CHAPTER-V

Synthesis of N-alkyl [1-(4-chlorophenyl) cyclopropyl] (piperazin-1-yl) methanone derivatives
INTRODUCTION

Piperazine is an interesting heterocyclic moiety as constituent of several biologically active molecules. The polar nitrogen atoms in the piperazine ring confer bioactivity to molecules and enhance favourable interaction with macromolecules [1-2]. Piperazinyl-linked ciprofloxacin dimers are potent antibacterial agents against resistant strains, antimalarial agents and potential antipsychotic agents [3-5]. Piperazine derivatives containing tetrazole nucleus have been reported as antifungal agents [6] Substituted benzamide piperazine derivatives have shown strong agonistic activity while the substituted acetamide piperazine derivative have better dopamine D receptor agonist activity as compared to substituted benzamide piperazine derivatives [7-8] Diphenyl piperazine derivatives possess broad pharmacological action on central nervous broad pharmacological action on central nervous system (CNS), especially on dopaminergic neurotransmission.

Heterocyclic systems are one of the most important classes of organic compounds present in nature or synthesized in laboratory. These compounds are known to possess an array of biological activities and are employed in the treatment of commonly occurring diseases. This has been the backbone for medicinal chemists to keep perpetuating interest to synthesize some novel derivatives of possible high biological activity. Literature survey revealed that piperazines, chalcones, pyrimidines and thiophene possess a broad spectrum of biological activities like, antibacterial [9], anti-inflammatory [10-11], antimalarial [12], antihistamine [13], antitubercular [14], anticancer [15-17] etc. The above classes of compounds are used extensively to elicit varied pharmacological responses. It has also been reported that substituted-piperazines possess antihistaminic [18-19], antioxidative [20], adrenolytic, hypotensive and CNS depressant activities [21]. As a part of our search
for potent H1-receptor antagonists, a novel series of various chalcone, pyrimidine and substituted-piperazines were synthesized [22-24] and considered for structure-activity relationship studies.

Malaria is a vector-borne infectious disease caused by protozoan parasites. It is widespread in tropical and subtropical regions, including parts of the Americas, Asia, and Africa. Every year, there is approximately 350–500 million cases of malaria [25], killing between one and three million people, and the majority of whom are young children in Sub-Saharan Africa [26]. Ninety percent of malaria-related deaths occur in Sub-Saharan Africa. The advent of long lasting insecticidal nets and Artemisinin-based combination therapy, plus a revival of support for indoor residual spraying of insecticide, presents a new opportunity for large-scale malaria control. Malaria is commonly associated with poverty, but is also a cause of poverty and a major hindrance to economic development [27]. People usually get malaria from the bite of Anopheles mosquitoes, the disease being caused by protozoan parasites of the genus Plasmodium. Five species of the Plasmodium parasite can infect humans; the most serious forms of the disease are caused by Plasmodium falciparum. Malaria caused by Plasmodium vivax, Plasmodium ovale and Plasmodium malariae causes milder disease in humans that is not generally fatal. A fifth species, Plasmodium knowlesi, causes malaria in macaques but can also infect humans. This group of human-pathogenic Plasmodium species is usually referred to as malaria parasites. Several antimalarial drugs have been formulated for the treatment and prevention of the disease, but these have led to development of resistance by the parasites to most of the drugs in use. Specifically, there is reportedly, rapid spread of P. falciparum resistance to available antimalarial drugs [28]. Thus, there is a constant need for developing new antimalarial compounds.
Ethnic medicine has provided two of the most efficacious drugs, Quinine and Artemisinin (and its analogs) and the ongoing screening of medicinal plants yields new lead compounds [29]. Work has been done on malaria vaccines with limited success and more exotic controls, such as genetic manipulation of mosquitoes to make them resistant to the parasite, have also been considered [30]. Although some vaccines are under development, none is currently available for malaria that provides a high level of protection [31]; preventive drugs must be taken continuously to reduce the risk of infection.

