ENTRAINMENT AND FLOODING IN VERTICAL STILL-HEADS

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An investigation has been made of the entrainment entering, and collected on, vertical still-heads from a boiling solution. The entrainment entering the still-heads increased gradually with increasing distillation rate until the Reynolds Number of the vapour in the still head reached a value of about 10,000. At this point a much greater rate of increase was noted and was accompanied by a greater rate of collection of the entrainment over all sections of the still-head. The value of 10,000 for the Reynolds Number was verified from the observations of others. The true flooding effect did not occur until much higher distillation rates, when there was a sudden increase in the volume of liquid collected at the top of the still-head and a corresponding decrease in that collected at the bottom. Increasing the length of the still-head from 30 to 60 inches showed an increase in maximum collecting efficiency from approximately 96 to 99 per cent. Decontamination factors compared favourably with those obtained by other workers using more complex packed columns.

When boiling any liquid, it is invariably found that the vapour immediately above the liquid surface contains varying proportions of droplets of the original liquid. The carriage of these droplets by the vapour stream is now generally referred to as entrainment, and it is in this context that the word is used throughout this paper.

The work was an extension of that of Shotton and Habeeb (1954) and Train and Velasquez-Guerrero (1957). They were concerned with the estimation of entrainment which had passed through a still-head of known characteristics, and had been collected in a centrifugal separator. Habeeb (1954) made an attempt to collect the liquid caught on the walls of the still-head but the results were found to be variable and could not be related to the distillation rate. This variability was due to an inadequate trapping channel, and to the design of the apparatus which allowed some direct splashing of the boiler liquid to a point above the channel.

Garner, Ellis and Lacey's work (1954), on the other hand, involved mainly an estimation of size and number of entrained liquid droplets. It is difficult, if not impossible, to give a full estimate of the total entrainment, since samples cannot be obtained from immediately above the surface of the boiling liquid. The higher the sampling level above the boiling liquid surface, the smaller will be the estimate of "total" entrainment, since a certain proportion of the droplets projected from the surface will fall back into the boiling liquid.

The principal aims of this work therefore were to study the "total" entrainment passing into given still-heads at various distillation rates and to determine the proportions collected over various sections of the still-head, together with that which passed through to a centrifugal separator.

Experimental

Apparatus

The design of the apparatus was based on that used by Shotton and Habeeb (1954) and a flow diagram is given in Fig. 1. The capacity was much increased so that conditions resembled those obtained in an industrial pilot-scale plant. Higher steam rates were used to cover a complete range of flow from the streamline conditions referred to by Shotton and Habeeb (1954), through the turbulent region to a point equivalent to full flooding conditions in a wetted-wall column.

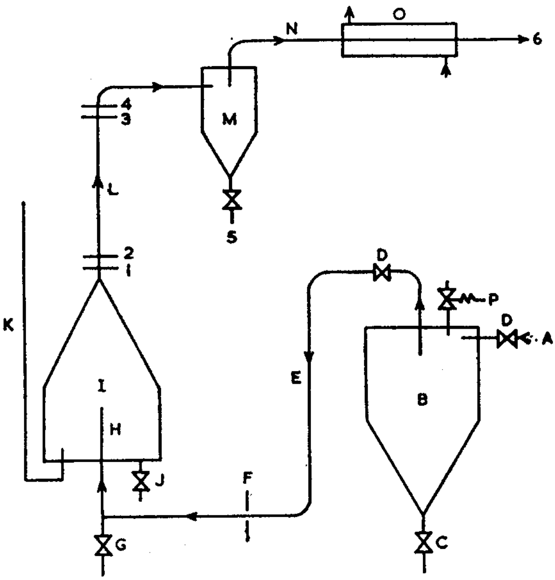

FIG. 1. Flow diagram of apparatus.

