CHAPTER 4

An efficient, surfactant mediated Biginelli condensation for the one pot synthesis of dihydropyrimidine derivatives
4.1 Introduction

The most powerful tool in the organic synthesis currently is multi-component reactions which have emerged as a ladder to the development of many important organic compounds. One of the important scaffold known is 3,4-dihydropyrimidin-2(1H)-ones (DHPM). Dihydropyrimidinones derivatives exhibit significant therapeutic and pharmacological properties such as antiviral, antitumour, antibacterial, antiinflammatory.\(^1\) In addition to this, some of DHMPS have been employed as calcium channel blockers, \(\alpha\)-1a-agonists and neuropeptide Y (NPY) antagonists.\(^2\) To name a few important DHMPS, Monastrol exhibits important biological property thereby blocking mitosis by specifically inhibiting the motor activity of the mitotic kinesin Eg5 and is a pilot molecule for the advancement of novel anticancer drugs, (R)-SQ 32926, SQ 32547, SWO2 also known to be antihypertensive agent with potent oral activity and Mon-97 shows promising anticancer activity (Fig. 4.1).\(^3\) Several alkaloids obtained from marine sources even found to possess the dihydropyrimidine core unit. Most noteworthy among these are the batzelladine alkaloids, which were found to be potent HIVgp-120-CD4 inhibitors.\(^4\)

![Monastrol](image1), ![SQ 32926](image2), ![SQ 32547](image3), ![SWO2](image4), ![Mon-97](image5)

Fig. 4.1 Pharmacologically important 3,4-dihydropyrimidin-2(1H)-ones/thiones.
The earliest attempt to synthesise the 3,4-dihydropyrimidin-2(1H)-ones was reported by Pietro Biginelli in 1893, he performed in a one-pot the three-component cyclocondensation reaction of ethyl acetoacetate, benzaldehyde and urea under Bronsted acid catalysis. However, this reaction has drawbacks like harsh conditions, high reaction time and low yields. Since the early days of the discovery of this reaction several reaction routes have been proposed but unfortunately, the synthesis of 3,4-dihydropyrimidin-2(1H)-ones often provides generally low to moderate yields. Therefore, this reaction has attained much attention of the researchers so as to develop more efficient protocol to synthesise the pharmacologically important DHMPs.

A number of catalysts have been employed for the Biginelli reaction which includes both homogenous and heterogeneous catalysts like p-toluene sulfonic acid, SiO₂-polyphosphoric acid (SiO₂-PPA), boric acid, HCl/FeCl₃, polyvinyl sulfonic acid, graphite, L-proline, silica sulfuric acid, zeolite, sulfated tungstate, xanthan sulfuric acid, montmorillonite KSF, sulfated silica tungstic acid, PPF–SO₃H, Al(HSO₄)₃, vanadium(III) chloride, bioglycerol-based sulfonic acid functionalized carbon catalyst. Other than classical method of heating, microwave and ultrasound irradiations have been used for the synthesis 3,4-Dihydropyrimidin-2(1H)-ones/thiones under different catalytic condition. A lot of improvised mode of catalysts like ionic liquids, heteropolyacids, and nanomaterials have been also utilised for the synthesis of these important heterocycles.

Khazaei et al. has reported 1-Methyl-3-nitro-1H-imidazol-3-ium tri nitro methanide \([\text{[MIMNO}_2\text{C(NO}_2\text{)_3]}]\) an unique nano structure ionic liquid (NIL) to catalyse the synthesis of 3,4-dihydropyrimidin-2(1H)-one derivatives (159) by the one-pot Biginelli-type three-component condensation reaction of aromatic aldehydes (55), urea (138) and 1,3-dione (158) at room temperature by reflux under solvent-free conditions (Scheme 4.1). The method uses reusable catalyst to furnish good yield of the product.
**Scheme 4.1** Synthesis of 3,4-dihydropyrimidin-2(1H)-one derivatives.

A Brønsted acidic ionic liquid 1-methyl-3-(3-trimethoxysilylpropyl) imidazolium hydrogen sulfate immobilized on magnetic Fe₃O₄ nanoparticles (MNPs-IL-HSO₄) was efficiently utilised by Safari and Zarnegar for the synthesis of a diverse array of 3,4-dihydropyrimidin-2(1H)-ones/thiones (161/162) by condensation of 55, 138/139 and acetylacetone (160) at 100°C under solvent-free condition (Scheme 4.2).²⁸ The authors used a novel catalyst for the Biginelli condensation.

**Scheme 4.2** Biginelli condensation for synthesis of 3,4-dihydropyrimidin-2(1H)-ones/thiones.

Recently, Khatri *et al.* reported a protocol to efficiently synthesise 3,4-dihydropyrimidin-2(1H)-ones/thiones (163/164) via a three-component Biginelli reaction with various aromatic aldehydes (55), β-ketoesters/β-diketones (10) and urea/thiourea (138/139) catalyzed by sulfated polyborate under solvent free conditions at 100°C (Scheme 4.3).²⁹ The protocol emphasis on the synthesis and characterisation of the catalyst there by efficiently deploying it for the synthesis.
Although a number of improved methods have been developed, most of them however suffer from drawbacks such as use of corrosive/toxic metal-based catalysts, volatile organic solvents, unsatisfactory yields, high cost, longer reaction times and harsh reaction conditions. Thus, there is still scope for the development of new and more efficient methods for the synthesis of these important heterocycles keeping in mind the green principle of chemistry.

The thrust for clean, green synthetic processes requires atom economy, minimal waste generation, environmentally benign reagents & solvents as the prime ingredients and water serves as the most preferred solvent whenever possible. The low solubility of organic reagents and the instability of the majority of catalysts in water mediated reaction is a concern. But in recent time, many surfactants have been used as phase transfer catalysts in a number of organic reactions having unique capabilities to dissolve both organic and aqueous solutions to enhance the rate of the reaction. The anionic surfactant sodium dodecyl sulfate (SDS), shows the catalytic activity almost equal with Brønsted acids, such as sodium hydrosulfate or p-toluene sulfonic acid. Moreover, its catalytic activity is also known be greater than that of most Lewis acids and therefore, SDS has been employed as catalyst in a variety of reaction schemes. In the reaction medium SDS forms micelles in water and can both solubilise the organic compounds and catalyse the reaction thus increasing the energy efficiency.

