Supporting Information

Eco-friendly syntheses of 2-substituted benzoxazoles and 2-substituted benzothiazoles from 2-aminophenols, 2-aminothiophenols and DMF derivatives in the presence of imidazolium chloride

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1. General information

All reagents were purchased from Ltd. (Shenzhen, China), Meyer Reagent Co., Ltd. (Shanghai, China), Macklin Reagent Co., Ltd. (Shanghai, China), Chongqing Chuandong Chemical Co., Ltd. (Chongqing, China), etc., and used without further purification. \textsuperscript{1}H and \textsuperscript{13}C NMR spectra were recorded on a Bruker AvanceIII NMR spectrometer (600MHz) in CDCl\textsubscript{3} internally referenced to tetramethylsilane (TMS) or CDCl\textsubscript{3} signals. Chemical shifts are reported in ppm and coupling constants (J) in Hz. Chromatography was carried out on silica gel (200-300 mesh, Merck) using gravity flow. All substrates are known compounds according to the literature. \textsuperscript{1}H and \textsuperscript{13}C NMR spectra were recorded in CDCl\textsubscript{3} and DMSO-d\textsubscript{6} on a Bruker Ascend-III 600 MHz and 600 MHz spectrometer using TMS as an internal standard. The residual solvent signals were used as references and the chemical shifts converted to the TMS scale (CDCl\textsubscript{3} : δ H = 7.25-7.26 ppm, δ C = 77.23 ppm; DMSO-d\textsubscript{6} : δ H = 2.51 ppm, δ C = 39.51 ppm).

2. General Procedures

2.1 General procedure for the synthesis of benzoxazole derivatives (2a - 2d)

\[
\begin{align*}
\text{1a} & \quad \begin{array}{c}
\text{H} \\
\text{Cl} \quad (0.1\text{eq})
\end{array} \\
\text{2a}
\end{align*}
\]

A mixture of \textbf{1a} (0.6g, 5.5 mmol, 1 equiv), imidazolium chloride (0.17g, 1.65mmol, 0.3equiv) and N,N-dimethylacetamide 5ml was stirred at 140°C for 8h. When the reaction was completed. Water (15ml) and ethyl acetate (20ml) were added with stirring to the reaction mixture. The organic layer was extracted and dried over anhydrous Na\textsubscript{2}SO\textsubscript{4}, filtered and concentrated under reduced pressure. The resulting residue was purified by column chromatography on silica gel using PE/EA as eluent to give the target product \textbf{2a}. 
2.2 General procedure for the synthesis of benzoxazole derivatives (2a, 2c, 2g and 2h) and benzothiazole derivatives (4a-4d).

\[
\begin{array}{c}
\text{R} & \text{NH}_2 & + & \text{O} & \text{N} & \text{R}_1 \rightarrow \text{R} & \text{N} & \text{R}_1 \\
3a & & & 4a & \text{8-10h, 160\degree C} & \\
\end{array}
\]

A tube-type schlenk flask was charged with 1a (0.6g, 5.5 mmol, 1 equiv), imidazolium chloride (0.28g, 1.65 mmol, 0.5equiv) and N,N-dimethylacetamide 5ml was stirred at 160\degree C for 8h. When the reaction was completed. Water (15ml) and ethyl acetate (20ml) were added with stirring to the reaction mixture. The organic layer was extracted and dried over anhydrous Na$_2$SO$_4$, filtered and concentrated under reduced pressure. The resulting residue was purified by column chromatography on silica gel using PE/EA as eluent to give the corresponding product 2a.

2.3 General procedure for the synthesis of benzoxazole derivatives (2b, 2d, 2e, 2f, 2i) and benzothiazole derivatives (4e-4k)

\[
\begin{array}{c}
\text{R} & \text{OH} & + & \text{O} & \text{N} & \text{R}_1 \rightarrow \text{R} & \text{O} & \text{R}_1 \\
1a & & & 2a & \text{8-10h, 160\degree C} & \\
\end{array}
\]

To a mixture of 1a (0.6g, 4.8 mmol, 1equiv), imidazolium chloride (0.25g, 2.4 mmol, 0.5equiv) and N,N-dimethylbenzamide (1.43g, 9.6 mmol, 2 equiv) was added. The mixture was stirred at 160\degree C for 10h. after completion of the reaction.15ml water was added and the resulting mixture was extracted with 20ml EA thrice, and the combined organic layers were dried over anhydrous Na$_2$SO$_4$ and concentrated. The residue was purified by column chromatography on silica gel using PE/EA as eluent to obtain the pure desired product.

