Transition-metal-free Regioselective Construction of 1,5-Diaryl-1,2,3-triazoles through Dehydrative Cycloaddition of Alcohols with Arylazides Mediated by SO$_2$F$_2$

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1. General Information

Unless otherwise specified, NMR spectra were recorded in CDCl$_3$ on a 500 MHz (for
1H), 471 MHz (for 19F), 126 MHz (for 13C) spectrometer. All chemical shifts were reported in ppm relative to TMS (1H NMR, 0 ppm) as internal standards. The HPLC experiments were carried out on a Waters e2695 instrument (column: J&K, RP-C18, 5 μm, 4.6 × 150 mm), and the yields of the products were determined by using the corresponding pure compounds as the external standards. The coupling constants were reported in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, dd = doublet of doublets. HRMS experiments were performed on a TOF-Q ESI or CI/EI instrument. Melting points were measured and uncorrected. Reagents used in the reactions were all purchased from commercial sources and used without further purification.

2. Screening the Optimized Reaction Conditions for One-Pot Synthesis of 1,5-
Disubstituted Triazole

Table 1 Screening the base

<table>
<thead>
<tr>
<th>Entry</th>
<th>Base (2.0 eq.)</th>
<th>Yield (3a, %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>t-BuOK</td>
<td>40</td>
</tr>
<tr>
<td>2</td>
<td>CsF</td>
<td>&lt;5</td>
</tr>
<tr>
<td>3</td>
<td>KOH</td>
<td>25</td>
</tr>
<tr>
<td>4</td>
<td>KNO3</td>
<td>n.d.</td>
</tr>
</tbody>
</table>
Reaction conditions: 4-biphenylethanol (1a, 0.4 mmol), K$_2$CO$_3$ (0.8 mmol, 2.0 eq.), DMSO (3.0 mL) were added to a reaction tube (10 mL) and SO$_2$F$_2$ was introduced into the reaction mixture through a balloon before reacting at room temperature for 2 h. The SO$_2$F$_2$ balloon was removed from reaction, phenyl azide (2a, 2 eq.), another base (2.0 eq.) and DMSO (0.5 mL) were subsequently added, then the reaction mixture was allowed to stir at room temperature for 12 h under argon atmosphere.

The yield was determined by HPLC using 3a as the external standard ($t_R$ = 5.509 min, $\lambda_{\text{max}}$ = 276.0 nm, acetonitrile/water = 70 : 30 (v / v)).

**Table 2 Screening the amount of base**

<table>
<thead>
<tr>
<th>Entry</th>
<th>t-BuOK (X eq.)</th>
<th>Yield (3a, %)$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.0</td>
<td>&lt;5</td>
</tr>
<tr>
<td>2</td>
<td>2.0</td>
<td>40</td>
</tr>
<tr>
<td>3</td>
<td>3.0</td>
<td>82</td>
</tr>
<tr>
<td>4</td>
<td>4.0</td>
<td>87</td>
</tr>
<tr>
<td>5</td>
<td>4.5</td>
<td>83</td>
</tr>
<tr>
<td>6</td>
<td>5.0</td>
<td>68</td>
</tr>
</tbody>
</table>

*Reaction conditions: 4-biphenylethanol (1a, 0.4 mmol), K$_2$CO$_3$ (0.8 mmol, 2.0 eq.), DMSO (3.0 mL) were added to a reaction tube (10 mL) and SO$_2$F$_2$ was introduced into the reaction mixture through a balloon before reacting at room temperature for 2 h. The SO$_2$F$_2$ balloon was removed from reaction, phenyl azide (2a, 2 eq.), t-BuOK (X eq.) and DMSO (0.5 mL) were subsequently charged, then the reaction stirred at room temperature for 12 h under argon atmosphere. $^b$The yield was determined by HPLC using 3a as the external standard ($t_R$ = 5.509 min, $\lambda_{\text{max}}$ = 276.0 nm, acetonitrile/water = 70 : 30 (v / v)).
Table 3 Screening the amount of azidobenzene$^a$

<table>
<thead>
<tr>
<th>Entry</th>
<th>Ph-N$_3$ (X eq.)</th>
<th>Yield (3a, %)$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.0</td>
<td>77</td>
</tr>
<tr>
<td>2</td>
<td>1.5</td>
<td>82</td>
</tr>
<tr>
<td>3</td>
<td>2.0</td>
<td>84</td>
</tr>
<tr>
<td>4</td>
<td>3.0</td>
<td>73</td>
</tr>
</tbody>
</table>

$^a$Reaction conditions: 4-biphenylethanol (1a, 0.4 mmol), K$_2$CO$_3$ (0.8 mmol, 2.0 eq.), DMSO (3.0 mL) were added to a reaction tube (10 mL) and SO$_2$F$_2$ was introduced into the reaction mixture through a balloon before reacting at room temperature for 2 h. The SO$_2$F$_2$ balloon was removed from reaction, phenyl azide (2a, X eq.), t-BuOK (3 eq.) and DMSO (0.5 mL) were subsequently charged, then the reaction stirred at room temperature for 12 h under argon atmosphere. $^b$The yield was determined by HPLC using 3a as the external standard ($t_R = 5.509$ min, $\lambda_{\text{max}} = 276.0$ nm, acetonitrile/water = 70 : 30 (v/v)).

3. General Procedures.

3.1 General Procedure for Synthesis Aryl Ethanol.

1a and 1c were prepared according to the literatures.$^{[1,2]}$ 1j-1t were prepared according to the literatures.$^{[3-9]}$ Others were all purchased from commercial sources and used without further purification.

3.2 General Procedure for Synthesis Aryl Azides.

2ba-2bx were all prepared according to the literatures.$^{[10,11]}$ 2by was prepared according to the literature.$^{[12]}$ (Caution: extraction should never be performed with halogenated solvents such as DCM or CHCl$_3$ as these solvents can react with residual sodium azide to form diazidomethane or azidoform which are highly shock sensitive.
and energetic explosives.

![Chemical Reaction](image)

3.3 General Procedure for Synthesis 1,5-Disubstituted Triazole.

**Procedure A**

Aryl ethanol (1, 1.0 mmol), K₂CO₃ (2.0 mmol, 2.0 eq., 277 mg), DMSO (0.13 M) were added to a reaction tube (25 mL) and SO₂F₂ was introduced into the reaction mixture through a balloon before reacting at room temperature for 2 h, the SO₂F₂ balloon was removed from reaction, then azido benzene (2a, 1.5 mmol, 1.5 eq., 179 mg), t-BuOK (3 mmol, 3.0 eq., 337 mg) were subsequently added and the mixture was stirred at room temperature for 12 h under argon atmosphere before extracted with ethyl acetate (3×50 mL) and the combined organic layers was dried over anhydrous sodium sulfate, filtered and concentrated under reduced pressure. The crude products were further purified by column chromatography on silica gel by gradient elution with petroleum ether/ethyl acetate (85/15 to 50/50) as eluent to obtain pure triazoles 3.