Therefore, in quest of improved green reaction protocol for the synthesis of the biologically important 3,4-dihydropyrimidin-2(1H)-ones/thiones, SDS an eco-friendly and cheap catalytic system was explored. This chapter outline the findings of one-pot three-component synthesis of 3,4-dihydropyrimidin-2(1H)-ones/thiones derivatives catalysed by SDS in aqueous medium.
4.2 Results and Discussion

As a part of our effort towards the development of environmentally benign synthetic methods for multi-component reactions (MCRs) to synthesize various biologically important heterocyclic compounds, this chapter delineates an efficient synthesis of 3,4-dihydropyrimidin-2(1H)-ones/thiones derivatives (167/168) using SDS a surfactant in water at 60 °C via the condensation of aldehyde (7), ethyl acetoacetate (165)/methyl acetoacetate (166), and urea/thiourea (138/139) (Scheme 4.4).

\[
\begin{array}{c}
\text{RCH} = \text{NG} \\
\text{R}_1 = \text{CH}_3 \cdot \text{C}_2 \text{H}_5 \\
\text{X} = \text{O}, \text{S}
\end{array}
\]

Scheme 4.4 SDS catalysed synthesis of 3,4-dihydropyrimidin-2(1H)-ones/thiones derivatives.

In our initial attempt to synthesise the 3,4-dihydropyrimidin-2(1H)-ones/thiones derivatives, we selected the model reaction of 4-Chloro benzaldehyde (7a) (2.0 mmol), urea (138) (3.0 mmol) and ethyl acetoacetate (165) (2.1 mmol) to yield ethyl 4-(4-chlorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (167a). To investigate the effect of catalyst, the model reaction was carried out in absence of any catalyst which resulted in no product formation even after 18 hrs. The reaction was further heated for 24 hrs which resulted in only trace amount of the product. In the next attempt we employed SDS a surfactant as catalyst in the model reaction at room temperature in water which resulted in very less product formation even after stirring for 12 hrs. The reaction was then heated to 60 °C which furnished the desired product in a speculated time of 4 hrs. Encouraged by this result, we carried out the reaction of 4-methyl benzaldehyde (7h) (2.0 mmol), thiourea (139) (3.0 mmol) and ethyl acetoacetate (165) (2.1 mmol) under identical conditions. Upon stirring the reaction at 60 °C excellent yield (92%) of the product (167h) was obtained in little longer time (5hrs). The structure of the compounds formed were confirmed by \(^1\)H & \(^{13}\)C NMR, Mass and IR analyses.
In order to evaluate the catalyst loading, the model reaction was carried out at different catalyst loading, viz. no catalyst, 05 mol%, 10 mol%, 15 mol%, 20 mol%, and 25 mol% of SDS in water at 60 °C. Except in case of no catalyst condition the reaction worked in all cases. In case of 5 mol% to 15 mol% catalyst loadings the expected product formation was only up to 55 % upon stirring for 4 hrs. To our delight, it was observed that when catalyst loadings was increased to 20 mol% and 25 mol% the reaction yielded 85% and 91% yield respectively in similar time of 4 hrs. Interestingly, enhancement of catalyst loading to 30 mol% did not result in reduction of the reaction time appreciably and gave almost similar yields to that of optimum catalyst loading (25 mol %) in 4 hrs (Fig 4.2).

The efficiency of nonionic surfactant PEG-600, Tween-80 and cationic surfactant TBAB, CTAB were also screened in order to evaluate and compare the catalytic activity for the three-component model reaction in water. It is evident from Fig 4.3 TBAB and CTAB was also efficient enough to catalyse the reaction to yield 69% and 75% respectively whereas PEG-600 and Tween-80 gave poor result under similar reaction condition yielding only 42% and 35% respectively proving SDS as the best surfactant for the mentioned reaction yielding 91% of the desired product.

The effect of solvent was then investigated by carrying out the model reaction in different solvents like DCM, THF, chloroform, DMF, H₂O and in EtOH:H₂O (1:1) ratio at fixed catalyst loading (25 mol%). It was observed that the reaction was efficient in protic solvents like EtOH, H₂O, EtOH:H₂O, whereas in other solvent the reaction proceeded sluggishly (Fig. 4.4), thus confirming water as the best suited solvent for the micelle formation. Micelles can be used to promote multi-component reactions by taking advantage of the confinement effect as well as of the presence of certain functional groups on the surface of the aggregates to improve the yield and reaction time.

The catalyst recyclability was investigated for the model reaction (Fig.4.5). Catalyst was recovered from the reaction mixture by simple workup where the catalyst was retained in the aqueous layer leaving behind the product in organic layer. The regenerated catalyst in aqueous solution was then reused for the same reaction and it was observed the efficiency of catalyst did not change appreciably even after using for 4 consecutive times for the model reaction.
After the optimization of the reaction parameters such as catalyst, catalyst loading and solvent, the efficiency of the optimised protocol was tested for a number of aromatic aldehydes having varying substituent. In each case the reaction proceeded smoothly at optimum reaction condition to give the desired product in moderate to excellent yields. The presence of electron withdrawing or releasing substituent in the ortho-, meta- and para-positions do not appear to have any effect on the overall reaction yields (Table 4.1). While in case of thiourea derivatives product formation took longer time as compared to that of urea derivatives.
Table 4.1 SDS catalysed synthesis of dihydropyrimidinones derivatives

