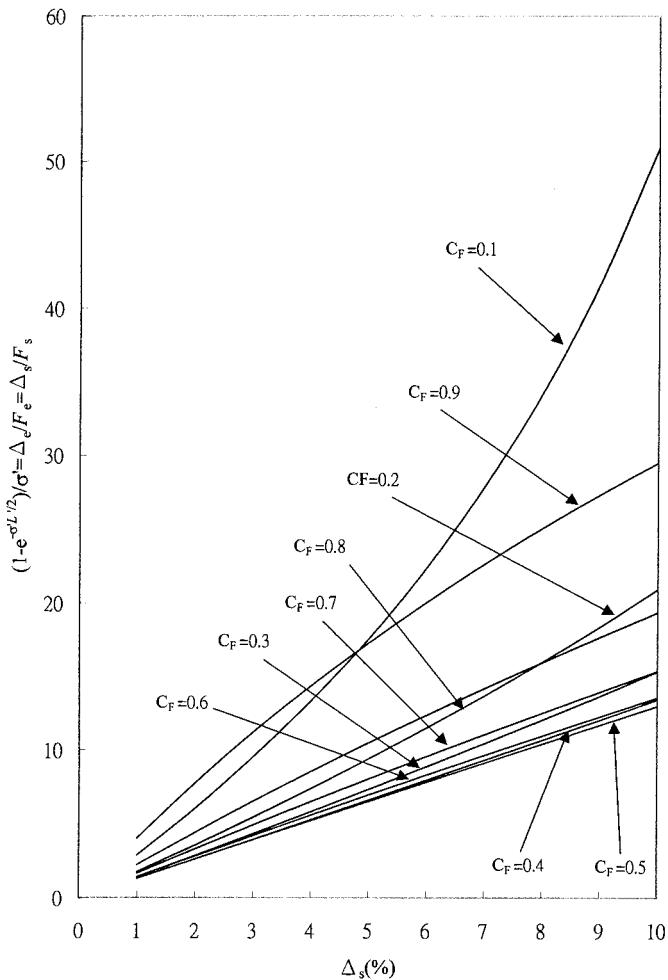


Figure 3.  $\Delta_s/F_s$  vs.  $\Delta_s$  with  $C_F$  as a parameter.

#### The Effect of L on $\Delta$

Since  $(1 - e^{-\sigma'L'/2})/\sigma'$  increases with  $L'$ , it is easy to see from Fig. 4 that the degree of separation ( $\Delta$ ) also increases with  $L'$ . Actually, this result is well known for the performance in the column devices always improves as the operating length (column length  $L$ ) increases. The tendency of the increment of  $\Delta$  with  $\sigma'$  is shown in Fig. 5.



### The Effect of $\sigma$ on $\Delta$

Since

$$\begin{aligned} \frac{\partial}{\partial \sigma'} \left[ \frac{(1 - e^{-\sigma' L'/2})}{\sigma'} \right] &= \frac{e^{-\sigma' L'/2} [(\sigma' L'/2) + 1] - 1}{\sigma'^2} \\ &= \left[ \frac{(\sigma' L'/2) + 1}{1 + (\sigma' L'/2) + (\sigma' L'/2)^2/2! + (\sigma' L'/2)^3/3! + \dots} - 1 \right] / \sigma'^2 < 0 \quad (38) \end{aligned}$$

it is known from Eq. (38) and Fig. 4 that  $\Delta$  decreases when  $\sigma'$  (or flow rate,  $\sigma$ ) increases. The tendency of the change of  $\Delta$  with  $\sigma'$  is shown in Fig. 6.

### The Effect of $C_F$ on $\Delta$

The variation of  $\Delta$  with  $C_F$  is rather complicated and hard to show mathematically. However, their relationship can be presented graphically as shown in Fig. 7. It is seen that  $\Delta$  increases when  $C_F$  goes away from both 0 and 1.0, and the optimal value for maximum  $\Delta$  is around 0.4–0.5.

