THE CATALYTIC ACTIVITY OF PALLADIUM(II)-1,3- Diaryl-4-nitrophenyl methyl]-1-benzimidazolium bromide complexes in Heck reaction

NUR ATIKAH SYAHUDAH BINTI ROSLI

Final Year Project Report Submitted in Partial Fulfilment of the Requirements for the Degree of Bachelor of Science (Hons.) Chemistry in the Faculty of Applied Sciences Universiti Teknologi MARA

JULY 2017
This Final Year Project Report entitled "The Catalytic Activity of Palladium(II)-1,3-Bis[(4-nitrophenyl)methyl]-1-Benzimidazolium Bromide Complexes in Heck Reaction" was submitted by Nur Atikah Syahidah binti Rosli, in partial fulfillment of the requirements for the Degree of Bachelor of Science (Hons.) Chemistry, in the Faculty of Applied Sciences, and was approved by

Dr Nur Rahimah binti Said
Supervisor
B. Sc. (Hons.) Chemistry
Faculty of Applied Sciences
Universiti Teknologi MARA
72000 Kuala Pilah
Negeri Sembilan

Nurul Huda binti Abdul Halim
Project Coordinator
B. Sc. (Hons.) Chemistry
Faculty of Applied Sciences
Universiti Teknologi MARA
72000 Kuala Pilah
Negeri Sembilan

Mazni binti Musa
Head of Programme
B. Sc. (Hons.) Chemistry
Faculty of Applied Sciences
Universiti Teknologi MARA
72000 Kuala Pilah
Negeri Sembilan

Date: ________________
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>ACKNOWLEDGEMENTS</th>
<th>iii</th>
</tr>
</thead>
<tbody>
<tr>
<td>TABLE OF CONTENTS</td>
<td>iv</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>vi</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>vii</td>
</tr>
<tr>
<td>LIST OF ABBREVIATIONS</td>
<td>viii</td>
</tr>
<tr>
<td>ABSTRACT</td>
<td>ix</td>
</tr>
<tr>
<td>ABSTRAK</td>
<td>x</td>
</tr>
</tbody>
</table>

## CHAPTER 1 INTRODUCTION
1.1 Background of Study 1
1.2 Problem Statement 2
1.3 Significant of Study 3
1.4 Objectives 4

## CHAPTER 2 LITERATURE REVIEW
2.1 Heck Reaction 5
2.2 N-Heterocyclic Carbenes 7
2.3 Metal Complexes in Organic Synthesis 8
   2.3.1 Palladium Catalyzed C-C Coupling Reaction 8
2.4 Characterization Method
   2.4.1 Fourier Transform Infrared Spectroscopy (FTIR) 9
   2.4.2 Nuclear Magnetic Resonance (NMR) Spectroscopy 9
   2.4.3 Gas Chromatography (GC) 10

## CHAPTER 3 METHODOLOGY
3.1 Materials 11
3.2 Synthesis of Catalyst 12
   3.2.1 Preparation of 1,3-bis[(4-nitrophenyl)methyl]-1-benzimidazolium bromide 13
   3.2.2 Synthesis of Palladium(II)-1,3-bis[(4-nitrophenyl)methyl]-1-benzimidazolium bromide 13
3.3 Catalytic Testing
   3.3.1 General Procedure of Heck Reaction 14
   3.3.2 Effect of Catalyst Loading 15
   3.3.3 Effect of Time 15

## CHAPTER 4 RESULTS AND DISCUSSION
4.1 Characterization of 1,3-bis[(4-nitrophenyl)methyl]-1-benzimidazolium bromide 16
ABSTRACT

THE CATALYTIC ACTIVITY OF PALLADIUM(II)-1,3-BIS[4-NITROPHENYL]METHYL]-1-BENZIMIDAZOLIUM BROMIDE COMPLEXES IN HECK REACTION

N-heterocyclic carbene ligand is used to synthesize palladium(II) complex which involves in Heck reaction as catalyst. In this study, the 1,3-bis[(4-nitrophenyl)methyl]-1-benzimidazolium bromide which is one of N-heterocyclic carbene (NHC) ligand had been successfully synthesized with reaction between benzimidazole and 1,4-nitrobenzyl bromide. The percentage yield of the ligand and complex had been calculated which is 84.21 % and 17.05% respectively. The compound of 1,3-bis[(4-nitrophenyl)methyl]-1-benzimidazolium bromide and palladium(II)-1,3-bis[(4-nitrophenyl)methyl]-1-benzimidazolium bromide complex were characterized by using Fourier Transfer Infrared (FTIR) and Nuclear Magnetic Resonance (NMR). The palladium(II)-1,3-bis[(4-nitrophenyl)methyl]-1-benzimidazolium bromide have been used as homogeneous catalyst which improves the catalytic activity in Heck reaction. The catalytic activity of complex was evaluated in Heck reaction. In Heck reaction, the synthesized complex was tested in the reaction between 1-bromo-4-nitrobenzene and styrene to produce 3-(4-nitrophenyl)-benzene. Besides, by using sodium carbonate, Na₂CO₃ as a base, the effect of the catalyst loading had been studied by changing the percentage of catalyst loading. Also, the reaction that had been monitored every 15 minutes which until 60 minutes is to study the effect of time for the production of product obtained from Gas Chromatography (GC) analysis. Therefore, the optimum condition for catalytic testing is 1.0 mmol%, 46.55 TON and 46.55% conversion rate at 45th minutes.