THE ENTRAINMENT OF LIQUID DURING DISTILLATION

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The process of distillation involves vaporisation of a liquid followed by removal and subsequent condensation of the vapour formed. In practice this is not always achieved, and it has been found that the distillate may be contaminated with the original liquid. This contamination has been traced to a variety of causes such as foaming and in particular to entrainment.

The term entrainment was used by Cessna and Badger[1] to signify the phenomenon whereby a small proportion of the liquor undergoing evaporation was carried over by the vapour as droplets into the condenser resulting in a loss of the material contained in the evaporator and contamination of the condensate. For example, in concentrating sugar juice, entrainment results in the loss of sugar from the evaporators[2] and in the production of distilled water the prevention of entrainment has been shown to be essential for precise *p*H work[3], conductivity experiments[4,5,6] and for water used for surface chemistry. In pharmacy it is important that water intended for the preparation of injection solutions should be free from pyrogens. The most probable source of pyrogen contamination of freshly distilled water is in the carry-over by entrainment of the water being evaporated. To reduce this to a minimum, the British Pharmacopæia directs that in preparing water for injection the still used should be fitted with an efficient device for preventing entrainment.

The mechanism whereby droplets of liquid and bubbles are thrown into the vapour above the boiling liquid has been studied and appears to be mainly due to the bursting of vapour bubbles and as the cavity left in the liquid surface collapses inwards droplets may be projected from the rising centre. If the boiling liquid foams small bubbles may be released into the vapour and also fragments from the breaking foam films.

The liquid droplets and bubbles formed are then carried away by the rising vapour and may thus reach the condenser. The factors governing the conveyance of the entrained droplets by the vapour have been the subject of much published work and variously ascribed to the rate of evaporation[2,7] the velocity of the vapour[1,8,9,10,11,12] and, particularly in the case of fractionating columns, to the vertical linear path of the vapour, i.e., plate spacing[8,9,10,11,12].

Much of the published work has been carried out on commercial and pilot plants which do not seem very suitable for a close control of the experimental conditions; we have, therefore, re-examined this problem using a relatively simple system, the entrainment being followed through straight vertical tubes.

EXPERIMENTAL

A very sensitive method of detecting entrainment was necessary since the contamination in the distillate represents only a very small fraction of the original liquid. Various methods have been reported, such as using electrolyte and detecting by conductivity methods[1,7] or by volumetric analysis[9,11]. These methods were not considered satisfactory in the circumstances, and it was decided to use fluorescein sodium as the indicator and to estimate this fluorimetrically.

A Spekker fluorimeter (model H.760)[13] was used for the estimations with a Wood's glass filter[14] on either side of the mercury-vapour lamp and

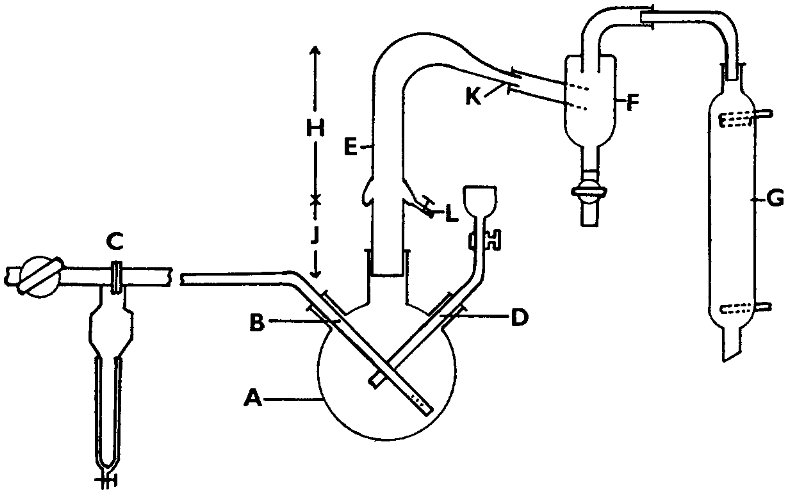

FIG. 1. Distillation apparatus.

A	5-l. flask.	D	Water inlet.
B	Steam inlet tube.	E	Stillhead.
C	Orifice meter.	F	Separator.
		G	Condenser.

Chances glass filters OG1 between the cuvette and the photocells. We found that fluorescein solutions exhibited the maximum fluorescence when buffered at *p*H 6. The fluorescein sodium used for the solutions was found to contain a small amount of fluorescent material volatile in steam and this was first removed by drying the solid at 105° C. to constant weight. Solutions containing 0·1, 0·01 and 0·001 per cent. of fluorescein sodium were unaffected by boiling under reflux for 80 hours, 8 hours and 8 hours respectively. Also exposure to diffuse daylight for 14 days did not affect the fluorescence of these solutions. For solutions containing 1·5 μg. to 0·02 μg. per ml. of fluorescein sodium the error of the estimation was found to be less than 6 per cent., but below 0·02 μg. per ml. the error was much greater. Fluorescence could be detected with concentrations as low as 0·001 μg. per ml.

