# Operating Characteristics of a Centrifugal Extractor

F. M. JACOBSEN and G. H. BEYER

Iowa State College, Ames, Iowa

The operating characteristics of a Podbielniak model 5,000 centrifugal extractor, having a combined-stream capacity of 450 cc./min. at 5,000 rev./min. and a rotor holdup of 529 cc., were investigated. Variables studied were density difference, rotor speed, light-liquid-out pressure, flow rates, holdup, and number of stages.

A technique for holdup determination that comprises displacing either phase in the extractor with the other phase is described and experimentally demonstrated. An equation useful in predicting flooding limits is verified. It is suggested that the effective values of the rotor dimensions in this equation be determined by the behavior of the extractor.

A relationship among number of stages, light-liquid-out pressure, and flow-rate ratio is demonstrated by extracting boric acid from isoamyl alcohol with water. With only a few runs this relationship should permit one to map in a family of curves for a particular system and thus rapidly to estimate optimum operating conditions.

A procedure is outlined for applying the methods to other systems using similar extractors. First the constants in an equation are estimated from holdup measurements in order to predict flooding limits. Then a few extraction runs are made to estimate optimum operating conditions.

The centrifugal extractor is a relatively new countercurrent liquid-liquid contacting device consisting of a series of concentric stages in which both mixing and settling are accelerated by a readily variable centrifugal-force field. The centrifugal-force field enables the extractor to handle systems of low density difference more efficiently than do conventional extractors. The low holdup and high throughput capacity of the centrifugal extractor lead to short contact times, which have proved useful in the pharmaceutical industry. Low holdup and rapid attainment of steady state operation may considerably reduce process- and feedstock-switching losses in petroleum refining. For comparable capacity the centrifugal extractor occupies a fraction of the volume required for conventional extractors. Additional stages beyond the extraction capacity of a particular unit are obtainable only by duplication of the unit.

The Podbielniak "Pup" model 5,000 laboratory extractor used in this study has been described previously (1). It is convenient for studying many systems in a relatively short time because of its low holdup, rapid attainment of steady state operation, and substantial capacity. The manufacturer claims to be able to extrapolate data obtained with this model to commercial-size units.

F. M. Jacobsen is at present with American Oil Company, Texas City, Texas, and G. H. Beyer at the University of Missouri, Columbia, Missouri. A literature review indicated few articles (1, 5) on the operating characteristics of centrifugal extractors. The first objective of this study, therefore, was to investigate these characteristics.

The literature review also led to an equation that would be useful in predicting operating limits if it could be experimentally verified. Inferences drawn from reported data (2, 3, 4) suggested a method of relating the number of stages with several operating variables. Additional objectives of this study, therefore, were to test the validity of an equation for predicting operating limits and to investigate the feasibility of relating stages and operating variables. Attainment of these objectives led to a procedure for rapidly predicting operating limits and for estimating optimum operating conditions.

#### **DESCRIPTION OF EXTRACTOR**

# **Operating Principles**

The centrifugal extractor studied had a rotor approximately 18 in. in diam. and 2 in. thick. The rotor was constructed by machining concentric annuli in two mating disks and then bolting the disks together. The annuli were connected at 180° intervals by slots, with the slots in adjacent rings rotated 90° to assure radial flow in each annulus. Figure 1 is a photograph of one of these disks. Figure 2 is a schematic diagram of the extractor, showing for the sake of clarity only five of the annuli.

Figure 2 is useful in explaining the opera-

tion of the extractor. The heavy liquid is fed axially through a rotary seal at one side of the extractor so that it enters the contacting section of the rotor near the axis. The light liquid is fed axially through a similar seal at the opposite side of the extractor and then into a small radial channel leading from the axis to the periphery of the rotor so that it enters the contacting section near the periphery. Centrifugal force causes the heavy liquid to be thrown outward, thereby displacing the light liquid inward. The heavy liquid leaves the contacting section near the periphery and is channeled radially to the axis, where it leaves through a rotary seal. The light liquid leaves the contacting section near the axis and is discharged through a similar seal.

The light-liquid-in pressure is determined by the density difference of the light-liquidin and heavy-liquid-out phases, the speed of rotation, and the rotor radius. The heavyliquid-out pressure is atmospheric; the heavy-liquid-in pressure is essentially the same as the light-liquid-out pressure, which is adjusted by a back-pressure regulator.

Two important differences between the centrifugal and conventional tower extractors should be noted (6). The first is that the settling effect of density difference is multiplied by the centrifugal force. The product of these factors is sufficient to force the two phases past one another through the small slots shown in Figures 1 and 2. This procedure permits the extractor effectively to handle systems of very low density difference. The second difference is that the liquid is either accelerated or decelerated radially and the process of acquiring or losing energy creates turbulence.

