For every non-technique, synthetic experiment you will be required to submit a typed formal report. Follow the guidelines below.


Pre-lab, notebook and lab technique are all worth 5 points each.

**Heading (5 points):**

Each formal report should begin with a heading, which includes the title of the experiment performed, your name, the name of your teaching assistant, and the submission date of the report.

**Purpose (5 Points):**

The purpose statement should include the names of reactants, reagents, and products and explain the type of reaction that is being performed (for example: alkylation, acylation, condensation, amidation, esterification, nitration, substitution). When appropriate, use the phrases “acid-catalyzed” or “base-catalyzed” to speak more generally about conditions, but detailed descriptions of solvents or other reaction conditions should be avoided. Overall, the purpose should contain enough information to draw the reactants and product(s) but not give specific details of how the experiment was conducted.

**Reaction Scheme (5 Points):**

This graphical representation is the most clear and straightforward means to describe a chemical reaction. The reactants (starting materials AKA substrates) are shown to the left of the arrow, reagents in excess, catalysts, and solvents as well as temperature and reaction time are written above and below the arrow, and the products and byproducts are shown to the right of the arrow. There is only one accepted mean to present a reaction scheme: use of a chemical structure drawing program,
such as ChemDraw\textsuperscript{1}, to create your own graphic. Do not hand-draw or cut and paste the reaction scheme from the lecture notes or an internet source. You should read the document “ChemDraw Tutorial” posted in the general handouts portion of the course website. There is educational value in creating the scheme yourself and your TA will award no points for inclusion of someone else’s work.

**Experimental Procedure (25 Points)**

This section should be as necessarily detailed as a recipe so that another organic chemist could reproduce the experiment. See the example on page 5-6 of this handout for a well-written procedure.

**Results and Discussion (35 Points) – 15 points for results portion, 20 for discussion portion**

Results Portion:

You should restate all product results (BPs, MPs, yields, etc.) in tabular form, including any characterization (IR, GC) that was obtained and discuss the actual results and what they mean. E.g., “The results of this experiment are summarized in the table below.”

<table>
<thead>
<tr>
<th>Reaction Product</th>
<th>Melting Point (°C)</th>
<th>Boiling Point (°C)</th>
<th>Percent yield</th>
<th>Characterization Methods Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-methyl-1-butene</td>
<td>Not Obtained</td>
<td>Not Obtained</td>
<td>75</td>
<td>GC and \textsuperscript{1}H-NMR</td>
</tr>
</tbody>
</table>

Discussion Portion:

**First Sentence:** “The starting materials were reacted using conditions specific to the reaction being performed and the target product was obtained in XX% yield.”

The first sentence of the discussion is a more detailed version of the purpose statement but distinct since it refers to the physical state and percent yield of the product.

\textsuperscript{1} ChemDraw is available free of charge using your umass email address. See the document “ChemDraw Tutorial” posted on the course website for instructions on how to obtain this software.
End Sentence(s): “The identity and purity of the product was assessed.”

Discussion Portion (continued):

Product identity is most often supported by the relative closeness of your observed mp, or bp, value to the literature value for the target compound. The most straightforward situation is when your observed range overlaps the range of the literature value, which confirms the identity of your product with little uncertainty. For mp determination: when the observed range is below that of the literature value, it is best to suggest that slight contaminates may be depressing the melting point value. For bp determination: deviation from the literature values usually indicates systematic error in thermometer placement. The purity of your isolated product can also be established by noting a relatively narrow melting/boiling range observed. A narrow range is ideally between 1–2 °C, widening slightly with an increase in the magnitude of the value. An observed range of 5 °C or more warrants a qualification of the product purity. For TLC analysis, the product should contain only one product band (ideally in a divergent pair of solvent conditions) to be considered pure.

