DISTILLATION; PURIFICATION and SEPARATION OF of LIQUID ORGANIC COMPOUNDS
Part 2

REF: "*Aldrich*", "the *CRC*", "the *Merck*", and/or "*Lange's*"
The Organic Chem Lab Survival Manual, 7th ed., J. Zubrick, Wiley, Hoboken, NJ

EQUIPMENT: equipment in student lab drawers
Thermowell heating mantle with controller (*ref. Zubrick ch. 18*)
250-mL or 500-mL graduated cylinder
digital thermometers (*thermocouple probe & temp. display*)
Abbe' Refractometer (*ref. Zubrick ch. 30*)

MATERIALS: ethanol / water mixture from fermentation flask
boiling chips

INTRODUCTION

Yeast ferments sugars to produce ethanol, per the following (*unbalanced*) equation:

$$C_{12}H_{22}O_{11}(aq) + H_2O(liq) \xrightarrow{yeast} CH_3CH_2OH(aq) + CO_2(g)$$

Today your task is to get the yeast to start working for you. After the yeast has done its job over the next week, you'll recover the ~~hootch~~ ethanol product via distillation (ref. Zubrick ch. 20).

The production of alcohol via fermentation is anaerobic; if O_2 is freely available, only CO_2 and H_2O are produced. Therefore, you'll need to set up equipment to allow the CO_2 to escape, but not allow any O_2 to reach the sugar and yeast.

SAFETY & WASTE

Review Zubrick, chapter 1. If you have any questions about lab safety, ask your instructor before proceeding with any experiment.

The barium hydroxide solution should not be discarded in the sink. If you have extra $Ba(OH)_2$ solution, place it into the waste beaker in the fume hood or share it with another group. As always, wash your hands before you leave the lab.

As always, goggles must be worn when anyone is performing any experimental procedure in the lab.

PROCEDURE, Part 1 (*from last week*)

1. Make a neat, legible list of properties for sucrose, ethanol, mineral oil, and $Ba(OH)_2$. This should include (ref. Zubrick ch. 3): systematic (IUPAC) name, molecular & structural formulas, melting & boiling points, solubility information, and safety and toxicity information.

2. Start the fermentation process.

One week later…

PROCEDURE, Part 2

While you're performing this procedure, you'll need to record data and answer questions on pages 5-6. Failure to do so could mean that you'll need to re-do this experiment.

3. Refer to Zubrick ch. 20 to determine what type of distillation – *Class 1: Simple* or *Class 3: Fractional* – that you'll need to use to separate your mixture of water and ethanol. Before you proceed, check your decision with your instructor. (*If you simply ask your instructor what you should do, your lab report will be awarded a 1 point penalty.*)

4. Gently decant most (~400 mL) of your liquid from the fermentation flask into a 500-mL round-bottomed flask.
5. Add a few (~5-10) boiling chips to the flask.

6. READ "CLAMPING A DISTILLATION SETUP" in Zubrick, ch. 19.
7. Assemble your distillation per fig. 20.10 in Zubrick, ch. 20.

8. In Zubrick, ch. 20 READ:
    a. "CLASS 1: SIMPLE DISTILLATION"
    b. "THE DISTILLATION EXAMPLE" & "THE DISTILLATION MISTAKE"
    c. "CLASS 3: FRACTIONAL DISTILLATION"

9. Start heating and distilling the water/ethanol mixture. Record the temperature when the first drop of distillate is produced, and continue to record the distillation temperature every minute or so.
10. Collect about 150-200 mL of distillate into a preweighed flask or beaker. DO NOT BOIL THE DISTILLING FLASK DRY.
11. Determine and record the volume and mass of the crude distillate you've collected.

12. READ Zubrick, ch. 30.
13. Determine and record the refractive index of this crude distillate.

14. Empty the remaining waste liquid and solids from the 500-mL distillation flask (down the drain) and thoroughly wash it out.

15. Pour the crude distillate back into the clean 500-mL round-bottomed flask.
16. Add a few boiling chips to the flask, and start heating to maintain a slow, steady boil.
17. Monitor the temperature throughout the entire distillation process, beginning when the first drop of distillate is produced; record this temperature on a regular basis, such as every 60 seconds.

18. Collect the fraction that boils at about 72-78°C into a pre-weighed 50- or 100-mL graduated cylinder.
19. In any case, DO NOT BOIL THE DISTILLING FLASK DRY.