## EXPERIMENTAL STUDIES

The experimental apparatus employed in this study is almost the same as that in Yeh and Ward's work (13), except that no wire spiral was inserted in the annular space. Figure 8 presents the flow diagram of a continuous-type concentric-tube thermal-diffusion column employed in the experimental work. A concentric-tube thermal-diffusion column having column length,  $L$ , of 122 cm and annular space,  $2w$ , of 0.04 cm was employed to separate heavy water from water–isotope mixtures. Since the column length is large compared to the annular spacing and the tube diameters, the end effects may be neglected. Hot and cold waters were supplied from two tanks with temperature controlled, to heat and cool the tube surfaces. Hot water was circulating through the inner tube, while cold water was circulating countercurrently with hot water through the jacket. Four copper constantan thermocouples located on the surface of each plate were used to measure the surface temperature. The mean surface temperatures of hot and cold surfaces were 47 and 14°C, respectively and, thus, the temperature difference between hot and cold surfaces and mean temperatures of the solution were 33 and 30.5°C (303.5K), respectively. The mean temperature of the solution,  $30.5 = [(47 + 14)/2]^\circ\text{C}$ , so selected is due to the reason that there is only the equilibrium constant  $K_{\text{eq}}$  of 30.5°C available, while the hot and cold surface temperatures of 47 and 14°C were chosen to achieve the mean



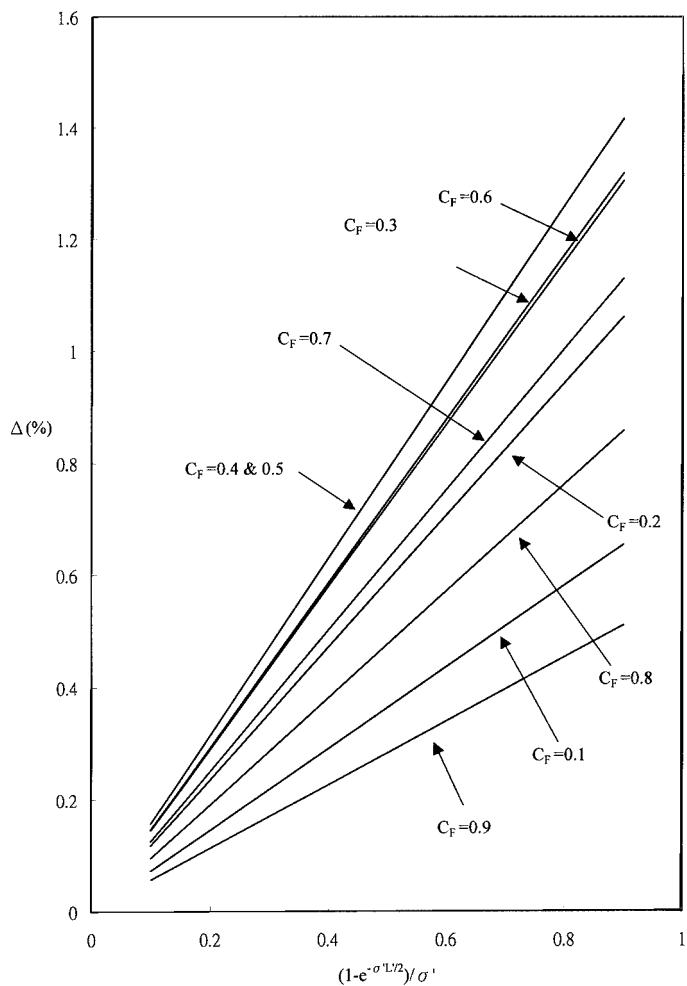


Figure 4.  $\Delta$  vs.  $(1 - e^{-\sigma' L/2})/\sigma'$  with  $C_F$  as a parameter.

temperature reaching  $30.5^\circ\text{C}$  easily. The steady feed rate,  $2\sigma$ , with temperature controlled at  $30.5^\circ\text{C}$ , was designed by a constant-head tank into the midpoint of the column due to gravitational force. The two product streams were adjusted by needle valves, withdrawn continuously with the same rate,  $\sigma$ , through cooling coils and rotameters to the collectors. The effluent samples were analyzed at  $25 \pm 0.05^\circ\text{C}$  by an automatic density meter (model DA-210, Kyoto Electronics, Tokyo, Japan). The precision of  $\pm 1.0 \times 10^{-4} \text{ g/cm}^3$  as well as  $\pm 5.0 \times 10^{-4} \text{ D}_2\text{O}$



