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A radiotracer determination of entrainment in a bubble-cap column

Vernon Paul Dorweiler
Iowa State University

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A RADIOTRACER DETERMINATION OF ENTRAINMENT
IN A BUBBLE-GAP COLUMN

by

Vernon Paul Dorweiler

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of
DOCTOR OF PHILOSOPHY

Major Subject: Chemical Engineering

Approved:

Signature was redacted for privacy.

In Charge of Major Work

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Signature was redacted for privacy.

Dean of Graduate College

Iowa State College
Ames, Iowa
1959
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INTRODUCTION

Purpose of the Research

The radiotracer has made great inroads as a research tool in the biological and physical sciences. Seemingly then the tracer holds great promise of application in chemical engineering research. Yet comparatively little has been done to date toward developing its potential in this area. It is the purpose of this research generally to develop some of the facets of tracer potential, and specifically to develop a tracer technique for the quantitative determination of entrainment rate in a bubble-cap column.

An important analytic tool in chemical engineering is the application of the law of the conservation of mass, the familiar material balance. The general utility of the material balance naturally suggests its application in this study. The question arises as to whether activity is directly amenable to treatment in a material balance. This question and the subsequent considerations required in such an application will be developed in this investigation.

Study of the entrainment phenomenon is by no means a new topic of interest to investigators. The literature reveals a continuous probing of the problem, but with the general trend being toward more complex techniques for correlating data, without any appreciable improvement in
experimental methods of determining entrainment rate. Some recent work has indicated a fundamental approach to the determination of the mechanism of entrainment, using advanced experimental methods. This analysis is still quite removed from the complexity of an operating column. The work here is aimed at an area intermediate to the two: application of a technique which permits determination of entrainment rate with markedly higher accuracy than the classical method, and use of an experimental system which closely simulates an operating column.

Entrainment may be defined as the upward displacement of droplets of liquid due to the dynamic action of vapor rising through the liquid. This phenomenon is characteristic of those unit operations in which a gas or vapor is dispersed through a liquid, and is, of course, of primary concern in distillation. Contamination of product streams and decrease in plate efficiency and column capacity are general effects of entrainment in a distillation operation. Any or all of these factors may concern the design engineer both in the total rate of entrainment to be expected, and in the effect of various design and operating variables on the rate. It is these two areas that are of specific interest in this study: the determination of the rate of entrainment for various design and operating conditions, and the determination of the effect produced in varying these conditions.
Some Aspects of the Tracer Technique

The classical technique reported in the literature for determining the entrainment rate is the entrainment of a dilute, non-volatile salt solution from one tray to the next, and subsequent analysis of samples of the higher tray solution to determine the amount of salt entrained. In this manner an average rate of entrainment is obtained. The sampling procedure is usually examined carefully since inference from the small samples is necessary. A device was recently patented (1) which permits determination of the entrainment through one riser by drawing off the vapor through the riser and trapping the entrained liquid. The obvious question of inference is pointed here.

Application of a tracer was initiated in this work on the basis of two factors: increased accuracy in determining results, and the instantaneous nature of the determination. Both of these follow directly from the nature of the radioisotope. Since the tracer can be detected external to the column, no sampling per se is needed; consequently error from this source is eliminated. Monitoring the higher tray yields a continuous record of activity on the tray. The rate at which the activity level increases is a measure of the rate of entrainment. In this way a continuous record of the entrainment rate is obtainable.
The tracer technique developed here is not completely divorced from the classical methods. First, the monitoring geometry of a tray involves in a real sense a selected "sampling" of the activity dispersed throughout the liquid contained within the solid angle subtended by the collimating path. The "sample" is, however, a continually averaging one due to the turbulence on the tray. Further, the system is not disturbed in this "sampling". Second, while the monitoring of a steady-state operation can feasibly yield instantaneous entrainment rate with relatively simple equipment, it readily becomes apparent that complex electronic circuitry would be required to achieve this for the operating periods involved here. Hence, by measuring the net increase in activity level, an average entrainment rate was determined using simple detection equipment.

In the preliminary formulation of this tracer technique the need for monitoring activity level at several locations on the column became apparent. To accomplish this with a single detection system required the development of a feature novel to quantitative monitoring procedures. Generally monitoring procedures are either the rigid, fixed-geometry type used in quantitative work or the movable, survey type used for qualitative indication. The technique here, being quantitative in nature, would require a movable, highly reproducible geometry scheme to take accurate activity measurements at the several locations. Success achieved in this
work in developing a scheme for reproducing geometry is believed to be a significant advance toward wider utilization of tracer methodology in engineering applications.

Scope of the Research

The system studied was an air-water, non-overflow simulation of a bubble-cap distillation system. Isolation of the entrainment phenomenon and obvious simplicities of operation prompted the choice of this system. A large portion of the entrainment literature has been developed in studying this type of system.

Both design and operating variables were selected for study. Tray spacing and slot area were chosen as the design variables to be studied, and vapor velocity and static submergence as the operating variables. Submergence is probably more properly classified as a design variable since the level is established by design of weir height. However the classification here, for the non-overflow system, is correct.

The following magnitudes of the four variables were chosen to demonstrate the several effects:

1. Tray spacings of 9 and 12 inches were selected. Since this variable is known to have a pronounced effect, the difference in levels was kept small so as to prevent masking the lesser effects.
(2) Slot heights (areas) of 1/2 and 1 inches (by 1/4 inch wide) were chosen mainly on the basis of the availability of the bubble-caps. However, the effect to be demonstrated here was the difference between a slot operating full at all velocity levels and one whose height of opening varied with velocity.

(3) Static submergences of 7/8 and 1 1/2 inches were chosen on the basis of these being general industrial conditions. Lesser submergence levels would tend to permit the slots to blow free, and greater submergence levels would tend to cause back-trapping of liquid.

(4) Six vapor velocities, equally spaced between 0.9 and 2.6 ft per sec, were selected to cover the range of industrial conditions. Higher velocities create very severe conditions on the tray. Lower velocities give poor vapor-liquid contact, and also present detection problems which would require an adjustment of the tracer technique. The entrainment rate detectable using the technique described covers a 400 fold range of entrainment rate.

In the interests of covering this range of variables, described in (1) through (4) above, with a replication of all conditions, a primary concern became the problem of minimizing the quantity of contaminated liquid to be contained, and to an extent required modifications in the experimental
system. Initially a specialized overflow scheme was devised to provide for this feature of column operation. However, the quantity of radioactive liquid which would be required became quite extensive. The concept of a static system therefore was adopted.

A further consideration was the effect of neighboring bubble-caps on a plate, and, akin to this, the effect of column wall. Obviously increasing column diameter would permit the use of several caps, but would again increase liquid volume. Since the alternatives were the use of quite small bubble-caps versus the troublesome containment problem, a single bubble-cap was used.
LITERATURE REVIEW

Two general areas of the literature are germane to this investigation: the field of radiochemistry as applied to tracer technology, and that area in which the tracer technique developed herein is applied, entrainment in bubble-cap columns. Emphasis was placed upon a review of the entrainment literature in which evaluation of this work was directly concerned. Less effort was devoted to a formal review of the tracer literature since the tracer technique developed in this work is quite independent of the basic features of other tracer work. The underlying science of radiochemistry is fully treated in current texts (2, 3).

Tracer Technology

While several tracer investigations of chemical engineering topics have been reported, only one has a similarity to this study. Wagner et al. (4) applied a Ba-140 isotope to study entrainment of pitch in a flash distillation unit used for the separation of the more volatile constituents of a crude oil residue. A non-radioactive barium salt of a high molecular weight phenol, containing a homogeneous distribution of trace amounts of Ba-140, was dissolved in flasher pitch, and the radioactive pitch was injected into the unit at any of five different positions. The source of entrainment
was located by monitoring the distillate stream after an injection at any of the five positions. Cause and magnitude of the entrainment were thereby determined. Both counting of samples and monitoring of streams were reported as amenable to calibration against standards, although quantitative measurements were not obtained.

Maloney and Hughes (5) reported the development of a Geiger-Mueller tube for counting static liquid samples and one for monitoring liquid streams on a quantitative basis, in a distillation of methanol-ethanol mixtures.

Several other tracer works have been reported in the study of several of the chemical engineering unit operations (6, 7, 8).

Entrainment Studies

A survey of the literature concerned with entrainment work begins with Peters (9) in 1922 and extends up to the recent issues of the chemical engineering journals, a span of nearly four decades. Two significant flurries of activity are readily noticeable in this period. Early work, 1933-1937, which accompanied the transmutation of distillation from an art into a science, took the classical chemical engineering approach in studying the entrainment phenomenon. Rate of entrainment in large scale equipment was measured, and generalized correlations were developed. More recent work, dating
from 1950, applied a more fundamental approach of studying a system in which the entrainment phenomenon is well-defined. The recent work is presented as a series of successive approximations to industrial practice. The results of both approaches have been applied in analysis of the results of this work.

**Classical entrainment studies**

The first complete, quantitative study of entrainment in a bubble-cap column was presented by Holbrook and Baker (10) in 1933. The effect on entrainment of tray spacing, superficial column velocity, slot velocity and the rate of liquid overflow were determined. A steam-water system in an eight-inch diameter test column, with two bubble-caps on each of three plates, was studied. Data were reported for 59 different operating conditions, a major contribution to an area otherwise barren of quantitative data.

In 1934, Souder’s and Brown (11), in analyzing the dynamics of liquid particle suspension, developed a relationship between the suspending velocity and vapor and liquid densities, \( \rho_v \) and \( \rho_l \). Empirical coefficients for this relationship, \( C \), were developed as a function of the system from data of operating commercial petroleum columns. The relationship

\[
\dot{w} = C \rho_v \left( \rho_l - \rho_v \right)^{0.5}
\]
although somewhat vague, is suggested for calculating the maximum allowable vapor velocities, \( W \), that can be used in commercial petroleum columns to obtain "satisfactory" products, and has, in fact, been widely used by designers in determining column capacity. A discussion of some of the more salient factors affecting the rate of entrainment is also presented by Souders.

Sherwood and Jenny (12), in 1935, studied the effect of vapor and slot velocities, plate spacing and liquid properties on entrainment of water by air, and analyzed the relation between entrainment and column performance. Two criteria for determination of the effect of entrainment on column performance were the appearance of non-volatile impurities in the product and modification of a McCabe-Thiele diagram to allow for entrainment and effect on operating efficiency. A relatively small increase in the total number of plates was found to correspond to large entrainment rates, and the efficiency was not materially reduced by entrainment until an entrainment of about 10% was reached. Rather high vapor rates are required to cause 10% entrainment rates; these considerably alter the vapor-liquid contact which may result in a decrease in efficiency apart from that caused by entrainment. Consequently the factor limiting column capacity was proposed to be pressure drop across the plates rather than entrainment.
The final significant work of this early group is that of Peavy and Baker in 1937 (13). Entrainment data are reported for the distillation of ethyl alcohol-water in an 18-inch diameter, three-plate column. The variables studied were plate spacing, static submergence, slot area and vapor velocity. A correlation is presented between the reduction in plate efficiency and entrainment.

Several authors have presented theoretical treatments of the effect of entrainment on efficiency (14, 15), and have derived equations for calculating the reduction in plate efficiency for a given quantity of entrainment. Colburn (16) reviewed the various methods and presented the following simplified equation for calculating the effect of entrainment on dry plate efficiency:

$$E_a = \frac{E_v}{1 + \frac{e E_v}{R}}$$

where $E_v$ is the dry vapor efficiency, $E_a$ is the apparent plate efficiency, $e$ is the entrainment rate, and $R$ is the liquid overflow rate, with $e$ and $R$ in consistent units. Colburn also found that the limiting factor in column operation is loading or excessive pressure drop across a plate, and not entrainment.

Eduljee (17), Simkin et al. (18) and Fair and Matthews (19) present recent correlations of entrainment rate with the various design, operating and system variables.
Entrainment mechanism studies

Recent British publications have introduced a significant, new trend in studying the entrainment phenomenon. Work, coordinated under the Institution of Chemical Engineers (London) (20), has been concentrated on defining bubble formation at slots, bubble dynamics at the slot and in traveling through the liquid, and bubble collapse at the liquid vapor interface. While this definitive work has many ramifications in chemical engineering, an obvious application is in explaining the entrainment phenomenon.

Spells (21) and Spells and Bakowski (22, 23) reported an extensive study of bubble formation at single and multiple slot arrangements. With a single slot, a basic pattern of behavior was definable. Two mechanisms of bubble formation were defined in describing this behavior. For a "deep mechanism", at low flow rates and sufficiently deep slot submergence, bubble development was found to follow a predictable pattern of bubble volume, time of formation and channel length of bubble in the liquid. An important part of bubble development occurred after the bubble had risen above the top of the slot due to a channel formed between bubble and slot. For a "shallow" mechanism, at increased air rates or decreased slot submergence, a series of similar bubbles was obtained forming a fairly continuous connection between the slot and the surface. No consistency as obtained above was found,
indicating considerable leakage of vapor from the slots to the vapor space through the bubbles. Bubbling from multiple slots was shown to be essentially the same for single slots, with the "shallow" mechanism predominating in conditions similar to normal bubble-cap operation. In both cases bubble sizes were found to be much larger than those predictable from surface tension and density considerations.

Slot action has been investigated by Cross and Ryder (24) for both low and high flow rates. From simple hydrodynamic theory, slot opening was predicted above a critical air rate through the slot. Below this critical air rate the slot opening was predictable from surface tension effects alone.

Knelman (25) presents a classic study of the collapse of surface bubbles at the liquid-vapor interface. The sequence of events associated with the collapse of the bubbles was described in the following manner. A bubble, when reaching the surface of the liquid, oscillates with decreasing amplitude until, just before collapse, it comes to rest with the upper part, a hemispherical dome, projecting above the surface. Collapse of the bubble is associated with a release of energy sufficient to impart a comparatively high velocity to any drops formed. The surrounding liquid moves inward to fill the depression caused by the bubble. As the crater fills in, the momentum of the inflowing liquid produces a jet, which rises at a high velocity and in certain circumstances
detaches one or more drops which are responsible for the main losses by entrainment. The number and the size of these drops projected into the vapor space are the main source of entrainment. Number, size and trajectories of drops formed were determined. Drop size distribution and drop characteristics are discussed. Excellent high-speed photographs of the various phases of the bubble collapse adds much to the overall presentation.

Garner et al. (26) established that droplets are formed both by collapse of the bubble dome and by disintegration of the jet of liquid arising from the bubble crater. Ninety-five percent of the entrained droplets were below 20 microns, including all from the collapse of the bubble dome, but due to their low mass formed a very small fraction of the total entrained liquid. Surface tension, viscosity and bubble size were found to control the projection of drops by the jet, with a reduction in any of the factors increasing the entrainment.

A summary paper is presented by Newitt et al. (27) developing an overall analysis of the mechanism of entrainment. The function of baffles in entrainment separators is also analyzed.
THE TRACER TECHNIQUE

The development of a technique for the determination of entrainment rate utilizing a radioactive tracer was based upon a variety of considerations. New ideas were developed in describing tracer characteristics. Well-established principles of material balances were likewise applied. Obviously further work will improve on the concepts developed here, so this initial amalgamation of new and old will be described in detail to aid such future work.

A word about nomenclature is appropriate before proceeding since a great deal of new symbolism is necessarily introduced. Standard chemical engineering nomenclature is used wherever possible. Also standard practice in the field of statistics is used in designation of variable magnitude and variable effect. The variables tray spacing, slot height, static submergence and velocity will be designated by the lower case letters z, h, s and v, respectively. Ascending magnitudes, or levels, of these variables will be denoted by subscribing the letters with numbers starting with unity as the lowest magnitude. Effects of the variables on entrainment rate will be designated by the upper case letters Z, H, S and V.

In introducing symbolism for count rate, capital letters are used to represent the count rate "effect" of a certain amount of activity. Since use of the letter C for all count
rate terms would require double subscripting, for location (at the test tray or the monitor tray) and for time (initial or final), the symbols T and M are used to denote count rate at the two locations. When any general count rate expression is derived, the symbol C is used.

Isotope Considerations

Selection of the isotope to be used in this work was approached chemically on the basis of use in a wide system of liquids, and physically on the basis of energy and penetration of the emitted radiation.

A basic reason for utilizing a radioisotope in this work was the unique ability to detect the presence and concentration of the tracer on the various trays without resorting to sampling. Characteristically then, the detecting device would be external to the system, and the radioactivity, to be suitably detected, would have to penetrate the column wall. The type of radiation indicated was either gamma or very strong beta radiation. The experimental arrangement favored the use of a gamma emitter since a depth of liquid equal to the column diameter, a bubble-cap and riser and a 1/4-inch wall were interposed in the scanning plane of the detector.

Selection of a particular gamma emitter was made consistent with the following criteria:

(1) Solubility of the tracer in the liquids to be used
without altering the physical characteristics of the liquids, i.e., density, viscosity and surface tension.

(2) Non-volatility of the tracer so that movement of the tracer is directly associated with the physical movement of the liquid, and not with vapor movement of a volatile tracer compound.

(3) Half-life sufficiently long so that time correction during a run would not be required, but also sufficiently short so as to permit simple disposal procedures.

(4) Ready availability.

(5) Radiological safety consistent with the frequency and period of exposure during experimental runs.

