Batch Distillation

➢ ABSTRACT :

In this experiment, it is required to carry out the distillation of a methanol and water mixture to determine the variation in the top concentration. In order to find the density, the samples of the top product that is collected are weighted. After recording the results, they are then subtracted from the mass of bottle that contained the samples and the results are recording again, these results are divided by volume of the bottle to get the density value. The methanol percentage determined from the graph paper attached with this report. In this report, the above information will be shown in much more detail.

➢ OBJUCTIVE:

To carry out the distillation of a methanol and water mixture to determine the variation in the top concentration.

➢ INTORDACTION:

Distillation is the most widely used method of separating mixture of liquids into their component chemicals, and it's an important process in the chemical industry. The process uses the fact that when a liquid containing tow or more components is heated to its boiling points, the composition of the vapour is usually different that to the liquid, (richer in the chemical components with the lower boiling point).

The most common type of distillation column is the 'Fractionating Column' which can achieve good separation of the liquid mixture. Batch distillation is used extensively in the laboratory and in small production unit that may be used to produce a variety of chemicals.

➢ THEORY:

The composition, (purity), of the top product from a batch fractionating still, depends on both the composition of the original charge to the boiling vessel, and the effectiveness of the fractionating column.

When distilling a mixture of two components, such as methanol and water, initially the top product will contain a relatively high % of the more volatile component (i.e. the one with the lower boiling point – methanol in this case). How ever, (based on mass balance considerations), as top product rich in methanol is continuously removed from the column, the liquid remaining in the batch still must contain less and less methanol. When the fractionating column is operated at a constant reflex ratio, it cannot compensate for the steady the top product must also get less and less.

The boiling point temperature of a mixture of two liquid components depends on both the boiling points of the pure components and the proportion of each component in the mixture. A methanol and water mixture with high methanol content has a boiling point close to that of methanol, but as the methanol content gets less and less, the boiling point temperature increases towards the boiling point of water. Hence, as the top product is always richer in methanol than the liquid in the boiling vessel, so the temperature at the fractionating column should always be lower than the temperature in the boiling vessel.

Thus, as the distillation proceeds, it is expected that: -

The Methanol content in the top product will steadily decrease,
The temperature both at the column and in the boiling vessel will steadily increase.
Also the volume of the top product collected should increase steadily as the distillation proceeds.

➢ PROCEDER:

The 'batch still' consists of an electrically heated boiling vessel linked to a fractionation column packed with the glass cylinders. Above the column is a water cooled condenser, and a 'reflux divider' allows top product to be removed at different rates. Vapour from the boiling vessel rises up through the fractionation column and condenses in the condenser. This liquid condensate is returned to the top of the fractionate column and flow down the column to the boiling vessel. Under 'total reflux 'conditions all of the condensate is returned to the column. (These conditions are used when the still starting up).

During the main distillation process the still was operated at the particular 'reflux ratio', and under these conditions some liquid top product was removed from the condenser and flows to a product receiver vessel. Samples could be taken of the top product, and the temperature at the top of the column and in the boiling vessel could be measured.

The boiling vessel was filled to approximately above the top of heating jacket with a mixture of methanol and water (about 20% methanol, 80%water on a volume basis).

The cooling water to the condenser was turned on, then the heating jacket was turned on, (water on the first for safety). With the reflux divider switched to manual – total reflux, allow the system to operate until equilibrium has been reached, (when the temperatures at the top of the column and in the boiling vessel became constant).

When the system has been reached equilibrium, the temperatures at the top of the column and in the boiling vessel were noted and the reflux divider was switched to auto. This automatically divides the condensate into two parts for top products and one part for return to the top of the column. A 400 cm^2 simple of the top product was taken then the simple was placed in cold water to cool it down so that could be analysed.

The distillation was carried out for approximately 2 hours, a 400 cm^2 simple was taken every 15 minutes of the top product, the total amount of product collected up to that time was measured, and the temperatures at the top of the column and in the boiling vessel were noted. Each simple was placed in cold water to cool it down for analyzing.

