A MODIFIED DISTILLERY PROCEDURE:
THE EFFECT OF RUN-BACK OF HEADS AND TAILS TO SUBSEQUENT

WINE CHARGES ON THE EFFICIENCY AND YIELDS OF POT STILL

BRANDY PRODUCTION.

B. HICKIN.

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I

#### ABSTRACT.

The aim of this experiment was to examine the effect of running back heads and tails to subsequent wine charges. By addition of heads and tails from the previous brandy run to the next wine charge, it was hoped that the yield of brandy heart would be substantially increased. Also it was hoped that this procedure would not have a detrimental effect on the quality of the brandy distillate.

The results show, however, that addition of feints to the wine charge only slightly increased the strength of the wine charge, by 1 to 1.5%  $^{V}/v$ .

This resulted in a small increase in the size of the brandy heart. Thus, addition of heads and tails to a wine charge does increase the size of the brandy heart, but only to a minor extent. INTRODUCTION.

II

Distillation of brandy in Australia involves the separation of head and tail fractions from a brandy "heart" during a brandy run. The heads and tails are then usually put aside and distilled at a later date for the purpose of alcohol recovery.

The experiment sought to find out what effect the running of heads and tails to subsequent wine charges has, on the efficiency and yields of Pot Still Brandy distillation.

The run back of heads and tails to wine charges is a well established practice in Cognac production in France. This practice is claimed to increase the quality of the Cognac brandy, as well as increasing the size of the brandy heart.

#### LITERATURE REVIEW.

## (a) THE THEORY OF DISTILLATION:-

Distillation involves the separation of the components of a liquid mixture (e.g. Wine) by partial vapourisation of the mixture and separate recovering of vapour and residue liquid. (2)

The more volatile constituents of the original mixture tends to concentrate in the vapour, whereas the less volatile constituents concentrate in the liquid residue.

The ease with which these constituents may be separated depends on the <u>nature</u> of the mixture, and the manner of distillation. (2)

#### IMMISCIBLE LIQUIDS.

A mixture of immiscible liquids will boil at a lower temperature than that at which either of the component liquids would boil alone. In other words, the vapour pressure of the mixture of immiscible liquids is greater than the vapour pressure of either component. (2)

#### MISCIBLE LIQUIDS.

With two liquids that are mutually soluble, the vapour pressure of each is decreased by the presence of the other, and therefore the sum of their vapour pressures is less than the sum of the vapour pressures of the two individual liquids. This will effectively increase the boiling point of the mixture. Ethyl alcohol and water form such a system. (2)

# ALCOHOL AND WATER MIXTURES.

The graph on page 8 shows the relationship between the alcoholic strength of a boiling ethonol/water solution and the strength of the vapour given off from that solution.

The vapour given off from an ethonol/water solution will have a greater concentration of alcohol than the solution it is leaving, providing the ethonol concentration of that solution is less than 96.0%  $^{\text{V}}/_{\text{V}}$ .

If the strength of the solution is 96%  $^{V}/_{V}$ , then the vapour given off from that solution will have the same strength as the solution it is leaving, and so concentration of alcohol will no longer occur. Thus the maximum alcoholic strength that can be attained by distillation is 96%  $^{V}/_{V}$ . (2)

Points to note from the grash are:-

(a) Up to 10% V/v alcohol of the solution, the gradient of the curve is very steep. This means that an increase in the strength of the solution will result in a much larger relative increase in strength of the vapour given off.

For example, a solution of 3%  $^{\vee}/v$  alcohol, gives off a vapour of 27%  $^{\vee}/v$ . If the strength of the solution was increased to 5%  $^{\vee}/v$ , the strength of the vapour would jump to 47%  $^{\vee}/v$ .

Thus, within this range (i.e. up to 10%) a much more efficient extraction of alcohol can be obtained by raising the strength of the solution only slightly. In terms of practical distillation, it is quite easy to obtain a brandy distillate up to a strength of 70%  $^{\text{V}}/\text{v}$ .

