Limiting Velocities, Holdup, and Pressure Drop at Flooding in Packed Extraction Columns

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Criterion for flooding which was found to be reproducible in packed columns has been described. Limiting velocities, holdup, and pressure drop at flooding have been experimentally determined for binary liquid systems in a countercurrent extraction column packed with Raschig rings, Berl saddles, Lessing rings, and spheres. Generalized correlations have been suggested, with the experimental data taken into account along with, published data of other workers for the estimation of limiting velocities, holdup, and pressure drop at flooding.

The operation of liquid-liquid extraction columns ordinarily involves the countercurrent flow of two immiscible liquids in a tower usually packed with materials specially designed to give a large surface area with the minimum amount of resistance to the flow of liquids. For a given flow rate of one of the phases in a column it has long been recognized that there will be a maximum flow rate of the other phase, which if exceeded will result in accumulation of one of the phases in the column and failure of the column operation. Further, in the operation of these columns it is observed that a definite amount of the dispersed phase is always present in the interstices of the packing and the amount of the dispersed phase thus held up is dependent upon the flow rates of the two phases and other physical variables. It is also noticed that there is always a pressure drop between any two sections of the packed column and that the holdup of the dispersed phase and the pressure drop attain a maximum value at flooding. Empirical correlations relating flooding velocities in extraction columns have been suggested by a number of authors (*2, 5, 7, 13, 15*). Elgin and Browning (*9*) made a theoretical study of spray-column operation giving an explanation of the familiair square-root plots. Minard and Johnson (*14*) gave an improved theoretical analysis of spray-column operation by extending the theory proposed by Bertitti (*3*) for gas-liquid absorption towers. Dell and Pratt (*8*) have reported theoretical correlations for flooding rates in packed columns. Very little information has appeared in literature on the determination of holdup in extraction columns. Appel and Elgin (*1*) gave some data of holdup in the course of their work on the rate of extraction of benzoic acid from water with toluene. Pratt and co-workers have suggested theoretical correlations for the estimation of holdup in packed columns (*15, 16*).

It has been the aim of the present investigation to determine the three important column characteristics: flooding rates, holdup, and pressure drop at flooding for various conditions of flow, and to correlate these in terms of the known variables of flow, physical properties of the liquid systems, and the packing characteristics.

EXPERIMENTAL

Apparatus

In many of the descriptions of packed columns used for liquid-liquid extraction studies (*1, 6, 16, 17*), much attention has been focused on the design of headers and the distributor. Dell and Pratt (*8*) indicate that there is no justification for increasing the diameter of the column at the dispersed-phase entry by more than 30 to 40%. However in the present study enlarged endpieces were used to obviate any reduction in the free cross-section due to the presence of the packing support at the bottom.

The column used in the investigation was a 2-in. I.D. Pyrex-glass tube, 3 ft. in length fitted with expanded endpieces 6-

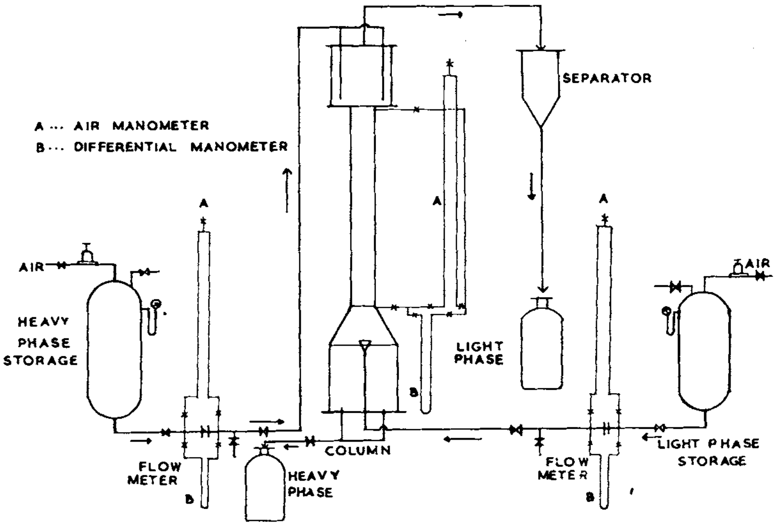

Fig. 1. Experimental set up.

in. I.D. at the bottom and 4½-in. I.D. at the top. The bottom endpiece was made of a truncated cone mounted on a cylindrical endpiece. The cylindrical portions of the endpieces were provided with a pair of sight glasses at diametrically opposite ends and also with outlet and inlet connections. Pressure tappings were made in the endpieces. The conical section at the bottom was also fitted with sight glasses for observation of the column behavior near the packing support and distributor. A distributor having 40 holes of 1/16-in. diameter was used to disperse the lighter phase liquid. The schematic flow diagram of the experimental setup is shown in Figure 1.

