

Nuffield Advanced Chemistry
CHEMICAL ENGINEERING
A Special Study

EXPERIMENTS

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Experiment 7 Reflux ratio

These are the experiments for the Chemical engineering Special Study
The text is supplied as a separate document

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Health and Safety

See the safety notes given with each experiment.

Health and safety in school and college science affects all concerned: teachers and technicians, their employers, students, their parents or guardians, as well as authors and publishers.

As part of the reviewing process, these publications have been checked for health and safety. In particular, we have attempted to ensure that:

- all recognized hazards have been identified,
- suitable precautions are suggested,
- where possible, the procedures are in accordance with commonly adopted model (general) risk assessments,
- if a special risk assessment is likely to be necessary this has been pointed out
- where model (general) risk assessments are not available, we have done our best to judge the procedures to be satisfactory and of an equivalent standard.

It is assumed that:

- practical work is conducted in a properly equipped and maintained laboratory,
- rules for student behaviour are strictly enforced,
- mains-operated equipment is regularly inspected, properly maintained and appropriate records are kept,
- care is taken with normal laboratory operations such as heating substances and handling heavy objects,
- good laboratory practice is observed when chemicals are handled,
- eye protection is worn whenever risk assessments require it,
- any fume cupboard required operates at least to the standard of Building Bulletin 88,
- students are taught safe techniques for such activities as heating chemicals, smelling them, or pouring from bottles,
- hand-washing facilities are readily available in the laboratory.

Under the COSHH and the Management of Health and Safety at Work regulations, employers are responsible for carrying out risk assessments before hazardous procedures are undertaken or hazardous chemicals used or made. Teachers are required to co-operate with their employers by complying with such risk assessments.

However, teachers should be aware that mistakes can be made and, in any case, different employers adopt different standards.

Therefore, before carrying out any practical activity, teachers should always check that what they are proposing is compatible with their employer's risk assessments and does not need modification for their particular circumstances. Any local rules issued by the employer must always be followed, whatever is recommended here.

Model (general) risk assessments have been taken from, or are compatible with:

CLEAPSS *Hazcards* (see annually updated CD-ROM)
CLEAPSS *Laboratory handbook* (see annually updated CD-ROM)
CLEAPSS *Recipe cards* (see annually updated CD-ROM)
ASE *Safeguards in the school laboratory* 10th edition 1996
ASE *Topics in Safety* 3rd edition, 2001
ASE *Safety reprints*, 2000 or later

Clearly, teachers must follow whatever procedures for risk assessment their employers have laid down. As far as we know, all the practical work and demonstrations in this course are covered by the model (general) risk assessments detailed in the above publications, and so, in most schools and colleges, you will not need to take further action.

If teachers or students decide to try some procedure with hazardous substances beyond what is in this course, and you cannot find it in these or other model (general) assessments, then the teachers' employer will have to make a special risk assessment. If the employer is a member, then CLEAPSS will act for them. Otherwise the ASE may be able to help.

Only the teacher can know when the school or college needs a special risk assessment. But thereafter, the responsibility for taking all the steps demanded by the regulations lies with the employer.

Investigations will involve independent action by the student. Our notes on investigations warn students to carry out a risk assessment; students should be responsible for safety in the first instance and credited in any assessment for making safe plans. Nevertheless, proposals must be seen by the teacher and who must make an appropriate check, particularly with respect to safety, on what will go on. The teacher will need to take particular care if students consult library books published before modern safety standards came into force or get ideas from the internet.

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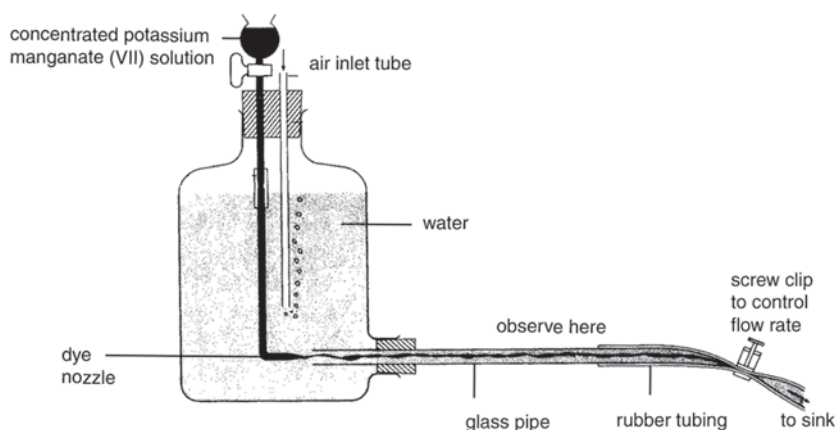
Experiments: *Chemical engineering*

Experiment 1 Investigation of flow patterns

(see Chapter 3)

In this experiment, water is discharged from a constant head device through a circular glass pipe of known diameter. You vary the water flow rate and observe the flow pattern within the glass pipe, by injecting a fine tracer stream of coloured liquid into the water. In this way, you can investigate the relationship between pipe diameter, flow rate, and flow pattern.

Procedure



CAUTION

Floors may get wet and slippery. All spillages *must* be mopped up immediately to avoid accidents.

In this and subsequent experiments involving aspirators, do not attempt to lift the previously filled aspirators. Put the empty vessel in place first, then fill it.

a Assemble the apparatus as shown in the diagram.

Check that the dye nozzle is correctly aligned with the end of the glass pipe. The bottom of the air inlet tube must be at least 5 cm above this pipe to give a reasonable head of pressure.

b Open the screw clip slightly to give a small flow rate of water through the glass pipe. Once a stream of bubbles begins to emerge from the bottom of the air inlet tube, the effective head of pressure of water within the apparatus will remain constant until the water level falls to this point.

c Slightly open the tap on the funnel containing the dye (potassium manganate(VII) solution), so that a fine stream of coloured liquid is injected into the water as it enters the glass pipe.

Observe the flow pattern within the pipe as you gradually increase the water flow rate. Attempt to identify the flow rate at which the flow pattern changes from streamline to turbulent.

The change-over from one pattern to the other is not sharp. Try to identify the point where the flow is definitely turbulent.

Measure the flow rate at this point using a measuring cylinder and stopwatch.

continued ...

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Experiments: *Chemical engineering*

Experiment 1 Investigation of flow patterns *continued*

(see Chapter 3)

d Use this mass flow rate, m (in kg per second), the internal diameter of the tube, d (in metres), and the viscosity of the water, η , (in kg per metre per second) to calculate the Reynolds Number at which the flow pattern finally changes from streamline to turbulent in your experiment.

The viscosity of water varies greatly with temperature.

Measure the temperature of the water you are using, and select the value of η from this table:

Temperature/ °C	Viscosity of water η / kg m ⁻¹ s ⁻¹
10	0.001307
11	0.001271
12	0.001235
13	0.001202
14	0.001169
15	0.001139
16	0.001109

Notes:

A reasonable value of the Reynolds Number, Re , for the change-over of flow patterns is 1200, but there are obvious uncertainties in identifying the change-over flow rate.

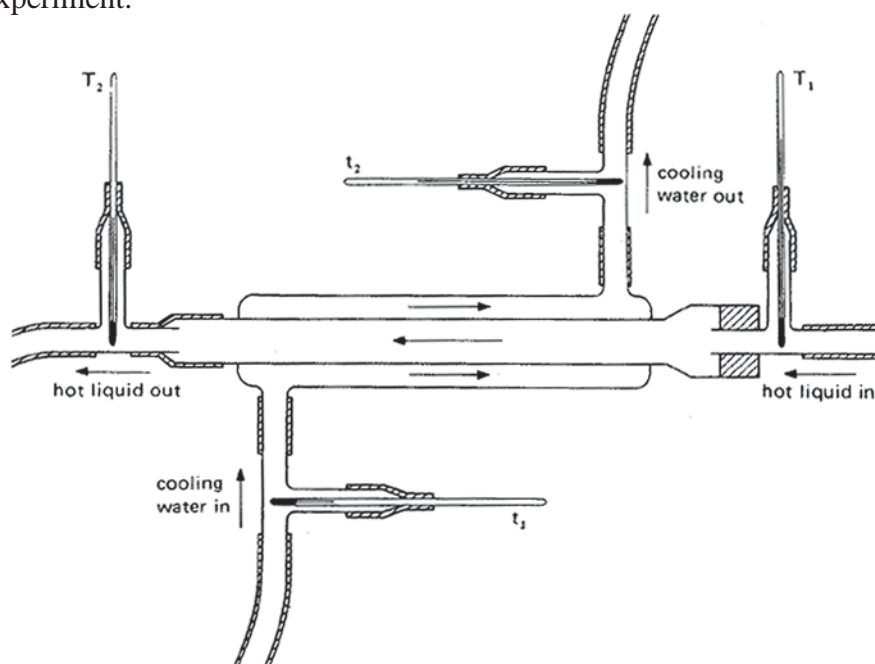
You should be able to explain which kind of flow is encouraged by increases in flow rate, viscosity and pipe diameter.

$$Re = \frac{4m}{d\pi\eta}$$

Experiment 2 Investigating heat transfer in a Leibig condenser

(see Chapter 4)

In this experiment, you will use a laboratory water-cooled Liebig condenser to reduce the temperature of a stream of hot liquid. You will determine the 'duty' of the heat exchanger. You measure the inlet and outlet temperatures, and the mass flow rate of the liquid stream. From your results, you will estimate the heat transfer coefficient across the heat exchanger surface under the conditions of the experiment.



Procedure

a Fit a laboratory condenser with thermometers at each inlet and outlet, so that you can measure the temperature of both the hot liquid stream and the cooling water before and after passing through the apparatus.

This is readily achieved by fitting plastic T-pieces into the rubber tubing, as shown in the diagram above. Take care to avoid leaks at joints.

b Pass the hot liquid which is to be cooled through the central 'tube' of the heat exchanger. Pass the cooling water through the outer 'shell' in a counter-current direction.

Control the flow rates of both liquids by means of screw clips attached to the outlet hoses. Ideally, both liquids should be supplied from constant head tanks, so that their flow rates remain steady throughout the experiment.

continued

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Experiments: *Chemical engineering*

Experiment 2 Investigating heat transfer in a Leibig condenser *continued*

(see Chapter 4)

C Adjust the flow rates of both liquid streams to give a temperature drop of at least 5 °C for the hot liquid.

When conditions are steady, record the hot liquid inlet temperature T_1 and outlet temperature T_2 ; also the cooling water inlet temperature t_1 and the outlet temperature t_2 .

Use an appropriate measuring cylinder and stopclock to measure the mass flow rate of hot liquid and cooling water through the heat exchanger.

After dismantling the apparatus, measure the length and average diameter of the 'tube' across which heat transfer takes place.

Treatment of results

The 'duty' of the heat exchanger (Q) is the amount of heat being transferred per hour (in kJ hr^{-1}). This is calculated from the results for the hot liquid as follows:

$$\begin{aligned} & \text{Heat transfer per hour } (Q)/\text{kJ hr}^{-1} \\ &= \text{mass flow per hour/kg hr}^{-1} \times \text{specific heat capacity of liquid/kJ kg}^{-1} \text{K}^{-1} \times \text{temperature change } (T_1 - T_2)/\text{K} \end{aligned}$$

The performance of a heat exchanger is described by the equation:

$$Q = UA \Delta t_m \quad \text{where } U \text{ is the heat transfer co-efficient}$$

The area of the heat transfer surface (A/m^2) is calculated from the tube length (l/m) and average diameter (d_{av}/m).

$$A = \pi d_{\text{av}} \times l$$

Δt_m , the log mean temperature difference across the heat exchanger, is calculated from t_1 , t_2 , T_1 , and T_2 .

$$\Delta t_m = \frac{(T_1 - t_2) - (T_2 - t_1)}{\ln [(T_1 - t_2) / (T_2 - t_1)]}$$

Experiment 3 Finding the concentration of a solution of hydrogen peroxide

(see Chapter 5)

The idea of this experiment is to design conditions for using hydrogen peroxide to convert potassium iodide into iodine, in a continuous flow tubular reactor (CSTR) using the reaction:



You will monitor the extent of the conversion by titration with sodium thiosulphate solution.

You will need a solution of hydrogen peroxide of known concentration. It is notoriously difficult to prepare such a solution by accurate dilution, because hydrogen peroxide decomposes spontaneously during storage.