- (b) With solutions containing more than 10%  $^{\text{v}}/_{\text{v}}$  ethanol, the curve flattens out very quickly, and further increases in strength of the solution result in a relatively small increase in alcoholic strength of the vapour given off.
- (c) When the strength of the solution is greater than 80%  $^{\text{V}}/\text{v}$ , the process of distillation becomes relatively inefficient, and a fractionating column has to be incorporated to reach a final strength of 96%  $^{\text{V}}/\text{v}$  (i.e. SVR).

## (b) THE METHOD OF DISTILLATION OF FRENCH COGNAC.

Cognac brandy is produced mainly from the St. 'Emillion grape variety in the Departments of Charente and Charente Maritime. (3) Their cool climate limits ripening so that the wines for distillation have a relatively low alcohol content. (3) The average for the last sixty years is 8.6% V/v alcohol. (3)

The years with the higher alcohol content were generally found to be of lower quality. The wines also have a naturally high acid content. (3)

When the wines are distilled, only Pot Stills are used.

The new wine (with its lees) is distilled until the vapour contains negligible alcohol. The distillate contains about 24-32% V/v alcohol. (3) A tails fraction may be separated. The "low wine" is then distilled and approximately 1-2% heads are separated. A small tails fraction is separated. The main distillate then averages 58-60% V/v. (3)

The heads and tails separated are then added back to the next wine charge.

Thus, the characteristics of this method of distillation are:-

- (a) The method of Double Distillation
- (b) The manner in which the method of distillation is carefully executed.
- (c) Adding of Heads and Tails back to the next wine charge.
  - (d) Presence of some lees with each wine charge.

The presence of lees, and the run-back of heads and tails are claimed to raise the quality of the resultant brandy. This will be discussed in detail later.

Besides the improvement of quality claimed by the Cognac people, run-back of heads and tails will also greatly improve the efficiency of distillation, when the wines are of a relatively low alcohol strength.

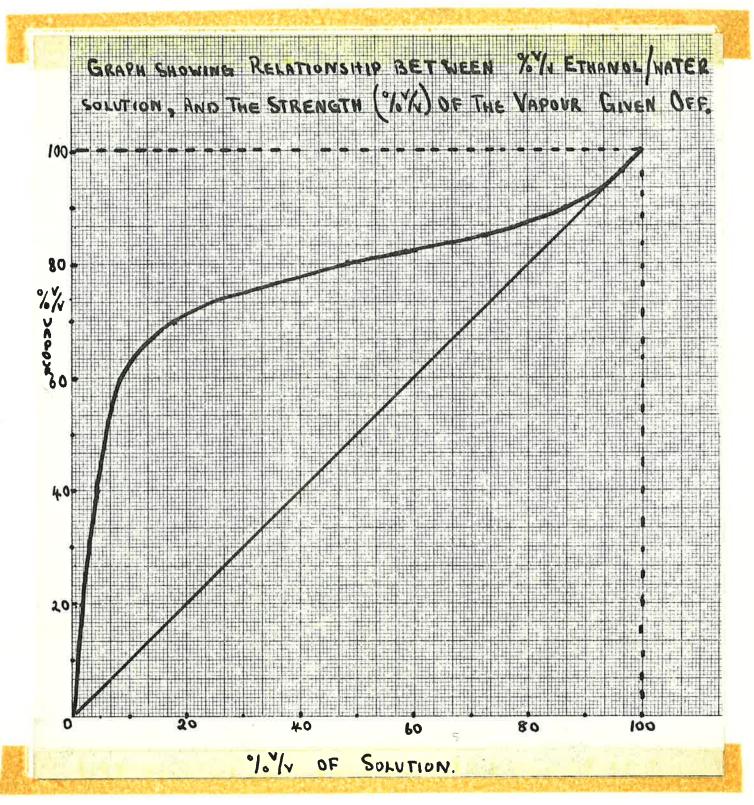
# (c) THE METHOD OF DISTILLATION OF AUSTRALIAN BRANDY USING THE POT STILL.

In Australia, the base wine for distillation is of a much higher strength, and consequently the low wines are of a higher strength. Distillation practice differs throughout Australia. However, a brandy spirit is generally obtained by cutting in at about 82%  $^{V/}v$  and cutting out at 68%  $^{V/}v$  as a fairly typical Australian brandy.

