Experiment 3 Laboratory Report (40 pts)

Directions: Make a copy of this document and save it to your Google Drive. Type into the designated areas. Boxes can be expanded, but your answers must be in boxes. Answers in the tables can be words or phrases and do not have to be complete sentences. All answers for questions not in tables must be answered in complete sentences. Points will be deducted for excessively wordy answers or changing the format of the report, although table boxes can be made bigger if necessary. Avoid having tables or responses to questions going from one page to the other to facilitate grading.

<u>Upload your report as a .pdf to Gradescope and make sure to carefully mark which questions are on each page. Please note that you can be asked for access to the Google Doc version of this assignment if there is suspicion of cheating or plagiarism.</u>

<u>Question 1</u> Complete the reaction table below. (*3.5 pts*)

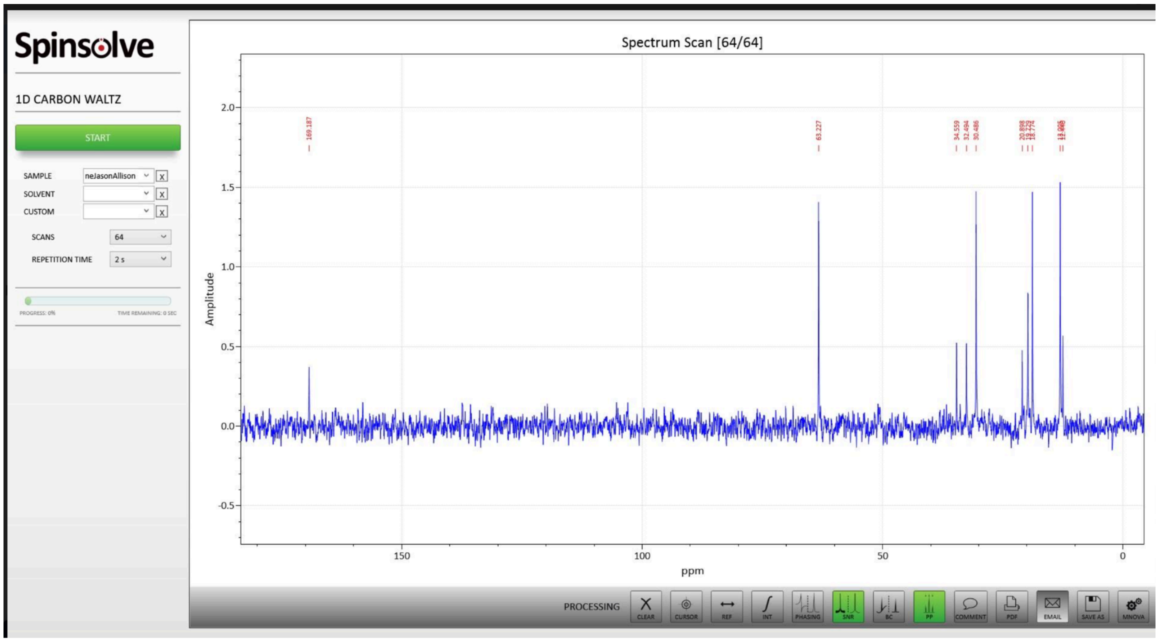
Compound	Molar mass (g/mol)	Amount used (mL or g)	Moles used	Density@T (^oC)
Butyl bromide	137.0183 g/mol	2.80 ml	0.0204 mols	1.27 g/cm³
Potassium acetate	98.15g/mol	1.502 g	0.0153 mols	---

Identify the limiting reactant (*0.5 pt*): Potassium Acetate

<u>Question 2</u> IR analysis - What new peaks appeared in your product's IR spectrum compared to the spectrum of butyl bromide that indicate the new functional group that was formed? You should list peak positions (cm^{-1}) and qualitative intensities (strong, weak, broad, etc.) and discuss how this tells you which new functional group was formed. (4 pts)

In our product's IR spectrum we saw a very strong, new peak at about 1743 cm^-1, medium 1366 cm^-1, and a weak peak at 1031 - 1042 cm^-1. These seem to indicate the formation of the functional groups of esters, bent alkanes, and amines. I was able to identify these functional groups through comparing the new peaks listed with the list of intensity to peak measurements of the functional groups as listed on the module assignment on canvas.

