Section C

7.0 Chemistry

The synthetic route for substituted 2-[(1H-benzo[d]imidazol-2-yl)amino]-pyrimidine derivatives are shown in Scheme 2. The known 2-benzimidazolylguanidine \( 53a,b \) was synthesized from substituted \( o \)-phenylenediamine \( 51a,b \) and cyanoguanidine \( 52 \) by following literature procedure.\(^9^4\) Compound \( 53 \) was treating with tri ethyl orthoformate \( \text{CH(OC}_2\text{H}_5)_3 \) and active methylene compounds containing carbonyl function \( 54, 1,3 \)-diketones \) to furnish substituted 2-[(1H-benzo[d]imidazol-2-yl)amino]-pyrimidine \( 55a-h \). The synthetic route for carboxamide derivatives are shown in Scheme 3. The substituted acetoacanilide \( 57 \) was directly prepared from substituted aniline \( 56 \) by our laboratory established method.\(^9^5\) The compounds \( 57 \) were reacted with tri ethyl orthoformate \( \text{CH(OC}_2\text{H}_5)_3 \) and 2-benzimidazolylguanidine \( 53a,b \) to give regioisomers intermediate \( 58a–n \). The cyclization of regioisomers intermediate was carried out by heating in glacial acetic acid with sodium acetate to furnish 2-[(1H-benzo[d]imidazol-2-yl)amino]-4-methyl-N-(substituted)-phenyl-pyrimidine-5-carboxamide derivatives \( 59a–n \). Table 7.1 and 7.2 show the yields and the physical data of these compounds.
7.1 Reaction Scheme

7.1.1 Scheme 1  Synthetic route for 2-benzimidazolylguanidine (53a, b).

\[
\begin{align*}
\text{51a, b} & \quad + \quad \text{52} \\
& \rightarrow \\
\text{53a, b} & \quad \text{HCl, reflux 3 h} \\
& \quad 10 \text{% KOH}
\end{align*}
\]

\(a; R^1 = H, \quad b; R^1 = Cl\)

7.1.2 Scheme 2  Synthetic routes for substituted 2-[(1H-benzo[d]imidazol-2-yl)amino]-pyrimidine derivatives (55a–h).

\[
\begin{align*}
\text{53} & \quad + \quad \text{54} \\
& \rightarrow \\
\text{55a-h} & \quad \text{Reflux 30 - 60 min} \\
& \quad \text{CH(OC}_2\text{H}_3}_3\text{, tri ethyl orthoformate}
\end{align*}
\]

\(a: R^1 = H; R^2 = R^3 = Me, \quad c: R^1 = Cl; R^2 = R^3 = Me, \quad e: R^1 = Cl; R^2 = Me; R^3 = OMe, \quad f: R^1 = Cl; R^2 = Me; R^3 = OMe, \quad g: R^1 = Cl; R^2 = Me; R^3 = OEt, \quad h: R^1 = H; R^2 = i\text{-Pr}; R^3 = OMe, \quad b: R^1 = H; R^2 = Me; R^3 = OEt, \quad d: R^1 = H; R^2 = CF}_3; R^3 = OEt, \quad i: R^1 = H; R^2 = i\text{-Pr}; R^3 = OMe, \quad j: R^1 = H; R^2 = Me; R^3 = OEt, \quad k: R^1 = H; R^2 = Me; R^3 = OEt, \quad l: R^1 = H; R^2 = Me; R^3 = OEt, \quad m: R^1 = H; R^2 = Me; R^3 = OEt, \quad n: R^1 = H; R^2 = Me; R^3 = OEt, \quad o: R^1 = H; R^2 = Me; R^3 = OEt, \quad p: R^1 = H; R^2 = Me; R^3 = OEt, \quad q: R^1 = H; R^2 = Me; R^3 = OEt, \quad r: R^1 = H; R^2 = Me; R^3 = OEt, \quad s: R^1 = H; R^2 = Me; R^3 = OEt, \quad t: R^1 = H; R^2 = Me; R^3 = OEt, \quad u: R^1 = H; R^2 = Me; R^3 = OEt, \quad v: R^1 = H; R^2 = Me; R^3 = OEt, \quad w: R^1 = H; R^2 = Me; R^3 = OEt, \quad x: R^1 = H; R^2 = Me; R^3 = OEt, \quad y: R^1 = H; R^2 = Me; R^3 = OEt, \quad z: R^1 = H; R^2 = Me; R^3 = OEt,
7.1.3 Scheme 3 Synthetic routes for 2-[(1H-benzo[d]imidazol-2-yl)amino]-4-methyl-N-(substituted)phenylpyrimidine-5-carboxamide derivatives (59a–n).
Table 7.1  Yields and physical data of the compounds 55a–h.

