

NOTES ON EXPERIMENT 8

Wittig Reagents provide a valuable method for converting a C=O bond to a C=C bond (synthetically equivalent to the reverse of ozonolysis). Although an equivalent conversion can be achieved using $\text{RMgX} + \text{C=O}$ followed by dehydration, this often gives mixtures of products; only the Wittig provides a single alkene with the double bond in a known place; however it may give E/Z isomer mixtures in some cases.

A Wittig reagent is conveniently generated by deprotonation of a phosphonium salt (made from $\text{Ph}_3\text{P} + \text{RX}$) using a strong base such as NaOH; the C-H group is rendered highly acidic by the nearby positive charge (on phosphorus). The reagent has two resonance forms (shown below), demonstrating the types of bonding available to a third row non-metal such as phosphorus. The carbon is nucleophilic (from the partial negative charge), and it attacks the electrophilic carbon of the C=O compound. The resultant intermediates decompose finally to give the alkene and triphenylphosphine oxide ($\text{Ph}_3\text{P=O}$). The driving force for the reaction is the formation of the strong P=O bond.

Experimental:

- Try to get the reaction started first thing, as there is a 30 min. stir time.
- If you have problems with separating the two layers (there is often a fine yellow emulsion of the two layers that looks like colored wool) you can try adding a little water and/or CH_2Cl_2 . If this fails, filter the solution then try separating again.
- When removing the CH_2Cl_2 , warm the flask with a beaker of warm water.
- Take the mass & melting point of your (dry) crystals after recrystallization.
- We will also look at an ethanol soln. of the product under short & long wave UV.

Assigned problems:

1. How might the same product be prepared using a different Wittig reagent, made from 9-chloromethylantracene?
2. Why is rapid stirring necessary in this procedure, more than is often the case?
3. For making a simple Wittig reagent (e.g. $\text{Ph}_3\text{P=CH}_2$), a stronger base such as *n*-butyllithium is needed. Why is a weaker base (NaOH) satisfactory in our experiment?
4. Deduce the structure of compound Z (formula C_9H_{10}) from the NMR (^1H , ^{13}C) spectra:

