INNOVATIVE TECHNIQUES IN SCHOOL LEVEL MICRO-SCALE CHEMISTRY EXPERIMENTS PART (I)

COLLECTION OF DISTILLATE AT THE SOURCE (CDS) TECHNIQUE [1]

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ABSTRACT

Traditional distillation setup collects distillate at a certain distance from the boiling source. This results in loss of product and the situation could be very serious if micro-scale method is employed. By using an innovative design, it is possible to collect nascent distillate immediately after vapor condensation at the boiling source. This is the rationale of designing the Collection of Distillate at the Source ("CDS") technique. The design not only significantly improves product yield, because cooling water used for condensation can be dispensed with or recycled. This offers a helpful means to enhance students' "Green awareness". Two designs using the "CDS" technique are introduced, namely (i) Micro-scale water-less reflux and distillation and (ii) Micro-scale recycled coolant and all-glass reflux and distillation. The first design can be totally homemade with local resources and by acquiring decent workshop skills, details of construction of its various components are provided. *[African Journal of Chemical Education—AJCE 11(2), July 2021]*

INTRODUCTION

Innovation of techniques in practical chemistry at school level has not been an important issue, compared with the counterparts at university or research level. Recent worldwide promotion of micro-scale techniques had prompted numerous workshops and publications on micro-scale chemistry experiments. These instrument-based innovations depend heavily on school curricula and commercial kits. They may not meet the requirements of individual public examination boards. Technique, instead of instrument, seems to be a more universal area of effective innovations. In fact, newly designed micro-scale instruments stemming from these techniques are simple, based on elementary principles and capable of enhancing student motivation in performing experiments. They also serve as tools for better understanding of chemistry principles.

"CDS" DESIGN (A) – Micro-scale water-less reflux and distillation

Performing group-based small scale organic distillation experiments in school laboratories often encounter the problem of improper sets and poor experimental yield. All-glass "Quick-fit" distillation apparatus [2] does not help much, because if micro-scale amount of reactant is used and after passing the condensation passage, the resulting yield of distillate is usually zero.

The problem can be solved by using the following method: (i) use traditional test tube for reaction, (ii) do not use water for condensation purpose, use a special "coolant" (Fig. 3) purchasable from super-markets to act as strong cooling agent instead, (iii) make a device to act as "cold finger", (iv) make a special small distillate cup to collect "nascent" distillate, (v) use a pin-shaped digital thermometer to register temperature and (vi) make a mini homemade low voltage heater to cater for naked flame-free heating. The paper includes two experiments specifically designed for ***water-less*** micro-scale reflux and distillation as an alternative to

traditional distillation by water condensation. Experimental results showed the set could perform with higher efficiency and reliability.

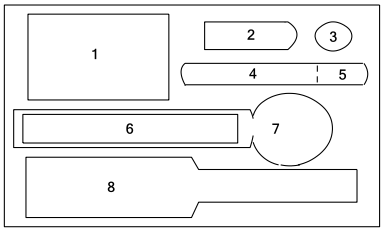
Micro-scale approaches at university level for short-path distillation commonly used the "Hickman Still Head" [3], [4], [5] (Fig. 1). This piece of apparatus and the accompanying set is obvious not on school chemistry laboratory equipment stock list.

(Fig. 1) A Hickman Still Head

(Fig. 2) Combostill setup

Recently, a micro-scale reflux and distillation instrument known as "Combostill" (Fig. 2) was available commercially [6]. The working principle is based on flame heating an outer oil containing glass vial which in turn heats up an inner glass vial containing the reactants. Air condensation by a delivery tube collects the distillate. The involved technique is in fact quite traditional. Naked-flame heating is at a disadvantage. The inefficiency of vapor condensation and collection of distillate makes quantitative investigations barely feasible.