These prophylactic drug treatments are often too expensive for most people living in endemic areas. Most adults from endemic areas have a degree of long-term infection, which tends to recur, and also possess partial immunity (resistance); the resistance reduces with time, and such adults may become susceptible to severe malaria if they have spent a significant amount of time in non-endemic areas. In last decades, Quantitative Structure-Activity Relationships (QSAR) [32], have been applied in many areas enabling to prevent time consuming and cost during the analysis of biological activities of interest. The main hypothesis involved in any QSAR is the assumption that the variation of the behavior of chemical compounds, as expressed by any experimentally measured biological or physicochemical property, can be correlated with numerical entities related to some aspect of the chemical structure termed molecular descriptors [33-34]. Descriptors are generally used to describe different characteristics/attributes of the chemical structure in order to yield information about the activity/property being studied. In general, QSAR studies are effected by various factors from which the most relevant
PRESENT WORK

In our present research we are planned to synthesis of derivatives of Chlorophenyl cyclopropyl piperazines with substituted alkyl and aryl halides. 1-(4-Chlorophenyl) cyclopropyl] (piperazin-1-yl) methanone (1.0 eq) was dissolved in dry DMF solvent (10 mL). The solution was stirred for 10 min at ambient temperature. Then cesium carbonate (1.5 eq) was added followed by alkyl or aryl halide (1.1 eq). The reaction mixture was heated to 45°C for 3h, the completion of reaction was monitored by TLC. The reaction mixture was diluted with ethyl acetate, the organic layer was washed water followed by brine solution and dried over anhydrous sodium sulphate. The organic layer was evaporate under reduced pressure and the crude was purified by column chromatography using 60-120 mesh silica gel.

All the synthesized compounds have been purified by column chromatography. The structures have been confirmed by elemental analysis and spectroscopic techniques like IR, $^1$H-NMR, LC-MS. The series of reactions carried out have been depicted in scheme 4 and derivatives are depicted in table-4.

Scheme 4
Table 4 Synthesis of piperazine methanone derivatives

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EXPERIMENTAL

t-butyl 4-(1-(4-Chlorophenyl)cyclopropanecarbonyl)piperazine-1-carboxylate (1)

1-(4-Chlorophenyl)cyclopropanecarboxylic acid (2.00 g, 7.55 mmol, 1.0 eq) was dissolved in dry DMF (20 mL). The solution was stirred for 10 min at ambient temperature. To this added HATU (4.30 g, 11.33 mmol) and N,N-diisopropylethylamine (3.905 g, 30.2 mmol). The reaction mixture was stirred for 20 min at ambient temperature, and then it was cooled to 0°C. Boc-piperazine (tert-
butyl piperazine-1-carboxylate) (1.406 g, 7.55 mmol,) was added portion-wise to the mixture and stirring was continued for 5 h at ambient temperature. The completion of the reaction was monitored by TLC. The reaction mixture was diluted with ethyl acetate and washed with water followed by saturated sodium bicarbonate solution followed by water and brine solution. It was finally dried over sodium sulphate and evaporate under reduced pressure. The crude mass was purified by column chromatography using silica gel and 10% ethyl acetate in hexane to get 3.3 g of t-butyl 4-(1-(4-chlorophenyl)cyclopropanecarbonyl) piperazine-1-carboxylate 1.

![1](image)

**1**

LC-MS (ESI, Positive): m/z: [M+H]^+: 365.2; ^1^H NMR: (400 MHz, DMSO-d_6): δ 7.40 (d, J = 2.0 Hz, 2H), 7.17 (d, J = 3.0 Hz, 2H), 3.63 (m, 2H), 2.96 (m, 2H), 1.37 (q, 2H), 1.18 (q, 2H), 1.2 (s, 9H); Elemental analysis: Calculated (%) for C_{14}H_{17}ClN_2O: C 62.54, H 6.91, N 7.68; Found: C 62.55, H 6.94, N 7.62

**[1-(4-Chlorophenyl)cyclopropyl](piperazin-1-yl)methanone (2)**

Compound 1 (3g 8.22mmol) was dissolved in Dry dichloromethane (30 ml) cooled the reaction mixture to 5°C slowly added 1.5N HCl (15 mL) to the cooled reaction mixture. The reaction was allowed to room temperature and stirred for 4 hr. The completion of the reaction was monitored by TLC. After completion of the reaction. The mixture was diluted with dichloromethane the organic layer was washed with water, saturated sodium bicarbonate solution followed by brine solution the organic layer was dried with sodium sulphate and evaporate under reduced pressure to get crude product of 1-(4-Chlorophenyl) cyclopropyl] (piperazin-1-yl) methanone.2
LC-MS (ESI, Positive): m/z: [M+H]^+ 265.2; ¹H NMR: (400 MHz, DMSO-d₆): δ 9.32 (s, 1H), 7.40 (d, J = 2.0 Hz, 2H), 7.17 (d, J = 3.0 Hz, 2H), 3.63 (m, 2H), 2.96 (m, 2H), 1.37 (q, 2H), 1.18 (q, 2H); Elemental analysis: Calculated for C₁₄H₁₇ClN₂O: C 63.51, H 6.47, N 10.58; Found: C, 63.53 H 6.46, N 10.57.

