

Triphenylmethanol

Johnson, Chad Philip; T/Th Lab, 8:00am

Submitted March 29th, 2012

Introduction

The preparation of a Grignard reagent is an important laboratory technique and is a required method in the synthesis of numerous organic molecules. Discovered by the French chemist Victor Grignard, it involves the formation of a temporal organometallic compound which contains a carbon-metal bond involving a carbon atom, magnesium metal and a halogen anion (McMurry 345). Many different kinds of halides can be used in these reactions, including alkyl and vinylic chains, and aromatic rings.

In this experiment I synthesized triphenylmethanol over the course of three lab periods through separate, stepwise reactions—each of which was followed by careful separation of impurities. The Grignard reagent phenylmagnesium bromide was first prepared from bromobenzene and pure magnesium solid (see Figure 1) and was then reacted with the single ester functional group found on

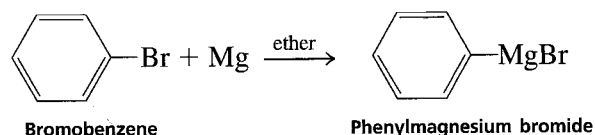


Figure 1. Preparation of the Grignard reagent phenylmagnesium bromide (Pavia 303).

benzophenone to form triphenylmethanol (see Figure 2). This product is a tertiary alcohol with three benzene rings, all of which are attached to a central carbon atom.

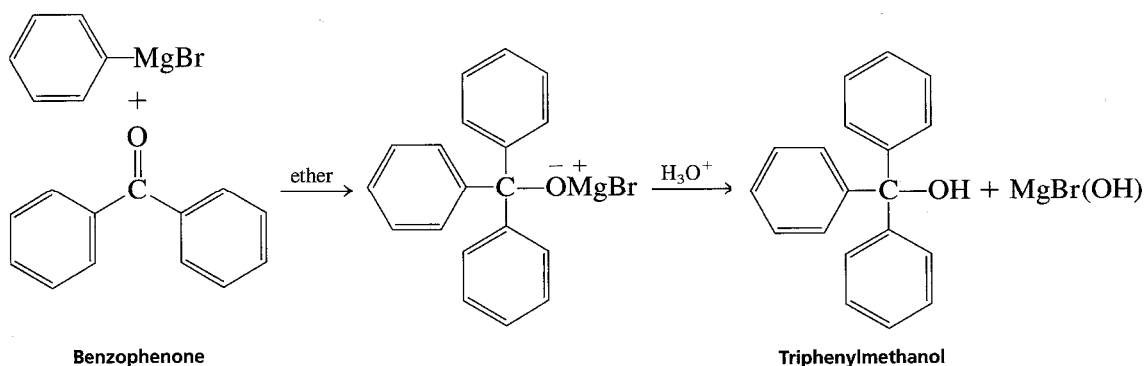


Figure 2. Reaction of benzophenone with phenylmagnesium bromide (Pavia 303).

There are no common uses or commercial applications for triphenylmethanol. Its value seems to be solely in the sphere of academia where the necessary steps for synthesis serve as an effective way to practice necessary lab procedures in preparing Grignard reagents.

Experimental Procedure

Assembling the apparatus

The first portion of the experiment to prepare the Grignard reagent required assembly of a special apparatus made up of the following components: glass separatory funnel with air shaft, claisen adapter, 100mL round bottom flask, condenser, drying tube filled with calcium chloride (CaCl_2), thermometer adapter, and stirring hot plate (see Figure 3). I combined 0.506g of magnesium (Mg) turnings, one very small flake of iodine and one large magnetic stirring bar into the round bottom flask before attaching it to the apparatus. Because Grignard reagents react so readily with H^+ ions, all of the glassware was thoroughly flame dried with a Bunsen burner before introducing additional reactants.

Preparation of the Grignard reagent

To start the reaction I poured a mixture of 2.381g (1.8mL) of bromobenzene and 10.0mL of anhydrous ether into the separatory funnel. Half of this amount was quickly added to the round bottom flask, and stirring and light heat was applied from the plate; this produced a reddish brown mixture.