**Procedure B**

Phenyl ethanol (1b, 1.0 mmol), K₂CO₃ (2.0 mmol, 2.0 eq., 277 mg), DMSO (0.13 M) were added to a reaction tube (25 mL) and SO₂F₂ was introduced into the reaction mixture through a balloon before reacting at room temperature for 2 h, the SO₂F₂ balloon was removed from reaction, then aryl azide (2, 1.5 mmol, 1.5 eq., 179 mg), t-
BuOK (3 mmol, 3.0 eq., 337 mg) were subsequently added and mixture was stirred at room temperature for 12 h under argon atmosphere. The reaction mixture was extracted with ethyl acetate (3×50 mL) and the combined organic layers was dried over anhydrous sodium sulfate, filtered and concentrated under reduced pressure. The crude products were further purified by column chromatography on silica gel by gradient elution with petroleum ether/ethyl acetate (85/15 to 50/50) as eluent to obtain pure triazoles 3.

3.4 General Procedure for Synthesis 1,5-Disubstituted Triazole through Alkyl Alcohol.

In first step, 4-phenylbutan-1-ol (1ag, 1.0 mmol), K$_2$CO$_3$ (1.2 mmol, 1.2 eq., 166 mg), DMSO (0.13 M) were added to a reaction tube (25 mL) and SO$_2$F$_2$ was introduced into the reaction mixture through a balloon before reacting at room temperature for 12 h, then added DBU (6 eq., 6 mmol, 914 mg) into the reaction tube and SO$_2$F$_2$ was introduced into the reaction mixture through a balloon stirred for another 12 h. The reaction mixture was extracted with ethyl acetate (3×50 mL) and the combined organic layers was dried over anhydrous sodium sulfate, filtered and concentrated under reduced pressure. The crude products were further purified by column chromatography on silica gel by gradient elution with petroleum ether/ethyl acetate (90/10 to 80/20) as eluent to obtain isomer 4.

In second step, 4-phenylbut-1-en-1-yl sulfurofluoridate (4, 1 mmol, 230.3 mg) was added to a reaction tube (25 mL), then t-BuOK (2 mmol, 2.0 eq., 225 mg) and azido benzene (2a, 1.5 mmol, 1.5 eq., 179 mg) were subsequently added at 0 ºC and mixture was stirred at room temperature for 12 h under argon atmosphere in mixed solvent DMSO : THF = 5:2 (v / v). The reaction mixture was extracted with ethyl acetate
(3×50 mL) and the combined organic layers was dried over anhydrous sodium sulfate, filtered and concentrated under reduced pressure. The crude products were further purified by column chromatography on silica gel by gradient elution with petroleum ether/ethyl acetate (85/15 to 50/50) as eluent to obtain pure triazoles 3ag.


5-([1,1'-biphenyl]-4-yl)-1-phenyl-1H-1,2,3-triazole (3a).

\[
\begin{align*}
\text{Ph} & \quad \text{N} \\
\text{N} & \quad \text{N}
\end{align*}
\]

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 214 mg, 72% yield, mp 157.8-159.0 °C.

\[\text{^1H NMR (500 MHz, CDCl}_3\text{) } \delta 7.92 \text{ (s, 1H), 7.59-7.57 (m, 4H), 7.47-7.41 (m, 7H), 7.37 (t, } J = 7.3 \text{ Hz, 1H), 7.30 (d, } J = 8.2 \text{ Hz, 2H).} \]

\[\text{^13C NMR (126 MHz, CDCl}_3\text{) } \delta 142.0, 139.9, 137.5, 136.7, 133.5, 129.5, 129.4, 128.98, 128.96, 128.0, 127.5, 127.0, 125.6, 125.3.}\]

\[\text{HRMS-ESI (m/z): [M+H]^+ \text{calcd for } C_{20}H_{15}N_3: 298.1266, \text{ found: } 298.1265.}\]

1,5-diphenyl-1H-1,2,3-triazole (3b). \[\text{[13]}\]
According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 190 mg, 86% yield, mp 104.0-106.0 °C (lit. 113-114 °C).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.86 (s, 1H), 7.44-7.41 (m, 3H), 7.38-7.32 (m, 5H), 7.23-7.21 (m, 2H).

1-phenyl-5-(p-tolyl)-1H-1,2,3-triazole (3c).  

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 183 mg, 78% yield, mp 142.0-143.5 °C (lit. 141-142 °C).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.84 (s, 1H), 7.45-7.43 (m, 3H), 7.38-7.36 (m, 2H), 7.16-7.11 (m, 4H), 2.36 (s, 3H).

5-(4-(tert-butyl) phenyl)-1-phenyl-1H-1,2,3-triazole (3d).  

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange liquid, 140 mg, 51% yield, mp 157.0-158.0 °C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.84 (s, 1H), 7.45-7.43 (m, 3H), 7.38-7.36 (m, 2H), 7.15 (d, $J$ = 8.3 Hz, 2H), 1.31 (s, 9H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 152.5, 137.8, 136.8, 133.3, 129.4, 129.3, 128.2, 125.9,
125.3, 123.7, 34.8, 31.2.

**HRMS-ESI** (m/z): [M+H]^+ calcd for C\textsubscript{18}H\textsubscript{19}N\textsubscript{3}: 278.3710, found: 278.3712.

5-(4-methoxyphenyl)-1-phenyl-1H-1,2,3-triazole (3e). \[16\]

\[
\text{H}_2\text{CO} \quad 3e
\]

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange liquid, 226 mg, 90% yield, mp 61.0-63.0 °C (lit. 97-98 °C).

\(^1\text{H NMR}\) (500 MHz, CDCl\textsubscript{3}) δ 7.79 (s, 1H), 7.43-7.42 (m, 3H), 7.37-7.35 (m, 2H), 7.13 (d, \(J = 8.7\) Hz, 2H), 6.85 (d, \(J = 8.7\) Hz, 2H), 3.79 (s, 3H).

5-(4-(benzyloxy) phenyl)-1-phenyl-1H-1,2,3-triazole (3f).

\[
\text{BnO} \quad 3f
\]

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 186 mg, 57% yield, mp 95.8-97.3 °C.

\(^1\text{H NMR}\) (500 MHz, CDCl\textsubscript{3}) δ 7.81 (s, 1H), 7.45-7.34 (m, 10H), 7.15 (dt, \(J_1 = 8.8\) Hz, \(J_2 = 2.8\) Hz, 2H), 6.94 (dt, \(J_1 = 8.9\) Hz, \(J_2 = 2.6\) Hz, 2H), 5.06 (s, 2H).