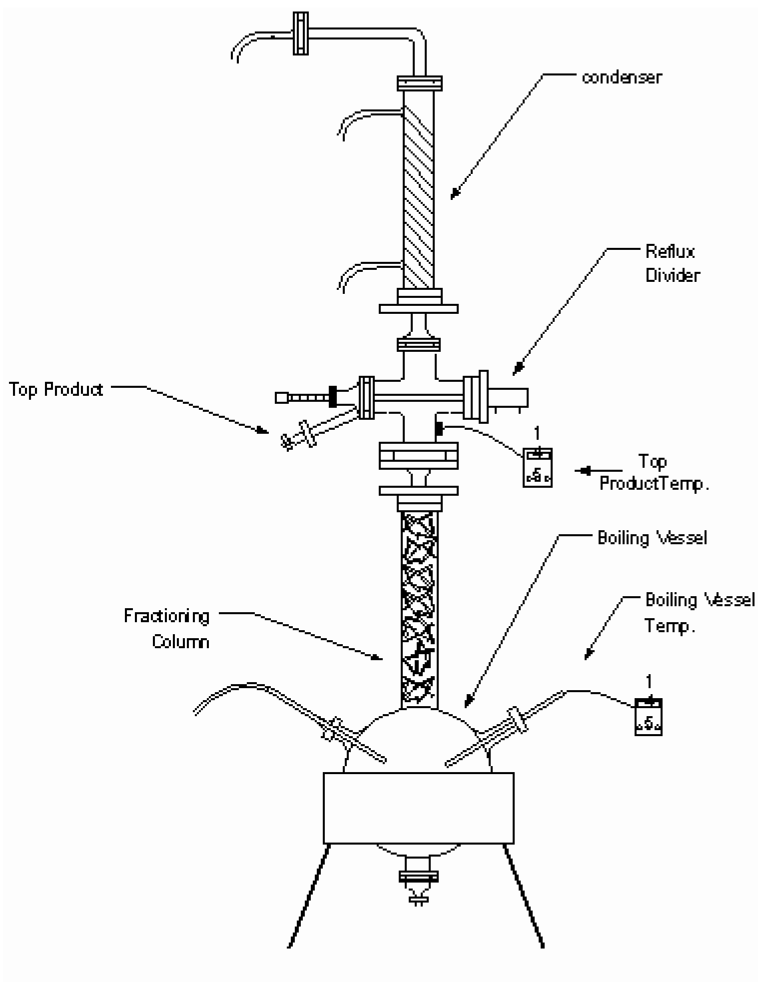
The reflux divider to 'total reflux' was switched at the end of the distillation, and the electric heating jacket was switched off, but the cooling water flowing *through the condenser was left until the system was completely cooled down, (for safety!).*

The samples of the top product for methanol content was analysed by using the fact that mixture of water and methanol of different methanol content have different density values.

➢ HAZZARDS:

- *Don't touch the boiling vessel**, because it's too hot.***
- *Don't let methanol to got to your eyes*

➢ Diagram:



➢ THE RESULT:

Time (min)	*Top Temp.(C^o)*	*Boling vessel Temp.(C^o)*	*Volume (cm^3)*
0	66	89	0
15	67	90	250
30	67	91	545
45	68	92	835
60	71	92	1115
75	72	93	1380
90	74	94	1640
105	75	95	1890
120	83	95	2125

Table (1)

Note:

Volume of the gravity bottle (V) =25 cm^2
Mass of the gravity bottle (m_1) = 23.2028 g
Gravity bottle + sample = (m_2)
Density = $\frac{mass}{Volume}$

m_2(g)	*(m_2-m_1)g*	*Density (Kg/m^3)*	*Methanol Percentage*
43.4792	20.2764	881.056	92.5%
43.5363	20.3335	813.34	90.5%
43.5820	20.3792	815.168	89%
43.6714	20.4686	818.744	87.5%
43.8655	20.6627	826.608	48%
44.1295	20.9267	837.068	79.5%
44.3893	21.1865	847.46	74%
44.6199	21.4171	856.684	70%
44.8895	21.6867	867.468	64%

Table (2)

➢ CALCULATION:

- ***To got the density of water for each run we used this equation:-***

$$\textit{Density} = \frac{\textit{Mass of sample in the bottle, Kg}}{\textit{Volume of the bottle, } m^3}$$