An additional 7.0mL of anhydrous ether was poured into the funnel and its contents were slowly added to the flask over the course of about five minutes. During this time the magnesium metal began reacting with the solvent and small bubbles evolved from the surfaces of each piece. The mixture also changed colors many times from brown to dark grey to light brown and finally settled on dark brown. At this point enough heat was being created by the exergonic

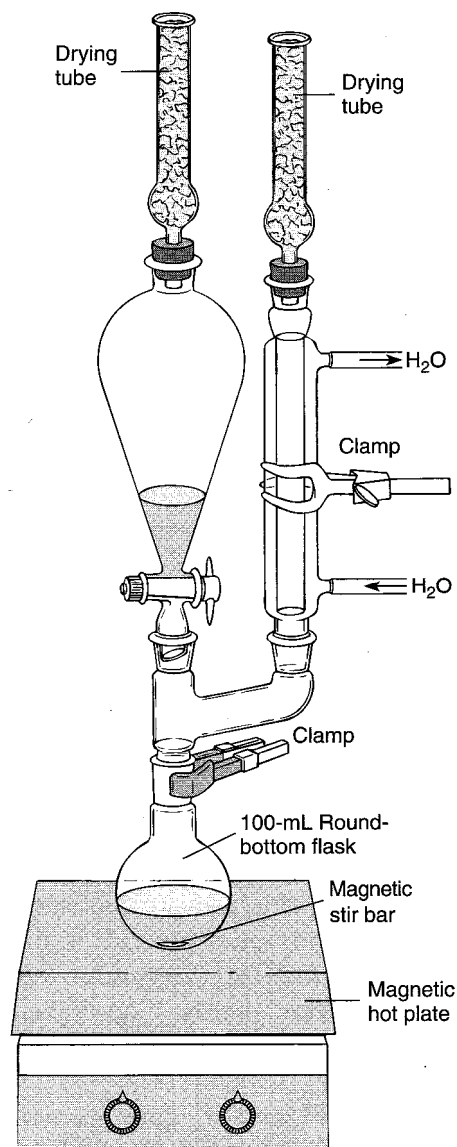


Figure 3. Apparatus (Pavia 307).

reaction to sustain the overall process without any external heat being provided from the plate. A final amount of 2mL anhydrous ether was poured into the funnel for rinsing and quickly added to the flask. I next removed the separatory funnel and stoppered the open joint of the Claisen adapter. Heat from the plate was reapplied as the reaction slowed and the process was allowed to finish over the course of about 15 minutes, with the slowly evaporating anhydrous ether being replaced in 1mL increments.

Use of Grignard reagent

I combined 2.409g bromobenzene and 10mL of anhydrous ether in the separatory funnel and then reattached it to the apparatus. This new mixture was added to the round bottom flask which caused a dramatic change in the color to a deep reddish-pink color. The external heat from the plate was again suspended and another treatment of anhydrous ether in the amount of 5mL was used to rinse the funnel and be combined with contents of the flask. Stirring was turned up to the highest setting and the mixture was allowed to cool to room temperature and crystallize. During this time the adduct slowly faded from its pink color and became cloudy white.

Hydrolysis and formation of triphenylmethanol

After crystallization 7mL of 6M hydrochloric acid was added by drops to this solid in the 100mL round bottom flask. This caused it to break up and dissolve, forming a light brown and opaque liquid. During this process a side reaction also occurred where unreacted magnesium metal began to bubble again forming hydrogen gas ($2\text{Mg} + 2\text{HCl} \rightarrow \text{H}_2 + 2\text{MgCl}$). After enough HCl was added for the mixture to become acidic I found that two phases had formed: a clear yellow-brown colored top and a clear and colorless bottom. The two phases were then separated with the top ether layer being retained and dried with 2.0g of anhydrous sodium sulfate. The ether liquid was removed by using a rotary evaporator and dried with a high vacuum system. This resulted in 3.188g of crude product, a clumpy orange colored solid.