\(^{13}\text{C NMR}\) (126 MHz, CDCl\textsubscript{3}) δ 159.5, 137.6, 136.7, 136.4, 133.0, 130.0, 129.4, 129.2, 128.7, 128.2, 127.5, 125.3, 119.2, 115.2, 70.1.

**HRMS-ESI** (m/z): [M+H]^+ calcd for C\textsubscript{21}H\textsubscript{17}N\textsubscript{3}O: 328.1372, found: 328.1370.

5-(4-(methylthio) phenyl)-1-phenyl-1H-1,2,3-triazole (3g).

\[
\text{S} \quad 3g
\]
According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 165 mg, 62% yield, mp 103.8-105.3 °C.

\( ^1\text{H NMR} \) (500 MHz, CDCl\(_3\)) \( \delta \) 7.82 (s, 1H), 7.43 (t, \( J = 2.5 \) Hz, 3H), 7.36-7.34 (m, 2H), 7.19-7.15 (m, 2H), 7.11 (d, \( J = 8.6 \) Hz, 2H), 2.45 (s, 3H).

\( ^{13}\text{C NMR} \) (126 MHz, CDCl\(_3\)) \( \delta \) 140.7, 137.4, 136.6, 133.2, 129.5, 129.3, 128.8, 126.1, 125.3, 122.9, 15.1.

\( \text{HRMS-ESI} \) (m/z): [M+H]\(^+\) calcd for C\(_{15}\)H\(_{13}\)N\(_3\)S: 268.0830, found: 268.0830.

5-(4-fluorophenyl)-1-phenyl-1H-1,2,3-triazole (3h). \[^{15}\]  

\[ \text{3h} \]  

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 175 mg, 74% yield, mp 89.0-91.0 °C (lit. 100-103 °C).

\( ^1\text{H NMR} \) (500 MHz, CDCl\(_3\)) \( \delta \) 7.82 (s, 1H), 7.43-7.33 (m, 5H), 7.21-7.16 (m, 2H), 7.03 (t, \( J = 8.4 \) Hz, 2H).

\( ^{19}\text{F NMR} \) (471 MHz, CDCl\(_3\)) \( \delta \) –110.9- –111.0 (m, Ar-F).

5-(4-chlorophenyl)-1-phenyl-1H-1,2,3-triazole (3i). \[^{16}\]  

\[ \text{3i} \]  

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 190 mg, 75% yield, mp 114.0-116.0 °C (lit. 105-106 °C).

\( ^1\text{H NMR} \) (500 MHz, CDCl\(_3\)) \( \delta \) 7.85 (s, 1H), 7.45-7.42 (m, 3H), 7.34-7.30 (m, 4H), 7.15 (d, \( J = 8.3 \) Hz, 2H).

5-(4-bromophenyl)-1-phenyl-1H-1,2,3-triazole (3j). \[^{13}\]
According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 178 mg, 60% yield, mp 139.5-141.5 °C (lit. 151-152 °C).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.87 (s, 1H), 7.49-7.44 (m, 5H), 7.36-7.34 (m, 2H), 7.09 (dt, $J_1 = 8.4$ Hz, $J_2 = 1.7$ Hz, 2H).

1-phenyl-5-(4-(trifluoromethoxy) phenyl)-1H-1,2,3-triazole (3k).

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange liquid, 175 mg, 58% yield.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.87 (s, 1H), 7.47-7.43 (m, 3H), 7.36-7.34 (m, 2H), 7.27-7.25 (m, 2H), 7.19 (d, $J = 8.2$ Hz, 2H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 149.8, 136.4 (d, $J = 22.7$ Hz), 133.6, 130.2, 129.6, 125.4, 125.3, 121.2.

In the $^{13}$C NMR spectrum of 3k, theoretically, there should be eleven peaks. Due to the compact overlaying, it is difficult to specify the overlaying peaks.

$^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ -57.8 (s, 3F).


1-phenyl-5-(4-(trifluoromethyl)phenyl)-1H-1,2,3-triazole (3l).

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as
eluent for column chromatography. Orange liquid, 140 mg, 49% yield.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.91 (s, 1H), 7.59 (d, $J$ = 8.3 Hz, 2H), 7.47-7.43 (m, 3H), 7.36-7.33 (m, 4H).

$^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ -62.9 (s, 3F).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 136.4, 136.3, 133.8, 131.4, 131.1, 130.4, 129.64, 129.61, 128.9, 125.9 (q, $J$ = 3.6 Hz), 125.3, 124.8, 122.6.

HRMS-ESI (m/z): [M+H]$^+$ calcd for C$_{15}$H$_{10}$F$_3$N$_3$: 290.0827, found: 290.0830.

1-phenyl-5-(m-tolyl)-1H-1,2,3-triazole (3m).

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 173 mg, 74% yield, mp 104.0-106.0 °C (lit. 109-113 °C).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.85 (s, 1H), 7.44-7.42 (m, 3H), 7.38-7.36 (m, 2H), 7.21-7.18 (m, 2H), 7.08 (s, 1H), 6.96 (d, $J$ = 7.4 Hz, 1H), 2.30 (s, 3H).

5-(3-methoxyphenyl)-1-phenyl-1H-1,2,3-triazole (3n).

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 189 mg, 76% yield, mp 95.5-97.0 °C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.85 (s, 1H), 7.44-7.43 (m, 3H), 7.38-7.36 (m, 2H), 7.24 (t, $J$ = 8.0 Hz, 1H), 6.89 (dd, $J_1$ = 8.3 Hz, $J_2$ = 2.0 Hz, 1H), 6.80 (d, $J$ = 7.6 Hz, 1H), 6.72 (t, $J$ = 1.9 Hz, 1H), 3.67 (s, 3H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 159.7, 137.6, 136.6, 133.4, 130.0, 129.4, 129.3, 127.9,
125.3, 121.0, 115.0, 114.0, 55.2.


5-(3-fluorophenyl)-1-phenyl-1H-1,2,3-triazole (3o).

![Structure of 5-(3-fluorophenyl)-1-phenyl-1H-1,2,3-triazole (3o)](image)

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 172 mg, 72% yield, mp 116.0-117.2 °C.

**$^1$H NMR** (500 MHz, CDCl$_3$) $\delta$ 7.87 (s, 1H), 7.45-7.44 (m, 3H), 7.35-7.28 (m, 3H), 7.05 (t, $J = 7.7$ Hz, 1H), 7.00 (d, $J = 7.5$ Hz, 1H), 6.91 (d, $J = 9.4$ Hz, 1H).

