Background and Case Study Introduction

Green Wittig Reaction for the Undergraduate Organic Chemistry Lab

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Background

The Wittig reaction has been used as a good general method for preparing alkenes from aldehydes or ketones. A phosphine ylide is formed *in situ* by treating the stable triphenylphosphonium chloride with a base that can deprotonate the carbon bonded to phosphorus.

The ylide will then react with aromatic aldehydes to form a 4 membered ring – oxaphosphetane- intermediate. This intermediate then eliminates triphenylphosphine oxide to give an alkene.

Both the E and Z forms of the alkene will form in this reaction. *If you only take into account product stability, which one would you expect to be the major product?*

Green chemistry techniques and processes can be applied to a Wittig reaction to make it safer for the environment and efficient for Organic Chemistry labs. Various changes made to the traditional procedure include using less harmful chemicals, lowering stir times, no reflux, along with replacing the organic solvent with water. The products can be analyzed via ^{1}H Nuclear Magnetic Resonance Spectroscopy (^{1}H NMR), Infrared Spectroscopy (IR), Thin Layer Chromatography (TLC), and Gas Chromatography—Mass Spectrometry (GCMS). Most products have sufficient yields to be acceptable for both analysis and identification in an Undergraduate Organic Chemistry lab setting.

Pre-lab Case-Study:
For this case study, you will play the part of chemists working for a company that produces stilbene derivatives. This case study will expose you to the topic of green chemical synthesis as well as helping you understand some of the value of green chemistry in industry.

Your lab has been using a traditional Wittig reaction to make stilbene derivatives for several years. Your lab manager tells you that she has found a recently published method that could improve on your product production time and potential cost to produce. She would like you to submit an analysis of the two processes to help her decide whether or not to change your production plan.

Traditional Wittig: The aldehyde and benzyltriphenylphosphine are stirred for 4 hours at room temperature in N,N-dimethylformamide with n-butyllithium as the base. The mixture is then heated to reflux overnight. Upon completion, the product is extracted with diethyl ether and recrystallized from propanol.

New Method overview: The aldehyde is mixed with the benzyltriphenylphosphonium chloride. Sodium hydroxide is added and the suspension is stirred for 30 minutes. The product is filtered, washed with water and recrystallized from ethanol.

The following are some questions you should address to help your manager understand the potential effects of this new green methodology.

What are the 12 principles of green chemistry? (if you are not familiar with them, they can be found on the American Chemical Society's ACE Green Chemistry Institute website) (accessed April 30, 2013) *http://portal.acs.org/portal/acs/corg/content?_nfpb=true&_pageLabel=PP_ARTICLEMAIN&node_id=1415&content_id=WPCP_007504&use_sec=true&sec_url_var=region1&__uuid=7f174e78-d85f-486f-b801-e17b77cc6df9*
Prevention, Atom Economy, Less Hazardous Chemical Synthesis, Designing Safer Chemicals, Safer Solvents, Design for Energy Efficiency, Use of Renewable Feedstocks, Reduce Derivatives, Catalysis, Design for Degradation, Real-time Analysis, Inherently Safer Chemistry for Accident Prevention

Identify 3 of the principles that are present in this experiment. Give a short explanation of how they are being applied.
Prevention, Less Hazardous Chemical Synthesis, Safer Solvents, Design for Energy Efficiency, Use of Renewable Feedstocks, Inherently Safer Chemistry for Accident Prevention