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# Aluminum Chloride–Catalyzed C-Alkylation of Pyrrole and Indole with Chalcone and Bis-Chalcone Derivatives

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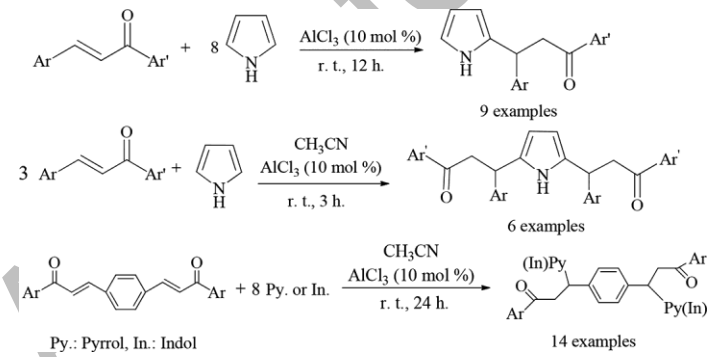
## Abstract

The $AlCl_3$-catalyzed alkylation of pyrrole (**2**) with chalcone (**1a-i**) at a ratio of 8:1 in the presence of 10 mol % $AlCl_3$ gave the solely 2-alkyl pyrroles (**3a-i**) at room temperature for 12 hours in good yields. The same reaction was performed with pyrrole (**2**) and chalcone at a ratio of 1:3 in $CH_3CN$ at r.t. for 3 h. to achieve the only 2,5-dialkyl pyrroles (**4a-f**). In addition, the reaction of the pyrrole (**2**) and indole (**7**) on 1.4-phenylene bis-chalcones (5**a-g**) at the ratio of 8:1 at r.t. for 24 h. gave the double-addition products **6a-g** and **8a-g** in high yields, respectively. The structure of the products was confirmed by $^1H$ and $^{13}C$ NMR spectroscopy and the elemental analysis.




**KEYWORDS:** Aluminum (III) chloride, Michael addition, Pyrrole, Indole, Chalcone, Bis-chalcone

## 1. INTRODUCTION

Pyrrole, indole, and their C-alkylated derivatives are important building blocks in respect to many biologically active compounds such as chlorophyll, porphyrin, hemoglobin, Vitamin B12, indigo, and bile pigment.[1,2] The alkylation of pyrrole and indole have thus been attracting much attention in recent years.[3,4] The Michael addition is one of the most useful reactions in the alkylation of pyrrole and indole.[5,6] This reaction is traditionally catalyzed by either strong bases or acids. Recently, a variety of Lewis acids have been found to catalyze this reaction and have been used for the alkylation of pyrrole with □,□-unsaturated ketones. These acids are $InCl_3$,[7,8] $InBr_3$,[9] $BiCl_3$,[10] $Bi(NO_3)_3$,[11,12] Bi(OTf)[3,13] $Yb(OTf)_3$,[14] $CuBr_2$,[15,16] $GaI_3$,[17] $SmI_3$,[18] and $HfCl_4$.[19] In the alkylation of pyrrole with these catalyses, the 2- and 2,5-dialkylated products have generally been obtained as a mixture. In addition, a number of these procedures have had some disadvantages such as a longer reaction time, strong acidic conditions, the use of an excessive expensive catalyst, and harsh reaction conditions.

Although several methods have been developed for the indirect synthesis of 2-alkyl pyrroles, in the literature there are few methods for the direct synthesis of 2-alkyl pyrrole.[4,20] Thus, developing a useful method for the direct synthesis of 2-alkyl pyrroles still remains a challenge for the synthetic chemist.

Aluminum chloride was widely used as a powerful and effective catalyst for various organic transformations.[21] For example, $AlCl_3$ has been used as a catalyst for Michael addition of indoles and pyrroles to maleimides.[22] However, there has been no report

related to $AlCl_3$ when used as a catalyst in addition to pyrrole and indole with chalcones. In this paper, an efficient method was presented for the alkylation of pyrrole and indole by chalcone and 1.4-phenylene bis-chalcones using aluminum chloride.