**$^{19}$F NMR** (471 MHz, CDCl$_3$) $\delta$ −111.30- −111.35 (m, Ar-F).

**$^{13}$C NMR** (126 MHz, CDCl$_3$) $\delta$ 162.6 (d, $J = 247.9$ Hz), 136.60, 136.58, 136.3, 133.6, 130.7 (d, $J = 9.1$ Hz), 129.5 (d, $J = 2.8$ Hz), 128.7 (d, $J = 8.1$ Hz), 125.2, 124.4 (d, $J = 2.7$ Hz), 116.3 (d, $J = 20.8$ Hz), 115.5 (d, $J = 23.6$ Hz).

**HRMS-ESI** (m/z): [M+H]$^+$ calcd for C$_{14}$H$_{10}$FN$_3$: 240.0859, found: 240.0860.

5-(3-chlorophenyl)-1-phenyl-1H-1,2,3-triazole (3p).

![Structure of 5-(3-chlorophenyl)-1-phenyl-1H-1,2,3-triazole (3p)](image)

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 186 mg, 73% yield, 78.0-80.0 °C, (lit. 85-87 °C).

**$^1$H NMR** (500 MHz, CDCl$_3$) $\delta$ 7.88 (s, 1H), 7.47-7.44 (m, 3H), 7.36-7.34 (m, 3H), 7.26 (t, $J = 7.8$ Hz, 2H), 7.06 (d, $J = 7.8$ Hz, 1H).

1-phenyl-5-(3-(trifluoromethoxy) phenyl)-1H-1,2,3-triazole (3q).
According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Red liquid, 150 mg, 50% yield.

**^1H NMR** (500 MHz, CDCl$_3$) $\delta$ 7.91 (s, 1H), 7.49-7.44 (m, 3H), 7.39 (t, $J=8.1$ Hz, 1H), 7.35 (d, $J=7.7$ Hz, 2H), 7.21 (t, $J=8.1$ Hz, 2H), 7.03 (s, 1H).

**^19F NMR** (471 MHz, CDCl$_3$) $\delta$ –57.97 (s, 3F).

**^13C NMR** (126 MHz, CDCl$_3$) $\delta$ 149.3, 136.2 (d, $J=17.2$ Hz), 133.6, 130.5, 129.64, 129.58, 129.3, 128.7, 126.9, 125.3, 125.2, 121.7, 121.0.

**HRMS-ESI** (m/z): [M+H]$^+$ calcd for C$_{15}$H$_{10}$F$_3$N$_3$O: 306.0776, found: 306.0776.

**1-phenyl-5-(o-tolyl)-1H-1,2,3-triazole (3r).** [16]

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 200 mg, 86% yield, mp 61.0-63.0 °C (lit. 81-82 °C).

**^1H NMR** (500 MHz, CDCl$_3$) $\delta$ 7.75 (s, 1H), 7.32-7.28 (m, 6H), 7.20-7.14 (m, 3H), 1.96 (s, 3H).

**5-(2-methoxyphenyl)-1-phenyl-1H-1,2,3-triazole (3s).** [15]

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 185 mg, 74% yield, mp 86.5-88.0
°C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.82 (s, 1H), 7.39-7.30 (m, 6H), 7.23 (dd, $J_1 = 7.6$ Hz, $J_2 = 1.6$ Hz, 1H), 6.97 (td, $J_1 = 7.7$ Hz, $J_2 = 0.9$ Hz, 1H), 6.83 (d, $J = 8.3$ Hz, 1H), 3.40 (s, 3H).

5-(2-fluorophenyl)-1-phenyl-1H-1,2,3-triazole (3t). [17]

![5-(2-fluorophenyl)-1-phenyl-1H-1,2,3-triazole (3t)](image)

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 181 mg, 76% yield, mp 103.0-105.0 °C (lit. 97-99 °C).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.91 (s, 1H), 7.41-7.33 (m, 6H), 7.19-7.07 (m, 3H).

$^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ –111.87-–111.92 (m, Ar-F).

1-phenyl-5-(2-(trifluoromethyl)phenyl)-1H-1,2,3-triazole (3u).

![1-phenyl-5-(2-(trifluoromethyl)phenyl)-1H-1,2,3-triazole (3u)](image)

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 100 mg, 35% yield.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.84 (s, 1H), 7.79 (d, $J = 7.6$ Hz, 1H), 7.54 (t, $J = 7.6$ Hz, 1H), 7.49 (t, $J = 7.5$ Hz, 1H), 7.35-7.33 (m, 3H), 7.30-7.27 (m, 2H), 7.15 (d, $J = 7.5$ Hz, 1H).

$^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ –58.7 (s, 3F).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 136.3, 135.2, 134.1, 132.7, 131.8, 130.1, 129.8, 129.3 129.1, 126.7 (q, $J = 3.5$ Hz), 125.9, 124.6, 122.3.

HRMS-ESI (m/z): [M+H]$^+$ calcd for C$_{15}$H$_{10}$F$_3$N$_3$: 290.0827, found: 290.0827.

5-(2-nitrophenyl)-1-phenyl-1H-1,2,3-triazole (3v).[26]
According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Black solid, 45 mg, 17% yield.

\[ ^1H \text{ NMR} \ (500 \text{ MHz, CDCl}_3) \delta 8.24 \ (s, 1H), 8.12 \ (d, J = 7.7 \text{ Hz, 1H}), 7.85 \ (d, J = 8.1 \text{ Hz, 1H}), 7.78 \ (d, J = 7.9 \text{ Hz, 2H}), 7.70 \ (t, J = 7.5 \text{ Hz, 1H}), 7.54 \ (q, J = 8.2 \text{ Hz, 3H}), 7.47 \ (t, J = 7.3 \text{ Hz, 1H}). \]

5-(2,5-dimethylphenyl)-1-phenyl-1H-1,2,3-triazole (3w).

According to general procedure A, 4 eq. \(-\text{BuOK} \) was used, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 150 mg, 61% yield, mp 121.8-123.2 °C.

\[ ^1H \text{ NMR} \ (500 \text{ MHz, CDCl}_3) \delta 7.74 \ (s, 1H), 7.36-7.31 \ (m, 5H), 7.13 \ (d, J = 7.2 \text{ Hz, 1H}), 7.08 \ (d, J = 7.7 \text{ Hz, 1H}), 7.02 \ (s, 1H), 2.30 \ (s, 3H), 1.89 \ (s, 3H). \]

\[ ^{13}C \text{ NMR} \ (126 \text{ MHz, CDCl}_3) \delta 137.1, 136.9, 135.8, 134.4, 134.1, 131.0, 130.61, 130.57, 129.2, 128.7, 126.6, 123.7, 20.8, 19.3. \]

\[ \text{HRMS-ESI} \ (m/z): [M+H]^+ \text{ calcd for } C_{16}H_{15}N_3: 250.1266, \text{ found: } 250.1260. \]

5-(3,5-dimethoxyphenyl)-1-phenyl-1H-1,2,3-triazole (3x).