In Australia the base wines are not "cared for" to anywhere near the extent as the base wines used in Cognac manufacture. The base wines used in Australia are generally of inferior quality, and often are oxidized.

The Cognac people take special care to ensure the base wine is safe from oxidation, but at the same time free from SO2. (3)

Also Australian brandy manufacture does not involve the addition of heads and tails back to the next wine charge. Australian base wines generally are greater than 10% \(^{\text{V}}\)\/v alcohol, and so, an increase in the alcoholic strength of that wine will not improve the efficiency of distillation to any great extent, as the relative increase in vapour strength will be slight.



# (d) MODIFIED DISTILLERY PROCEDURE, IN RELATION TO THE SECONDARY CONSTITUENTS OF BRANDY.

The method of Cognac manufacture in France has envolved over many hundreds of years by method of trial and error. (1)

In Australia, the industry is a comparatively now one, and the technique employed differs in important respects from that used in France. (1) Certain French practices are not widely known or well understood in Australia, and it is uncertain whether brandles have ever been made in this country in strict accordance with French practice. (1)

The main factors influencing the quality of brandy are:-

- (1) The base wine,
- (2) The type of Still,
- (3) The distillation of the low wines.
- (4) The distillation of the latter to Brandy,
- (5) Maturation.

The finer French brandies are aged in Wood for periods from 15 to 30 years. (1) Apart from this, the points in which French methods differ from those commonly used in Australia are:-

- (1) Strength of Wine.
- (2) Rate of Distillation.
- (3) Procedure in separating heads and tails.
- (4) The use of lees.
- (5) Return of heads and tails to subsequent charges, and
- (6) The amount of reflux which is controlled by length of column, use of a brandy ball, or rectification by plates in the column or a condenser. (1)

Research indicates that by reducing;-

- (1) the strength of the wine
- (ii) the rate of distillation
- (111) modifying the separation of heads and tails to agree more closely with methods used in France, a brandy with a composition which corresponds much more closely to that of classical French brandies is obtained. (4)

This review will be looking at two practices carried out in Cognac manufacture. The use of lees will be looked at briefly; and the run-back of heads and tails will be discussed.

#### THE USE OF LEES.

The normal procedure in Cognac manufacture is to distil off second lees. (5) When the wine is of inferior quality, however, the practice is to run trial distillations off varying quantities of lees in order to find the level where maximum benefit can be derived from them. (5)

The base wine used in Cognac manufacture is allowed to complete rermentation; is then racked off first less; allowed to stand for 15 to 20 days to ensure completeness of fermentation, and is then distilled in contact with the full second less. (6)

The wines distilled from wine with thick lees give a brandy higher in acids and aldehydes, and lower in esters and higher alcohols. Also, 'lees' brandies have only a 40% variation in non-alcohol constituents, while the wine brandies have a 350% variation - i.e. approximately nine times as great. (7)

Thus the presence of less in the base wine appears to increase the proportion of non-alcohols, and to stabilize the composition. Both effects must have an important effect on quality. The increase in non-alcohols, provided

they are of a desirable type, should increase quality. (7)

The inclusion of lees is regular practice in France, and so it is reasonable to assume that this practice improves the quality of the brandy. (7)

Thus it may be desirable to include as much less to base wine as possible, provided they do not import taints to the spirit, such as earthiness or mouldiness, as would be found in wines from inferior or damaged fruit. (5)

Experiments carried out by T. Angove (1) with reference to inclusion of less in wine charges indicate that inclusion of less gives a cleaner separation between desirable and undesirable head products.

#### RETURN OF HEADS AND TAILS TO SUBSEQUENT WINE CHARGES.

The return of heads and tails to subsequent wine charges is a well established practice with Cognac production in France.

Experiments carried out by T. Angove (1) with reference to run-back of heads and tails to wine charges, show that this procedure causes the heads and tails product to pass more and more completely towards the first and last stages of distillation. Thus the effect of run-back of heads and tails to wine charges is to effectively increase the size of the brandy heart, as a larger brandy cut can be taken. The brandy produced by this method is much richer in esters and to a lesser extent aldehydes, but contains much the same quantities of other constituents. (1)

Analytically, brandy produced by this modified procedure corresponds very closely with genuine French Cognacs.