## 2. RESULTS AND DISCUSSION

The general synthetic strategy employed to prepare the chalcone and bis-chalcone derivatives were based on the Claisen-Schmidt condensation, which was previously reported.[23,24] The chalcone derivatives were synthesized by adding acetophenone derivatives to the benzaldehyde derivatives at the basic medium (ethanol-NaOH) at room temperature in high yields. Additionally, 2 mol acetophenone derivatives were used in the case of the bis-chalcone. The chalcone derivatives **1a**,[25] **1b**,[26] **1c**,[23] **1d**,[23] **1e**,[27] **1f**,[23] **1g**,[28] **1h**[29] and **1i**[29] are well-known in the literature. Bis-chalcones **5f** except for; **5a-d**,[30] **5e**[31] and **5g**.[32]

In previous reported studies, the 2-alkyl and 2,5-dialkyl pyrroles have been generally obtained as a mixture in the alkylation of pyrrole by chalcone. In this study, the synthesis of the 2-alkyl and 2,5-dialkyl pyrroles as a sole product was focused upon separately. Hence, a systematic study was carried out for determination of optimized conditions. Initially, the pyrrole was reacted with chalcone (in a ratio of pyrrole:chalcone 1:1, 2:1 and 4:1) in the presence of $AlCl_3$ in various solvents such as $CH_2Cl_2$, EtOH, THF and $CH_3CN$, and also without solvent. All the reactions performed in the solvent gave the mixture of 2- and 2,5-dialkyl pyrroles in low yields, and the chalcone remained unconsumed; however, in contrast, the products' yields increased in the solvent-free

reaction. Thus, the reactions were examined increasing the pyrrole ratio to consume the chalcone under solvent-free conditions. The reactions were performed with the pyrrole:chalcone 6:1, 8:1 and 10:1 ratios and 5%, 10% and 15% mol of the $AlCl_3$ under solvent-free conditions. The best condition to approach 2-alkyl pyrroles was obtained: solvent-free, the ratio of pyrrole:chalcone 8:1, 10% mol of the $AlCl_3$, reaction temperature of r.t. and reaction time of 12 hours.

In these optimized conditions, the 2-alkyl pyrroles (**3a-i**) were synthesized in high yields of 84%-95% (Sch. 1, Tab. 1).

The structures of compounds (**3a-i**) were explained on the basis of the spectral (NMR and elemental analysis) data and a comparison with the literature data.[10,15,33]

After, in order to obtain the 2,5-dialkyl pyrroles (**4a-f**) as a sole product, some reactions were examined by increasing the chalcone ratio in the different solvents. The reactions of the pyrrole with chalcone at the ratio of 1:3 were performed in the presence of 10% mol of $AlCl_3$ in various solvents such as $CH_2Cl_2$, EtOH, THF, and $CH_3CN$ at room temperature. Among these reactions, the 2,5-dialkyl pyrroles was obtained as a sole product with the best yield in $CH_3CN$ at room temperature for 3 hours. In these conditions, the 2,5-dialkyl pyrroles (**4a-f**) were obtained in yields of 76%-93% (Sch. 2, Tab. 2). The structures of compounds (**4a-i**) were explained on the basis of the spectral (NMR and elemental analysis) data and comparison with the literature data. [10,15,33]

This study was extended to the alkylation of the pyrrole and indole with 1,4-phenylene bis-chalcones. The reactions of the pyrrole and/or indole with 1,4-bis-chalcones at a ratio of 8:1 and in the presence of 10% mol of $AlCl_3$ gave the double-addition products **6a-g** (67-89%) (Sch. 3, Tab. 3) and **8a-g** (69-83 %) (Sch. 4, Tab. 4), respectively, in $CH_3CN$ at room temperature for 24 hours.

The structure of all the synthesized compounds was confirmed by $^{1}H$-, $^{13}C$-NMR spectroscopy and the elemental analysis.