You are provided with an approximately '1 volume' solution of hydrogen peroxide – so-called because if 1 cm³ of it were decomposed fully into water and oxygen, 1 cm³ of oxygen would be produced.

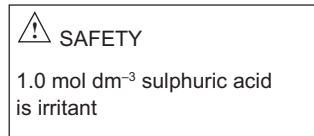
The object of this part of the experiment is to find the concentration of the '1 volume' hydrogen peroxide as accurately as possible.

Procedure

- a** Add about 1 g of potassium iodide (an excess) to about 20 cm³ of water, and about 10 cm³ of 1.0 mol dm⁻³ sulphuric acid in a conical flask. Swirl the flask to dissolve the solid.
- b** Using a pipette and filler, add 10.0 cm³ of '1 volume' hydrogen peroxide.
- c** Warm the reaction mixture to about 50 °C, and allow it to stand for about 30 minutes for the reaction to be complete.
- d** Titrate the liberated iodine with 0.20 mol dm⁻³ sodium thiosulphate, using starch as indicator in the conventional way.
- e** From your titration result, calculate the concentration of the hydrogen peroxide solution in mol dm⁻³.

During the rest of the available class time, while this part of the experiment is going on, you should discuss the conditions to be used in the CSTR (see the *Chemical engineering* text page 23).

You will be using the design equation to work out flow rates for achieving a desired 'conversion' of hydrogen peroxide into iodine.



continued

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Experiments: *Chemical engineering*

Experiment 3 Finding the concentration of a solution of hydrogen peroxide *continued*

(see Chapter 5)

For this purpose it will be necessary to know the rate constant for the reaction.

The rate constant for the reaction varies with temperature, so choose an appropriate value from the following table.

Temperature/°C	Rate constant k /min ⁻¹
14	0.016
15	0.017
16	0.019
17	0.020
18	0.022

If you need to go outside of this range, the formula for calculating k , based on an activation energy of 56.1 kJ mol⁻¹ is:

$$\ln k = 19.69 - \frac{6841}{T}$$

where T is the temperature in Kelvin

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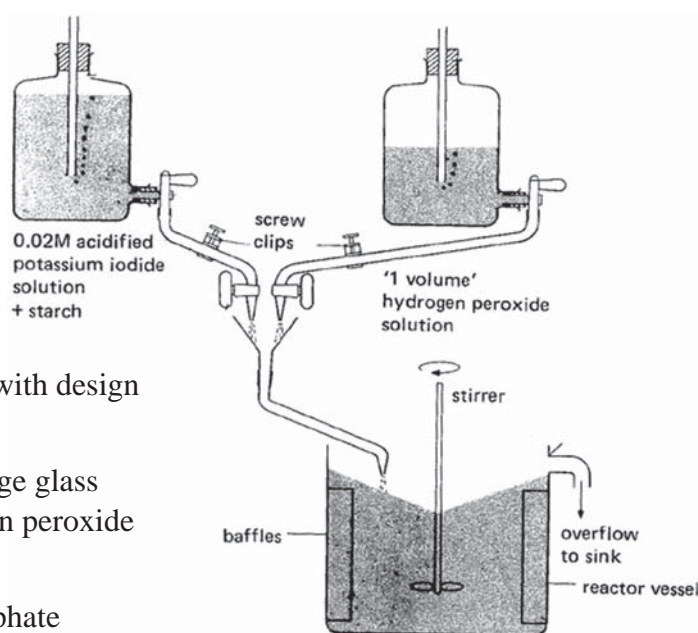
Experiments: *Chemical engineering*

Experiment 4 The continuous-flow stirred tank reactor

(see Chapter 5)

In this experiment, you will design a continuous stirred tank reactor (CSTR) to produce a certain percentage conversion of reactants to products.

You will then construct the reactor to your own specifications, and compare its operating performance with your design calculations. The reagents will be '1 volume' hydrogen peroxide solution and acidified potassium iodide solution (0.02 mol dm^{-3}) as used in Experiment 3.



a Set up an apparatus like the one in the diagram (with design variations or improvements as you see fit).

The potassium iodide solution needs to go in the large glass flask which you used in Experiment 1. The hydrogen peroxide will go in a smaller container.

