Grain Irish Whiskey Distillation

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Distillation

What is Distillation?

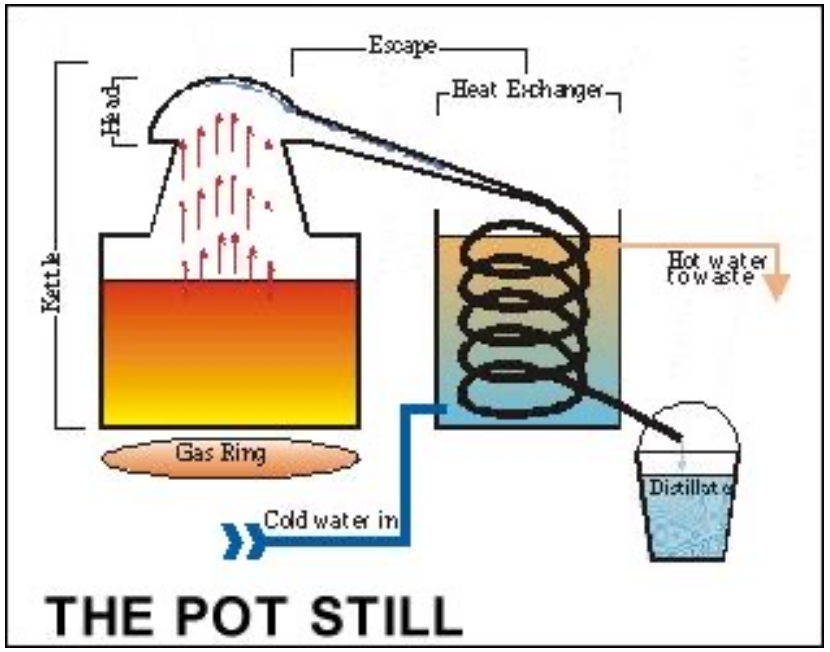
- Distillation is the process of separating the components or substances from a liquid mixture by using selective boiling and condensation.
- It is a Separation technique
- It exploits the difference in boiling points of the components in the liquid mixture being distilled.
- Boiling points
  - Pure Water 100°C
  - Pure Ethanol 78.37 °C
  - 50/50 Water/Ethanol 83 °C

Distillation

Types of Distillation

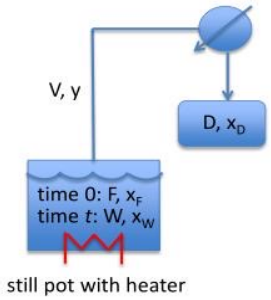
- Simple Distillation
  - Pot stills-no columns
    - Double distilled
    - Two and a half times distilled
    - Triple distilled
- Fractional Distillation
  - Column Stills
    - Wash still
    - Extractive distillation still
    - Rectifier still
- Vacuum Distillation
  - Use for High BP >150 °C
- Steam Distillation
  - Use for heat sensitive compounds that are not water-miscible

Simple distillation



Simple batch distillation

no rectification (= no column)



Characteristics:

- *no* column; a single equilibrium stage (= the still pot)
- single charge (F) to still pot at time = 0
- vapor is withdrawn continuously
- composition of liquid in still pot (x_W) changes continuously
- composition of liquid distillate (x_D) changes continuously