Question 3 NMR analysis - How many peaks were in your products ^{13}C NMR spectrum? How many carbons does this mean your product had in comparison to the starting alkyl bromide. What do the chemical shifts of the product peaks tell you about the functional group present in the product? (4 pts)



In our product we saw about 5 major peaks and about 5-6 minor peaks. This would indicate that our product had 5 carbons which is greater than our starting butyl bromide, which would have 4 carbons. Thus it seems our product had gained a carbon. One of these carbons is at the 169.2 ppm mark, much more compared to the rest of the carbons which is to indicate it is experiencing great electron shielding, most likely a result of an ester bond. The carbon at the 63.2 ppm seems to be bonded to an oxygen based on the literature. The Carbons at 30.4 and 19.729 seem to be bonded to CH2 and the 12.446 carbon bonded to a CH3.

Question 4 - Identification of **product by CAS-RN** (*2 pt*): 109-60-4 (Propyl Acetate)

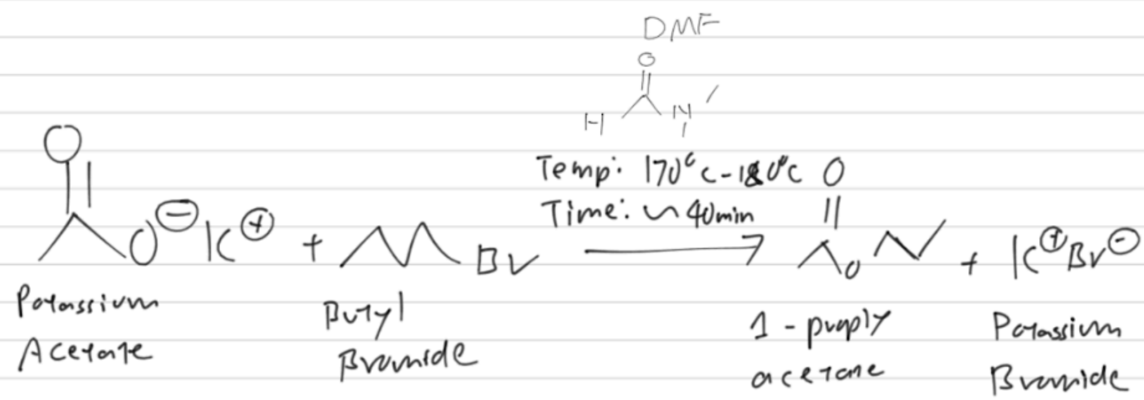
Complete the table below for the product. (*5 pts*)

Molar mass (g/mol)	Amount obtained (g)	Moles obtained	Experimental Boiling point, °C	Literature Boiling Point, °C
102.131 g/mol	**0.697g**	**0.00682 mols**	**96-126.34**	**101.24 C***

Citation for Boiling Point - Use the Reaxys database to obtain all the reference values. Citations from any other source will NOT be accepted. **See the Reaxys tutorial for specific information about this assignment.** (1 pt)

Xiaobin Liu, Yukai Zhang, Min Li, Xin Li, Guoxuan Li, Yinglong Wang, and Jun Gao
Journal of Chemical & Engineering Data 2018 63 (5), 1588-1595
DOI: 10.1021/acs.jced.7b01107

Question 5 Draw the structure of the overall reaction, including the reactants and products, solvent, and reaction conditions. Replace my generic image below with your reaction. You can do this using ChemDraw, or drawing the structure neatly, either is fine. (3 pts)



Question 6 Show all the calculations to determine:

a) the theoretical yield of the product in grams (*1 pts*)

0.0153 mols (Potassium acetate) * 102.131g /1 mol (Potassium acetate) = 1.562 g

b) the % yield of the reaction. (*1 pts*)

0.697 g / 1.563 g = 44.6%

Question 7

Insert your GC report below: (1 pts)