![55a-h](image)

<table>
<thead>
<tr>
<th>Compd.</th>
<th>Substitute</th>
<th>Yield %</th>
<th>MP °C</th>
<th>Analysis</th>
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<td>H Me Me</td>
<td>88</td>
<td>&gt;300</td>
<td>CHN</td>
</tr>
<tr>
<td>55b</td>
<td>H Me OMe</td>
<td>79</td>
<td>210–211</td>
<td>CHN</td>
</tr>
<tr>
<td>55c</td>
<td>H Me OEt</td>
<td>76</td>
<td>221–222</td>
<td>CHN</td>
</tr>
<tr>
<td>55d</td>
<td>H CF₃ OEt</td>
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<td>55h</td>
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Table 7.2  Yields and physical data of the compounds 59a–n.

![59a-n](image)

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</tr>
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<td>59b</td>
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<td>H</td>
<td>4-MeO</td>
<td>77</td>
<td>240–241</td>
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<tr>
<td>59d</td>
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<td>H</td>
<td>4-F</td>
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<td>H</td>
<td>2-Cl</td>
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<td>3-CF₃</td>
<td>61</td>
<td>272–273</td>
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</table>
7.2 Experimental

7.2.1 General methods and materials

All commercial chemicals and solvents were reagent grade and used without further purification unless otherwise specified. Melting points were determined on a Fargo melting point apparatus and are uncorrected. Thin-layer chromatography was performed on silica gel G60 F<sub>254</sub> (Merck) with short-wavelength UV light for visualization. All reported yields are isolated yields after chromatography or crystallization. Elemental analyses were done on a Heraeus CHN-O Rapid instrument. Mass spectra were recorded on Shimadzu GC-MS QP-2010 model using direct injection probe technique. The molecular ion peak was found in agreement with molecular weight of the respective compound. <sup>1</sup>H NMR spectra were recorded on a 400 MHz, Brucker Top-Spin spectrometers in the indicated solvent. The chemical shifts were reported in ppm (δ) relative to TMS and coupling constants (J) in Hertz (Hz) and s, d, t, m, brs, refer to singlet, doublet, triplet, multiplet, broad respectively.

All synthesized compounds were characterized by using <sup>1</sup>H NMR, <sup>13</sup>C NMR, Mass and Elemental analysis. For compounds 55a–h, the characteristic proton signals for pyrimidine ring (Ar-CH) appeared at the range of 8.96–9.09 δ ppm as singlet. The ethyl ester (OC<sub>2</sub>H<sub>5</sub>) proton appeared at the range of 1.31–1.35 and 4.30–4.34 δ ppm as triplet and quartet respective. The characteristic proton for benzimidazole ring and bridge (NH) appeared at the range of 11.87–12.24 δ ppm as singlet and D<sub>2</sub>O exchangeable single. While the compounds 59a–n, the characteristic proton signals for pyrimidine ring (Ar-CH) appeared at the range of 8.73–8.79 δ ppm as singlet and amide (CONH) proton appeared at the range of 10.19–10.51 δ ppm as singlet D<sub>2</sub>O exchangeable single. The characteristic proton for benzimidazole ring and bridge (NH) appeared at the range of 11.89–12.01 δ ppm as singlet and D<sub>2</sub>O exchangeable single. The molecular ion peak was found in agreement with molecular weight of the respective compound. The elemental analysis of the newly synthesized derivatives was within ±0.4% range of the calculated C, H, N data.
Synthesis of 2-benzimidazolylguanidine (53a). A mixture of o-phenylenediamine (51, 10.8 g, 100 mmol), cyanoguanidine (52, 8.4 g, 100 mmol) and concd HCl (20 mL) in H₂O (200 mL) was heated under reflux for 1 h. The reaction mixture was cooled at 0 °C and KOH (10%; 50 mL) was added slowly. The precipitates of 2-guanidinobenzimidazole were collected by filtration, washed with H₂O, dried, and used in next reactions without further purification. Yield 14 g (80 %); mp 240–242 °C (lit. mp 243–244 °C); MS $m/z = 175$ (M⁺).