The apparatus is represented diagrammatically in Figure 1. The 5-l. flask (A) was charged with 3000 ml. of a 0·1 per cent. w/v solution of fluorescein sodium in distilled water which was maintained at its boiling point with a heating mantle having two heating circuits (500 W. and 300 W.). Steam was injected into the fluorescein solution through a ⅝-in. glass tube (B), the rate of flow of the steam being measured by a simple orifice meter (C). 4 stainless-steel orifice plates were used with holes ⅛ in., ¼ in., 5/16 in. and ⅜ in. diameter for measuring distillation rates of 0 to 1·5, 1·5 to 4, 4 to 8 and 8 to 17 l. per hour. The tube (B) which dipped below the surface of the fluorescein solution was sealed at the end having 6 holes (¼ in. diameter) in the side of the tube. It was considered that by these means, reproducible conditions in the still would be achieved and for a given rate of distillation the amount of entrainment produced in the still would be constant. The vapour rising from the solution passed through stillhead (E) into a centrifugal separator (F), where entrained droplets were collected, and then to the condenser (G). The still was constructed of Pyrex pipeline equipment, reduction joints were used to prevent the solution creeping along the walls and to avoid possible constrictions in the pipes by the gaskets used in butt-ended joints. Stillheads of 4 diameters, ⅝ in., 1 in., 1½ in. and 2 in. were used. The length of the stillheads (H) being 5 in., 10 in., 15in. and 30 in. for each diameter. Part J at the bottom was standard in all cases—2 in. diameter and 5 in. long; part K was ⅝ in. diameter for all stillheads. The apparatus was assembled so that the stillhead was vertical and channel and tapping (L) was included so that the liquid trapped on the walls of the vertical section could be collected as it drained down. The still, stillhead and the separator were lagged to prevent undue condensation in the system.

The fluorescein sodium used in preparing the 0·1 per cent. w/v solution in distilled water was dried to a constant weight at 105° C. in order to remove moisture, which may be as much as 10 per cent., and to remove impurities which have been shown to be fluorescent and steam volatile. The still was heated by the mantle (800 W.) until the solution was at the boiling point, the loading on the mantle was then reduced to 300 W. and the level of the solution at 100° C. noted. A loading of 300 W. was sufficient to maintain the solution at the boiling point.

Steam was allowed to pass for 20 minutes to ensure equilibrium conditions in the apparatus as indicated by maintenance of water level in the boiler and a constant steam rate through the orifice meter. During an experiment the steam was fed into the still at atmospheric pressure at a controlled rate and the distillation timed for 20 minutes by a stop-clock. After the run the level of the liquid in the still was checked and if a variation occurred, corrections for the change in concentration of the solution were applied to the figures obtained for entrained fluorescein. Liquid was collected separately from the base of the stillhead, from the separator and from the condenser. The volumes were measured, and the concentration in each was estimated fluorimetrically by comparison with standard solutions of fluorescein, 2 standard solutions being used for the liquid from the separator. All solutions were buffered to *p*H 6 for the estimation.

The rate of distillation was expressed as ml. per hour, being the sum of the volumes of the liquids collected from the separator and condenser. It was found that the concentration of fluorescein in the liquid collected from the condenser was constant for all rates of distillation irrespective of length and diameter of stillhead. Since this concentration was equal to the figure obtained when the volatility of dried fluorescein in steam was determined, it was assumed that the centrifugal separator was acting efficiently and separating the droplets from the vapour stream. Fluorescein equivalent to that collected during the run was returned to the still before another experiment was commenced.

Results

Figures 2, 3, 4 and 5 summarise the experimental results obtained and these curves represent a total of 1067 distillations. The points plotted on these curves are the averaged results for increments of 500 ml./hour in the distillation rate. By this means the scatter of the points around each curve was reduced, which simplified the drawing of the curves where they lie close together, but this did not alter their shape. The figures obtained for the concentration and quantity of fluorescein collected from the base of the stillhead varied very widely and showed little correlation with the rate of distillation. The quantity of fluorescein collected in the separator was expressed as μg./l. of distillate, which was a measure of the entrainment taking place during the distillation, this representing the degree of contamination of the distillate, each μg. of fluorescein being equivalent to 0·001 ml. of the original solution.

Discussion

The results have been treated in a number of ways in an attempt to demonstrate the effect of various factors influencing entrainment.

Rate of Distillation. This treatment is the simple experimental plot given in Figures 2, 3, 4 and 5. These curves may be divided into three distinct stages.

(*a*) Initially, entrainment increases linearly with distillation rate.

(*b*) The middle portion tends to a constant rate of entrainment which may be preceded by a region where entrainment decreases with increasing distillation rate.

(*c*) The third stage is characterised by a sudden and spectacular increase in the amount of fluorescein carried over. It was observed that this increase was due to a film of solution being swept along the walls of the stillhead by the vapour at high distillation rates. This phenomenon is referred to subsequently as "gross carry-over."