"Micro-scale Water-less Distillation Set"



Code	Description
1	Mini-heater
2	"Distillate Cup"
3	"Cold Ring"
4	Small test tube
5	Silicone teat
6	Pasteur pipette
7	Digital thermometer "Summit 310"
8	"Cold Stick"

(A) Construction of the "Cold Stick"

Running water used as condensation coolant in traditional distillation is dumped into the sink and wasted. Such practice is really not environmentally good. A simple solution is not using water as coolant. A special reusable coolant pack (Fig. 3) used to keep food chilled during picnic time is readily available from supermarkets. Simply keep it cool in a freezer overnight and you will get a powerful cooling device like a big piece of ice. The chemical used inside the plastic pack is a strong organic cooling agent which is very safe and unharmful. It is an ideal coolant for condensation in place of water.

The "Cold Stick" (Fig. 4) consists of a brass rod and a transparent plastic tube containing the strong coolant as mentioned earlier. It works like a "cold finger". The position of copper in the electrochemical series is below hydrogen. Metallic copper or brass has substantial strength of resistance to simple organic reaction corrosion. As such, we found "Cold Stick" is safe for handling simple organic substances like alkanols, esters, haloalkanes or alkanoic acids. Unlike university level organic chemistry practicals, only limited simple organic reactions requiring distillation like preparations

of esters and haloalkanes are included in senior secondary school chemistry experiments and that makes the design suitable for use at school level.

(Fig. 3) Coolant pack

(Fig. 4) Completed "Cold Stick"

First prepare a 100 mm (high) x 35 mm (diameter) transparent acrylic tube. Two amber acrylic 5 mm thick disc with the same diameter dimension as the transparent tube are needed. Each amber disc has one central pre-drilled 8 mm diameter hole, one disc has one small hole for liquid coolant injection. Carefully and repeatedly cement the amber discs onto the two ends of the transparent tube with drops of trichloromethane from a disposable syringe (Fig. 5 and 6). Insert the brass rod into the holes of the two amber discs and cement both ends with epoxy glue. Using a 50 cm^3 plastic syringe and by inserting into the small hole of one of the amber discs, fill the cavity of the tube 8/10 full with the liquid coolant. Finally, seal the small hole with epoxy glue. A finished "Cold stick" is shown in Fig. 7.

(Fig. 5)

(Fig. 6)

(Fig. 7)

(B) Construction of the "Distillate Cup"

Traditional condensation (Fig. 8) uses a water condenser to cool the vapor to form distillate. The vapor has to travel a certain distance before the distillate drops to the collector and the situation becomes crucial for micro-scale distillation. As the initial mass of reactants are small, the final yield could be very limited or even no distillate would be collected. Targeting this problem, a straight forward solution is to collect the "nascent" distillate where it is formed. A special homemade "Distillate Cup" (Fig. 20) when capped to the end of the brass rod (Fig. 9 and 10) can receive all the initial distillate right after the reaction starts. The nascent distillate collected by the "Distillate Cup" is 100% pure reaction product.

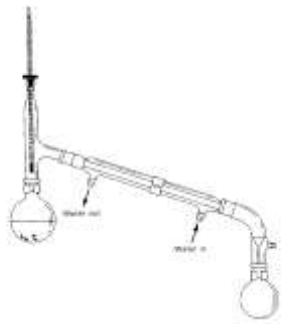
Prepare the following materials:

1. A section of 10 cm long 8 mm diameter brass rod (L)
2. A section of 8 mm long 8 mm internal diameter brass tube (L1)
3. A section of 11 mm long 10 mm internal diameter brass tube (L2)
4. A brass or copper disc 8 mm diameter 0.2 mm thick with a central hole of diameter 0.8 mm
5. A copper sheet measuring 29 mm long, 15 mm wide and 0.1 mm thick.
6. A small 0.2 mm thick copper sheet
7. A section of 25 mm long thick connecting wire
8. A small working platform

Wrap the 0.1 mm thick copper sheet round the brass rod (L) one turn, forming a cylindrical wrap (Fig. 11). Insert L2 (just fit, Fig. 12). Place the brass disc over one end of the cylindrical wrap (Fig. 13). Clamp the combination and place it on a small

working platform (Fig. 14). Solder two suitable points (one is the junction between the disc and the copper wrap and the other is just the opposite, Fig. 15). Remove the brass rod from the wrap/disc combination. Finally close the copper wrap with another solder joint. The completed cap (Fig. 16) is just good for capping the brass rod. Solder the thick connecting wire onto the wrap/disc combination to form another part as shown in Fig. 17.