Of these criteria all except the last are fixed in the properties of the isotope chosen. The last item is related to the concentration of tracers and, as noted, the time of exposure involved.

Selection of either I-131 or La-140 would have fulfilled the experimental requirements. The 131 isotope of iodine emits a variety of gamma rays and several high energy beta particles. The decay scheme (28) is shown in Figure 1. Half life of the isotope is 8.1 days, with decay to xenon. The 140 isotope of lanthanum emits a variety of gamma rays with energies from 0.090 to 1.60 Mev, and has a half-life of 40.2 hours. Comparison of the solubility data of these two elements indicates some advantage for the iodides, particularly in the liquids familiar in distillation.
Figure 1. Decay scheme for I-131 (28)
Iodides are available from the Oak Ridge National Laboratories, in either the sodium or the potassium forms. The lanthanum isotope due to its short half life is not conveniently available. Rather the $^{140}$ isotope of barium is purchased which decays by beta emission with a 12.8 day half-life to the $^{140}$ isotope of lanthanum. This mother-daughter decay scheme necessitates a "milking" separation of lanthanum from the barium, and the subsequent formation of a lanthanum compound to be used. Selection of the iodine isotope was made on the basis of ready availability and higher solubility of the iodides.

Quantitative Considerations

In applying a tracer technique in measuring quantitatively the entrainment rate, only the method of analysis is changed from the classical method. The equations developed for entrainment rate are based upon material balances describing the movement of activity. Detection and analysis of count rate data introduce novel concepts. The quantitative development will now be presented.

Material balances

The following terms are defined:

- $a$ total activity on a tray, millicuries (mc)
- $A$ activity concentration on a tray, mc per liter
C  count rate, counts per minute (cpm)

$E_t$  entrainment rate from a tray, pounds of liquid entrained per minute

K  geometry factor, cpm per mc

L  volume on a tray, liters

$\Theta$  operating time, minutes

$\rho$  liquid density, pounds per liter

The following relations follow directly from the above definitions:

$$C = Ka$$  \hspace{1cm} (1)

$$a = AL$$  \hspace{1cm} (2)

$$C = KAL$$  \hspace{1cm} (3)

Consider the system shown schematically in Figure 2. Initially only the pool on the test tray contains activity. As liquid is entrained up the column, with no back-trapping of liquid down the column, the monitor tray will contain activity, and the buffer tray will contain only water. The activity on the monitor tray at any time will equal that entrained from the test tray to the monitor tray less that entrained from the monitor tray to the de-entraining tray:

$$a_M = (a_T)_{T\rightarrow M} - (a_M)_{M\rightarrow D}$$  \hspace{1cm} (4a)

or
Figure 2. Schematic of test column showing entrainment between trays
where \(M, T\) and \(D\) refer to the monitor, test and de-entraining trays, respectively. Rearranging Equation 4b,

\[
A_M L_M = \frac{E_T \theta}{\rho} A_T - \frac{E_M \theta}{\rho} A_M
\]

(4b)

The activity on the test tray at any time will equal that initially present less than that entrained to the monitor tray:

\[
a_T = (a_T)_i - (a_T)_{T\rightarrow M}
\]

(5a)

or

\[
A_T L_T = (A_T L_T)_i - \frac{E_T \theta}{\rho} A_T .
\]

(5b)

Writing a liquid balance in terms of volume about the test tray,

\[
L_T = (L_T)_i - \frac{E_T \theta}{\rho} + \frac{E_B \theta}{\rho}
\]

(6)

\[= (L_T)_i\]

if \(E_B = E_T\). Substituting Equation 6 into Equation 5b, and solving for \(A_T L_T\),

\[
A_M L_M \left[1 + \frac{E_M \theta}{L_M \rho}\right] = A_T L_T \left[\frac{E_T \theta}{L_T \rho}\right].
\]

(4c)
\[ A_{T}L_{T} = \frac{(A_{T})_{1}L_{T}}{1 + \frac{E_{T} \Phi}{(L_{T})_{1}}} . \] \hspace{1cm} (7)

Substituting Equation 7 into Equation 4c and rearranging,

\[ \frac{A_{M}L_{M}}{(A_{T}L_{T})_{i}} = \frac{E_{T} \Phi}{\left[ 1 + \frac{E_{T} \Phi}{(L_{T})_{1}} \right] \left[ 1 + \frac{E_{T} \Phi}{(L_{M})_{1}} \right]} . \] \hspace{1cm} (8)

If \( E_{T} = E_{M} = E_{t} \), then the above reduces to

\[ \frac{A_{M}L_{M}}{(A_{T}L_{T})_{i}} = \frac{E_{t} \Phi}{\left[ 1 + \frac{E_{t} \Phi}{(L_{T})_{1}} \right]^{2}} = \frac{p \cdot E_{t}}{(1 + pE_{t})^{2}} \] \hspace{1cm} (9)

where the \( p \) parameter is defined by

\[ p = \frac{\Phi}{(L_{T})_{1}} . \] \hspace{1cm} (10a)

(This parameter represents a grouping of the physical measurements, as opposed to the count rate measurements which will be grouped together also.)
Rewriting Equation 3 for the two trays,

\[ C_T = T = K_T A_T I_T \quad (11a) \]

and

\[ C_M = M = K_M A_M I_M \quad. \quad (11b) \]

Solving for the respective AL products and substituting into Equation 9,

\[ \frac{\Delta M_C}{T} \frac{K_T}{K_M} = \frac{p E_t}{(1 + p E_t)^2} \quad. \quad (12) \]

(The use of \( \Delta M_C \) instead of \( M \) or \( \Delta M \) alone will be explained in the subsequent section, Monitoring measurements.) Defining the \( q \) parameter as

\[ q = \frac{\Delta M_C}{T} \frac{K_T}{K_M} \quad. \quad (10b) \]

Equation 12 takes the form

\[ E_t^2 (p^2q) + E_t (2pq - p) + q = 0 \quad. \quad (13a) \]

or

\[ E_t = \frac{(1 - 2q) - (1 - 4q)^{0.5}}{2pq} \quad. \quad (13b) \]

(The \( q \)-parameter represents a grouping of the count rate terms.)
Equation 13b then is the expression used to calculate entrainment rate.

**Monitoring measurements**

The general experimental scheme is illustrated in Figure 3. An analysis of this illustration will give a clear appreciation for necessary count rate measurements.

Some specific nomenclature is required to define the initial increase in activity level on the monitor tray. This increase is due entirely to the geometric location of activity on the test tray after injection. This will be termed the "geometric" effect $G$. The term "geometric" effect must be clearly differentiated from the term "geometry" factor. The latter is used (Equation 1) to designate the relationship between activity on a tray and count response from monitoring the same tray. The former is used to designate the count rate on the monitor tray due to the presence of activity on the test tray.

The initial geometric effect, $G_1$, is measured as the initial count rate on the monitor tray

$$G_1 = M_1 .$$

This effect for a given physical arrangement will be directly proportional to the amount of activity on the test tray, $a_T$. By analogy to the geometry factor, this geometric effect can be considered as the response at the monitor tray for an
Figure 3. Recorder history of the monitor tray during a typical run.
amount of pseudo-activity \( a_M' \) on the monitor tray. The amount of pseudo-activity then is directly proportional to the amount of activity on the test tray \( a_T \). The geometric ratio \( g \) is defined then as the ratio of these two

\[
g = \frac{a_M'}{a_T} = \frac{(G/K_M)}{(T/K_T)} . \tag{15a}
\]

To evaluate directly from count rate data, this term is modified slightly:

\[
g' = g \frac{K_M}{K_T} = \frac{G}{T} . \tag{15b}
\]

The geometric ratio \( g' \) can be evaluated by

\[
g' = \frac{G_i}{T_i} . \tag{15c}
\]

The change of count rate above noted in Figure 3 corresponds to the effective entrainment of activity to and from the monitor tray.

As activity is depleted to the test tray (due to entrainment) the geometric effect will diminish. Consequently the measured change of count rate on the monitor tray, \( \Delta M = M_T' - M_i \), will be somewhat less than the true change due to entrainment alone. This difference being the diminished geometric effect. An obvious technique for
evaluation of this diminished effect is to employ the Equations 15b and 15c, that is

$$\frac{G_i}{T_i} = g' = \frac{G_f}{T_f}$$

$T_f$ can be measured, and hence $G_f$ can be determined from the right-hand equality. The geometric effect then has diminished by

$$G_i - G_f = -\Delta G \quad (16)$$

(The negative sign is adopted here to maintain the standard notation of final minus initial states.) Consequently the true change of count rate due to entrainment is

$$\Delta M_c = \Delta M - \Delta G \quad , \quad (17)$$

where this is the appropriate term used in Equation 12.

In summary, the monitoring measurements required are, in sequence,

(1) Background at the monitor tray, $M_b$
(2) Background at the test tray, $T_b$
(3) Activity level after injection at the test tray, $T_i$
(4) Geometric level at the monitor tray, $M_i$
(5) Activity level after entrainment at the monitor tray, $M_f$
(6) Depleted activity level after entrainment at the test tray, $T_f$
It will be noted that all of the count rate terms used in the development here are assumed corrected for background. Separate background count rates are appropriate for the two monitoring locations as will be developed later (see section on Activity contamination of column).

**Activity balance**

Equation 13b is based on material balances for both liquid and activity. A much simpler type of balance might well be of value in checking the accuracy of the several assumptions. The loss of activity on the test tray should approximately equal the gain on the monitor tray:

\[ a_T + a_M = 0 \]

This balance becomes, from Equation 1,

\[ \frac{T_i - T_f}{K_T} = \frac{M_f - M_i}{K_M} \]  \hspace{1cm} (18)

Obviously this is only a first order approximation, since a more detailed analysis would lead again to Equations 4a and 5a.

**Evaluation of measurements**

Three aspects of the monitoring procedure are of significance here: application of the correction term, accuracy
of determining the basic count rate measurements and multiple use of these measured quantities.

Correction of the measured change on the monitor tray may seem, and is, in essence, straight-forward. However, several arguments might be advanced: that this is but a first order correction, that some experimental exploration of the nature of the true correction is needed. While several schemes were formulated to obtain more information concerning the geometric effect, no workable procedure could be incorporated into the routine procedure. However, several factors to be discussed subsequently did lead to a verification of this procedure.

To minimize any serious error due to deviation from a true correction, the magnitude of the $\Delta G$ term was kept small; in general,

$$\frac{-\Delta G}{\Delta M_c} \ll 15\%$$

Since

$$\Delta G = \beta' \Delta T$$

then $\Delta T$ is also kept small, and the assumed relationship, Equation 15a, can be expected to represent the true function.

The accuracy of determining the basic count rate measurements, $T_1$, $M_1$ and $M_2$, is of great interest. These three quantities, used in Equation 12, are measured in the most accurate manner. Referring to the sequence of measurements,
$T_i$ is determined directly after $T_b$, without moving the counter. Hence its absolute value will not be influenced by any error from reproducibility. Likewise $M_i$ and $M_f$ are determined in direct sequence so that this difference $\Delta M$ is independent of $M_b$ and reproducibility of counter geometry. Consequently it may be seen that the critical need for precise reproduction of geometry is somewhat relaxed. Obviously some dependence is still expressed in the use of constant geometry factors.

It is significant to point out the multiple use of the count rate measurements, and the consequent necessity for devoting sufficient counting time to reduce the error of each measurement to a minimum. $T_i$ and $M_i$ are used in the entrainment rate equation, in the geometric ratio determination and in the activity balance. $M_f$ is used in the entrainment rate equation and in the activity balance. $T_f$ is used in determining the final geometric effect and in the activity balance.

Isotope Specifications

Activity data, either as absolute amount of activity or as activity concentrations, are extremely poor. Specific activity is specified by the Oak Ridge National Laboratories to a $\pm 10\%$. Even this low order of accuracy is further reduced in the dilution procedure followed in preparing the individual activity samples (see Dilution procedure section).
As is true of much analytical work and tracer work, however, the use of ratios of related quantities are involved so that specification of absolute values is not necessary.

As will be noted, the q parameter involves a ratio of both count rate and geometry factors. The ratio of count rates

$$\frac{\Delta M}{T_1}$$

implies that the system is self-calibrating, i.e., independent of the amount of activity initially introduced on the test tray. $T_1$ is a direct measure of the concentration of the solution being entrained, the noted response on the monitor tray, $\Delta M$, is directly proportional to this concentration, and consequently the ratio is independent of the amount of activity used. Demonstration of the independence of the ratio of geometry factors is somewhat more lengthy.

Consider the mechanics of determining the two geometry factors $K_T$ and $K_M$:

$$K = \frac{C}{a}$$

Calculation of the geometry factors then requires determination of the count rate for a specified quantity. Obviously activity can be calculated from the specific activity of the solution resulting from dilution as corrected for time decay. By taking exact aliquots of the activity solution, the relative accuracy of the samples can be maintained at a high
level. Then, since "a" is a fixed portion of the total activity present in the shipment, insured by quantitative transfer procedures, the true amount of activity present, \(a^*\), is

\[a^* = \frac{A^*}{A} = \frac{A^*}{A_0} \exp(t/8.1)\]

where \(A^*\) is the true specific activity of the solution and \(A\) is the specific activity based on the shipment data. The ratio of geometry factors is

\[
\frac{K_T}{K_M} = \frac{C_T}{C_M} \frac{a^*_M}{a^*_T} = \frac{C_T}{C_M} \frac{a^*_M}{a^*_T} \left(\frac{A^*}{A}\right) = \frac{C_T}{C_M} \frac{a^*_M}{a^*_T} \left(\frac{A^*}{A}\right)
\]

Hence, the ratio of geometry factors have been shown to be independent of the absolute specific activity. Procedurally this means that both geometry factors must be determined from the same shipment.

Statistical Considerations

Excellent discussions of the statistical nature of radioactive decay and the application of these principles in
analyzing the action of the count rate meter are presented in (29) and (30), respectively. The discussion here will be limited to considering the reflection of error of count rate data on the calculated value of entrainment rate, and to considering the application of a statistical design in carrying out the experimental work.

"Confidence" intervals on calculated data

Equation 13b, used in calculating the entrainment rate, contains the p and q parameters. These parameters were defined so as to separate the count rate terms from the other physical measurements. These latter items can be measured very accurately so that no error here will be considered. The q parameter, being a function of count rate, is subject to its inherent error. It is of interest then to determine the effect of errors in the q parameter as reflected in the calculated values of entrainment rate.

While a strict statistical estimation of the variance of the q parameter and the resultant reflection in the entrainment rate is nearly prohibitive, a secondary statistical procedure is readily available. That is the procedure of fitting the entrainment rate-velocity data to "best", least squares curves. This procedure develops the standard deviations and hence the confidence bands for the curves. However, as is characteristic of the least squares procedure, the confidence bands have minimum breadth at the several means
of the curve (simple mean, mean of the square terms, cube terms, etc.). This type of confidence band is difficult to interpret. A much simpler procedure was adopted here, calculation of a "confidence" interval about each calculated point.

The problem addressed was this: Count rate data were read from recorder paper. A non-linear scale of the paper rendered useless the graphical integration method for averaging the recorder trace. Hence the "best" estimated average would at time be in error. Also slight peculiarities of the trace would raise further questions regarding the true values to be used in Equation 13b. A calculation procedure was desired which would permit a determination of the magnitude of error that would be reflected in entrainment rate by an error in count rate. Since either a positive or negative deviation in count rate could be expected, an interval about the observed entrainment rate could be obtained. By analogy to the statistical term, this interval was denoted as the "confidence" interval about the observed value. (The use of the word confidence here is not in accordance with the statistical meaning. Consequently quotations will be used to designate this altered meaning.)

Consider the form of the q parameter:

\[
q = \frac{\Delta M_c}{T_i} \frac{K_T}{K_M}.
\]
Let a quantity with a prime denote an observed value, and without a prime a true value. Then define \( \alpha \) and \( \beta \) by

\[
\frac{\Delta M}{K_M} = \alpha \frac{M'}{K_M}
\]

and

\[
\frac{T_1}{K_T} = \beta \frac{T_1'}{K_T}.
\]

Then

\[
q = \frac{\alpha}{\beta} q'.
\]

The expression for the true entrainment rate (measured without error) can then be written in terms of observed quantities

\[
E_t = \frac{(1 - \frac{\alpha}{\beta} q') - (1 - \frac{\alpha}{\beta} \bar{q} q')^{0.5}}{2pq' \frac{\alpha}{\beta}}
\]

\[
= \frac{(\frac{\beta}{\alpha} - 2q') - (\left[\frac{\beta}{\alpha}\right]^2 - \frac{\beta}{\alpha} \bar{q} q')^{0.5}}{2pq'}.
\]  \hspace{1cm} (19)

The principal utility of Equation 19 is in determining a "confidence" interval on the observed entrainment rate for an assumed range of error in the observed quantities. The range considered in this work was a ± 5\% error in each of the terms. Thus
Calculation of $E_t$ values for these extreme values will establish a "confidence" interval not on the entrainment rate-velocity curve but about each point. Hence a "best" curve should then lie completely within the individual intervals.

**Experimental design**

Since the purposes of this investigation were to evaluate the application of the tracer technique in measuring the entrainment rate and to determine the effect of several variables on the rate, the experimental work was conducted within a statistical design. This approach permitted a quantitative determination of the effects (and their interactions) of the several design and operating variables. Also the statistical analysis of data yielded a quantitative evaluation of the reproducibility of data obtained in applying this technique, that is, the precision attainable.