The slope of the line in the first part of the curve is related to the diameter and entrainment increases with increasing diameter. This is probably due to the greater radial path offering less chance for the ascending droplet to be trapped on the wall. In the second portion of the curve the entrainment tends to a value which is nearly constant for all diameters as the rate of distillation increases, except for the $\frac{5}{8}$-in. diameter stillhead, and this entails a reduction in entrainment for the $1\frac{1}{2}$-in. and 2-in. diameter stillheads. This "constant" value for entrainment was of the order of 8 μg. of fluorescein, although the stillheads 5 in. long and those $\frac{5}{8}$ in. diameter deviated from the figure. Rhodes and Slachman[15] obtained results which

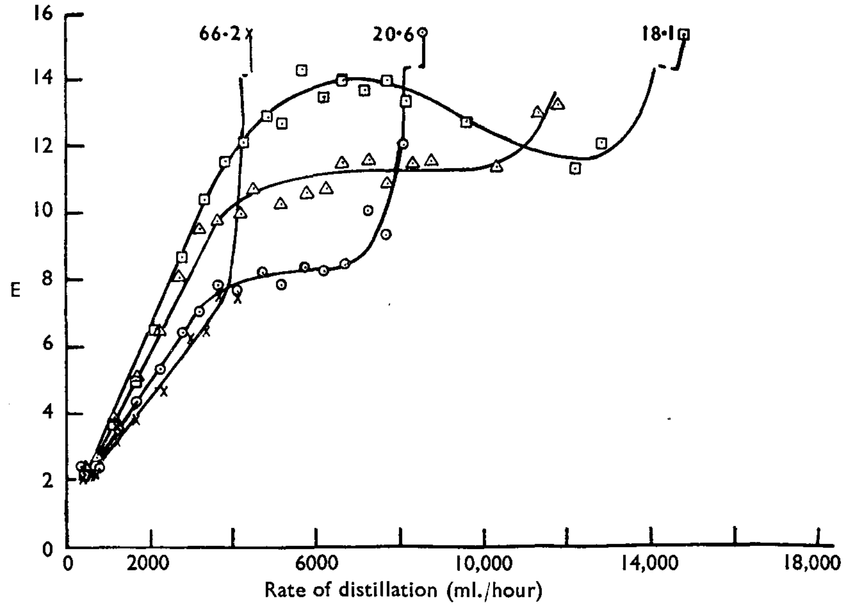

FIG. 2. The effect of the rate of distillation on entrainment in stillheads 5 in. long.

× $\frac{5}{8}$ in. diameter. △ $1\frac{1}{2}$ in. diameter.
⊙ 1 in. diameter. ⊡ 2 in. diameter.

E = entrainment as μg. of fluorescein sodium per litre.

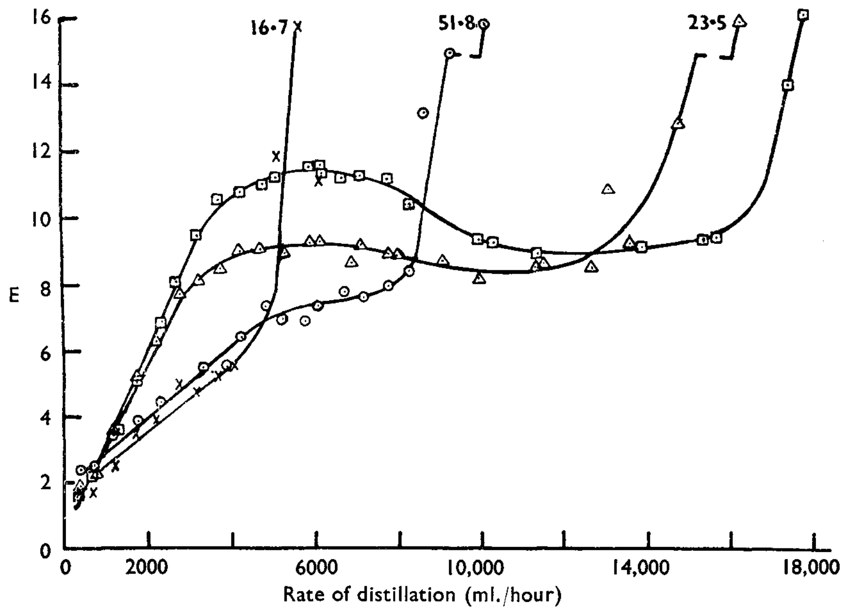

FIG. 3. The effect of the rate of distillation on entrainment in stillheads 10 in. long.

× $\frac{5}{8}$ in. diameter. △ $1\frac{1}{2}$ in. diameter.
⊙ 1 in. diameter. ⊡ 2 in. diameter.

E = entrainment as μg. of fluorescein sodium per litre.