<table>
<thead>
<tr>
<th>Entry</th>
<th>X</th>
<th>Aldehyde</th>
<th>Producta</th>
<th>Time(hrs)</th>
<th>Yieldb</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>O</td>
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<td>167a</td>
<td>4</td>
<td>91</td>
</tr>
<tr>
<td>2</td>
<td>O</td>
<td><img src="167b.png" alt="Image" /></td>
<td>167b</td>
<td>4</td>
<td>89</td>
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<tr>
<td>3</td>
<td>O</td>
<td><img src="167c.png" alt="Image" /></td>
<td>167c</td>
<td>4</td>
<td>87</td>
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<tr>
<td>4</td>
<td>O</td>
<td><img src="167d.png" alt="Image" /></td>
<td>167d</td>
<td>4</td>
<td>90</td>
</tr>
<tr>
<td>5</td>
<td>O</td>
<td><img src="167e.png" alt="Image" /></td>
<td>167e</td>
<td>4</td>
<td>92</td>
</tr>
<tr>
<td>6</td>
<td>S</td>
<td><img src="167f.png" alt="Image" /></td>
<td>167f</td>
<td>5</td>
<td>88</td>
</tr>
<tr>
<td>7</td>
<td>S</td>
<td><img src="167g.png" alt="Image" /></td>
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<td>85</td>
</tr>
<tr>
<td>8</td>
<td>S</td>
<td><img src="167h.png" alt="Image" /></td>
<td>167h</td>
<td>5</td>
<td>91</td>
</tr>
<tr>
<td>9</td>
<td>S</td>
<td><img src="167i.png" alt="Image" /></td>
<td>167i</td>
<td>5</td>
<td>89</td>
</tr>
<tr>
<td>10</td>
<td>S</td>
<td><img src="167j.png" alt="Image" /></td>
<td>167j</td>
<td>5</td>
<td>86</td>
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</table>

Reaction Condition: aldehyde (2.0 mmol), ethyl acetoacetate (2.1 mmol), urea/thiourea (3.0 mmol), and SDS (25 mol%) at 60 °C.

aIsolated yields

bProducts were characterized by 1H & 13C NMR, Mass and IR analyses.

Likewise, almost identical results were obtained when methyl acetoacetate (166) was replaced in place of ethyl acetoacetate (165) and the three-component reaction with urea (138) and 4-Chloro benzaldehyde (7a) was carried out in presence of 25 mol% of SDS in water under similar condition. To study the scope of the reaction thiourea (139) and varying substituted aldehydes (7) were employed. The electrophilicity of the aldehydes resulting from the +I or -I effect of substituent on the phenyl ring did not much effected the overall reaction yield in all the cases (Table 4.2). In case of thiourea
derivatives also gave very good yields under similar reaction conditions, albeit taking longer times than their urea counterpart. The products were successfully isolated and purified by recrystallisation from ethanol without the use of chromatographic method.

The catalytic property of SDS to accelerate the rate of reaction can be explained as follows. In the above discussed reaction aldehyde, urea/thiourea, 1,3-diones condensed to produce dihydropyrimidinones derivatives which are hydrophobic in nature. Surfactant forms micelle at a concentration above its CMC, thus in the micellar solution of SDS the hydrophobic moieties escape away from water molecules thereby encircling the micelle core of SDS. Moreover, the reactant molecules are pushed by the water surrounding the micelle to enter inside the hydrophobic core of the micelle and resulting more efficient reactant collisions takes place to thus facilitating the reaction which can be schematically represented as Fig.4.6.

![Fig.4.6 Schematic representation of role of micellar SDS droplets on the reaction in aqueous media.](image)

Table 4.2 SDS catalysed synthesis of dihydropyrimidinones derivatives

<table>
<thead>
<tr>
<th>Entry</th>
<th>X</th>
<th>Aldehyde</th>
<th>Product</th>
<th>Time(hrs)</th>
<th>Yield&lt;sup&gt;b&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
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<td>Cl</td>
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<td>4</td>
<td>93</td>
</tr>
<tr>
<td>2</td>
<td>O</td>
<td>O&lt;sub&gt;N&lt;/sub&gt;</td>
<td>168b</td>
<td>4</td>
<td>89</td>
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Chapter 4  

SDS catalysed synthesis

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<thead>
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<th>O</th>
<th>168c</th>
<th>4</th>
<th>85</th>
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<tbody>
<tr>
<td>4</td>
<td>O</td>
<td>168d</td>
<td>4</td>
<td>90</td>
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<tr>
<td>5</td>
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<td>91</td>
</tr>
<tr>
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<td>168f</td>
<td>5</td>
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</tr>
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<td>5</td>
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<tr>
<td>9</td>
<td>S</td>
<td>168i</td>
<td>5</td>
<td>93</td>
</tr>
<tr>
<td>10</td>
<td>S</td>
<td>168j</td>
<td>5</td>
<td>86</td>
</tr>
</tbody>
</table>

Reaction Condition: aldehyde (2.0 mmol), methyl acetoacetate (2.1 mmol), urea/thiourea (3.0 mmol), and SDS (25 mol %) at 60 °C.  

Isolated yields  

Products were characterized by $^1$H & $^{13}$C NMR, Mass and IR analyses.

The plausible mechanism is depicted in Scheme 4.5. The reaction presumably proceeds via the reaction between aldehyde (A) with urea/thiourea (B) to form the acylimine intermediate D which is the key step, which subsequently reacts with the 1,3 diketone derivatives (E) followed by intramolecular cyclisation and dehydration afforded the corresponding 3,4-dihydropyrimidinones/thione derivatives (G)(167/168). The mechanism is in good agreement to that reported in the literature.

Scheme 4.5 Plausible mechanism for the formation of dihydropyrimidinones/thione.
In conclusion, an efficient, facile, one-pot three-component method for the synthesis of biologically important dihydropyrimidinones/thione derivatives via SDS a surfactant, a green and mild catalyst has been developed. The methodology offers high convergence, cost effectiveness and high throughput in short reaction time. In addition to this, use of environmentally safer reagents, solvent, recyclable catalysts, non chromatographic method of purifications are the add-on of this protocol which supersedes many pre-existing methodologies.