20. Determine and record the volume and mass of the second distillate you've collected.

21. Determine and record the refractive index of the second distillate. (ref. Zubrick, ch. 30)

22. Perform the calculations and answer the questions on pages 5-7.

23. **ON OR BEFORE THE DUE DATE**, turn in this report, in this order: (*If your lab report is not neatly assembled in this order and stapled, your lab report will be awarded a 1 point penalty.*)
    a. pages 5-7
    b. your Table of Physical and Chemical Properties (Procedure, step 1)
    c. pages 1-4

Aqueous Ethanol					
Density	**% by wt**	**g/100 mL**	**Density**	**% by wt**	**g/100 mL**
0.989	5	4.9	0.856	75	64.2
0.982	10	9.8	0.843	80	67.5
0.975	15	14.6	0.831	85	70.6
0.969	20	19.4	0.818	90	73.6
0.962	25	24.0	0.815	91	74.2
0.954	30	28.6	0.813	92	74.8
0.945	35	33.1	0.810	93	75.4
0.935	40	37.4	0.807	94	75.9
0.925	45	41.6	0.804	95	76.4
0.914	50	45.7	0.801	96	76.9
0.903	55	49.6	0.798	97	77.4
0.891	60	53.5	0.795	98	77.9
0.880	65	57.1	0.792	99	78.4
0.868	70	60.7	0.789	100	78.9

TABLE 1 - % Ethanol by Water/Ethanol Mixture Density
g/100 mL = grams ethanol / 100 mL of solution; **% by wt** = mass of ethanol / mass of solution

Alcohol concentration	**Refractive index to air (20°C, Na light)**
0%	1.3333
10%	1.3395
20%	1.3469
30%	1.3535
40%	1.3583
46%	1.3604
50%	1.3616
60%	1.3638
70%	1.3652
80%	1.3658
90%	1.3650
100%	1.3614

TABLE 2 - Mass % Ethanol by Water/Ethanol Mixture Refractive Index

NAME:_____

DATA, CONCLUSIONS & QUESTIONS

If you need more space to perform calculations, you may attach additional pages to this lab report.

Theoretical Yield Calculations

1. balanced chemical equation for fermentation of sucrose to produce ethanol:

2. mass of sucrose used..... _____ g
3. theoretical yield of ethanol (*ref. Zubrick, ch. 2*) ..... _____ g

First (Crude) Distillation

4. temperature when distillation began (*ref. Zubrick, ch. 20*)..... _____ °C
5. temperature when distillation was stopped..... _____ °C

6. mass of ethanol/water mixture collected (*ref. Zubrick, ch. 20*)..... _____ g
7. volume of ethanol/water mixture collected ..... _____ mL

Mass of Ethanol in Crude Distillation Solution by Density:

8. density of ethanol/water mixture collected ..... _____ g/mL
9. % ethanol from Table 1 ..... _____ %
10. mass of ethanol from density ..... _____ g

Mass of Ethanol in Crude Distillation Solution by Refractive Index:

11. refractive index of ethanol/water mixture collected (*ref. Zubrick, ch. 30*) ..... _____
12. % ethanol from Table 2 ..... _____ %
13. mass of ethanol from refractive index ..... _____ g

14. estimated % ethanol from first (crude) distillation (*ref. items 9 & 12*) ..... _____ %
15. estimated mass of ethanol collected in first (crude) distillation (*ref. items 10 & 13*) .... _____ g
16. % yield of ethanol from first (crude) distillation (*ref. Zubrick, ch. 2*) ..... _____ %

Second Distillation

17. temperature when distillation began (*ref. Zubrick, ch. 20*)..... _____ °C

18. temperature when distillation was stopped (*ref. Zubrick, ch. 20*) ..... _____ °C

19. mass of ethanol/water mixture collected ..... _____ g

20. volume of ethanol/water mixture collected ..... _____ mL

21. density of ethanol/water mixture collected ..... _____ g/mL

Mass of Ethanol after Second Distillation by Density:

22. % ethanol from Table 1 ..... _____ %

23. mass of ethanol from density ..... _____ g

Mass of Ethanol after Second Distillation by Refractive Index:

24. refractive index of ethanol/water mixture collected (*ref. Zubrick, ch. 30*) ..... _____

25. % ethanol from Table 2 ..... _____ %

26. mass of ethanol from refractive index ..... _____ g

27. estimated % ethanol from second distillation (*ref. items 22 & 25*) ..... _____ %

28. estimated mass of ethanol collected in second distillation (*ref. items 23 & 26*) ..... _____ g

29. % yield of ethanol from second distillation (*ref. Zubrick, ch. 2*)..... _____ %

QUESTIONS

30. Why did you have to set up a trap to keep air out of the fermentation flask?

31. Compare and contrast your % yield and % purity (% yield) from the first and second distillations. What generalization can you make about % yield and % purity from distillation processes?

32. Why were you not able to recover 100% pure ethanol despite twice-distilling your product? (*ref. Zubrick, ch. 20*)

Attach your table of physical and chemical properties (Procedure, step 1) behind this page.