The study of a series of variables is usually treated in a factorial experiment. (The words variable and factor, and magnitude and level, take on synonymous meanings.) In such an experiment each level of each factor is combined in one of the experimental combinations with each level of every other factor. Information about the effect of each factor, and about the interrelationships between the factors, is
obtained. In this investigation, four variables, or factors, \( z, h, s \) and \( v \), were studied, the first three at two levels and the last one at six. The number of different experimental combinations was \( 2^3 \times 6^1 \) or 48.

The type of statistical design to be applied to this factorial experiment is indicated by the logical procedure that can be used in performing the forty eight runs required to examine all of the possible experimental combinations. The obvious procedure is to assemble the test column for a given combination of design variables, and then to run the twelve operating variable combinations for this one combination. The combination of design variables would then be changed and the same twelve operating variable combinations used, repeating this procedure until all possible combinations of the design variables had been studied. Change of column design variables for each combination of operating variables would require reassembly of the test column, and would also require a determination of a set of geometry factors. Such a procedure would be prohibitive. Consequently the former procedure was followed in this investigation.

A statistical design to treat this experimental procedure, and to also provide the desired factorial information, is the split-plot design. Two classifications of plots are made: main plots and sub-plots. Superimposed upon each main plot is a series of the sub-plots. In this application, the main plot is the column assembled with the
two design variables specified, e.g., $z_{m}^{h} r_{n}$; the sub-plot is the column assembled with all four variables specified, e.g., $z_{m}^{h} s_{j}^{v_{k}}$. Thus all of the $s_{j}^{v_{k}}$ are nested within each of the $z_{m}^{h} r_{n}$.

The split-plot design as applied to this research is displayed in Figure 13. The main plots are designated at the left of the display, and the sub-plots at the top. As described, the experimental procedure was to specify the row indices $m$ and $n$, and then to allow $j$ to take on the values 1 and 2, and $k$ the values 1, 2, 3, 4, 5 and 6, all of the possible column indices within this row.

The predominant feature of the split-plot design is that two different experimental error terms are generated, one for the main plots and one for the sub-plots. More precise information can be obtained about the effect of the sub-plot variables, and their interrelationships with the main plot variables, than can be obtained for the main plot variables. This higher precision follows from the characteristically smaller variation among the sub-plots within a larger main plot than among the main plots themselves.

Since the validity of statistical inference is based upon the concept of randomization of the experimental plan, the order of the individual runs, within the limitation of the split-plot design, was scheduled by a randomization procedure. First the order of the main plots was determined and then, for each of these main plots, a separate order of the sub-
plots was determined. Specifically, the order of the $s_jv_k$ was not the same for different $z_{m,n}$.

The physical process of randomization used was that of employing random numbers. For the main plot order, doubles of random digits were used. The first digit indicated tray spacing and the second slot height. For an example, the double 74 indicated $z_1$ and $h_2$ since 7 is a multiple of 2 with an odd remainder and 2 is a multiple of 2 with an even remainder. For sub-plot order, triples of random digits were used. The first digit of a triple indicated submergence level and the other two digits velocity. For an example, the triple 069 would specify $s_2$ and $v_3$ since 0 is a multiple of 2 with an even remainder and 69 is a multiple of 6 with a remainder of 3. The even-odd remainder was sufficient to denote the two level variables while the numerical remainder was used to denote the six level variable, velocity. Since the six velocity levels would not be uniformly represented by the random numbers 01 to 00, the excess numbers 97, 98, 99 and 00 were neglected as they appeared.

The random order was followed in the experimental work with one necessary modification. When the random order of the $z_m$ variable did not result in a change of the $z$ level, such a change was inserted. This effectively meant an alternation of tray spacings in the order of main plots. Necessity of this modification will be discussed in a subsequent section (Activity contamination of test column).
To provide an estimate of the experimental error terms to be used in the statistical tests, the forty eight experimental combinations were studied twice; and, to test the reproducibility of the data within this same duplicate study, the experimental plan was traversed once entirely before the repeat of the plan was started. Separate randomizations of the two experimental plans were made.

This complete traversal of the experimental plan is termed a replicate. Further repetitive investigations of the experimental plan in this manner are called replications. A comparison of the results of two or more replicates permits an evaluation of the reproducibility of the data.
EQUIPMENT AND PROCEDURE

Test Column

The test column for this investigation was designed for flexibility since several design variables were to be studied, for stability of the entrainment phenomenon since a well-defined system was essential to interpretation of results, and for minimum liquid hold-up since containment of contaminated liquid was required. Column development proceeded through the use of two, three and finally four trays. The four tray system, shown schematically in Figure 4, is described below.

With a simulated system (air-water) being studied, no column auxiliary equipment was necessary. Two test columns were constructed, a 9-inch and a 12-inch tray spacing system. Each column consisted of four cannister-type tray sections. Each section, of 8-inch nominal pipe, was cut to length to provide the desired tray spacing. A tray with a riser welded onto the bottom surface was welded into each section. Section ends were grooved, male at the top of a section and female at the bottom. This overlapping groove system provided accurate column alignment. A thin, rubber O-ring between the exterior bearing surface of the male-female grooves provided a vapor-liquid seal at the column joints. Column details are shown in Figure 4.
Figure 4. Design details for the test column
Each tray contained a single, 3-inch o.d., 16-gauge bubble-cap. The caps with sixteen square end, 1-inch by 1/4-inch slots, and the 2-inch i.d., 1/8-inch wall risers were of 410 stainless steel. A 5/16-inch bolt welded to a cross bar in the riser permitted vertical adjustment of the cap over the tray. The cap was pulled tightly to the tray for all runs. Variation of slot height (area) was obtained by reducing the 1-inch slot height, covering slot bottoms with adhesive tape.

No overflow from plate to plate was provided for in the simulated system; instead the column was equipped with a series of valves for charging liquid to the trays and for draining the trays. Sight glasses were attached to each column section to permit determination of liquid level on a tray.

A compressed air source provided the necessary vapor velocities. The air was humidified by channelling it through a 6-inch section of ceramic packing in the bottom of the column. This packing was wetted before each run so that no, or only slight, loss of liquid from the trays due to evaporation was experienced. A section of Yorkmesh was inserted directly above the ceramic packing. Mesh was also inserted at the discharge end of the column to prevent entrainment from being carried out of the column. This was considered very important in eliminating discharge of radioactive liquid into the room.
All of the interior surfaces of the column, with one exception, were painted to prevent excessive corrosion of the black-iron pipe. The one exception was the bottom surface of each tray. This was left to develop a rusted surface since it was felt that a painted surface would affect the rate of entrainment. After a week of use and a week of storage the interior of each column section was re-painted. Of course, the stainless steel of the bubble-caps and risers were not painted.

The test and monitor trays were equipped with tracer injection systems to provide uniform distribution of activity on a tray. A system consisted of a perforated dispersion tube and an injection port. The injection port housed a soft rubber ampule stopper and contained a 1/16-inch passage seating the dispersion tube. The tube was 1/16-inch o.d., of stainless steel and was soldered to the brass housing of the injection port. Holes, 0.27 mm diameter, were drilled at 1-inch intervals on the top of the curved portions of the tubes. Ends of the tubes were soldered closed. Details of the system are shown in Figure 4.

Monitoring Equipment

Developments in the past decade in the application of scintillation counters for gamma ray detection have demonstrated the superiority of this method over all others. The
radiation detector used in this work was the Model DS-1 scintillation counter of the Nuclear-Chicago Corporation. The scintillation crystal was a thallium-activated sodium iodide crystal, 3/4-inch by 3/4-inch.

A modified nose piece was formed which conformed to the curvature of the column wall. The collimating path was 1/4-inch by 2 9/16-inches slot which was particularly well suited for viewing the depth of liquid on a tray. The nose piece wall was a 3-inch i.d., 3/8-inch wall steel tube. Lead was cast in the tube with graphite inserts firmly held in place to form the collimating slot and the recess needed to fit over the basic scintillation probe. Four 1-inch machine bolts protruded through the tube wall into the lead to prevent the lead from shrinking away from the steel during cooling. The face of the head was cut with an 8 1/2-inch radius so that it would fit tightly against the column wall. The nose piece is shown schematically in Figure 5.

A Model 1615-A, analytic count-rate meter of the Nuclear-Chicago Corporation was used in conjunction with the scintillation counter. The meter is equipped with five counting rate ranges, 500, 1500, 5000, 15,000 and 50,000 cpm. Statistical accuracies of 2, 5 and 15% are provided. A Model 4312, Esterline-Angus milliammeter-recorder was used to record detector impulse.

The device designed to permit reproduction of geometry is shown in Figure 6. A 1-inch precision drill rod was used
Figure 5. Nose piece for scintillation counter
Figure 6. Geometry device for positioning scintillation counter
as the vertical guide post. Reproduction of monitoring elevations was obtained by positioning the elevation guide rings on the guide post, one above the upper monitoring location and the other below the lower monitoring location. Monitoring angle of the counter was reproduced by twisting the angle guide until the lugs fit firmly into place in the keyway cut in the elevation guide, and then locking the angle guide to the drill rod. Radial distance from the column was readily reproduced by slipping the scintillation counter through the retaining rings of the holding device. Rotation of the counter about its axis until the face of the nose piece fit the radius of the column established the final radial positioning.

The assembled monitoring equipment is shown in Figure 7.

Experimental Procedure

As outlined under the Experimental design section, the overall experimental plan was to study the forty eight variable combinations, with two complete replications of the plan. The plan used was to assemble the test column for a given tray spacing and slot height, \( z_m h_n \). Entrainment rates were obtained for the twelve submergence-velocity combinations, within this one column assembly. The four geometry factors were determined then, before the column was disassembled. This procedure was repeated until all four \( z_m h_n \)
Figure 7. Assembled monitoring equipment and test column
had been studied and the two replications had been com-
pleted.

A set of twelve entrainment points was obtained for
each assembly of the column. This number of data points re-
quired a reasonable activity shipment, and also provided a
logical division for reporting the data. Each set of twelve
data points within one column assembly, or within one main
plot, was designated a "series".

Details of the entrainment and geometry procedures, as
well as isotope handling techniques, will be described here.

**Operation of test column**

The use of a non-overflow experimental system reduced
considerably the operating considerations. Operating proce-
dure for the entrainment runs, along with the monitoring
procedure introduced for continuity of operations, was the
following:

1. Water volume necessary to give the static submerg-
   ence level was charged to each tray.
2. Background count was taken on the monitor tray and
   then on the test tray.
3. Activity was charged to the test tray while a low
   air flow rate was maintained through the column. The
   initial activity level on the test tray, Tᵢ, was deter-
mined.
4. The initial geometric effect on the monitor tray,
M₁ or G₁, was determined, and liquid levels on each of the operating trays were recorded.

(5) Air flow was adjusted to provide the desired velocity level, and then admitted to the test column for an accurately measured operating time.

(6) Final activity level on the monitor tray and on the test tray were determined, and final liquid levels on the trays were recorded.

(7) Contaminated liquid was drained from the column into a 20-liter carboy. The column was then flushed vigorously with tap water, with the water admitted at the top of the column. This flush water was drained through a 1/2-inch gate valve in the bottom of the column into a 55-gallon overflow drum for ultimate dilution before being discharged to the sanitary sewer. Approximately a thirty minute flush was necessary to reduce the column to a stable background.

A second procedure was involved in operating the test column in determination of geometry factors at the two monitoring positions. During the geometry runs the scintillation counter was fixed at one tray, and all of the runs necessary to determine the geometry factor were performed. The counter would then be moved to the second monitoring position and fixed there during its series of geometry runs. The procedure was as follows:
(1) Water volumes were charged to the trays as before, however only to the trays lower than the one being tested. To this tray water was charged to provide the lower submergence level.

(2) Background count was taken on the tray.

(3) Activity was charged to the tray as in (3) above, and the count corresponding to this activity was then recorded.

(4) Sufficient extra water was charged to the tray to increase the submergence level to the second level. Again a low air flow rate was maintained through the column. The corresponding count was again recorded.

(5) Contaminated liquid was drained from the column into a 20-liter carboy and the column flushed as before. Minimum counting periods at the test tray were 20 to 30 minutes and at the monitor tray 30 to 40 minutes. Count data were recorded on the 500 cpm range for monitor tray and on the 1500 cpm range for the test tray. This was done even with the test tray background below 100 cpm so as to provide an automatic zero adjust for each range on the recorder.

The one most important part of column operation was maintaining an air flow rate through the column during injection of activity on a tray, and also during dilution of a geometry run to the second submergence level. This flow rate, maintained at a low value with very, very low entrainment potential, provided sufficient turbulence on a tray so that
homogeneous distribution of activity was obtained. The initial distribution of activity by the dispersion tubes proved to be grossly unsatisfactory. A five minute flow period was found adequate to thoroughly mix the solution on the tray.

**Isotope handling**

**Dilution procedure**

Shipments of about 40 millicuries of I-131 isotope, sodium iodide in basic sodium sulfite solution (pH of 8), were received from the Oak Ridge National Laboratories. Shipment volumes were of the order of 1.0 milliliters in glass, screw-cap vials of about 15 ml volume. The shipment volume was diluted in its vial to 10.0 ml with a caustic solution, keeping the pH at 8. Ten samples of 1.25 mc, one of 1.50 mc and one of 1.75 mc, corrected for decay and based on activity specifications provided by the Oak Ridge Laboratories, were prepared for the entrainment runs, and ten samples of activity varying from 0.1 to 1.5 mc were similarly prepared for the geometry runs. Any remaining activity was used to prepare further samples for low velocity entrainment runs.

The samples were transferred from the shipping vial by a 1.0 ml pipette, with 0.01 ml graduations, to 20 ml sample, glass vials provided with polyethylene stoppers. The volume of the sample, after repeated washing of the pipette with caustic solution were added to the sample, was brought up to
about 10 ml. All transfers were performed behind suitable lead shielding. Samples were drawn up into the pipette by securing a hypodermic syringe to the top of the pipette. Dilution and transfers were performed by the Radiological Services Group of the Ames Laboratory.

**Injection procedure** The activity sample in the sample vial was first diluted with distilled water to a 20 ml volume. This volume was taken up into two 10 ml hypodermic syringes. Twenty-two gage hypodermic needles were used with the syringes. The activity was injected onto a tray by inserting the needle through the ampule stopper and lodging it inside the dispersion tube (see Figure 4). The activity was then discharged into the dispersion tube by hypodermic pressure. A rolled tissue was kept pressed under the needle at its penetration into the ampule stopper to prevent any losses of minute droplets during injection and during removal of the needle.

Walls of the sample vial were washed briskly with distilled water. This wash solution was then injected into the column, serving to rinse the vial, the syringe barrel and the dispersion system.

Throughout the injection procedure, rubber surgical gloves were worn by the operator to prevent direct contact of activity on the skin.

The injection is demonstrated pictorially in Figure 8.
Figure 8. Injection of activity into the test column
Activity recovery

After an entrainment run some slight amounts of activity were to be found on the de-entraining tray, and on the monitor tray for the lowest velocity entrainment runs. The activity levels were sufficiently low to permit drainage to the 55-gallon overflow drum for dilution and subsequent disposal to the sanitary sewer. (See section below for discussion of disposal requirements.) The great bulk of the activity remaining on the test and monitor trays was drained thoroughly into a 20-liter carboy and stored. After drainage was completed, the column was flushed and the effluent carefully discharged through the overflow system.

Activity contamination of test column

As is generally true in handling radioisotopes, contamination presents a continual and perplexing problem. Once the tracer was introduced into the test column no amount of flushing could reduce the empty column activity levels to the original background. Instead it was found that a highly stable quasi-background was attainable, that is, after a thorough flushing a stable level could be reached which would not change with repeated flushing. The concept of this quasi-background, or memory, was accepted here on the basis of its stability. Since stability of the count rate was demonstrated during flushing, this stability was assumed during the entrainment and geometry runs.
In some preliminary work one test column was used for a three week period. This resulted in a rapid build-up of the quasi-background. Examination of the column revealed a highly corroded interior. The build-up of quasi-background was concluded to be caused by this rusting and roughening of the column walls, and subsequent adsorption of activity. Consequently it was found necessary to alternate the 9-inch and the 12-inch test columns each week. Repainting of the column sections after a week of use and a week of storage was found to provide a workable schedule.

**Activity accumulation and disposal** Since a rather extensive isotope usage rate was established, the total possible accumulation of activity was of some interest. A rather simple expression was found to represent the amount of activity on hand after receiving a weekly shipment. This is the geometric series with its sum, \( a_\Sigma \), after \( N \) weeks given by

\[
a_\Sigma = a_s \frac{(1 - e^{-\lambda N})}{(1 - e^{-\lambda})}
\]

where \( a_s \) is the activity per shipment, and \( \lambda \) is the decay constant in weeks\(^{-1}\).
The contaminated liquid recovered from the test column was held in storage for decay through ten half-lives (81 days), and then turned over to the Radiological Services Group of the Ames Laboratory for disposal. Disposal of the decayed activity was in accordance with the regulations set forth in (31).
EVALUATION OF THE TRACER TECHNIQUE

Evaluation of the technique developed here takes on two aspects: an analysis of the quantitative development, and an even more significant evaluation of the applicability and limitations of the technique. Both are important in outlining suggested future work in application, or further development, of the technique.