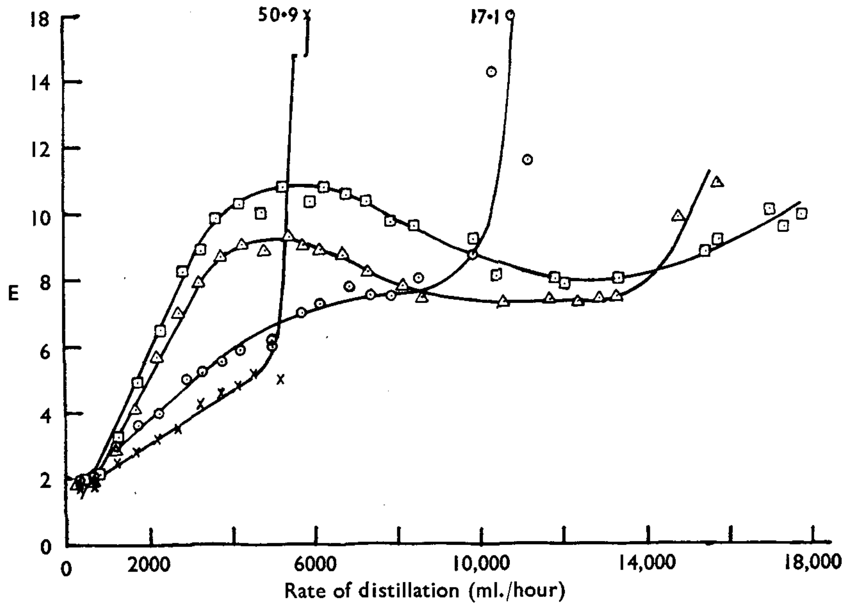

FIG. 4. The effect of the rate of distillation on entrainment in stillheads 15 in. long.

× $\frac{5}{8}$ in. diameter. △ $1\frac{1}{2}$ in. diameter.
⊙ 1 in. diameter. ⊡ 2 in. diameter.

E = entrainment as μg. of fluorescein sodium per litre.

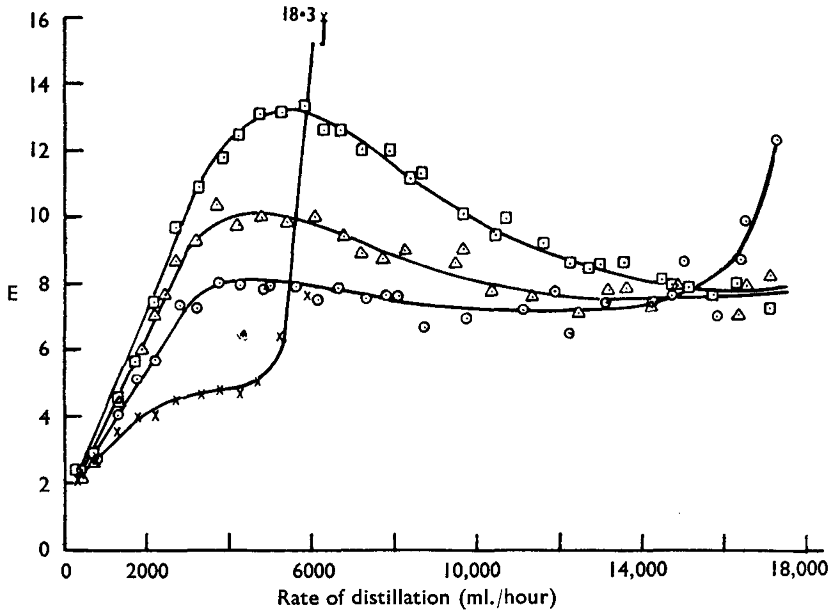

FIG. 5. The effect of the rate of distillation on entrainment in stillheads 30 in. long.

× $\frac{5}{8}$ in. diameter. △ $1\frac{1}{2}$ in. diameter.
⊙ 1 in. diameter. ⊡ 2 in. diameter.

E = entrainment as μg. of fluorescein sodium per litre.

were very similar for the effect of the rate of distillation on entrainment, using benzene-toluene and also ethanol-water mixtures.

Effect of Vapour Velocity. The mean velocity of the vapour through each stillhead was calculated from the rate of distillation and Figure 6 illustrates the effect of vapour velocity on entrainment in stillheads 15 in. long. These curves are of the same general form as Figures 2, 3, 4 and 5, but emphasise more strongly the effect of the diameter of the stillhead on entrainment.

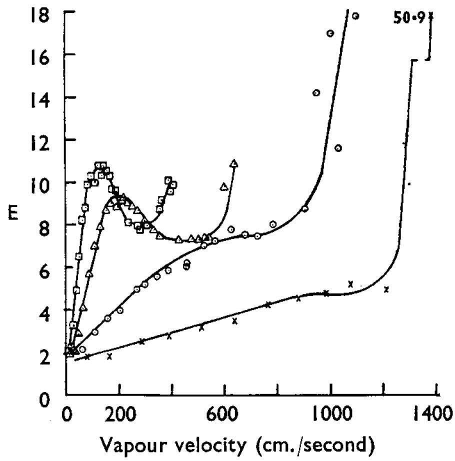

FIG. 6. The effect of vapour velocity on entrainment in stillheads 15 in. long.

