

# The wittig reaction – lab report essay sample

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The purpose of this experiment is to synthesize trans-9-(2-phenylethenyl)anthracene from benzyltriphenylphosphonium chloride and 9-anthraldehyde through the reaction mechanism recognized as the Wittig Reaction. The Wittig Reaction allows the chemist to synthesize phosphoranes in the lab with relative ease. A more recent and inexpensive version of the reaction is the Wittig-Horner reaction (1).

#### ABSTRACT

Georg Wittig was a German chemist and Nobel Prize winner in 1979 for the Wittig reaction (1). He was born in Berlin, on June 16, 1897, and died August 26, 1987 (1). Wittig discovered the route to alkenes through ylide molecules (1). Wittig was educated originally at Tübingen; Wittig spent periods at Braunschweig, Freiburg, back to Tübingen again before taking up the post as director of the organic chemistry department at Heidelberg (1). Wittig became an emeritus professor in 1967, where he remained until the end of his notable career (1). In 1967, he won the Otto Hahn Prize, and in 1979, he and Herbert C. Brown were jointly awarded the Nobel Prize for their development of the use of boron and phosphorus-containing compounds for important reagents in organic synthesis (1).

The foundation of the Wittig reaction is not complex. Phosphorus is a second row element - in-group 5 - like nitrogen, but unlike nitrogen, has the ability to expand its valency from 3 to 4, 5, or even 6 (1). The stable 5 valencies are met in compounds like phosphoric acid and PCl<sub>5</sub> (1). Wittig discovered that phosphines, which are the phosphorus equivalent of amines, easily form phosphonium salts with alkyl halides and that these salts readily lose HX

with strong base. This product is called an ylide or a phosphorane. The ylide is a polar molecule with a carbanionic carbon.

Figure 1. How to make a phosphorus ylid (1)

In relation to stereochemistry, such as cis/trans, or E/Z, is still not fully understood in the reaction (2). Triphenylphosphines tend normally to give the Z- (Cis) isomer. Trialkylphosphines or the presence of groups that stabilize the ylide tends to give E- (Trans) geometry (2). The Wittig reaction can tolerate all types of functionality. The trans-isomer is more stable and the preferred product or the only product in certain reactions due in part to the reduction of steric hindrance and eliminating orbital overlap.

Figure 2. Mechanism for phosphorus ylids denoting cis/trans-isomers (1)

The Wittig is basically used to convert a carbonyl group,  $C=O$ , into an alkenes,  $C=C$ . A phosphorus ylid is formed and obtained by the treatment of a phosphonium salt with a strong base. These ylids are very stable due to resonance and highly reactive (2). Phosphorus ylids are generally not isolatable and are treated with carbonyl compounds.

Once the ylid has been generated, it is added to a carbonyl group to give the intermediate known as betaine and is followed by the elimination of the phosphine oxide. This elimination has been calculated to occur after the formation of a four-membered ring known as an oxaphosphetane. Based on the latest laboratory evidence, such as x-ray diffraction, the Wittig reaction may proceed directly through the oxaphosphetane intermediate (2).

### Figure 3. How to make alkenes using the Wittig reaction (1)

This reaction goes so well due in part to the formation of the immensely strong P-O bond. The strong bases used to make the ylid can be the sodium hydride (NaH), and sodium amide (NaNH<sub>2</sub>). If we have a more acidic H to abstract a weaker base such as NaOEt or even Na could be used in the reaction.

Another variant on the Wittig reaction that is much easier to control and cheaper to carry out is the Wittig-Horner reaction (1). This reaction uses a phosphate ester instead of a phosphine, manufacturing a more reactive ylid.

### Figure 4. Mechanism for Wittig-Horner reaction (1)

In the Wittig reaction, an organic phosphorus compound with a formal double bond between phosphorus and carbon is reacted with a carbonyl compound. The oxygen of the carbonyl compound is exchanged for carbon, forming a product known as an olefin (2). The method for making olefins has opened up new possibilities, especially for the synthesis of biologically active substances containing carbon-to-carbon double bonds. For example, vitamins such as vitamin A are synthesized industrially using the Wittig reaction.

## DATA & RESULTS

The Wittig reaction-synthesis of trans-9-(2-phenylethenyl) anthracene yielded .067 g of crystals and a 47 % yield. Under Wittig-Horner parameters, we could hypothesize achieving a yield in the 65 % range. After 30 minutes

of reaction time, the solution was removed and 1.5 ml of water and dichloromethane were added to the vial. The organic (bottom) layer was extracted and placed in a test tube. Calcium chloride pellets were added to dry the dichloromethane layers and the solvent was removed under vacuum. After approximately 15 minutes a solid had formed and 3 ml of 1-propanol were added and heated on a hot plate in the hood. The solution was allowed to cool to room temperature then placed in ice to reform crystals. The crystals were vacuum filtered using the Hirsch funnel apparatus and washed with cold dichloromethane. The crystals were removed and were yellow-gold in color and shiny, somewhat metallic in nature. A melting point was obtained and fell in the range of 126-128 as compared to the literature value of 130-132 (2). The melting point could have been lowered due in part to some residual solvent.

## DISCUSSION

The basis of conducting this Wittig reaction was to use benzyltriphenylphosphonium chloride with 50% NaOH, Wittig reagent (ylid) and 9-anthraldehyde to yield the trans-9-(2-phenylethenyl) anthracene. This molecule has a melting point of 130-132 C. Upon extraction and purification of crystals produced by the reaction, a melting point was obtained along with the mass of the crystals. The reaction yielded 0.067 grams of crystals and a melting point of 126-128 was obtained as compared to the literature value of 130-132 (2). One can only conclude that the melting point was off due to either some residual moisture or additional solvent present on the crystals. It was evident that the product did form or the

melting point would have not been as close as it was. The additional spectrographic information from IR, MS or NMR was not needed due to the melting point achieved. The crystals were gold or silver-yellow metallic in nature. After removing from the vacuum apparatus, the crystals fell directly out of the filter paper and held their form, a very interesting structure. A percent yield has been calculated below for reference.

Figure 5. Calculation of yield

#### REFERENCE

1. The Wittig Reaction. Available at <http://www.sunderland.ac.uk/~hs0bcl/org8.htm>. Downloaded 2 March 2004.
2. Salvatore RN, Jung KW. experimental organic chemistry: A synthetic & mechanistic perspective. 222 Berkley St. Boston: Houghton Mifflin; 1999. 102p.