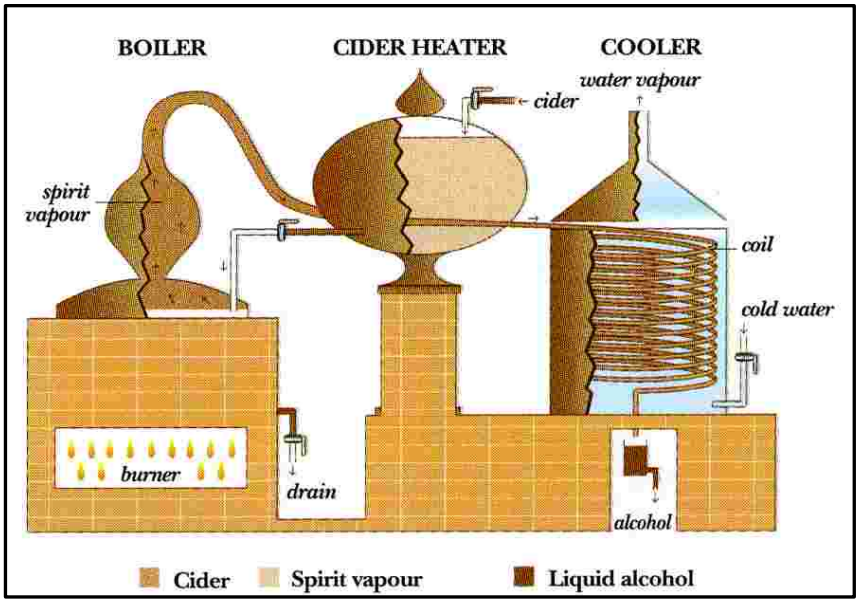
Distillation

Simple Distillation

One single pot distillation

Cider Distillation

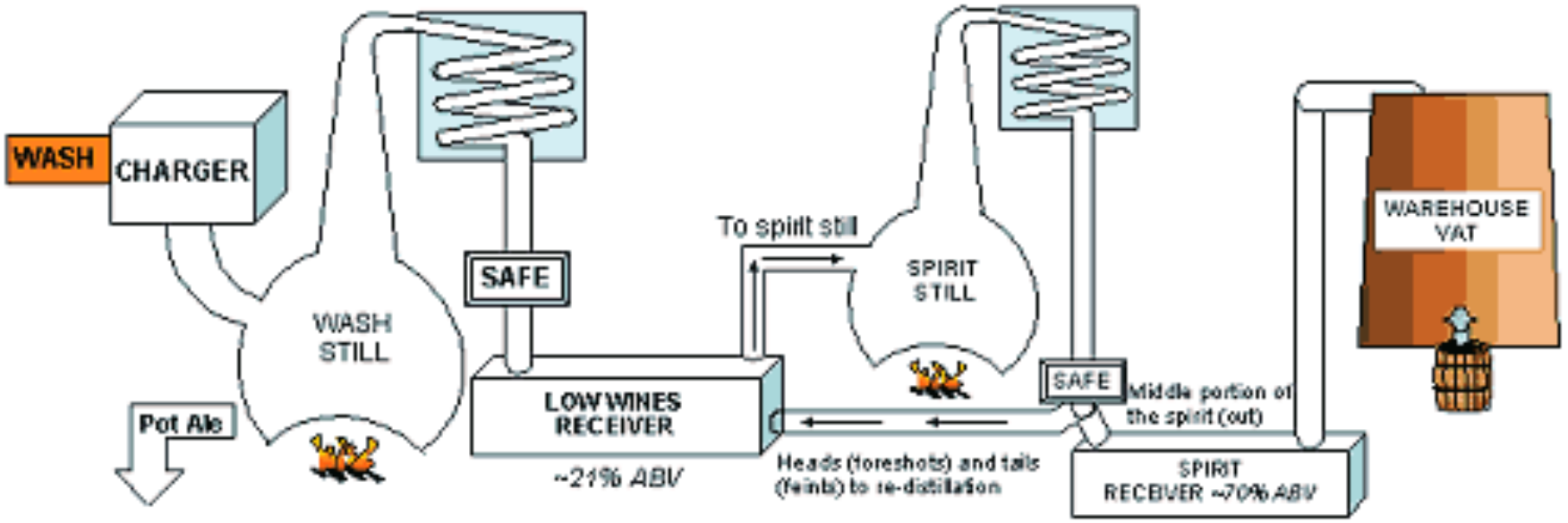
French Cognac Distillation



Distillation

Simple Distillation

Double Distillation: Malt, Peated Malt, Pot Still.

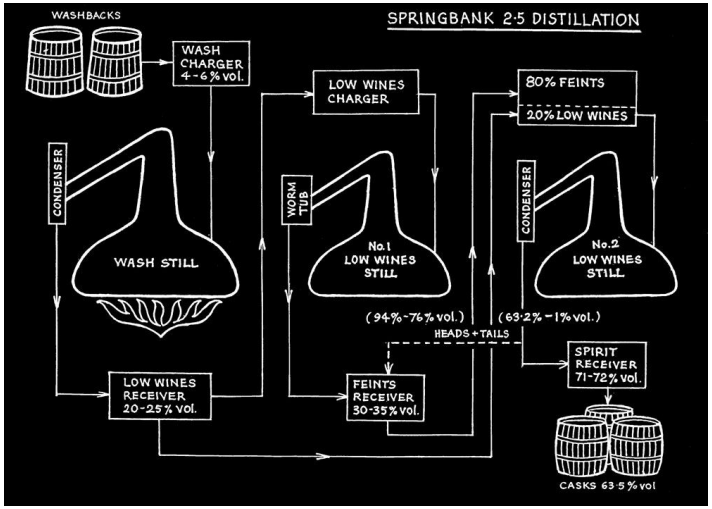


Cooley Distillery

Kilbeggan Distillery

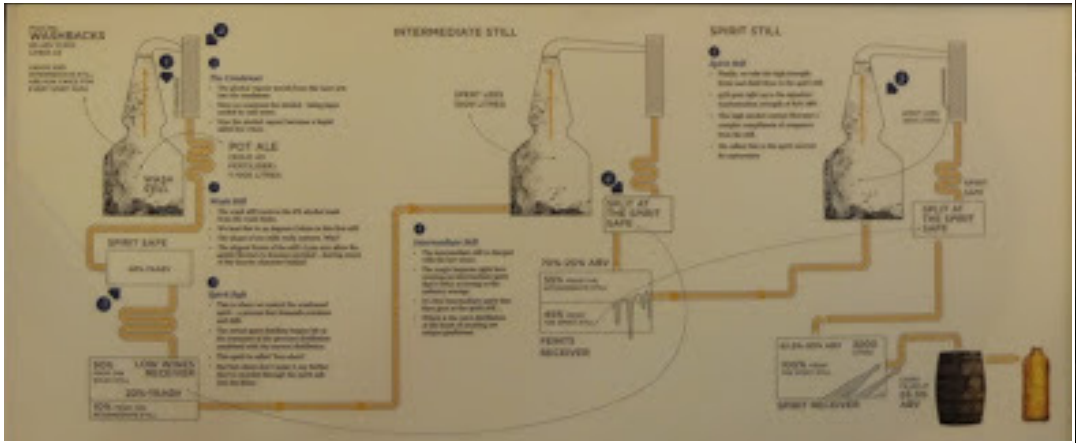
Distillation

Simple Distillation



Springbank 2.5 Distillation

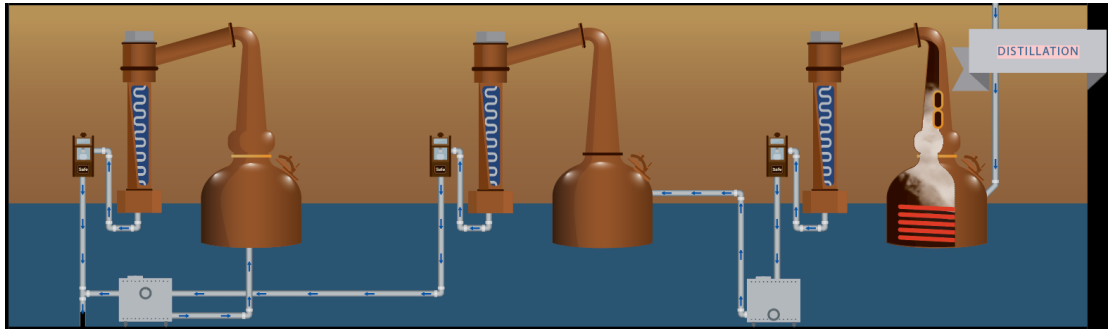
Simple Distillation Auchentoshan Triple Distillation