## 3. CONCLUSION

As a consequence, in this paper, a useful method was reported for the direct synthesis of 2- and 2,5-dialkyl pyrroles from the $AlCl_3$-catalyzed addition of pyrrol to chalcone derivatives. In addition, the $AlCl_3$ was used as the catalyst for the first time in this reaction. The 2-alkyl pyrrolles were obtained in pyrrole, chalcone and $AlCl_3$ 8:1:10% mol ratios, respectively, at room temperature for 12 hours in high yields (84%-95%). When the same reaction was performed using the ratio of pyrrole, chalcone and $AlCl_3$ at 1:3:10% mol, respectively, in $CH_3CN$ at room temperature for 3 hours, the 2,5-dialkyl pyrroles were obtained in good yields (76-93%). Moreover, the addition of pyrrole and indole to 1,4-phenylene bis-chalcone was performed for the first time. The reaction of pyrrole and/or indole with 1,4-phenylene bis-chalcone at a ratio of 8:1 in the presence of 10% mol of $AlCl_3$ at room temperature for 24 hours gave the double additional products in good yields (67-89% and 69-83%, respectively).

## 4. EXPERIMENTAL

The melting points were determined on a Electrothermal 9100 apparatus and were uncorrected. The $^{1}H$ and $^{13}C$ NMR spectra were recorded on a Bruker Avance DPX-400 instrument. As internal standards served TMS ($\delta$ 0.00) for $^{1}H$ NMR and $CDCl_3$ ($\delta$ 77.0) for $^{13}C$ NMR spectroscopy, the *J* values are given in Hz. The multiplicities of the signals in the $^{1}H$ NMR spectra were abbreviated by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad) and combinations thereof. The elemental analyses were obtained from a LECO CHNS 932 Elemental Analyzer.

### 4.1. General Procedure For The Synthesis Of 3a-I:

A mixture of pyrrole (**2**) (10 mmol) and chalcone (**1a-i**) (1 mmol) and $AlCl_3$ (10%, mol) was stirred at room temperature for 24 h until the starting material disappeared on the TLC. After the completion of the reaction, the catalyst was separated by filtration. The reaction mixture was then diluted with water and extracted with $CHCl_3$. Subsequently, the organic layer was dried over anhydrous sodium sulfate. The evaporating of the solvent under reduced pressure gave the corresponding viscous oil products.

#### 4.1.1. 1,3-Di(Furan-2-Yl)-3-(1H-Pyrrol-2-Yl)Propan-1-One (3a)

Viscous oil; Yield (84%). $^{1}H$-NMR (400 MHz, $CDCl_3$) $\delta$ (ppm): 8.63 (s, 1H), 7.59 (br s, 1H), 7.40 (br s, 1H), 7.23 (d, *J* = 3.2 Hz, 1H), 6.71 (t, *J* = 1.2 Hz, 1H), 6.56 (t, *J* = 1.6 Hz, 1H), 6.33 (t, *J* = 1.4 Hz, 1H), 6.18-6.14 (m, 2H), 5.98 (br *s*, 1H), 4.89 (t, *J* = 7.0, Hz, 1H), 3.58 (dd, *J* = 6.4; 4.0; 2.4 Hz, 2H). $^{13}C$-NMR (100 MHz, $CDCl_3$) $\delta$ (ppm): 187.4, 157.4, 152.5, 151.5, 146.7, 141.6, 131.5, 117.2, 112.5, 110.3 (2C), 105.5, 105.3, 44.1,

33.4. Anal. cald. for $C_{15}H_{13}NO_3$: C, 70.58; H, 5.13; N, 5.49. Found: C, 70.46; H, 5.09; N, 5.37.

### 4.2. General Procedure For The Synthesis Of 4a-F

To a mixture of pyrrole (**2**) (1 mmol) and chalcone (**1**) (3 mmol) in $CH_3CN$ (5 mL) and $AlCl_3$ (10%, w/w) was added, and stirred at room temperature for 3 h. The reaction was followed by TLC. After the completion of the reaction, the catalyst was separated by filtration. The reaction mixture was then diluted with water and extracted with $CHCl_3$. Subsequently, the organic layer was dried over anhydrous sodium sulfate. The evaporating of the solvent under reduced pressure gave the corresponding viscous oil products.