Conditions in the contacting section during operation may be thought of in terms of a "principal" interface, to describe the behavior of liquids in the contacting section as a whole, and of a series of "minor" interfaces, to describe the behavior of liquids in the individual stages of the contacting section (1). Considering the entire contacting section, the heavy liquid will predominate radially outward from the principal interface to the periphery; whereas the light liquid will predominate radially inward from the principal interface to the axis. Within each annulus there is a minor interface because enough of the dispersed phase must build up to develop sufficient head to pass through the slots.

The minor interfaces in the annuli are one source of interfacial area through which mass transfer may take place. Another, and perhaps more important, source of interfacial area is provided by the dispersing action of the slots; for example, as the heavy liquid passes outward through a slot it forms droplets which are dispersed in the continuous light-liquid layer on the outer face of the annulus wall. These droplets continue radially until they coalesce on the inner face of the next annulus wall. The lightliquid droplets moving inward through the slots and heavy-liquid layers exhibit similar behavior. The net effect is the continuous creation of fresh mass transfer area. The principle is that of repeated mixing and settling, except that the settling is accelerated by centrifugal force and that the two layers must flow past each other radially until they escape into another annulus.

The position of the principal interface between the light and heavy liquid is controlled by the light-liquid-out pressure. Thus, changing this pressure is analogous to opening and closing the outlet valve in the exit line of a tower extractor. At low light-liquid-out pressures the heavy-liquid phase predominates in the contacting section; whereas at high light-liquid-out pressures the light-liquid phase predominates. Therefore, as the light-liquid-out pressure is varied, a change may be expected in both types of mass transfer area.

# Flooding

Two types of flooding have been observed with this extractor. The first type, caused by too large a throughput, is fixed for centrifugal extractors by their design. The second type of flooding is analogous to level control in a tower extractor. It is caused by movement of the principal interface to either of the effluent stream take-offs. For a particular centrifugal extractor this type of flooding depends primarily on rotor-speed and phasedensity difference. At a sufficiently low light-liquid-out pressure the rotor will be essentially full of heavy liquid, and so the principal interface will spill into the lightliquid-out line. This condition has been defined as flooding in the light liquid out. Likewise, at a sufficiently high lightliquid-out pressure, the rotor will be essentially full of light liquid, and so the principal interface will spill into the heavy-liquid-out line. This condition has been defined as flooding in the heavy liquid out. The useful operating range of the extractor for a particular system may

be described by light-liquid-out pressures between flooding limits, because at and outside these limits stable operation is difficult.

#### Holdup

The liquid volume or holdup in this extractor is contained in two functional sections. One is the rotor or contacting section; the other comprises the seals and feed and product lines. If  $V_E$  is the total extractor volume and  $V_C$  the rotor volume, then the difference

$$V_E - V_C = V_S \tag{1}$$

will be called the seal volume. The subscript L denotes the light-liquid phase and the subscript H the heavy-liquid phase. The following equations are then implied by Equation (1):

$$V_{E} = V_{H} + V_{L} \tag{2}$$

$$V_c = V_{CH} + V_{CL} \qquad (3)$$

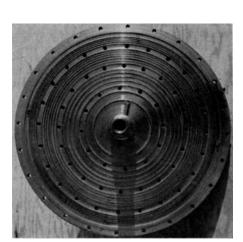


Fig. 1. Extractor disk.

$$V_s = V_{sH} + V_{sL} \tag{4}$$

$$V_L = V_{CL} + V_{SL} \tag{5}$$

$$V_H = V_{CH} + V_{SH} \tag{6}$$

In these equations  $V_E$ ,  $V_C$ ,  $V_S$ ,  $V_{SH}$ , and  $V_{SL}$  are constants; whereas  $V_L$ ,  $V_H$ ,  $V_{CL}$ , and  $V_{CH}$  are variables dependent upon experimental conditions.

Previous workers (1, 5) reported the extractor holdup as  $V_E = 600$  cc., and the contacting section holdup as  $V_C = 65$  cc. Values experimentally observed during this investigation were  $V_E = 638$  and  $V_C = 529$  cc.

#### **EXPERIMENTAL INVESTIGATION**

Morgenthaler *et al.* (5) derived the following equation for light-liquid pressure drop across the extractor:

$$P_{LI} - P_{LO} = \Delta \rho w^2 V_{CH} / 2\pi b g_c \quad (7)$$

Barson and Beyer (1) derived the following equation for light-liquid-in pressure:

$$P_{LI} = \Delta \rho w^2 r^2 / 2g_c \tag{8}$$

and evaluated r as 7.71 in.

Eliminating  $P_{LI}$  from Equations (7) and (8) and solving for  $P_{LO}$ , one obtains

$$P_{LO} = \Delta \rho w^2 (r^2 - V_{CH}/\pi b) / 2g_c \quad (9)$$

Equation (9) has two important uses. The first is to predict operating limits for any system. The second is to estimate optimum operating conditions in conjunction with a relationship between rotor inventory and number of stages that will be demonstrated below.