4.3 Experimental Section

All chemicals and reagents available commercially were purchased from Sigma Aldrich, Merck and were used without further purification. Purity of the products were confirmed by infrared (IR), $^1$H-NMR, $^{13}$C-NMR and mass spectra besides melting point data. Melting points were measured in open capillary tubes using Optics Technology melting point instrument and are uncorrected. IR spectra were recorded in KBr pellets on a Perkin Elmer Spectrum 400 FTIR instrument and the frequencies are expressed in cm$^{-1}$. $^1$H-NMR and $^{13}$C-NMR spectra were recorded on Bruker Avance II-400 spectrometer in DMSO-$d_6$/CDCl$_3$ (Chemical shifts in $\delta$ with TMS as internal standard). Mass spectral data were obtained with a JEOL D-300 (ESI) mass spectrometer. All reactions were monitored by thin layer chromatography (TLC) using precoated aluminum sheets (silica gel 60 F$_{254}$ 0.2-mm thickness).

**General procedure for the synthesis of dihydropyrimidinones/thione derivatives (167a-167j, 168a-168j)**

A mixture of aldehyde (7) (2.0 mmol), ethyl acetoacetate (165) /methyl acetoacetate (166) (2.1 mmol) and urea/tiourea (138/139) (3.0 mmol) in 5 mL millipore water was stirred at 60°C for appropriate time in the presence of SDS (25 mol %). The completion of the reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was worked up using ethyl acetate. The organic layer was dried over anhydrous Na$_2$SO$_4$ and concentrated in vacuo to give the solid product. The solid obtained was recrystallized from ethanol to give the pure product.
4.4 Physical, Spectral and Analytical data

**Ethyl 4-(4-chlorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167a)**

Appearance: White solid.

Yield: 91%.

Mp: 209–211°C.

\[ \text{IR} \nu_{\text{max}} \ (\text{KBr}) : 3457, 3248, 1706, 1647, 1604, 1226 \text{ cm}^{-1}; \ \text{\textsuperscript{1}H NMR} \ (400 \text{ MHz, CDCl}_3) : \delta_\text{H} \ (\text{ppm}) \ 1.17-1.21 \ (\text{m, 3H, CH}_3), \ 2.35 \ (\text{s, 3H, CH}_3), \ 4.20-4.23 \ (\text{m, 2H, CH}_2), \ 5.16 \ (\text{s, 1H, 4H}), \ 7.29 \ (\text{d, 2H, } J = 8.0 \text{ Hz, Ar-H}), \ 7.37 \ (\text{d, 2H, } J = 8.0 \text{ Hz, Ar-H}), \ 9.92 \ (\text{s, 2H, NH}); \ \text{\textsuperscript{13}C NMR} \ (100 \text{ MHz, CDCl}_3+\text{DMSO-d}_6) : \delta_\text{C} \ (\text{ppm}) \ 13.67, 17.74, 53.61, 61.06, 99.07, 128.60, 128.90, 130.43, 130.57, 132.06, 139.34, 143.24, 148.01, 166.67; \ \text{MS (ES\textsuperscript{+}) calcd. for C}_{14}\text{H}_{15}\text{ClN}_2\text{O}_3 : 294.08 found m/z 295.10 (M + H\textsuperscript{+}).

**Ethyl 4-(4-methoxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167b)**

Appearance: Yellow solid.

Yield: 89%.

Mp: 202–204°C.

\[ \text{IR} \nu_{\text{max}} \ (\text{KBr}) : 3449, 3249, 1724, 1658, 1619, 1219 \text{ cm}^{-1}; \ \text{\textsuperscript{1}H NMR} \ (400 \text{ MHz, CDCl}_3+\text{DMSO-d}_6) : \delta_\text{H} \ (\text{ppm}) \ 1.12-1.16 \ (\text{m, 3H, CH}_3), \ 2.27 \ (\text{s, 3H, CH}_3), \ 3.73 \ (\text{s, 3H, CH}_3), \ 3.97-4.00 \ (\text{m, 2H, CH}_2), \ 5.17 \ (\text{s, 1H, 4H}), \ 6.77 \ (\text{d, 2H, } J = 8.0 \text{ Hz, Ar-H}), \ 7.17 \ (\text{d, 2H, } J = 8.0 \text{ Hz, Ar-H}), \ 9.01 \ (\text{s, 1H, NH}), \ 9.84 \ (\text{s, 1H, NH}); \ \text{\textsuperscript{13}C NMR} \ (100 \text{ MHz, CDCl}_3+\text{DMSO-d}_6) : \delta_\text{C} \ (\text{ppm}) \ 13.74, 17.67, 53.50, 55.31, 60.42, 99.80, 114.09, 127.37, 136.81, 147.41, 152.52, 158.34, 165.28; \ \text{MS (ES\textsuperscript{+}) calcd. for C}_{15}\text{H}_{18}\text{N}_2\text{O}_4 : 290.13 found m/z 291.00 (M + H\textsuperscript{+}), 313.00 (M + Na\textsuperscript{+}).

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**Ethyl 4-(3-bromophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate**, (167c)

Appearance: White solid.

Yield: 87%.

Mp: 208–210°C.

IR\text{\nu} \text{max} (KBr): 3452, 3257, 1708, 1642, 1610, 1222 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)+DMSO-\(d_6\)): \(\delta_H \text{ (ppm)}\) 1.32-1.35 (m, 3H, CH\(_3\)), 2.40 (s, 3H, CH\(_3\)), 4.34-4.39 (m, 2H, CH\(_2\)), 4.92 (s, 1H, 4\(H\)), 7.23-7.49 (m, 3H, Ar-H), 7.54 (s, 1H, Ar-H), 10.00 (s, 2H, NH); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)+DMSO-\(d_6\)): \(\delta_C \text{ (ppm)}\) 13.42, 17.86, 54.00, 61.25, 99.96, 122.16, 125.80, 127.60, 128.10, 132.85, 140.97, 146.58, 152.53, 166.63; MS (ES\(^+\)) calcd. for C\(_{14}\)H\(_{15}\)BrN\(_2\)O\(_3\): 238.03 found m/z 239.11 (M + H)\(^+\).