Before this evaluation is undertaken, however, some general discussion of the underlying premise of this work, that activity is directly amenable to treatment in a material balance, is appropriate. The only firm evidence that can be offered in establishing its validity lies in two areas: the adaptability of the several quantitative concepts developed in the treatment of data, which will be discussed here, and the evaluation of the entrainment data, which will be treated under Results of the Entrainment Study. However, in preview of these discussions, it seems evident that in this work the utilization of activity in a material balance has lead to a satisfactory analysis of this physical system.

Evaluation of the Technique

Quantitative considerations

The entrainment data obtained in this work were calculated from Equation 13b. A sample calculation for a series
of runs is presented in Table 1. The question underlying the application of this equation is: How well does the mathematical expression describe the physical system? Answers to this necessarily incorporate a critical review of the assumptions involved in the derivation of this equation and the assumptions inherent in some phases of the technique. These will now be discussed.

**Geometry factor** Geometry factor, defined by Equation 1, was calculated for each series of runs from five sets of activity-count rate points. The calculation procedure used was the least squares fitting of the experimental data to the linear relation

\[ C_r = K a_r + e_r \]  

(20)

where \( e_r \) is the error observed in the \( r \)th determination. The slope, the geometry factor, was calculated from the relation

\[ K = \frac{\sum_r C_r a_r}{\sum_r a_r^2} \]  

(21)

Scatter of experimental data about the calculated line was in all cases very slight. An example is shown in Figure 9, for a typical series of runs. The detailed calculations
Table 1. Calculation of results for a series of runs ($z_2^h_1$)

<table>
<thead>
<tr>
<th>$s_j^V_k$</th>
<th>$M_1$</th>
<th>$T_1$</th>
<th>$g'$</th>
<th>$\Delta T$</th>
<th>$\Delta G$</th>
<th>$\Delta M$</th>
<th>$\Delta M_c$ ($K_T/K_M$)</th>
<th>$q$</th>
<th>$2p$</th>
<th>$E_t$</th>
<th>$E_t^x$</th>
<th>$g'$</th>
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<td>$s_1^V_1$</td>
<td>154</td>
<td>1203</td>
<td>0.1280</td>
<td>24</td>
<td>0.1042</td>
<td>0.02665</td>
<td>16.5289</td>
<td>0.00340</td>
<td>0.0104</td>
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<td>0.1221</td>
<td>54</td>
<td>0.1042</td>
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<td>8.8548</td>
<td>0.00966</td>
<td>0.0296</td>
<td>1.402</td>
<td>0.00507</td>
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<tr>
<td>$s_1^V_3$</td>
<td>133</td>
<td>873</td>
<td>0.1523</td>
<td>63</td>
<td>0.1042</td>
<td>0.05192</td>
<td>7.0838</td>
<td>0.01642</td>
<td>0.0504</td>
<td>2.413</td>
<td>0.00680</td>
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</tr>
<tr>
<td>$s_1^V_4$</td>
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<td>822</td>
<td>0.1338</td>
<td>51</td>
<td>0.1042</td>
<td>0.09549</td>
<td>5.0177</td>
<td>0.04771</td>
<td>1.465</td>
<td>2.918</td>
<td>0.01635</td>
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<tr>
<td>$s_1^V_5$</td>
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<td>0.1256</td>
<td>45</td>
<td>0.1042</td>
<td>0.10900</td>
<td>3.5419</td>
<td>0.08029</td>
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<td>3.459</td>
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<tr>
<td>$s_1^V_6$</td>
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<td>768</td>
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<td>0.1042</td>
<td>0.08205</td>
<td>2.0661</td>
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<tr>
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<td>7.0291</td>
<td>0.01022</td>
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<td>$s_2^V_2$</td>
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<td>4.2174</td>
<td>0.02972</td>
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<tr>
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<td>0.106</td>
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<td>4.2174</td>
<td>0.06286</td>
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<td>2.936</td>
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</tr>
<tr>
<td>$s_2^V_5$</td>
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<td>642</td>
<td>0.1636</td>
<td>45</td>
<td>0.106</td>
<td>0.12010</td>
<td>2.8116</td>
<td>0.11537</td>
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<td>0.03309</td>
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</tr>
<tr>
<td>$s_2^V_6$</td>
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<td>672</td>
<td>0.1518</td>
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<td>0.106</td>
<td>0.08528</td>
<td>1.3705</td>
<td>0.15170</td>
<td>4.659</td>
<td>4.017</td>
<td>0.03776</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
for the series are presented in Appendix A along with a listing in Table 6 of all $K_T$ and $K_M$ values for the eight series of runs.

The geometry factor is a function of the geometry of the activity being viewed. In this application the geometry is a dispersion of activity in a liquid pool. The height of liquid on the tray is a critical dimension, with the depth fixed by column diameter. Variation in the height of this pool is reflected in a variation of the magnitude of the geometry factor. Consequently it was necessary to determine separate geometry factors for the two submergence levels at each of the two monitoring positions.

As discussed in the procedure section, the geometry factor was determined for the higher submergence level by diluting the individual runs from the lower submergence level after the initial count rate response had been recorded for the activity injected. This procedure would be suspect statistically since a high correlation exists between $(K_M)_1$ and $(K_M)_2$ and between $(K_T)_1$ and $(K_T)_2$. However, only the ratio of geometry factors at the different locations is used in the calculations, and the geometry factors in the ratio are determined from independent activity injections.

A second procedure followed in determining geometry factor might be questioned. This is the determination of the geometry factors by fixing the counter at one monitoring position and running through the necessary data points.
Figure 9. Geometry factors for a typical series
Since the technique is based upon the reproducibility of geometry, a more severe test would be to reset the counter for each set of data points. The latter procedure was eliminated for two reasons. First, it could only introduce error in the determination without any increase of confidence that a better estimate of the true, effective geometry factor was being generated. Second, the data given in Table 4, Appendix A, indicates that the ratio of geometry factors, determined without resetting the counter, was essentially constant for both submergence levels, and also from series to series. In fact the use of a value of 1.1 for the ratio would be justified for all work except the first series listed in Table 6.

**Entrainment rate** In proceeding from Equation 8 to Equation 9, the assumption is made that the entrainment rates for the monitor and the test trays are the same. Special care was taken to provide identical tray sections, so that this assumption seems well-founded. If this were not exactly true, the rate determined from Equation 13b would represent some average of the two rates.

A further assumption is that the entrainment rate for the buffer tray equals that for the test tray. This is a minor assumption involved only in the liquid balance about the test tray. It will be considered later in a discussion of liquid level stability on the trays.
Geometric ratio  This concept was developed as a correction term, and was not independently demonstrated in the research. However, the total data of the eight series of runs have demonstrated substantially its validity.

As was developed in the earlier discussion, the geometric ratio is entirely a function of the location of activity on the test tray. Assuming a homogeneous distribution of activity throughout the pool of liquid on the tray, the geometric ratio could be expected to be different for level \((s_T)_1\) than for level \((s_T)_2\), due to the different proximity of the activity to the counter in the two cases. Such indeed was found to be the case.

In a series of twelve runs, six values of the ratio were obtained for each submergence level. Since variation in the six values at each level was experienced, an obvious technique for evaluating the soundness of the concept, and also the accuracy of determining the value, was the application of a statistical test to determine whether the means at the two levels were significantly different. However, some variation of liquid level from the desired operating levels, \((s_T)_1 = 7/8\) inches and \((s_T)_2 = 1 1/2\) inches, was also noted, thereby introducing some explanation for the noted variation of ratio values. The \(g' - s_T\) data were consequently fitted to the linear relationship

\[
g' = \gamma_0 + \gamma_1 s_T \tag{22}\]
where \( \gamma_0 \) and \( \gamma_1 \) are the intercept and slope, respectively. These data were then subjected to a statistical test to determine whether the variation in \( g' \) values due to variations in the \( s_T \) values was significantly greater than the variation in \( g' \) values with this \( s_T \) variation removed.

Figure 10 presents the \( g'-s_T \) data for a series of runs. The results of the fitting of \( g' \) and \( s_T \) to the linear relationship of Equation 22 are presented in Table 7 of Appendix A. The results of the statistical tests of fit are also tabulated there. In each case, variation in the value of \( g' \) was found to be due principally to the variation in submergence level.

The final factor in substantiation of the use of the geometric ratio, and its resultant correction term, is the control of the magnitude of the correction term. As previously stated, the correction term was kept below 15% of the total corrected change \( \Delta M_c \). This is believed to have kept the error in the correction term within the count rate error involved in determining \( \Delta M \).

**Liquid level** Stability of liquid level on each operating tray was a major control sought in the experimental work. In fact, two of the four trays in the test column were inserted solely for this purpose: the buffer tray to absorb entrance effects and to provide entrainment \( E_B \) to the test tray, and the de-entraining tray to receive entrainment from
Figure 10. Modified geometric ratio-submergence level least squares line (Equation 22).
the monitor tray. Accumulation of liquid on a tray was considered as serious as depletion.

Operation of the monitor and the test trays was considered satisfactory if a stable liquid level within 3/16 inches of the initial level on a tray. Heavier losses were expected from the buffer tray, and were experienced. (This would tend to decrease the rate of entrainment from the buffer tray.) The de-entraining tray was not used as an operating tray since it served the further purpose of eliminating entrainment out of the column.

The principal concern is the effect of variation of liquid level on the calculation of entrainment rate. As has already been discussed, K and \( g' \) are functions of the liquid level. Both appear in the equation used in calculating entrainment rate, so it is of some interest then to re-evaluate Equation 13b in terms of a variation of the K and \( g' \) factors with liquid levels.

The method used proceeded from the same material balances, but introduced initial and final values for K and \( g' \). The results were reflected in the following corrected q-parameter:

\[
q^* = \frac{\left[ \frac{M_f}{(K_M)^f} - \frac{M_1}{(K_M)^i} \right] + g_1 \left[ \frac{T_1}{(K_M)^i} \right] - (g_1 + \Delta g) \left[ \frac{T_f}{(K_M)^f} \right]}{\frac{T_i}{(K_M)^i}}
\]

(23)
Linear interpolation between the values of $K_M$ and $K_T$ determined for the two reference submergence levels was used to calculate the individual values of $K_M$ and $K_T$ at intermediate submergence levels. The value of $\Delta g$ was obtained from Equation 22:

$$\Delta g = -\gamma_1 \left[ (s_T)_i - (s_T)_f \right].$$

Using the corrected $q$-parameter, $q_3E$, entrainment rate was again calculated from Equation 13b. A comparison of the corrected data with the original data is presented in Tables 8, 9, 10 and 11 of Appendix B, for the eight series of runs. No significant change in entrainment rate was observed. Consequently it may be assumed that the variation in liquid level observed, and its effect on geometry factor and geometric ratio, did not have a significant effect on the calculation of entrainment rate.

"Confidence" intervals Uncertainty in recorder data analysis prompted the development of the simple scheme, defined by Equations 19a and 19b, to evaluate the error reflected in the calculated entrainment rate by error in count rate data. This calculation procedure was applied to the data to determine whether the scatter of points about a smoothed curve could be accounted for in this way. Results of the application of this procedure to the data of a series of runs is shown in Figure 11.
Figure 11. "Confidence" interval about the calculated entrainment data for one set of column conditions.
As could be expected, this work was effective in accounting for the lesser scatter of data about the line. The more serious deviations were probably generated by some operational disturbance rather than error in determining the count rate terms of the q-parameter.

**Activity balance** The utility of Equation 18 in the work initially was to check the uniformity of activity distribution on the test tray. Either very large or very small $\Delta T$ values, corresponding to a moderate $\Delta M$ value would be indicative of poor distribution. Subsequently the plotted activity balances were used only as a general check of a series of runs.

The $\Delta M - \Delta T$ data for a series of runs are shown in Figure 12, with the theoretical slope of Equation 18, $(K_T/K_M)$, indicated.

**Precision of the technique** The precision of the data obtained in applying the tracer technique was tested by the reproducibility of data that was achieved and by the magnitude of the experimental error. The statistical analysis of the data, which assigns a quantitative measure to these two concepts, will be considered fully under the following main heading. It will be stated here, and left for proof later, that the technique affords a high degree of precision in measuring the entrainment rate.
Figure 12. Activity balance for a typical series, with the $(K_T/K_M)$ ratio shown.
General considerations

The tracer technique was developed in this work for a well-defined system to cover a rather broad scope of variables (forty eight different conditions). In this work the underlying goal was the development of some facets of tracer application so that a well-defined system was essential to logical interpretation of cause and effect. Questions concerning applicability and limitations of the technique in other systems, however, must be considered for a proper evaluation of its true potential.

A single criterion seems definitely applicable in basic analysis of the technique. The criterion is this: a definite balance exists between the breadth of system and the scope of variables that can be investigated for equal operating difficulty. This balance can be best illustrated by an example.

Consider that the same air-water system is to be studied with the added feature of overflow across each tray. Obviously this broadens the system to include such variables as hydraulic gradient and net motion of the fluid. A specialized overflow system would be necessary to prevent activity dispersion throughout the column. By overflowing to the de-entraining tray, from there to the monitor tray,
from the monitor tray directly to the buffer tray and from
the buffer tray, plus a special overflow of activity solution
to and from the test tray, a realistic operating scheme
could be achieved. The addition of this variable however in-
creases considerably the activity inventory to be considered
in both material balances and containment. Also shielding
and equipment contamination for the activity flow system be-
come very real problems. Nevertheless, the operating scheme
could be handled by reducing somewhat the scope of conditions
to be studied.

In comparison with the classical method the tracer tech-
nique provides a marked increase in the precision of deter-
mining entrainment rate. Whether this high precision can be
maintained for a broader system can only be speculated here.
Using two characteristics, severity and quantity, some foun-
dation can be established.

The measured quantity, entrainment rate, was satis-
factorily determined over a 400-fold range. Upper limits of
the range imposed very severe operating conditions. Still no
failing of the technique was detected. In this regard the
technique seems well adapted.

Quantitative developments were based on the premise
that activity could be treated in material balances, exactly
like an ordinary component. Further the assumption was made
that the count rate analysis was based on simple geometric
considerations. Nothing entirely obvious would indicate a
failure of either as the volume, or magnitude, of the system increases. (Depth of liquid pool in the scanning plane of
the counter increases with column diameter, and would be one
factor to be studied in expansion of column size.) The
adaptability of these premises to the work here generally
suggests their extension would be equally suitable.

Limitations of the technique are two in nature. First,
it is inherently limited to a measurement of a transfer from
some reference region to another reference region. (This
can obviously be a negative type of transfer, as in the case
of de-entraining.) Proximity of the regions introduces
problems with difficulty as some inverse function of dis­tance. In this application the geometric ratio \( g' \) reflects
this principle. In a mixing problem, where transfer within
a single liquid region is to be determined, a zero distance
is involved. This would introduce infinite complexity in
applying the technique.

Second, the technique as described herein is limited to
the system studied. Such a limitation is inherent only in
the limitation of effort invested in bringing this initial
study to some degree of completion. Removal of the limita­
tion will be left to those who may consider further develop­
ment of this type of procedure, some proposals for which will
now be considered.
Proposals for Future Work

Two avenues of development are perceivable: further development of the quantitative aspects of this tracer application, and further development of the technique and system. Unfortunately no elaboration can be presented relative to the quantitative work other than the knowledge that a deeper scrutiny of the various data will certainly generate improved characterizations of the technique.

Several schemes seem evident for the development of the technique and the system. System and technique, which are not independent, will necessitate modification of technique with alteration of system. Some general remarks pertinent to both will be presented for each suggested scheme.

First of these schemes, an overflow system, has already been outlined. Besides the change in system needed to handle and meter activity solution flow across the test tray, the technique would have to be altered to determine concentration of the activity solution.

A second scheme would be a heated, distilling system. This would probably be without overflow to minimize system changes, or with overflow if considered as a study subsequent to the overflow system. Some further study of the isotope would be needed to assure stability of the tracer, the sodium iodide, under the column conditions. An elementary distillation system would be steam-water.
Obviously the technique as developed here could be applied in further study of column and design variables or liquid properties (density, viscosity and surface tension). This would not require any serious modification of either system or technique. The isotope was specifically chosen to be compatible with a large selection of liquid systems.
RESULTS OF ENTRAINMENT STUDY

The data presented here are limited to the results of the entrainment study. Those data concerned more directly with the tracer were considered previously. The original data for both of these sections are presented in (32).

A preliminary matter of some importance is the selection of a suitable parameter to describe the entrainment rate. The term used in developing Equation 13b was $E_t$, in pounds of liquid entrained per minute. This has the disadvantage of being applicable only to the specific tray dimensions encountered in this work. Consequently a more general term $E_{t*}$, pounds of liquid entrained per minute per square foot of free tray area, was also considered. The final form of the rate to be considered was the dimensionless $E$, pounds of liquid entrained per pound of vapor flowing. For completeness all three parameters were used in presenting the data.

Two general views of the entrainment data obtained in this work will be considered here: a graphical and tabular presentation of the entrainment data, and a statistical analysis of the data.
Entrainment Rate

Entrainment rate data, are presented in Figure 13, a display of the split plot design. The two figures entered in each sub-plot represent duplicate entrainment rate values for the same combination of variables. These data, in terms of the three parameters, $E_t$, $E_t^c$ and $E$, are plotted in Figures 14 and 16, and are tabulated in Tables 8, 9, 10, and 11 of Appendix B.

