Course: CH 224-54

Instructor Name: Dr. Kwan

Experiment 3: Esterification

This is a brief & good objective of the experiment

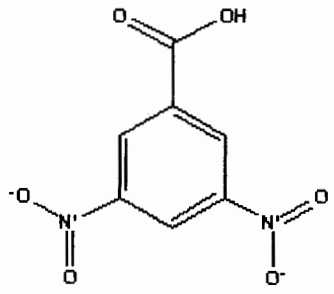
Introduction:

In this lab an ester was made using an unknown alcohol and an unknown acid, both of low molecular weight. The low molecular weight caused them to have a terrible smell. On the other hand, the esters had a sweet, fruity smell and were commonly used as natural products.[1] Esters ~~were~~ are carboxylic acid derivatives. The –OH group of a carboxylic acid was replaced a different electron-withdrawing group. The electron-withdrawing group in esters was alkoxy groups (–O–R). The alkoxy group commonly came from an alcohol.[2] The common formula of an ester is RCOOR. To identify the ester, it was transesterified with 3,5-dinitrobenzoic acid, Figure 1. Nitro groups are very polar and their ester derivatives usually have sharp melting solids. These properties made the ester identifiable.[1]

good & brief description of the reaction. Including a mechanism would also be good to put in for a reaction.

These statements give specific information about the lab being completed. This is always a good idea to include.

Figure 1: 3,5-dinitrobenzoic acid[3]



computer generated graphics!

Experimental:

An unknown mixture (#305) of an acid and an alcohol, each of low molecular weight, was placed in a 100-mL round bottom flask containing a boiling stone and a stir bar. While stirring, approximately 4 mL of concentrated H_2SO_4 was added slowly to the flask. The reaction was heated under reflux, Figure 2, for 1-½ hours. The mixture was allowed to cool to room temperature and then transferred to a 250-mL separatory funnel and 20 mL of diethyl ether was added. The mixture was washed by shaking the

Notice how All figures are referenced in the text! You MUST do this. DO NOT attach figures which are not in your text!!!

also, notice the specific details in the procedure. It is detailed & flows in a narrative, do not write choppy procedures which do not follow a LOGICAL order!

[1] Handout. *Esterification.* 38

[2] Wade, L. G. *Organic Chemistry Fifth Edition.* Prentice Hall: New Jersey. 2003. 426, 474.

[3] Structures taken from chemfinder.com

separatory funnel vigorously with one 15-mL portion of deionized water. Twenty milliliters of ice were added to the funnel and the mixture was washed with 20-mL portions of 20% NaOH until a basic pH was obtained. If the solution became warm more ice was added to lower the heat. Once a basic pH was obtained in the aqueous layer, the mixture was washed once more with 15 mL of deionized water. The aqueous layer was drained off and the ether was dried over anhydrous $MgSO_4$ for 10 minutes. The ether was gravity filtered into a clean, dry 100-mL round bottom flask.

The ether mixture was distilled by simple distillation, Figure 3, into fractions. The distillation continued until a plateau was reached, Table 1. When a distillation temperature reached plateau and then escalated, the distillation was complete. The fractions of pure ester were combined and a micro boiling point (124.3° - 127.1°C) and a refractive index (n^{20} = 1.3815) were obtained.

NOTICE physical data is in this section!

Two milliliters of the pure ester were put in a 50-mL round bottom flask. Two drops of concentrated H_2SO_4 and approximately 1.5 g of 3,5-dinitrobenzoic acid (1.512 g) were added to the flask. The mixture was heated under reflux as in Figure 2 for 60 minutes. The reaction mixture was then dissolved in 25 mL of diethyl ether and extracted with two 15-mL portions of 5% Na_2CO_3 followed by one 10-mL portion of deionized water. The ether layer was dried over anhydrous $MgSO_4$ for ten minutes and allowed to evaporate. The ether was either evaporated under reduced pressure or for one week. If evaporation was performed under reduced pressure, an oily residue formed. The residue was dissolved in 3-5 mL of boiling absolute ethanol. Boiling deionized water was added until the 3,5-dintrobenzoate ester began to separate. The mixture should become cloudy. The mixture was allowed to cool to induce crystallization. If the reaction mixture were allowed to evaporate for a week, then crystals would have formed (m.p. = 64.6° – 66.8°C).