Before the validity of Equation (9) is tested, it seemed wise to check both Equation (8) and the effects of flow rates on the light-liquid-in and light-liquid-out pressures. Recent data (4), such as those shown in Figure 3, indicate that Equation (8) is an excellent approximation. It has

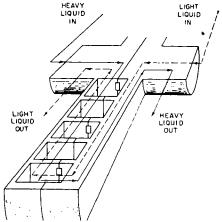


Fig. 2. Schematic diagram of flow through extractor.

also been shown (4) that the change in light-liquid-out pressure at either flooding limit with flow rates is small compared with the light-liquid-out pressure range between flooding limits. Data at incipient flooding in the light liquid out are shown in Figure 4. The light-liquid-out pressure between flooding limits is of course operationally independent of flow rates because one may choose it and the heavy- and light-liquid flow rates independently of one another.

# **Holdup Studies**

If the two extractor parameters, r and b, in Equation (9) are known, that equation may be used to predict operating limits. Thus, specification of  $\Delta \rho$  and w fixes  $P_{LO}$  at both flooding limits because  $V_{CH}$  is constant at either flooding limit. At flooding in the light liquid out,  $V_{CH}/V_C$  will be essentially unity, and at flooding in the heavy liquid out,  $V_{CH}/V_C$  will be

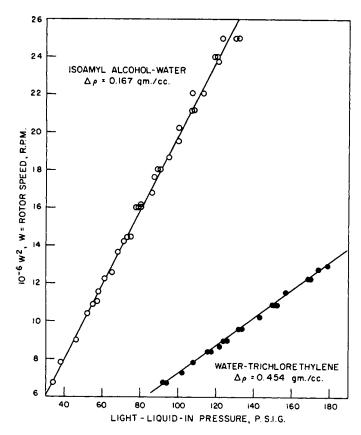


Fig. 3. Relation among rotor speed, density difference, and light-liquid-in pressure.

essentially zero. This section outlines methods by which the constants in Equation (9) were evaluated. The application of these methods to other centrifugal extractors is discussed in a later section.

The application of Equation (9) is not limited by a varying rotor width if b is defined as the effective rotor width and evaluated numerically from the behavior of the extractor rather than from its dimensions. Defining b in this manner should permit the application of Equation (9) independently of the internal arrangement of the contacting sections of similar extractors.

To test the validity of Equation (9) it was necessary to make direct holdup measurements. Previous workers (1, 5) attempted to measure holdup by valving off both the feed and effluent streams, stopping the rotor, and then draining it. The limitation of this method is evident from Figures 1 and 2, which indicate the tortuous path either liquid has to follow in leaving the rotor. A more promising approach appeared to be displacing either phase from the extractor with the other phase. Equation (2) could then be employed to check the consistency of the results since the sum of  $V_L$  and  $V_H$  must be constant at all operating conditions.

An aspect of Equation (9) useful in demonstrating its validity and evaluating the constants in Equations (1) through (6) is seen by solving Equation (9) for  $V_{CH}$ :

$$V_{CH} = \pi b (r^2 - 2g_c P_{LO} / \Delta \rho w^2)$$
 (10)

and substituting into Equation (3) to obtain

$$V_{CL} = (V_C - \pi b r^2) + 2\pi b g_c P_{LO} / \Delta \rho w^2 \quad (11)$$

Since r and b are defined as effective rotor dimensions and  $\pi b r^2$  is therefore rotor holdup  $V_C$ , the first two terms on the right-hand side of Equation (11) vanish, giving

$$V_{GL} = 2\pi b g_c P_{LO} / \Delta \rho w^2 \quad (11a)$$

Substituting Equations (10) and (11a) into Equations (6) and (5), respectively, one obtains

$$V_{H} = (\pi b r^{2} + V_{SH})$$
  
-  $2\pi b g_{c} P_{LO} / \Delta \rho w^{2}$  (12)

$$V_L = V_{SL} + 2\pi b g_c P_{LO} / \Delta \rho w^2 \qquad (13)$$

Equations (12) and (13) are of the form

$$V_X = a_X \pm c_3 P_{LO} \tag{14}$$

where the  $a_X$ 's are constants and  $c_3$  is constant at constant  $\Delta \rho$  and w. Equations (12) and (13) predict that the volume of either phase in the extractor should vary linearly with  $P_{LO}$ .

Before the data used to verify these equations is discussed, however, two other points should be considered. The first point is concerned with the rotor inven-

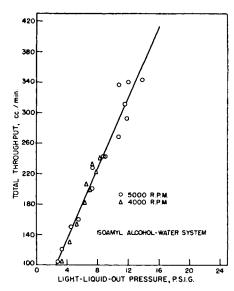


Fig. 4. Relation between total throughout and light-liquid-out pressure at incipient flooding in the light liquid out.

tory at either flooding limit. The second is concerned with the effect of rotor construction on holdup-determination technique.