Packing Used

Two sizes of Raschig rings ⅜ and ¼-in.; three sizes of Lessing rings 3/16, ⅜, and ¼-in.; ¼-in. Berl saddles; and three sizes of glass balls, ½-in. 5 mm and 8 mm. were used in this investigation. The physical properties of the packings are listed in Table 1.* In the calculation of a the wetted area of the column has also been included.

Liquid Systems Studied

The different liquid systems studied were water–methyl *i*-butyl ketone, 10 and 20% glycerol with methyl *i*-butyl ketone, water-kerosene, 10 and 20% glycerol with kerosene, water-toluene, and nitrobenzene-water. The heavier phase was always made the continuous phase and the lighter the dispersed phase. Before the experiment was started each phase was saturated with respect to the other, and the physical properties of the saturated phases were determined. The values are recorded in Table 2.*

Experimental Procedure

Each time a new liquid system was used, the phases were circulated through the column until saturation was attained. Samples were taken out, and their physical properties were determined. The orifice meters were then calibrated by direct measurement.

The procedure for making a run was as follows. At first the continuous phase was let in and rate of flow maintained constant at a desired value. The dispersed phase was then let in and its rate gradually increased. After each change in the dispersed-phase flow rate some time was allowed for the column operation to reach equilibrium, indicated by the interface at the top separating section being constant and the manometer readings also remaining constant. The dispersed-phase flow rate was steadily increased until the flooding point was reached. The flooding point has been described in various ways by many workers. In the present work the approach of the flooding point could easily be judged by the rapid increase in the manometer recording of the pressure drop. It was observed that at the flooding point a thin layer of dispersed phase collected and tended to move below the packing support.

* Tabular material has been deposited as document No. 6258 with the American Documentation Institute, Photoduplication Service, Library of Congress, Washington 25, D. C., and may be obtained for $2.50 for photoprints or $1.75 for 35-mm. microfilm.

TABLE 4

System: Methyl isobutyl ketone–water
Dispersed phase: Methyl isobutyl ketone
Continuous phase: Water

Packing: ⅜-in. Raschig rings.
$a = 159.3$ $e = 0.616$ $a/e^3 = 676$
Temperature: 28°C.

Expt.	V_c	V_d	x	Δp	$\phi_1\phi_2 \cdot 10$	$\frac{ae\gamma}{\rho_c V_c^2}$	$\frac{\Delta p}{\Delta\rho/\rho_c}$	$\frac{V_d^2 a}{ge^3}\frac{\rho_d}{\Delta\rho}\cdot 10^4$
136	15.52	48.98	0.590	0.131	5.08	1655.0	0.664	158.0
140	30.06	35.08	0.540	0.119	5.42	440.0	0.605	81.0
143	40.83	30.9	0.473	0.102	5.77	238.5	0.518	62.7
147	61.38	22.18	0.455	0.109	6.17	128.2	0.551	32.4

System: Nitrobenzene-water
Dispersed phase: Water
Continuous phase: Nitrobenzene

Packing: 8-mm. glass beads
$a = 154.1$ $c = 0.463$ $a/e^3 = 1{,}552$
Temperature: 28°C.

Expt.	V_c	V_d	x	Δp	$\phi_1\phi_2 \cdot 10$	$\frac{ae\gamma}{\rho_c V_c^2}$	$\frac{\Delta p}{\Delta\rho/\rho_c}$	$\frac{V_d^2 a}{ge^3}\frac{\rho_d}{\Delta\rho}\cdot 10^4$
232	13.25	58.80	0.644	0.136	6.75	2313.0	0.821	650.0
234	20.70	43.80	0.533	0.105	6.80	954.0	0.627	361.0
236	32.20	33.30	0.441	0.115	7.10	393.0	0.698	207.5
239	50.00	18.65	0.388	0.082	7.26	164.0	0.496	65.2

System: Kerosene–water
Dispersed phase: Kerosene
Continuous phase: Water

Packing: ¼-in. Berl saddles:
$a = 227.2$ $e = 0.649$ $a/e^3 = 831.4$
Temperature: 27°C.