**Ethyl 4-(4-fluorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate**, (167d)

Appearance: White solid.

Yield: 90%.

Mp: 174–176°C.

IR\text{\nu} \text{max} (KBr): 3443, 3248, 1728, 1711, 1687, 1227 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)+DMSO-\(d_6\)): \(\delta_H \text{ (ppm)}\) 1.16-1.24 (m, 3H, CH\(_3\)), 2.32 (s, 3H, CH\(_3\)), 4.04-4.10 (m, 2H, CH\(_2\)), 4.90 (s, 1H, 4\(H\)), 6.94 (d, 2H, \(J = 8.0 \text{ Hz, Ar-H}\)), 7.23 (d, 2H, \(J = 8.0 \text{ Hz, Ar-H}\)), 8.06 (s, 1H, NH), 8.65 (s, 1H, NH); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)+DMSO-\(d_6\)): \(\delta_C \text{ (ppm)}\) 13.77, 17.71, 53.40, 58.81, 101.84, 113.84, 114.80, 128.05, 128.98, 140.77, 145.32, 148.06, 159.24, 166.88; MS (ES\(^+\)) calcd. for C\(_{14}\)H\(_{15}\)FN\(_2\)O\(_3\): 278.11 found m/z 279.04 (M + H)\(^+\).
**Ethyl 4-(4-hydroxy-3-methoxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167e)**

Appearance: Off white solid.

Yield: 92%.

Mp: 232–234°C.

\[
\text{IR}_{\text{max}} \text{ (KBr): 3447, 3254, 1707, 1698, 1646, 1277 cm}^{-1}; \ \text{^1H NMR (400 MHz, CDCl}_3+\text{DMSO-}d_6\text{): } \delta_{\text{H}} \text{ (ppm) 1.17-1.20 (m, 3H, CH}_3\text{), 2.32 (s, 3H, CH}_3\text{), 3.82 (s, 3H, CH}_3\text{), 4.02-4.08 (m, 2H, CH}_2\text{), 5.22 (s, 1H, 4H), 6.72-6.77 (m, 2H, Ar-H), 6.85 (s, 1H, Ar-H), 7.20 (s, 1H, OH), 7.77 (s, 1H, NH), 8.87 (s, 1H, NH); ^13C NMR (100 MHz, CDCl}_3+\text{DMSO-}d_6\text{): } \delta_{\text{C}} \text{ (ppm) 13.89, 17.72, 53.84, 55.37, 59.00, 99.87, 110.34, 114.86, 118.40, 135.78, 145.45, 146.96, 147.22, 152.55, 165.48; MS (ES^+) calcd. for C_{15}H_{18}N_{2}O_{5} : 306.12 found m/z 307.16 (M + H)^+ , 329.13 (M+Na)^+ .}
\]

**Ethyl 6-methyl-4-(3-nitrophenyl)-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167f)**

Appearance: White solid.

Yield: 88%.

Mp: 203–206°C.

\[
\text{IR}_{\text{max}} \text{ (KBr): 3457, 3176, 1732, 1630, 1591, 1211 cm}^{-1}; \ \text{^1H NMR (400 MHz, CDCl}_3+\text{DMSO-}d_6\text{): } \delta_{\text{H}} \text{ (ppm) 1.37-1.35 (m, 3H, CH}_3\text{), 2.26 (s, 3H, CH}_3\text{), 3.87-3.93 (m, 2H, CH}_2\text{), 5.44 (s, 1H, 4H), 7.59-7.71 (m, 2H, Ar-H), 8.14-8.17 (m, 1H, Ar-H), 8.75 (s, 1H, Ar-H), 9.71 (s, 1H, NH), 10.19 (s, 1H, NH); ^13C NMR (100 MHz, CDCl}_3+\text{DMSO-}d_6\text{): } \delta_{\text{C}} \text{ (ppm) 13.76, 17.27, 51.47, 66.35, 99.97, 121.10, 122.31, 130.56, 134.99, 141.10, 147.54, 159.54, 168.21, 172.59; MS (ES^+) calcd. for C_{14}H_{15}N_{3}O_{4}S : 321.08 found m/z 322.11(M + H)^+ .}
\]
**Chapter 4**  
**SDS catalysed synthesis**

**Ethyl 4-(3,4-dimethoxyphenyl)-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167g)**

![Chemical structure](image)

Appearance: Yellow solid.

Yield: 85%.

Mp: 216–218°C.

**IR**ν<sub>max</sub> (KBr): 3427, 3238, 1711, 1645, 1608, 1234 cm<sup>−1</sup>; ¹H NMR (400 MHz, CDCl<sub>3</sub>+DMSO-d<sub>6</sub>): δ<sub>H</sub> (ppm) 1.22-1.28 (m, 3H, CH₃), 2.20 (s, 3H, CH₃), 3.87 (s, 3H, CH₃), 3.91 (s, 3H, CH₃), 4.09-4.14 (m, 2H, CH₂), 4.92 (s, 1H, 4H), 6.99 (d, 1H, J=8.0 Hz, Ar-H), 7.34 (s, 1H, Ar-H), 7.43-7.45 (M, 1H, Ar-H), 9.79 (s, 2H, NH); ¹³C NMR (100 MHz, CDCl<sub>3</sub>+DMSO-d<sub>6</sub>): δ<sub>C</sub> (ppm) 13.64, 17.62, 49.46, 55.40, 55.67, 60.62, 89.24, 110.21, 111.43, 123.98, 140.88, 145.53, 148.08, 158.94, 163.93, 171.68; MS (ES<sup>+</sup>) calcd. for C<sub>16</sub>H<sub>20</sub>N<sub>2</sub>O<sub>4</sub>S: 336.11 found m/z 337.20 (M + H)<sup>+</sup>.