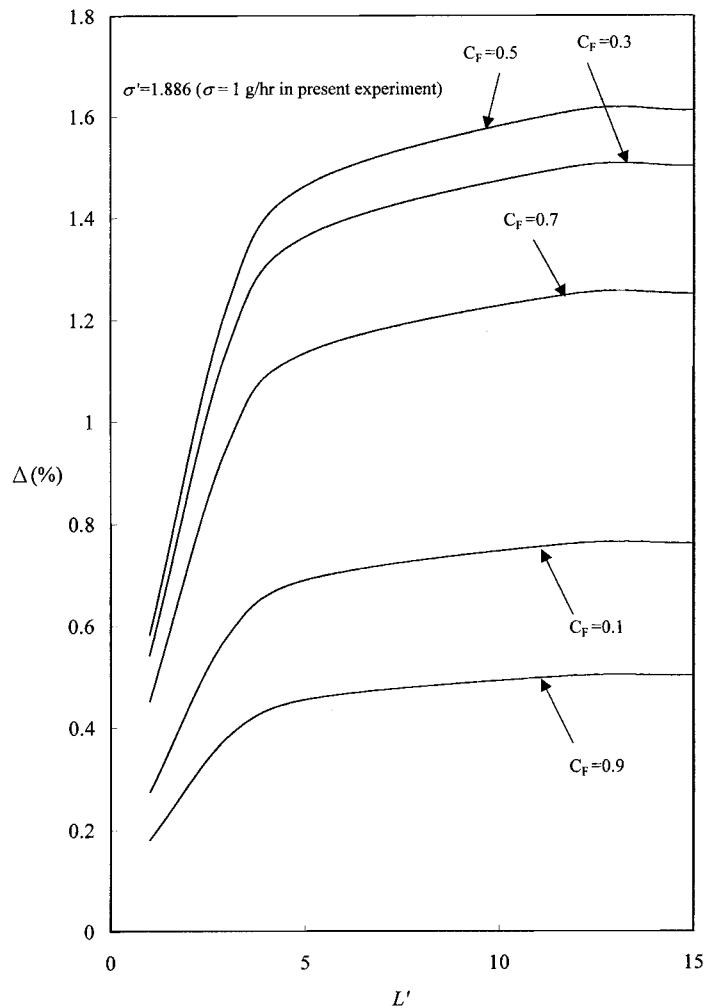


Figure 5.  $\Delta$  vs.  $L'$  with  $\sigma' = 1.886$  and  $C_F$  as parameters.

mass fraction was obtainable in this instrument. The experimental results are shown in Fig. 9 for  $C_F = 0.1$  and  $C_F = 0.381$ .

The present theory for predicting the degree of separation in a continuous-type thermal-diffusion column will be compared with the experimental results performed in this study. The theoretical predictions for the degree of separation may be obtained by substituting  $L = 122$  cm as well as the following physical