Place L1 over the 0.2 mm thick small copper sheet. Apply solder and trim spare parts (Fig. 18) to form a small cup as shown in Fig. 19. Make a small hole at the centre of the cup, allowing the connecting wire to go through.



(Fig. 8)

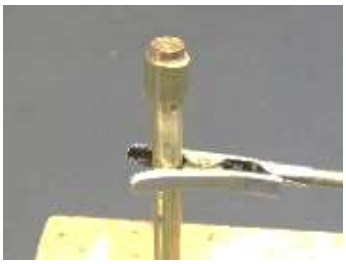
(Fig. 9)

(Fig. 10)

(Fig. 11)

(Fig. 12)

(Fig. 13)



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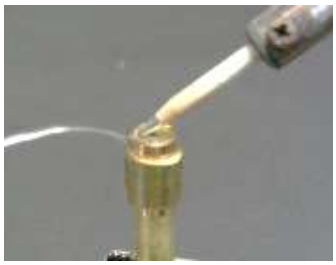
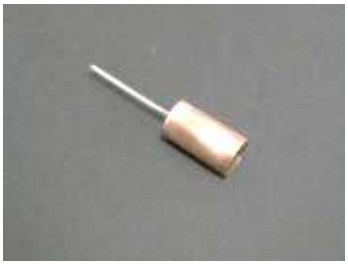
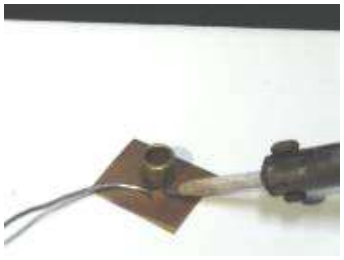


(Fig. 15)

(Fig. 16)



(Fig. 17)



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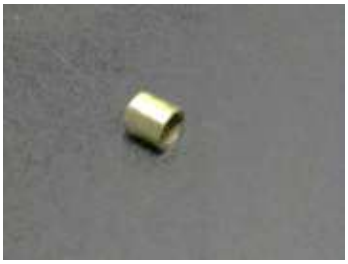
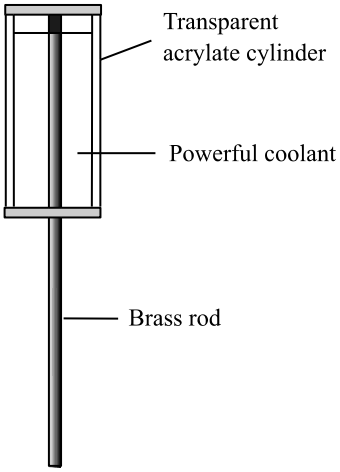


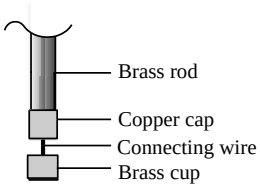
(Fig. 19)

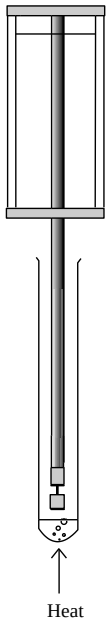
Finally solder and trim the cap and the cup together to form a “Distillate Cup” (Fig. 20).

(Fig. 20) Finished “Distillate Cup”

Schematic diagrams of the Micro-Scale Water-less Distillation Set:







Cold Sick: a transparent plastic cylinder 8/10 filled with a strong liquid coolant. A central brass rod acts as a "cold finger". Can be reused by placing in a freezer.