**Ethyl 6-methyl-2-thioxo-4-(p-tolyl)-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167h)**

![Chemical structure](image)

Appearance: White solid.

Yield: 91%.

Mp: 188–190°C.

**IR**ν<sub>max</sub> (KBr): 3431, 3246, 1718, 1660, 1608, 1254 cm<sup>−1</sup>; ¹H NMR (400 MHz, CDCl<sub>3</sub>+DMSO-d<sub>6</sub>): δ<sub>H</sub> (ppm) 1.21-1.26 (m, 3H, CH₃), 2.20 (s, 3H, CH₃), 2.34 (s, 3H, CH₃), 4.24-4.28 (m, 2H, CH₂), 5.20 (s, 1H, 4H), 7.24 (d, 2H, J = 8.0 Hz, Ar-H), 7.73 (d, 2H, J = 8.0 Hz, Ar-H), 9.91 (s, 2H, NH); ¹³C NMR (100 MHz, CDCl<sub>3</sub>+DMSO-d<sub>6</sub>): δ<sub>C</sub> (ppm) 13.63, 17.17, 21.32, 54.11, 60.56, 101.15, 126.27, 128.52, 133.63, 140.87, 160.01, 165.48, 171.69; MS (ES<sup>+</sup>) calcd. for C<sub>15</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>S: 290.11 found m/z 291.01 (M + H)<sup>+</sup>.
Ethyl 4-(4-chlorophenyl)-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167i)

![Chemical structure image]

Appearance: White solid.

Yield: 89%.

Mp: 193–195°C.

\[\text{IR}_{\text{max}} \text{(KBr): 3449, 3250, 1724, 1700, 1646, 1277 cm}^{-1}; \] \[\text{H NMR (400 MHz, CDCl}_3+\text{DMSO-}d_6\): } \delta \text{H (ppm) 1.16-1.19 (m, 3H, CH}_3\), 2.30 (s, 3H, CH}_3\), 4.01- 4.04 (m, 2H, CH}_2\), 4.96 (s, 1H, 4H), 7.56 (d, 2H, J = 8.0 Hz, Ar-H), 8.08 (d, 2H, J = 8.0 Hz, Ar-H), 10.21 (s, 2H, NH); \] \[\text{C NMR (100 MHz, CDCl}_3+\text{DMSO-}d_6\): } \delta \text{C (ppm) 13.76, 17.83, 51.90, 59.59, 104.26, 123.52, 123.81, 128.33, 129.87, 130.29, 139.14, 160.33, 165.97, 170.08; MS (ES\textsuperscript{+}) calcd. for C\textsubscript{14}H\textsubscript{15}ClN\textsubscript{2}O\textsubscript{2}S : 310.05 found m/z 311.08 (M + H\textsuperscript{+}).

Ethyl 4-(4-methoxyphenyl)-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167j)

![Chemical structure image]

Appearance: Yellow solid.

Yield: 86%.

Mp: 155–157°C.

\[\text{IR}_{\text{max}} \text{(KBr): 3454, 3241, 1724, 1718, 1659, 1215 cm}^{-1}; \] \[\text{H NMR (400 MHz, CDCl}_3+\text{DMSO-}d_6\): } \delta \text{H (ppm) 1.13-1.17 (m, 3H, CH}_3\), 2.32 (s, 3H, CH}_3\), 3.37 (s, 3H, CH}_3\), 4.18- 4.20 (m, 2H, CH}_2\), 5.18 (s, 1H, 4H), 7.30 (d, 2H, J = 8.0 Hz, Ar-H), 7.75 (d, 2H, J = 8.0 Hz, Ar-H), 9.88 (s, 2H, NH); \] \[\text{C NMR (100 MHz, CDCl}_3+\text{DMSO-}d_6\): } \delta \text{C (ppm) 13.72, 18.16, 53.58, 60.14, 61.08, 100.56, 114.19, 128.76, 135.66, 157.78, 163.49, 166.70, 171.20; MS (ES\textsuperscript{+}) calcd. for C\textsubscript{15}H\textsubscript{18}N\textsubscript{2}O\textsubscript{3}S : 306.10 found m/z 307.02 (M + H\textsuperscript{+}).
**Methyl 4-(4-chlorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168a)**

Appearance: White solid.
Yield: 93%.
Mp: 203–205°C.

IR ν<sub>max</sub> (KBr): 3439, 3242, 1725, 1705, 1662, 1240 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>+DMSO-<em>d<sub>6</sub></em>): δ<sub>ν</sub> (ppm) 2.38 (s, 3H, CH<sub>3</sub>), 3.78 (s, 3H, CH<sub>3</sub>), 5.24 (s, 1H, 4H), 7.57 (d, 2H, <em>J</em> = 8.0 Hz, Ar-H), 7.80 (d, 2H, <em>J</em> = 8.0 Hz, Ar-H), 8.85 (s, 1H, NH), 9.93 (s, 1H, NH); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>+DMSO-<em>d<sub>6</sub></em>): δ<sub>C</sub> (ppm) 17.79, 52.06, 53.42, 98.91, 127.83, 127.97, 128.63, 128.94, 131.16, 139.87, 143.06, 148.29, 167.20; MS (ES<sup>+</sup>) calcd. for C<sub>13</sub>H<sub>13</sub>ClN<sub>2</sub>O<sub>3</sub>: 280.06 found m/z 281.04 (M + H)<sup>+</sup>.

**Methyl 6-methyl-4-(4-nitrophenyl)-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168b)**

Appearance: White solid.
Yield: 89%.
Mp: 253–255°C.