Removal of biphenyl contaminant and crystallization

I next poured 10mL of petroleum ether into the round bottom flask which produced a pink hued liquid. The flask was quickly transferred to a lukewarm water bath which caused a small amount of the petroleum ether to evaporate. During this time another crude solid formed—a white precipitate—which was retrieved through vacuum filtration using a Büchner funnel. The weight of this solid was 2.174g.

Finally, to purify triphenylmethanol, crystallization techniques were used with isopropyl alcohol solvent. The crystals were again retrieved through vacuum filtration and a Büchner funnel and, after transferring to a round bottom flask, dried in a high vacuum system.

Test for impurities

I measured the melting point and compared it to the literature value. The HNMR spectrum was analyzed by preparing a sample of product dissolved in deuteriochloroform. Also, IR spectroscopy was used to make additional observations about the products by using a KBr pellet.

Results

Yield, melting point and appearance

Table 1. Data for the synthesis of triphenylmethanol using a Grignard reagent

Product	Yield (g)	Yield (%)	Experimental Melting Point	Literature Melting Point	Appearance
Triphenylmethanol (Pure)	1.214g	35.05%	161.8°C – 162.4°C	162°C [†]	White solid

[†] (Pavia 311)

HNMR and IR Data Summary

HNMR results are entirely consistent with the expected results and could not be more telling: 15 hydrogens with identical signals in the 7.2ppm range with a lone signal in the 2.9ppm range (see Figure 4). IR results were also as expected with two areas of interest: the C—H signals for an aromatic ring at 3024.1cm⁻¹ and 3059.6cm⁻¹, and the —OH signal at 3465.3cm⁻¹ (see Figure 5).

Discussion and Conclusion

Though lengthy and complicated, the experiment was finished successfully and my results were quite agreeable. The melting point matched almost exactly with the literature value. All procedures were performed without incident or unforeseen difficulties. The only aspect I feel could be improved is the final yield: there were many intermediate steps and better handling techniques combined with more precise instruments would have prevented at least some of the nearly 65% loss of product.

