UNIVERSITI TEKNOLOGI MARA

# DICYCLOHEXYLPHENYLPHOSPHINE IN HECK COUPLING

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**Dissertation submitted in partial fulfillment of the requirement for the Bachelor Pharmacy (Honours)**

**Faculty of Pharmacy**

**November 2008**

**Acknowledgements**

*In the name of Allah, the Most Compassionate, the Most Merciful*

It is only by the grace of Allah, and prayers softly whispered by those near and far, and the helping hand extended by generous friends, course mates, and my family as well as the continuous understanding and immense support from my supervisor, Dr. Ibtisam Abdul Wahab, and other lecturers at the Faculty of Pharmacy, UiTM who directly and indirectly have taught me the invaluable learning during this PHM555 Course. Thank you. Such understanding, help and consideration mean a lot to me especially after my long journey of undergoing undergraduate study. Thank you for helping me become a better person. May Allah bless us all with knowledge and good judgement.

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## Abstract

This study investigates the efficiency of dicyclohexylphenylphosphine **1** among the ligands used in the Heck coupling reaction. The method used includes the protection of iodophenol **3** by acetylation prior to the Heck reaction. 1.0 g of 4-iodoacetoxybenzene **5** (0.0038 mole) was reacted with 0.6 ml of 3, 4-dimethoxystyrene **6** (0.0038 mole) in the presence of 0.0673 g of palladium (II) dichloride (0.00038 mole), 0.2085 g of dicyclohexylphenylphosphine **1** (0.00076 mole), 0.6455 g of mercury nitrate (0.0038 mole), and 0.4848 g of potassium acetate (0.00494 mole). The reaction was left stirred under Nitrogen flow for 24 hours. The product obtained, 3, 4-dimethoxy-12-acetoxystilbene **7** (0.0038 mole) was extracted using liquid-liquid chromatography and was purified by using preparative TLC with 80 hexane: 20 ethyl acetate solvent system. Three bands were discovered: band 1 (0.0260 g), band 2 (0.0143 g) and band 3 (0.0029 g). They were then dissolved in chloroform-D and put into NMR tubes to be analyzed by using the $^{1}$H NMR spectrometer. The $^{1}$H NMR results show that the first band is best to represent 3, 4-dimethoxy-12-acetoxystilbene **7** (0.0038 mole) with the percentage yield of 2.3%. Another study that investigated the efficacy of cyclohexyldiphenylphosphine **8** as a ligand in Heck coupling produced 39.83% yield of the same product. Thus, dicyclohexylphenylphosphine **1** can still be used in Heck coupling reaction, even though it does not perform satisfactorily. However, the low yield of 3, 4-dimethoxy-12-acetoxystilbene **7** might be due to the short time spent for the reactants to optimize their coupling reaction (1 day), compared with the study on cyclohexyldiphenylphosphine **8** where the reaction was left for 7 days. Therefore, increasing the reaction time to about 7 days may increase the percentage yield of the 3, 4-dimethoxy-12-acetoxystilbene **7**.

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

The palladium-catalyzed and alkenylation of olefins was first discovered independently by Mizoroki *et al.* in 1971 in Japan and Heck and Nolley in 1972 in the United States (Schmidt, A. F. *et al.* 2008). Generally, it is referred as Heck Coupling and it has become the most frequently applied for the carbon-carbon bond formation.

Generally, the Heck reaction is catalyzed by palladium species generated by palladium(0) compounds such as tetrakis(triphenylphosphine)-palladium [$Pd(PPh_3)_4$] (Heck, 1982) and tri(1-naphtyl)phosphine (Shaw, B. L. *et al.* 1998). The Heck reaction is attractive from the synthetic point of view because of high selectivity and mild reaction condition with low toxicity and low cost of reagents.

Heck reaction is widely used in the production of hydrocarbons, polymers, pharmaceutical dyes, and other natural products (Beletskaya and Cheprakov, 2000; Whitcombe, N. J. *et al.* 2001). Our interest in this type of Heck-coupling reactions between 4-iodoacetoxybenzene **4** and 3, 4-dimethoxystyrene **5** arose in the context