IR ν<sub>max</sub> (KBr): 3439, 3241, 1714, 1698, 1648, 1280 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>+DMSO-<em>d<sub>6</sub></em>): δ<sub>H</sub> (ppm) 2.28 (s, 3H, CH<sub>3</sub>), 3.82 (s, 3H, CH<sub>3</sub>), 5.26 (s, 1H, 4H), 8.10 (d, 2H, <em>J</em> = 8.0 Hz, Ar-H), 8.25 (d, 2H, <em>J</em> = 8.0 Hz, Ar-H), 8.34 (s, 2H, NH); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>+DMSO-<em>d<sub>6</sub></em>): δ<sub>C</sub> (ppm) 18.61, 52.16, 53.26, 100.75, 123.73, 131.29, 144.26, 145.77, 149.05, 152.01, 168.57; MS (ES<sup>+</sup>) calcd. for C<sub>13</sub>H<sub>13</sub>N<sub>3</sub>O<sub>5</sub>: 291.09 found m/z 292.14 (M + H)<sup>+</sup>.
Methyl 4-(3-fluorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168c)

Appearance: White solid.

Yield: 85%.

Mp: 194–196°C.

IR ν\text{max} (KBr): 3452, 3257, 1708, 1689, 1642, 1240 cm\textsuperscript{-1}; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}+DMSO-d\textsubscript{6}): \text{δ}_\text{H} (ppm) 2.29 (s, 3H, CH\textsubscript{3}), 3.70 (s, 3H, CH\textsubscript{3}), 5.20 (s, 1H, 4H), 6.89-7.09 (m, 3H, Ar-H), 7.40 (s, 1H, Ar-H), 9.85 (s, 2H, NH); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}+DMSO-d\textsubscript{6}): \text{δ}_\text{C} (ppm) 17.84, 52.06, 52.11, 99.03, 113.64, 115.10, 121.15, 130.29, 145.82, 146.70, 148.19, 163.29, 169.07; MS (ES\textsuperscript{+}) calcd. for C\textsubscript{13}H\textsubscript{13}FN\textsubscript{2}O\textsubscript{3}: 264.09 found m/z 265.15 (M + H\textsuperscript{+}).

Methyl 6-methyl-2-oxo-4-(3,4,5-trimethoxyphenyl)-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168d)

Appearance: White solid.

Yield: 90%.

Mp: 194–196°C.

IR v\text{max} (KBr): 3378, 3251, 1701, 1654, 1642, 1213 cm\textsuperscript{-1}; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}+DMSO-d\textsubscript{6}): \text{δ}_\text{H} (ppm) 2.60 (s, 3H, CH\textsubscript{3}), 3.75 (s, 3H, CH\textsubscript{3}), 3.87 (s, 6H, CH\textsubscript{3}), 3.96 (s, 3H, CH\textsubscript{3}), 5.51 (s, 1H, 4H), 6.60 (s, 2H, NH), 6.75 (s, 2H, Ar-H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}+DMSO-d\textsubscript{6}): \text{δ}_\text{C} (ppm) 17.70, 50.65, 53.72, 55.89, 60.10, 103.22, 106.54, 131.51, 142.74, 148.46, 152.64, 153.19, 165.84; MS (ES\textsuperscript{+}) calcd. for C\textsubscript{16}H\textsubscript{20}N\textsubscript{2}O\textsubscript{6}: 336.13 found m/z 337.18 (M + H\textsuperscript{+}).
**Methyl 6-methyl-2-oxo-4-(p-tolyl)-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168e)**

Appearance: White solid.

Yield: 91%.

Mp: 171–173°C.

\[
\text{IR } \nu_{\text{max}} (\text{KBr}): 3440, 3251, 1724, 1652, 1610, 1234 \text{ cm}^{-1}; \ \text{\textsuperscript{1}H NMR (400 MHz, CDCl}_3+\text{DMSO-d}_6): } \delta (\text{ppm}) 2.29 (s, 3H, CH}_3), 2.35 (s, 3H, CH}_3), 3.62 (s, 3H, CH}_3), 5.22 (s, 1H, 4H), 7.10 (d, 2H, J = 8.0 Hz, Ar-H), 7.15 (d, 2H, J = 8.0 Hz, Ar-H), 8.22 (s, 1H, NH), 9.16 (s, 1H, NH); \ \text{\textsuperscript{13}C NMR (100 MHz, CDCl}_3+\text{DMSO-d}_6): } \delta (\text{ppm}) 18.05, 22.01, 50.42, 50.62, 99.33, 126.01, 126.83, 128.4, 128.76, 136.37, 141.43, 145.52, 152.36, 167.54; \ \text{MS (ES}^+\text{) calcd. for C}_14\text{H}_16\text{N}_2\text{O}_3:\text{S : 260.12 found m/z 261.14 (M + H)}^+.\]

**Methyl 4-(4-methoxyphenyl)-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168f)**

Appearance: White solid.

Yield: 84%.

Mp: 148–150°C.

\[
\text{IR } \nu_{\text{max}} (\text{KBr}): 3439, 3242, 1734, 1678, 1628, 1245 \text{ cm}^{-1}; \ \text{\textsuperscript{1}H NMR (400 MHz, CDCl}_3+\text{DMSO-d}_6): } \delta (\text{ppm}) 2.25 (s, 3H, CH}_3), 3.45 (s, 3H, CH}_3), 3.59 (s, 3H, CH}_3), 4.87 (s, 1H, 4H), 6.77 (d, 2H, J = 8.0 Hz, Ar-H), 7.55 (d, 2H, J = 8.0 Hz, Ar-H), 9.57 (s, 2H, NH); \ \text{\textsuperscript{13}C NMR (100 MHz, CDCl}_3+\text{DMSO-d}_6): } \delta (\text{ppm}) 17.10, 51.99, 55.38, 59.60, 101.21, 114.15, 124.74, 135.47, 158.60, 161.39, 167.93, 173.99; \ \text{MS (ES}^+\text{) calcd. for C}_14\text{H}_16\text{N}_2\text{O}_3\text{S : 292.09 found m/z 293.17 (M + H)}^+.\]
Methyl 4-(4-bromophenyl)-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168g)

Appearance: White solid.
Yield: 92%.
Mp: 190–191°C.