Distillation

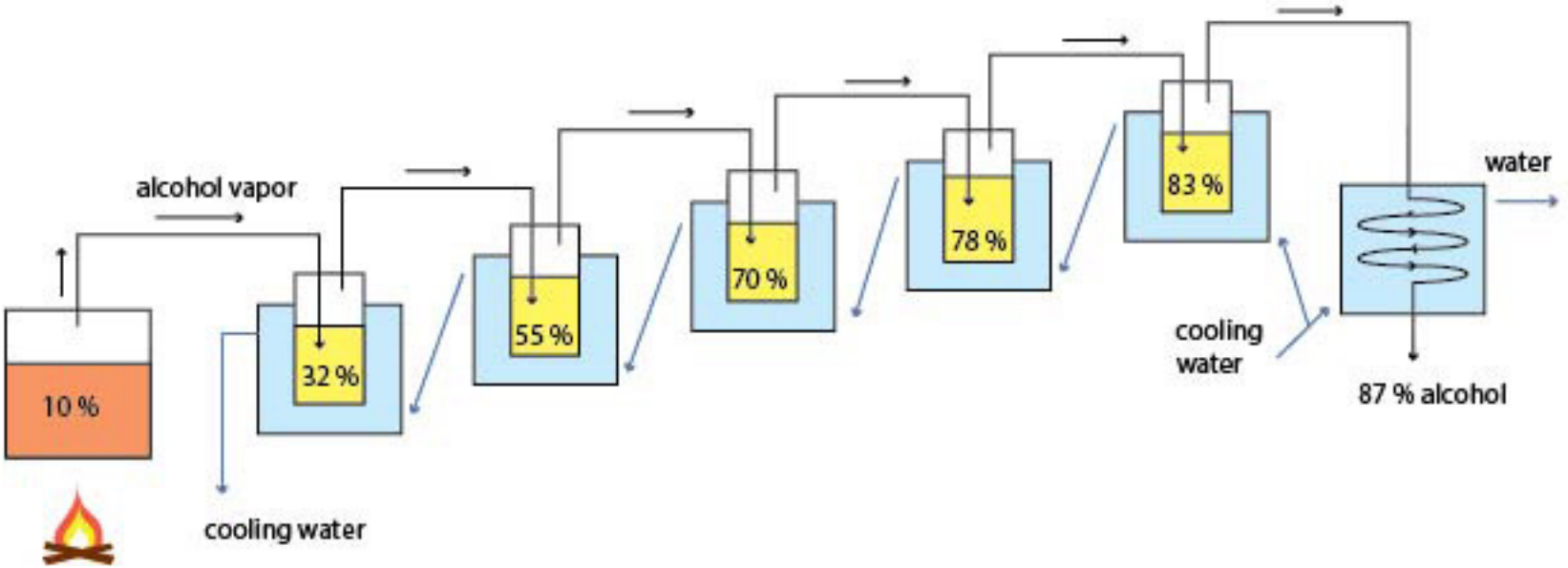
Simple Distillation Powerscourt

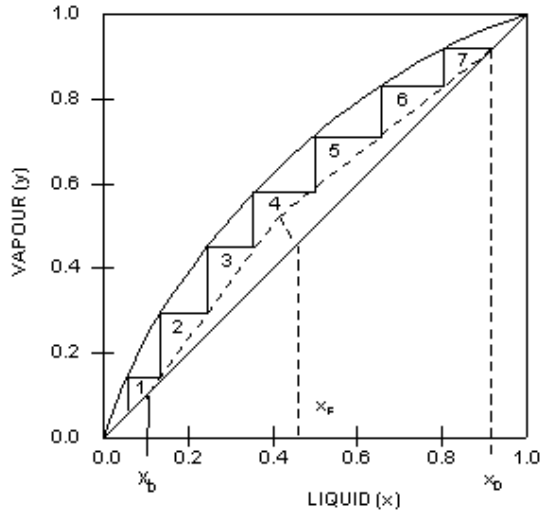
Triple and Double Distillation possible

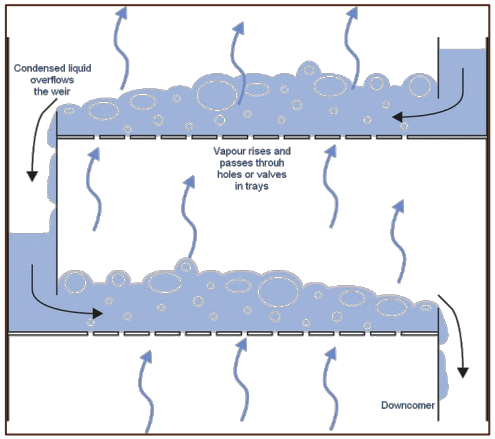


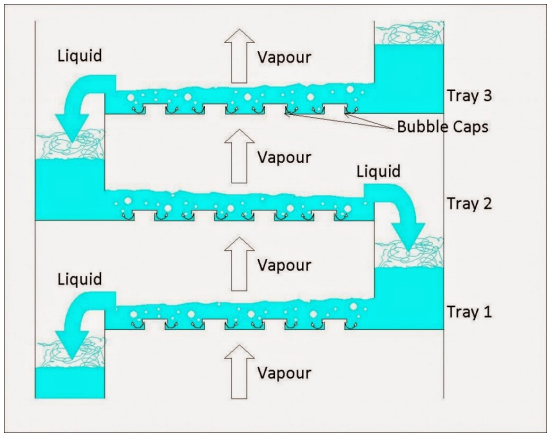
Distillation

Fractional Distillation









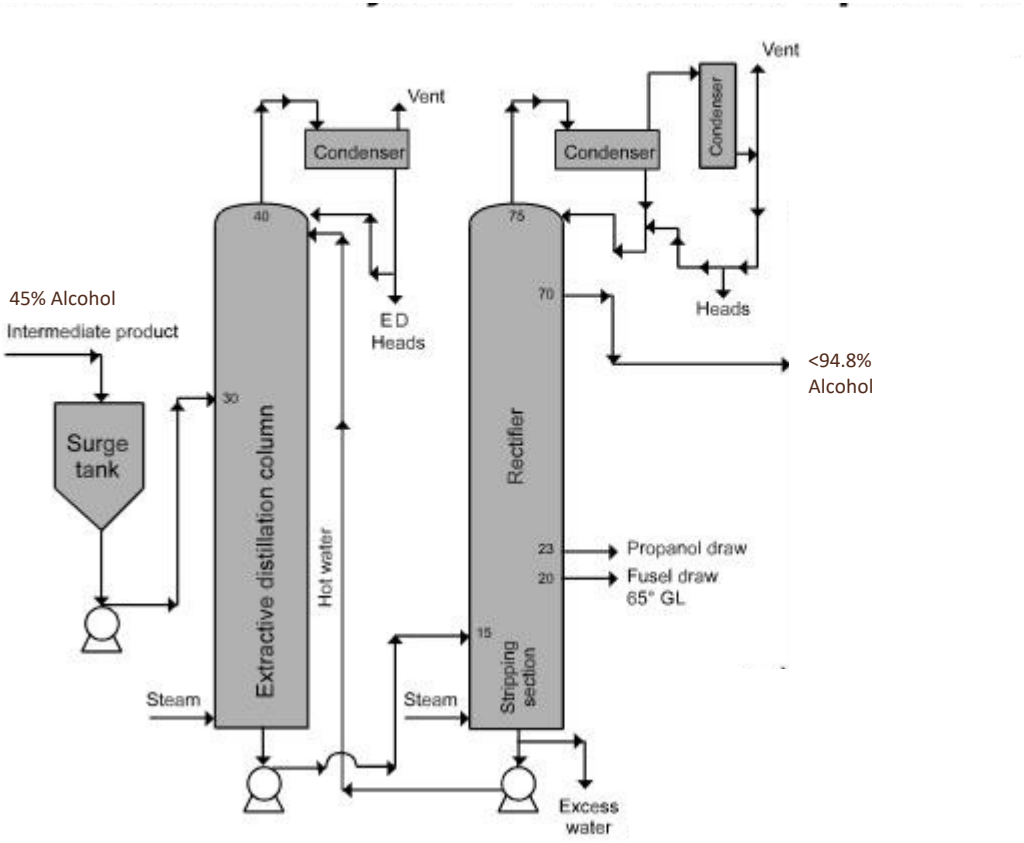
Fractional Distillation

Fractional or column distillation

Bubble Cap tray

Fractional Distillation

Extractive and Rectifier column System for Grain whiskey Production in a 3 still configuration



Distillation

- What's the story about copper?
- Pot stills are made of a very high grade of copper
- Column stills can be made from stainless steel however they typically have sections made of copper or have sacrificial copper mesh or pieces in some sections of the rectifier
- Copper reacts with Sulphur which can be present in the cereals but is also produced by yeast in fermentation.
- The copper used in the distillation stills reduces the sulphur content of the whisky most likely by acting as a catalyst in processes resulting in insoluble copper sulphates (Blue stone)

Distillation

Sulphur compounds in unaged and aged whiskey

No.	Compound (common name)[a]	Formula	Unaged "A"		Aged "A"		Unaged "G"		Aged "G"	