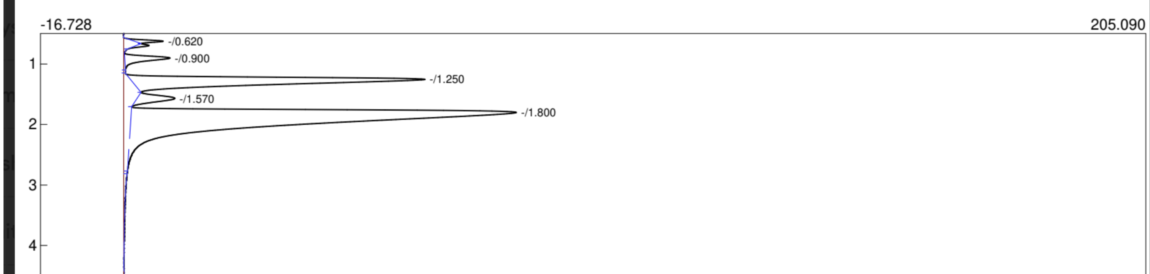
Lab name: SRI Instruments
Client: YourLabName
Client ID: N12000
Analysis date: 03/11/2024 15:23:11
Method: 1ul syringe on-column
Description: FID Detector hi gain
Column: 30MXT502 1 mic
Carrier: H2 from BIHG
Integration: Peak sens=95.0 Base sens=60.0 Min area= 10.00 Standard= 1.000 Sample= 1.000 Tangents=off
Data file: ChannelOne-18.chr ()
Sample: 333 calibration sd

Temperature program:

Init temp	Hold	Ramp	Final temp
50.00	1.000	10.000	110.00

Events:

Time	Event
0.000	ZERO
0.000	SOUND



Component	Retention	Area	Internal	Units	Norm area %
-	0.620	14.5101	0.0000		N/D
-	0.900	49.0770	0.0000		N/D
-	1.250	436.7382	0.0000		N/D
-	1.570	52.5571	0.0000		N/D
-	1.800	1160.3294	0.0000		N/D
		1713.2118	0.0000		100.0000

<GC Report>

What is the purity of your ester product based on your GC trace? Show your calculation (if relevant), and briefly indicate how you knew which peak was your ester product and which were impurities (if relevant). (2 pts)

The purity of our ester came out to be about (1160.329/1713.212) = 0.677 or about 67.7%.

The second peak is our ester product since out it has a comparatively higher boiling point compared to the reactant. Thus the first peak is our reactants of possible leftover butyl bromide and potassium acetate and impurities, while the second peak is thus our ester product. So we take the value of that and divide it by the total to get 67.7%

Question 8

Learning Link: List two techniques from previous experiments that were performed in this experiment. Briefly explain why we used each of the techniques in the current experiment. Be specific. (*60 words or fewer; 4 pts*)

Liquid-liquid extraction, was used to separate our product from unreacted reactants, water, and DMF which allowed for purification. IR spectroscopy was used for product and functional group identification as well as purity assessment.

Question 9

Intermolecular forces and polarity are topics we have discussed throughout lab this semester. Discuss how these concepts were important in terms of the experiment we did in lab this week? (*At least 3 sentences; 3 pts*)

In this experiment we used many concepts within intermolecular forces and polarity in order to perform our isolations and confirm our experimental results. Notably, the polarity of the molecules in this lab allowed us to use the seperatory funnel and hexane to separate between the non-polar and polar. Furthermore, the intermolecular forces between our substances direclty influence their boiling point which is another measurement we took advantage of in our distillation process to separate our product and purify it. Boiling point was also used in our gas chromatography for a similar use in determining our product purity. The difference in structure was also essential for the use of IR and NMR spectroscopy which uses differing structure groups and their effects on carbon electrons respectively to help us identify the product at hand.

By digitally signing below, I verify that all data collected, observations, and answers provided on this lab report are my own and are not duplications of another student's report either at Brown or at other institutions.

Digital Signature: JASON LIN

Banner ID: B01881807