

The Laboratory Notebook for Chem 269.

Use only the required notebook, one that allows a copy of each page to be made and torn out. The copy is given to the TA for grading and the original is kept by the student. Entries should be dated and entered in ink. There is no separate lab report. The entire write-up is done in the notebook.

Note that there are two basic types of experiments – Techniques and Reactions. Each will have a slightly different format. Technique experiments are those that are done to learn a new lab technique. These are done in the first half of the semester. In these types of experiments, lab chemicals are used but no reactions are carried out so the chemicals do not chemically change from beginning to end. An example of such an experiment would be the purification of a crude compound by a technique called recrystallization. Impure compound X is recrystallized to produce pure compound X. No chemical change has occurred. In a reaction type of experiment, starting with compound Y, one carries out a chemical reaction to produce compound Z. In both, one would make a table of all chemicals used, along with their physical properties. The difference lies in the measure of loss of product during the operation. In a technique experiment, one simply calculates a % recovery. If you start with 0.060 g of compound X, and recover 0.045 g after carrying out the technique, the % recovery is simply $(0.045/0.060) \times 100 = 75\%$ recovery. The loss is simply a result of mechanical losses that occur in carrying out the technique. In a chemical reaction type of experiment, the molecular weight (MW) of product is usually different than the MW of reactant, so a % yield must be calculated to measure the effectiveness of the experiment. The loss in such a case is not only the result of mechanical losses but also of side reactions occurring that would consume reactant. The first technique experiment is “distillation” and the first reaction type of experiment is the “preparation of cyclohexene”. An example showing how to calculate a % yield is given in the “cyclohexene” experiment.

Prelab Entries.

Title of experiment and **short description of the purpose** of the experiment.

Balanced reaction using structural formulas. Include side reactions and products.

Table of reactants, products, and side-products. This should include molecular weight (MW), relevant physical properties such as melting point (MP), boiling point (BP), density, solubility information, toxicity, and quantities used (moles, grams, milliliters). All physical properties are not always needed for all the compounds. For example, BPs would be needed if a distillation were to be carried out, but including the MP of a liquid would not be useful. Solvent and catalyst data should also be noted (e.g., BP, toxicity, density). Such information can be found in the “CRC Handbook of Chemistry and Physics”, available in the library, in the organic lab, and on the library website. Chemfinder.com and web searches in general can also be helpful here.

Prelab outline. This is the heart of the prelab preparation. It is a short version of the lab procedure, written in outline form, in your own words. It is based on the experiment handout and information gained from the OWL prelab exercises. Read the handout carefully, do the

OWL assignments and synthesize a prelab outline. The purpose of the outline is to help you prepare for the experiment and carry it out in the lab. Trying to do the experiment directly from the handout, without having prelab outline is inefficient and ineffective. You will likely need more time to do the experiment that way and will likely have errors. It is also very unsafe to work in an unprepared manner. **Work will not be allowed until the TA has checked and accepted the prelab outline.** If you come to lab without having an acceptable outline, you will be asked to leave and return only after you have an acceptable outline. One point of ten will automatically be deducted. Also, you will unlikely have sufficient time to complete the experiment, leading to further loss of credit. Include safety and waste disposal instructions in the outline, as well as sketches of equipment.

In-lab Entries.

As you work in the lab, enter directly into the notebook, the actual procedure that you follow. This will differ somewhat from the prelab outline. The prelab outline is what you expect to do. The in-lab entry is what you actually do. Measurements such as weighings, MPs, BPs, and chromatographic and spectroscopic data should be entered as the data is determined. Careful observations should be noted (“the flask contents turned deep red, smoked profusely, and exploded, blowing a two foot hole in the lab bench.” The idea here is to provide as much detail as you can so that another worker could exactly reproduce what you did and get the same results. When you work in a research lab, this is extremely important so learn it and practice it here.

Before leaving the lab, clean up your workspace and any common workspaces that your TA may ask you to clean up, and **get the TA to sign the notebook right after the last entry.** **Unsigned work will not be given credit.**

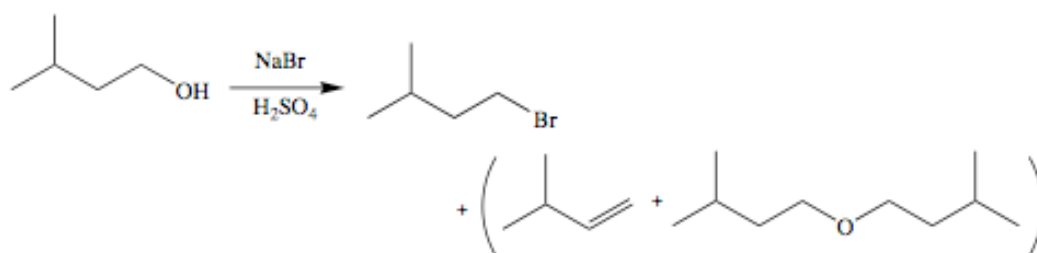
Postlab Entries. Results, Discussion, Answers to Assigned Questions.