A further grouping of the four variables will be helpful. The term "column conditions" will be used to denote a specifying of the $m$, $n$, and $j$ indices. In this way, the entrainment data can be parameterized by the eight different column conditions represented by $z_m h_n s_j$, with the entrainment rate-velocity relation more fully considered as a continuous curve. "Best" visual curves are shown drawn through the data in Figures 14 and 16.

The smoothed curves, representing the entrainment rate-velocity functions for the eight column conditions, are shown in composite in Figures 15 and 17. Several pronounced features of the family of curves are the following:

1. The data are bounded by the extreme column conditions, the higher tray spacing and lower submergence level and slot height, $z_2 h_1 s_1$, as the lower bound, and the lower tray spacing and higher submergence level and slot height, $z_1 h_2 s_2$, as the upper bound.
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Figure 13. Entrainment rate data ($E_t$) in a display of the split plot design.
Figure 14. Entrainment rate ($E_t$ and $E_t^*$) for the eight column conditions of tray spacing ($z_m$), slot height ($h_n$) and submergence level ($s_j$)
Figure 15. Smoothed curves of entrainment rate ($E_t$ and $E'_t$) for the eight column conditions of tray spacing ($z_m$), slot height ($h_n$) and submergence level ($s_j$).
Figure 16. Entrainment rate ($E$) for the eight column conditions of tray spacing ($z_n$), slot height ($h_n$) and submergence level ($s_j$).
Figure 17. Smoothed curves of entrainment rate \((E)\) for the eight column conditions of tray spacing \((z_n)\), slot height \((h_n)\) and submergence level \((s_j)\).
(2) At the higher velocities there is a marked uniformity of slope for all of the curves except two. The curves representing the \( h_2s_1 \) combinations are those with slopes which differ from this uniformity, but which do agree with each other.

(3) The \( h_2s_2 \) combinations caused a rather large vertical displacement from the narrower region of the other three curves, at both tray spacings. This effect at \( z_2 \) was sufficiently great to overcome the marked effect of difference in tray spacing, the \( z_2h_2s_2 \) curve generally agreeing with the lower three \( z_1 \) curves.

(4) A very rapid decrease in entrainment rate is noted at the lower velocities.

The two different representations of the data, \( E \) and \( E_t \) (or \( E^* \)), show different tendencies of the curves at the low velocity region. In Figure 17, the \( E \) data show the rate-velocity function for six column conditions tending toward a single curve. Data were not obtained for the other two column conditions, representing \( z_2s_1 \), but it is evident that these two curves tend also toward a rapid decrease with a horizontal displacement to the right of the single curve. In Figure 15, the \( E_t \) (or \( E^*_t \)) data show the two \( z_1s_1 \) curves also tend toward this horizontal displacement with position intermediate to the \( z_2h_1 \) curves and a single curves now representing four curves. It is believed that the \( E_t \) form of the
data gives a clearer representation since the velocity vari-
atation is not entered on both axes of the plot.

Results of a special series of runs concentrating on the
low velocity region are shown in Figure 18. This work was
done principally to demonstrate the application of the tracer
technique at the low detection level

Analysis of Variance

The data were analyzed using techniques appropriate to
the split-plot design. Before the results of this analysis
can be considered, several concepts must be developed:
statistical meaning of effect and interaction, analysis of
variance and test of significance. These concepts are dis-
cussed in Appendix B. The reader who is unfamiliar with
statistical terminology is invited to refer there since these
specific meanings will be implied in subsequent discussion.

Analysis of the entrainment data

The form of the entrainment rate term is critical in
considering the meaning of the results of a statistical
analysis of this data. Since the effect of velocity V is
to be determined, an analysis of the data in the $E$ form would
differ substantially from that in the $E_t$ form. This is true
because $E$, given by
Figure 18. Entrainment rate (E) for one set of column conditions and low vapor velocities.
\[ E = \frac{E_t}{\rho v}, \] (24)

has a certain velocity variation removed. Hence the \( E_t \) form is considered the more suitable. Data in the \( E_t \) form were actually used in the analysis since the two forms differ only by a constant multiplier, and their F-ratios in the analysis of variance are identical.

**Error terms** As discussed earlier (see Experimental Design), the split-plot design is less sensitive to the main plot effects than it is generally to the sub-plot effects. The error (b) term is an estimate of the experimental error generated among sub-plots, \( \sigma_s^2 \). The error (a) term, \( \sigma^2 \), is an estimate of the following linear combination of the experimental errors generated among sub-plots and main plots

\[ \sigma^2 = \sigma_s^2 + 12 \sigma_w^2 \]

where there are twelve sub-plots per main plot. It is apparent that error (a) is characteristically larger than error (b).

A formal analysis of variance for the split-plot is presented in Table 2a. The mean square for error (b) is 0.00220 while the mean square for error (a) is 0.00022. Since there are no reasons why error (a) should be less
Table 2a. Analysis of variance for split-plot design
(Entrainment rate is in $E_t$ form)

<table>
<thead>
<tr>
<th>Sources of variation</th>
<th>D. F.</th>
<th>Mean square</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Main plots</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replicates</td>
<td>1</td>
<td>.00804</td>
</tr>
<tr>
<td>Z</td>
<td>1</td>
<td>.622142</td>
</tr>
<tr>
<td>H</td>
<td>1</td>
<td>.51789</td>
</tr>
<tr>
<td>ZH</td>
<td>1</td>
<td>.02205</td>
</tr>
<tr>
<td>Error (a)</td>
<td>3</td>
<td>.00022</td>
</tr>
<tr>
<td><strong>Sub-plots</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S</td>
<td>1</td>
<td>.62292</td>
</tr>
<tr>
<td>V</td>
<td>1</td>
<td>.50510</td>
</tr>
<tr>
<td>SV</td>
<td>1.05</td>
<td>.05491</td>
</tr>
<tr>
<td>SZ</td>
<td>1</td>
<td>.08715</td>
</tr>
<tr>
<td>SH</td>
<td>1</td>
<td>.13154</td>
</tr>
<tr>
<td>VZ</td>
<td>1.5</td>
<td>.07823</td>
</tr>
<tr>
<td>VH</td>
<td>1</td>
<td>.08233</td>
</tr>
<tr>
<td>SVZ</td>
<td>1</td>
<td>.00633</td>
</tr>
<tr>
<td>SVH</td>
<td>1</td>
<td>.00859</td>
</tr>
<tr>
<td>SZH</td>
<td>1</td>
<td>.00176</td>
</tr>
<tr>
<td>VZH</td>
<td>1</td>
<td>.00838</td>
</tr>
<tr>
<td>SVZH</td>
<td>1</td>
<td>.00139</td>
</tr>
<tr>
<td>Error (b)</td>
<td>1.44</td>
<td>.00220</td>
</tr>
</tbody>
</table>

than error (b), and indeed there is a substantial reason for it to be larger, the preferable procedure is to properly pool the error terms, assuming now that there is no difference between error (a) and error (b).

A second analysis of variance, ignoring the split-plot structure of the experimental design, and using a joint
error term, is presented in Table 2b. F-ratios and the results of the F-tests of significance are also shown in the table. Significance at the 2.5% and 0.5% probability levels are indicated by single and double asterisks, respectively.

Table 2b. Analysis of variance with split-plot structure ignored (Entrainment rate is in $E_T$ form)

<table>
<thead>
<tr>
<th>Sources of variation</th>
<th>D. F.</th>
<th>Mean Square</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Replicates</td>
<td>1</td>
<td>.008014</td>
<td>4.45</td>
</tr>
<tr>
<td>Z</td>
<td>1</td>
<td>.62242</td>
<td>12.2</td>
</tr>
<tr>
<td>H</td>
<td>1</td>
<td>.51789</td>
<td>28.7</td>
</tr>
<tr>
<td>ZH</td>
<td>1</td>
<td>.02205</td>
<td>12.2</td>
</tr>
<tr>
<td>S</td>
<td>1</td>
<td>.62292</td>
<td>34.5</td>
</tr>
<tr>
<td>V</td>
<td>5</td>
<td>.50510</td>
<td>280</td>
</tr>
<tr>
<td>SV</td>
<td>5</td>
<td>.05491</td>
<td>10.4</td>
</tr>
<tr>
<td>SZ</td>
<td>1</td>
<td>.08742</td>
<td>18.4</td>
</tr>
<tr>
<td>SH</td>
<td>1</td>
<td>.13154</td>
<td>72.7</td>
</tr>
<tr>
<td>VZ</td>
<td>5</td>
<td>.07823</td>
<td>13.3</td>
</tr>
<tr>
<td>VH</td>
<td>5</td>
<td>.08232</td>
<td>45.6</td>
</tr>
<tr>
<td>SVZ</td>
<td>5</td>
<td>.00633</td>
<td>3.51</td>
</tr>
<tr>
<td>SVH</td>
<td>5</td>
<td>.00859</td>
<td>4.76</td>
</tr>
<tr>
<td>SZH</td>
<td>1</td>
<td>.00176</td>
<td>0.976</td>
</tr>
<tr>
<td>VZH</td>
<td>5</td>
<td>.00838</td>
<td>4.67</td>
</tr>
<tr>
<td>SVZH</td>
<td>5</td>
<td>.00139</td>
<td>0.772</td>
</tr>
<tr>
<td>Joint error</td>
<td>47</td>
<td>.001805</td>
<td>-</td>
</tr>
</tbody>
</table>

**Significant at 0.5% probability level.
*Significant at 2.5% probability level.
Reproducibility  The F-ratio for replicates is shown, in Table 2b, to be insignificant at the 2.5% probability level. The meaning of this is that the data of the first replicate of the forty eight variable combinations are not significantly different from the data of the second replicate, that is, the data are reproducible with a high degree of precision. This fact is demonstrated at the higher of the two probability levels.

Effects and interactions of the variables  All four of the effects of the variables studied were found to be significant at the 0.5% probability level. Also all first order interactions of the variables were found to be significant at the 0.5% level. Of the higher-order interactions, the SVH and VZH interactions were found significant at the 0.5% level and SVZ at the 2.5% level, while only SZH and SVZH were not found significant.

The analysis of variance here is based on the linear model

\[(E_t)_{mnjk} = \mu + Z_m + H_n + S_j + V_k + (ZH)_{mn} + \ldots + (SHV)_{njk} + (ZHSV)_{mnjk} + e_{mnjk},\]

where \(\mu\) is the mean effect and \(e_{mnjk}\) is the experimental error. Results of the analysis indicate that this model is not appropriate to permit a representation of the entrainment rate by addition of simple effects, with negligible inter-
action terms. While physical interpretation can be es-
tablished for the effects and interactions, the use of such
an unwieldy model to characterize the entrainment rate is
certainly of little value. The problem of finding and ana-
lyzing a form of the data, or a scale of measurement, for
which a simple model, linear in the effects alone, will be
considered next.

Analysis of transformed data

The extreme interaction of the variables of the linear
model suggests the advisability of seeking a transformation
of the data. Figures 14, 15, 16, and 17 indicate that the
logarithm of entrainment rate is a suitable parameter, and
this is further substantiated by the usual procedure recom-
mended for interactive (non-additive) systems (40).

As before, the choice of the entrainment rate form is
critical. Since, from Equation 24

\[
\log E = \log \frac{E_t}{\rho v} = \log E_t - \log v - \log \rho
\]

a velocity variation is removed from the data to be ana-
lyzed if this form were used. Consequently the \(E_t^*\) form is
considered the more suitable. The parameter actually used
in the analysis is

\[
3 + \log E_t = \log 1000 E_t
\]
This is related to \( \log E_t^* \) by

\[
3 + \log E_t = 3 + \log E_t^* - \log 0.326
\]

where there are 0.326 ft\(^2\) of free tray area per tray. Analysis of the \( \log 1000 E_t \) form is exactly identical to the analysis of \( \log E_t^* \) since the mean of the transformed data and all of the individual transformed data are shifted by \((3 + \log 3.071)\). The analysis of variance is concerned only with deviations about the mean, and these remain unchanged. Analysis of the transformed data is not, however, the same as the analysis of the original data.

The problem addressed in transforming the data is the determination of a scale of measurement for which the response (entrainment rate) can be obtained by a simple addition of effects, with small deviations from additivity (negligible interaction). The transformed data are presented in Figure 19. From the results of the analysis of variance for the transformed data, in Table 3, it is obvious that the logarithmic transformation has been very effective in eliminating non-additivity (interaction).

Error term On the transformed scale, the main plot and sub-plot error terms assume their normal relationship:

\[
\text{error (c)} > \text{error (d)}
\]

The mean square for error (c) is 0.02138 while the mean
\[
\begin{array}{cccccc|cccccc}
& S_1 & & & & & S_2 & & & & & \\
& V_1 & V_2 & V_3 & V_4 & V_5 & V_6 & V_1 & V_2 & V_3 & V_4 & V_5 & V_6 \\
\hline
z_1 & h_1 & 0.94151 & 1.41747 & 1.91270 & 2.06751 & 2.36229 & 2.42710 & 1.42619 & 1.93641 & 2.24284 & 2.38435 & 2.60382 & 2.74016 \\
& & 0.89432 & 1.61268 & 1.96137 & 2.19025 & 2.42453 & 2.28919 & 1.62118 & 1.89509 & 2.17316 & 2.39046 & 2.65005 & 2.85110 \\
& 0.88874 & 1.48954 & 1.97690 & 2.31956 & 2.40081 & 2.89317 & 1.84794 & 2.12620 & 2.56347 & 2.72960 & 2.86811 & 3.12267 \\
\hline
z_2 & h_1 & 0.53148 & 0.98498 & 1.21537 & 1.67861 & 1.90466 & 1.98186 & 1.00945 & 1.47305 & 1.60927 & 1.79837 & 2.06210 & 2.18099 \\
& 0.47129 & 1.20085 & 1.41095 & 1.74570 & 1.92293 & 2.00932 & 1.18184 & 1.54716 & 1.67952 & 1.88423 & 2.11625 & 2.39254 \\
h_2 & 0.98945 & 1.24428 & 1.65533 & 2.01047 & 2.22365 & 2.36033 & 1.69705 & 1.77093 & 2.08906 & 2.27855 & 2.65628 & 2.73024 \\
& 0.53908 & 1.19921 & 1.55425 & 1.77048 & 2.23379 & 2.46736 & 1.59628 & 1.86451 & 2.13242 & 2.55924 & 2.65878 & 2.82408 \\
\end{array}
\]

Figure 19. Transformed entrainment rate data (log 1000 \(E_t\)) in a display of the split plot design.
Table 3. Analysis of variance for split plot design
(Entrainment rate is in log (1000 $E_t$) form)

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>D. F.</th>
<th>Mean square</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Main plots</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replicates</td>
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<td>.00917</td>
<td>4.468</td>
</tr>
<tr>
<td>Z</td>
<td>1</td>
<td>3.80514</td>
<td>178</td>
</tr>
<tr>
<td>H</td>
<td>1</td>
<td>2.91280</td>
<td>103</td>
</tr>
<tr>
<td>ZH</td>
<td>1</td>
<td>.14518</td>
<td>6.79</td>
</tr>
<tr>
<td>Error (c)</td>
<td>3</td>
<td>.02138</td>
<td>-</td>
</tr>
<tr>
<td><strong>Sub-plots</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S</td>
<td>5</td>
<td>4.73938</td>
<td>529</td>
</tr>
<tr>
<td>V</td>
<td>5</td>
<td>4.43622</td>
<td>495</td>
</tr>
<tr>
<td>SV</td>
<td>5</td>
<td>.10224</td>
<td>11.4</td>
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<tr>
<td>SZ</td>
<td>1</td>
<td>.00114</td>
<td>1.16</td>
</tr>
<tr>
<td>SH</td>
<td>1</td>
<td>.23492</td>
<td>26.2</td>
</tr>
<tr>
<td>VZ</td>
<td>5</td>
<td>.01550</td>
<td>1.73</td>
</tr>
<tr>
<td>VH</td>
<td>5</td>
<td>.01797</td>
<td>2.01</td>
</tr>
<tr>
<td>SVZ</td>
<td>5</td>
<td>.001015</td>
<td>1.13</td>
</tr>
<tr>
<td>SUH</td>
<td>5</td>
<td>.01663</td>
<td>1.86</td>
</tr>
<tr>
<td>SZH</td>
<td>1</td>
<td>.00521</td>
<td>.582</td>
</tr>
<tr>
<td>VZH</td>
<td>5</td>
<td>.00989</td>
<td>1.10</td>
</tr>
<tr>
<td>SVZH</td>
<td>5</td>
<td>.00573</td>
<td>.640</td>
</tr>
<tr>
<td>Error (d)</td>
<td>44</td>
<td>.008954</td>
<td>-</td>
</tr>
</tbody>
</table>

**Significant at 0.5% probability level.**

The square for error (d) is 0.0089545. This smaller error (d) term makes the experiment more sensitive to the effect of the operating variables on the transformed scale.

**Reproducibility** The F-ratio for replicates is shown in Table 3 to be less than unity. This effectively indicates
that the mean square for replicates is completely insignificant.

