Sample Lab Report

Your name, the date the experiment was performed, your lab section, and your lab partner's name should be written at the top of the report.

Wittig Reaction

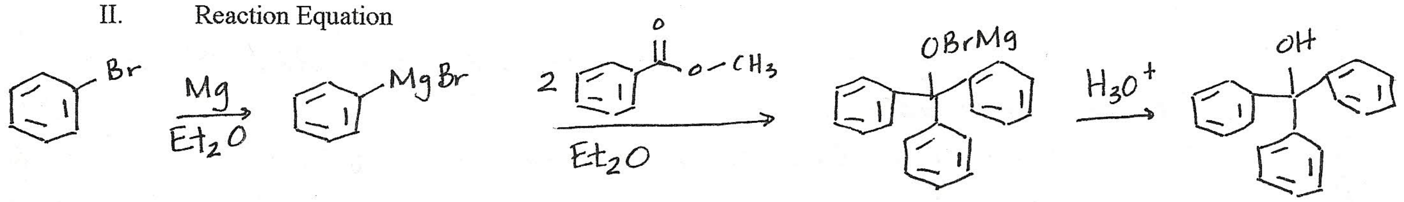
The title of the experiment should be **clear and concise**. Most TA's won't mind if you take your title directly from the lab manual.

I. Objective

The purpose of this experiment was to generate the alkene trans-9-(2-phenylethenyl) anthracene, from 9-anthradlehyde and benzyltriphenylphosphonium chloride via a Wittig Reaction. To assess the success of this reaction, melting point was taken and the yield calculated.

This section may also be labeled the "introduction" of the lab report. Include a brief statement of why this particular experiment is being performed and **what you hope to accomplish** by conducting this experiment. Like the rest of the lab report, this section should be written in **third person passive voice**. Include any necessary definitions here.

II. Reaction Equation



It is important to include this in your lab report to show the **chemical basis for the experiment** you are to conduct. This can be found in your lab manual and copied directly into your report.

III. Procedure

To begin, 200 mg of benzyltriphenylphosphonium chloride and 120 mg of 9-anthraldehyde were added to a clean, dry test tube. A magnetic stir bar and 0.6 mL of dichloromethane were added to the same test tube, which was then placed in a test tube rack on a stir plate. 10 drops of 50% sodium hydroxide were added to the reaction mixture to deprotonate the benzyltriphenylphosphonium chloride to generate the ylide. The in situ formation of the ylide in this manner led to the attack of the aldehyde. The solution was mixed by drawing up and own with a Pasteur pipette. The mixture was left stirring on the stir plate for 30 minutes. The color change indicated the ylide attack of the aldehyde.

After removing the test tube from the stir plate, 1.5 mL of dichloromethane and 1.5 mL of distilled water were added to the test tube to dilute the reaction with organic solvent as well as water and to form two distinct layers of solution in the test tube which were then separated. Directly following the addition of dichloromethane and water, the two layers of solution were mixed thoroughly using a Pasteur pipette. After allowing the solution to settle and the two distinct layers to separate completely, the bottom layer of the solution was removed using a Pasteur pipette and transferred to a clean, dry test tube. This bottom layer consisted of the dichloromethane and organic soluble solution as water is less dense than dichloromethane and therefore floats above it. A scoopful or magnesium sulfate solid was added to the test tube containing the dichloromethane in order to remove any remaining water. To remove any remaining dichloromethane solution from the water, 1.0 mL of distilled water were added to the reaction test tube and the lower layer was removed using a Pasteur pipette, as before. This solution was added to the test tube containing the dichloromethane and magnesium sulfate.

Using a Pasteur pipette, the dichloromethane was transferred to the bottom of a suction flask. The flask was sealed with a septum and attached to a vacuum aspirator. The flask was gently warmed using a hot plate and swirled constantly to facilitate the removal of dichloromethane. Once all of the dichloromethane solvent had been boiled off, the aspirator was turned off and the suction tube and septum removed. To dissolve the crude product, 5 mL of 1-propanol were added to the suction flask and warmed to dissolve. The warm solution was pipetted down the side of the suction flask to dissolve any crude product clinging to the sides. The solution was transferred to a clean and dry Erlenmeyer flask using a Pasteur pipette. The solution was allowed to cool to room temperature and then cooled further in an ice bath to cause re-crystallization of the product. Once the crystals had formed, they were filtered off using the microscale suction apparatus. The crystals were weighed to determine the yield of the reaction and a melting point taken to assess the purity of the product formed.

The water layer in the initial reaction test tube contained sodium hydroxide, both basic and corrosive, which was disposed of as hazardous waste in the proper waste receptacle. The 1-propanol used for re-crystallization and remaining in the bottom of the filter flask was disposed of as liquid waste. The magnesium sulfate and crystals of trans-9-(2-phenylethenyl) anthracene were disposed of as solid waste in the appropriate waste container.