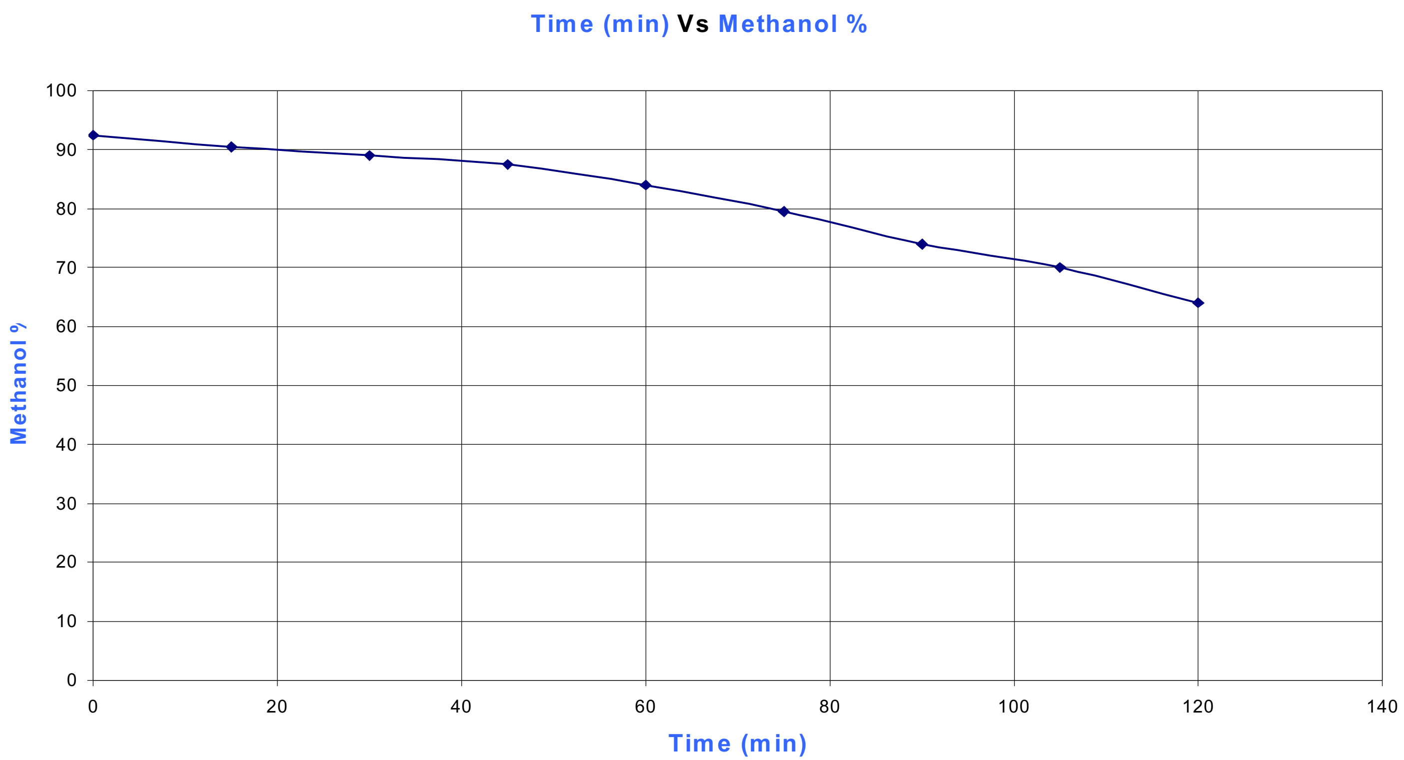
Example:

$$\textit{Density} = \frac{20.2764 \times 10^{-3}}{25 \times 10^{-6}} = 881.056 \text{ (for first run)}$$

- ***To get the methanol percentage for each run we used the graph which is attached with the report.***