Effects and interactions of the variables  Again all four of the effects of the variables studied were found significant at the $0.5\%$ probability level. Only the SV and SH interactions are significant at the $0.5\%$ level while all other interactions fail even at the $5\%$ level.

The analysis of variance of the transformed data is based on the linear model given by

\[
(\log 1000 E_t)_{mnjk} = \mu + (\log Z)_m + (\log H)_n + (\log S)_j + (\log V)_k + (\log ZH)_{mn} + \ldots + e_{mnjk}
\]

where $\mu$ is the mean effect on the transformed scale and $e_{mnjk}$ is the experimental error.

From the results of the analysis of variance the model which fits the data satisfactorily is

\[
(\log 1000 E_t)_{mnjk} = \mu + (\log Z)_m + (\log H)_n + (\log S)_j + (\log V)_k + (\log SV)_{mn} + (\log SH)_{jk} + e_{mnjk}.
\]

This result suggests that the model is multiplicative in the simple effects and the two remaining interactions:

\[
(E_t)_{mnjk} 10^3 = Z_m H_n S_j V_k (SV)_{jk} (SH)_{jn} \exp(e_{mnjk} + \mu).
\]
Since the data covers only the two levels of three variables, and six levels of the fourth, the model is restricted to this range. An extension of the number of levels of a variable would permit a fit of the effect of a variable to a relation with its magnitude, e.g.,

$$\log V = \delta_0 + \delta_1 v_1 + \delta_2 v_1^2 + \ldots + \epsilon_1$$

where the $\delta$ are coefficients and $\epsilon_1$ are deviations of observed values from the fitted curve.
DISCUSSION OF ENTRAINMENT STUDY

Whenever two phases are contacted, the process of disengaging is never entirely completed. Witness the turmoil at the top of a fluidized-bed, with the solid fines in various tailings in the gas stream. Similarly, in vapor-liquid contacting, the region above the liquid surface presents a heterogeneous region of drops and slugs of liquid. The term entrainment is applied to the resultant net transfer of some of this liquid from the region.

Prediction of entrainment for any set of conditions has been an important consideration in the design of vapor-liquid contacting equipment, in particular in the design of bubble-cap columns for optimum capacity and efficiency. Obviously this requires a fundamental understanding of the mechanism(s) by which entrainment takes place. Much of the work to date has been devoted to the task of accumulating entrainment data for various sets of conditions and systems, and drawing conclusions about the overall (effective) mechanism from the results. But now some preliminary studies have begun to appear which more directly attack the complexities of the causes of entrainment. A most heartening result is that in many phases inferences drawn from individual entrainment studies and conclusions reached from detailed mechanism work represent convergent views.
The work here was addressed to the intermediary task of measuring entrainment rate but with the potential of quantitatively evaluating the effect of several variables. From this stand the strength of the two approaches can be drawn upon, and perhaps re-enforced.

Theoretical Considerations

The sequence of events leading from the dispersion of vapor into the liquid pool to the entrainment of liquid seems to be assignable to two groupings of column variables which can then be used to describe the actual mechanisms involved. Conditions on the tray can be assigned the role of generating liquid drops and projecting them above the liquid surface. These "tray" conditions can be defined in terms of the factors affecting turbulence on the tray: liquid and vapor properties, velocity of vapor dispersion (slot velocity) and mechanical design of the tray. The actual transfer of liquid can be assigned to "disengaging" conditions, that is, distance to the next tray and superficial vapor velocity. The two groupings of conditions are not completely independent but are both effective in the entrainment of liquid. These two groupings will now be discussed in developing the entrainment process.
Tray conditions

Three basic conditions under which drops are formed are well recognized (26, 27, 34, 35): bursting of bubbles, foaming and splashing. These three represent varying conditions of arrival of vapor at the liquid surface. An evaluation of the tray conditions will permit an understanding of the three conditions, and subsequently an understanding of the formation of drops and their projection from the vapor-liquid interface.

Vapor is dispersed through slots into the liquid pool on a tray. Slot opening, bubble formation and bubble dynamics in the liquid become important considerations. For low air rates through the slots, detailed analyses of bubble characteristics have been obtained (23, 36, 37): bubble size and the velocity and path describing travel upward through the liquid pool are predictable from surface tension, viscosity and density of the liquid, slot opening and pressure of the dispersing vapor. For higher air rates through the slots, new factors are encountered (36, 37): kinetic energy of the gas, converted to pressure energy, controls bubble size, and swarm motion of the bubbles is characteristic. However, some semblance of a definable system still prevails, although not as clearly as for low air rates.

At high air flows, beginning at rates less than the $v_1$ level in this work (21), the term "momentum and frictional
effects" begins to appear. Slot opening is directly predictable from velocity (24). Bubbles are no longer formed individually but as a series of more or less similar bubbles, with the development of one bubble starting as soon as its predecessor is about to leave the slot; in fact, periodic direct leakage channels are formed through connected bubbles from slot to liquid surface (23). Physical properties of liquid and vapor, and slot shape, have little effect on the bubbles. In general, any relationship between air rates and bubble volumes and time periods of formation is destroyed by the leakage phenomenon.

Height of the pool, or submergence level, presents a conflicting picture. Much is known about the idealized case of a particle rising (or falling) in a medium. A terminal velocity is reached, given sufficient opportunity (time or distance). Such appears to be the case here also in analyzing bubble dynamics. Where its magnitude is sufficiently great, submergence level has little effect on the velocity with which a bubble rises in the liquid (36). However, this level was found to be important in development of the leakage channels (23). When the bubble meets the water surface before reaching maximum size (this occurs at low submergence levels) a certain amount of leakage takes place.

Arrival of vapor at the liquid surface then creates the three basic conditions under which liquid drops are formed. The basic unit in all three conditions seems to be the
arrival of an individual bubble and its subsequent bursting. Foaming is then definable independently only as to rate of occurrence of the basic unit; splashing presents an exaggerated analogy of the basic unit.

A very clear description of the series of events involved in the arrival of a bubble at the vapor-liquid interface, and its bursting, is available (25, 26, 27). The bubble penetrates the liquid surface, forming a dome-shaped film of liquid. As liquid drains away from the dome, a rupture occurs with the vapor escaping through the opening. Droplets formed by disintegration of the dome are projected upward. However, the vapor, escaping with an explosive force at the instant of burst, leaves a partial vacuum and a crater in the liquid surface. The motion of surrounding liquid rushing in to fill the crater produces a jet of liquid which rises above the liquid surface and disintegrates into drops. Two sources of drop formation are detected from this bubble bursting: rupture of the dome and the jet disintegration.

Swarm motion of bubbles at the liquid surface presents a frothing which is termed the foaming condition. Depth of the layer is controlled by the relative ratio of arrival and collapse of the bubbles at the surface (26). Under foaming conditions, drops are formed and projected only by the collapse of the domes (35).
The third condition, splashing, represents the release of a large volume of vapor at a localized area of the liquid surface. Agitation of the liquid becomes severe, and slugs of liquid are released (10, 34). An analogy of the bursting bubble is brought to mind: the periodic leakage of vapor directly from the slots to vapor spaces represents the large vapor release, and the slug action is due to the collapse of the tunnel with the resulting jet, or slug, being created by the violently agitated liquid.

In summary of the foregoing, tray conditions are defined basically by bubble mechanics and the burst phenomenon. The various factors involved in the bubble mechanics appear to control the characteristics of the bubble collapse, and hence control the size of drops formed and the velocities with which the drops are projected. The disengaging conditions then are effective in determining whether a drop will be entrained.

Transfer process

Whether liquid drops will be carried away depends on the three disengaging conditions: velocity of projection from the liquid surface, superficial vapor velocity and height of the disengaging space. Imposed upon these three factors is the concept of a size distribution of the drops being generated by the tray. Total entrainment is critically dependent on size of drops since the mass entrained is approximately
proportional to the cube of drop diameter. However this aspect of the entrainment process will not be considered here since only certain qualitative factors are understood at present (26). Instead the entrainment, or transfer, process will be examined in view of the several approaches presented in the literature, and with a view toward evaluating the role of the disengaging conditions in effecting the actual entrainment.

The superficial vapor velocity in the disengaging space was used by Souder's and Brown (11) in the initial characterization of the transfer process. The analysis consisted of a consideration of the theoretical velocity required to suspend liquid drops in the vapor stream. Throwing of drops by jet action of the vapor was recognized in the analysis but the form of the equation derived for suspending action was not altered to include this effect. Instead empirical coefficients were determined to correct the error introduced. This analysis minimized the significance of disengaging space since a drop once suspended by the vapor will be carried away regardless of the distance to the next tray.

The problem was next examined by Davis (38) who included both effects. The analysis considered a drop projected upward with velocity $p$ in a vapor stream. The vapor stream velocity $c$ was considered a sustaining velocity. A general equation was derived for the relationship between height of projection, drop size, projection velocity and
vapor velocity. For negligible resistance and zero vapor velocity this relation reduces to the very logical form \( p^2/2g \).

Application of the general equation derived by Davis requires a proper evaluation of the projection velocity and the drop size from a distribution. A satisfactory characterization of an effective projection velocity and an effective drop size, as functions of tray conditions, would permit a specification of the height (tray spacing) required to eliminate drops greater than a critical size. In this way entrainment rate could be theoretically predicted and controlled.

An alternate approach to defining the transfer process is suggested by the form of the Peavy velocity parameter (13), \( v \rho^{0.5} \). The square of this term, \( v^2 \rho \), represents the kinetic energy per unit volume of the vapor in the disengaging space (superficial vapor velocity is used). A portion of the change of kinetic energy of the vapor, from its initial value in the slots to its final value in the disengaging space, is exchanged with the liquid on the tray and subsequently transferred to the drops thus providing the impetus to elevate the drops to a given height.

Such an analysis is admittedly rudimentary since the direct form of the change in kinetic energy per mass of vapor in entraining a mass of liquid is not present; rather the square root of the function is. The important factors
present are that the parameter does correlate entrainment
data for different vapor densities (masses), and that the
use of superficial vapor velocity in the parameter does not
correlate for various tray conditions. The proper charac-
terization of initial velocity, in terms of tray conditions,
does seem to offer some merit in determining the portion of
the total energy change which is transferred, and hence the
amount of liquid entrained.

Discussion of Results

Analysis of variance results

The meaning of the effect of several variables has been
brought sharply into focus in the theoretical considerations
above, and has been demonstrated by the discussion of Fig-
ures 14, 15, 16 and 17. The two scales of measurement used
in analysis of the data present divergent views of the form
of the entrainment rate as a function of the effects of the
variables studied in this work: additive versus multiplica-
tive. The scale chosen for any analysis is evaluated in
terms of its success in simplifying the working model so
that examination of the experiment and presentation of re-
sults are straightforward. On the basis of this criterion
the log $E_t$ scale represents the more workable model in pre-
senting the entrainment data. However, the $E_t$ scale demon-
strates more clearly the complex physical mechanisms
involved in the entrainment phenomenon. Consequently both forms of the analysis will be explored in the following.

A most important factor relative to the analyses of the data should be considered before proceeding. The results of the analyses must be tempered by the fact that the submergence level effect includes a tray spacing effect. This becomes obvious by focusing attention on the disengaging space, or the free height between the liquid-vapor interface and the next tray surface. Disengaging space is defined (11) as the physical tray spacing \( z_m \) less twice the total liquid depth on the tray:

\[
    z_{mj}^* = z_m - 2(s_j + t)
\]  

(25)

where \( t \) is the distance from the tray surface to the top of the slots (1 3/16 inches in the test column), and \( s_j \) is the static submergence level. Disengaging space is then a function of both tray spacing and submergence level. The quantity subtracted in Equation 25 represents the height of the foaming liquid pool, and is estimated as twice the total liquid depth on the tray.

A change of submergence level affects a change in disengaging space for a constant tray spacing. Then, while the analysis evaluates the effect of a change in submergence level this extraneous change is included. The degree to which this affects the analysis is not known since the
experimental design originally planned gives an evaluation of the tray spacing-submergence level relationship. In shifting emphasis to a disengaging space-submergence level relationship the design becomes a fractional replication of a design similar to the original, but with four levels of $z_m$ studied: $z_{11}^*$, $z_{12}^*$, $z_{21}^*$ and $z_{22}^*$. The data collected here is of little value in estimating the effects and interactions on the basis of a fractional replication of the altered design.

**Analysis of entrainment data**

The extremely interactive nature of entrainment data (see Table 2b) is not surprising in view of the theoretical analysis of tray conditions and disengaging conditions. The $v$, $h$ and $s$ variables are involved basically in defining tray conditions; the $z$, $v$ and $s$ variables are dominant in the disengaging conditions. Hence interaction of the variables within these two groupings would be expected. Also since the tray conditions are effective within disengaging conditions, an interaction between groupings would also be expected.

Turbulence of liquid on the tray, due largely to bubble agitation, appears to be the key factor to a real understanding of the variables effective on the tray. Velocity of the vapor through the slots appears to be controlling. With an increase of vapor velocity or a decrease in slot area (height), the dispersing velocity through the slots, and
hence the tray turbulence, increases. The role of static submergence on the tray is somewhat obscure. Bubble motion appears unaffected by it (23) as does slot action (24). However, the quantity of liquid in motion is a function of the submergence level (see Equation 25), and in this way can act as a dampening factor in the velocity effects in the tray turbulence. These initial observations will give an appreciation of the complexities on the tray. The true role of each of the v, h and s variables cannot be specifically defined, and what is even more important, their effect on liquid surface conditions cannot be readily examined.

Disengaging conditions apparently are more properly centered about the projection of drops from the surface than the suspension of drops by the vapor stream (26). In this way z and s enter directly into the determination of disengaging space through Equation 25. Velocity, although entering directly in suspending-sustaining velocity, is perhaps more fundamentally present in tray conditions as they effect projection velocities. The former is characterized by superficial column velocity while the latter has already been considered as a slot velocity effect. Some s and v interplay can be visualized since the frothing of a liquid pool is a function of the liquid on the tray, s, and the air flow, v, passing through this liquid, a somewhat more strict interpretation than Equation 25 affords.
The interaction of the variables is quite obvious from the foregoing discussion. As has been introduced in the theoretical considerations, and enlarged somewhat above, the disengaging conditions include the effects of tray conditions. The s and v variables enter directly in both sets of conditions as well as by inclusion in the disengaging conditions. The further classification of v into slot and column velocity designations must be noted. The z and h variables seem more properly considered as a direct factor only in their respective sets of conditions. Again by inclusion of tray conditions effects in disengaging conditions an interaction can be expected.

Analysis of the transformed data

Value of analysis of the log $E_t$ data appears to lie in the simplification of the model which can be used to designate the response. There is merit in this approach since an extension of the range of the variables, as suggested in the results section, would effectively permit a prediction of entrainment rate for a given set of conditions. As has been stated at the outset of this section, it is this prediction of entrainment rate that is the basis for work in this general area.

The two remaining significant interactions on the transformed scale, SH and SV, cannot be readily interpreted for physical meaning. It will be remembered that the SH interaction has been demonstrated in the plots of log $E_t$
versus velocity, in Figures 14 and 15. No similar graphical evidence is obvious from the plots for the SV interaction. Its physical significance has been discussed in the preceding section: the froth height at the various levels of velocity increases with a resultant decrease in disengaging space. Hence the differential effect on the entrainment rate is noted by the interaction. It can only be suggested that these two interactions are sufficiently great as to be present in the multiplicative function.

**Entrainment data**

Smoothed curves representing the bounds of the 12-inch and the 9-inch spacings of this work are shown in Figure 20, compared with the low velocity work of Volante as quoted by Robinson and Gilliland (39, p. 426), and in Figure 21, compared with the 12-inch tray spacing data of Holbrook and Baker (10), Sherwood and Jenny (12) and Peavy and Baker (13).

It can be seen that the low velocity data agree substantially with that of Volante. The agreement of data at the higher velocity level is not particularly good at first glance. The lower, \( z_2 h_{1s} \) curve, although starting off somewhat higher than the Holbrook and the Sherwood data, does indicate a general agreement at the higher velocities. The \( z_2 h_{2s} \) curve evidently reflects the lesser disengagement space and greater froth height, and tends toward agreement with the data of Peavy. Note that the extreme point of the
Figure 20. Comparison of entrainment rate ($E$) with literature data for several tray spacings ($z_m$)
Figure 21. Comparison of entrainment rate (E) with literature data for 12-inch tray spacing ($z_2$)
Peavy data in Figure 21 suggest the high velocity trend of this data. The Sherwood data were fitted to the line shown by the least squares procedure whereas the other curves were transcribed from the original publications.