			Concentration ($ng\,mL^{-1}$)	RSD (%), $n=3$	Concentration ($ng\,mL^{-1}$)	RSD (%), $n=3$	Concentration ($ng\,mL^{-1}$)	RSD (%), $n=3$	Concentration ($ng\,mL^{-1}$)	RSD (%), $n=3$
3	Diethyl sulfite	$C_4H_{10}O_3S$	100	2.2	0.52	3.4	93	3.7	0.73	7.3
4	Ethyl methanesulfonate	$C_3H_8O_2S$	28	2.0	62	1.5	50	2.1	75	0.37
9	3-Methyl sulfanyl propyl acetate	$C_6H_{12}O_2S$	35	2.5	0.32	12	20	2.6	0.35	6.1
10	1-(1,3-thiazol-2-yl)ethanone (2-acetyl thiazole)	C_5H_5NOS	13	7.6	9.1	4.4	15	12	17	3.1
11	Thiophene-3-carbaldehyde (3-formyl thiophene)	C_5H_4OS	2.8	5.9	4.5	6.0	3.4	1.7	18	4.5
12	Thiophene-2-carbaldehyde (2-formyl thiophene)	C_5H_4OS	28	3.6	21	3.1	22	2.6	28	0.59
13	3-Methyl sulfanyl propan-1-ol (methionol)	$C_4H_{10}OS$	110	3.5	nd	–	32	5.1	nd	–
16	3-Methylthiophene-2-carbaldehyde (3-methyl-2-formyl thiophene)	C_6H_6OS	9.6	4.6	17	1.1	15	2.2	13	3.2
18 + S4	2-Methyl-1,3-benzothiazole + S4	$C_8H_7NS/C_3H_8OS_2$	26	4.1	nd	–	31	1.4	nd	–
19	1,3-Benzothiazole	C_7H_5NS	210	2.3	1.2	9.5	170	2.5	1.0	4.5
S1	S1	$C_5H_{10}O_2S$	11	3.5	nd	–	21	5.1	nd	–
S2	S2[b]	$C_5H_{12}OS_2$	6.4	4.3	0.41	1.3	9.5	2.6	0.30	10
S5	S5	$C_8H_{15}NOS$	110	6.0	nd	–	100	2.7	nd	–

[a] Common name was shown in a parenthesis.
[b] S2 was identified as 1-ethoxy-2-(methyldisulfanyl)ethane (3,4-dithiapentyl ethyl ether) from Refs. [4,22].