Expt.	V_c	V_d	x	Δp	$\phi_1\phi_2 \cdot 10$	$\frac{ae\gamma}{\rho_c V_c^2}$	$\frac{\Delta p}{\Delta\rho/\rho_c}$	$\frac{V_d^2 a}{ge^3}\frac{\rho_d}{\Delta\rho}\cdot 10^4$
328	25.81	41.3	0.179		5.54	2065	0.814	119.6
330	37.4	29.31	0.175		5.66	983	0.794	60.3
332	44.14	23.12	0.175		5.72	707	0.694	37.45
335	55.05	15.52	0.120		5.76	453	0.545	16.93

This was found to be exactly reproducible. The first appearance of the dispersed phase below the packing support was therefore taken as the criterion for flooding. Simultaneously the readings of all manometers were taken, and all flow valves were closed. It was found desirable to close the dispersed-phase supply valve first so that no dispersed phase was allowed to enter the column after the flooding point. As soon as the flow of the liquids was stopped to the column, the interface at the top descended, owing to the accumulation of the dispersed phase held inside the column. When no more movement of phases inside the column was observed, the continuous-phase supply was turned on gradually, and the discontinuous-phase liquid flowing out was collected in a weighed beaker until the interface regained its original level and the weight of the dispersed phase collected was determined. This was then used to determine percentage or fractional holdup of the dispersed phase in the column.

Column Performance

In the case of all systems with water as the continuous phase the dispersed phase could be seen flowing upward through the interstices of the packing in well-defined droplets of almost spherical shape. Considerable amount of channeling was observed particularly with larger sizes of packing. At higher dispersed-phase flow rates there was a tendency for the droplets to become bigger and to cling to packing interstices, especially with smaller packings. Holdup and pressure drop were found to increase gradually with increase in dispersed-phase flow, but close to flooding region holdup and pressure drop increased rapidly, attaining a maximum at flooding. Very near the flooding point much coalescence of the droplets particularly at the base of the column was observed.

Results

The experimental data on limiting flow rates, holdup of dispersed phase, and pressure drop at flooding in 2-in. I.D. column for a few runs are given in Table 3.* Table 4 gives sample data.*

CORRELATION OF RESULTS

Dell and Pratt (8) had derived the following relationship for flooding in packed liquid-liquid extraction columns:

* See footnote in column 1.

$$\left[1 + 0.835 \left(\frac{\rho_d}{\rho_c} \right)^{1/4} \left(\frac{V_d}{V_c} \right)^{1/2} \right] = C_2 \left[\left(\frac{V_c^2 a}{g e^3} \right) \left(\frac{\rho_c}{\Delta \rho} \right) \right]^{-1/4} \quad (1)$$

In order to take into account the effect of interfacial tension, a term γ was included within the bracket on the right-hand side of the correlation.

The above correlation was tested with published data (*7, 8*) and the data collected in this investigation. It was found that the plot of

$$\left[1 + 0.835 \left(\frac{\rho_d}{\rho_c} \right)^{1/4} \left(\frac{V_d}{V_c} \right)^{1/2} \right] \text{ vs. } \left[\left(\frac{V_c^2 a}{g e^3} \right) \left(\frac{\rho_c}{\Delta \rho} \right) \right]$$

did not give straight lines properly placed when γ was taken as the parameter. Likewise the plot of

$$\left[1 + 0.835 \left(\frac{\rho_d}{\rho_c} \right)^{1/4} \left(\frac{V_d}{V_c} \right)^{1/2} \right] \text{ vs. } \left(\left[\frac{V_c^2 a}{g e^3} \right) \left(\frac{\rho_c}{\Delta \rho} \right) \gamma \right]^{1/4}$$

did not give properly oriented straight lines when the packing size was the parameter. This observation has also been made by Ghosal and Dutt (*12*).

In the light of these results it was necessary to modify the equation to a generalized correlation. An attempt was made to express C_2 as a function of such dimensionless groups, for example $(V_c \rho_c)/(a u_c)$, $\gamma/(V_c u_c)$, and $(\gamma \rho_c)/(u^2_c a)$, but no satisfactory correlation was obtained. A resort was then made to the application of dimensional analysis.