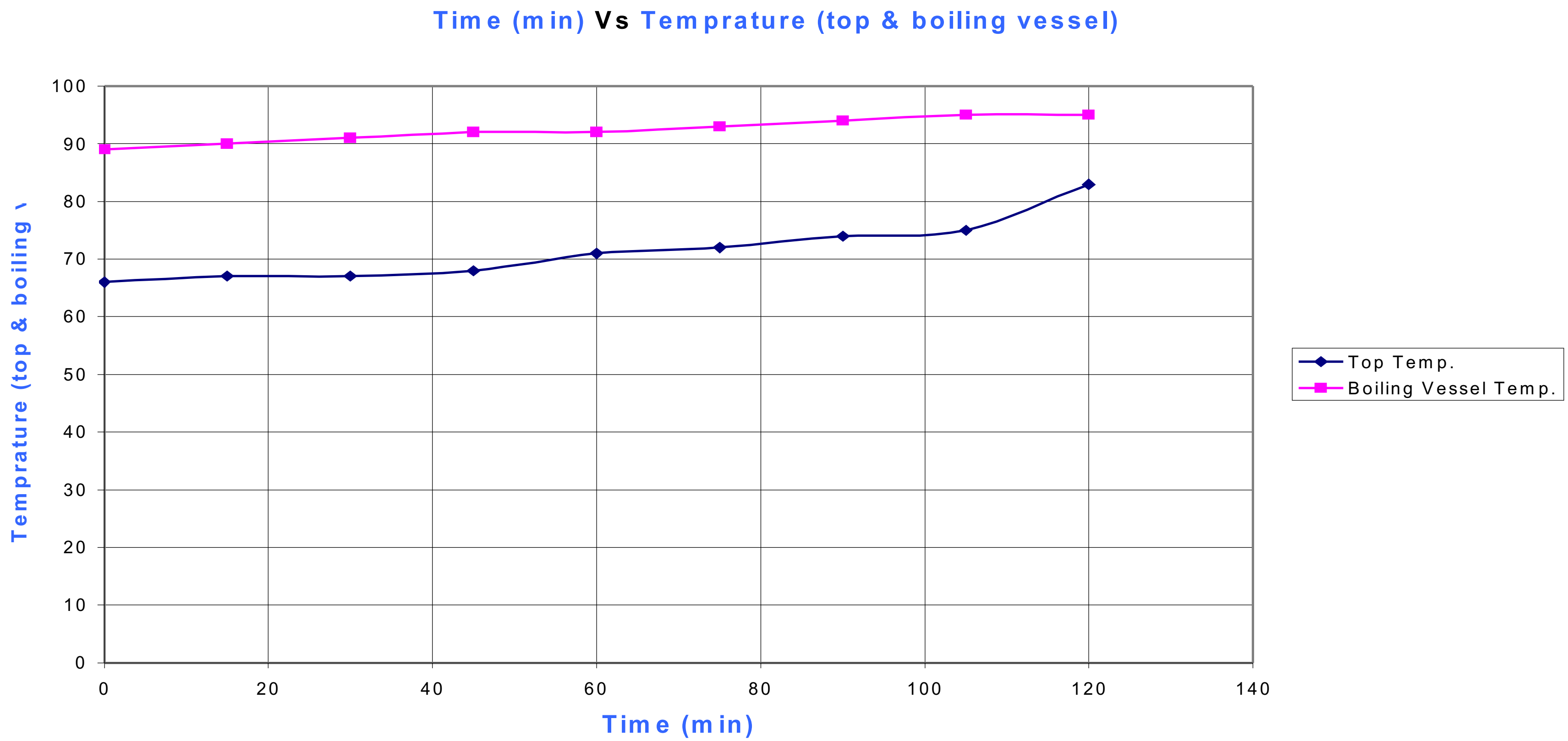
➢ GRAPHS:

- ***Graph (1):***



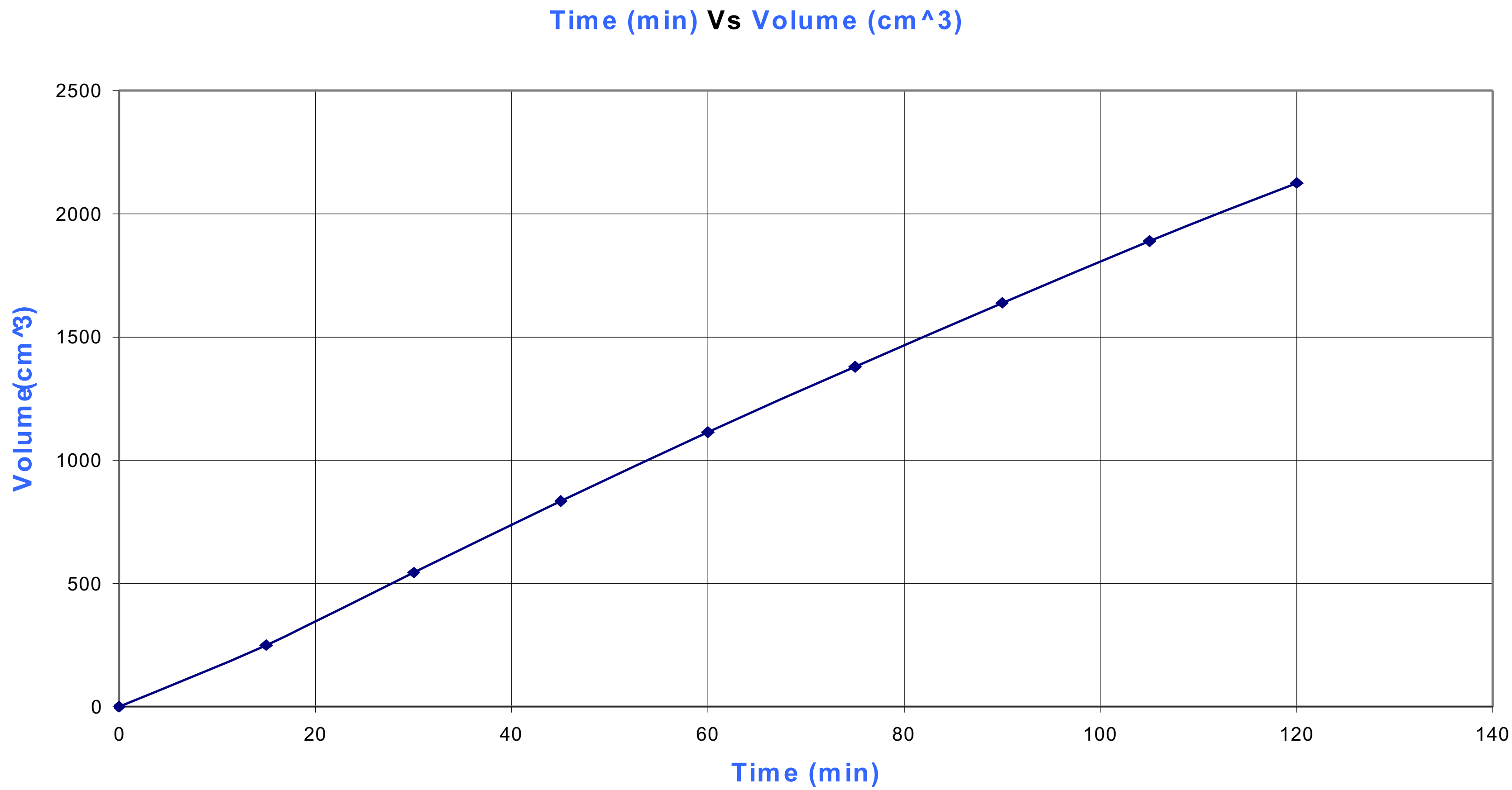
The above graph explains the relation between the time which is on the x-axis and the methanol percentage in the y-axes. We can see from the graph that when the time is increasing the methanol percentage is decreasing, and that what was discussed in the theory.

- ***Graph (2):***



From the previous graph that explains the relation between the both of the boiling vessel and the top temperature against time; we can observe that as the time increasing the temperature is increasing as well, and that what was discussed in the theory.

- *Graph (3)*



The above graph explains the relation between volumes which is on the y-axis against the time which is on the x-axis. We can see from the graph that as the time is increasing the volume is increasing as well, and that what was discussed in the theory.

- **DISSCUSSING THE RESULT& CONCOLUTION:**

I think the results I got are right, because when I compared my graphs to expected shape in the theory, I got the same shape. I didn't got a good percentage of water that because the temperature which I started from was low (89 C^o), if I started from higher temperature I think I was going to get better percentage of water in the last run.