Distillate Cup: Formed by a small cap connected to a small cup. The Cap end is to be capped onto the brass rod while the cup receives distillate.

Water-less micro-scale distillation setup

(C) Construction of the "Cold Ring"

The "Cold Ring" (Fig. 21) is used for cooling while taking the boiling point of the distillate. It provides a cooling surface for refluxing the distillate. Usually, time taken for measurement is short and this device is optional. Moist with cold water a small piece of 15 mm thick circular sponge with a central hole which fits a small test tube. It is placed near the mouth of the small test tube.

(Fig. 21) "cold Ring" for cooling

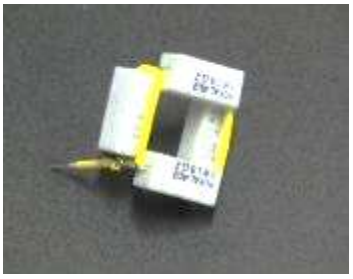
(D) Construction of the "Mini Low Voltage Heater"

(Fig. 22) Mini low voltage heater

Organic liquids are flammable and should not be heated by naked Bunsen flame. Using an electric heater is one way and using a hot sand bath is another. Commonly available cement resistors are suitable to act as small electrical heaters (Fig. 23). Four 5W 18Ω cement resistors connected in parallel to form a shape of 井 (Fig. 24) can function as a 20W heater, the central hollow part accommodates a test tube nicely.

All joints have to be soldered permanently. Sections of Teflon tubing (yellow tubing of Fig. 23) or ceramic tubing are used for heat insulation of exposed linings, as all parts will become very hot upon passage of current for some time. Normal solder has melting points above 200^{o} C and the cement resistor combination can attain a maximum temperature around 150^{o} C, after considering environmental cooling factors. The heating assembly can stay intact for an hour's operation.

(Fig. 23)

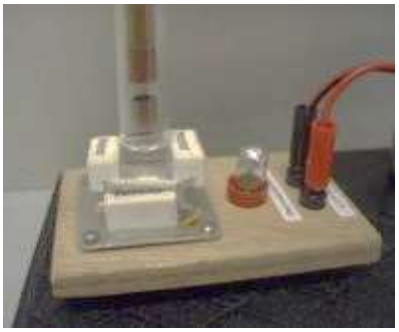


(Fig. 24)

(Fig. 25)

Cement the 井 like heater combination onto an aluminum sheet by strong metallic epoxy glue for heat dissipation (Fig. 25). Fix the entire combination onto a wooden plate. Assemble a panel lamp for illumination (Fig. 26). Start heating by applying a low voltage (12V) AC or DC current (3A). The device could supply heating power for test tube distillation for at least half an hour (Fig. 27).

(Fig. 26) Finished mini-heater



(Fig. 27) Setup for micro-scale distillation

(E) Pin-shaped digital thermometer

(Fig. 28) "Summit 310" pin-shaped thermometer

(Fig. 29) DMM with miniature thermocouple for temperature measurement

The Set uses a pin-shaped digital thermometer ("Summit 310", Fig. 28) for measuring boiling points of distillate. The thermometer has a resolution of ± 0.1°C and can be placed into a

small test tube. Alternatively, the miniature thermocouple as an accessory of a DMM can also be used. However, the latter can display only integer numbers (Fig. 29).

(i) Reflux, (ii) Distillation and (iii) Checking boiling points of distillate:

(Fig. 30) Reflux

(Fig. 31) Distillation

(Fig. 32) Boiling point determination

Experiment (1): Preparation of 2-chloro-2-methylpropane

Objective

To perform a micro-scale preparation of 2-chloro-2-methylpropane (t-butyl chloride) and determine the percentage yield

Experimental procedures

1. Place 0.280 g of 2-methylpropan-2-ol (usually in liquid form, m.pt. = 26oC) into a test tube.
2. Using a clean Pasteur pipette, add about 1.5 cm3 of conc. hydrochloric acid. Add also a small spatula measure of fine anti-bumping granules.