Also arrange for a burette to deliver sodium thiosulphate solution into the reactor tank for analysis purposes at the end of the experiment.

b Determine the working capacity of your reactor by filling it with water and switching on the stirrer. Water will overflow until a steady state is reached. Switch off and measure the volume of water left in the vessel. This is the working volume of the reactor ($V \text{ dm}^3$).

c For the sake of comparison, aim to work at similar concentrations to those used in Experiment 3. The inlet stream should be '1 volume' hydrogen peroxide mixed with 0.02 mol dm^{-3} acidified potassium iodide solution, containing some starch in a volume ratio of 1:10.

Allowing for dilution, this would make the initial hydrogen peroxide concentration $1/11$ th of that of your '1 volume solution' – you know this concentration accurately from Experiment 3.

d Use these conditions in the design equation for a CSTR. Planning for a conversion rate of, say, 15%, 20% or 25%, calculate the flow rate for the potassium iodide and hydrogen peroxide solutions, remembering that they are in a 10:1 ratio.

e Put water in the apparatus and set up the taps, screw clips etc so as to get as near as possible to your calculated flow rates.

continued

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Experiments: *Chemical engineering*

Experiment 4 The continuous-flow stirred tank reactor *continued*

(see Chapter 5)

f Fill up the apparatus with the proper reagents and start the flows going. You will find that counting the bubbles as they leave the bottom of the constant-head tube is a good way of checking to see that the flow rates are not changing.

g Allow the reactor vessel to fill up and reach a steady state. This will take about four times the mean residence time after the reactor tank is full.

h While the system is coming to a steady state, sodium thiosulphate should be added a few drops at a time to remove the blue colour each time it appears, so as to keep the potassium iodide concentration constant.

i Once the reactor has reached a steady state, add 0.1 mol dm^{-3} sodium thiosulphate from the burette, at such a rate that the colour of the reactor contents appears to 'hover' between blue and colourless. Measure the flow rate. From this the achieved percentage conversion can be calculated.

Treatment of results

a List the flow rates of sodium thiosulphate, potassium iodide, and hydrogen peroxide.

b Applying a mass balance:

$$\text{reactant in (B)} - \text{unchanged reactant out (C)} - \text{reactant reacting (A)} = 0$$

$$\text{Flow rate of thiosulphate} = \dots\dots\dots \text{ cm}^3 \text{ min}^{-1}$$

$$\begin{aligned} \text{rate of I}_2 \text{ formation} &= \dots\dots\dots \text{ mol min}^{-1} \\ &= \text{rate of H}_2\text{O}_2 \text{ consumption (A)} \end{aligned}$$

$$\text{Rate of H}_2\text{O}_2 \text{ supply (B)} = \text{conc. of H}_2\text{O}_2 \times \text{flow rate of H}_2\text{O}_2$$

$$\begin{aligned} \text{Rate of H}_2\text{O}_2 \text{ leaving unreacted (C)} \\ &= \text{final conc. of H}_2\text{O}_2 \times \text{total flow rate (in dm}^3 \text{ min}^{-1}) \end{aligned}$$

From this the final concentration of hydrogen peroxide may be calculated.

Calculate the % conversion from:

$$\% \text{ conversion} = \frac{\text{change of concentration}}{\text{original concentration}} \times 100$$