By following the same synthetic procedure as that for 53a, the following compounds were synthesized:

5-Chloro-2-benzimidazolylguanidine (53b). Yield, 79 %; mp 258–259 °C. MS $m/z = 209$ (M⁺).

1-[2-((1H-Benz[d]imidazol-2-yl)amino)-4-methylpyrimidin-5-yl]ethanone (55a). A mixture of 2-guanidinobenzimidazole (53a, 1.75 g, 10 mmol), acetylacetone (54, 1 g, 10 mmol) and tri ethyl orthoformate (15 mL) was stirred at reflux temperature for 40 min. Upon the completion of the reaction (monitored by TLC, ethyl acetate:hexane (1:1)), the reaction mixture was concentrated under reduced pressure, and 1 mL of water was added. The separated solid product was collected by filtration and recrystallized from DMF to give 55a 2.3 g (88 %); mp > 300 °C (lit. mp > 300 °C); MS $m/z = 267$ (M⁺). Anal. Calcd. for (C₁₄H₁₃N₅O₄): C, 62.91; H, 4.90; N, 26.20. Found: C, 62.78; H, 4.98; N, 26.44.

By following the same synthetic procedure as that for 55a, the following compounds were synthesized:

Methyl-2-((1H-benzo[d]imidazol-2-yl)amino)-4-methylpyrimidine-5-carboxylate (55b). Yield, 79 %; mp 210–211 °C. $^1$H NMR (DMSO-$d₆$) δ 2.78 (3H, s, Me), 3.85 (3H, s, OMe), 7.09–7.11 (2H, m, 2 × ArH), 7.51–7.53 (2H, m, 2 × ArH), 8.96 (1H, s, ArH), 11.99 (2H, br s, exchangeable NH). MS $m/z = 283$ (M⁺). Anal. Calcd. for (C₁₄H₁₃N₅O₂): C, 59.36; H, 4.63; N, 24.72. Found: C, 59.69; H, 4.54; N, 24.88.
Ethyl-2-((1H-benzo[d]imidazol-2-yl)amino)-4-methylpyrimidine-5-carboxylate (55c). Yield, 76 %; mp 221–222 °C. $^1$H NMR (DMSO-$d_6$) $\delta$ 1.34 (3H, t, $J = 7.2$ Hz, Me), 2.77 (3H, s, Me), 4.31 (2H, q, $J = 7.2$ Hz, OCH$_2$), 7.08–7.10 (2H, m, 2 × ArH), 7.47–7.49 (2H, m, 2 × ArH), 8.96 (1H, s, ArH), 11.87 (2H, br s, exchangeable NH). MS $m/z$ = 297 (M$^+$). Anal. Calcd. for (C$_{15}$H$_{15}$N$_5$O$_2$): C, 60.60; H, 5.09; N, 23.56. Found: C, 60.34; H, 5.31; N, 23.41.

Ethyl-2-((1H-benzo[d]imidazol-2-yl)amino)-4-(trifluoromethyl)pyrimidine-5-carboxylate (55d). Yield, 72 %; mp 214–215 °C. $^1$H NMR (DMSO-$d_6$) $\delta$ 1.32 (3H, t, $J = 7.2$ Hz, Me), 4.31 (2H, q, $J = 7.2$ Hz, OCH$_2$), 7.15–7.17 (2H, m, 2 × ArH), 7.45–7.47 (2H, m, 2 × ArH), 9.09 (1H, s, ArH), 12.24 (2H, br s, exchangeable NH). $^{13}$C NMR (DMSO-$d_6$) $\delta$ 13.7, 61.5, 112.6, 112.8, 1211.8, 133.3, 148.9, 161.5, 161.7, 162.7. MS $m/z$ = 351 (M$^+$). Anal. Calcd. for (C$_{15}$H$_{12}$N$_5$O$_2$F$_3$): C, 51.29; H, 3.44; N, 19.94. Found: C, 51.46; H, 3.57; N, 19.73.