× $\frac{5}{8}$ in. diameter. △ $1\frac{1}{2}$ in. diameter.
⊙ 1 in. diameter. ⊡ 2 in. diameter.

E = entrainment as μg. of fluorescein sodium per litre.

Sherwood and Jenny[11], using columns of 17·8 cm., 20·3 cm. and 25·4 cm. diameter, found that for a given gas velocity the entainment increased with increasing diameter, which is in agreement with our results.

Correlation with Reynolds Number. Reynolds[16] found that the flow of a fluid through a pipe is related to the density, viscosity and velocity of the fluid and also the diameter of the pipe in the following manner:—

$$\text{Reynolds Number: (Re)} = \frac{\rho d u}{\eta}$$

ρ = density of fluid.
d = diameter of pipe.
u = velocity of fluid.
η = viscosity of fluid.

For smooth-bore pipes such as the glass stillheads used the flow is streamline or laminar for a Reynolds number below 2000 to 4000, whereas above this range the flow becomes turbulent and full turbulence will develop when the Reynolds number reaches a value of 10,000 to 20,000.

The variation of entrainment with Reynolds number is given in Figures 7 and 8 for the 5 in. and 15 in. long stillheads respectively and here a relationship with the diameter of the stillhead is shown also since in these figures the entrainment divided by the diameter of the stillhead is plotted (i.e., entrainment per unit radial path). The curves for the 10 in. and 30 in. long stillheads are similar. From these curves it would seem that when the steam exhibits laminar flow, as indicated by Reynolds number, the entrainment increases linearly with Reynolds number and is greatest with the greatest diameter since if the wall of the tube is the main trapping agency then the wider the tube the greater is the radial path a droplet must traverse to reach the wall. The sudden reduction in diameter of the stillheads of $\frac{5}{8}$ in. and 1 in. diameter may have induced some turbulence at the lower values of Reynolds number which would account for the lower values obtained for entrainment.

When Reynolds number is increased and the vapour in the stillhead becomes more and more turbulent the effect of the diameter is decreased

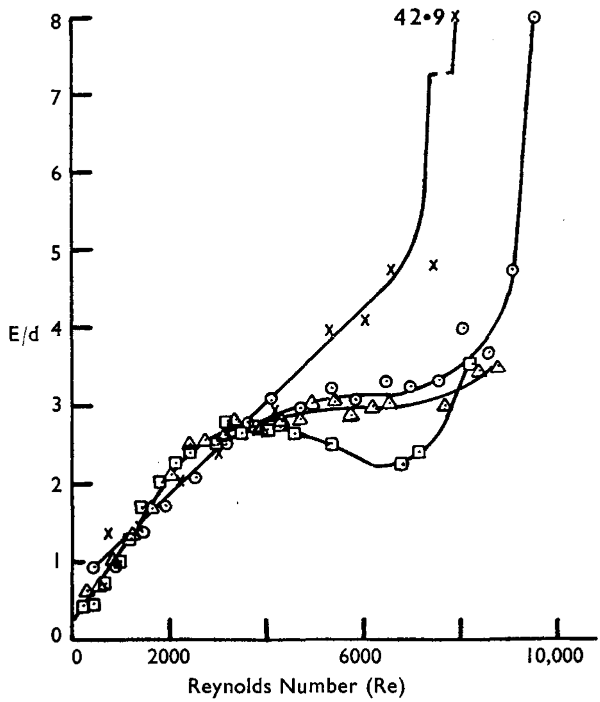

FIG. 7. The effect of Reynolds number on entrainment per unit diameter in stillheads 5 in. long.

× $\frac{5}{8}$ in. diameter. △ $1\frac{1}{2}$ in. diameter.
⊙ 1 in. diameter. ⊡ 2 in. diameter.

$\frac{E}{d} = \frac{\text{entrainment as } \mu\text{g. of fluorescein sodium per litre.}}{\text{diameter of stillhead}}$

so that as full turbulence is achieved the amount of entrainment is nearly the same for all diameters. This is shown for stillheads 15 in. long in Figure 9 and may be deduced for the 5 in., 10 in. and 30 in. long stillheads from Figures 2, 3 and 5.

The results published by O'Connell and Pettyjohn[7] have been converted into terms of Reynolds number and are shown in Figure 10 and 11. In drawing their curves these authors used only those points that are shown with continuous lines drawn through them and they ignored those points we have connected with a broken line. It is significant that the peaks in these curves occur at values for Reynolds number comparable with our results. The apparatus was a semi-commercial horizontal tube evaporator using solutions of salts.