3. Clamp the test tube and lower it into the mini-heater. Switch on the low voltage power supply. Clamp the prepared "Cold Stick" and lower it into the test tube until the end of the brass rod is about 1 cm above the surface of the solution mixture. Reflux for 5 minutes (Fig. 30).
4. Remove the "Cold Stick" and place the distillate cap with the cup onto the tip of the brass rod. Lower the arranged setup into the test tube again and start distillation (Fig. 31).
5. Wait until the small brass cup has collected enough distillate. Remove the distillation setup from the test tube. Transfer the distillate to a weighted small test tube with the help of a clean Pasteur pipette.
6. Repeat procedure (5) a number of times until all of the upper layer has been removed and collected.
7. Determine the mass of the distillate.
8. Using the pin-type digital thermometer and with the help of the "Cold Ring", determine the b.pt. of the distillate (Fig. 32).

Results

$$\underset{74}{(CH_3)_3COH} + HCl \longrightarrow \underset{92.6}{(CH_3)_3CCl} + H_2O$$

Mass of distillate (2-chloro-2-methylpropane) = *0.143 g*

No. of mole of 2-methylpropan-2-ol used = *0.28/74 = 0.00378*

No. of mole of 2-chloro-2-methylpropane formed = *0.143/92.6 = 0.00154*

% yield of 2-chloro-2-methylpropane = *(0.00154/0.00378) x 100 = 40.7%*

B.pt. of 2-chloro-2-methylpropane = *51.0^{o}C (Literature value: 50.8^{o}C)*

P.S. In the absence of advanced instruments for identifying organic compounds like GC-MS, NMR or IR spectrometers [7], checking of boiling point seems to be the only holistic identification tool at school level instead of discrete chemical means of functional group identifications.

Conclusion

Percentage yield of 2-chloro-2-methylpropane formed by the reaction of conc. HCl with 2-methylpropan-2-ol was determined to be 40.7%. The product was reasonably pure.

Remarks

Video clip *Micro-scale preparation of tert. Butyl chloride* [8] shows 2-chloro-2-methylpropane is insoluble and is less dense than aqueous solutions. Therefore, it floated atop of the reaction product mixture. One quick way of getting this layer is withdrawal by using a syringe instead of performing distillation.

The Set looks attractive to students. It serves as an attention focus for experimentation. The panel lamp of the mini-heater is a magic touch of the design; it adds weight for participant concentration. All in all, students are curious in operating a device as such.

Experiment (2): Preparation of ethyl methanoate

Choice of ester for preparation

Esters are formed by the reaction between alkanol and alkanoic acid with conc. sulphuric acid as catalyst. Esters of low molecular mass and their respective boiling points are listed in the following table (Fig. 33):

Preparation of esters of low RMM	
Ethyl ethanoate	Equation: $C_2H_5OH + CH_3COOH \rightleftharpoons CH_3COOC_2H_5 + H_2O$ B. Pt.: 78 °C 118 °C 77 °C 100 °C
Ethyl methanoate	Equation: $C_2H_5OH + HCOOH \rightleftharpoons HCOOC_2H_5 + H_2O$ B. Pt.: 78 °C 101 °C 54 °C 100 °C
Methyl methanoate	Equation: $CH_3OH + HCOOH \rightleftharpoons HCOOCH_3 + H_2O$ B. Pt.: 65 °C 101°C 32 °C 100 °C
Methyl ethanoate	Equation: $CH_3OH + CH_3COOH \rightleftharpoons CH_3COOCH_3 + H_2O$ B. Pt.: 65 °C 118 °C 57 °C 100 °C

(Fig. 33) Esters of low RMM, their preparations and boiling points

In the past, school chemistry practicals usually chose the formation of ethyl ethanoate as student experiment for the topic on ester. However, inspection of the above table revealed that the boiling points of the product (ethyl ethanoate, 77°C) and the reactant (ethanol 78°C) are so close that the two chemicals will appear in the distillate in appreciable proportion, leading to a very low yield of the desired ester.