1-[2-((5-Chloro-1H-Benzol[d]imidazol-2-yl)amino)-4-methylpyrimidin-5-yl]ethanone (55e). Yield, 78 %; mp > 300 °C. MS $m/z$ = 301 (M$^+$). Anal. Calcd. for (C$_{14}$H$_{12}$N$_5$OCl): C, 55.73; H, 4.01; N, 23.21. Found: C, 55.46; H, 3.89; N, 23.34.

Methyl-2-((5-Chloro-1H-benzo[d]imidazol-2-yl)amino)-4-methylpyrimidine-5-carboxylate (55f). Yield, 74 %; mp 222–223 °C. MS $m/z$ = 317 (M$^+$). Anal. Calcd. for (C$_{14}$H$_{12}$N$_5$O$_2$Cl): C, 52.92; H, 3.81; N, 22.44. Found: C, 52.78; H, 3.98; N, 22.32.


2-((1H-Benz[\textit{d}]imidazol-2-yl)amino)-4-methyl-N-phenylpyrimidine-5-carboxamide (59a). A mixture of 2-guanidinobenzimidazole (53a, 1.75 g, 10 mmol), 3-oxo-N-phenylbutanamide (57, 1.8 g, 10 mmol) and tri ethyl orthoformate (15 mL) was stirred at reflux temperature for 30 min. The precipitates were collected by filtration and recrystallized from dioxane to give (58a). Compound 58a was added into a mixture of anhydrous sodium acetate (3 g) in glacial acetic acid (30 mL) and the reaction mixture was boiled for 30 min. The reaction mixture was cooled to room temperature and dropped into cold water (100 mL). The precipitates was collected by filtration and recrystallized from DMF to give 59a 2.8 g (82 %); mp 268–270 °C; MS m/z = 344 (M+). Anal. Calcd. for (C19H16N6O): C, 66.27; H, 4.68; N, 24.40. Found: C, 66.43; H, 4.47; N, 24.59.

By following the same synthetic procedure as that for 59a, the following compounds were synthesized:

2-((1H-Benz[\textit{d}]imidazol-2-yl)amino)-N-(2-methoxyphenyl)-4-methylpyrimidine-5-carboxamide (59b). Yield, 75 %; mp 248–249 °C. MS m/z = 374 (M+). Anal. Calcd. for (C20H18N6O2): C, 64.16; H, 4.85; N, 22.45. Found: C, 64.38; H, 4.77; N, 22.31.

2-((1H-Benz[\textit{d}]imidazol-2-yl)amino)-N-(4-methoxyphenyl)-4-methylpyrimidine-5-carboxamide (59c). Yield, 77 %; mp 240–241 °C. MS m/z = 374 (M+). Anal. Calcd. for (C20H18N6O2): C, 64.16; H, 4.85; N, 22.45. Found: C, 64.01; H, 4.67; N, 22.28.


2-((1H-Benz[\textit{d}]imidazol-2-yl)amino)-N-(4-fluorophenyl)-4-methylpyrimidine-5-carboxamide (59e). Yield, 79 %; mp 247–248 °C. 1H NMR (DMSO-\textit{d6}) ð 2.66 (3H, s, Me), 7.06–7.10 (2H, m, 2 × ArH), 7.19–7.23 (2H, m, 2 × ArH), 7.46–7.49 (2H, m, 2 × ArH), 7.73–7.77 (2H, m, 2 × ArH), 8.73 (1H, s, ArH), 10.51 (1H, br s, exchangeable CONH), 11.98 (2H, br s, exchangeable NH). MS m/z = 362 (M+). Anal.
Calcd. for (C$_{19}$H$_{15}$N$_{6}$OF): C, 62.98; H, 4.17; N, 23.19. Found: C, 62.80; H, 4.30; N, 23.01.

2-((1H-Benzol[d]imidazol-2-yl)amino)-N-(2-chlorophenyl)-4-methylpyrimidine-5-carboxamide (59f). Yield, 69 %; mp 252–253 °C. $^1$H NMR (DMSO-$d_6$) $\delta$ 2.71 (3H, s, Me), 7.08–7.11 (2H, m, 2 × ArH), 7.28–7.32 (1H, m, ArH), 7.39–7.43 (1H, m, ArH) 7.51–7.58 (3H, m, 3 × ArH), 7.71–7.73 (1H, m, ArH), 8.79 (1H, s, ArH), 10.19 (1H, br s, exchangeable CONH), 11.90 (2H, br s, exchangeable NH). $^{13}$C NMR (DMSO-$d_6$) $\delta$ 23.1, 121.1, 121.9, 127.8, 127.9, 128.2, 129.0, 129.9, 134.9, 148.3, 157.5, 158.5, 165.0, 168.0. MS m/z = 378 (M$^+$). Anal. Calcd. for (C$_{19}$H$_{16}$N$_{6}$OCl): C, 60.24; H, 3.99; N, 22.19. Found: C, 60.41; H, 3.78; N, 22.03.