IV

#### EXPERIMENT.

# "RUN-BACK" OF HEADS AND TAILS TO SUBSEQUENT WINE CHARGES AND ITS EFFECT ON EFFICIENCY AND YIELDS OF POT STILL BRANDY DISTILLATION.

# (a) <u>INTRODUCTION TO EXPERIMENT</u>.

Normal brandy distillation in Australia involves the separation of head and tail fractions from a brandy "heart" during a brandy run. The heads and tails are then put aside and distilled at a later date purely for the purpose of alcohol recovery.

The aim of this experiment is to examine the effect of run-back of heads and tails to subsequent wine charges, on the efficiency and yields of Pot Still Brandy Distillation.

No attempt in this experiment has been made to study the quality aspects of this modified distillery procedure. This point has been reviewed previously from the experiments of Mr. T.W.C. Angove.

# (b) MATERIALS AND METHODS USED.

#### WINE USED FOR DISTILLATION.

As this experiment is not concerned with the quality aspects of run-back of feints, a synthetic wine was prepared, which contained all the volatile components of a wine (and thus distillable), except the fruity aroma. This was also necessary because no wines were available at the time of the experiment, which has low enough SO2 to make them suitable for distillation.

# COMPOSITION OF THE WINE.

The pH was adjusted to greater than 3.0 by addition of Potassium Hydroxide. Total Volume of The Synthetic Wine was 100 litres.

#### DISTILLATION EQUIPMENT USED.

Experimental glass stills were used.

The round 5 litre distillation flask was heated by a thermostatically controlled heating element to ensure a smooth distillation.

The distillation column (60cm) was insulated with paper to ensure minimal reflux.

A thermometer was placed at the head of the column. This recorded the temperature of the vapour, which was correlated back to the alcoholic strength of the vapour.

The impour person from the column, along a cross-quin and was seeled and condensed in the condenser. Sufficient water was passed through the condenser to ensure all the impour was condensed.

# ANALYSIS OF DISTILLATES, AND EQUIPMENT USED.

Analysis carried out were the measurement of volume and alcoholic strength of the wines and distillates.

The alcoholic strength of wine charges was measured with an Ebulliometer. The strength of all the distillates was measured with an Abbe Refractometer (by reference to the refractive index tables). The temperature was maintained at 20°c while using the refractometer.

# (c) PLAN OF EXPERIMENT.

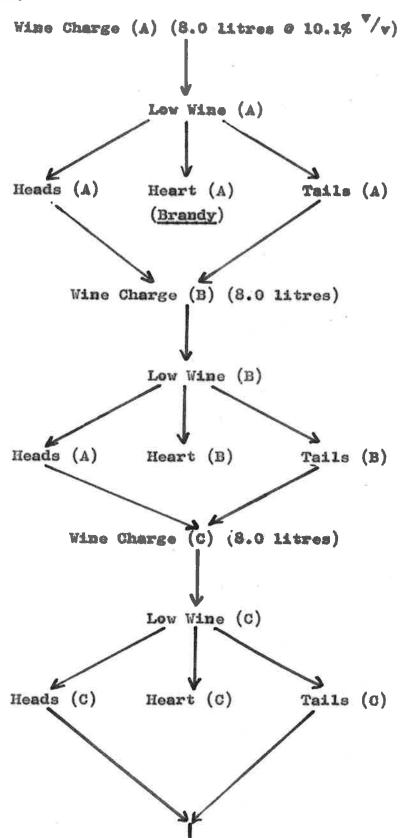
The experiment was laid out in such a way as to study the effect that run-back of heads and tails had on resultant volumes and strengths of brandy hearts.