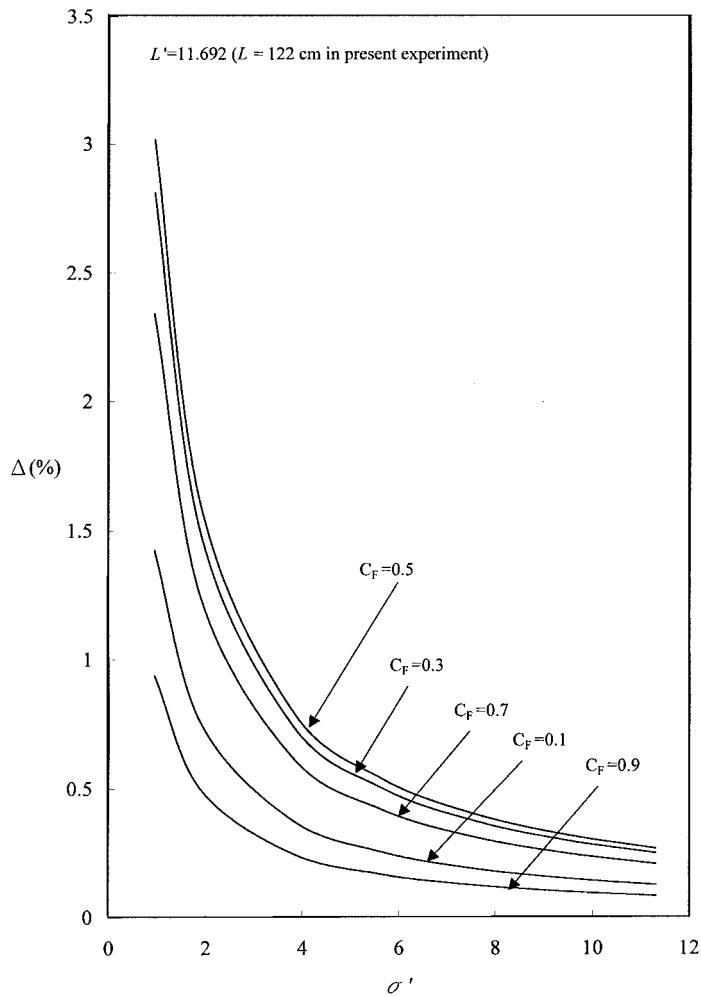


Figure 6.  $\Delta$  vs.  $\sigma'$  with  $L' = 11.692$  and  $C_F$  as parameters.

values (19) into Eq. (36):

$$H = -1.473 \times 10^{-4} \text{ g/sec} \quad (39)$$

$$K = 1.549 \times 10^{-3} \text{ g cm/sec} \quad (40)$$

$$K_{\text{eq}} = 3.793 \text{ (30.5°C)} \quad (41)$$



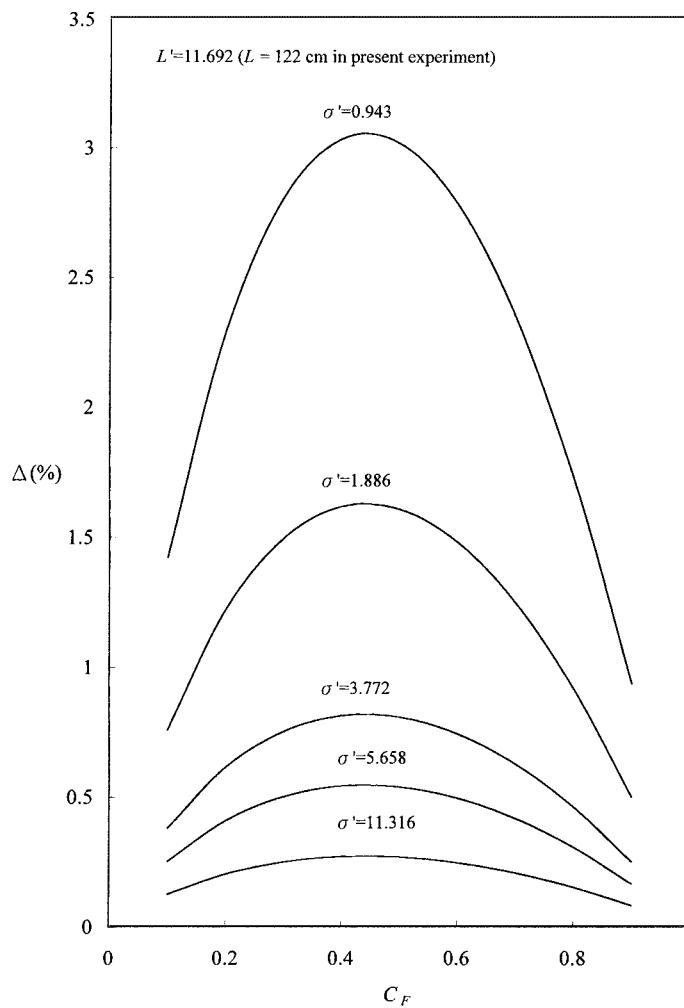


Figure 7.  $\Delta$  vs.  $C_F$  with  $L' = 11.692$  and  $\sigma'$  as parameters.

The theoretical results for the concentration of  $C_F = 0.1$  and  $C_F = 0.381$ , were also shown in Fig. 9 for comparison with the experimental data. It is found from this figure that the theoretical predictions confirm pretty well with the experimental results, especially for those obtained by Eq. (20) treating the column as two sections.