#### 4.2.1. 3,3'-(1H-Pyrrole-2,5-Diyl)Bis(1,3-Bis(4-Methoxyphenyl)Propan-1-One) (4a)

Viscous oil; Yield (85%). $^1$H-NMR (400 MHz, $CDCl_3$) δ (ppm): 8.53 (br s, 1H), 8.05 (d, *J* = 8.8 Hz, 4H), 7.87 (d, *J* = 8.4 Hz, 4H), 6.95 (d, *J* = 8.8 Hz, 4H), 6.84 (d, *J* = 8.4 Hz, 4H), 5.79 (d, *J* = 2.4 Hz, 1H), 5.75 (d, *J* = 4.0 Hz, 1H), 4.76 (t, *J* = 6.8 Hz, 2H), 3.79 (s, 12H), 3.65 (dd, *J* = 17.2, 7.6 Hz, 2H), 3.45 (dd, *J* = 16.8, 6.4 Hz, 2H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ (ppm): 197.4 (2C), 163.5 (2C), 158.1 (2C), 138.1 (2C), 135.5 (2C), 130.7 (2C), 128.9 (4C), 127.7 (4C), 114.4 (4C), 114.0 (4C), 107.7 (2C), 55.3 (2C), 55.0 (2C), 44.8 (2C), 38.9 (2C). Anal. cald. for $C_{38}H_{37}NO_6$: C, 75.60; H, 6.18; N, 2.32. Found: C, 74.98; H, 6.07; N, 2.28.

### 4.3. General Procedure For The Synthesis Of Bis-Chalcone Derivatives (5a-G)

To a solution of acetophenone derivatives (2 mmol) in EtOH (20 ml) was added NaOH (2.5 M, 6 ml). The mixture was stirred for 3 min and teraftaldehit was added (1 mmol) and stirred at r.t. for 4 h. After the reaction, the precipitate was filtered and washed with ethanol several times. The solid was crystallized by methylene chloride / hexane (3/7).

**4.3.1**. **3,3'-(1,4-Phenylene)Bis(1-(Thiophene-3-Yl)Prop-2-En-1-One (5f)**

Yield (90%); mp: 219-221 $^{o}$C. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ (ppm): 8.85 (s, 2H), 7.97 (s, 4H), 7.95-7.91 (d, *J*= 15.6 Hz, 2H), 7.76-7.72 (d, *J*= 15.6 Hz, 2H), 7.70-7.69 (m, 4H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ (ppm): 183.3, 143.3, 142.5, 137.0, 135.0, 129.7, 128.2, 127.5, 124.5. IR Spektrumu (KCl, $cm^{-1}$): 3077, 1656, 1600, 1511, 1423, 1396, 1230, 1203, 1072, 1035, 981, 804, 713, 690. Anal. cald. for $C_{20}H_{14}O_2S_2$: C, 68.54; H, 4.03. Found: C, 68.28; H, 4.02.

**4.4. General Procedure For The Synthesis Of 6a-G And 8a-G**

To a solution of pyrrole (**2**) (8 mmol) or indole (**7**) (8 mmol) and bis-chalcone (**5a-g**) (1 mmol) in $CH_3CN$ (5 mL) was added $AlCl_3$ (10%, w/w) and the mixture was stirred at room temperature for 24 h. The reaction was followed by TLC. After the completion of the reaction, the catalyst was separated by filtration. The reaction mixture was then diluted with water and extracted with $CHCl_3$. Subsequently, the organic layer was dried over anhydrous sodium sulfate. The evaporating of the solvent under reduced pressure gave the corresponding solid products. The crude products were purified by recrystallization from *n*-hexane-$CHCl_3$.

**4.4.1. 3,3'-(1,4-Phenylene)Bis(1-Phenyl-3-(1H-Pyrrol-2-Yl)Propan-1-One) (6a)**

Yield (85%); mp: 212-214 $^{o}$C. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ (ppm): 8.43 (br s, 2H), 7.97 (d, *J* = 7.2 Hz, 4H), 7.60-7.57 (m, 2H), 7.48 (t, *J* = 7.2 Hz, 4H), 7.27 (s, 4H), 6.68 (s, 2H), 6.11 (s, 2H), 5.87 (s, 2H), 4.77 (d, *J* = 7.2 Hz, 2H), 3.83-3.79(dd, *J* = 8.0, 17.6 Hz, 2H), 3.64-3.58 (dd, *J* = 5.6, 17.6 Hz, 2H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ (ppm): 197.7 (2C), 141.4 (2C), 139.8 (2C), 135.1 (2C), 134.1 (2C), 129.5 (4C), 128.9 (4C), 128.3 (4C), 117.2 (2C), 107.9 (2C), 105.4 (2C), 45.1 (2C), 39.0 (2C). Anal. cald. for $C_{32}H_{28}N_2O_2$: C, 81.33; H, 5.97; N, 5.93. Found: C, 81.27; H, 5.91; N, 5.86.