Since the data presented in Figure 21 represents a wide variation of experimental conditions, some of the important specifications of each system are given in Table 4. It will be noted that the $z_2h_2s_2$ curve represents a minimum disengagement space for the group, hence its elevated position in Figure 21. Furthermore, the $z_2h_1s_1$ curve, representing a 9 11/16-inch tray spacing, agrees very closely with the Sherwood data when corrected for the differential in disengaging space.
Table 4. Details for reported experimental entrainment systems (12-inch tray spacing)

<table>
<thead>
<tr>
<th>Ref.</th>
<th>Col.</th>
<th>System</th>
<th>Static dia. (in.)</th>
<th>Static disengag. (in.)</th>
<th>Static sub. space (in.)</th>
<th>Min. disengag. space (in.)</th>
<th>Overflow</th>
<th>Caps per tray</th>
<th>No. of plates</th>
<th>Slot dimensions</th>
</tr>
</thead>
<tbody>
<tr>
<td>(10)</td>
<td>8</td>
<td>Steam-water</td>
<td>9.5</td>
<td>.4-.1.2&lt;sup&gt;a&lt;/sup&gt;</td>
<td>9.5</td>
<td>Yes</td>
<td>2</td>
<td>3</td>
<td>1 1/8 x .264&lt;sup&gt;b&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>(12)</td>
<td>18</td>
<td>Air-water</td>
<td>11-s</td>
<td>.28-.10&lt;sup&gt;a&lt;/sup&gt;</td>
<td>10</td>
<td>No</td>
<td>1/7</td>
<td>2</td>
<td>3/4 x 1/8 x 3/16&lt;sup&gt;b&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>(13)</td>
<td></td>
<td>EtOH-water</td>
<td>10 11/16-s</td>
<td>0.5</td>
<td>1,2</td>
<td>Yes</td>
<td>10</td>
<td>3</td>
<td>1/2 x 1/8</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Steam-water</td>
<td>10 11/16-s</td>
<td>1,2</td>
<td>8 11/16</td>
<td>Yes</td>
<td>10</td>
<td>3</td>
<td>1/2 x 1/8</td>
<td></td>
</tr>
<tr>
<td>(32)</td>
<td>8</td>
<td>Air-water</td>
<td>10 9/16-s</td>
<td>7/8</td>
<td>9 1/16</td>
<td>No</td>
<td>1</td>
<td>4</td>
<td>1/2 x 1/4</td>
<td></td>
</tr>
</tbody>
</table>

<sup>a</sup>Dynamic submergence level.

<sup>b</sup>Tooth-shaped slots.
SUMMARY

A tracer technique was developed for the determination of entrainment in a bubble-cap column, using the I-131 isotope as the tracer. Quantitative relations were developed by treating the tracer directly in material balances. Entrainment rate was calculated from count rate data and simple geometric considerations.

The experimental system was an air-water, non-overflow simulation of a distillation system, in an eight-inch diameter, four plate test column. Activity was introduced on a tray in the sealed column through a hypodermic injection-dispersion system. Entrainment was determined by the transfer of activity from this tray to the next higher tray. The amount of activity transferred was detected by monitoring the upper tray with a scintillation counter. A device was developed to permit monitoring the two trays with the one unit, accurately reproducing monitoring geometry at the two locations.

The entrainment rate expression was found to be independent of the absolute amount of activity used in preparing samples for the experimental runs. Quantitative handling procedures were found adequate to transfer the differing amounts of activity required by decay corrections. Activity contamination of the test column interior was satisfactorily controlled for a series of experimental runs by vigorous
flushing of the column. A quasi-background count was established in the column which was somewhat higher than normal background count but which exhibited a rigid stability. Ninety six experimental conditions were studied with only moderate activity accumulation problems involved. The I-131 isotope proved to be a satisfactory tracer for detection through the column wall and for simple disposal procedures.

A 400-fold change of entrainment rate was determined for forty eight different operating combinations of four variables: vapor velocity, submergence level, slot area and tray spacing. This range was extended further in demonstrating the determination accuracy at very low entrainment rates. Duplication of the operating combinations established that the entrainment data were reproducible with a high degree of precision.

The tracer technique developed is suggested as a general method for measuring transfer between two regions, with difficulty of application as an inverse function of proximity of the two regions. Recommendations for extension of the experimental system and technique are presented.

Entrainment data collected for the forty eight operating conditions were analyzed statistically to evaluate the effect of the four variables, and the interrelationship between the variables. For the range of the variables covered, entrainment rate was found to be nearly a multiplicative function
of the variable effects alone. The mechanism of entrainment was described as primarily a projection of drops from the vapor-liquid interface to the next tray. A comparison of the entrainment data developed in this work with the literature data is presented.
LITERATURE CITED


31. Federal Register, 22, No. 19, Title 10, Chap. 1, Part 20, Par. 20.303. 1957.


NOMENCLATURE

a total activity on a tray, mc
A activity concentration, mc per ml
B subscript referring to buffer tray
C count rate, cpm
D subscript referring to de-entraining tray
e error in determination
E entrainment rate, lbs liquid entrained per lb vapor flowing
E_t entrainment rate, lbs liquid entrained per min
\( E_t^* \) entrainment rate, lbs liquid entrained per min per sq foot of free tray area
f subscript referring to final value
g geometric ratio
g' modified geometric ratio
h slot height, inches
H effect of slot height on entrainment rate
i subscript referring to initial value
j index for submergence level
k index for velocity level
K geometry factor, cpm per mc
L volume on a tray, liters
m index for tray spacing
M count rate at monitor tray; subscript referring to monitor tray
n index for slot height
p parameter defined by Equation 10a
q parameter defined by Equation 10b
r index for individual observations
s static submergence level, inches
S effect of submergence level on entrainment rate
T count rate at test tray; subscript referring to test tray
v vapor velocity, ft per sec
V effect of velocity on entrainment rate
z tray spacing, inches
z* disengaging space, inches
Z effect of tray spacing on entrainment rate
α error term defined by Equation 19a
β error term defined by Equation 19a
γ₀,γ₁ constants defined by Equation 22
ρ density
θ operating time, min
ACKNOWLEDGMENTS

The author wishes to thank Dr. George Burnet for his suggestion of the problem, and for his guidance and criticism during the investigation. The assistance of Dr. A. Voigt in an initial evaluation of the problem, and the assistance of Dr. D. V. Huntsberger with the statistical analysis is also gratefully acknowledged.

Appreciation is expressed to the members of the Chemical Engineering Division who assisted in the experimental work, and to the members of the Radiological Services Group for their patient assistance which included the preparation of over 600 activity samples.
APPENDIX A

Data pertaining to the Evaluation of the Tracer Technique section are presented here. More complete data can be obtained in (32). The correspondence between series designation here and in (32) is given in Tables 6 and 7.

Geometry Factor

Equation 21 was applied in the calculation of the four geometry factors for each series of runs. (The term series refers to the twelve $s_j v_k$ combinations studied within a single $z_{m,n}$.) A typical set of experimental data necessary for the calculation of the four geometry factors for a series of runs is listed in Table 5. Mechanics of the calculation

Table 5. Geometry factor data for a typical series of runs
of \((K_M) \) are illustrated by the following:

\[
(K_M)_1 = \frac{\sum_r c_r a_r}{\sum_r a_r^2} = \frac{(62)(.100) + \cdots + (304)(.538)}{(.100)^2 + \cdots + (.538)^2}
\]

\[
= \frac{367.968}{0.63006} = 584.021
\]

Results of the calculations for the three other geometry factors of this series are

\[
(K_M)_2 = \frac{307.734}{0.62725} = 490.608
\]

\[
(K_T)_1 = \frac{3927.003}{5.86883} = 669.129
\]

\[
(K_T)_2 = \frac{3246.660}{5.85588} = 554.427
\]

The two \((K_T/K_M)\) ratios then are

\[
\left(\frac{K_T}{K_M}\right)_1 = \frac{669.129}{584.021} = 1.146
\]

\[
\left(\frac{K_T}{K_M}\right)_2 = \frac{554.427}{490.608} = 1.130
\]

Results of the determination of geometry factors for the eight series of runs are listed in Table 6.
Table 6. Geometry factors for the eight series of runs

<table>
<thead>
<tr>
<th>Series in (32)</th>
<th>z_{m/n}</th>
<th>(K_T)_1</th>
<th>(K_M)_1</th>
<th>(K_T/K_M)_1</th>
<th>(K_T)_2</th>
<th>(K_M)_2</th>
<th>(K_T/K_M)_2</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>z_2h_2</td>
<td>814.575</td>
<td>485.995</td>
<td>1.67609</td>
<td>649.190</td>
<td>389.360</td>
<td>1.66732</td>
</tr>
<tr>
<td>Q</td>
<td>z_1h_1</td>
<td>796.502</td>
<td>763.236</td>
<td>1.04358</td>
<td>664.156</td>
<td>606.248</td>
<td>1.09551</td>
</tr>
<tr>
<td>R</td>
<td>z_2h_1</td>
<td>609.136</td>
<td>550.937</td>
<td>1.10563</td>
<td>505.620</td>
<td>485.223</td>
<td>1.04203</td>
</tr>
<tr>
<td>S</td>
<td>z_1h_2</td>
<td>858.285</td>
<td>795.236</td>
<td>1.07928</td>
<td>712.487</td>
<td>665.310</td>
<td>1.07090</td>
</tr>
<tr>
<td>T</td>
<td>z_2h_2</td>
<td>710.971</td>
<td>654.030</td>
<td>1.08706</td>
<td>579.515</td>
<td>531.908</td>
<td>1.08950</td>
</tr>
<tr>
<td>U</td>
<td>z_1h_1</td>
<td>631.028</td>
<td>560.151</td>
<td>1.06227</td>
<td>505.763</td>
<td>476.114</td>
<td>1.12653</td>
</tr>
<tr>
<td>V</td>
<td>z_2h_1</td>
<td>669.129</td>
<td>584.021</td>
<td>1.14572</td>
<td>554.427</td>
<td>490.608</td>
<td>1.13008</td>
</tr>
<tr>
<td>W</td>
<td>z_1h_2</td>
<td>721.215</td>
<td>629.733</td>
<td>1.14527</td>
<td>572.760</td>
<td>516.417</td>
<td>1.10910</td>
</tr>
</tbody>
</table>
Geometric Ratio

Modified geometric ratio $g'$ is related to static submergence level $s_T$ by Equation 22. Experimental values of $g'$ and $s_T$ were fitted to this linear relation by the least squares procedure. Least squares estimates of the $\gamma_0$ and $\gamma_1$ values, as defined for Equation 22, are listed in Table 7 for the eight series of runs.

Table 7. Least squares estimate of $\gamma_0$ and $\gamma_1$ parameters of Equation 22

<table>
<thead>
<tr>
<th>Series in (32)</th>
<th>$z_i^{h_n}$</th>
<th>$\gamma_0$</th>
<th>$\gamma_1$</th>
<th>$F_{e,f}$</th>
<th>$F$</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>$z_2^{h_2}$</td>
<td>0.08548</td>
<td>0.003203</td>
<td>$F_{1,11}$</td>
<td>121.6*</td>
</tr>
<tr>
<td>Q</td>
<td>$z_1^{h_1}$</td>
<td>0.09371</td>
<td>0.002605</td>
<td>$F_{1,11}$</td>
<td>51.47*</td>
</tr>
<tr>
<td>R</td>
<td>$z_2^{h_1}$</td>
<td>0.10091</td>
<td>0.001965</td>
<td>$F_{1,11}$</td>
<td>10.21*</td>
</tr>
<tr>
<td>S</td>
<td>$z_1^{h_2}$</td>
<td>0.09909</td>
<td>0.002754</td>
<td>$F_{1,13}$</td>
<td>107.5*</td>
</tr>
<tr>
<td>T</td>
<td>$z_2^{h_2}$</td>
<td>0.06482</td>
<td>0.003755</td>
<td>$F_{1,12}$</td>
<td>53.44*</td>
</tr>
<tr>
<td>U</td>
<td>$z_1^{h_1}$</td>
<td>0.11098</td>
<td>0.001889</td>
<td>$F_{1,10}$</td>
<td>12.96*</td>
</tr>
<tr>
<td>V</td>
<td>$z_2^{h_1}$</td>
<td>0.08678</td>
<td>0.002605</td>
<td>$F_{1,11}$</td>
<td>31.30*</td>
</tr>
<tr>
<td>W</td>
<td>$z_1^{h_2}$</td>
<td>0.09902</td>
<td>0.002388</td>
<td>$F_{1,11}$</td>
<td>21.75*</td>
</tr>
</tbody>
</table>

*Significant at the 0.5% level.
Each set of data was tested statistically to determine whether the variation in observed $g'$ data was due primarily to variations in $s_T$. Results of the tests, in the form of F-ratios, are presented in Table 7, with the significance probability levels indicated.
APPENDIX B

The data presented here pertain to the Results of Entrainment Study section.

Entrainment Rate

Applicability of the different forms of the entrainment rate term, $E_t$, $E_t^*$ and $E$, requires a tabulation of the several forms. Since this is also true of the air rate terms, $v$, $v\rho^{0.5}$ and $G'$, Tables 8, 9, 10 and 11 contain multiple listings. Outlined calculations of the $E_t$ and $E$ data are presented in (32). The title of each of these tables lists the correspondence between series designation in this paper and the designation used in (32).

An evaluation of the effect of changes in liquid level on the calculated entrainment rate was made using Equation 23. The resulting corrected values, $(E_t)_c$, are tabulated along with the original $E_t$ data for sake of comparison.

Analysis of Variance

The terms effect and interaction have developed specific statistical meanings. This terminology will be discussed here, along with the concepts of the analysis of variance and the F-test of significance.
Table 8. Entrainment data for 9-inch tray spacing and 1/2-inch slot height, z₁h₁ (Series Q and U (32))

<table>
<thead>
<tr>
<th>v</th>
<th>v</th>
<th>ρ</th>
<th>G</th>
<th>Et</th>
<th>Et⁺</th>
<th>E</th>
<th>(Et) c</th>
</tr>
</thead>
<tbody>
<tr>
<td>s₁ = 7/8 inches</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>.908</td>
<td>.248</td>
<td>1.409</td>
<td>.00874</td>
<td>.0268</td>
<td>.00620</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>.895</td>
<td>.244</td>
<td>1.389</td>
<td>.00784</td>
<td>.0230</td>
<td>.00538</td>
<td>.0112</td>
<td></td>
</tr>
<tr>
<td>1.219</td>
<td>.333</td>
<td>1.891</td>
<td>.02615</td>
<td>.01383</td>
<td>.0263</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.205</td>
<td>.329</td>
<td>1.870</td>
<td>.04099</td>
<td>.1259</td>
<td>.02191</td>
<td>.0397</td>
<td></td>
</tr>
<tr>
<td>1.566</td>
<td>.428</td>
<td>2.430</td>
<td>.08179</td>
<td>.2512</td>
<td>.03365</td>
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</tr>
<tr>
<td>1.543</td>
<td>.421</td>
<td>2.393</td>
<td>.09149</td>
<td>.2810</td>
<td>.03823</td>
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</tr>
<tr>
<td>1.857</td>
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<tr>
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<td>.15497</td>
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<tr>
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<td>.2170</td>
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</tr>
<tr>
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<td>.603</td>
<td>3.425</td>
<td>.26579</td>
<td>.8162</td>
<td>.07760</td>
<td>.2697</td>
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</tr>
<tr>
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<td>.708</td>
<td>4.026</td>
<td>.26736</td>
<td>.8211</td>
<td>.06610</td>
<td>.2630</td>
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</tr>
<tr>
<td>2.475</td>
<td>.676</td>
<td>3.841</td>
<td>.19462</td>
<td>.5977</td>
<td>.05066</td>
<td>.1938</td>
<td></td>
</tr>
<tr>
<td>s₂ = 1 1/2 inches</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>.641</td>
<td>.175</td>
<td>.994</td>
<td>.00183</td>
<td>.00562</td>
<td>.00184</td>
<td>.0028</td>
<td></td>
</tr>
<tr>
<td>.903</td>
<td>.247</td>
<td>1.401</td>
<td>.02668</td>
<td>.0819</td>
<td>.01901</td>
<td>.0287</td>
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</tr>
<tr>
<td>.922</td>
<td>.252</td>
<td>1.430</td>
<td>.04180</td>
<td>.1284</td>
<td>.02923</td>
<td>.0290</td>
<td></td>
</tr>
<tr>
<td>1.221</td>
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<td>.08638</td>
<td>.2653</td>
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<tr>
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<td>.331</td>
<td>1.879</td>
<td>.07954</td>
<td>.2412</td>
<td>.04179</td>
<td>.0717</td>
<td></td>
</tr>
<tr>
<td>1.536</td>
<td>.419</td>
<td>2.383</td>
<td>.17492</td>
<td>.5372</td>
<td>.07340</td>
<td>.1522</td>
<td></td>
</tr>
<tr>
<td>1.522</td>
<td>.416</td>
<td>2.362</td>
<td>.14499</td>
<td>.4754</td>
<td>.06307</td>
<td>.1452</td>
<td></td>
</tr>
<tr>
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<td>2.930</td>
<td>.24230</td>
<td>.7072</td>
<td>.08269</td>
<td>.2481</td>
<td></td>
</tr>
<tr>
<td>1.891</td>
<td>.516</td>
<td>2.935</td>
<td>.24573</td>
<td>.7546</td>
<td>.08372</td>
<td>.2435</td>
<td></td>
</tr>
<tr>
<td>2.245</td>
<td>.613</td>
<td>3.848</td>
<td>.40163</td>
<td>1.233</td>
<td>.11527</td>
<td>.4002</td>
<td></td>
</tr>
<tr>
<td>2.226</td>
<td>.608</td>
<td>3.456</td>
<td>.44673</td>
<td>1.372</td>
<td>.12926</td>
<td>.4788</td>
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<td>2.550</td>
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<td>3.956</td>
<td>.54974</td>
<td>1.688</td>
<td>.13896</td>
<td>.4963</td>
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<td>2.554</td>
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<td>3.963</td>
<td>.70974</td>
<td>2.180</td>
<td>.17909</td>
<td>.7360</td>
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</tbody>
</table>
Table 9. Entrainment data for 9-inch tray spacing and 1-inch slot height, $z_{1,h_2}$ (Series S and W (32))