A better idea is to choose an ester with a b.pt. quite different from that of the reactants and the product. Considering the b.pt. of ethyl methanoate being only 54°C, significantly different from that of ethanol (78°C), methanoic acid (101°C) and water (100°C), preparation of ethyl methanoate is obviously a good choice. In addition, methyl methanoate with a b.pt. of 32°C is too volatile to be considered.

Objective

To perform a micro-scale preparation of ethyl methanoate and determine its percentage purity

Experimental procedures

1. Using separate clean Pasteur pipettes, transfer 40 drops of 95% ethanol and 60 drops of 100% methanoic acid into a test tube followed by 3 drops of conc. sulphuric acid. Add a small spatula measure of fine anti-bumping granules.
2. Clamp the setup and lower it into the mini-heater. Switch on the low voltage power supply. Clamp the prepared "Cold Stick" and lower it into the test tube until the end of the brass rod is about 1 cm above the surface of the solution mixture. Reflux for 30 minutes.
3. Remove the "Cold Stick" and place the distillation cap with the cup onto the tip of the brass rod. Lower the arranged setup into the test tube again and start distillation.
4. Wait until the small brass cup has collected enough distillate. Remove the distillation setup from the test tube. Transfer the distillate to a small test tube with the help of a clean Pasteur pipette.
5. Using the pin-type digital thermometer and with the help of the "Cold Ring", determine the b.pt. of the distillate.

Results

Boiling point of distillate = *54.0°C*

(Literature value: b.pt. of ethyl methanoate = 54.0°C)

Conclusion

Micro-scale preparation of ethyl methanoate was performed and the product was determined to be pure ethyl methanoate.

Remarks

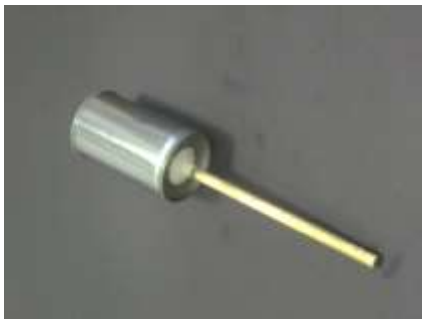
The micro-scale distillation set is very suitable for small scale simple organic reactions. The idea of water-less condensation not only saves tap water resources, it can also enhance students' recognition of environmental protection and hence fortify their "Green Awareness". The design to a large extent shortens the time required for experiment. Naked flame-free heating advantage gives extra emphasis to safety measures. The reason for using copper and brass as components is because of their easy availability and forming. As the reactivity of copper and brass are low, they are rather resistant to corrosion. Long exposure to low molecular mass organic chemicals makes them passive and more resistant to corrosion.

The set works effectively for organic chemicals with boiling point less than 100°C. Increasing current will enhance heating and can cater for temperatures more than 100°C. Placing the "Cold Stick" in a refrigerator can perform reflux for an hour while storing it in a freezer cabinet provides for a 3-hour reflux.

Ester formations are reversible reactions. Ethyl methanoate has a solubility of 9% [9]. Direct distillation after reflux is not a suitable method of getting the ester. The percentage yield of this experiment is purposely not included and the aim of the experiment is to identify and test purity of the product.