3. Characterization of products 2-substituted benzoxazoles and 2-substituted benzothiazoles.
2-methylbenzo[d]oxazole (2a): The product was obtained as yellow liquid in 80% yield. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.64 (d, $J = 7.2$ Hz, 1H), 7.45 (d, $J = 7.6$ Hz, 1H), 7.29 – 7.25 (m, 2H), 2.61 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 163.74, 150.94, 141.49, 124.39, 124.04, 119.38, 110.15, 14.46.

2-phenylbenzo[d]oxazole (2b): The product was obtained as white solid in 86% yield. MP: 101-103°C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 8.27 (d, $J = 6.3$ Hz, 2H), 7.80 – 7.78 (m, 1H), 7.60 – 7.58 (m, 1H), 7.53 (d, $J = 7.0$ Hz, 3H), 7.36 (dd, $J = 6.0$, 3.1 Hz, 2H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 162.02, 149.72, 140.96, 130.55, 127.90, 126.63, 126.07, 124.12, 123.59, 118.97, 109.58.

2, 6-dimethylbenzo[d]oxazole (2c): The product was obtained as yellow liquid in 86% yield. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.42 (d, $J = 8.1$ Hz, 1H), 7.17 (s, 1H), 7.01 (d, $J = 8.0$ Hz, 1H), 2.51 (s, 3H), 2.37 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 163.23, 151.26, 139.25, 134.71, 125.22, 118.70, 110.39, 21.63, 14.46.
6-methyl-2-phenylbenzo[d]oxazole (2d)\(^4\): The product was obtained as yellow solid in 83% yield. MP: 90-92°C. \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta 8.17 - 8.15\) (m, 2H), 7.57 (d, \(J = 8.1\) Hz, 1H), 7.44 (d, \(J = 1.6\) Hz, 3H), 7.31 (s, 1H), 7.10 (d, \(J = 8.1\) Hz, 1H), 2.43 (s, 3H).

\[
\begin{array}{c}
\text{N} \\
\text{OMe}
\end{array}
\]

2-(4-methoxyphenyl)benzo[d]oxazole (2e)\(^6\): The product was obtained as white solid in 88% yield. MP: 97-100. \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta 8.20\) (d, \(J = 8.5\) Hz, 2H), 7.74 (d, \(J = 8.7\) Hz, 1H), 7.56 (d, \(J = 6.8\) Hz, 1H), 7.32 (h, \(J = 6.7, 6.1\) Hz, 2H), 7.03 (d, \(J = 8.3\) Hz, 2H), 3.89 (s, 3H). \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta 163.17, 162.34, 150.66, 142.21, 129.42, 124.61, 124.43, 119.63, 114.37, 110.39, 55.47.

\[
\begin{array}{c}
\text{N} \\
\text{NO2}
\end{array}
\]

2-(4-nitrophenyl)benzo[d]oxazole (2f)\(^5\): The product was obtained as yellow solid in 52% yield. MP: 166-167°C. \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta 7.74\) (d, \(J = 6.9\) Hz, 2H), 7.52 (t, \(J = 7.5\) Hz, 2H), 7.42 (t, \(J = 7.8\) Hz, 2H), 7.30 - 7.26 (m, 2H). \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta 160.67, 151.04, 149.42, 141.90, 132.80, 128.42, 126.37, 125.25, 124.25, 120.70, 110.96.

\[
\begin{array}{c}
\text{N} \\
\text{O}
\end{array}
\]
2, 5-dimethylbenzo[d]oxazole(2g): The product was obtained as yellow liquid in 84% yield. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.42 (s, 1H), 7.30 (d, J = 22.9 Hz, 1H), 7.07 (d, J = 7.6 Hz, 1H), 2.60 (s, 3H), 2.44 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 163.85, 149.19, 141.68, 133.82, 125.43, 119.34, 109.51, 21.38, 14.49.

![2g](image)

5-bromo-2-methylbenzo[d]oxazole(2h): The product was obtained as yellow liquid in 87% yield. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.78 (d, J = 1.9 Hz, 1H), 7.39 (d, J = 8.6 Hz, 1H), 7.33 (d, J = 8.5 Hz, 1H), 7.33 (d, J = 8.5 Hz, 1H), 2.64 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 165.10, 149.94, 143.11, 127.45, 122.46, 116.83, 111.42, 14.56.