A — $\frac{3}{4}$ in. steam supply.
B — Expansion vessel.
C — Drain valve and steam trap.
D — Control valves.
E — 2 in. steam line.
F — 1 in. orifice.
G — Drain valve.
H — Steam injector.
I — Cone and boiler.
J — Drain valve.
K — Filling tube and level indicator.
L — Still-head.
M — Centrifugal separator.
N — 2 in. vapour line.
O — Tube and shell condenser.
P — Safety valve.
1, 2, 3, 4, 5—Entrainment collecting points.
6 — Condensate.

The boiler was of stainless steel with a total capacity of approximately 20 l. Vapour passed up through a cone, forming the upper part of the still into a 2 in. diameter still-head without obstruction to its free flow. At the base of the still-head were situated two collecting rings which were integrated in what is termed a collecting unit (Fig. 2). This unit was so designed that the lower ring S (collecting point 1, Fig. 1) collected any liquid rising up the wall of the cone and prevented its further passage up the still-head. The upper ring T (collecting point 2) was intended to collect any entrainment plus condensate caught on the walls of the still-head, which subsequently drained downwards, under the influence of gravity. This unit was designed to facilitate liquid collection without interfering with the flow pattern of the continuous steam phase.

A similar collecting unit was inserted at the top of the still-head, the lower part (collecting point 3) to trap any climbing film from the still-head and the upper part (collecting point 4) for entrainment plus condensate caught on and near the close angle bend just above it which would normally have drained back into the still-head. This general arrangement enabled trapping areas to be defined, and also showed whether liquid on the wall was moving up or down the still-head, under given flow conditions.

Two metal still-heads were used, giving a distance between the centres of the collecting units of 30 in. or 60 in. respectively; in addition a 30 in

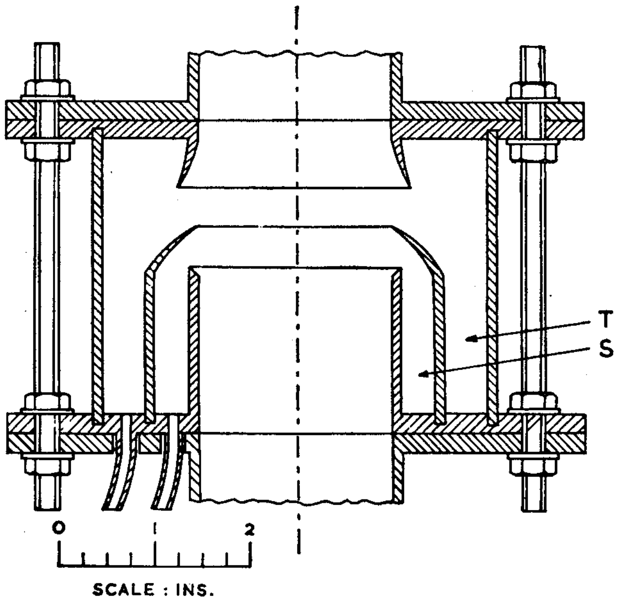

FIG. 2. Still-head collecting unit.

Pyrex glass still-head of the same diameter was also used to enable visual observations to be made. Experiments were also made with the close angle bend connected directly to the lower collecting unit.

Steam was introduced into the boiler using a steam sparger designed to eliminate violent bubbling action of the entering steam and to prevent loss of boiler contents when the steam was not flowing. The bulk of the apparatus, consisting of the steam sparger, cone, still-heads, collecting units, separator and their connecting pipes, was given a dull chrome finish to avoid contamination of the fluids contained in them, and to provide a surface which could be completely wetted. The apparatus was lagged to reduce the condensation, although it was appreciated that some condensate was necessary in order that efficient collection of the entrained droplets could be ensured.