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 190 mg, 68% yield, mp 148.0-149.2 °C.
1H NMR (500 MHz, CDCl₃) δ 7.84 (s, 1H), 7.44-7.37 (m, 5H), 6.43 (s, 1H), 6.32 (d, J = 2.0 Hz, 2H), 3.64 (s, 6H).

13C NMR (126 MHz, CDCl₃) δ 160.9, 137.7, 136.7, 133.3, 129.4, 129.3, 128.3, 125.3, 106.7, 101.3, 55.3.


5-(2,3-dimethoxyphenyl)-1-phenyl-1H-1,2,3-triazole (3y).

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 126 mg, 45% yield, mp 78.5-80.0 °C.

1H NMR (500 MHz, CDCl₃) δ 7.86 (s, 1H), 7.37 (s, 5H), 7.03 (t, J = 8.0 Hz, 1H), 6.97 (d, J = 8.0 Hz, 1H), 6.73 (d, J = 7.7 Hz, 1H), 3.85 (s, 3H), 3.51 (s, 3H).

13C NMR (126 MHz, CDCl₃) δ 153.0, 147.0, 137.2, 134.7, 134.1, 129.2, 128.9, 124.2, 124.1, 122.6, 121.4, 114.0, 60.5, 55.9.


5-(3,4-dichlorophenyl)-1-phenyl-1H-1,2,3-triazole (3z).

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 130 mg, 45% yield, mp 125.3-126.7 °C.

1H NMR (500 MHz, CDCl₃) δ 7.89 (s, 1H), 7.49 (d, J = 5.2 Hz, 3H), 7.41-7.36 (m, 4H), 7.00 (d, J = 7.5 Hz, 1H).

13C NMR (126 MHz, CDCl₃) δ 136.1, 135.6, 133.8, 133.7, 133.3, 131.0, 130.3, 129.8,
129.7, 127.7, 126.7, 125.2.

**HRMS-ESI (m/z):** [M+H]$^+$ calcd for C$_{14}$H$_9$Cl$_2$N$_3$: 290.0174, found: 290.0170.

1-phenyl-5-(3,4,5-trimethoxyphenyl)-1H-1,2,3-triazole (3aa).

![3aa](image)

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange liquid, 127 mg, 41% yield.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.85 (s, 1H), 7.46-7.45 (m, 3H), 7.39-7.38 (m, 2H), 6.38 (s, 2H), 3.84 (s, 3H), 3.65 (s, 6H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 153.4, 138.8, 137.8, 136.7, 132.9, 129.4, 129.3, 125.5, 121.8, 105.9, 60.9, 56.0.

**HRMS-ESI (m/z):** [M+H]$^+$ calcd for C$_{17}$H$_{17}$N$_3$O$_3$: 312.1270, found: 312.1270.

5-(benzo[d] [1,3] dioxol-5-yl)-1-phenyl-1H-1,2,3-triazole (3ab).

![3ab](image)

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 195 mg, 74% yield, mp 133.0-134.5 $^\circ$C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.79 (s, 1H), 7.44 (t, $J$ = 3.4 Hz, 3H), 7.38-7.36 (m, 2H), 6.77 (d, $J$ = 8.1 Hz, 1H), 6.73 (dd, $J_1$ = 8.3 Hz, $J_2$ = 1.7 Hz, 1H), 6.63 (d, $J$ = 1.6 Hz, 1H), 5.98 (s, 2H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 148.5, 148.1, 137.6, 136.6, 133.2, 129.4 (d, $J$ = 17.2 Hz), 125.2, 122.9, 121.9, 120.2, 109.4, 108.8 (d, $J$ = 7.3 Hz), 101.6.

**HRMS-ESI (m/z):** [M+H]$^+$ calcd for C$_{15}$H$_{11}$N$_3$O$_2$: 266.0851, found: 266.0850.

5-(naphthalen-1-yl)-1-phenyl-1H-1,2,3-triazole (3ac).
According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow liquid, 200 mg, 74% yield.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.95 (s, 1H), 7.92 (d, $J$ = 8.2 Hz, 1H), 7.89 (d, $J$ = 8.1 Hz, 1H), 7.64 (d, $J$ = 8.2 Hz, 1H), 7.50 (td, $J_1$ = 6.9 Hz, $J_2$ = 1.1 Hz, 1H), 7.44 (td, $J_1$ = 7.2 Hz, $J_2$ = 1.5 Hz, 2H), 7.32-7.22 (m, 6H).

3-(1-phenyl-1H-1,2,3-triazol-5-yl) pyridine (3ad).

According to general procedure A, 4 eq. $t$-BuOK was used, petroleum ether / ethyl acetate = 50 : 50 (v / v) as eluent for column chromatography. Yellow solid, 115 mg, 72% yield, mp 152.9-154.0 °C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.57 (dd, $J_1$ = 4.9 Hz, $J_2$ = 1.4 Hz, 1H), 8.51 (d, $J$ = 1.7 Hz, 1H), 7.91 (s, 1H), 7.48-7.41 (m, 4H), 7.33-7.31 (m, 2H), 7.24 (t, $J$ = 4.1 Hz, 1H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 150.3, 149.1, 136.1, 135.7, 134.7, 133.7, 130.0, 129.7, 125.3, 123.5, 123.2.

HRMS-ESI (m/z): [M+H]$^+$ calcd for C$_{13}$H$_{10}$N$_4$: 223.0905, found: 223.0900.

1-phenyl-5-(thiophen-3-yl)-1H-1,2,3-triazole (3ae).

According to general procedure A, 4 eq. $t$-BuOK was used, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 155 mg, 69% yield, mp 89.5-91.0 °C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.87 (s, 1H), 7.48 (d, $J$ = 5.5 Hz, 3H), 7.41-7.40 (m,
2H), 7.32-7.31 (m, 1H), 7.14 (s, 1H), 6.92 (d, J = 4.8 Hz, 1H).

\[\text{\textsuperscript{13}C NMR (126 MHz, CDCl}_3\text{) }\delta \ 136.7, 133.6, 132.8, 129.7, 129.5, 127.0, 126.8, 126.7, 125.6, 124.7.\]

\[\text{HRMS-ESI (m/z): [M+H]}^+ \text{ calcd for C}_{12}\text{H}_9\text{N}_3\text{S: 228.0517, found: 228.0515.}\]

\textbf{1-phenyl-5-(thiophen-2-yl)-1H-1,2,3-triazole (3af).} \[^{[18]}\]

According to general procedure A, 4 eq. \textit{t}-BuOK was used, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 186 mg, 82% yield, mp 97.5-99.0 °C.