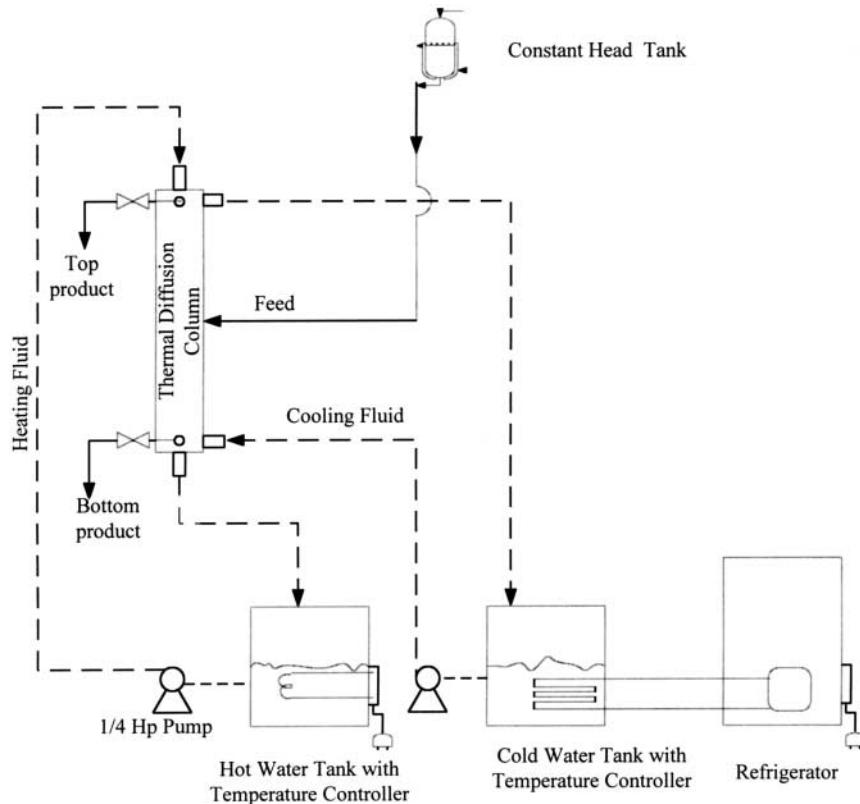
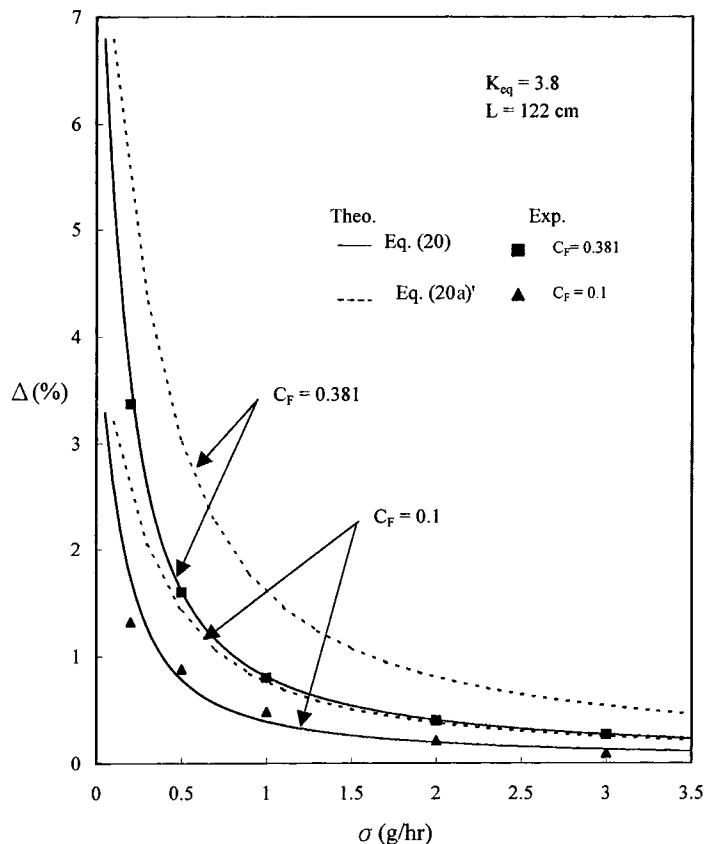


Figure 8. Flow diagram of the experimental system.