Procedure

The method of estimation of entrainment was essentially the same as that used by Shotton and Habeeb (1954) and by Train and Velasquez-Guerrero (1957). This consisted of the use of a boiler solution containing 0·1 per cent (≡1,000 μg./ml.) of Fluorescein Sodium B.P. By this means droplets of original solution, which were entrained and subsequently

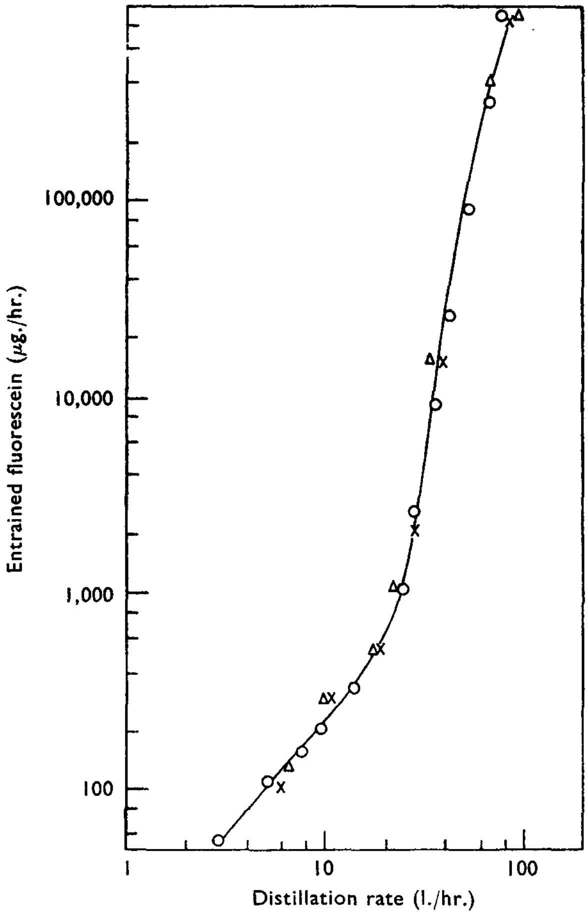

FIG. 3. "Total" entrainment collected—from points 2, 3, 4, 5 and 6.
X "Blank" still-head.
○ 30 in. still-head.
△ 60 in. still-head.

collected over various sections of the apparatus, would mix with any condensate collected from the same section, and form a comparatively dilute solution, on which a quantitative estimate of fluorescein content could be made by means of a Spekker Fluorimeter. This method of estimation has been fully described by Shotton and Habeeb (1955).

The still was initially heated by two 1 kilowatt heating elements for approximately 30 min. or until the solution was boiling, the apparatus warmed through and some condensate obtained. The liquid in the boiler was then readjusted to a predetermined level, the heaters switched off, and the steam introduced at the required rate. 30 min. was allowed for the

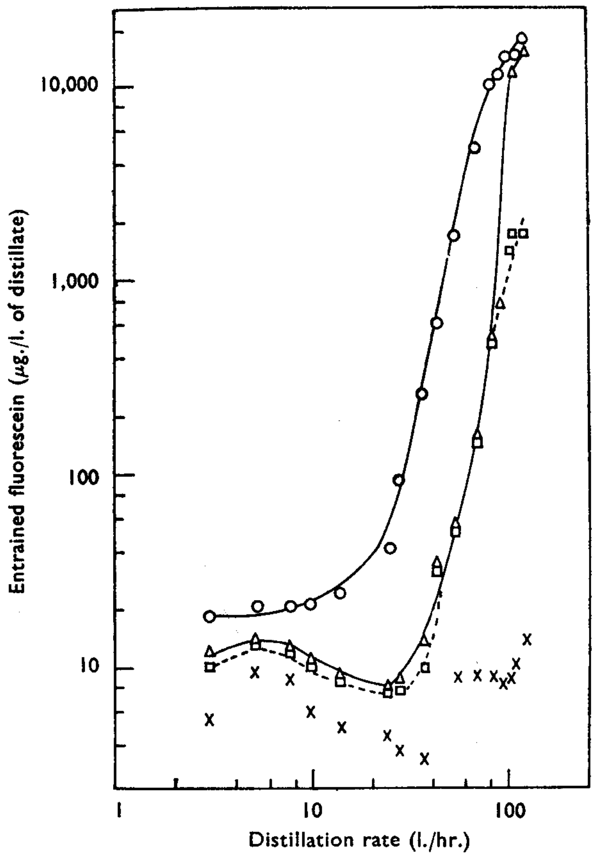

FIG. 4. Entrainment collected from succeeding sections of 30 in. still-head.
○ From points 2 + 3 + 4 + 5 + 6.
△ From points 3 + 4 + 5 + 6.
□ From points 5 + 6.
X From point 6.

system to reach equilibrium and an experimental run made during the succeeding 30 min. At the end of a run the solutions from the five collecting points and the condensate were measured and retained for fluorimetric estimation, and the liquid level in the still checked.

