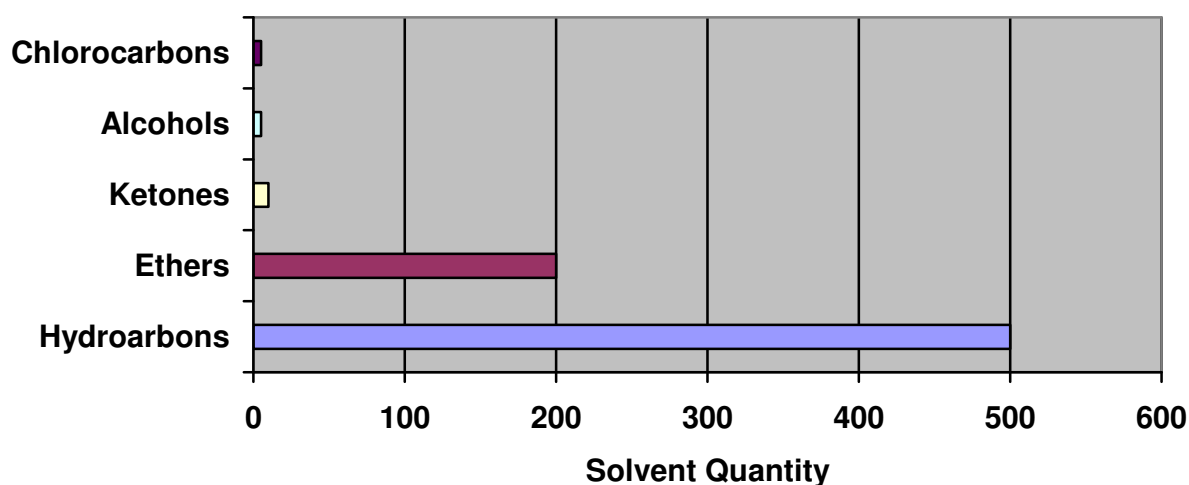


Studies on Removal of Triphenylphosphine Oxide

The wittig olefination results in a very stable triphenylphosphine oxide (TPPO) **6c** as a byproduct, which is the driving force for the reaction. Separation of **6c** from the reaction mass is required to improve quality of desired olefin compound **6**. The control strategy we adopted was to interplay between solubility of product and byproduct and to treat the reaction mass in a solvent wherein **6b** is practically insoluble and product is soluble due to its liquid nature.

Solubility of **6c** was examined in various solvents isothermally. It was observed that **6c** was relatively more soluble in chloroalkanes than alcohols than ketones. Solubility studies in ethers and both alicyclic and aliphatic hydrocarbons revealed that **6c** was practically insoluble in hydrocarbons than ethers. The solubility details of **6c** in various classes of solvents can be represented in a graphical mode as follows.

Measured solubility of 6c in various classes of solvents



Based on the above solubility data, **6c** has less solubility in Ethers and Hydrocarbons. Further, between both the classes of solvents, Hydrocarbons were found to be preferable for **6c** removal. Specifically, among Hydrocarbons group n- heptane was selected for **6c** removal. Further extensive studies suggested treatment of reaction mass with 4 volumes of n-heptane between 25 and 35⁰C for 1-1 ½ hrs was the choice of solvent and process parameters, which was based on product solubility while byproduct was insoluble. So, based on the experimental results the by-product can be isolated at ambient temperatures.