\textbf{1H NMR (500 MHz, CDCl}_3\text{) }\delta \ 7.89 (s, 1H), 7.53-7.48 (m, 3H), 7.42 (d, J = 7.2 Hz, 2H), 7.34 (d, J = 5.0 Hz, 1H), 6.98 (t, J = 3.8 Hz, 1H), 6.92 (d, J = 3.3 Hz, 1H).

\textbf{5-phenethyl-1-phenyl-1H-1,2,3-triazole (3ag).}

According to \textbf{general procedure 3.4}, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 167 mg, 67% yield.

\textbf{1H NMR (500 MHz, CDCl}_3\text{) }\delta \ 7.69-7.67 (m, 2H), 7.56 (s, 1H), 7.52-7.48 (m, 2H), 7.43-7.40 (m, 1H), 7.32-7.29 (m, 2H), 7.24-7.21 (m, 3H), 3.16-3.13 (m, 2H), 3.09-3.06 (m, 2H).

\textbf{13C NMR (126 MHz, CDCl}_3\text{) }\delta \ 148.0, 141.1, 137.3, 129.7, 128.53, 128.50, 126.2, 120.4, 119.2, 35.5, 27.5.

\[\text{HRMS-ESI (m/z): [M+H]}^+ \text{ calcd for C}_{16}\text{H}_{15}\text{N}_3\text{: 250.1266, found: 250.1260.}\]

\textbf{1-(4-fluorophenyl)-5-phenyl-1H-1,2,3-triazole (3ba).} \[^{[22]}\]
According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 190 mg, 80% yield, mp 155.0-157.0 °C (lit. 140-141 °C).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.85 (s, 1H), 7.40-7.33 (m, 5H), 7.21 (d, $J = 6.9$ Hz, 2H), 7.12 (t, $J = 8.4$ Hz, 2H).

$^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ –111.12- –111.18 (m, Ar-F).

1-(4-bromophenyl)-5-phenyl-1H-1,2,3-triazole (3bb). \textsuperscript{[21]}

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 272 mg, 91% yield, mp 88.0-90.0 °C (lit. 100-102 °C).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.83 (s, 1H), 7.52 (d, $J = 8.5$ Hz, 2H), 7.35 (q, $J = 7.2$ Hz, 3H), 7.23-7.20 (m, 4H).

1-(4-nitrophenyl)-5-phenyl-1H-1,2,3-triazole (3bc). \textsuperscript{[22]}

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Red solid, 185 mg, 70% yield, mp 151.0-153.0 °C, (lit. 164-165 °C).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.29 (d, $J = 8.9$ Hz, 2H), 7.88 (s, 1H), 7.58 (d, $J = 8.9$ Hz, 2H).
Hz, 2H), 7.47-7.40 (m, 3H), 7.24 (d, J = 7.3 Hz, 2H).

4-(5-phenyl-1H-1,2,3-triazol-1-yl) benzonitrile (3bd). [23]

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 170 mg, 70% yield, mp 115.0-117.0 °C.

\[
\text{\textbf{1H NMR}} \ (500 \text{ MHz, CDCl}_3) \ \delta \ 7.87 \ (s, \ 1H), \ 7.75-7.70 \ (m, \ 2H), \ 7.52 \ (d, \ J = 8.5 \text{ Hz}, \ 2H), \ 7.46-7.39 \ (m, \ 3H), \ 7.23 \ (d, \ J = 7.2 \text{ Hz}, \ 2H).
\]

5-phenyl-1-(4-(trifluoromethoxy) phenyl)-1H-1,2,3-triazole (3be).

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 245 mg, 81% yield, mp 89.7-91.5 °C.

\[
\text{\textbf{1H NMR}} \ (500 \text{ MHz, CDCl}_3) \ \delta \ 7.87 \ (s, \ 1H), \ 7.43-7.37 \ (m, \ 5H), \ 7.28 \ (d, \ J = 8.6 \text{ Hz}, \ 2H), \ 7.23 \ (d, \ J = 6.5 \text{ Hz}, \ 2H).
\]

\[
\text{\textbf{13C NMR}} \ (126 \text{ MHz, CDCl}_3) \ \delta \ 149.4, \ 137.9, \ 134.9, \ 133.7, \ 129.6, \ 129.1, \ 128.7, \ 126.6, \ 126.4, \ 122.4, \ 121.7.
\]

\[
\text{\textbf{19F NMR}} \ (471 \text{ MHz, CDCl}_3) \ \delta \ -57.9 \ (s, \ 3F).
\]

\[
\text{\textbf{HRMS-ESI (m/z): [M+H]^+}} \ \text{calcd for C}_{15}\text{H}_{10}\text{F}_{3}\text{N}_3\text{O: 306.0776}, \ \text{found: 306.0775}.
\]

5-phenyl-1-(m-tolyl)-1H-1,2,3-triazole (3bf). [24]
According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange liquid, 230 mg, 98% yield.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.83 (s, 1H), 7.34-7.30 (m, 3H), 7.25-7.20 (m, 5H), 7.04 (d, $J = 7.6$ Hz, 1H), 2.33 (s, 3H).

1-(3-chlorophenyl)-5-phenyl-1H-1,2,3-triazole (3bg).

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange liquid, 254 mg, 99% yield.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.85 (s, 1H), 7.46 (s, 1H), 7.41-7.31 (m, 5H), 7.22 (d, $J = 6.9$ Hz, 2H), 7.18 (d, $J = 7.9$ Hz, 1H).

5-phenyl-1-(3-(trifluoromethoxy) phenyl)-1H-1,2,3-triazole (3bh).

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow liquid, 190 mg, 63% yield.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.85 (s, 1H), 7.46 (t, $J = 8.2$ Hz, 1H), 7.40-7.34 (m, 4H), 7.28 (d, $J = 8.3$ Hz, 1H), 7.23-7.22 (m, 3H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 149.3, 136.2 (d, $J = 20.0$ Hz), 133.7, 130.5, 129.65, 129.58, 128.7, 126.9, 125.3, 121.7, 121.0.

In the $^{13}$C NMR spectrum of 3bh, theoretically, there should be thirteen peaks. Due to
the compact overlaying, it is difficult to specify the overlaying peaks.

\[^{19}\text{F NMR}\] (471 MHz, CDCl\textsubscript{3}) \(\delta -58.0\) (s, 3F).

HRMS-ESI (m/z): [M+H]\(^+\) calcd for C\textsubscript{15}H\textsubscript{10}F\textsubscript{3}N\textsubscript{3}O: 306.0776, found: 306.0780.