## DISCUSSION AND CONCLUSION

The theoretical and experimental studies of the separation efficiency for the enrichment of heavy water in a continuous-type thermal diffusion column, have been investigated in the present study. The theoretical values of separation for various feed concentrations and feed rates were calculated from Eqs. (20), (34), and (35), by using the given transport coefficients and equilibrium constant. Eqs. (20), (34), and (35) are also presented graphically in Figs. 2–4 for convenience. The effects of column length, flow rate, and feed concentration on the degree of separation are shown in Figs. 5–7, respectively. The feed concentration and feed rate are also the parameters of experimental runs. The experimental results thus obtained and the corresponding values of the theoretical predictions, calculated either from Eq. (20) or from Eq. (20a), are plotted in Fig. 9 for comparison.





**Figure 9.** Comparison of separation obtained from theoretical predictions and experimental results.

Performance of the experimental result is in agreement with the theoretical predictions, especially with those derived in the present study. The improvement on theoretical results of the degree of separation can be obtained by using two average concentrations calculation, instead of the average value of concentration-product term as simply taken as that at the feed position. Considering the case of  $C_F = 0.381$  and  $\sigma = 0.5$  g/hr for example, the theoretical prediction of  $\Delta$  by present study, Eq. (20), is nearly the same as that of the experimental result, while the predicted value calculated by the theory of previous work, Eq. (20a), is approximately 100% erroneous from the experimental result. Theoretical and experimental results, as shown in Fig. 9, indicate that the advantage of present results is evident.



## NOMENCLATURE

$B$	column width, $2\pi R_1$ for concentric-tube column (cm)
$C$	fraction concentration of D <sub>2</sub> O in H <sub>2</sub> O–HDO–D <sub>2</sub> O system
$D$	mass diffusivity (cm <sup>2</sup> /sec)
$C\hat{C}$	pseudo product form of concentration for D <sub>2</sub> O defined by Eq. (5)
$F_e, F_s$	appropriate values of $C\hat{C}$ in enriching section, in stripping section
$g$	gravitation acceleration (m/sec <sup>2</sup> )
$H$	transport coefficient defined by Eq. (3) (g/sec)
$K$	transport coefficient defined by Eq. (4) (g cm/sec)
$K_{eq}$	mass-fraction equilibrium constant of H <sub>2</sub> O–HDO–D <sub>2</sub> O system
$L$	length of column (cm)
$M$	defined by Eq. (28)
$N$	defined by Eq. (29)
$P$	defined by Eq. (30)
$Q$	defined by Eq. (31)
$R$	residue defined in least-squares method
$R_1$	outside radius of inner tube of concentric-tube column
$\bar{T}$	reference temperature (K)
$\Delta T$	difference in temperature of hot and cold surfaces (K)
$z$	axis parallel to the plates in the direction of convective flow
$z'$	dimensionless coordinate defined by Eq. (13)

### Greek symbols

$\alpha$	reduced thermal diffusion constant for D <sub>2</sub> O in H <sub>2</sub> O–HDO–D <sub>2</sub> O system
$\bar{\beta}_T$	( $\partial\rho/\partial T$ ) evaluated at $\bar{T}$ (g/cm <sup>3</sup> K)
$\Delta$	degree of separation, $C_B - C_T$
$\Delta_e, \Delta_s$	$C_B - C_F, C_F - C_T$
$\bar{\rho}$	mass density evaluated at $\bar{T}$ (g/cm <sup>3</sup> )
$\mu$	absolute viscosity (g cm/sec)
$\tau$	transport of D <sub>2</sub> O along $z$ -direction (g/sec)
$\sigma$	mass flow rate (g/sec)
$\sigma'$	dimensionless mass flow rate defined by Eq. (13)
$\omega$	half of plate spacing, or half of annular space (cm)

### Subscripts

$e$	for the enriching section
$B$	in the bottom product
$F$	in the feed stream
$s$	for the stripping section
$T$	in the top product



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