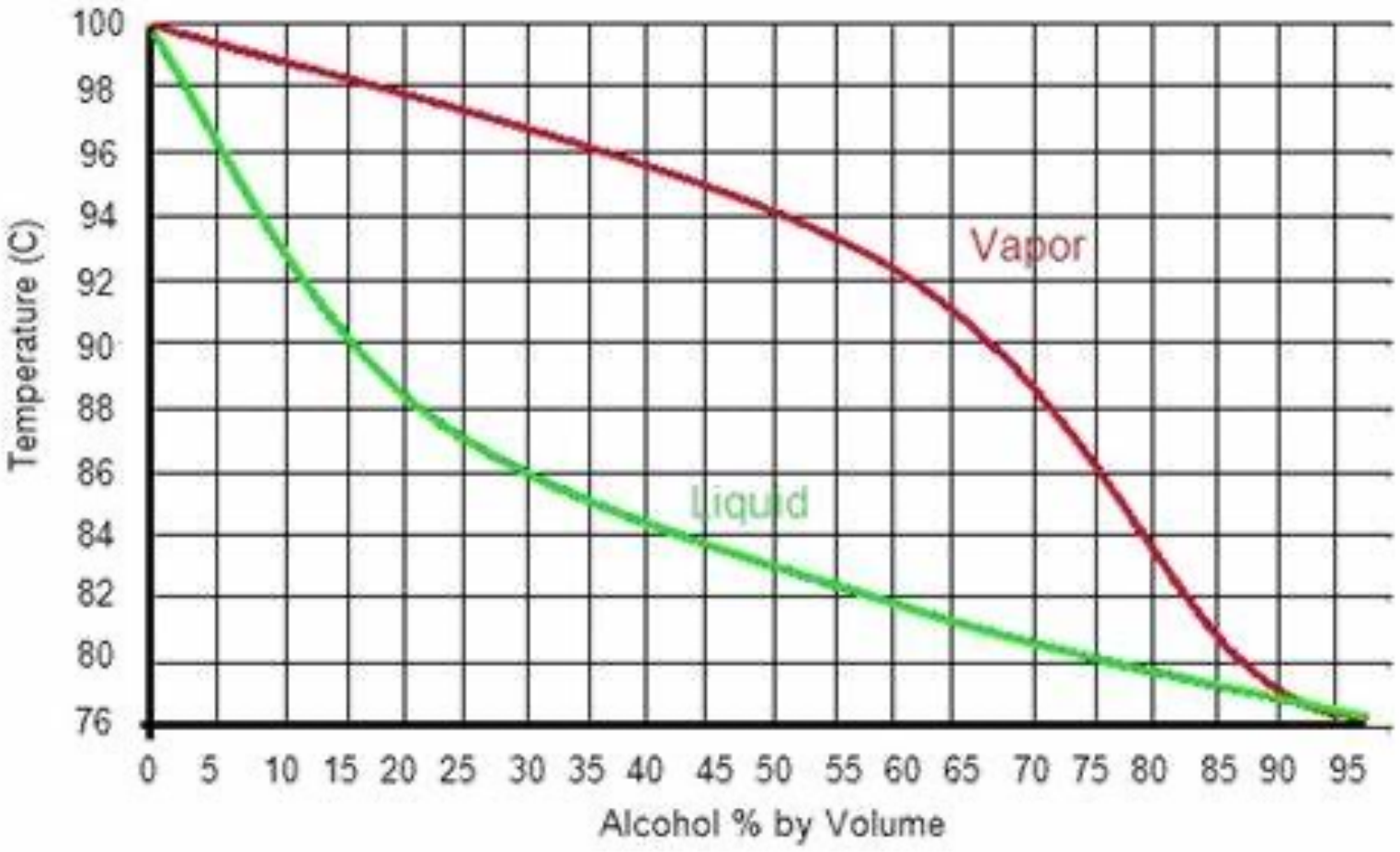
Sulphur compounds can also be present wine casks