Postscript

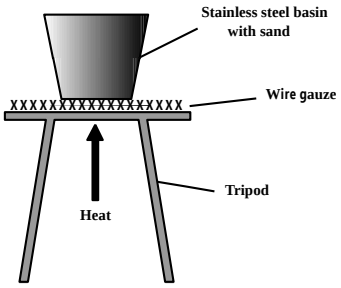
(1) The idea of using the "Cold Stick" is creative, but the design is a bit fancy. If aiming at getting the result and not caring much about appearance, the design can be simplified to just using an opened tin with a hole at the bottom and attaching to a brass rod. A brass rod is a very good conductor of heat and is not suitable for large area soldering. (A+B) type metallic epoxy cement binds the rod and the tin hole nicely. The cooling agent is a handful of small ice cubes or simply cold water (Fig. 34, 35).



(Fig.34) Drill a central hole at the bottom of the tin, allowing a brass rod to go through. Fix by (A+B) type metallic epoxy cement.

(Fig. 35) Add small ice cubes

(2) The Mini-heater is also a fancy design. It could be completely done away with by using a traditional sand bath (Fig. 36, 37) which does not use electricity or require a naked flame.



(Fig. 36) A stainless steel basin containing sand pre-heated to around 300°C

(Fig. 37) Setup for reflux/distillation

"CDS" DESIGN (B) – Micro-scale recycled coolant and all-glass reflux and distillation ("Green Distelector")

The instrument

Traditional distillation setups use a water condenser to condense vapor and collect distillate. However, if the amount of reactants is small and the vaporized product has to diffuse sometime before being condensed, we usually do not obtain any product yield. Unfortunately, schools do not afford university level micro-distillation equipment. In fact, condensation of vapor does not limit one only to traditional methods. The innovative micro-scale instrument used in the experiment -"Green Distelector"(distelector meaning distillate collector) (Fig. 38) employs a new way of condensing vapor and collecting distillate right at the spot. Apart from the micro-scale

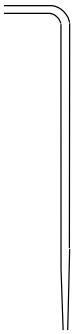
feature of the instrument, recycling of cooling water also contributes to enhance students'"Green Awareness".

(Fig. 38) "Green Distelector" setup

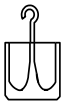
"Green Distelector" is provided in a plastic box containing:

	Description	Qty
1.	L glass tubing with silicone rubber fitting	1
2.	T tube with one opening and a ring at the bottom plus silicone rubber fitting	1
3.	Test-tube	1
4.	Silicone rubber circular-wrap fitting for test tube	1
5.	Silicone rubber tubing	2
6.	Aquarium pump with accessories	1 set
7	Stainless steel basin 4/5 filled with sand	1
8.	Small cylindrical glass bowl with a hook	1

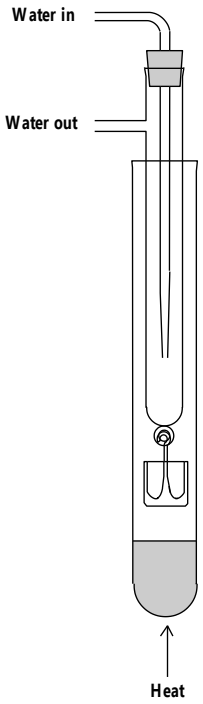
Schematic diagrams of the Micro-scale recycled coolant and all-glass reflux and distillation Set:

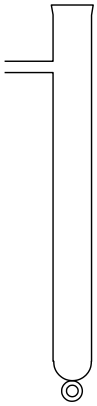


(Fig. 39) Inverted L tube

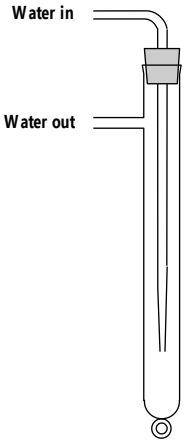


(Fig. 40) glass bowl with a hook





(Fig. 41) T tube with a ring at the bottom



(Fig. 42) A "cold finger"

(Fig. 43) Complete setup for distillation

(Fig. 44) Inverted L tube and T tube combination. Water comes from the top opening and leaves from

(Fig. 45) The base of the T tube has a glass ring which attaches to a small

(Fig. 46) Test tube, inverted L tube and T tube combination with

(Fig. 47) Sand bath and "Green Distelector" setup

the next opening. It serves as a "cold finger"	cylindrical glass bowl with a hook	small cylindrical glass bowl

As shown in Fig. 38, water for cooling does not come from the water tap but from a pool of cold water contained in a plastic box. A small aquarium pump is used to inject water into the "Green Distelector". Used water is directed back to the plastic box and recycled.