The expression

$$\left[1 + 0.835 \left(\frac{V_d}{V_c} \right)^{1/2} \left(\frac{\rho_d}{\rho_c} \right)^{1/4} \right]$$

denoted by ϕ_1 is dimensionless and may be assumed to vary with V_c, a, ρ_c, density difference, and γ. Thus

$$\phi_1 = f(V_c, a, \rho_c, \Delta\rho, g, \gamma) \quad (2)$$

It can be shown that the following equation results from dimensional analysis:

$$\phi_1 = C \left[\left(\frac{\rho_c}{\Delta \rho} \right)^l \left(\frac{V_c^2 a}{g} \right)^m \left(\frac{a \gamma}{V_c^2 \rho_c} \right)^n \right] \quad (3)$$

The actual velocity of the continuous phase through the interstices of the packing is V_c/e and the equivalent diameter e/a. The group $(V_c^2 a)/g$ will then become $(V_c^2 a)/(g e^3)$, and the group $(a \gamma)/(V_c^2 \rho_c)$ becomes $(ae\gamma)/(\rho_c V_c^2)$. When one substitutes these in the correlation, it becomes

$$\left[1 + 0.835 \left(\frac{\rho_d}{\rho_c} \right)^{1/4} \left(\frac{V_d}{V_c} \right)^{1/2} \right] = C \left[\left(\frac{V_c^2 a}{g e^3} \right)^m \left(\frac{\rho_c}{\Delta \rho} \right)^l \left(\frac{ae\gamma}{\rho_c V_c^2} \right)^n \right] \quad (4)$$

With the value of l and m taken as —λ ¼ as in Pratt's correlation, a study was made to determine the effect of the modified Weber group $(ae\gamma)/(\rho_c V_c^2)$ by plotting

$$\left[1 + 0.835 \left(\frac{\rho_d}{\rho_c} \right)^{1/4} \left(\frac{V_d}{V_c} \right)^{1/2} \right] \left[\left(\frac{V_c^2 a}{g e^3} \right) \left(\frac{\rho_c}{\Delta \rho} \right) \right]^{1/4} \text{ vs. } \frac{ae\gamma}{\rho_c V_c^2}$$

on logarithmic coordinates.

The data obtained from more than 250 runs of the present investigation along with the published data of other authors (*4, 5, 8, 16*) can be represented by the following correlation:

$$\left[1 + 0.835 \left(\frac{\rho_d}{\rho_c} \right)^{1/4} \left(\frac{V_d}{V_c} \right)^{1/2} \right] \left[\left(\frac{V_c^2 a}{g e^3} \right) \left(\frac{\rho_c}{\Delta \rho} \right) \right]^{1/4} = C \left[\frac{ae\gamma}{\rho_c V_c^2} \right]^n$$

The values of c and n as obtained by the method of least squares for the various types of packings are given below:

Type of packing	c	n
Raschig rings	0.894	—0.078
Berl saddles	0.882	—0.052
Lessing rings	0.853	—0.046
Spheres	0.839	—0.029

It is obvious that though the magnitude of n is not large, n varies significantly with the packing type. If the respective values of c and n are used in the flooding correlation for different packings, the average deviation of all points from those predicted by the correlation for Raschig rings, Berl saddles, Lessing rings, and spheres is 7.6, 8.8, 3.7, and 5.0% respectively. However if all the data of the present investigation along with data of other workers are subjected to statistical analysis, with all the types and sizes of packing, different tower diameters, and different liquid systems taken into account, the values of c and n are found to be 0.812 and 0.048 respectively. With these values of c and n the average deviation of all the points is 12.6%, and maximum deviations are +32 and —29% from those predicted by the correlation. It may be observed that introduction of the modified Weber Group $(ae\gamma)/(\rho_c V_c^2)$ brings together data for various liquid systems, different types and shapes of packings, and tower diameters ranging from 2 to 22-in. under one generalized correlation. It may be observed here that the percentage of average deviation of data is greater for the generalized correlation, and hence it may be preferable to use the values of c and n as obtained for each packing for estimation of limiting velocities wherever greater accuracy is desired for design purposes.

The correlation for flooding is shown in Figure 2*, where the data of Crawford and Wilke for a 12-in. column, data of Dell and Pratt for 3- and 6-in. columns, and data of the authors for 12-in. column have been plotted for Raschig rings. It may be mentioned that modified Weber group ranges from a value of 30 to 15,000. In Figure 3* the published data obtained by Breckenfeld and Wilke (*5*); Blanding and Elgin (*4*); and Row, Koffolt, and Withrow (*16*) are compared with the results of the present investigation. Figure 4* shows a similar correlation for Berl saddles, where the data of Dell and Pratt for 3- and 6-in. diam. column have been presented. The data of Dell and Pratt and those of the authors for Lessing rings are plotted in Figure 5*. The correlation for the spheres, including the data of Blanding and Elgin (*4*) is presented in Figure 6*. In Figures 2 through 6, $\phi_1 \phi_2$ is plotted vs. the modified Weber group.