It was observed earlier that when the principal interface spills into either effluent stream the rotor will be essentially full of one phase. At flooding in the light liquid out, for example, when the rotor is essentially full of heavy liquid, Equation (13) indicates that if the rotor were completely full of heavy liquid,  $V_L$  would be equal to  $V_{SL}$ , and  $P_{LO}$  would be zero. Experimentally, it was observed that the light-liquid-out pressure was not zero at flooding in the light liquid out. The amount of light liquid in the rotor under these conditions was therefore estimated to be the difference between  $V_L$  at flooding in the light liquid out and  $V_{SL}$ . The light-liquid seal volume  $V_{SL}$  was estimated from Equation (13) when  $P_{LO}$  was zero. Evaluating  $V_{SL}$  in this manner assumes that the rotor is full of heavy liquid when the light-liquid-out pressure is zero. Likewise, the heavy-liquid seal volume  $V_{SH}$  was estimated from Equation (12) on the assumption that at flooding in the heavy liquid out  $V_H =$ 

V<sub>SH</sub>.

The second point is concerned with the effect of rotor construction on holdup-determination technique. This point will be discussed with reference first to heavy-liquid and then to light-liquid holdup.

Tabulated heavy-liquid holdups are actually the sum of two heavy-liquid volumes. The first of these volumes was displaced while the rotor was spinning. The extractor was permitted to attain stable operation at the desired values of the operating variables:  $w, \Delta \rho$ , and flow rates. Attainment of stable operation was judged by the constancy of these variables for a 10-min. interval. The heavy-liquid feed was then turned off and light.

Table 1. Variation of Holdup with Light-liquid-out Pressure for Light-liquid-out Pressures Less Than 100 lb./sq. in.

Isoamyl alcohol-water system Rotor speed = 4,950 to 5,000 rev./min. H flow rate = 90 to 105 cc./min. L flow rate = 125 to 150 cc./min. Volumes = cc. at 25° C.  $P_{LO}$  = lb./sq. in. gauge

Light-liquid holdup

$\operatorname{Run}H$	$P_{LO}$	$V_L$	$\operatorname{Run} H$	$P_{LO}$	$V_L$
56	9.3	123.55	84	59	392.16
57	8.7	121.01	86	59	395.59
<b>5</b> 8	8.6	127.91	79	77.7	479.03
92	23.8	203.10	81	79	473.30
93	23.8	210.65	75	98	573.67
68	39	287.41	78	98	573.76
69	39	283.67			

## Heavy-liquid holdup

Run $H$	$P_{LO}$	$V_H$	$\operatorname{Run} H$	$P_{LO}$	$V_H$
60	9	622.60	39	77	266.12
61	9	617.93	40	79	265.51
62	9	622.18	41	80	268.73
63	9	622.05	42	76	268.74
94	23.8	541.29	43	77	272.73
95	23.8	537.70	44	79	272.72
70	40	466.91	77	98	178.60
67	41	460.39	88	98	174.27
85	59	353.49	89	98	175.77
87	59	352.50			

liquid was introduced into both the heavy- and light-liquid-in lines. Closing a valve in the light-liquid-out line forced the total input to the extractor to leave through the heavy-liquid-out line, facilitating displacement of the heavy liquid. The rotor speed was not reduced while the heavy liquid was being displaced. The displaced heavy liquid was collected, separated from the light liquid, and weighed to determine its volume.

The second of these heavy-liquid volumes was obtained by stopping the rotor after no more heavy liquid was being displaced while it was spinning and by flushing the extractor with light liquid while the rotor was slowing down and after it had stopped. The origin of this volume of heavy liquid may be seen by

observing in Figure 1 that centrifugal force will prevent displacement of the heavy liquid normally contained in the rotor at a radial distance from the center of the rotor greater than the radial distance of the heavy-liquid take-off. The average volume of heavy-liquid in the rotor that could not be displaced by a less dense liquid while the rotor was spinning was approximately 25 cc. This volume is the heavy-liquid rotor inventory at flooding in the heavy liquid out,

$$(V_{CH})_{FH} = 25 \text{ cc.}$$
 (15)

Similar considerations indicated that the rotor might contain light liquid that could not be displaced by heavy liquid while the rotor was spinning. Attempts to

Table 2. Variation of Holdup with Light-liquid-out Pressure for Light-liquid-out Pressures Greater Than 96 lb./sq. in.

Isoamyl alcohol-water system
Rotor speed = 4,950 to 5,000 rev./min.

H flow rate = 90 to 105 cc./min.

L flow rate = 125 to 150 cc./min.

Volumes = cc. at 25° C.

 $P_{LO} = \text{lb./sq. in. gauge}$ 

55.	Light-liqui	d holdup			
$P_{LO}$	$V_L$	Run	$P_{LO}$	$V_L$	
98	573.67	G6	113	590.61	
98	573.76	G8	118	618.86	
104 580.70		H53	120	607.23	
111	591.21	H55	122	615.03	
113	590.02				
	Heavy-liqu	id holdup			
$P_{LO}$	$V_H$	Run	$P_{LO}$	$V_H$	
98	178.60	H50	114	154.02	
98	174.27	H49	116	147.66	
98	175.77	H51	118	150.44	
	98 98 104 111 113 P <sub>Lo</sub> 98 98	$\begin{array}{cccc} & & & \text{Light-liqui} \\ P_{Lo} & V_L \\ 98 & 573.67 \\ 98 & 573.76 \\ 104 & 580.70 \\ 111 & 591.21 \\ 113 & 590.02 \\ & & & \\ & & \\ & & \\ & & \\ P_{Lo} & V_H \\ 98 & 178.60 \\ 98 & 174.27 \\ \end{array}$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	

displace additional light liquid from the rotor while it was slowing down and after it had stopped, after the spinning rotor had been thoroughly flushed, were unsuccessful. This indicated that substantially all light liquid may be displaced from a spinning rotor by a denser liquid.

Selected data (4) indicating the volume by either phase in the extractor as a function of  $P_{LO}$  are given in Table 1 for  $P_{LO} < 100$  lb./sq. in. gauge and in Table 2 for  $P_{LO} > 96$  lb./sq. in. gauge. The following equations were obtained from these data by least squares:

$$V_H = 663.14 - 5.03 P_{LO}$$
 (12a)

$$V_L = 84.38 + 5.04 P_{LO}$$
 (13a)

for light-liquid-out pressures less than 96 lb./sq. in. gauge and

$$V_H = 314.53 - 1.41P_{LO}$$
 (12b)

$$V_L = 417.83 + 1.57 P_{LO}$$
 (13b)

for light-liquid-out pressures greater than 96 lb./sq. in. gauge. The foregoing equations and the data from which they were obtained are plotted in Figure 5.

The reason two sets of equations are required for the light-liquid-out pressure is that the constant b is common to both the slope and intercept of Equation (10) and therefore of Equations (12), (12a), and (12b). The data in Figure 5 indicate that the effective rotor thickness probably changes sharply somewhere between the center and periphery, as at a horizontal heavy-liquid take-off.

By substituting Equations (12a) and (13a) and Equations (12b) and (13b) into Equation (2), one obtains, respectively,

$$V_E = 747.52 + 0.01P_{LO} \quad (2a)$$

$$V_E = 732.36 + 0.16 P_{LO} \quad (2b)$$

These equations indicate that the experimentally determined sum of the heavy-and light-liquid volumes in the extractor is sufficiently constant to warrant placing considerable confidence in the methods used to determine holdup.

A comparison of the first terms of the right-hand sides of Equations (13) and (13a) shows that

$$V_{SL} = 84.38 \text{ cc.}$$
 (16)

This value is 46 cc. lower than the value of  $V_L = 130$  cc. calculated from Equation (13a) at incipient flooding in the light liquid out, for which  $P_{LO}$  is about 9 lb./sq. in. gauge. This 46 cc. represents the light liquid in the rotor at incipient flooding in the light liquid out,

$$(V_{CL})_{FL} = 46 \text{ cc.}$$
 (17)

At flooding in the heavy liquid out,  $P_{LO} = 128$  lb./sq. in. gauge for this system at 5,000 rev./min. Under these conditions the rotor contains essentially

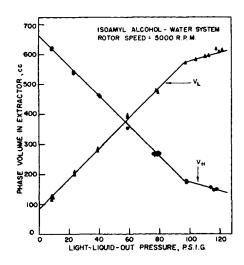


Fig. 5. Variation of rotor inventory with light-liquid-out pressure.

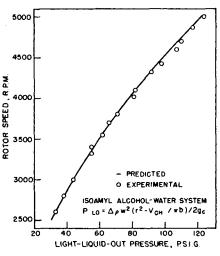
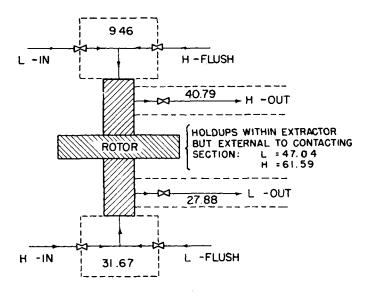


Fig. 7. Relation between rotor speed and light-liquid-out pressure at flooding in the heavy liquid out.