Fractional Distillation

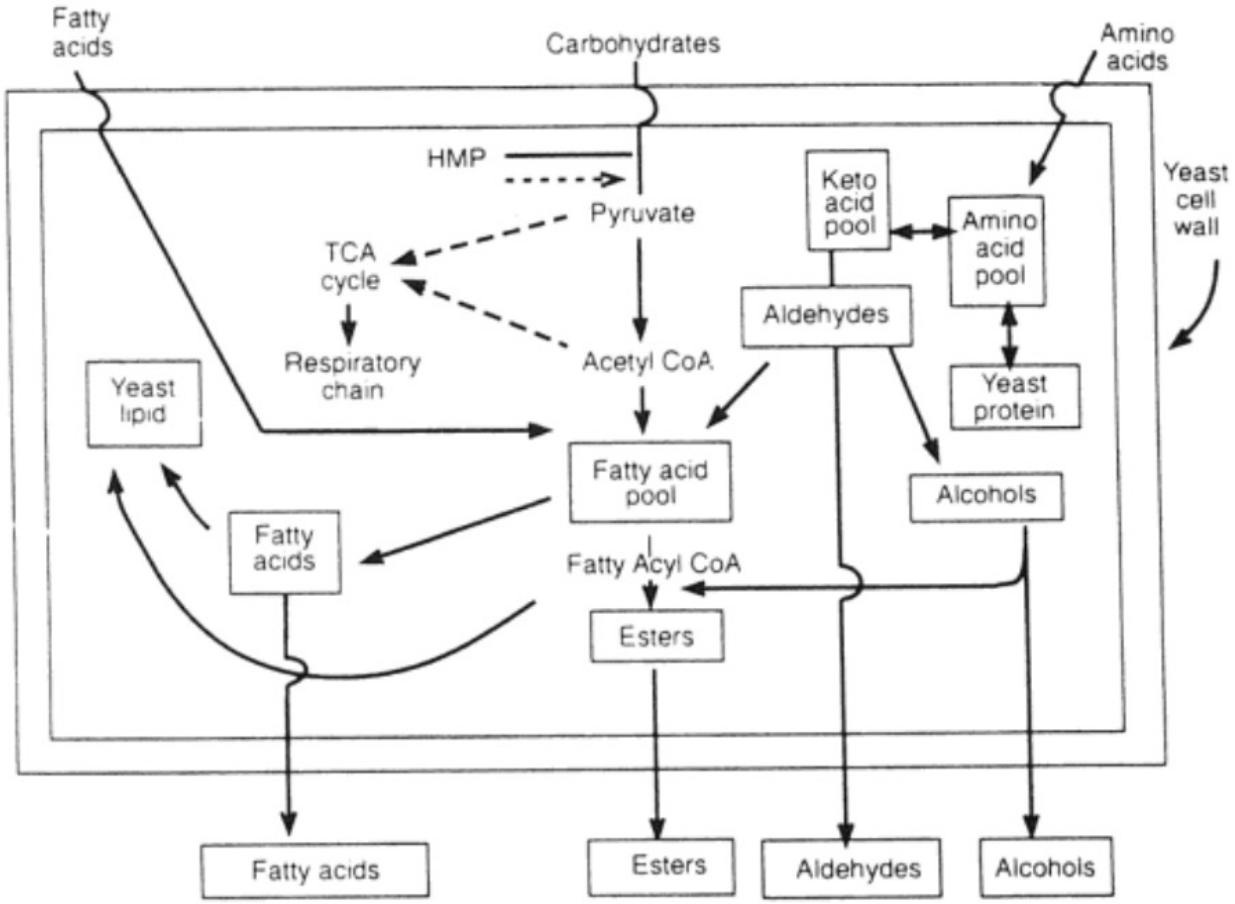
Fractional Distillation

- Coffee still named after Aeneas Coffee 1780-1852 Revenue Officer
- Probably made for India
- Currently in Kilbeggan

Fractional Distillation

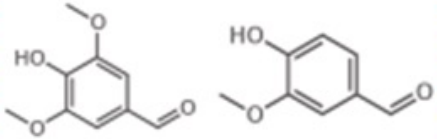


The picture below sums the simple reactions involved in the flavour formation during alcoholic fermentation.



Distillation

ALDEHYDES

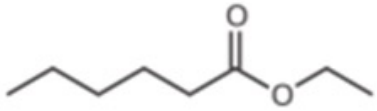


SYRINGALDEHYDE & VANILLIN

A number of different aldehydes can be extracted from oak barrels into the whisky. Syringaldehyde lends a spicy, woody aroma, whilst vanillin gives a vanilla tone. Furfural adds an almond-like grainy flavour, while hexanal offers grassy notes.

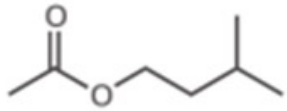
FURFURAL & HEXANAL

ESTERS



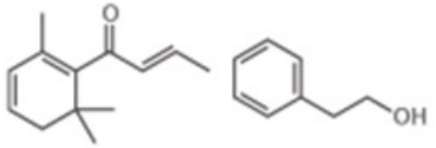
ETHYL HEXANOATE

Esters in whisky can add fruity flavours, such as ethyl hexanoate, which gives a sweet apple flavour. Isoamyl acetate, on the other hand, gives a banana-like, pear drop aroma. Some whiskies are chill filtered to remove the majority of esters, as they can cause cloudiness in the whisky.



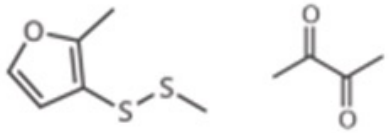
ISOAMYL ACETATE

OTHER COMPOUNDS



ß-DAMASCENONE & PHENETHYL ALCOHOL

ß-damascenone and phenethyl alcohol add floral notes to whisky. Diacetyl, a common off-flavour in beer, contributes a buttery taste. Other burnt, rubbery off-flavours can be produced by sulfur compounds, though they can also add meaty tones.



2-METHYL-3-(METHYLDISULFANYL)FURAN & DIACETYL

Fatty Acids

The short chain fatty acid esters (C6, C8) are formed early in the fermentation.
The medium chain esters (C10,C12) quite evenly throughout the fermentation. The longer chain esters (C16) mostly at the cell-death phase.

Increased cell growth usually results in lower levels of esters, due to lower levels of free fatty acids in the wort, as fats are used to build cell membranes.

Organic acids are formed throughout the fermentation and at high levels they produce notes of vinegar, vomit and barnyard.

The right proportion of fusel alcohols and free fatty acids or acetate is crucial when producing estery wort and avoiding the solventy off-notes from the excess alcohols and on the other hand the rancid aromas from the excess free fatty acids.

An estery, fruity wort can be produced with warm long fermentations, high original gravities, high pitching rates with aerobically grown yeast and low nitrogen barley.

Increased glucose levels tend to produce more short chain esters, for example isoamyl acetate with a typical banana aroma.

High fermentation temperatures usually produce more acetate esters with mainly fruity aromas, but also medium-long chain ethyl esters, which can give an oily and waxy texture to the flavour.

Esters

Ester formation depends on the amount of fusel alcohols and organic acids in the wort, but also on the activity of alcohol acetyltranferase enzymes (ATAase I and II), which in turn depends greatly on the yeast strain.

Esters in the fermentation can be classified into two groups:

- The acetate esters (acetate+alcohol)
- The ethyl esters (ethanol+fatty acid).

The acetate esters are usually formed in greater amounts, but the ethyl esters can be very aromatic even in low concentrations.

Esters contd.

Fusel alcohols themselves are not a desired flavour in the wort - producing usually a sharp, solventy notes - but together with acids they form esters, which are important and desired flavour compounds in whisky as they produce various fruity and flowery notes.

The table below shows Aminoacids which metabolise into different fusel alcohols

Amino acid	Fusel alcohol
Leucine	Isoamyl alcohol
Valine	Isobutanol
Isoleucine	Active amyl alcohol
Phenyl	2-phenylethanol
Tyrosine	p-hydroxyphenylethanol / tyrosol
Tryptophan	Tryptophol
Methionine	Methionol

Common descriptors for the aromas of esters are listed in the table below

Esters Contd.