HOLDUP OF DISPERSED PHASE AT FLOODING

Gayler and Pratt (*10*) have derived the following theoretical correlation for estimation of holdup at flooding:

$$x_f = C' \left[\left(\frac{V_d^2 a}{g e^3} \right) \left(\frac{\rho_d}{\Delta \rho} \right) \right]^{1/4} \quad (6)$$

Figure 7* shows a plot of holdup x_f at flooding vs. the group $[(V_d^2 a)/(g e^3)](\rho_d/\Delta\rho)$ on logarithmic coordinates for the various systems and packing under investigation. This correlation may be represented by the following equation derived by mathematical analysis of all the data:

$$x_f = 0.753 \left[\left(\frac{V_d^2 a}{g e^3} \right) \left(\frac{\rho_d}{\Delta \rho} \right) \right]^{0.11} \quad (7)$$

The average deviation of all the points is 15.7%. The maximum deviations are +36 and —35%. As this equation brings under one generalized correlation all data for various systems and for shapes and sizes of various packings, the correlation may be regarded as satisfactory for design purposes. It

* See footnote on page 356.

may be noted here that the exponent 0.11 is the same as that obtained by Gayler and Pratt (*10*), but they obtained a value of 0.62 for the constant C'. Whereas the data reported here represent values taken exactly at flooding, Gayler and Pratt prepared their plot by extrapolation.

PRESSURE DROP AT FLOODING

Dell and Pratt (*8*) in their work have indicated that when the column is operating close to flooding, Bernoulli's theorem can be applied separately to the continuous- and discontinuous-phase channels in the column. In the case of the continuous phase the pressures P_1 and P_2 at two points separated by an arbitrary distance h are related as follows:

$$P_2 = P_1 + \rho_c h - \Delta P_c \qquad (8)$$

Similarly for the dispersed phase assumed to be flowing upwards

$$P_1 = P_2 - \rho_d h - \Delta P_d \qquad (9)$$

where ΔP_c and ΔP_d are due to flow of the continuous and discontinuous phase.

If Equation (8) is multiplied by $(1-x)$ and (9) by (x), one gets

$$(1-x)P_2 = (1-x)P_1 + \rho_c h(1-x) - (1-x)\Delta P_c \qquad (10)$$

and

$$xP_1 = xP_2 - x\rho_d h - x\Delta P_d \qquad (11)$$

Subtracting (11) from (10) one gets

$$P_2 = P_1 + \rho_c h(1-x) + \rho_d hx + x\Delta P_d - (1-x)\Delta P_c \qquad (12)$$

It may be observed that the term $[x\,\Delta P_d - (1-x)\Delta P_c]$ measures the net thrust. If at flooding the net thrust is assumed to be negligible (inasmuch as the dispersed phase is not allowed to go up in the column), Equation (12) simplifies to

$$P_1 - P_2 + \rho_c h = hx\Delta\rho$$

or

$$\Delta p' = \frac{P_1 - P_2 + \rho_c h}{\rho_c} = hx\,\Delta\rho/\rho_c \qquad (13)$$

The expression on the left side represents the actual pressure drop measured in terms of head of the continuous phase for the height. If Δp represents head loss per foot length of the column for the continuous phase, the following relationship

$$\frac{\Delta p'/h}{\Delta\rho/\rho_c} = \frac{\Delta p}{\Delta\rho/\rho_c} = x \qquad (14)$$

suggests that plot of $\Delta p/(\Delta\rho/\rho_c)$ vs. holdup should give a straight line. However it may be observed that at flooding there is always some net thrust present, and the values of holdup and pressure drop at flooding do not change appreciably. It is therefore not desirable to have a plot of two quantities which do not change appreciably for obtaining a generalized correlation. Since it has been shown that x is a function of the group $[(V_d^2 a)/(ge^3)](\rho_d/\Delta\rho)$, $\Delta p/(\Delta\rho/\rho_c)$ may be plotted against this group on logarithmic coordinates. It may be noticed that the range of the experimental values of the group $[(V_d^2 a)/ge^3](\rho_d/\Delta\rho)$ is quite large, from about 5 to 1,000.