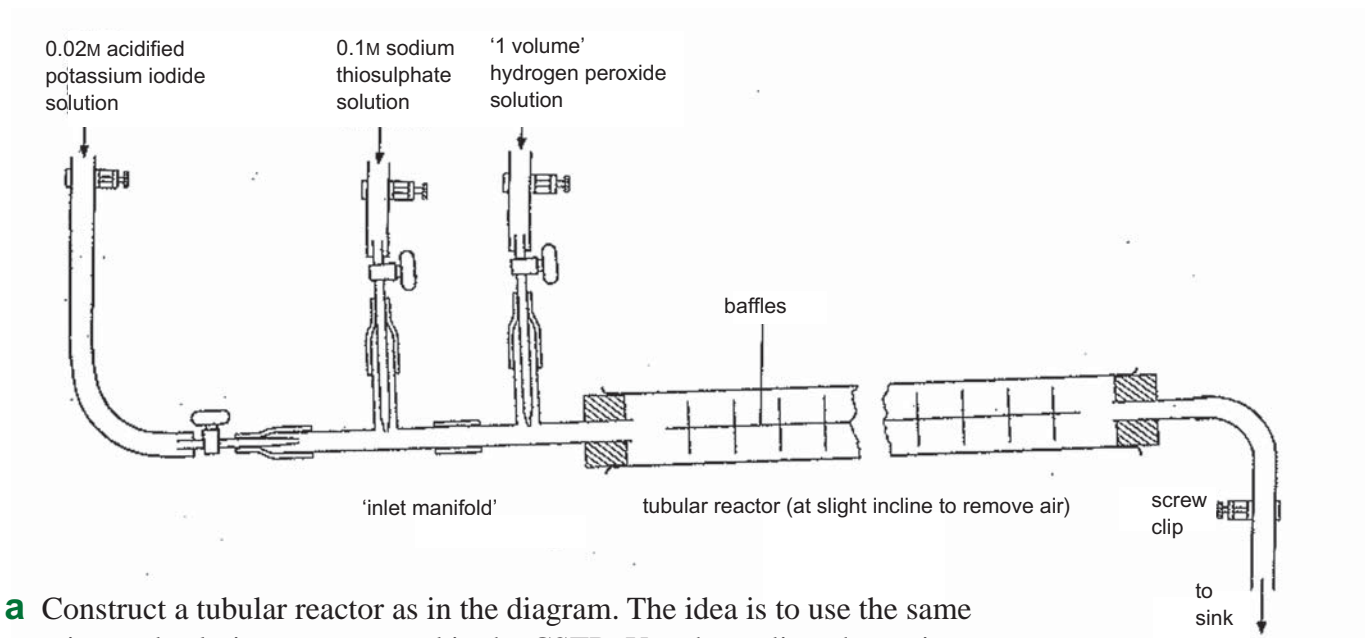
Compare this value with your intended % conversion.

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Experiments: *Chemical engineering*

Experiment 5 The continuous-flow tubular reactor

(see Chapter 5)



a Construct a tubular reactor as in the diagram. The idea is to use the same reaction and solutions as you used in the CSTR. You then adjust the various flow rates until the contents of the tube turn blue about half way up the tube.

b Record all the flow rates when this is achieved.

Measure the distance up the tube where the change-over occurs, and also measure the internal radius of the tube.

Treatment of results

The design equation for a tubular reactor is essentially the same as that for a batch reactor, since each disc of solution passing up the tube operates like a little self-contained batch of reaction mixture.

This design equation is

$$t = \frac{1}{k} \times \frac{\ln[\text{H}_2\text{O}_2]_{\text{initial}}}{[\text{H}_2\text{O}_2]_{\text{initial}} - [\text{H}_2\text{O}_2]_{\text{consumed}}}$$

At the point where the change-over occurs:

$$\begin{aligned} [\text{H}_2\text{O}_2]_{\text{consumed}} &= \frac{\Omega \times 0.1 \times \text{flow rate of thiosulphate}}{\text{total flow rate}} \\ &= \dots \text{ mol dm}^{-3} \end{aligned}$$

$$\begin{aligned} [\text{H}_2\text{O}_2]_{\text{initial}} &= [\text{H}_2\text{O}_2]_{\text{'1 volume'}} \times \frac{\text{flow rate H}_2\text{O}_2}{\text{total flow rate}} \\ &= \dots \text{ mol dm}^{-3} \end{aligned}$$

continued

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Experiments: *Chemical engineering*

Experiment 5 The continuous-flow tubular reactor *continued* (see Chapter 5)

Substituting in the design equation (and using an appropriate value for k), you can calculate the value of t .

The volume of liquid going up the tube in time t minutes is

$$= \text{total flow rate} \times t$$

Hence the distance d up the tube at which the changeover occurs will be

$$= \frac{\text{volume of liquid}}{\pi \times \text{radius}^2}$$

Calculate this distance d and compare it with the value which you measured.