Calculate % recoveries (starting with compound X, doing an operation on it, and recovering compound X, what % did you recover?) and/or % yields (starting with compound X, carrying out a reaction, recovering compound Y, how much did you recover and how did that compare to the theoretical amount? Moles and stoichiometry needed.) Summarize results in an easy to read format (e.g., compound X, MP 101.5-103.5° C, 0.067 g obtained, 56% yield). Interpret spectra and chromatograms, write a brief (less than 1 page) conclusion, and answer any assigned questions.

A partial sample report for a reaction type of experiment.

Synthesis of 1-Bromo-3-methylbutane.

The purpose of this experiment is to synthesize 1-bromo-3-methylbutane by an S_N2 reaction of 3-methylbutanol with hydrobromic acid. The HBr is produced in the reaction flask by reaction of sodium bromide with sulfuric acid. The product is purified by distillation and analyzed by gas chromatography (GC).



Reagents

Reagent	MW	Density	BP (°C)	wt used (g)	mmoles theory	mmoles used
3-methyl-1-butanol	88.2	0.809	129	0.900	10.2	10.2
NaBr	102.9	-	-	1.18	10.2	11.5
H ₂ SO ₄	98.1	1.84	-	1.96	10.2	20

Products and By-products

			BP (°C)	BP (°C)	Theoretical Yield	Theoretical Yield	Found	Found
Compound	MW	Density	actual	found	mmoles	g	g	g
1-bromo-3-methylbutane	151.0	1.207	120.4		10.2	1.54 (100%)		
3-methyl-1-butene			20					
isobutyl ether			124					

PRELAB OUTLINE.

(draw sketch of set-up)

- weigh 1.18 g NaBr into a 5 mL round-bottomed flask.
- add 0.90 g 3-methyl-1-butanol and 1 mL water to flask
(USE EXTREME CAUTION IN USING SULFURIC ACID)
- carefully add 1.07 mL (1.96 g) conc sulfuric acid with constant swirling.
- add a boiling chip
- connect distilling head and attach receiving vial.

- place receiving vial in 50 mL beaker containing ice-water. Beaker held in place with large three-pronged clamp.

- heat flask for 1 hr

- increase heat to distill crude product.

- (and so on and so on.)

- distill dried product into a clean, dry tared vial using a fractional distillation set-up (sketch set-up), collecting product over a temperature range of about 117-121°C.

- measure amount obtained

- carry out GC

- WASTE: place all liquid waste into the ORGANIC LIQUID WASTE container in the fume hood.

PROCEDURE AND OBSERVATIONS.

NaBr 5.252 g

4.072 g tare

1.180 g

write down all weighings as shown for NaBr directly into the notebook as they being done

A sand bath was set to just under 40 to warm up. To a 5 mL rb flask, was added 1.180 g NaBr, 1 mL water, and 0.90 g 3-methylbutanol. The flask was swirled well to mix the contents. With extreme caution, 1.07 mL conc sulfuric acid was added dropwise with a pipet, with constant swirling. The flask became quite warm and the mixture turned yellow. A small amount of sulfuric acid was accidentally spilled on the desktop. This was diluted with a large amount of water and wiped up with a paper towel, which was then rinsed thoroughly with copious amounts of water and disposed of in the solid waste container in the hood. The flask was clamped securely to a ring stand using a small three-necked clamp. A black plastic connector was used to attach the distilling head and a thermometer was attached to the head with a thermometer adaptor. The thermometer was placed so that the top of the bulb was level with the outlet on the distilling head. A large sample vial was attached to the outlet of the distilling head with copper wire. The flask was set down into a depression in the sand bath and a 50

mL beaker with ice-water was brought up to the bottom of the vial. The reaction mixture began to boil in about 4.5 minutes. The mixture turned darker and darker brown as the reaction proceeded, and a faint acrid smell was noticed (probably HBr).

(and so on and so on)

..... The fractional distillation resulted in 0.95 g of 1-bromo-3-methylbutane, which was collected over a temperature range of $119.5 - 121^{\circ}C$. The sample was injected into the GC with the help of the TA, resulting in the following chromatogram. (sketch or tape GC trace to notebook)

(clean up work space and ask TA for signature)

RESULTS AND DISCUSSION.

% yield calculation

expected: 1.54 g

obtained: 0.95 g

GC indicated purity of 88% so actual amount of product is $0.95 \times .88 = 0.84$ g

$$(0.84/1.54) \times 100 = 55\% \text{ yield}$$

Summary of results:

1-bromo-3-methylbutane: 0.84 g (88% purity by GC, 55% yield), BP
119.5 - 121°C

1-bromo-3-methylbutane was synthesized in 55% yield. Because of the possible side-reactions shown above, some of the reactant alcohol was converted to byproducts instead of the desired product. Mechanical losses due to evaporation of product and leaving behind small amounts of product in each step are to be expected in extractions and distillations. The

*product was shown to be relatively pure by GC and by the observed BP.
The IR spectrum showed no alcohol group in the distilled product.*

Using a higher-boiling chaser in the distillation would have led to a larger amount of product being distilled, resulting in a higher yield. (and so on and so on)

ANSWERS TO ASSIGNED QUESTIONS.

1. blah blah blah

2. (and so on and so on)