Such plots for each type of packings give fairly good correlations. Figure 8* shows the composite plot for all the packings and systems studied. The over-all relationship obtained for the combined correlation for all packings and systems by mathematical analysis of the data following the method of least squares may be expressed as

$$\frac{\Delta p}{\Delta\rho/\rho_c} = 0.83\left[\left(\frac{V_d^2 a}{ge^3}\right)\left(\frac{\rho_d}{\Delta\rho}\right)\right]^{0.081} \qquad (15)$$

The average deviation of all the points is 23.2%. The maximum deviations are +55 and −58%.

Data show an appreciable amount of scatter due partly to representing data of many liquid system and packings of different shapes and sizes by one generalized correlation and also due to a larger experimental error involved in the accurate measurement of pressure drop at flooding in liquid-liquid extraction columns. In view of the fact that the exponent on the group $[(V_d^2 a)/ge^3](\rho_d/\Delta\rho)$ is small, one may assume that the pressure drop at flooding is nearly independent of the dispersed-phase velocity, a condition reminiscent of constant pressure drop at flooding in gas-liquid absorption towers (*18*).

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NOTATION

- a = superficial area of packing, sq.ft./cu.ft.
- C, C', C_2 = constants
- f = functional operator
- g = gravitational acceleration
- h = height of the column
- l, m, n = constants
- P = pressure, lb./sq.ft.
- Δp = actual pressure drop meassured at ft. of continuous phase/ft. height of packing
- ΔP = differential pressure lb./sq.ft.
- V = superficial velocity of the phase (empty tower), ft. hr.
- x = fractional holdup of dispersed phase in column free space

Greek Letters

- ρ = density of phase, lb./cu.ft.
- $\Delta\rho$ = density difference, lb./cu.ft.
- γ = interfacial tension, dynes/cm. (lb./hr.^{2} in modified Weber group)
- ϕ = functional operator
- ϕ_1 = $\left[1 + 0.835\left(\frac{\rho_d}{\rho_c}\right)^{1/4}\left(\frac{V_d}{V_c}\right)^{1/2}\right]$ dimensionless
- ϕ_2 = $\left[\left(\frac{V_c^2 a}{ge^3}\right)\left(\frac{\rho_c}{\Delta\rho}\right)\right]$ dimensionless
- e = fractional voids, dimensionless
- u = viscosity of phase, lb./(hr.)(ft.)

Subscripts

- c = continuous phase
- d = dispersed phase
- f = flooding state

* See footnote on page 356.

LITERATURE CITED

1. Appel, F. J., and J. C. Elgin, *Ind. Eng. Chem.*, **29**, 451 (1927).
2. Ballard, J. H., and E. L. Piret, *ibid.*, **42**, 1088 (1950).
3. Bertetti, J. W., *Trans. Am. Inst. Chem. Engrs.*, **38**, 1023 (1942).
4. Blanding, F. H., and J. C. Elgin, *ibid.*, **33**, 305 (1942).
5. Breckenfeld, R. R., and C. R. Wilke, *Chem. Eng. Progr.*, **46**, 187 (1950).
6. Colburn, A. P., *Trans. Am. Inst. Chem. Engrs.*, **35**, 211 (1939).
7. Crawford, J. W., and C. R. Wilke, *Chem. Eng. Progr.*, **47**, 423 (1951).
8. Dell, F. R., and H. R. C. Pratt, *Trans. Inst. Chem. Engrs. (London)*, **29**, 89 (1951).
9. Elgin, J. C., and F. M. Browning, *Trans. Am. Inst. Chem. Engrs.*, **31**, 639 (1935); **32**, 105 (1936).
10. Gayler, R., and H. R. C. Pratt, *Trans. Inst. Chem. Engrs. (London)*, **29**, 110 (1951).
11. Gayler, R., N. W. Roberts, and H. R. C. Pratt, *ibid.*, **31**, 57 (1954).
12. Ghosal, S. R., and D. K. Dutt, *Trans. Ind. Inst. Chem. Engrs.*, **8**, 36 (1955-56).
13. Hoffing, E. M., and F. J. Lockhart, *Chem. Eng. Progr.*, **50**, 94 (1954).
14. Minard, G. W., and A. I. Johnson, *ibid.*, **48**, 62 (1952).
15. Rae, H. K., *ibid.*, **47**, 431 (1951).
16. Row, S. B., J. H. Koffolt, and J. R. Withrow, *Trans. Am. Inst. Chem. Engrs.*, **37**, 559 (1941).
17. Varteressian, K. A., and N. R. Fenske, *Ind. Eng. Chem.*, **28**, 928, 1153 (1936).
18. Zenz, F. A., *Chem. Eng. Progr.*, **43**, 415 (1947).

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