2-((5-Chloro-1H-benzo[d]imidazol-2-yl)amino)-N-(3-chlorophenyl)-4-methylpyrimidine-5-carboxamide (59h). Yield, 74 %; mp 255–256 °C. MS m/z = 413 (M$^+$). Anal. Calcd. for (C$_{19}$H$_{14}$N$_{6}$OCl$_2$): C, 55.22; H, 3.41; N, 20.34. Found: C, 55.03; H, 3.21; N, 20.25.

2-((1H-Benzol[d]imidazol-2-yl)amino)-N-(4-bromophenyl)-4-methylpyrimidine-5-carboxamide (59i). Yield, 82 %; mp 262–263 °C. MS m/z = 423 (M$^+$). Anal. Calcd. for (C$_{19}$H$_{16}$N$_{6}$OBr): C, 53.91; H, 3.57; N, 19.86. Found: C, 53.74; H, 4.18; N, 19.73.

2-((1H-Benzol[d]imidazol-2-yl)amino)-N-(2-methylphenyl)-4-methylpyrimidine-5-carboxamide (59j). Yield, 72 %; mp 286–287 °C. MS m/z = 358 (M$^+$). Anal. Calcd. for (C$_{20}$H$_{18}$N$_{6}$O): C, 67.02; H, 5.06; N, 23.45. Found: C, 67.18; H, 5.19; N, 23.34.


### 7.3 Conclusion

In present chapter, first time reported three component condensations of benzoimidazole-2-guanidines, orthoester and active methylene carbonyl compounds leading to several novel new chemical entities substituted-(1H-benzo[d]imidazol-2-yl)amino-pyrimidine derivatives. The biological activity of newly synthesized compounds is under investigation.
7.4 Representative Spectra

7.4.1 Mass Spectrum for compound 53a.

7.4.2 $^1$H NMR Spectrum for compound 55b.
Chapter 7

7.4.3 $^1$H NMR Spectrum for compound 55c.

D$_2$O exchange $^1$H NMR Spectrum for compound 55c.
7.4.4 $^1$H NMR Spectrum for compound 55d.

D$_2$O exchange $^1$H NMR Spectrum for compound 55d.
7.4.5 \( ^1\text{H} \) NMR Spectrum for compound 57.

7.4.6 \( ^1\text{H} \) NMR Spectrum for compound 59e.
D$_2$O exchange $^1$H NMR Spectrum for compound 59e.

7.4.7 $^1$H NMR Spectrum for compound 59f.
D$_2$O exchange $^1$H NMR Spectrum for compound 59f.

7.4.8 $^{13}$C NMR Spectrum for compound 55d.
7.4.9  $^{13}$C NMR Spectrum for compound 59f.

![13C NMR Spectrum](image)

7.4.10  Mass Spectrum for compound 55a.

![Mass Spectrum](image)
7.4.11 Mass Spectrum for compound 55g.

7.4.12 Mass Spectrum for compound 55h.
7.4.13 Mass Spectrum for compound 59e.

\[
\text{MW: 362.36}
\]

7.4.14 Mass Spectrum for compound 59g.

\[
\text{MW: 412.06}
\]
7.4.15 Mass Spectrum for compound 59j.

[Diagram of Mass Spectrum for compound 59j]

MW: 358.40

7.4.16 Mass Spectrum for compound 59k.

[Diagram of Mass Spectrum for compound 59k]

MW: 392.84
7.4.17 Mass Spectrum for compound 59l.

7.4.18 Mass Spectrum for compound 59m.
7.4.19 Mass Spectrum for compound 59n.
Table 7.3  Elemental analysis of compounds 55a–h and 59a–n.

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<th>CHN Found (%)</th>
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</tr>
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Section C

References
References


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