**4.4.8. 3,3'-(1,4-Phenylene)Bis(3-(1H-Indol-3-Yl)-1-Phenylpropan-1-One) (8a)**

Yield (75%); mp: 230-232 $^{o}$C. $^{1}$H-NMR (400 MHz, $CDCl_3$) δ (ppm): 10.81 (s, 2H), 7.97–7.94 (μ, 4H7.60-7.58 (m, 2H), 7.46 (t, *J* = 7.6 Hz, 4H), 7.39-7.36 (m, 2H), 7.26 (s, 8H), 7.01-6.97 (m, 2H), 6.86-6.82 (m, 2H), 4.79 (t, *J*= 7.2 Hz, 2H), 3.93-3.87 (dd, *J*= 7.6, 17.2 Hz, 2H), 3.69-3.63 (dd, *J*= 6.8, 17.2 Hz, 2H). $^{13}$C-NMR (100 MHz, $CDCl_3$) δ (ppm): 199.3 (2C), 143.6 (2C), 137.8 (2C), 137.2 (2C), 134.0 (2C), 129.5 (4C), 128.9 (4C), 128.4 (4C), 127.3 (2C), 122.7 (2C), 121.8 (2C), 119.6 (2C), 119.1 (2C), 118.9 (2C),112.2 (2C), 45.3 (2C), 38.1 (2C). Anal. cald. for $C_{40}H_{32}N_2O_2$: C, 83.89, H, 5.63; N, 4.89. Found: C, 83.77; H, 5.58; N, 4.81.



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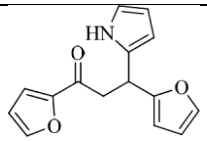
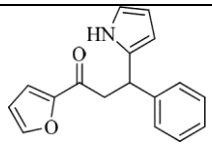
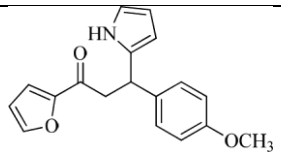
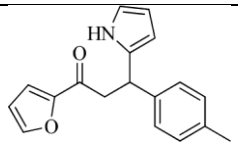
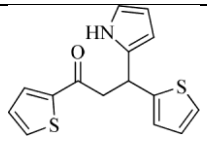
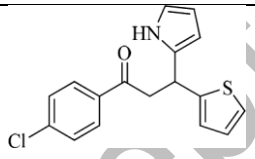
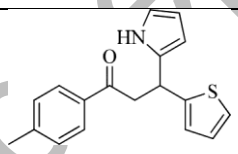
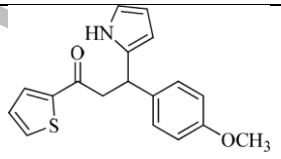
Supplemental data for this article can be accessed on the publisher's website.

Table 1. $AlCl_3$-Catalyzed addition of pyrrol to chalcones in ratio of 8:1

| Entry | Product | Yield (%) |
|---|---|---|
| 1 |  | 84 |
| 2 |  | 89 |
| 3 |  | 95 |
| 4 |  | 93 |
| 5 |  | 91 |
| 6 |  | 89 |
| 7 |  | 93 |
| 8 |  | 84 |