Effect of the Length of the Stillhead. A consideration of the factors governing the height to which a drop may be carried by vapour has been given by Hausbrand[17]. Figure 12 shows the effect of the rate of distillation on entrainment for stillheads of different lengths of 1 in. diameter. Curves obtained for $\frac{5}{8}$ in., $1\frac{1}{2}$ in. and 2 in. diameter stillheads are similar. At the

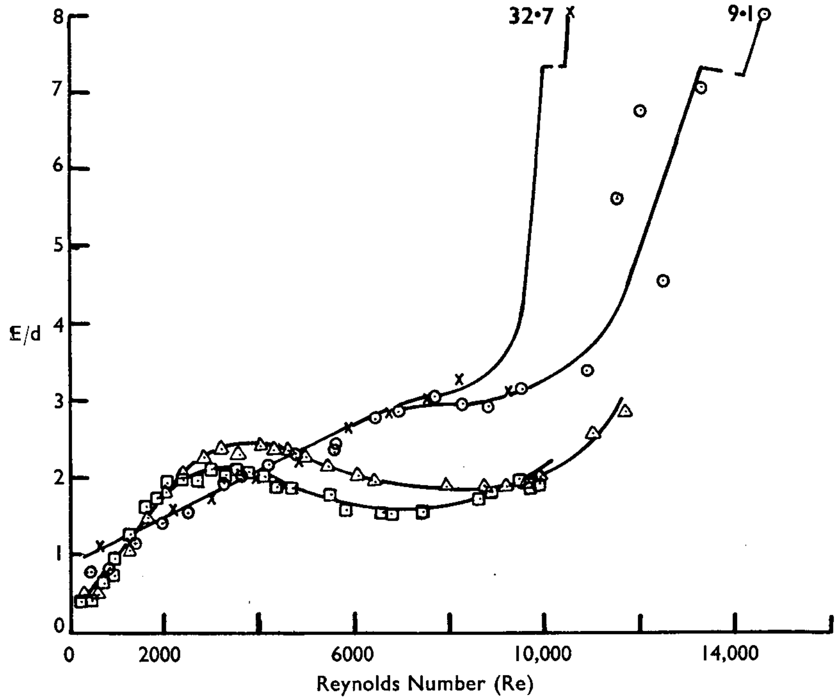

FIG. 8. The effect of Reynolds number on entrainment per unit diameter in stillheads 15 in. long.

× $\frac{5}{8}$ in. diameter. △ $1\frac{1}{2}$ in. diameter.
⊙ 1 in. diameter. ⊡ 2 in. diameter.

$$\frac{E}{d} = \frac{\text{entrainment as } \mu\text{g. of fluorescein-sodium per litre}}{\text{diameter of stillhead}}$$

lower rates of distillation the amount of fluorescein entrained decreases as the length of stillhead increases from 5 in. to 15 in. as may be expected, but is greatest for the 30 in. stillhead. Then as the rate of distillation increases entrainment through the 30 in. stillhead falls to values below that of the others.

This apparent anomaly with the 30 in. stillhead at the lower rate of distillation is surprising, but was found to occur with the stillheads of $\frac{5}{8}$ in., $1\frac{1}{2}$ in. and 2 in. diameter also. Further work may give rise to a reasonable explanation.

Much of the published work on the effect of the vertical path has been carried out in fractionating columns of the bubble-cap type by varying the distance between the plates[8,9,10,11,12]. In general an increase in the plate spacing produces a decrease in entrainment, but the conditions in a bubble-cap column will be very different from the conditions in our apparatus.

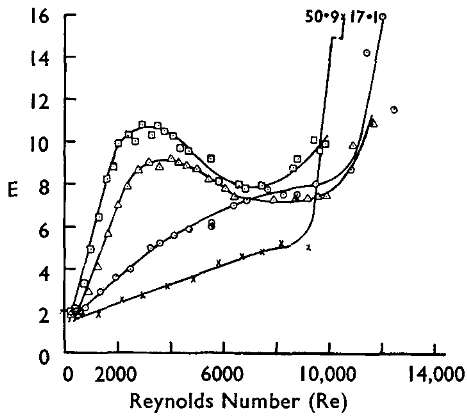

FIG. 9. The effect of Reynolds number on entrainment in stillhead 15 in. long.

× ⅝ in. diameter. △ 1½ in. diameter.
⊙ 1 in. diameter. ⊡ 2 in. diameter.

E = entrainment as μg. of fluorescein sodium per litre.

Gross Carry-Over. At the higher distillation rates it was observed that fluorescein solution was swept upwards along the wall of the stillhead resulting in a large increase in the quantity of fluorescein collected in the separator. The onset of this phenomenon was sudden and Table I gives in terms of Reynolds number the lowest experimental results at which an ascending film was obtained in each stillhead.

The maximum Reynolds numbers obtained for the 1½ in. and 2 in. stillheads (30 in. long) were 12,735 and 9545 respectively and no gross carry-over occurred, these figures corresponding to the maximum distillation rate of the apparatus, approximately 17·1 l./hour.