**General procedure for 10a-o**

Synthesis of derivatives of chlorophenyl cyclopropyl piperazines with substituted alkyl and aryl halides. 1-(4-Chlorophenyl) cyclopropyl] (piperazin-1-yl) methanone (1.0 eq) was dissolved in dry DMF solvent (10 mL). The solution was stirred for 10 min at ambient temperature. Then cesium carbonate (1.5 eq) was added followed by alkyl or aryl halide (1.1 eq). The reaction mass was heated to 45°C for 3h, the completion of reaction was monitored by TLC. The reaction mixture was diluted with ethyl acetate, the organic layer was washed with water followed by brine solution and dried over anhydrous sodium sulphate. The organic layer was evaporate under reduced pressure and the crude was purified by column chromatography using 60-120 mesh silica gel. Pure compound obtained after column purification.

**SPECTRAL DATA**

4-(4-Allylpiperazin-1-yl)(1-(4-chlorophenyl)cyclopropyl)methanone (10a)
LC-MS (ESI, Positive): m/z: [M+H]^+: 306.4; $^1$H NMR (400 MHz, DMSO-$d_6$): δ 7.37-7.33 (m, 2H), 7.18-7.14 (m, 2H), 5.78-5.72 (m, 1H), 5.16-5.08 (m, 2H), 3.42-3.37 (m, 4H), 2.89-2.87 (d, $J$=6.4 Hz, 2H), 2.26-2.13 (m, 4H), 1.29-1.27 (m, 2H), 1.17-1.14 (m, 2H); Elemental analysis: Calculated (%) for $C_{17}H_{21}ClN_2O$: C 66.99, H 6.94, N 9.19; Found: C 66.85, H 6.82, N 9.10.

**(4-Benzylpiperazin-1-yl)(1-(4-chlorophenyl)cyclopropyl)methanone (10b)**

![Image of 10b](image)

LC-MS (ESI, Positive): m/z: [M+H]^+: 357.8; $^1$H NMR (400 MHz, CDCl$_3$): δ 7.31-7.29 (m, 4H), 7.28-7.23 (m, 3H), 7.09-7.06 (m, 2H), 3.64-3.46 (m, 4H), 3.44 (s, 2H), 2.60-2.39 (m, 4H), 1.42-1.39 (m, 2H), 1.14-1.11 (m, 2H); Elemental analysis: Calculated (%) for $C_{21}H_{23}ClN_2O$: C 71.07, H 6.53, N 7.89; Found: C 69.92, H 6.43, N 7.56.

**(4-(4-(Benzyloxy)benzyl)piperazin-1-yl)(1-(4-chlorophenyl)cyclopropyl)methanone (10c)**

![Image of 10c](image)

LC-MS (ESI, Positive): m/z: [M+H]^+: 463.0; $^1$H NMR (400 MHz, DMSO-$d_6$): δ 7.44-7.36 (m, 4H), 7.33-7.29 (m, 3H), 7.23-7.14 (m, 3H), 6.90-6.82 (m, 3H), 5.08 (s,
2H), 3.45-3.38 (m, 4H), 3.33 (s, 2H), 2.50-2.27 (m, 4H), 1.30-1.27 (m, 2H), 1.17-1.14 (m, 2H); Elemental analysis: Calculated (%) for C_{28}H_{29}ClN_{2}O_{2}: C 72.95, H 6.34, N 6.08; Found: C 72.62, H 6.23, N 5.96.

**(1-(4-Chlorophenyl)cyclopropyl)(4-propylpiperazin-1-yl)methanone (10d)**

![10d](image)

LC-MS (ESI, Positive): m/z: [M+H]^+: 307.2; \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}): \(\delta\) 7.27-7.25 (m, 2H), 7.09-7.07 (m, 2H), 3.64-3.42 (m, 4H), 2.37-2.13 (m, 6H), 1.47-1.40 (m, 4H), 1.15-1.12 (m, 2H), 0.91-0.87 (t, \(J=7.2\) Hz, 3H); IR (KBr) \(\nu(\text{cm}^{-1})\): 1604 (C=O), 776 (C-Cl); Elemental analysis: Calculated (%) for C_{17}H_{23}ClN_{2}O: C 66.55, H 7.56, N 9.13; Found: C 66.41, H 7.32, N 9.09.