Ester Name	Odor or occurrence	Ester Name	Odor or occurrence
Allyl hexanoate	pineapple	Linalyl butyrate	peach
Benzyl acetate	pear, strawberry, jasmine	Linalyl formate	apple, peach
Bornyl acetate	pine	Methyl acetate	glue
Butyl butyrate	pineapple	Methyl anthranilate	grape, jasmine
Ethyl acetate	nail polish remover, model paint, model airplane glue	Methyl benzoate	fruity, ylang ylang, feijoa
Ethyl butyrate	banana, pineapple, strawberry	Methyl butyrate (methyl butanoate)	pineapple, apple, strawberry
Ethyl hexanoate	pineapple, waxy-green banana	Methyl cinnamate	strawberry
Ethyl cinnamate	cinnamon	Methyl pentanoate (methyl valerate)	flowery
Ethyl formate	lemon, rum, strawberry	Methyl phenylacetate	honey
Ethyl heptanoate	apricot, cherry, grape, raspberry	Methyl salicylate (oil of wintergreen)	Modern root beer, wintergreen
Ethyl isovalerate	apple	Nonyl caprylate	orange
Ethyl lactate	butter, cream	Octyl acetate	fruity-orange
Ethyl nonanoate	grape	Octyl butyrate	parsnip
Ethyl pentanoate	apple	Amyl acetate (pentyl acetate)	apple, banana
Geranyl acetate	geranium	Pentyl butyrate (amyl butyrate)	apricot, pear, pineapple
Geranyl butyrate	cherry	Pentyl hexanoate (amyl caproate)	apple, pineapple
Geranyl pentanoate	apple	Pentyl pentanoate (amyl valerate)	apple
Isobutyl acetate	cherry, raspberry, strawberry	Propyl acetate	pear
Isobutyl formate	raspberry	Propyl hexanoate	blackberry, pineapple, cheese, wine
Isoamyl acetate	pear, banana (flavoring in Pear drops)	Propyl isobutyrate	rum
Isopropyl acetate	fruity	Terpenyl butyrate	cherry
Linalyl acetate	lavender, sage		

Alcohols

Homogeneous Azeotropes	**Boiling Point °C**
Ethanol	78.5
Iso-propanol (2-propanol)	82.3
Tert-butanol (2-me-2-propanol)	82.8
Heterogeneous Azeotropes	
Iso-butanol	108.4
2-methyl-1-propanol (tert amyl)	102.3
2-butanol (sec)	99.5
2-methyl-1-butanol	128.0
3-methyl-2-butanol	112.9
3-pentanol	115.6
2-pentanol	119.3
1-butanol	117.7
Iso-pentanol (Iso-amyl)	132.0
Pentanol (n-amyl)	138.0
c-pentanol	140.9
2-ethyl-1-butanol	148.9
Hexanol	158.0
1-octanol	195.0
2-butyl-octanol	253.4
Decanol	283.0

If the constituents of a mixture are completely miscible in all proportions with each other, the type of azeotrope is called a ***homogeneous azeotrope***.

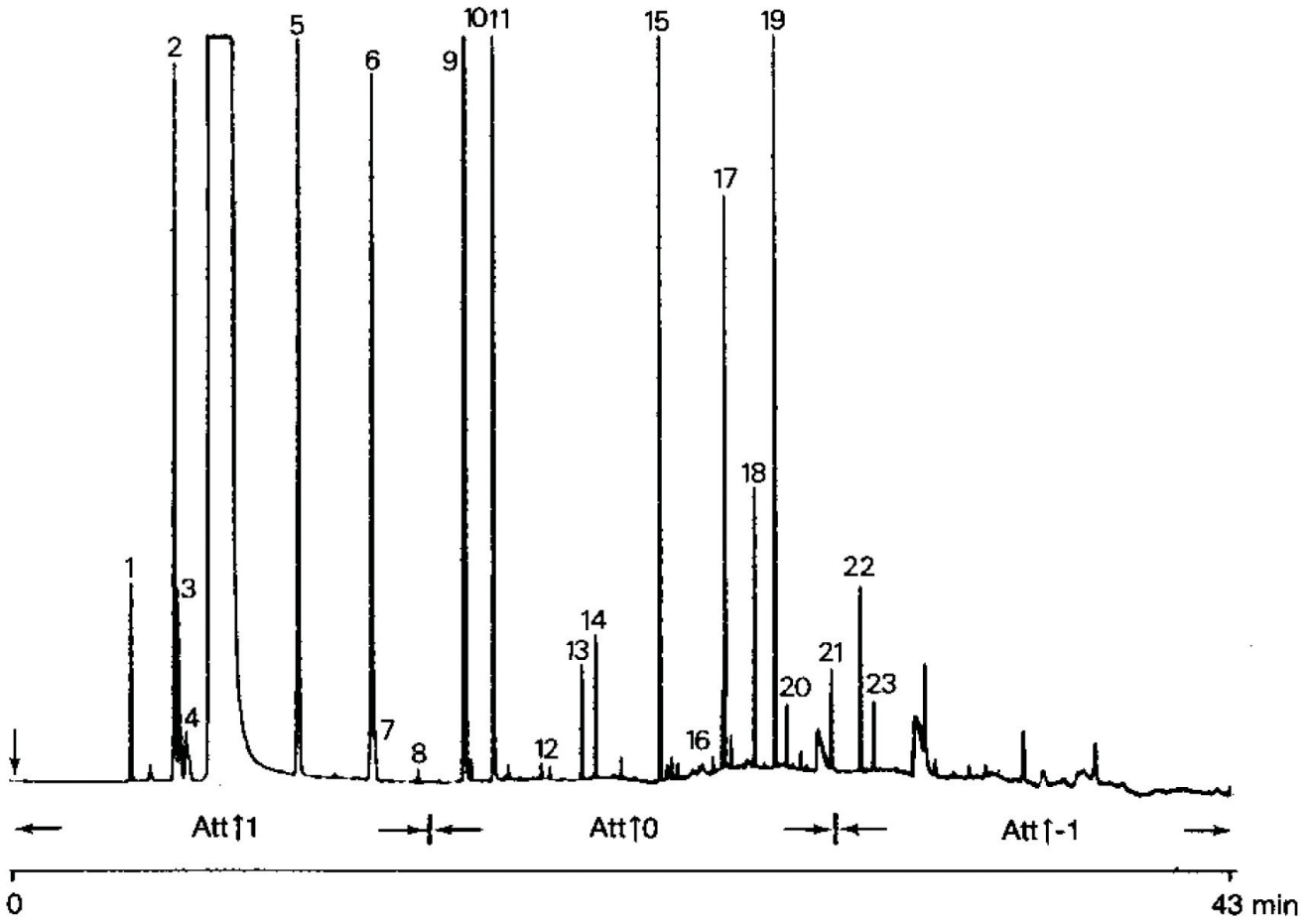
If the constituents are not completely miscible, an azeotrope can be found inside the miscibility gap. This type of azeotrope is called ***heterogeneous azeotrope***