<table>
<thead>
<tr>
<th>$v$</th>
<th>$v^0.5$</th>
<th>$G'$</th>
<th>$E_t$</th>
<th>$E^*$</th>
<th>$E$</th>
<th>$(E_t)_g$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.915</td>
<td>0.250</td>
<td>1.420</td>
<td>0.01167</td>
<td>0.0358</td>
<td>0.00821</td>
<td>0.0128</td>
</tr>
<tr>
<td>0.913</td>
<td>0.249</td>
<td>1.417</td>
<td>0.00774</td>
<td>0.0238</td>
<td>0.00516</td>
<td>0.0091</td>
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<tr>
<td>1.201</td>
<td>0.328</td>
<td>1.863</td>
<td>0.04213</td>
<td>0.1294</td>
<td>0.02261</td>
<td>0.0417</td>
</tr>
<tr>
<td>1.211</td>
<td>0.381</td>
<td>1.879</td>
<td>0.03087</td>
<td>0.0948</td>
<td>0.01642</td>
<td>-</td>
</tr>
<tr>
<td>1.532</td>
<td>0.418</td>
<td>2.378</td>
<td>0.10364</td>
<td>0.3183</td>
<td>0.06358</td>
<td>0.1060</td>
</tr>
<tr>
<td>1.559</td>
<td>0.425</td>
<td>2.396</td>
<td>0.09482</td>
<td>0.2912</td>
<td>0.03923</td>
<td>0.0482</td>
</tr>
<tr>
<td>1.894</td>
<td>0.517</td>
<td>2.938</td>
<td>0.15941</td>
<td>0.4895</td>
<td>0.05425</td>
<td>0.2150</td>
</tr>
<tr>
<td>1.887</td>
<td>0.515</td>
<td>2.928</td>
<td>0.20872</td>
<td>0.6110</td>
<td>0.07128</td>
<td>0.2172</td>
</tr>
<tr>
<td>2.239</td>
<td>0.611</td>
<td>3.475</td>
<td>0.34193</td>
<td>1.050</td>
<td>0.09839</td>
<td>0.3387</td>
</tr>
<tr>
<td>2.132</td>
<td>0.582</td>
<td>3.308</td>
<td>0.25166</td>
<td>0.7728</td>
<td>0.07607</td>
<td>0.2779</td>
</tr>
<tr>
<td>2.533</td>
<td>0.692</td>
<td>3.931</td>
<td>0.60515</td>
<td>1.859</td>
<td>0.15401</td>
<td>0.6225</td>
</tr>
<tr>
<td>2.564</td>
<td>0.700</td>
<td>3.978</td>
<td>0.76414</td>
<td>2.347</td>
<td>0.19209</td>
<td>0.7783</td>
</tr>
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</table>

$s_1 = \frac{7}{8}$ inches

<table>
<thead>
<tr>
<th>$v$</th>
<th>$v^0.5$</th>
<th>$G'$</th>
<th>$E_t$</th>
<th>$E^*$</th>
<th>$E$</th>
<th>$(E_t)_g$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.623</td>
<td>0.170</td>
<td>0.996</td>
<td>0.00232</td>
<td>0.00712</td>
<td>0.00240</td>
<td>0.0025</td>
</tr>
<tr>
<td>0.907</td>
<td>0.218</td>
<td>1.408</td>
<td>0.05794</td>
<td>0.1779</td>
<td>0.14115</td>
<td>0.0589</td>
</tr>
<tr>
<td>0.900</td>
<td>0.216</td>
<td>1.397</td>
<td>0.07046</td>
<td>0.2164</td>
<td>0.15043</td>
<td>-</td>
</tr>
<tr>
<td>1.227</td>
<td>0.335</td>
<td>1.901</td>
<td>0.27302</td>
<td>0.8384</td>
<td>0.11339</td>
<td>0.2349</td>
</tr>
<tr>
<td>1.190</td>
<td>0.325</td>
<td>1.846</td>
<td>0.13372</td>
<td>0.4106</td>
<td>0.07243</td>
<td>0.12292</td>
</tr>
<tr>
<td>1.545</td>
<td>0.422</td>
<td>2.397</td>
<td>0.29373</td>
<td>0.9020</td>
<td>0.12624</td>
<td>0.2808</td>
</tr>
<tr>
<td>1.514</td>
<td>0.422</td>
<td>2.396</td>
<td>0.36599</td>
<td>1.124</td>
<td>0.15275</td>
<td>-</td>
</tr>
<tr>
<td>1.895</td>
<td>0.517</td>
<td>2.940</td>
<td>0.54215</td>
<td>1.666</td>
<td>0.18450</td>
<td>0.5136</td>
</tr>
<tr>
<td>1.875</td>
<td>0.512</td>
<td>2.910</td>
<td>0.53654</td>
<td>1.648</td>
<td>0.18437</td>
<td>0.6273</td>
</tr>
<tr>
<td>2.256</td>
<td>0.616</td>
<td>3.500</td>
<td>0.81638</td>
<td>2.507</td>
<td>0.23325</td>
<td>0.7942</td>
</tr>
<tr>
<td>2.103</td>
<td>0.574</td>
<td>3.263</td>
<td>0.73809</td>
<td>2.267</td>
<td>0.22169</td>
<td>0.6895</td>
</tr>
<tr>
<td>2.562</td>
<td>0.699</td>
<td>3.975</td>
<td>1.11616</td>
<td>3.428</td>
<td>0.28079</td>
<td>1.0277</td>
</tr>
<tr>
<td>2.537</td>
<td>0.693</td>
<td>3.936</td>
<td>1.32644</td>
<td>4.075</td>
<td>0.33700</td>
<td>1.2455</td>
</tr>
</tbody>
</table>
Table 10. Entrainment data for 12-inch tray spacing and 1/2-inch slot height, \( z_{2h_1} \) (Series R and V (32))

<table>
<thead>
<tr>
<th>( v )</th>
<th>( \sqrt{\rho} )</th>
<th>( G' )</th>
<th>( E_t )</th>
<th>( E^* )</th>
<th>( E )</th>
<th>( (E_t)_c )</th>
</tr>
</thead>
<tbody>
<tr>
<td>.907</td>
<td>.248</td>
<td>1.407</td>
<td>.00340</td>
<td>.0104</td>
<td>.00241</td>
<td>.0036</td>
</tr>
<tr>
<td>.904</td>
<td>.247</td>
<td>1.403</td>
<td>.00296</td>
<td>.0091</td>
<td>.00210</td>
<td>.0038</td>
</tr>
<tr>
<td>1.226</td>
<td>.335</td>
<td>1.902</td>
<td>.00966</td>
<td>.0296</td>
<td>.00507</td>
<td>.0098</td>
</tr>
<tr>
<td>1.212</td>
<td>.331</td>
<td>1.880</td>
<td>.01588</td>
<td>.0488</td>
<td>.00844</td>
<td>.0167</td>
</tr>
<tr>
<td>1.555</td>
<td>.425</td>
<td>2.413</td>
<td>.01642</td>
<td>.0504</td>
<td>.00680</td>
<td>.0156</td>
</tr>
<tr>
<td>1.534</td>
<td>.419</td>
<td>2.380</td>
<td>.02576</td>
<td>.0791</td>
<td>.01082</td>
<td>.0266</td>
</tr>
<tr>
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<td>.513</td>
<td>2.918</td>
<td>.04771</td>
<td>.1465</td>
<td>.01635</td>
<td>.0505</td>
</tr>
<tr>
<td>1.878</td>
<td>.513</td>
<td>2.915</td>
<td>.05568</td>
<td>.1710</td>
<td>.01910</td>
<td>.0596</td>
</tr>
<tr>
<td>2.229</td>
<td>.609</td>
<td>3.459</td>
<td>.08029</td>
<td>.2466</td>
<td>.02321</td>
<td>.0822</td>
</tr>
<tr>
<td>2.217</td>
<td>.605</td>
<td>3.439</td>
<td>.08374</td>
<td>.4015</td>
<td>.02435</td>
<td></td>
</tr>
<tr>
<td>2.527</td>
<td>.590</td>
<td>3.921</td>
<td>.09591</td>
<td>.2945</td>
<td>.02446</td>
<td>.0987</td>
</tr>
<tr>
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<td>.695</td>
<td>3.953</td>
<td>.10217</td>
<td>.3176</td>
<td>.02584</td>
<td>.0847</td>
</tr>
</tbody>
</table>

\( s_1 = 7/8 \) inches

| .626  | .171   | .971 | .00129| .00396| .00132| .0026  |
| .910  | .248   | 1.411| .01022| .0314 | .00724| .0116  |
| .907  | .247   | 1.407| .01520| .0467 | .01080| .0188  |
| 1.211 | .331   | 1.879| .02972| .0913 | .01581| .0312  |
| 1.235 | .337   | 1.916| .03525| .1082 | .01839| .0406  |
| 1.537 | .420   | 2.385| .04067| .1249 | .01705| .0414  |
| 1.521 | .415   | 2.359| .04781| .1468 | .02026| .0483  |
| 1.892 | .517   | 2.936| .06286| .1930 | .02141| .0619  |
| 1.878 | .513   | 2.914| .07660| .2353 | .02628| .1088  |
| 2.247 | .613   | 3.486| .11537| .3543 | .03309| .1223  |
| 2.178 | .595   | 3.380| .13089| .4013 | .03866| .1343  |
| 2.589 | .707   | 4.017| .15170| .4659 | .03776| .1529  |
| 2.661 | .726   | 4.129| .21691| .7583 | .05979| .1827  |

\( s_2 = 1 \frac{1}{2} \) inches
Table 11. Entrainment data for 12-inch tray spacing and 1-inch slot height, $z_2h_2$ (Series P and T (32))

<table>
<thead>
<tr>
<th>$v$</th>
<th>$vP^{0.5}$</th>
<th>$G'$</th>
<th>$E_t$</th>
<th>$E^\infty$</th>
<th>$E$</th>
<th>$(E_t)_c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$s_1 = 7/8$ inches</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.914</td>
<td>250</td>
<td>1.418</td>
<td>0.00976</td>
<td>0.01000</td>
<td>0.00688</td>
<td>0.0098</td>
</tr>
<tr>
<td>0.899</td>
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<td>0.01060</td>
<td>0.00240</td>
<td>0.0035</td>
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<tr>
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<td>341</td>
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<td>0.05390</td>
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<td>0.0236</td>
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<tr>
<td>1.208</td>
<td>330</td>
<td>1.875</td>
<td>0.01582</td>
<td>0.04860</td>
<td>0.00843</td>
<td>0.0155</td>
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<tr>
<td>1.552</td>
<td>424</td>
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<td>0.13890</td>
<td>0.01877</td>
<td>0.0501</td>
</tr>
<tr>
<td>1.534</td>
<td>419</td>
<td>2.380</td>
<td>0.03583</td>
<td>0.11000</td>
<td>0.01505</td>
<td>0.0476</td>
</tr>
<tr>
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<td>511</td>
<td>2.907</td>
<td>0.10214</td>
<td>0.31860</td>
<td>0.03528</td>
<td>0.1170</td>
</tr>
<tr>
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<td>2.907</td>
<td>0.05895</td>
<td>0.18100</td>
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</tr>
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</tr>
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<td>0.05794</td>
<td>0.2524</td>
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<tr>
<td>2.578</td>
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<td>0.90080</td>
<td>0.07331</td>
<td>0.2908</td>
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<tr>
<td>$s_2 = 1 1/2$ inches</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>0.638</td>
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<td>0.00485</td>
<td>0.00160</td>
<td>0.0016</td>
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<tr>
<td>0.628</td>
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<td>0.00273</td>
<td>0.00838</td>
<td>0.00280</td>
<td>0.0027</td>
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<tr>
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<td>0.03550</td>
<td>0.0453</td>
</tr>
<tr>
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<td>0.02814</td>
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<td>0.03161</td>
<td>0.0572</td>
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<td>0.03912</td>
<td>0.0727</td>
</tr>
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<td>0.1399</td>
</tr>
<tr>
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</tr>
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<td>0.3117</td>
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<tr>
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<td>0.4680</td>
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<tr>
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<td>0.66693</td>
<td>2.04800</td>
<td>0.16854</td>
<td>0.4530</td>
</tr>
</tbody>
</table>
Effect and interaction

Effect of a variable is defined simply as the difference between entrainment rate at one level of the variable and the entrainment rate at the second level, averaged over all levels of the other variables. (This refers specifically to variables at two levels. A definition of effect for a six level variable is rather complex, and will be left only as an intuitive extension of the definition of effect given.) The question which effect answers is this: did the change from one level of the variable to the second level cause a change in entrainment rate? Such a change can be either positive or negative once the uniform practice of comparing the higher level with the lower level is adopted. The effect of tray spacing, \( Z \), from Figures 15 and 17, obviously, is negative.

Interaction refers to a dependence of the variables. Specifically interaction is the differential effect of the levels of one variable over several levels of the other variable(s). The effect of a variable which interacts then is dependent on the particular combination of the other variables with which they are produced. An extension of the simple, first-order interaction to higher-order interactions considers the differential effect of the combination of groups of variables over several levels of another variable.
As an example of a first-order interaction, consider the SZ interaction. This means that the effect on entrainment rate brought about by changing from $s_1$ to $s_2$ is different at the $z_1$ level than the effect brought about by the same change at the $z_2$ level. Alternately this also means that the effect on entrainment rate brought about by changing from $z_1$ to $z_2$ at the $s_1$ level is different from the effect brought about by this change at the $s_2$ level, with the result that

$$SH = HS.$$ 

Analysis of variance and F-test

The analysis of variance is basically a procedure for partitioning the total sum of squares according to the sources of variation present in the experiment. These sources of variation arise from the forty eight treatment combinations and the replication procedure, and are listed in Table 12. The portion of the total sum of squares assigned to a source of variation, or component, is termed the sum of squares "for" this variation, e.g., the sum of squares assigned to the variation in tray spacing is denoted by the sum of squares "for" $Z$. Any residual from the assignment to all of the possible sources of variation is termed the error, or residual, sum of squares. Mean squares for each component are obtained by dividing the individual sum of squares by their respective degrees of freedom. The
Table 12. Degrees of freedom for the analysis of variance

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>Degrees of freedom</th>
</tr>
</thead>
<tbody>
<tr>
<td>Replicates</td>
<td>(r-1)</td>
</tr>
<tr>
<td>Z</td>
<td>(m-1)</td>
</tr>
<tr>
<td>H</td>
<td>(n-1)</td>
</tr>
<tr>
<td>S</td>
<td>(j-1)</td>
</tr>
<tr>
<td>V</td>
<td>(k-1)</td>
</tr>
<tr>
<td>ZH</td>
<td>(m-1)(n-1)</td>
</tr>
<tr>
<td>ZS</td>
<td>(m-1)(j-1)</td>
</tr>
<tr>
<td>ZV</td>
<td>(m-1)(k-1)</td>
</tr>
<tr>
<td>HS</td>
<td>(n-1)(j-1)</td>
</tr>
<tr>
<td>HV</td>
<td>(n-1)(k-1)</td>
</tr>
<tr>
<td>SV</td>
<td>(j-1)(k-1)</td>
</tr>
<tr>
<td>ZHS</td>
<td>(m-1)(n-1)(j-1)</td>
</tr>
<tr>
<td>ZHV</td>
<td>(m-1)(n-1)(k-1)</td>
</tr>
<tr>
<td>ZSV</td>
<td>(m-1)(j-1)(k-1)</td>
</tr>
<tr>
<td>HSV</td>
<td>(n-1)(j-1)(k-1)</td>
</tr>
<tr>
<td>ZHSV</td>
<td>(m-1)(n-1)(j-1)(k-1)</td>
</tr>
<tr>
<td>Error (a)</td>
<td>by difference (in main plots) 3</td>
</tr>
<tr>
<td>Error (b)</td>
<td>by difference (in sub-plots) 44</td>
</tr>
<tr>
<td>Total</td>
<td>(rmnjk-1)</td>
</tr>
</tbody>
</table>

degrees of freedom associated with the various components are listed in Table 12. The tabulation of the components, or source of variation, with their respective degrees of freedom, sums of squares and mean squares, is popularly referred to as "the analysis of variance" of the data.

The statistical test of significance used in this work is the F-test. The F-test consists of forming the ratio of the mean square for a given source of variation to the error
mean square and then determining the probability of obtaining an F as large or larger than the calculated value. This probability is determined from the F-distribution with the source and error degrees of freedom as parameters and under the hypothesis that the component or source of variation produced no effect. Values of F corresponding to several probability levels, 0.5, 1.0, 2.5, 5.0, 10.0 and 25%, are tabulated in (40). When a test is significant at the 0.5% level, for example, as denoted by the F-test, the hypothesis that the component produced no effect is rejected with the condition that the hypothesis will be rejected once in two hundred times when it is in fact true. The level of probability used in the various tests of significance in this work is noted with each value of the F-ratio.

Details of the calculations involved in the analysis of variance for a split-plot design are given by Cochran and Cox (33). A complete tabulation of the analysis of variance calculations for the results listed in Tables 2a, 2b and 3 is given in (32).