Entrainment was calculated as the total amount of fluorescein collected over the various parts of the still-head in μg./hr. All figures for entrained fluorescein were related to a constant concentration of 1,000 μg./ml. in the still throughout each individual experiment (Evans, 1961).

The rate of distillation was determined from the sum of the volumes of solution collected at points 2, 3, 4 and 5, together with the final distillate. Solution collected from point 1 was not included, since, particularly at the high rates of distillation, the bulk of it entered the collecting duct as a result of a climbing film effect up the cone from the boiling solution, and not as true condensate.

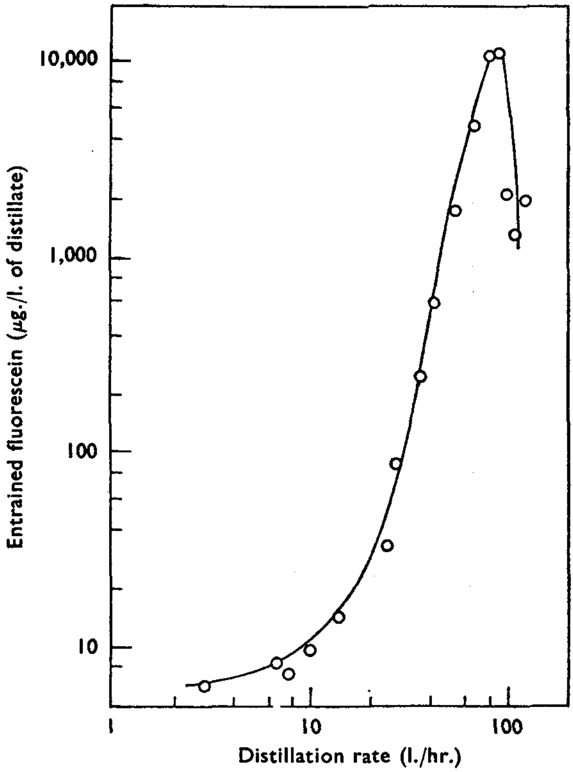

FIG. 5. Entrainment collected from bottom of 30 in. still-head (Point 2).

Quantities of liquid held on the still-head surface at distillation rates near the flooding point were determined by rapidly shutting off the steam supply and collecting the liquid draining down the still-head.

RESULTS

Entrainment entering the three still-heads over the full flow range used in the experiments is shown in Fig. 3, showing that the entrainment passing from the boiler into the various still-heads was constant for a given rate of distillation. The entrainment passing through to succeeding sections of the apparatus is exemplified by the results shown in Fig. 4 using the 30 in. still-head. Detailed results of other lengths are available (Evans, 1961). The entrainment collected on any particular section is represented by the vertical height between adjoining plots, and the amount falling down the still-head surface is shown in Fig. 5. The changes in mass movement of liquid on the wall of the still-head are tabulated in Table I, and the movement could also be seen in the glass still-head, corresponding to the normal flooding conditions in a wetted-wall column.

TABLE I

VOLUMES COLLECTED ON 30 IN. STILL-HEAD

Distillation rate (l./hr.)	Collecting points 2 (ml./hr.)	Collecting points 3 (ml./hr.)	Reynolds Number of vapour
2·9	81	24	1,617
13·86	76	13	7,720
24·08	77	16	13,420
35·75	87	18	19,920
53·01	174	31	29,500
68·32	390	33	38,050
82·15	920	36	45,750
89·9	988	39	50,050
91·16	1,074	30	50,800
97·4	532*	568	54,300
101·1	300	1,052	56,300
107·82	216	1,116	60,500

* Hold-up volume of liquid on still-heads at flooding rate = 9·7 ml.