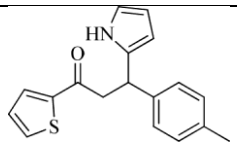
| | | |
|---|---|---|
| 9 |  | 86 |

Table 2. $AlCl_3$-Catalyzed addition of pyrrol to chalcones in ratio of 1:3

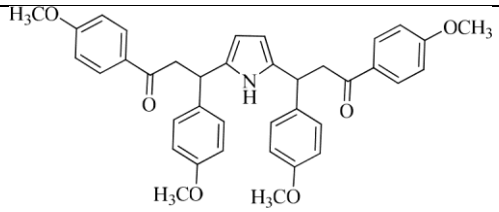
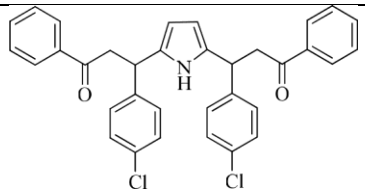
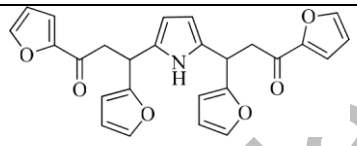
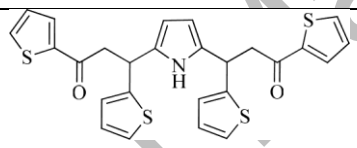
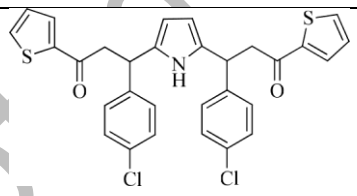
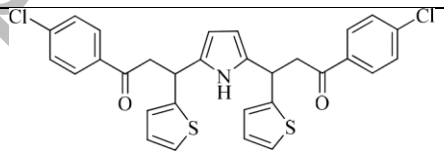
| Entry | Product | Yield (%) |
|---|---|---|
| 1 |  | 85 |
| 2 |  | 93 |
| 3 |  | 76 |
| 4 |  | 82 |
| 5 |  | 85 |
| 6 |  | 84 |

Table 3. $AlCl_3$-Catalyzed addition of pyrrol to bis-chalcones

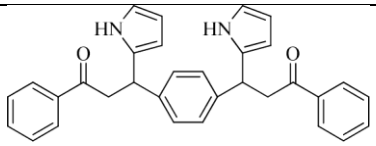
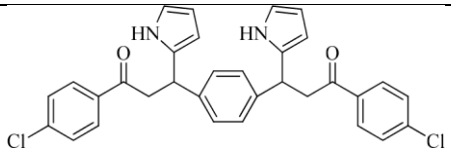
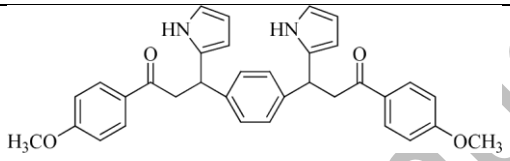
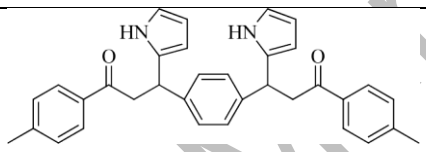
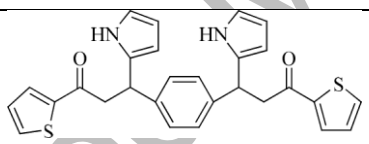
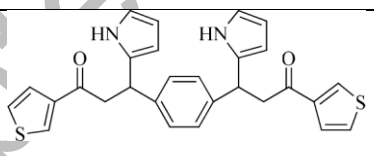
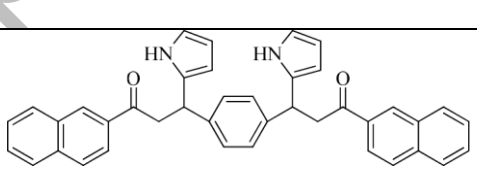
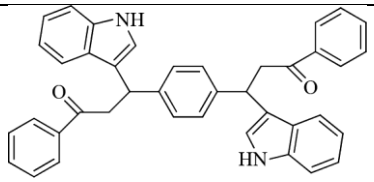
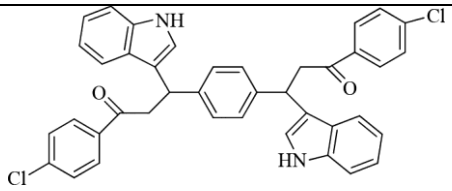
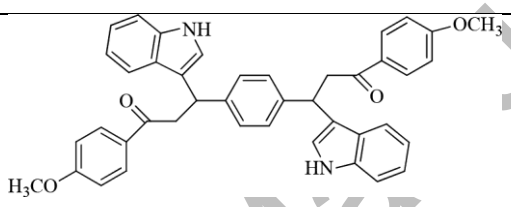
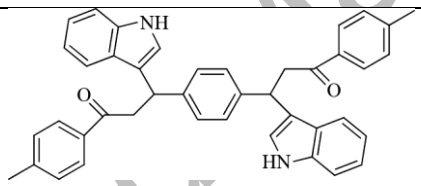
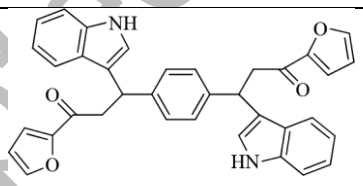
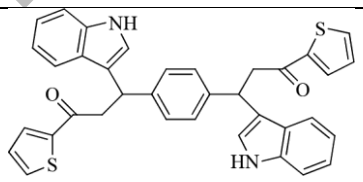
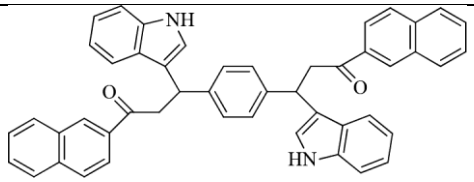
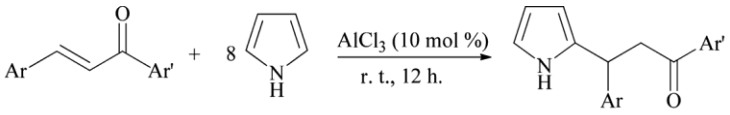
| Entry | Product | Yield (%) |
|---|---|---|
| 1 |  | 85 |
| 2 |  | 89 |
| 3 |  | 74 |
| 4 |  | 71 |
| 5 |  | 89 |
| 6 |  | 76 |
| 7 |  | 81 |