NOTE : ALL VOLUMES ARE IN CC

Fig. 6. Holdup of external extractor piping and internal lines and seals exclusive of contacting section.

no heavy liquid, and from Equation (12b)

$$V_H = V_{SH} = 134.05 \text{ cc.}$$
 (18)

Substituting Equations (16) and (18) into Equation (4) gives

$$V_s = 84.38 + 134.05$$
  
= 218.43 cc. (4a)

Comparing Equation (18) and the first term on the right-hand side of Equations (12) and  $(12a)_1$  one obtains

$$V_c = 663.14 - 134.05$$
  
= 529.09 cc. (19)

Substituting Equations (4a) and (19) into Equation (1) gives

$$V_E = 529.09 + 218.43$$
  
= 747.52 cc. (1a)

The constants in Equations (1) through (6) have now been evaluated.

Since that portion of  $V_S$  outside the extractor was peculiar to the particular feed- and product-piping arrangement used in this investigation, it is of interest to report the portions of  $V_S$  both inside and outside the extractor. The quantities and locations of holdup in the piping external to the extractor are outlined by the broken lines in Figure 6. The volumes were obtained by measuring the liquid required to fill the particular lines. Holdup data are summarized in Table 3.

From Equation (19),

$$V_c = \pi b r^2 = 529 \text{ cc.}$$
 (19)

and for r = 7.71 in., b = 0.173 in.

# **Prediction of Operating Limits**

With numerical values of  $V_{CH}$  at either flooding limit and b, one may predict

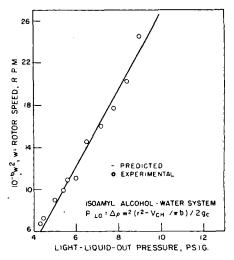


Fig. 8. Relation between rotor speed and light-liquid-out pressure at flooding in the light liquid out.

operating limits for various systems by substitution of the appropriate constants into Equation (9); for example, the rotor constants are r=7.71 in. and b=0.173 in., and the system constant is  $\Delta \rho=0.167$  g./cc. At flooding in the heavy liquid,  $V_{CH}=25$ , and Equation (9) yields

$$(P_{LO})_{FH} = (4.86 \times 10^{-6} w^2)$$

lb.-force/sq. in. (20a)

At flooding in the light liquid,  $V_{CH} = 529 - 46 = 483$  cc., and Equation (9) yields

$$(P_{L0})_{FL} = (0.447 \times 10^{-6} w^2)$$

lb.-force/sq. in. (20b)

where w is the rotor speed in revolutions per minute. These equations are plotted, in Figures 7 and 8, respectively, together with experimental data. The agreement between predicted and experimental val-

ues shown in Figures 7 and 8 indicates that Equation (9) predicts operating limits satisfactorily.

#### **EXTRACTION STUDIES**

# Proposed Relation Among Stages, $P_{LO},\,\,$ and Flow-rate Ratio

The data of Barson (2, 3) at low lightliquid-out pressures and the data of Jacobsen (4) at high light-liquid-out pressures suggested Figure 9A, which shows lines of constant light-liquid flow rate  $R_L$  at constant heavy-liquid flow rate  $R_H$  on a plot of number of stages against  $P_{LO}$ . The relationship shown is based upon the idea that  $P_{LO}$  is an indication of mass transfer area. The curves shown are members of a family of curves, each representing a different operating line slope. Other systems might show an increase in number of stages with increasing light-liquid-out pressure. One may be able to estimate the qualitative effect of increasing light-liquid-out pressure from a knowledge of the relative resistances to mass transfer in the two phases. The useful feature of this relationship is that with a small number of laboratory runs one should be able to map in a family of curves for a particular system and thus rapidly determine optimum operating conditions.

Since the heavy-liquid rate is the same for both curves shown in Figure 9A, these curves are also lines of constant-flow-rate ratio. A cross plot of Figure 9A would give Figure 9B, which shows lines of constant  $P_{L0}$  (constant rotor inventory) at constant heavy-liquid flow rate on a plot of number of stages against flow-rate ratio. These curves are similar to those in Figure 8 of Barson and Beyer's paper (1) for conditions of incipient flooding in the light liquid out, as under these conditions the rotor inventory is essentially constant.

# Materials and Methods

Each stream experiences a temperature rise of approximately 10°C, in passing

through the extractor. Reagent-grade boric acid was used as a solute in the isoamyl alcohol-water system for all extraction studies because this system has a straight equilibrium curve independent of temperature between 15° and 45°C. The equilibrium data for this system were obtained from Barson's work (2).

The number of theoretical stages extracting boric acid from isoamyl alcohol with water was calculated from the expression

$$N = \frac{\log\left(1 - \frac{(y_2 - y_1)(m - a)}{a(y_1 - mx_1)}\right)}{\log(a/m)}$$
(21)

Representative data are given in Table 4. A complete discussion of compositionanalysis procedure, material-balance errors, and attainment of steady state is given in reference 4.