For the 15 in. long stillhead of 2 in. diameter an increase in entrainment was found at Reynolds number 9870, but the rising film was not seen in the stillhead.

TABLE I

REYNOLDS NUMBERS AT WHICH GROSS CARRY-OVER OCCURRED CALCULATED FROM EXPERIMENTAL DISTILLATION RATES

Length of stillhead in.	Diameter of stillhead			
	⅝ in.	1 in.	1½ in.	2 in.
5	7,875	8,090	8,425	8,230
10	9,190	9,250	9,732	9,650
15	10,500	10,830	10,970	9,870
30	11,235	18,280	No gross carry-over	

From Table I there is good agreement for the Reynolds number at which gross carry-over occurred for each length of stillhead except for those 30 in. long. For the two in which gross carry-over was obtained, however, the agreement was better if vapour velocity was compared. For the ⅝ in. and 1 in. diameter stillheads the Reynolds numbers given correspond to vapour velocities of 1480 cm./sec. and 1507 cm./sec. respectively.

SUMMARY

1. The entrainment of liquid droplets through straight vertical stillheads during distillation has been investigated using fluorescein sodium as an indicating substance.

2. When the Reynolds number for the vapour flowing through the stillhead is below 2000, indicating streamline or laminar flow, the entrainment has been found to be directly proportional to the rate of distillation

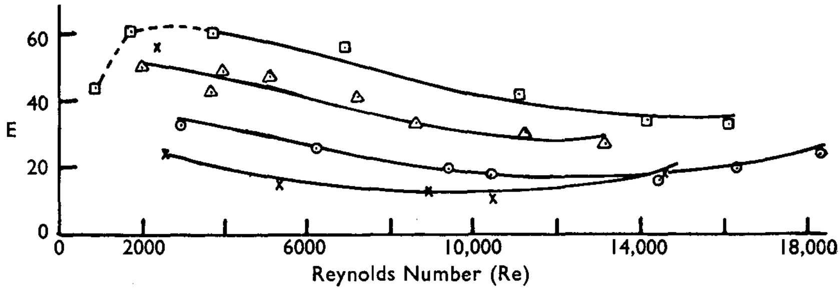

FIG. 10. The effect of Reynolds number on entrainment (O'Connell and Pettyjohn[7]). Distillation of 20 per cent. sodium chloride.

× = solution boiling at 140° F.
⊙ = solution boiling at 167° F.
△ = solution boiling at 190° F.
⊡ = solution boiling at 219° F.
E = entrainment as pounds of liquid per 10^6 pounds of vapour.

and to the diameter of the stillhead. As the wall of the stillhead constitutes the main trapping agency it is postulated that the entrainment will increase as the diameter increases since under these flow conditions the vapour has little radial movement. The entrainment per unit diameter is directly proportional to the Reynolds number in the 30 in. stillheads, but deviations occur in the shorter stillheads of $\frac{5}{8}$ in. and 1 in. diameter, which is probably due to the constriction from 2 in. inducing some turbulence.

3. As the Reynolds number is increased to 6000 to 8000 the entrainment approaches a constant value, irrespective of the diameter, for a given length of stillhead. Under these conditions the flow of the vapour is probably fully turbulent and has a high radial velocity which carries the droplets to the wall.

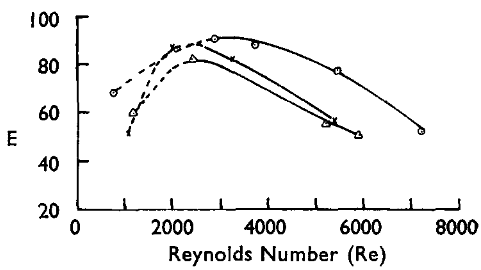

FIG. 11. The effect of Reynolds number on entrainment (O'Connell and Pettyjohn[7]). Distillation of solutions of sodium sulphate).

× = 20 per cent. sodium sulphate boiling at 140° F.
⊙ = 24 per cent. sodium sulphate boiling at 166° F.
△ = 24 per cent. sodium sulphate boiling at 140° F.
E = entrainment as pounds of liquid per 10^6 pounds of vapour.

4. Entrainment has been found to decrease as the length of the stillhead increases from 5 in. to 15 in. for each diameter, but in the 30 in. stillhead entrainment was highest.

