**The Grignard Reaction: Preparation of Triphenylmethanol**

**Introduction**

The main purpose of this lab is to create and learn the process of making a Grignard reagent. In which later we will use in our own reaction to a carbonyl in a nucleophilic reaction resulting in the creation of triphenylmethanol.

**Reaction**

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**Mechanism**



**Theoretical Yield**

730mg benzophenone/ 182.22 (g/mol) benzophenone = 0.00401 mol benzophenone.  
Since there is a 1:1 ratio between benzophenone and triphenylmethanol,

0.00401 mol triphenylmethanol \* 260.33 (g/mol) triphenylmethanol = 1.043g triphenylmethanol.

**Physical Data:**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Name** | **Molecular weight**  **(g/mol)** | **Boiling point**  **(oC)** | **Melting**  **Point**  **(oC)** | **Density**  **(g/cm3)** | **Solubility (g/L)** | **Safety Hazards** |
| ***Bromobenzene*** | 157.01 | 156.00 | -30.80 | 1.50 | 0.0041 | Irritant |
| ***Phenylmagnesium bromide*** | 181.31 | - | - | 1.14 | Reacts with Water | Volatile, Flammabl |
| ***Benzophenone*** | 182.22 | 305.40 | 48.50 | 1.11 | Organic solvents/ insoluble with water | Harmful |
| ***Magnesium*** | 24.31 | 1091.00 | 650.00 | 1.74 |  | - |
| ***Sulfuric acid*** | 98.08 | 337.00 | 10.00 | 1.84 | Miscible | Corrosive |
| ***Diethyl ether*** | 74.12 | 34.60 | -116.30 | 0.71 | 69 | Irritant, Flammabl |
| ***Triphenylmethanol*** | 260.33 | 160-163.00 | 360-380.00 | 1.199 | - |  |

*Possible unknowns are given on pg.186 of the Experiments Book with MP and Structure.*

*Sources: Handbook for Organic Chemistry,* ***CRC Handbook of Chemistry and Physics*** *(especially Section C: "Physical Constants of Organic Compounds" ), available at the information desk in the Science Library (in Norlin) and in the Organic Chemistry Stockroom.*

***Wastes***

*Aqueous Waste: All aqueous layers acquired through the extraction process, 5 % sulfuric acid, saturated sodium chloride should go into the aqueous waste.*

*Organic Waste: Unused hexanes and ether into organic waste jar.*

*Solid Waste: The reaction product, used filter papers, pipets, melting point tubes, and drying agents into the solid waste bin.*

***Safety Precautions***

*Diethyl ether is extremely flammable. Corrosive: Sulfuric acid. Mild irritants: Benzophenone and Bromobenzene.*

**Procedure**

1. Make sure all glassware is clean and dried, if not dried then add acetone and then diethyl ether before usage.
2. Weigh out 145mg of Magnesium
   1. Place it in a test tube.
3. Measure 0.6mL of bromobenzene into another test tube, mix it with 1mL of anhydrous diethyl ether.
4. Use the pipet, with the mixture, to cover the entire magnesium.
5. Cover the test tube with a small piece of foil
6. Place the reaction test tube into the sonicator and turn it on.
   1. Check after 2 minutes to see whether the liquid is brown.
   2. Continue until the liquid is turned brown.
7. Add 1mL of ether to the bromobenzene and ether mix in the test tube.
   1. Pipet over a few drops to the reaction tube.
   2. Continue adding bromobenzene/ther so that the reaction keeps on proceeding.
   3. Dissolve some benzophenone in ether for the next part.
8. Make sure the reaction is continuing after adding all the bromobenzene/ether.
   1. If it drops below the 2mL line, add diethyl ether to the tube.
9. When the reaction stopped bubbling and the magnesium does not seem to be present, place the tube into the sonicator for 2 minutes.
10. Weigh 730mg of benzophenone in 4ml of anhydrous diethyl ether in a flask and the stir bar in it.
    1. Add an ice bath over the stir motor
11. Stir the solution with minimum speed
12. Add the Grignard reagent through the pipet drop by drop.
13. Chill 5mL of 5% Sulfuric Acid, simultaneously remove the reaction from the ice bath once the Grignard reagent has been completely added.
    1. You should see crystals being formed
    2. If you do not see crystals, heat the reaction on a hotplate for several minutes.
14. In order for the reaction to be quenched, add the chilled 5mL of Sulfuric acid to the reaction flask (whiles its still stirring).
15. Agitate the flask to make sure that there are no undissolved solids.
16. Transfer it to the separatory funnel, add a little bit more ether.
17. Shake the funnel (mixing it) until the layers have been separated.
18. Drain the bottom aqueous layer.
19. Wash the other layer with 3mL of Sulfuric Acid.
20. Drain the bottom layer again.
21. Wash the ether layer with 3mL saturated Sodium Chloride.
22. Collect the ether solution and dry it with sodium sulfate.
23. Decant the solution into an Erlenmeyer Flask.
24. Add 12mL of hexanes to the ether solution.
    1. Place it on a hotplate, heat it until the solution froths.
25. As soon as the crystals are formed, let it cool down to room temperature.
26. Weigh out the crystals
27. Obtain a melting point of crude crystals.
28. Recrystallize 100mg from hexanes if the crude MP indicates impure crystals.