1-(3-nitrophenyl)-5-phenyl-1H-1,2,3-triazole (3bi). \[^{25}\]

According to general procedure B, 4 eq. \(t\)-BuOK was used, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 160 mg, 61% yield, mp 110.0-112.0 °C (lit. 133-134 °C).

\[^{1}\text{H NMR}\] (500 MHz, CDCl\textsubscript{3}) \(\delta 8.31-8.30\) (m, 2H), 7.90 (s, 1H), 7.70 (d, \(J = 8.1\) Hz, 1H), 7.63 (t, \(J = 8.7\) Hz, 1H), 7.46-7.39 (m, 3H), 7.25 (d, \(J = 7.0\) Hz, 2H).

5-phenyl-1-(o-tolyl)-1H-1,2,3-triazole (3bj). \[^{19}\]

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 160 mg, 68% yield, mp 73.0-75.0 °C (lit. 71-73 °C).

\[^{1}\text{H NMR}\] (500 MHz, CDCl\textsubscript{3}) \(\delta 7.93\) (s, 1H), 7.39 (t, \(J = 6.3\) Hz, 1H), 7.30-7.25 (m, 6H), 7.16 (d, \(J = 6.9\) Hz, 2H), 1.93 (s, 3H).

1-(2-isopropylphenyl)-5-phenyl-1H-1,2,3-triazole (3bk).

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 220 mg, 84% yield, mp 107.5-108.8
°C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.94 (s, 1H), 7.49 (t, $J = 7.5$ Hz, 1H), 7.42 (d, $J = 7.6$ Hz, 1H), 7.31-7.24 (m, 5H), 7.17-7.16 (m, 2H), 2.46 (hept, $J = 6.8$ Hz, 1H), 0.97 (br s, 6H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 145.7, 139.0, 134.5, 132.2, 130.7, 129.1, 128.8, 127.9, 127.8, 127.1, 126.7, 126.6, 28.0, 22.3.

HRMS-ESI (m/z): [M+H]$^+$ calcd for C$_{17}$H$_{17}$N$_3$: 264.1422, found: 264.1425.

1-(2-nitrophenyl)-5-phenyl-1H-1,2,3-triazole (3bl).

![3bl](image)

According to general procedure A, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Black liquid, 70 mg, 27% yield.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.07 (dt, $J = 8.0$ Hz, $J = 1.4$ Hz, 1H), 7.89 (s, 1H), 7.74-7.66 (m, 2H), 7.45 (dt, $J = 7.6$ Hz, $J = 1.2$ Hz, 1H), 7.38-7.31 (m, 3H), 7.21 (d, $J = 7.0$ Hz, 2H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 145.4, 139.4, 133.9, 133.7, 132.7, 131.6, 131.0, 130.1, 129.7, 129.6, 129.1, 128.4, 125.7, 124.3.

HRMS-ESI (m/z): [M+H]$^+$ calcd for C$_{14}$H$_{10}$N$_4$O$_2$: 267.0804, found: 267.0810.

1-(2,6-dimethylphenyl)-5-phenyl-1H-1,2,3-triazole (3bm).

![3bm](image)

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 130 mg, 53% yield, mp 58.8-60.5 °C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.02 (s, 1H), 7.33-7.28 (m, 4H), 7.16 (d, $J = 7.5$ Hz, 4H), 1.93 (s, 6H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 138.3, 135.9, 135.3, 132.2, 130.2, 129.2, 129.0, 128.7,
126.9, 126.6, 17.6.
HRMS-ESI (m/z): \([M+H]^+\) calcld for \(C_{16}H_{15}N_3\): 250.1266, found: 250.1268.

1-(2,6-diethylphenyl)-5-phenyl-1H-1,2,3-triazole (3bn).

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v/v) as eluent for column chromatography. Orange solid, 145 mg, 53% yield, mp 78.9-80.6 °C.

\(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.03 (s, 1H), 7.44 (t, \(J = 7.8\) Hz, 1H), 7.32-7.23 (m, 5H), 7.16 (d, \(J = 7.5\) Hz, 2H), 2.18 (hept, \(J = 7.4\) Hz, 4H), 1.01 (t, \(J = 7.5\) Hz, 6H).

\(^1\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 141.5, 138.6, 134.3, 132.1, 130.6, 129.2, 129.0, 127.0, 126.8, 126.6, 24.1, 14.1.

HRMS-ESI (m/z): \([M+H]^+\) calcld for \(C_{18}H_{19}N_3\): 278.1579, found: 278.1580.

1-(2,3-dimethylphenyl)-5-phenyl-1H-1,2,3-triazole (3bo).

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v/v) as eluent for column chromatography. Yellow liquid, 127 mg, 51% yield, mp 61.7-63.2 °C.

\(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.93 (s, 1H), 7.29-7.25 (m, 4H), 7.20-7.16 (m, 3H), 7.11 (d, \(J = 7.8\) Hz, 1H), 2.29 (s, 3H), 1.80 (s, 3H).

\(^1\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 138.79, 138.76, 136.0, 133.9, 132.3, 131.6, 129.1, 128.9, 127.7, 126.7, 126.3, 125.3, 20.3, 14.1.

HRMS-ESI (m/z): \([M+H]^+\) calcld for \(C_{16}H_{15}N_3\): 250.1266, found: 250.1270.

1-(3,5-dimethylphenyl)-5-phenyl-1H-1,2,3-triazole (3bp).
According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 185 mg, 75% yield, mp 85.5-87.0 °C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.85 (s, 1H), 7.37-7.33 (m, 3H), 7.24 (d, $J = 7.0$ Hz, 2H), 7.07 (s, 1H), 6.97 (s, 2H), 2.30 (s, 6H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 139.3, 137.7, 136.5, 133.2, 130.9, 129.2, 128.8, 128.5, 126.9, 123.0, 120.5, 21.2.

HRMS-ESI (m/z): [M+H]$^+$ calcd for C$_{16}$H$_{15}$N$_3$: 250.1266, found: 250.1265.

1-(2-methoxy-5-methylphenyl)-5-phenyl-1H-1,2,3-triazole (3bq).

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 216 mg, 82% yield, mp 157.7-159.2 °C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.86 (s, 1H), 7.29 (d, $J = 5.4$ Hz, 4H), 7.24-7.21 (m, 3H), 6.81 (d, $J = 8.4$ Hz, 1H), 3.40 (s, 3H), 2.34 (s, 3H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 151.4, 139.3, 132.1, 131.8, 130.7, 129.1, 128.9, 128.7, 128.6, 127.6, 127.4, 125.4, 55.5, 20.3.