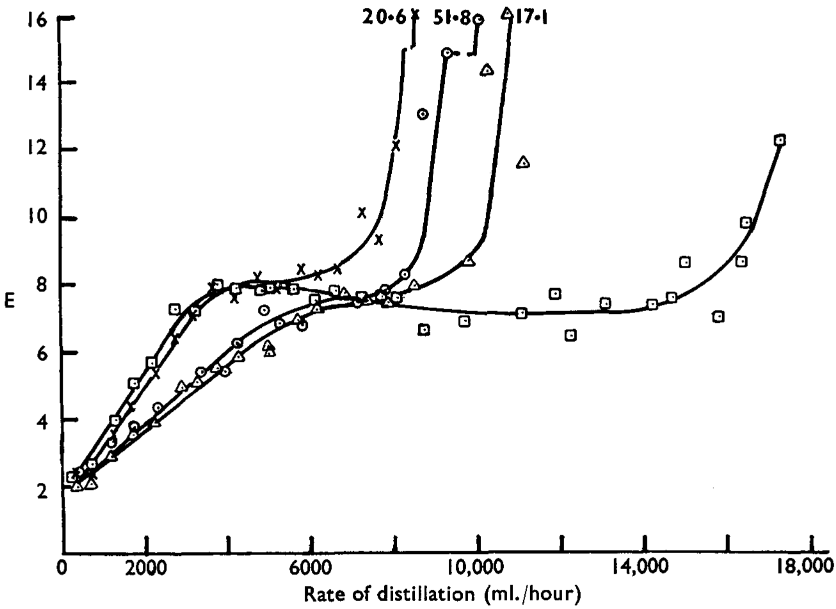

FIG. 12. The effect of the length of stillhead on entrainment in stillheads of 1 in. diameter.
× Stillheads 5 in. long.
⊙ Stillheads 10 in. long.
△ Stillheads 15 in. long.
⊡ Stillheads 30 in. long.
E = entrainment as μg. of fluorescein sodium per litre.

5. Gross carry-over (climbing film effect) occurred in the 5 in., 10 in. and 15 in. stillheads at an approximately constant value for Reynolds number for each length and was unaffected by the diameter. For the 30 in. stillheads gross carry-over was not achieved for diameters of $1\frac{1}{2}$ in. and 2 in. and in the case of the $\frac{5}{8}$ in. and 1 in. diameter tubes gross carry-over was more nearly related to vapour velocity.

We would like to thank Professor H. Berry for suggesting the use of fluorescein sodium, Dr. F. Wokes for advice on the fluorimetric estimations and Mr. D. Train for help and advice throughout this work.

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DISCUSSION

The paper was presented by MR. E. SHOTTON.

DR. W. MITCHELL (London) asked whether the results obtained would apply to apparatus of different design, in particular to large-scale apparatus? It would be of interest to know if the ordinary gross carry-over would be different if a material other than glass were used. This could be tested by replacing the glass stillheads—part E in Figure 1—by similar metal stillheads.

DR. F. HARTLEY (London) said that for the particular stillhead there might be a critical length between 30 in. and 15 in. at which entrainment began to increase. This might be due to the thermal losses occurring in the longer stillheads. Were the authors satisfied with the lagging of the stillheads? If lagging were inefficient and thermal losses high, a cloud would tend to build up and to adsorb the entrainment. The obvious pharmaceutical application of the work was in the design of stills for the preparation of pyrogen-free water. In industrial practice two stills were often used for this purpose, the distillate from the first being redistilled in the second. It seemed possible that they might find a critical length of stillhead for their equipment beyond which, relatively, no further reduction in entrainment occurred.

MR. W. C. PECK (London) described the paper as one of the most fundamental studies which had been made in the design of entrainment separators. It would affect not only the distillation of water but also the design of fractionating columns, and in fact all the processes where a vapour and a liquid were concerned. The relationship of the entrainment to the Reynolds Number was of first-rate importance. He suggested that dimensional analysis might have revealed that apart from the Reynolds Number there was another factor, the height of the column, which would have explained anomalous results. Some of the most economic processes for the production of distilled water, such as that of vapour recompression, had not been investigated in this country.

MR. J. H. OAKLEY (London) said that by widening the still orifice one would expect entrainment to be reduced because the rate of flow of the vapour is lowered, but at the same time condenser effect is also lowered, which would tend to increase entrainment. At the other extreme, if the orifice were too narrow one would reach a climbing film evaporator. Was the latitude between these two extremes very critical?

DR. A. F. S. A. HABEEB, in reply, said that the curves in Figures 10 and 11 showed results by the American workers, O'Connell and Pettyjohn, using a semi-commercial horizontal evaporator. After a critical Reynolds Number of about 2000 the amount of entrainment was reduced, which was in agreement with their results, but in fractionating columns other workers got different results. The distances between the plates and the splashing of the liquid on the plates might account for these differences. Work on different materials for stillheads was in hand. Speaking of the abnormality of the curves for 30-in. stillheads, he said that if they had been able to see inside they might have seen whether there was clouding.

Note by the Editor.—Subsequent to the meeting the following note was received from MR. SHOTTON:—

"Although some condensation in the stillhead would take place, this would not be a governing factor since the entrainment falls with an increase in the distillation rate and vapour velocity, which is the reverse of what may be expected if the condensation due to heat loss was a governing factor. Regarding the point raised by Mr. Oakley, there was sufficient latitude between these two extremes, this latitude increasing with increasing diameter of pipe."