Table 4. $AlCl_3$-Catalyzed addition of indole to bis-chalcones

| Entry | Product | Yield (%) |
|---|---|---|
| 1 |  | 75 |
| 2 |  | 69 |
| 3 |  | 73 |
| 4 |  | 81 |
| 5 |  | 77 |
| 6 |  | 83 |
| 7 |  | 78 |

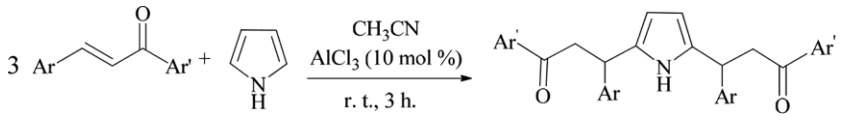
Scheme 1.




**1** **2** **3**

| | **3a** | **3b** | **3c** | **3d** | **3e** | **3f** | **3g** | **3h** | **3i** |
|---|---|---|---|---|---|---|---|---|---|
| Ar | 2-furyl | Ph | 4-MeOPh | 4-MePh | 2-thienyl | 2-thienyl | 2-thienyl | 4-MeOPh | 4-MePh |
| Ar' | 2-furyl | 2-furyl | 2-furyl | 2-furyl | 2-thienyl | 4-ClPh | 4-MePh | 2-thienyl | 2-thienyl |

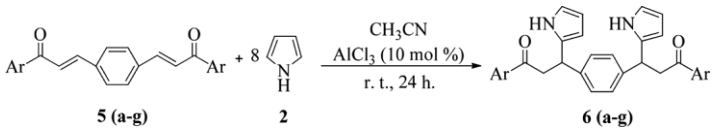
Scheme 2.




1 2 4

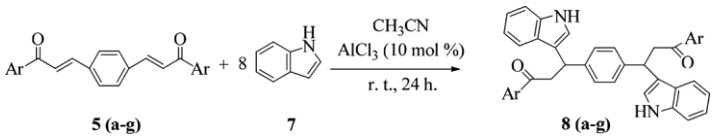
| | 4a | 4b | 4c | 4d | 4e | 4f |
|---|---|---|---|---|---|---|
| Ar | 4-MeOPh | 4-ClPh | 2-furyl | 2-thienyl | 4-ClPh | 2-thienyl |
| Ar' | 4-MeOPh | Ph | 2-furyl | 2-thienyl | 2-thienyl | 4-ClPh |

Scheme 3.




**5 (a-g)** **2** **6 (a-g)**

Scheme 4.




**5 (a-g)** **7** **8 (a-g)**