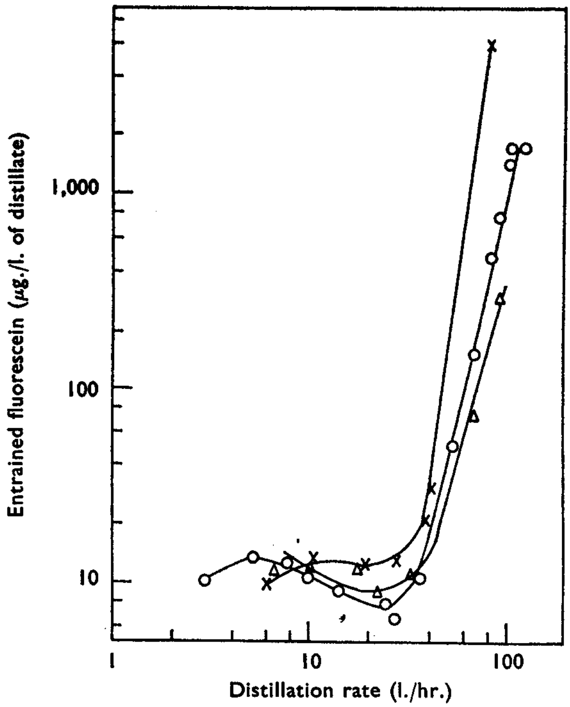

FIG. 6. Entrainment passing through still-heads.
X "Blank" still-head.
O 30 in. still-head.
Δ 60 in. still-head.

An example of the hold-up volumes of liquid held on the still-head at the flooding rate is given as a footnote to Table I.

It was noticed in the glass still-head at distillation rates of about 20 litre/hr. that there was a wave motion on the surface of the liquid film on the wall; the wave motion ascended the column against the downward flow of the liquid due to gravity.

The entrainment passing through the various length still-heads is shown in Fig. 6.

Discussion

This work has shown that the total entrainment entering three different length still-heads is substantially the same for a given rate of distillation

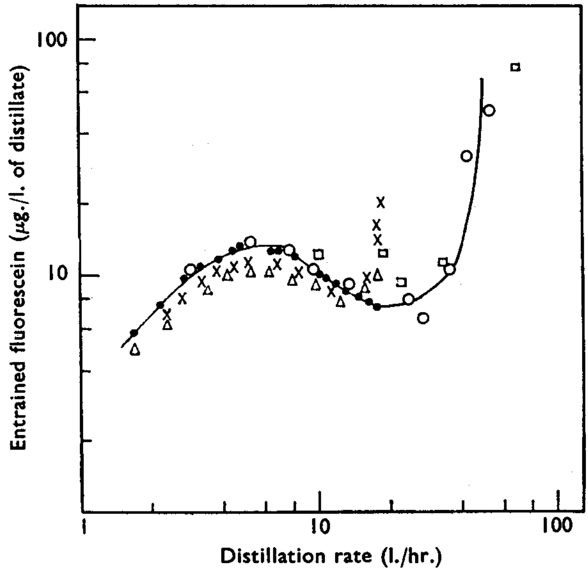

FIG. 7. Correlation of results of entrainment passing through various still-heads.

× 10 in. still-head }
△ 15 in. still-head } Shotton and Habeeb (1954).
● 30 in. still-head }

○ 30 in. still-head }
□ 60 in. still-head } This work.

(Fig. 3), thus verifying the assumption of both Shotton and Habeeb (1954) and Train and Velasquez-Guerrero (1957). The plot of the entrainment from points 5 and 6 (Fig. 1) for the 30 in. still-head is similar to that obtained by Shotton and Habeeb (1954) for their 30 in. × 2 in. still-head, even though the apparatus differed. A direct comparison of these results is given in Fig. 7. The comparison could not be drawn directly from the figures for entrainment collected in the centrifugal separators, since at low distillation rates the separator used in this work was not working at its maximum efficiency and a certain proportion passed into the condensate.