![2h](image)

5-bromo-2-phenylbenzo[d]oxazole(2i): The product was obtained as white solid in 80% yield. MP: 108-110 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 8.24 (d, J = 7.5 Hz, 2H), 7.91 (s, 1H), 7.58 – 7.52 (m, 3H), 7.46 (s, 2H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 164.16, 149.74, 143.64, 131.97, 128.99, 128.10, 127.78, 126.60, 122.96, 117.33, 111.81.

![2i](image)

2-methylbenzo[d]thiazole(4a): The product was obtained as yellow liquid in 82% yield. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.95 (d, J = 8.0 Hz, 1H), 7.80
(d, J = 7.3 Hz, 1H), 7.43 (t, J = 7.7 Hz, 1H), 7.34 – 7.31 (m, 1H), 2.81 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl₃) δ 166.90, 153.36, 135.64, 125.90, 124.68, 122.37, 121.38, 20.09.

6-chloro-2-methylbenzo[d]thiazole(4b)⁴: The product was obtained as yellow solid in 85% yield. MP: 79-82 °C. $^1$H NMR (600 MHz, CDCl₃) δ 7.80 (d, J = 8.6 Hz, 1H), 7.74 (s, 1H), 7.35 (d, J = 8.6 Hz, 1H), 2.78 (s, 3H). $^{13}$C NMR (151 MHz, CDCl₃) δ 167.78, 151.49, 136.64, 130.79, 126.85, 123.05, 121.08, 20.13.

5-chloro-2-methylbenzo[d]thiazole(4c)⁵: The product was obtained as white solid in 80% yield. MP: 60-62 °C. $^1$H NMR (600 MHz, CDCl₃) δ 7.93 (d, J = 2.0 Hz, 1H), 7.73 (d, J = 8.5 Hz, 1H), 7.33 (d, J = 6.5 Hz, 1H), 2.84 (s, 3H). $^{13}$C NMR (151 MHz, CDCl₃) δ 169.04, 154.13, 133.85, 132.00, 125.24, 122.29, 122.09, 20.23.

2-methyl-6-nitrobenzo[d]thiazole(4d)¹¹: The product was obtained as yellow solid in 75% yield. MP: 161-162 °C. $^1$H NMR (600 MHz, CDCl₃) δ 8.78 (s, 1H), 8.34 (d, J = 6.7 Hz, 1H), 8.04 (d, J = 8.9 Hz, 1H), 2.93 (s, 3H).
$^{13}$C NMR (151 MHz, CDCl$_3$) δ 173.30, 157.09, 144.81, 136.02, 122.65, 121.59, 118.01, 20.71. 

![Diagram of 4e]

**2-(4-methoxyphenyl)benzo[d]thiazole (4e)**: The product was obtained as white solid in 87% yield. MP: 119-120 °C. $^1$H NMR (600 MHz, CDCl$_3$) δ 8.04 (d, J = 8.6 Hz, 3H), 7.87 (d, J = 7.6 Hz, 1H), 7.47 (t, J = 7.4 Hz, 1H), 7.35 (t, J = 7.6 Hz, 1H), 7.00 (d, J = 8.8 Hz, 2H), 3.87 (s, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) δ 167.91, 161.96, 154.11, 134.80, 129.15, 126.36, 126.25, 124.83, 122.80, 121.53, 114.57, 114.39, 77.26, 77.13, 55.49. 

![Diagram of 4f]

**2-phenylbenzo[d]thiazole (4f)**: The product was obtained as white solid in 79% yield. MP: 111-113 °C. $^1$H NMR (600 MHz, CDCl$_3$) δ 8.12 – 8.08 (m, 3H), 7.91 (d, J = 8.0 Hz, 1H), 7.50 (p, J = 4.1 Hz, 4H), 7.39 (t, J = 7.6 Hz, 1H). $^{13}$C NMR (151 MHz, CDCl$_3$) δ 168.15, 153.99, 135.00, 133.52, 131.06, 129.07, 127.62, 126.39, 125.26, 123.23, 121.66.

![Diagram of 4g]

**2-(4-nitrophenyl)benzo[d]thiazole (4g)**: The product was obtained as yellow solid in 60% yield. MP: 224-226°C. $^1$H NMR (600 MHz, CDCl$_3$) δ 8.29 (d, J = 8.9 Hz, 2H), 8.21 (d, J = 8.8 Hz, 2H), 8.07 (d, J = 8.2 Hz, 1H), 7.89 (d, J = 7.8 Hz, 1H), 7.51 – 7.48 (m, 1H), 7.42 – 7.39 (m, 1H). $^{13}$C NMR
(151 MHz, CDCl$_3$) $\delta$ 163.82, 153.08, 148.02, 138.16, 134.46, 127.23, 125.90, 125.21, 123.30, 122.92, 120.82.