**(1-(4-Chlorophenyl)cyclopropyl)(4-methylpiperazin-1-yl)methanone (10e)**

![10e](image)

LC-MS (ESI, Positive): m/z: [M+H]^+: 280.2; \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}): \(\delta\) 7.27-7.25 (m, 2H), 7.10-7.08 (m, 2H), 3.64-3.44 (m, 4H), 2.37-2.13 (m, 4H), 2.62 (s, 3H), 1.43-1.40 (m, 2H), 1.16-1.13 (m, 2H); Elemental analysis: Calculated (%) for C_{15}H_{19}ClN_{2}O: C 64.63, H 6.87, N 10.05; Found: C 64.52, H 6.75, N 9.98.
(1-(4-Chlorophenyl)cyclopropyl)(4-(pent-4-enyl)piperazin-1-yl)methanone (10f)

10f

LC-MS (ESI, Positive): m/z: [M+H]^+: 334.8; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.27-7.24 (m, 2H), 7.11-7.07 (m, 2H), 5.81-5.74 (m, 1H), 5.01-4.92 (m, 2H), 3.64-3.42 (m, 4H), 2.29-2.25 (m, 4H), 2.12-2.01 (m, 4H), 1.56-1.50 (m, 2H), 1.43-1.40 (m, 2H), 1.17-1.14 (m, 2H); Elemental analysis: Calculated (%) for C\(_{19}\)H\(_{25}\)ClN\(_2\)O: C 68.56, H 7.7, N 8.42; Found: C, 68.23, H 7.46, N 8.23.

(4-Butylpiperazin-1-yl)(1-(4-chlorophenyl)cyclopropyl)methanone (10g)

10g

LC-MS (ESI, Positive): m/z: [M+H]^+: 324.0; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.29-7.24 (m, 2H), 7.12-7.06 (m, 2H), 3.64-3.43 (m, 4H), 2.29-2.25 (m, 6H), 1.59-1.30 (m, 4H), 1.27-1.12 (m, 4H), 0.94-0.89 (t, \(J=7.2\) Hz, 2H); Elemental analysis: Calculated (%) for C\(_{18}\)H\(_{25}\)ClN\(_2\)O: C 67.38, H 7.85, N 8.73; Found: C 67.11, H 7.75, N 8.65.

(4-(But-3-enyl)piperazin-1-yl)(1-(4-chlorophenyl)cyclopropyl)methanone (10h)

10h

LC-MS (ESI, Positive): m/z: [M+H]^+: 320.4; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.28-7.24 (m, 2H), 7.10-7.07 (m, 2H), 5.77-5.73 (m, 1H), 5.06-5.00 (m, 2H), 3.64-3.42
(1-(4-Chlorophenyl)cyclopropyl)(4-ethylpiperazin-1-yl)methanone (10i)

LC-MS (ESI, Positive): m/z: [M+H]^+: 296.3; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.27-7.25 (m, 2H), 7.10-7.08 (m, 2H), 3.64-3.44 (m, 4H), 2.37-2.32 (m, 4H), 2.16-2.14 (m, 2H), 1.43-1.40 (m, 2H), 1.16-1.13 (m, 2H), 1.05-1.02 (t, \(J=7.2\) Hz, 3H); IR (KBr) \(\nu\) (cm\(^{-1}\)): 1602 (C=O); Elemental analysis: Calculated (%) for C\(_{18}\)H\(_{23}\)ClN\(_2\)O: C 67.81, H 7.27, N 8.79; Found: C 67.75, H 7.20, N 8.58.

(4-(4-Methoxybenzyl)piperazin-1-yl)(1-(4-chlorophenyl)cyclopropyl)methanone (10j)

LC-MS (ESI, Positive): m/z: [M+H]^+: 386.2; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.27-7.25 (m, 2H), 7.10-7.06 (m, 2H), 6.99-6.96 (m, 2H), 6.61-6.58 (m, 2H), 3.65-3.46 (m, 4H), 3.87 (s, 3H), 3.46 (s, 2H), 2.60-2.39 (m, 4H), 1.42-1.39 (m, 2H), 1.14-1.11 (m, 2H); Elemental analysis: Calculated (%) for C\(_{22}\)H\(_{25}\)ClN\(_2\)O\(_2\): C 68.65, H 6.55, N 7.28; Found: C 68.61, H 6.32, N 7.15.
(1-(4-Chlorophenyl)cyclopropyl)(4-isopropylpiperazin-1-yl)methanone (10k)

\[ \text{Cl-} \overset{\text{O}}{\text{N}} \text{N} \]

**10k**

LC-MS (ESI, Positive): m/z: [M+H]^+ : 307.8; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.25-7.23 (m, 2H), 7.09-7.07 (m, 2H), 3.65-3.44 (m, 4H), 2.95 (m, 1H), 2.47-2.30 (m, 4H), 1.45 (s, 6H), 1.43-1.40 (m, 2H), 1.16-1.13 (m, 2H); Elemental analysis: Calculated (%) for C\(_{17}\)H\(_{23}\)ClN\(_2\)O: C 66.55, H 7.56, N 9.13; Found: C 66.58, H 7.52, N 9.15.