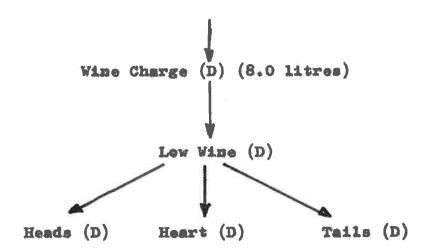
The experiment involved four consecutive distillations of wine charges as follows:-

- (1) 8.0 litres of Wine (at 10.1%  $^{\vee}/_{\nu}$ ) was distilled to give a low wine, which was then distilled to give heads and tails (A), and Brandy heart (A).
- (11) To a second charge of wine, heads and tails (A) were added. After mixing, the volume was bought back to 8.0 litres by removing that volume of wine and feints mixture which was equivalent to the volume of heads and tails added. The 8.0 litres was then distilled to low wines and then redistilled to give heads and tails (B), and brandy heart (B).
- (111) To a third wine charge heads and tails (B) were added and the volume was brought back to 8.0 litres as before. The wine was then distilled to low wine, which was redistilled to heads and tails (C) and brandy heart (C).
- (iv) A fourth wine charge was prepared by addition of heads and tails (C); the volume brought back as previously described, and the wine was distilled to low wine which was redistilled to give heads and tails (D) and brandy heart(D).

Thus the volume of the wine charge was kept constant to study the effect that run-back of feints had on strength and volume of brandy heart.

The Plan of The experiment can be represented diagramatically below:-



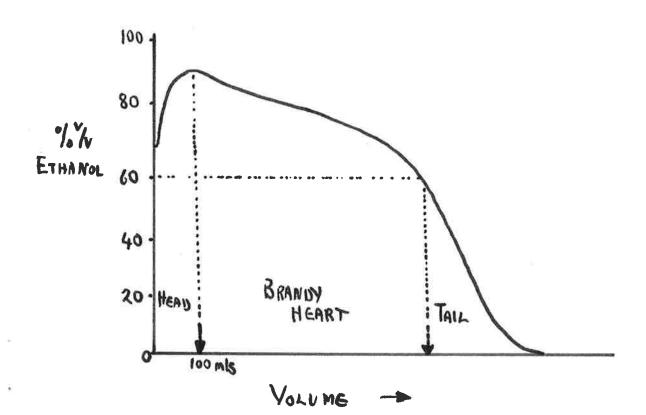


# STANDARD PROCEDURE FOR EXTRACTION OF BRANDY HEART.

As the brandy runs were carried out, the distillate was separated into 25ml pertions. The portions were numbered, and the strengths of all the 25ml portions were measured.

A standard head Cut was made at 100mls. Also a standard tail out was made, when the strength of the distillate dropped below 60%  $^{\rm V}/{\rm v}$ .

This can be represented below:-



The 25ml portions comprising the brandy heart were then mixed together and the strength of that brandy heart was measured.

Heads and tails separated were mixed together thus:—All the heads and that portion of the tails which had a strength greater than the original wine (i.e. 10% V/v).

The heads and tails mixture was then added back to the next wine charge.

N.B. The first head cut made, i.e. (A) was only 50mls. However, head cuts B C and D were all of 100mls.

# (d) RESULTS OF THE EXPERIMENT.

# EFFECT OF RUN-BACK OF HEADS AND TAILS ON WINE CHARGE.

|                 | Strength (% <sup>v/</sup> v) Original Duplicate |      | Volume<br>Original | (L) Duplicate | Litres Abs. Alcohol<br>Original Duplicate |       |  |
|-----------------|---|------|--------------------|---------------|---|-------|--|
| Wine Charge (A) | 10.1  | 10.1 | 8.0                | 8.0           | 0.808                                     | 0.808 |  |
| Wine Charge (B) | 11.0  | 11.0 | 8.0                | 8.0           | 0.880                                     | 0.880 |  |
| Wine Charge (C) | 11.5  | 11.7 | 8.0                | 8.0           | 0.920                                     | 0.936 |  |
| Wine Charge (D) | 11.4  | 11.4 | 8.0                | 8.0           | 0.912                                     | 0.912 |  |

#### EFFECT OF RUN-BACK ON LOW WINES.

| smith Living | Strength (% V/v). |           | Volume (1) |           | LAL      |           |
|--------------|-------------------|-----------|------------|-----------|----------|-----------|
|              | Original          | Duplicate | Original   | Duplicate | Original | Duplicate |
| Low Wine A   | 45.8              | 47.0      | 1.83       | 1.73      | 0.838    | 0.813     |
| Low Wine B   | 39.6              | 47.4      | 2.24       | 1.88      | 0.887    | 0.891     |
| Low Wine C   | 56.4              | 56.0      | 1.58       | 1.63      | 0.891    | 0.913     |
| Low Wine D   | 48.8              | 49.1      | 1.76       | 1.77      | 0.859    | 0.869     |