IR\textsubscript{\nu max} (KBr): 3462, 3236, 1736, 1718, 1638, 1233 cm\textsuperscript{-1}; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}+DMSO-\textit{d}_6): \delta_H (ppm) 2.25 (s, 3H, CH\textsubscript{3}), 3.75 (s, 3H, CH\textsubscript{3}), 5.08 (s, 1H, 4\textit{H}), 6.94 (d, 2H, \textit{J} = 8.0 Hz, Ar-H), 7.49 (d, 2H, \textit{J} = 8.0 Hz, Ar-H), 9.65 (s, 1H, NH), 9.67 (s, 1H, NH); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}+DMSO-\textit{d}_6): \delta_C (ppm) 18.06, 49.15, 51.58, 100.73, 121.54, 128.93, 129.14, 130.67, 130.81, 139.90, 158.65, 167.18, 170.14; MS (ES\textsuperscript{+}) calcd. for C\textsubscript{13}H\textsubscript{13}BrN\textsubscript{2}O\textsubscript{2}S: 339.99 found m/z 340.84 (M + H\textsuperscript{+}).

Methyl 4-(3,4-dimethoxyphenyl)-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168h)

Appearance: yellow solid.
Yield: 83%.
Mp: 192–194°C.

IR\textsubscript{\nu max} (KBr): 3390, 3172, 1675, 1631, 1600, 1238 cm\textsuperscript{-1}; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}+DMSO-\textit{d}_6): \delta_H (ppm) 2.08 (s, 3H, CH\textsubscript{3}), 3.76 (s, 3H, CH\textsubscript{3}), 3.85 (s, 3H, CH\textsubscript{3}), 3.89 (s, 3H, CH\textsubscript{3}), 5.01 (s, 1H, 4\textit{H}), 6.97 (d, 1H, \textit{J} = 8.0 Hz, Ar-H), 7.31 (s, 1H, Ar-H), 7.43 (d, 1H, \textit{J} = 8.0 Hz, Ar-H), 9.77 (s, 2H, NH); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}+DMSO-\textit{d}_6): \delta_C (ppm) 18.90, 53.61, 59.14, 60.67, 60.93, 100.48, 113.88, 115.49, 134.70, 147.02, 154.21, 159.19, 169.28, 174.41; MS (ES\textsuperscript{+}) calcd. for C\textsubscript{15}H\textsubscript{18}N\textsubscript{2}O\textsubscript{4}S: 322.10 found m/z 323.17 (M + H\textsuperscript{+}).
**Methyl 6-methyl-4-(4-nitrophenyl)-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168i)**

![Chemical Structure](image)

Appearance: Off white solid.

Yield: 93%.

Mp: 235–237°C.

**IR v<sub>max</sub> (KBr):** 3432, 3219, 1714, 1659, 1632, 1269 cm<sup>-1</sup>; ¹H NMR (400 MHz, CDCl<sub>3</sub>+DMSO-d<sub>6</sub>): δ<sub>H</sub> (ppm) 2.27 (s, 3H, CH<sub>3</sub>), 3.78 (s, 3H, CH<sub>3</sub>), 5.12 (s, 1H, 4H), 7.57 (d, 2H, J = 8.0 Hz, Ar-H), 8.16 (d, 2H, J = 8.0 Hz, Ar-H), 10.12 (s, 2H, NH); ¹³C NMR (100 MHz, CDCl<sub>3</sub>+DMSO-d<sub>6</sub>): δ<sub>C</sub> (ppm) 18.13, 53.28, 59.14, 100.23, 125.65, 125.78, 128.13, 128.24, 145.29, 150.21, 158.61, 167.38, 173.24; MS (ES<sup>+</sup>) calcd. for C<sub>13</sub>H<sub>13</sub>N<sub>3</sub>O<sub>4</sub>S: 307.06 found m/z 308.04 (M + H)<sup>+</sup>.

**Methyl 4-(4-fluorophenyl)-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (168j)**

![Chemical Structure](image)

Appearance: Off white solid.

Yield: 86%.

Mp: 215–217°C.

**IR v<sub>max</sub> (KBr):** 3413, 3238, 1714, 1689, 1637, 1238 cm<sup>-1</sup>; ¹H NMR (400 MHz, CDCl<sub>3</sub>+DMSO-d<sub>6</sub>): δ<sub>H</sub> (ppm) 2.24 (s, 3H, CH<sub>3</sub>), 3.75 (s, 3H, CH<sub>3</sub>), 5.24 (s, 1H, 4H), 7.25 (d, 2H, J = 8.0 Hz, Ar-H), 7.35 (d, 2H, J = 8.0 Hz, Ar-H), 9.97 (s, 1H, NH), 10.36 (s, 1H, NH); ¹³C NMR (100 MHz, CDCl<sub>3</sub>+DMSO-d<sub>6</sub>): δ<sub>C</sub> (ppm) 18.19, 52.28, 58.14, 100.13, 116.65, 117.18, 128.74, 128.84, 139.10, 158.15, 160.25, 167.18, 174.14; MS (ES<sup>+</sup>) calcd. for C<sub>13</sub>H<sub>13</sub>FN<sub>2</sub>O<sub>2</sub>S: 280.07 found m/z 281.10 (M + H)<sup>+</sup>.
4.5 Representative $^1$HNMR, $^{13}$CNMR, IR and Mass Spectra

$^1$H NMR Spectrum of Ethyl 4-(4-hydroxy-3-methoxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167e)
$^{13}$C NMR Spectrum of Ethyl 4-(4-hydroxy-3-methoxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167e)
IR Spectrum of Ethyl 4-(4-hydroxy-3-methoxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167e)
Mass Spectrum of Ethyl 4-(4-hydroxy-3-methoxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (167e)
4.6 References


Chapter 4  

**SDS catalysed synthesis**