Distillation

Peak identification

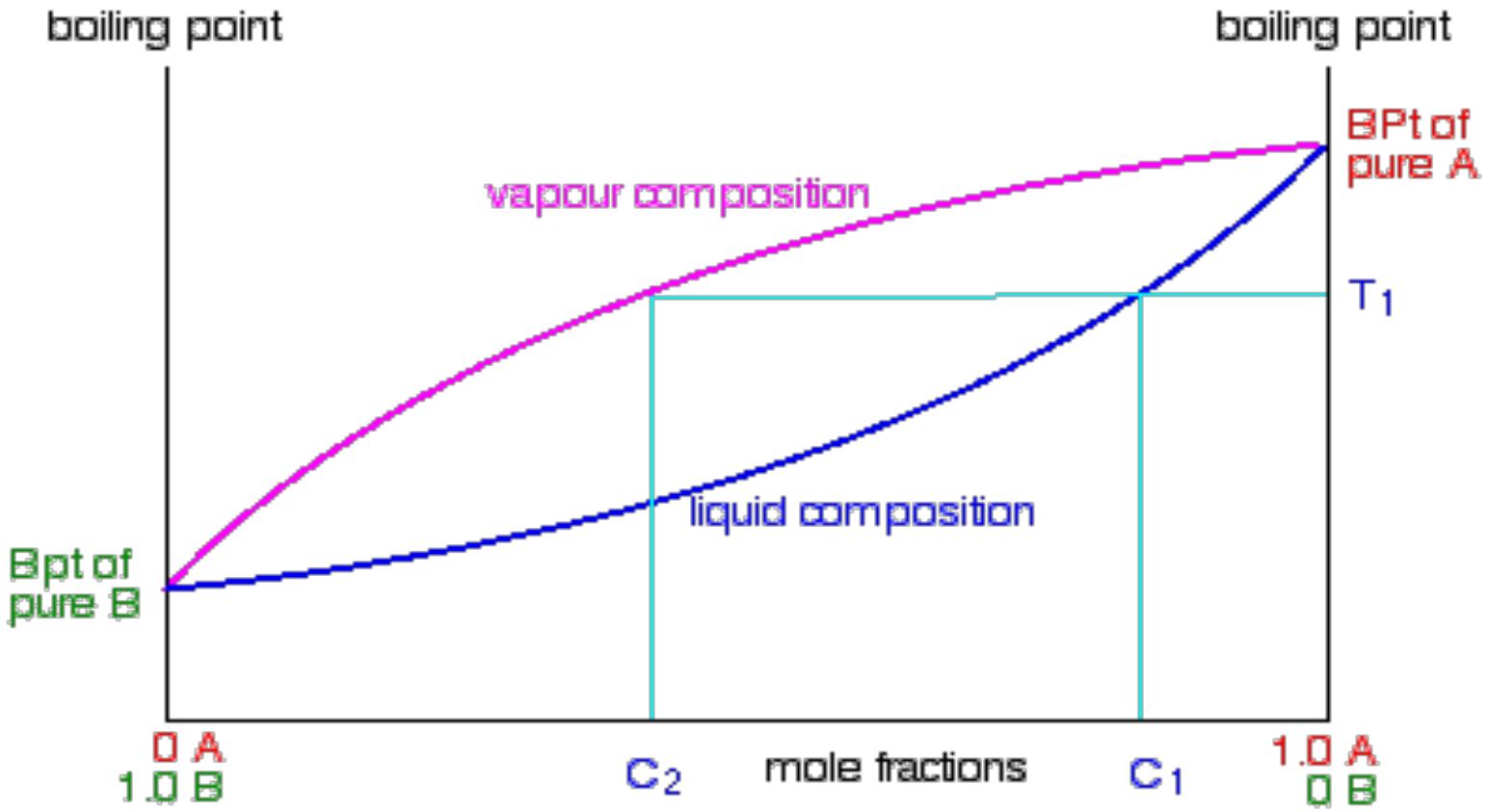
1.	acetaldehyde
2.	ethylacetate
3.	acetal
4.	methanol
5.	propanol
6.	iso-butanol
7.	iso-amylacetate
8.	n-butanol
9.	2-methyl-1 -butanol
10.	3-methyl-1-butanol
11.	n-pentanol
12.	ethylactate
13.	ethylcaprylate
14.	furfural
15.	ethylcaprate
16.	2-phenylethylacetate
17.	ethyllaurate
18.	2-phenylethylalcohol
19.	methylmyristate
20.	ethylmyristate
21.	myristylalcohol
22.	ethylpalmitate
23.	ethylpalmitoleate
24.	ethylstearate
25.	ethyloleate
26.	ethyllinoleate
	Peaks 11 and 19 are standards

Gas chromatograph of Malt Whiskey



DISTILLATION

In this diagram, if you boil a liquid mixture C_1, it will boil at a temperature T_1 and the vapour over the top of the boiling liquid will have the composition C_2.



Fractional Distillation

Fractional distillation
Azeotropic Distillation