2-(4-chlorophenyl)benzo[d]thiazole (4h)$^{14}$: The product was obtained as white solid in 82% yield. MP: 114-115 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 8.07 (d, $J = 8.2$ Hz, 1H), 8.04–8.01 (m, 2H), 7.90 (dd, $J = 8.0$, 1.1 Hz, 1H), 7.50 (ddd, $J = 8.2$, 7.1, 1.2 Hz, 1H), 7.47–7.45 (m, 2H), 7.41–7.38 (m, 1H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 166.65, 154.02, 137.06, 135.04, 132.08, 129.29, 128.73, 126.52, 125.45, 123.30, 121.67.

2-(2-chlorophenyl)benzo[d]thiazole (4i)$^{2}$: The product was obtained as white solid in 79% yield. MP: 81-83 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 8.24–8.19 (m, 1H), 8.16–8.12 (m, 1H), 7.95 (dd, $J = 8.1$, 1.1 Hz, 1H), 7.57–7.51 (m, 2H), 7.47–7.39 (m, 3H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 164.20, 152.38, 136.05, 132.71, 132.19, 131.76, 131.17, 130.81, 127.12, 126.32, 125.47, 123.43, 121.40.

2-(pyridin-2-yl)benzo[d]thiazole (4j)$^{15}$: The product was obtained as white solid in 75% yield. MP: 133-135 °C. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 8.70 (d, $J = 4.0$ Hz, 1H), 8.39 (d, $J = 8.0$ Hz, 1H), 8.10 (d, $J = 8.2$ Hz, 1H), 7.97 (d, $J =...
7.6 Hz, 1H), 7.86 (t, J = 7.7 Hz, 1H), 7.51 (t, J = 7.0 Hz, 1H), 7.44 – 7.38 (m, 2H). $^{13}$C NMR (151 MHz, CDCl$_3$) δ 169.35, 154.22, 151.37, 149.65, 137.05, 136.11, 126.30, 125.67, 125.29, 123.57, 122.02, 120.81.

3-Benzothiazol-2-yl-1-phenyl-propan-1-one (4k) $^{17}$: The product was obtained as white solid in 82% yield. MP: 93-95 °C. $^1$H NMR (600 MHz, CDCl$_3$) δ 8.02 (d, J = 7.8 Hz, 2H), 7.95 (d, J = 8.1 Hz, 1H), 7.83 (d, J = 8.0 Hz, 1H), 7.57 (t, J = 7.4 Hz, 1H), 7.50 – 7.40 (m, 3H), 7.34 (t, J = 7.6 Hz, 1H), 3.66 (t, J = 7.1 Hz, 2H), 3.57 (t, J = 7.1 Hz, 2H). $^{13}$C NMR (151 MHz, CDCl$_3$) δ 197.87, 170.59, 153.09, 136.50, 135.24, 133.36, 128.69, 128.14, 125.97, 124.85, 122.52, 121.54, 37.64, 28.29.

4. References


5. $^1$H NMR and $^{13}$C NMR spectra of
Figure 1: $^1$H NMR spectra of 2-methylbenzo[d]oxazole (2a) (solvent CDCl$_3$)
Figure 2 $^{13}$C NMR spectra of 2-methylbenzo[d]oxazole (2a) (solvent CDCl$_3$)

Figure 3 $^1$H NMR spectra of 2-phenylbenzo[d]oxazole (2b) (solvent CDCl$_3$)
Figure 4 $^{13}$C NMR spectra of 2-phenylbenzo[d]oxazole (2b) (solvent CDCl$_3$)
Figure 5 $^1$H NMR spectra of 2, 6-dimethylbenz[d]oxazole(2c) (solvent CDCl$_3$)

![H NMR spectra](image)

Figure 6 $^{13}$C NMR spectra of 2, 6-dimethylbenz[d]oxazole(2c) (solvent CDCl$_3$)

![C NMR spectra](image)
Figure 7: $^1$H NMR spectra of 6-methyl-2-phenylbenzo[d]oxazole (2d) (solvent CDCl$_3$)
Figure 8: $^{13}$C NMR spectra of 6-methyl-2-phenylbenzo[d]oxazole (2d) (solvent CDCl$_3$)

Figure 9: $^1$H NMR spectra of 2-(4-methoxyphenyl)benzo[d]oxazole (2e) (solvent CDCl$_3$)
Figure 10 $^{13}$C NMR spectra of 2-(4-methoxyphenyl)benzo[d]oxazole (2e) (solvent CDCl$_3$)
Figure 11 ¹H NMR spectra of 2-(4-nitrophenyl)benzo[d]oxazole (solvent CDCl₃)