HRMS-ESI (m/z): [M+H]$^+$ calcd for C$_{16}$H$_{15}$N$_3$O: 266.1215, found: 266.1215.

1-(2,6-difluorophenyl)-5-phenyl-1H-1,2,3-triazole (3br).

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as
eluent for column chromatography. Yellow solid, 233 mg, 91% yield, mp 117.5-119.0 ℃.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.92 (s, 1H), 7.51-7.45 (m, 1H), 7.39-7.33 (m, 3H), 7.25 (d, $J = 7.8$ Hz, 2H), 7.06 (t, $J = 8.2$ Hz, 2H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 158.0 (dd, $J_1 = 256.2$ Hz, $J_2 = 2.7$ Hz), 140.3, 132.2 (d, $J = 9.1$ Hz), 132.1 (d, $J = 10.0$ Hz), 129.7, 129.0, 127.7, 126.0, 112.4 (d, $J_1 = 3.7$ Hz), 112.3 (d, $J = 3.6$ Hz).

$^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ –117.44–117.47 (m, Ar-F).

HRMS-ESI (m/z): [M+H]$^+$ calcld for C$_{14}$H$_9$F$_2$N$_3$: 258.0765, found: 258.0760.

1-(3,4-dichlorophenyl)-5-phenyl-1H-1,2,3-triazole (3bs).

According to general procedure B, 4 eq. t-BuOK was used, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Yellow solid, 125 mg, 44% yield, mp 96.3-97.5 ℃.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.85 (s, 1H), 7.59 (d, $J = 2.3$ Hz, 1H), 7.48 (d, $J = 8.5$ Hz, 1H), 7.43-7.39 (m, 3H), 7.24 (d, $J = 6.7$ Hz, 2H), 7.14 (dd, $J_1 = 8.6$ Hz, $J_2 = 2.3$ Hz, 1H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 137.8, 135.7, 133.7, 133.64, 133.61, 131.0, 129.8, 129.2, 128.7, 126.9, 126.1, 124.1.

HRMS-ESI (m/z): [M+H]$^+$ calcld for C$_{14}$H$_9$Cl$_2$N$_3$: 290.0174, found: 290.0170.

1-(3,5-bis(trifluoromethyl)phenyl)-5-phenyl-1H-1,2,3-triazole (3bt). [20]

According to general procedure B, 4 eq. t-BuOK was used, petroleum ether / ethyl acetate = 65 : 35 (v / v) as eluent for column chromatography. Yellow solid, 206 mg,
58% yield, mp 140.0-142.0 °C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.92 (s, 1H), 7.89 (s, 1H), 7.85 (s, 2H), 7.49-7.42 (m, 3H), 7.24 (d, $J$ = 7.4 Hz, 2H).

$^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ –63.2 (s, 6F).

1-(naphthalen-1-yl)-5-phenyl-1H-1,2,3-triazole (3bu).

According to general procedure B, 4 eq. t-BuOK was used, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Orange solid, 225 mg, 83% yield, mp 128.0-130.0 °C (lit.127-129 °C).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.05 (s, 1H), 8.01 (d, $J$ = 8.2 Hz, 1H), 7.94 (d, $J$ = 8.3 Hz, 1H), 7.56-7.43 (m, 4H), 7.38 (d, $J$ = 8.4 Hz, 1H), 7.23 (d, $J$ = 7.4 Hz, 1H), 7.18 (t, $J$ = 7.6 Hz, 2H), 7.13 (d, $J$ = 7.5 Hz, 2H).

3-(5-phenyl-1H-1,2,3-triazol-1-yl) quinoline (3bv).

According to general procedure B, petroleum ether / ethyl acetate = 65 : 35 (v / v) as eluent for column chromatography. Orange solid, 180 mg, 67% yield, mp 134.5-136.0 °C.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.80 (d, $J$ = 2.1 Hz, 1H), 8.29 (d, $J$ = 2.0 Hz, 1H), 8.14 (d, $J$ = 8.4 Hz, 1H), 7.93 (s, 1H), 7.85-7.79 (m, 2H), 7.63 (t, $J$ = 7.3 Hz, 1H), 7.41-7.34 (m, 3H), 7.26 (d, $J$ = 7.1 Hz, 2H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 147.6, 146.3, 146.3, 138.2, 133.8, 131.2, 130.9, 130.2, 129.8, 129.6, 129.3, 128.7, 128.3, 128.1, 127.3, 126.2.

HRMS-ESI (m/z): [M+H]$^+$ calcd for C$_{17}$H$_{12}$N$_4$: 273.1062, found: 273.1060.
3-(5-phenyl-1H-1,2,3-triazol-1-yl) pyridine (3bw).\textsuperscript{[24]}

According to general procedure B, petroleum ether / ethyl acetate = 50 : 50 (v / v) as eluent for column chromatography. Yellow solid, 174 mg, 79% yield, mp 112.0-114.0 °C (lit. 112.0-114.0 °C).

\textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}) \(\delta\) 8.68 (d, \(J = 4.8\) Hz, 1H), 8.63 (d, \(J = 2.0\) Hz, 1H), 7.88 (s, 1H), 7.74 (d, \(J = 8.2\) Hz, 1H), 7.42-7.36 (m, 4H), 7.22 (d, \(J = 7.0\) Hz, 2H)

4-(5-phenyl-1H-1,2,3-triazol-1-yl) pyridine (3bx).\textsuperscript{[19]}

According to general procedure B, petroleum ether / ethyl acetate = 50 : 50 (v / v) as eluent for column chromatography. Yellow solid, 180 mg, 81% yield, mp 204.0-206.0 °C (lit. 199-201 °C).

\textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}) \(\delta\) 8.67 (d, \(J = 6.0\) Hz, 2H), 7.85 (s, 1H), 7.47-7.40 (m, 3H), 7.33 (d, \(J = 6.1\) Hz, 2H), 7.26 (d, \(J = 7.1\) Hz, 2H).

5-phenyl-1-(thiophen-3-yl)-1H-1,2,3-triazole (3by).

According to general procedure B, petroleum ether / ethyl acetate = 85 : 15 (v / v) as eluent for column chromatography. Black solid, 180 mg, 80% yield, mp 103.0-105.0 °C.

\textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}) \(\delta\) 7.83 (s, 1H), 7.42-7.38 (m, 3H), 7.36-7.34 (m, 1H), 7.32-7.28 (m, 3H), 7.06 (d, \(J = 5.0\) Hz, 1H).
$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 137.7, 134.7, 133.2, 129.6, 128.9, 128.7, 126.7, 126.4, 123.7, 119.3.

HRMS-ESI (m/z): [M+H]$^+$ calcd for C$_{12}$H$_9$N$_3$S: 228.0517, found: 228.0517.

5. References.


6. NMR Spectra.