- The procedure may also be labeled the "methods" section of your lab report.
- This section should be written in **paragraph form** and **ALWAYS in third person passive voice.**
- Your procedure needs to be **clear and precise** enough so that someone reading it could successfully re-create your experiment, so be sure to include how much of each substance was used.
- Make sure to **note any changes to the experimental procedure** from what is outlined in the lab manual. For example, if you heated a solution for twenty minutes instead of the fifteen indicated by the manual, make sure to note this alteration.
- Also include a **discussion of waste disposal** in this section; it is incredibly important that the hazardous chemical materials generated in these lab experiments are disposed of properly.

IV. Data and Observations

-After the test tube was removed from the stir plate, a very thick, gelatinous layer had formed on the bottom of the test tube

-Once the dichloromethane and water were added to the test tube, two very distinct layers formed with the darker layer on the bottom

-5 mL rather than the 3 mL (suggested by the lab manual) of 1-propanol were necessary to dissolve the crude product

-Despite the warmer and increased amount of 1-propanol added, not all of the chunks of crude product dissolved

-Some of the chunks that did not dissolve looked as if they were burnt

-The funnel did not fit tightly into the suction flask and had to be held in place

-Because of the poor fit, the filtration took a long time and some crystals may have been lost in adjusting the funnel to get the suction apparatus to work correctly

-The empty filter funnel weighed 7.771 grams

-The filter funnel and crystals weighed 7.844 grams

-The product generated melted between 131°C and 134°C

- *It is important to include observations so that someone re-creating the experiment can judge whether or not the experiment is progressing as it should. These observations may also indicate where an experiment has gone wrong due to error, so should be* ***brief and objective.***
- *The data collected and recorded in this section will be* ***used to complete the necessary calculations and to draw conclusions*** *on the efficacy of the experiment.*
- *The data may be* ***organized into tables*** *to make it more manageable. In this case, only a few pieces of data are recorded, so a table is unnecessary.*

V. Calculations

-For more detailed calculations, please see attached-

1.a) Using the molar ratios provided by the reaction equation in the lab manual, the limiting reagent was determined to be benzyltriphenylphosphonium chloride. The amount of benzyltriphenylphosphonium chloride used to perform this experiment was enough to generate 5.1 x 10^{-4} mol of trans-9-(2-phenylethenyl) anthracene product, theoretically. Enough 9-anthradlehyde was used to generate 5.83 x 10^{-4} mol of product; therefore benzyltriphenylphosphonium chloride was the limiting reagent.

1.b) Subtracting the weight of the funnel from the weight of the funnel an crystals, the yield of the reaction was found to be 0.073 grams of trans-9-(2-phenylethenyl) anthracene. Converting to mol, this value is 2.607 x 10^{-4} mol of trans-9-(2-phenylethenyl) anthracene.

1.c) Comparing this value to the theoretical yield determined by the stoichiometric ratio of limiting reagent, benzyltriphenylphosphonium chloride, to product, a percent yield of 50.7% was calculated.

2.) Using the melting point apparatus, the melting point of the product synthesized in this laboratory experiment was found to be between 131°C and 134°C. The lab manual reports the melting point of trans-9-(2-phenylethenyl) anthracene as 131° C. The product fits neatly within this range. It therefore can be concluded that the product synthesized by in this experiment was indeed trans-9-(2-phenylethenyl) anthracene with few impurities.

- This section is the bulk of the report, where you prove that you understand the material and the experimental procedures used.
- Some TAs will prefer that you keep your results separate from your discussion and conclusions sections; make sure to consult the guidelines they distribute and separate or combine these sections according to their expectations.
- Your **results should be stated outright and briefly.**
- Your discussion and/or conclusion sections should **include an assessment of the experiment conducted** and should qualify the objectives stated in the first section of your lab report. To assess your experiment, answer the questions: How successful was the experiment? Did you reach all of the experimental goals you wanted to?
- This section should also include a discussion of the errors that may have affected the efficacy of your results and some TAs will also want you to include a discussion of the quality of the experiment and suggest ways in which the experiment could be improved. For example, consider whether or not you think the experiment was sufficient in clarifying lecture material and whether or not you were able to complete it on time.
- Your discussion/conclusion section should **explain your results**. What does it mean, for example, that your percent yield was over 100% ? Why does it matter that the melting point of your product was so far below that of a pure product? How do your results relate to the amount of error encountered in this experiment?
- This section should also include a **description of the theory behind the experiment**. What should have happened under perfect conditions and why? What theorems or formulas is this experiment testing? How do the important steps in the experimental procedure work to prove these theorems? Why are the steps in the experimental procedure performed in a particular order?
- This is where you **tie the experiment together**, so make sure to be very detailed and thorough in your explanations.
- If your TA has assigned **post-lab questions**, you may include them in your report or hand them in as a separate document; ask your TA which they prefer.
- Remember, your TA has to read at least twenty reports per week, so make sure yours is **well-written** and easy to follow and that you have proofread and corrected your errors. Good writing is always appreciated, no matter what the context!

Created by: Rachel Conrad