The entrainment passing over into the separator and condenser for all three still-heads is shown in Fig. 6, and it may be seen that the entrainment per unit volume of distillate passing through the still-head decreased within the range 10–30 litre/hr. It has already been shown in Fig. 3, however, that the "total" entrainment per unit time continually increases, and as a corollary to this the entrainment collected in unit time from the still-head, that is, the summation from points 2 and 3 (Fig. 1), also continually increased.

It may be seen from Fig. 6 that the entrainment passing completely through the 60 in. still-head is greater than that through the 30 in. below a distillation rate of 35 litre/hr. This apparent anomaly is similar to that reported by Shotton and Habeeb (1954), who found that although, in general, entrainment passing through the still-heads decreased with increasing length, the material passing through their 30 in. still-head was greater than that passing through shorter length still-heads over lower ranges of distillation rates.

These apparent anomalies in both this and Shotton and Habeeb's work are thought to be due to the development of a turbulent boundary layer of flowing steam at the upper ends of the longer still-heads. In the presence of turbulent eddies of vapour, any entrained droplets are liable to be given a radial component of velocity and thus would proceed in the general direction of the still-head wall. Only an opposing eddy would alter this radial direction and if, therefore, the droplet entered a streamline boundary layer it would continue along a path approximate normal to axial flow and thus be caught on the walls of the still-head.

As full turbulent conditions are progressively developed in the tube and the annular streamline boundary layer is reduced to the buffer and laminar sub-layers, there is the increasing possibility of the droplets being deflected by further eddies which effectively hold them within the main vapour stream, and therefore reduce their chance of being caught on the walls. The presence of a turbulent boundary layer may also result in the re-entrainment of previously collected droplets because of the turbulent action of the flowing steam on the boundary layers of *liquid* on the walls of the still-head.

It has been shown by both Goldstein (1938) and Coulson and Richardson (1955a), in their textbooks, that a transition takes place at some critical value of the Reynolds number involving the distance from the leading edge. This modified Reynolds number is usually denoted by Re_x, where the length factor x is the distance from the leading edge (in this example the entry point into the still-head) and x replaces the more usual diameter dimension.

The critical value of Re_x for the development of a turbulent boundary layer is usually considered to be about 10^5, although values as high as $1{\cdot}1 \times 10^6$ have been observed, depending on the disturbances present and on the conditions of entry at the leading edge.

It may be expected, therefore, that the longer the still-head the greater the chance of the development of a turbulent boundary layer resulting in the re-entrainment of liquid as described above.

When considering the visual observations, in the glass still-head the liquid caught on the walls did not immediately drain down, but quite large volumes oscillated over several centimetres. The effect of this oscillation, plus any wave formation on the surface, would greatly enhance the possibility of re-entrainment in this region.

"Climbing Film Effect"

To avoid any discrepancies due to variation in the quantity of total entrainment entering the still-head, the efficiency of collection of the three still-heads was represented by Fig. 8. The efficiency was approximately

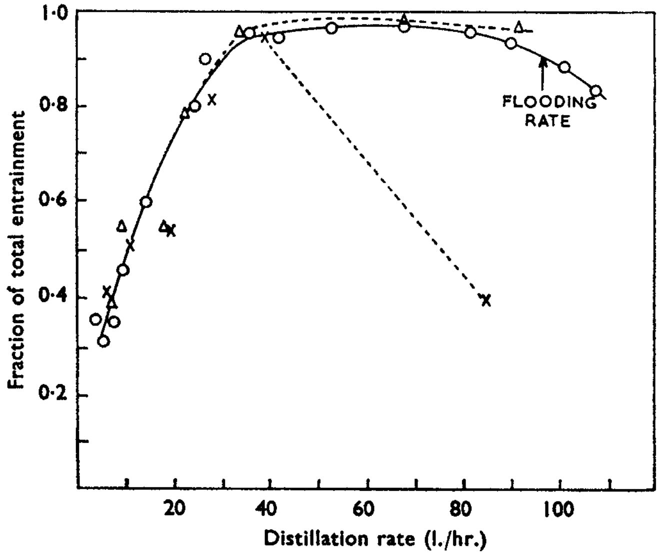

FIG. 8. Fraction of "total" entrainment caught on walls of still-head.
X "Blank" still-head.
O 30 in. still-head.
△ 60 in. still-head.