The excerpts below are in order of decreasing adequacy with respect to the start of discussing results:

1. “In this lab 2-methyl-1-butene was synthesized via the elimination reaction of 1-bromo-2-methylbutane in the presence of potassium tert-butoxide. Product identified to be 2-methyl-1-butene via \(^1\)H-NMR. The signal at 4.7 ppm is a singlet as expected as the terminal alkene protons are equivalent and do not experience spin-spin coupling. The chemical shift of these protons is also indicative of a typical alkene. The starting material, 1-bromo-2-methylbutane, would not show any signal this far downfield (halide-bearing carbon protons generally 3 ppm).”
2. "2-methyl-1-butene was synthesized. Product identified to be 2-methyl-1-butene via \(^1\)H-NMR. The signal at 4.7 ppm is a singlet as expected.” Notice a significant amount of interpretation is missing.

3. “2-methyl-1-butene was synthesized. Product identified to be 2-methyl-1-butene via \(^1\)H-NMR.” Even more interpretation is missing.

4. “Product identified to be 2-methyl-1-butene.” is completely inadequate.

1. DISCUSSION does not mean CONFESSION: avoid discussion of mistakes, accidents, spills, and human error.

2. Avoid discussion of mass loss through transfers, recrystallization, or weighing.

3. DO challenge assumptions made about the procedure.

4. DO consider this modification to be something that you would actually try if you were to repeat the experiment in our lab.

5. DO discuss specifics about the reaction equation and/or the stoichiometry table

6. DO justify changes in catalyst, solvent, reagent, temperature, time, and/or molar ratio using your chemical/mechanistic understanding of the reaction.

7. DO be clear and logical about what the change would accomplish.

8. Express your understanding of what a catalyst does and how it cannot improve the percent yield; it only allows a reaction to reach completion, or alternatively reach the equilibrium mixture at a faster rate (which could produce more molecules of final product in a limited reaction time).

9. Express your understanding of what reactions are in equilibrium and when Le Chatelier’s principle can be considered.

10. Express your understanding of the affect of concentration on the reaction rate.
11. Express your understanding of increasing/decreasing temperature on the reaction rate, or formation of products/side products.

12. Express your understanding of how unstable reagents may result in lower than expected yield.

**Post-lab Questions (10 Points):** Provide written answers to these in your notebook as the last page of the experiment. Attach as the last page to the typed report.

**The Most Common Mistakes**

- Spaces follow every unit of measure, and before the degree (°) symbol, except the percent sign and in the event of describing the size of a piece of equipment (125-mL Erlenmeyer flask). The degree symbol in MS Word . . . Insert tab|Symbol|More Symbols, and find it. It is NOT a superscript zero or letter "o".
- Chemical names are only capitalized when they appear at the beginning of a sentence or when they appear in a title (title case).
- Leading zeros are required before all decimal places. For example 1.6 x 10⁻¹ should be written 0.16.
- Maintain a consistent number of decimal places in all of your stoichiometrically-relevant values. More than two decimal places and three significant figures are too much for most applications.
- Melting points values can contain a great deal of useful data, but be careful not to overstate their importance when discussing purity and identity.
- Use of incorrect abbreviations, for example, min means both minute and minutes, h is hour and hours, and rt is room temperature (not RT).

**Example Experimental Procedure:**

**Procedure:** To a 250 mL round bottom flask equipped with a magnetic stir bar was added acetic anhydride (60.0 mL, 638 mmol) with a catalytic amount (1 drop) of 70% perchloric acid. The solution was cooled to 0 °C and D-mannose (12.0 g, 66.6 mmol) was added, in ~1 g portions and not further until complete dissolution, over the course of 1 h while maintaining the temperature of the reaction below 50 °C. Upon completion of the addition, the solution was allowed to stir at rt for 1 h. After this time, the solution was light yellow. The reaction was poured over crushed ice and saturated aqueous NaHCO₃ (100 mL) was added. The resulting mixture was diluted with CH₂Cl₂
(200 mL) and stirred for 2 h. The organic layer was washed with saturated aqueous NaHCO$_3$ (3 x 50 mL) and water (2 x 50 mL). The solution was dried with MgSO$_4$, filtered and concentrated under reduced pressure to yield the crude product (24.3 g, 93%) as a light clear oil. The resulting oil was recrystallized from diethyl ether to obtain the product as a white crystalline solid (20.4 g, 78%). mp 148 – 149 °C (insert literature value in parenthesis if known). R$_f$ (6:1 hexanes:ethyl acetate) = 0.63. (Note that this last piece of data is for TLC.)