# Experimental Relation Among Stages, $P_{LO},\,\,$ and Flow-rate Ratio

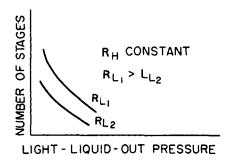
Figure 10, a plot of number of stages against light-liquid-out pressure for several flow-rate ratios, supports the relationship proposed in Figure 9A. The following additional conclusions may be drawn from Figure 10. First, more stages are obtained with this system when extracting from alcohol to water at low light-liquid-out pressures than at high light-liquid-out pressures. Second, increasing the flow-rate ratio increases the number of stages at low light-liquid-out pressures but has little effect at high light-liquid-out pressures. Third, the effect of flow-rate ratio on the relationship between number of stages and lightliquid-out pressure becomes increasingly important at higher flow-rate ratios. The effect of total throughput on the relationship between number of stages and lightliquid-out pressure was found to be small experimentally. Although the rate of mass transfer should increase with increasing throughput because of greater turbulence, the contact time in any one

stage would be correspondingly shorter so that stage efficiency might remain unchanged.

#### APPLICATION TO OTHER SYSTEMS

The procedure for applying the methods presented above to other systems using similar extractors involves two steps. First, the constants in an equation for predicting flooding limits are estimated from holdup measurements. Then a few extraction runs are made to estimate optimum operating conditions.

For an extractor having the same effective rotor dimensions and holdup as the



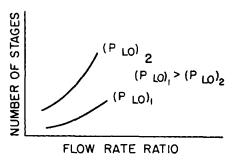


Fig. 9. Correlation among number of stages, flow-rate ratio, and light-liquid-out pressure.

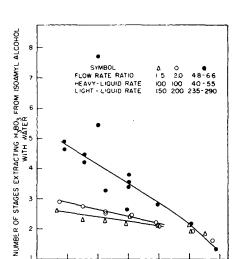


Fig. 10. Variation of number of stages with light-liquid-out pressure and flow-rate ratio.

20 40 60 80 100 LIGHT-LIQUID-OUT PRESSURE, PSIG

# TABLE 3. SUMMARY OF EXTRACTOR HOLDUP DATA

Source of holdup										Holdup, cc.
								4	7.04	529.09
Total internal line holdup										
Extractor holdup External $LL$ line holdup External $HL$ line holdup								3	7.34	637.72
Total external line holdup										
Equipment holdup, $V_E$								8-	4.38 -	747.52
Total line holdup, $V_S$										218.43
L in rotor at flooding in the light lique H in rotor at flooding in the heavy lid	uid o guid	ut, ( out.	$V_C$	L) <sub>F L</sub>	٠,,					46 25

All concentrations expressed as grams of HaBO2 per liter Rotor speed: 4,950 to 5,000 rev./min.

Run	Feed conc.	Solvent conc.	Raffinate conc.	Extract cone.	Material balance, $\%$ error	H flow rate, cc./min.	L flow rate, cc./min.	Flow- rate ratio	$P_{oldsymbol{LO}}, \  ext{lb./} \  ext{sq.in. } oldsymbol{ ext{gauge}}$	Number of stages
A9	7.628	0.096	1.164	13.64	10.6	103.5	193.9	1.873	116	2.36
A13	2.207	0.079	0.572	4.462	5.1	102.9	261.8	2.544	108	2.03
A 20-1	8.037	0.083	0.153	4.291	0.6	106.2	<b>57</b> .0	0.537	120	2.13
B12	7.923	0.042	0.696	10.54	4.9	98.6	150.4	1.523	44	2.27
B17	7.878	0.063	0.981	13.51	5.3	99.1	203.6	2.054	62	2.49
B36	7.858	0.319	4.240	23.30	7.4	40.1	273.4	5.917	101	2.19
B40-1	7.845	0.288	3.683	25.17	2.1	46.5	283.7	6.101	61	3.48
B40-2	7.845	0.288	3.705	25.31	2.9	45.5	282.9	6.213	61	3.57
B40-3	7.845	0.288	3.627	25.45	4.7	45.9	286.7	6.252	61	3.78
B41-1	7.845	0.288	3.527	26.42	4.9	45.4	288.6	6.355	40	5.45
B41-2	7.845	0.288	3.503	26.83	3.7	45.1	286.1	6.343	40	7.71
B41-3	7.845	0.288	3.643	26.50	1.8	45.3	287.4	6.349	40	5.48

extractor used in this investigation, one may use r = 7.71 in., b = 0.173 in.,  $V_C = 529$  cc.,  $(V_{CL})_{FL} = 46$  cc., and  $(V_{CH})_{PH} = 25$  cc. to calculate the maximum permissible rotor speed (within the pressure limitations of the equipment) from Equation (8) and to estimate both flooding limits as a function of rotor speed from Equation (9).

For an extractor having different rotor dimensions and holdup from those of the extractor in this investigation it is suggested that the procedure outlined below be followed, a two-phase system in which no extraction takes place being used. Working with such a simple two-phase system will provide a background of experience that will be useful in explaining and circumventing apparently anomalous behavior with more complex systems.