Notice the 3rd person passive! Do not forget to write in this tense!

Figure 2: Heating under reflux[4]

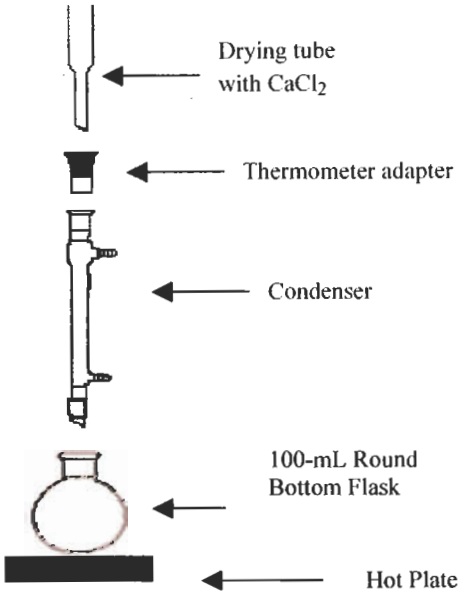
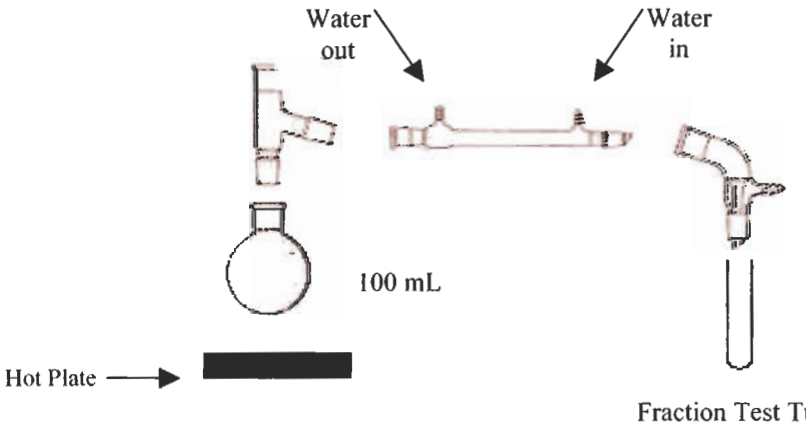


Figure 3: Simple Distillation[4]



[4] K. Poleski. Glassware taken from previous lab reports.

Yellow Sheet

observations
measurement
procedures

Figure 4: n-butyl acetate[3]

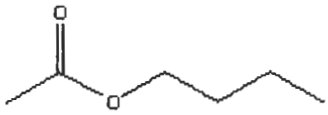


Table 1

Fraction	Temperature, °C
1	100
2	110
3	112
4	114
5	114
6	114
7	115
8	115
9	115
10	115

The physical data can be repeated in this section where you EXPLAIN your results. Do not simply state them

Discussion:

The ester that was formed in this experiment was believed to be n-butyl acetate, Figure 4. The literature value for the boiling point of n-butyl acetate was 127° C. The micro boiling point obtained in this experiment was 124.3° - 127.1° C. The refractive index for this ester was $n^{20} = 1.334$. The refractive index obtained in the lab was $n^{20} = 1.38315$. The melting point of the 3,5-dinitrobenzoate crystals should be around 63° C. The crystals formed in the lab melted around 64.6° - 66.8° C. The increase in the melting point could have been due to error in reading the melting point apparatus at the right time. The data that helped to identify the ester the most was the melting point of the crystals. This narrowed the field of the number of choices; the boiling point of the ester was next helpful. The NMR matches nicely with the structure of n-butyl acetate also. Identifying the ester and then matching the peaks of the graph to the structure determined this. There were 5 peaks on the NMR spectrum, which corresponded to the 5 sets of non-chemical shift equivalent hydrogens. This is an explanation of why.

This is an explanation of WHY or HOW.

[3] Structures taken from chemfinder.com

NMR Spectrum