the same in all still-heads for distillation rates up to 20–30 litre/hr. Just before the onset of flooding conditions within the still-heads, the efficiency of collection begins to decrease. Since the onset of flooding indicates that the vapour flowing through the still-head is capable of exerting sufficient drag force on the liquid on the walls to reverse its direction completely, it is probable that this reduction in efficiency is a result of re-entrainment of some of the collected liquid into the vapour stream. The apparent sudden decrease in efficiency of the "blank" still-head is not strictly comparable with the results from the other still-heads because only one set of collecting ducts was used, corresponding to points 1 and 4, and liquid which was either re-entrained or reversed in its direction of flow would be passing directly into the centrifugal separator (point 5, Fig. 1).

The sudden increase in entrainment collected at points 5 and 6 (Fig. 5) can be compared with the increases found by Shotton and Habeeb (1954) and Train and Velasquez-Guerrero (1957). They found that in all sizes of still-head they used, the increase in entrainment passing through the still-heads began when the distillation rate corresponded to a value of the Reynolds number of the vapour in the tube in the range 9,000–10,000 (Habeeb, 1954, p. 82). In our work the increase began at distillation rate equivalent to an *R*e value of approximately 11,000. These previous workers referred to the increase in entrainment collection in the separator as the beginning of a climbing film effect or gross carry-over of fluorescein solution.

Our results show that the "climbing film effect" was not present at these distillation rates, and that mass movement of liquid on the wall of the still-head was still downwards (Table I). The apparent "climbing film effect" is due to wave motion on the surface of the liquid film on the

TABLE II

VELOCITIES AT BOILER SURFACES AND IN STILL-HEADS IN THIS AND COMPARATIVE WORK AT POINTS OF SUDDEN INCREASE IN ENTRAINMENT

Reference	Diameter of boiler surface (in.)	Velocity at boiler surface (cm./sec.)	Diameter of still-head (in.)	Velocity in still-head (cm./sec.)	Reynolds Number of vapour in still-head
This work	12	12·7	2	459	11,140
Garner, Ellis and Lacey (1957) ..	12	72·3	12	72·3	10,500
Shotton and Habeeb (1954) ..	9	18·8	2	381	9,240
	9	14·8	1½	534	9,712
	9	9·6	1	777	9,390
	9	6·15	⅝	1,278	9,700

wall, the wave motion ascending the column against the downward flow of the liquid. This phenomena has been reported by other workers (Semenov, 1944, and Thomas and Portalski, 1958).

The sudden increase of entrainment entry into the separator is accompanied by a similar increase in collection of entrainment over all other sections of the still-head. The graph of "total" entrainment against distillation rate (Figs. 3 and 4) summarises these effects and indicates that the initial cause of these increased entrainment collections over different parts of the still-head is due to a sudden increase of total entrainment entering the still-head and not differing conditions within the still-head itself.

Garner, Ellis and Lacey (1954) comment on this phenomenon and have shown that above a certain critical distillation rate increase of entrainment is very rapid. In their work, this sudden increase was more marked in the determination of entrainment entering the vapour line at the top of the vapour space than on results from measurements of "total" entrainment near the surface of the boiling liquid. The critical distillation rate in their work was approximately 250 lb./hr., which, in their 12 in. diameter evaporator, corresponded to a value of *R*e of 10,500. They commented that this sudden increase may be caused by a change in boiling conditions at the liquid surface, but since corresponding points of sudden increase of entrainment have been noticed in two other completely different sets of apparatus using widely differing rates of vapour release from the surface this would not appear to be the controlling factor (Table II). Garner, Ellis and Lacey also considered the terminal velocities of the various size droplets, but again the sudden increase of entrainment could not be directly related with this or the standard theories of elutriation because of the wide divergencies of flow velocities in the different sets of apparatus, the relevant dimensions of which being included in Table II.