The procedure is as follows: the lightliquid-in pressure is measured at several rotor speeds; the light-liquid-out pressure is measured at both incipient flooding and flooding in the heavy liquid out at a single rotor speed; and the holdup of each phase is measured at this latter rotor speed and at least two rather high and two rather low light-liquid-out pressures, including incipient flooding in the light liquid out. These data are then used to estimate r, b,  $V_C$ ,  $(V_{CL})_{FL}$ ,  $(V_{CH})_{FH}$ and the holdups of the various sections of the extractor.

The rotor radius r is estimated by substituting the data for light-liquid-in pressure as a function of rotor speed into Equation (8).

The equipment holdup  $V_E$  is estimated as the sum of V<sub>L</sub> and V<sub>H</sub> at each lightliquid-out pressure, according to Equation (2).

The light-liquid-holdup measurements as a function of light-liquid-out pressure are used to obtain the equation

$$V_L = a_1 + a_2 P_{LO} (14a)$$

for low light-liquid-out pressures and the equation

$$V_L = a_3 + a_4 P_{L0} (14b)$$

for high light-liquid-out pressures, where the  $a_i$ , i = 1 to 4, are estimated from the data when plotted as in Figure 5.

The light-liquid line volume is estimated as  $a_1$ . The light liquid in the rotor at flooding in the light liquid out  $(V_{CL})_{FL}$ is estimated as  $a_2P_{LO}$  when  $P_{LO}$  is given its value at flooding in the light liquid out. The rotor holdup  $V_c$  is estimated as the difference between V<sub>L</sub> at flooding in the heavy liquid out and  $V_{SL}$ , where  $V_L$  at flooding in the heavy liquid out is computed from the measured value of  $P_{LO}$ under these conditions and Equation

The effective rotor width b is estimated from  $\pi b r^2 = V_C$ .

The heavy-liquid line volume is esti-

$$V_{SH} = V_E - V_C - V_{SL}$$
 (22)

The heavy liquid in the rotor at incipient flooding in the heavy liquid out  $(V_{CH})_{FH}$ is estimated as the heavy liquid flushed from the extractor while it is being stopped, after no more heavy liquid can be flushed from the spinning rotor. This step completes the estimation of constants required for the prediction of flooding limits.

The second part of the procedure for applying the methods presented above to other systems involves making at least nine extraction runs: at three flow-rate ratios and at low, intermediate, and high light-liquid-out pressures. Such data should permit one to map in a family of curves on a plot of stages against lightliquid-out pressure, such as shown in Figures 9 and 10.

It should be noted that the system studied was characterized by a small change in density difference between the inlet and effluent streams. When large changes in density occur as the result of solute transfer, the proper method for evaluating the  $\Delta \rho$  term has not been established.

## NOTATION

 $V_E$  = extractor holdup, cc.

 $V_C$  = rotor holdup, cc.  $V_S$  = line and seal holdup, cc.

 $V_H$  = heavy-liquid holdup, cc.  $V_L$  = light-liquid holdup, ec.

 $V_{CH}$  = heavy-liquid rotor holdup, cc.  $V_{CL}$  = light-liquid rotor holdup, cc.  $V_{SH}$  = heavy-liquid line and seal holdup,

 $V_{SL} =$ light-liquid line and seal holdup,

 $R_L = \text{light-liquid flow rate, cc./min.}$ 

 $R_H$  = heavy-liquid flow rate, cc./min.

 $P_{LI} = \text{light-liquid-in pressure, lb./sq. in.}$ 

 $P_{LO} = \text{light-liquid-out pressure, lb./sq.}$ in. gauge

= density difference between two  $\Delta \rho$ phases, g./cc.

= rotor speed, rev./min.

= rotor radius, in.

= rotor width, in.

= gravitational constant

= number of theoretical stages

= slope of equilibrium line

= slope of operating line

= feed concentration, g./liter  $y_2$ 

= solvent concentration, g./liter  $x_1$ 

= raffinate concentration, g./liter  $u_1$ 

= extract concentration, g./liter

## LITERATURE CITED

- 1. Barson, N., and G. H. Beyer, Chem. Engr. Progr., 49, 243 (1953).
- -, M.S. thesis, Iowa State College Library, Ames, Iowa (1952).
- -, personal communication, Institute for Atomic Research, Iowa State College, Ames (1953).
- Jacobsen, F. M., Ph.D. thesis, Iowa State College Library, Ames, Iowa (1954).
- Morgenthaler, J. H., G. T. Keene, W. L. Mann, and A. A. Wasserman, U. S. Atomic Energy Commission Rept. KT-110 (1951).
- 6. Poffenberger, N., personal communication, Dow Chemical Company, Midland, Michigan (1955).

Presented at A.I.Ch.E. Houston meeting