(4-(4-Fluorobenzyl)piperazin-1-yl)(1-(4-chlorophenyl)cyclopropyl)methanone (10l)

\[ \text{Cl-} \overset{\text{O}}{\text{N}} \text{N} \]

**10l**

LC-MS (ESI, Positive): m/z: [M+H]^+ : 373.8; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.27-7.25 (m, 2H), 7.12-7.04 (m, 4H), 6.99-6.96 (m, 2H), 3.68-3.51 (m, 4H), 3.55 (s, 2H), 2.62-2.37 (m, 4H), 1.42-1.39 (m, 2H), 1.14-1.11 (m, 2H); Elemental analysis: Calculated (%) for C\(_{21}\)H\(_{22}\)ClF\(_2\)N\(_2\): C 67.65, H 5.95, N 7.51; Found: C 67.66, H 5.94, N 7.53.

(4-(4-Methoxybenzyl)piperazin-1-yl)(1-(4-chlorophenyl)cyclopropyl)methanone (10m)

\[ \text{Cl-} \overset{\text{O}}{\text{N}} \text{N} \]

**10m**
LC-MS (ESI, Positive): m/z: [M+H]^+: 390.3; ^1H NMR (400 MHz, CDCl$_3$): δ 7.57-7.55 (m, 2H), 7.42-7.34 (m, 4H), 7.25-7.21 (m, 2H), 3.78-3.65 (m, 4H), 3.60 (s, 2H), 2.65-2.40 (m, 4H), 1.42-1.39 (m, 2H), 1.14-1.11 (m, 2H); Elemental analysis: Calculated (%) for C$_{21}$H$_{22}$Cl$_2$N$_2$O: C 64.79, H 5.70, N 7.20; Found: C 64.82, H 5.75, N 7.23.

Ethyl(4-[[1-(4-chlorophenyl)cyclopropyl]carbonyl]piperazin-1-yl)acetate (10n)

![Image of 10n](image)

LC-MS (ESI, Positive): m/z: [M+H]^+: 351.8; ^1H NMR (400 MHz, CDCl$_3$): δ 7.45-7.43 (m, 2H), 7.29-7.27 (m, 2H), 4.12-4.09 (q, J=7.2Hz, 2H), 3.68-3.46 (m, 4H), 3.20 (s, 2H), 2.49-2.31 (m, 4H), 1.43-1.40 (m, 2H), 1.21-1.18 (t, J=7.2Hz, 3H), 1.16-1.13 (m, 2H); Elemental analysis: Calculated (%) for C$_{18}$H$_{23}$ClN$_2$O$_3$: C 61.62, H 6.61, N 7.98; Found: C 61.64, H 6.64, N 7.96.

(4-(4-Methylbenzyl)piperazin-1-yl)(1-(4-chlorophenyl)cyclopropyl) methanone (10o)

![Image of 10o](image)

LC-MS (ESI, Positive): m/z: [M+H]^+: 369.9; ^1H NMR (400 MHz, CDCl$_3$): δ 7.37-7.35 (m, 2H), 7.20-7.06 (m, 4H), 6.81-6.78 (m, 2H), 3.65-3.46 (m, 4H), 3.87 (s, 3H), 3.48 (s, 2H), 2.60-2.39 (m, 4H), 1.43-1.40 (m, 2H), 1.15-1.12 (m, 2H); Elemental analysis: Calculated (%) for C$_{22}$H$_{25}$ClN$_2$O: C 71.63, H 6.83, N 7.59; Found: C 71.64, H 6.84, N 7.56.
REFERENCES


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<td>Deviation, abs.</td>
<td>0.02</td>
<td>0.05</td>
<td>0.10</td>
<td>0.002</td>
</tr>
<tr>
<td>Delta(%)</td>
<td>0.04</td>
<td>0.10</td>
<td>0.20</td>
<td>0.004</td>
</tr>
</tbody>
</table>

**Statistic report**