Figure 12 ¹³C NMR spectra of 2-(4-nitrophenyl)benzo[d]oxazole (solvent CDCl₃)
Figure 13. $^1$H NMR spectra of 2, 5-dimethylbenzo[d]oxazole (2g) (solvent CDCl$_3$)
Figure 14 $^{13}$C NMR spectra of 2, 5-dimethylbenzo[d]oxazole (solvent CDCl$_3$)

![13C NMR spectrum of 2, 5-dimethylbenzo[d]oxazole](image)

Figure 15 $^1$H NMR spectra of 5-bromo-2-methylbenzo[d]oxazole (solvent CDCl$_3$)

![1H NMR spectrum of 5-bromo-2-methylbenzo[d]oxazole](image)
Figure 16. $^{13}$C NMR spectra of 5-bromo-2-methylbenzo[d]oxazole (2h) (solvent CDCl$_3$)
Figure 17 \( ^1H \) NMR spectra of 5-bromo-2-phenylbenzo[d]oxazole(2i) (solvent CDCl$_3$)

Figure 18 \( ^13C \) NMR spectra of 5-bromo-2-phenylbenzo[d]oxazole(2i) (solvent CDCl$_3$)
Figure 19: $^1$H NMR spectra of 2-methylbenzo[d]thiazole (4a) (solvent CDCl$_3$).
Figure 20 $^{13}$C NMR spectra of 2-methylbenzo[d]thiazole(4a) (solvent CDCl$_3$)

Figure 21 $^1$H NMR spectra of 6-chloro-2-methylbenzo[d]thiazole(4b) (solvent CDCl$_3$)
Figure 22. $^{13}$C NMR spectra of 6-chloro-2-methylbenzo[d]thiazole (4b) (solvent CDCl$_3$).
Figure 23: $^1$H NMR spectra of 5-chloro-2-methylbenzo[d]thiazole(4c) (solvent CDCl$_3$)

Figure 24: $^{13}$C NMR spectra of 5-chloro-2-methylbenzo[d]thiazole(4c) (solvent CDCl$_3$)
Figure 25: $^1$H NMR spectra of 2-methyl-6-nitrobenzo[d]thiazole (solvent CDCl$_3$)
Figure 26 ¹³C NMR spectra of 2-methyl-6-nitrobenzo[d]thiazole (solvent CDCl₃)

Figure 27 ¹H NMR spectra of 2-(4-methoxyphenyl)benzo[d]thiazole (solvent CDCl₃)
Figure 28 $^{13}$C NMR spectra of 2-(4-methoxyphenyl)benzo[d]thiazole (4e) (solvent CDCl$_3$)
Figure 29 $^1$H NMR spectra of 2-phenylbenzo[d]thiazole (4f) (solvent CDCl$_3$)

Figure 30 $^{13}$C NMR spectra of 2-phenylbenzo[d]thiazole (4f) (solvent CDCl$_3$)
Figure 31 $^1$H NMR spectra of 2-(4-nitrophenyl)benzo[d]thiazole (4g) (solvent CDCl$_3$)

Figure 32 $^{13}$C NMR spectra of 2-(4-nitrophenyl)benzo[d]thiazole (4g) (solvent CDCl$_3$)
Figure 33. $^1$H NMR spectra of 2-(4-chlorophenyl)benzo[d]thiazole (4h) (solvent CDCl₃)
Figure 34 $^{13}$C NMR spectra of 2-(4-chlorophenyl)benzo[d]thiazole (4h) (solvent CDCl$_3$)

Figure 35 $^1$H NMR spectra of 2-(2-chlorophenyl)benzo[d]thiazole (4i) (solvent CDCl$_3$)
Figure 36: $^{13}$C NMR spectra of 2-(2-chlorophenyl)benzo[d]thiazole(4i) (solvent CDCl$_3$)
Figure 37 $^1$H NMR spectra of 2-(pyridin-2-yl)benzo[d]thiazole(4j) (solvent CDCl$_3$)

Figure 38 $^{13}$C NMR spectra of 2-(pyridin-2-yl)benzo[d]thiazole(4j) (solvent CDCl$_3$)
Figure 39: $^1$H NMR spectra of 3-Benzothiazol-2-yl-1-phenyl-propan-1-one (4k) (solvent CDCl$_3$)
Figure 40 $^{13}$C NMR spectra of 3-Benzothiazol-2-yl-1-phenyl-propan-1-one (4k) (solvent CDCl$_3$)