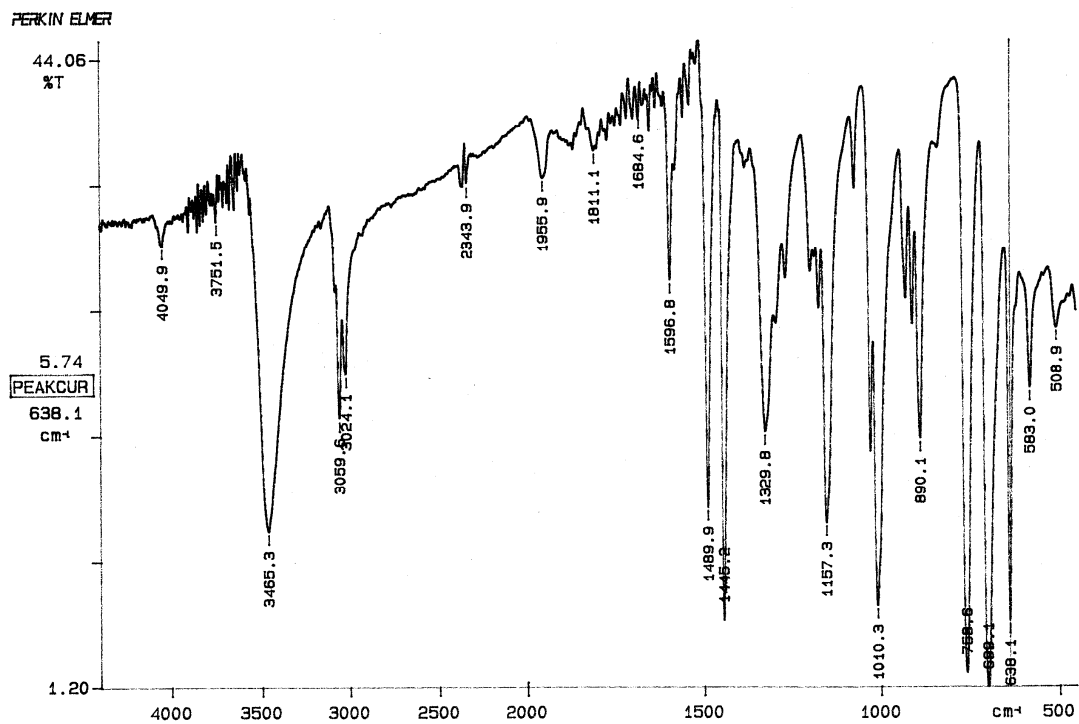


Figure 4. IR spectrum for triphenylmethanol.

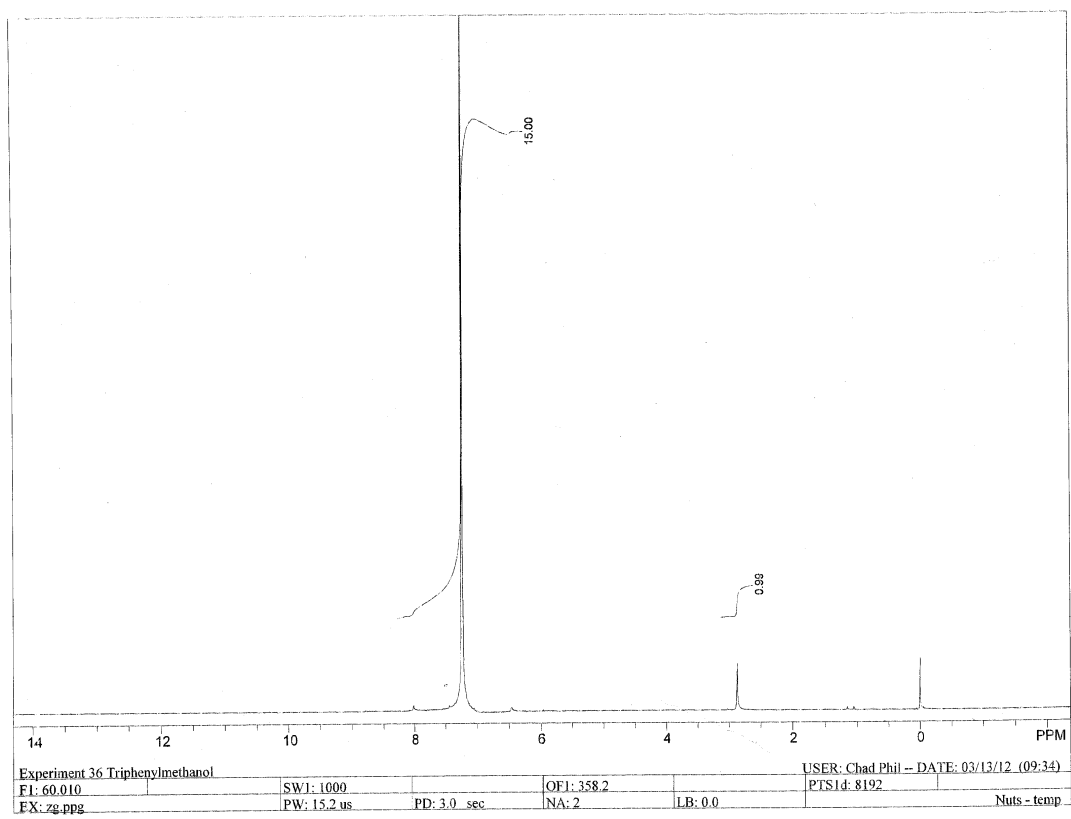


Figure 5. ¹H NMR spectrum for triphenylmethanol.

References

McMurry, J. *Organic Chemistry*. 7th ed. Belmont, CA: Brookes/Cole, 2008.

Pavia, D.L., Lampman, et al. *Introduction to Organic Laboratory Techniques, A Small Scale Approach*. 2nd ed. Belmont, CA: Brookes/Cole, 2005.