-19-EFFECT OF RUN-BACK ON HEADS AND TAILS.

| * * * * * * * * * * * * * * * * * * * | Strength (% V/v).  | Volume (1)         | LAL                |  |
|---------------------------------------|--------------------|--------------------|--------------------|--|
|                                       | Original Duplicate | Original Duplicate | Original Duplicate |  |
| Heads and Tails A                     | 35.7 40.0          | 0.232 0.235        | 0.083 0.094        |  |
| Heads and Tails B                     | 46.0 49.1          | 0.345 0.330        | 0.159 0.162        |  |
| Heads and Tails C                     | 49.6 56.2          | 0.272 0.202        | 0.135 0.114        |  |
| Heads and Tails D                     | 51.0 53.4          | 0.310 0.250        | 0.158 0.134        |  |

# EFFECT OF RUN-BACK ON BRANDY HEARTS.

|                |      | (% V/v). Duplicate |       | ume (1) Duplicate | LAL<br>Original | Duplicate |
|----------------|------|--------------------|-------|-------------------|-----------------|-----------|
| Brandy Heart A | 76.8 | 75.6               | 0.900 | 0.875             | 0.691           | 0.661     |
| Brandy Heart B | 76.0 | 75.8               | 0.875 | 0.825             | 0.665           | 0.625     |
| Brandy Heart C | 76.8 | 80.2               | 0.925 | 1.000             | 0.710           | 0.802     |
| Brandy Heart D | 80.2 | 80.6               | 0.950 | 0.925             | 0.762           | 0.746     |

V

# DISCUSSION OF RESULTS.

# EFFECT OF RUN-BACK OF FEINTS ON WINE CHARGE.

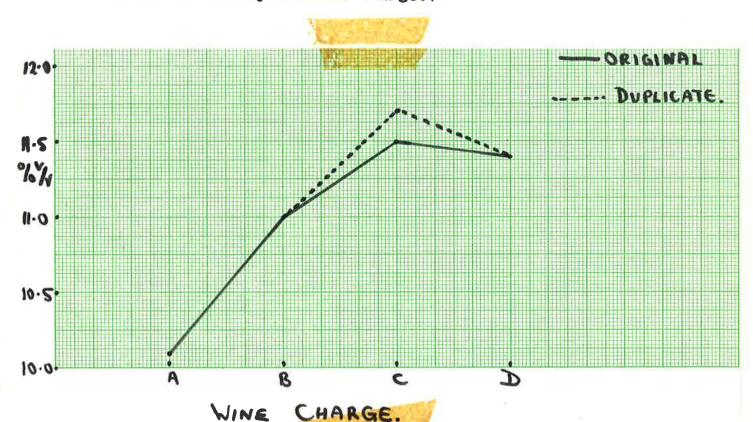
The results show that run-back of feints raised the alcoholic strength of wine charge (B). Subsequent wine charges, however, had approximately the same strength.

Thus, the effect of run-back was to initially raise the strength of the next wine charge, but the alcoholic strength of subsequent charges stabilized.

As previously mentioned, the first head cut made was only 50mls, but subsequent head cuts were 100mls. The size of the head cut was changed to 100mls, as 50mls was found to be unrealistic.

This of course meant that the strength of wine charge (B), was not as great as wine charges (C) and (D). Had the practice of 100ml head cuts been adopted initially, the strengths of the wine charges would have stabilized sooner.

Below is a graph showing the effect on run-back of feints on subsequent wine charges:-



Wine charge (A) - no heads and tails added.

Wine charge (B) - 50mls head cut plus Tails, added.

Wine charges (C) and (D) - 100mls head cut plus Tails, added.

Thus if 100mls head cut plus tails had have been added to wine charge (B), its strength would have been approximately the same as (C) and (D).

# EFFECT OF RUN-BACK ON LOW WINES.

The results show that on average the strength of the low wines increased as a result of run-back of feints to wine charges.

The results also show that the litres of absolute alcohol in the low wines increased initially, but stabilized in subsequent low wines.