The correlating feature of these various critical distillation rates appears to be a function of the Reynolds number of the vapour stream in the still-head. As a corollary to this, it was realised that the product of the mass flow rate Q (g./sec.) and velocity u (cm./sec.) is also a constant factor for this critical point.

That is $Qu = K(Re)^2$ where $K = \frac{\pi\eta^2}{4\rho}$

and is constant for all sets of apparatus.

It is thought that this product Qu may represent the total force available for lifting the particles into the still. It can be seen that the dimensions of the product are the same as that of a force unit, $\frac{ML}{T^2}$.

Garner, Ellis and Lacey (1957) stated that the bulk of the entrainment was caused by the larger droplets, in spite of the fact that over 95 per cent of the number of droplets collected were below 20 μ in diameter.

There appears to be a critical size range of about 17–20 μ above which the entrainment caused by these larger droplets increases approximately as the square of the diameter of the droplet.

It would follow that there is a possible correlation of these critical size ranges of droplets and the force available from the rising vapour, in that, when the force is sufficient to entrain the larger droplets then the "total" entrainment collected in the still-head and subsequent parts of the apparatus, will show a sudden increase.

Pyott, Jackson and Huntington (1935) in their investigations on a kerosene-air system showed that a sudden increase in entrainment occurred with increase of velocity at approximately 1·5 ft./sec. of air. Their experiments were made in a 12·5 in. diameter tower at 80° F. and thus the corresponding value of Reynolds number at the critical point is found to be approximately 7,500. Sherwood and Jenny (1935) working with a water-air system at 20° and in a 45·7 cm. diameter tower showed a more definite critical point to arise at the same vapour velocity as Pyott and others, but in a larger diameter tower. This corresponds to a Reynolds number of approximately 12,000. O'Connell and Pettyjohn (1946), who investigated liquid carry-over in a horizontal tube evaporator, obtained a figure for the allowable mass velocity to prevent splashing. This corresponded for boiling point conditions at 140° F. to a Reynolds number of 10,100 and for 164° F. to a figure of 15,100.

These values of Reynolds numbers are similar to those in Table II.

The Post-entrainment Stage

The true climbing film or flooding effect was clearly manifest when there was a sudden increase in the volume collected at point 3.

Correlation of these results and those of other workers using wetted-wall columns is almost impossible because values of liquid mass flow rates in these experiments were less than 150 lb./hr. per sq. ft. Without altering the standard distillation system the quantity of liquid on the walls of the still-head cannot be appreciably changed. It is dependent solely on the condensate forming on the still-head, and entrained solution collected at the specific flow rate. At flooding conditions the entrained portion contributes over 90 per cent of the total, and is itself directly dependent on the flow rate. Thus no relation was possible with work such as that of Holmes (1947), who investigated flooding velocities in vertical unpacked tubes using much higher liquid flow rates.

The relation between the momentum of the downcoming liquid and the upward resistance force from the rising steam was established, using the data at the flooding rate given in Table I. From the pipe friction chart of Coulson and Richardson (1955b) it was calculated that the resistance to flow, per unit area of still-head, at the flooding point would be 8·1 dynes/cm^2. The practical figure obtained by considering the momentum of the downcoming fluid was 7·9 dynes/cm^2.

Decontamination Factor

In industrial distillation plants the removal of entrained material is of practical concern and in certain instances, as when dealing with radioactive materials, the decontamination factor must be very high. The decontamination factor is defined as the ratio of the activity/ml. of the still pot solution to the activity/ml. of condensed vapour leaving the evaporator.

In our experiments, although only simple entrainment removal devices were used such as a straight length of vertical tubing and a centrifugal separator, the decontamination factors were about 10^5. This compares favourably with the figures 10^4 to 10^5 obtained by Manowitz, Bretton and Horrigan (1955) for example, who made experiments using a Raschig ring packed tower and a "Thermal Wool Fiber-Fiberglass" packed tower.

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