Experiment (3): Preparation of ethyl ethanoate

Objective

To perform a micro-scale preparation of ethyl ethanoate and determine the percentage yield

Experimental procedures

1. Assemble the setup of the "Green Distelector" and arrange for recycling cooling water. (initial installation of the small cylindrical bowl with a hook is not required).
2. Switch on the aquarium pump. Ensure that cold water is running into the inverted L tube, leaving through the outlet of the T tube and directed back to the plastic box containing the water reservoir.
3. Heat the sand bath to about 200^{o}C.
4. Using a clean Pasteur pipette, transfer 40 drops of 95% ethanol (absolute ethanol is the best but the chemical is less readily available) into a weighted test tube and determine the mass of ethanol.
5. Add 60 drops of 100% ethanoic acid (in slight stoichiometric excess).
6. Add 3 drops of conc. sulphuric acid followed by some anti-bumping granules.

7. Lower the inverted L tube and T tube combination into the test tube containing the reaction mixture. Secure the arrangement using an iron stand and clamp.
8. Place the test tube carrying the condensing device into a hot sand bath. Reflux for 15 minutes.
9. Remove the test tube which contains the product mixture. Add 3 cm^3 4M NaOH solution. Shake well and allow it to settle. Using a clean Pasteur pipette, transfer all of the ***top*** organic layer to another test tube.
10. Add enough spatula measures of solid calcium chloride. Hook the small cylindrical bowl onto the ring of the T tube and lower the combination into the test tube containing the dehydrated product. Place the whole assembly into the hot sand bath. Start micro distillation (Fig. 45, 46 and 47).
11. Transfer the content of the small cylindrical bowl when it is full to a weighted small test tube by using a clean Pasteur pipette.
12. Repeat the distillation procedure until ***all*** ethyl ethanoate has been collected.
13. Determine the mass of the ester collected.
14. Determine the b.pt. of the product ester.

Results

$$\underset{46}{C_2H_5OH} + \underset{60}{CH_3COOH} \rightleftharpoons \underset{88}{CH_3COOC_2H_5} + H_2O$$

Mass of ethanol = *0.64g*

No. of mole of ethanol = *0.64/46 = 0.0139*

Mass of prepared ethyl ethanoate = *0.66g*

No. of mole of ethyl ethanoate = *0.66/88 = 0.0075*

Percentage yield of ethyl ethanoate = *(0.0075/0.0139) x 100% = 54%*

B.pt. of prepared ethyl ethanoate = *74.0°C (Literature value: 78.3°C)*
Deviation of b.pt of product from literature value may indicate ether impurities.

Conclusion

Micro-scale preparation of ethyl ethanoate was performed with a percentage yield of 54%

Remarks

Ester formation reactions are reversible. The product formed after reflux was actually a mixture of ester, alcohol, acid and water. The boiling points of ethyl ethanoate and ethanol are so close to each other (ethanol: 78°C, ethyl ethanoate: 77°C) that direct distillation will give a very low yield of the ester. The mixture was first treated with NaOH(aq) to neutralize the acid, dissolve the ethanol and set free the ester from the aqueous solution mixture (with a slight loss of the product, as ethyl ethanoate has a solubility of 8%). The crude ester was dried by adding solid $CaCl_2$ and then distilled.

The small electrical aquarium pump must meet stringent safety requirements as it has to operate under water for a long period of time.

The all-glass design requires glass blowing expertise. Institutes with laboratory technicians equipped with such skills and tools can do the job.

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