## EFFECT OF RUN-BACK ON HEADS AND TAILS.

Here also the results clearly show that the strength and litres of absolute alcohol were initially increased (i.e. of the heads and tails mixture), but this trend flattened out with consequentive distillations.

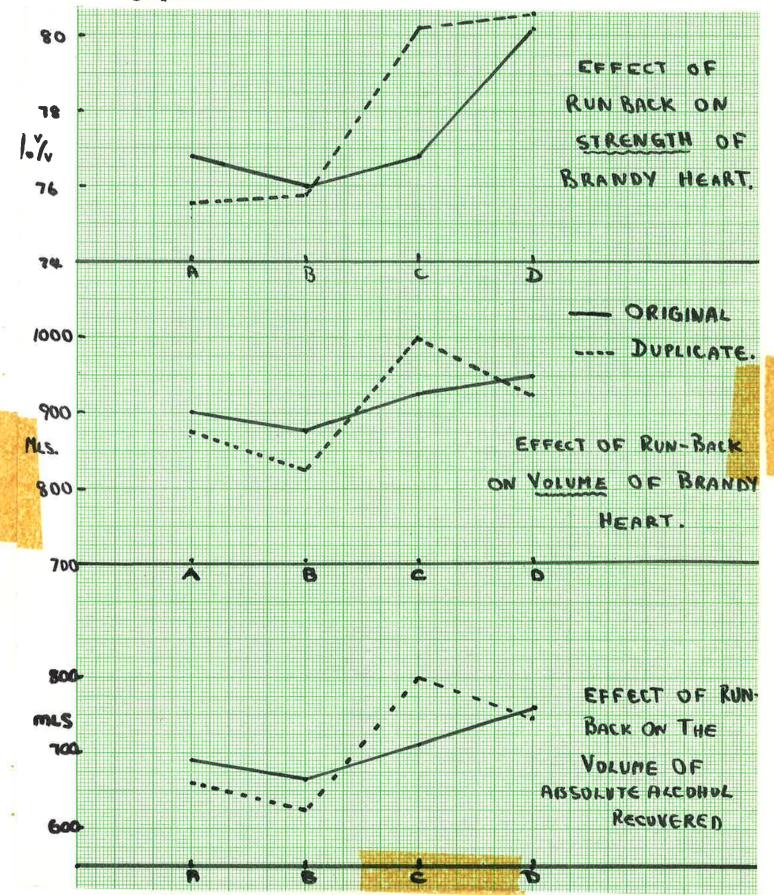
#### EFFECT OF RUN-BACK ON BRANDY HEARTS.

The overall trend of the results shown, indicate that run-back of feints increases the strength and LAL of the brandy heart. This increases only initial and after a number of subsequent distillations, the system reaches an equilibrium.

Thus run-back of heads and tails to subsequent wine charges increases the efficiency of brandy distillation, as it increases the strength of the wine charge and low wine, and thus the strength of the brandy.

The overall yield of alcohol of the brandy heart is increased. Therefore this modified distillery procedure results in a bigger brandy heart. This trend is only slight,

however, as the addition of heads and tails in terms of absolute alcohol, was relatively minor compared to the total alcoholic centent of the wine charge. These results are graphed below:



VI.

# CONCLUSION.

The overall effect of run-back of heads and tails to subsequent wine charges is to slightly increase the alcoholic content of the wine charges, low wines and brandy distillates. The system reaches an equilibrium after a number of consequentive distillations which is higher than the equilibrium of brandy distillation which does not involve this modified distillery procedure.

Thus the efficiency of brandy heart manufacture is slightly increased by this procedure.

This procedure would also eliminate the problem of accumulation of large quantities of heads and tails.

Consequently it also reduces the problem of distilling large volumes of heads and tails which have been put aside during distillery operation.

This modified procedure may fall down, however, if the wines to be distilled were of low quality and contained large amounts of SO<sub>2</sub> or ethyl acetate for example. Poor quality wines would have head and tail fractions of a much higher impurity, and run-back of these feints to subsequent wine charges might well taint the quality of the brandy distillates produced from them.

VII.

# ACKNOWLEDGEMENTS.

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