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Experiments: *Chemical engineering*

Experiment 6a A phase diagram for the propanone–ethanol system (Study Task)

(see Chapter 6)

Equilibrium data for the propanone–ethanol system are given in the table on the right.

a Use this data to plot the temperature/composition diagram, drawing both liquid and vapour lines as accurately as you can, with a flexible curve if available.

b Use your graph to determine the number of theoretical plates (*i.e.* distillation stages) necessary to obtain a distillate containing 95 mole % propanone, starting from a 50 mole % mixture.

c Carefully draw the appropriate steps on your graph, using a sharp pencil and ruler. You will use this diagram later to interpret your experimental results.

Mole % propanone in liquid	Mole % propanone in vapour	Temperature /°C
0.0	0.0	78.3
10.0	26.2	73.0
20.0	41.7	69.0
30.0	52.4	65.9
40.0	60.5	63.6
50.0	67.4	61.8
60.0	73.9	60.4
70.0	80.2	59.1
80.0	86.5	58.0
90.0	92.9	57.0
100	100.0	56.1

Liquid/vapour equilibrium data for mixtures of propanone and ethanol at standard atmospheric pressure.

Experiment 6b Verifying some points on the phase diagram for the propanone–ethanol system

a Use the apparatus shown opposite to verify some of the points on the phase diagram. Make up mixtures of ethanol and propanone with

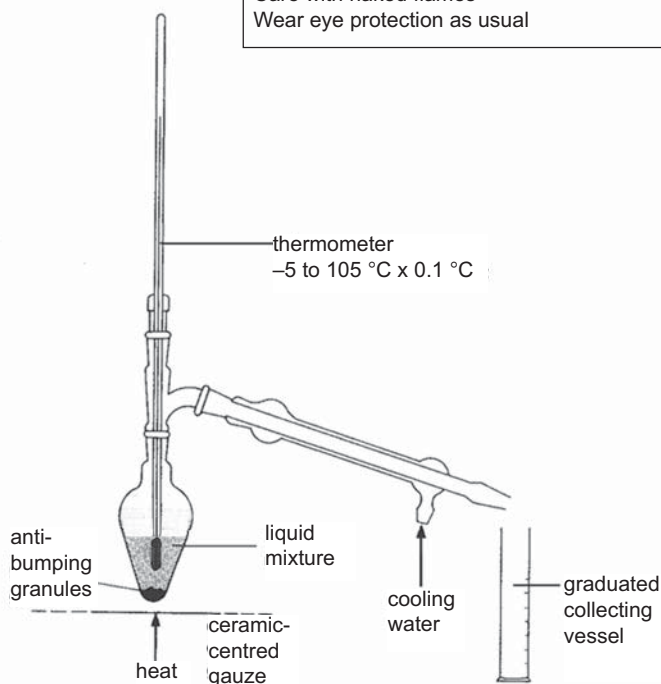
(i) 20% propanone

(ii) 60% propanone

b Distil a few drops of each mixture into a small test tube, record the temperature at which each boils, and then find the boiling point of each distillate. Do this by heating the distillate to boiling point in a beaker of hot water (fire precautions!)

c Relate your results to the phase diagram.

SAFETY
Propanone is irritant and highly flammable
Ethanol is highly flammable
Care with naked flames
Wear eye protection as usual



7 Reflux ratio

Procedure

a Set up the apparatus as in the diagram, but without the insulation at first.

b Make a mixture which is 20 mol per cent of propanone and 80 per cent ethanol; put the mixture into the distilling flask.

c Set the variable reflux ratio head to total reflux, and turn on the electrical heater. When the mixture boils, observe the counter-flow in the column with vapour going up and liquid running down.

d Insulate the column, leaving a 'window' to allow the reflux drip rate to be counted.

e Turn down the electrical heater so that 'logging' of the column does not occur.

f When the conditions are steady, record the temperature at the still-head and convert this into a percentage of propanone using your phase diagram. The reflux ratio is infinite because no liquid is being distilled over.

g Set the reflux ratio to some other value, and again record the temperature and % of propanone at the still-head. Do this for several reflux ratios if possible.

Results

You should see that, with increasing reflux ratio, the distillate quality improves but, of course, the rate of distillation slows. A balance between high quality and an economic distillation rate is one of the problems of large-scale distillation.

A further problem is that the composition of the liquid in the flask changes during the distillation. It would be much better if the distillation could be run continuously; see Chapter 6.

(see Chapter 6)

