Supporting Information

for

Visible-light-promoted decarboxylative addition cyclization of N-aryl glycines and azobenzenes to access 1,2,4-triazolidines

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1. General Information

$^1$H NMR, $^{13}$C NMR and $^{19}$F NMR spectra were recorded on a Varian Mercury-400 Plus spectrometer or an Agilent Technologies DD2 (600 MHz) in CDCl$_3$. Chemical shifts ($\delta$) for NMR were quoted in ppm relative to the solvent peak (7.26 ppm for $^1$H and 77.00 ppm for $^{13}$C in CDCl$_3$). Coupling constants $J$ are recorded in Hz. High-resolution mass spectra (HRMS) were reported from the Thermo Orbitrap Elite with an ESI source. UV-Visible absorption spectra were recorded on an Agilent 8453 spectrophotometer. Photoluminescence spectra were obtained with a HORIBA FluoMax-4 Spectrofluorometer. Melting points (m.p.) were measured on an XT4A apparatus (uncorrected). The X-ray single-crystal diffraction was performed on an Agilent SuperNOVA instrument. Reactions were monitored by thin layer chromatography (TLC) using pre-coated silica gel plates (GF254). Flash column chromatography was performed on silica gel 60 (particle size 200–400 mesh ASTM, purchased from Liangchen, China) and eluted with petroleum ether /ethylacetate. The symmetrical azobenzenes were prepared according to the literature procedure.$^{[1]}$ The unsymmetrical azobenzenes were synthesized according to the literature procedure.$^{[2]}$ The other materials and solvents obtained from commercial suppliers were used directly without further purification.

Unless otherwise noted, all photochemical reactions were carried out in Pyrex glass tube with magnetic stirring bar. The LED white lamps employed in this work were bought from Wuhan Jiushang Technology Co. LTD; Power (6 W); The setup of photocatalytic reaction as illustrated in Figure S1. The distance from the light source to the irradiation vessel is about 1.5 cm. The temperature is controlled by a fan. No filter was used in this reaction.

![Figure S1. Setup of Photocatalytic Reaction and Light Characteristics](image-url)
2. Optimization of Reaction Conditions

<table>
<thead>
<tr>
<th>Entry</th>
<th>Photocatalyst</th>
<th>Solvent</th>
<th>Yield(%)&lt;sup&gt;b&lt;/sup&gt;</th>
</tr>
</thead>
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<tr>
<td>1</td>
<td>MB</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN</td>
<td>95</td>
</tr>
<tr>
<td>2</td>
<td>Rose Bengal</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN</td>
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<tr>
<td>3</td>
<td>Eosin Y</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>Mes-Acr&lt;sup&gt;+&lt;/sup&gt;ClO&lt;sub&gt;4&lt;/sub&gt;&lt;sup&gt;-&lt;/sup&gt;</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN</td>
<td>trace</td>
</tr>
<tr>
<td>5</td>
<td>Rhodamine B</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>Ir[dF(CF&lt;sub&gt;3&lt;/sub&gt;)ppy]&lt;sub&gt;2&lt;/sub&gt;(dtbbpy)PF&lt;sub&gt;6&lt;/sub&gt;</td>
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<td>0</td>
</tr>
<tr>
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</tr>
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<td>11</td>
<td>MB</td>
<td>DMF</td>
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<td>MB</td>
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<td>13</td>
<td>MB</td>
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<td>–</td>
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<td>0</td>
</tr>
<tr>
<td>16&lt;sup&gt;d&lt;/sup&gt;</td>
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<td>0</td>
</tr>
<tr>
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<td>83</td>
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<td>81</td>
</tr>
<tr>
<td>20&lt;sup&gt;h&lt;/sup&gt;</td>
<td>MB</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN</td>
<td>72</td>
</tr>
</tbody>
</table>

<sup>a</sup>Reaction conditions: A solution of 1 (0.2 mmol), 2 (0.5 mmol, 2.5 equiv.) and MB (3.0 mol%) in CH<sub>3</sub>CN (3.0 mL) is irradiated by 6 W white LEDs at room temperature for 4 h.<sup>b</sup>Isolated yield.<sup>c</sup>Without photocatalyst. <sup>d</sup>In the dark. <sup>e</sup>2 (0.4 mmol, 2 equiv.).<sup>f</sup>2 (0.6 mmol, 3 equiv.).<sup>g</sup>6 W blue LEDs.<sup>h</sup>6 W green LEDs

3. General Procedure for the Synthesis of 1,2,4-Triazolidines

Azobenzenes (0.2 mmol), N-aryl glycines (0.5 mmol, 2.5 equiv.), MB (1.9 mg, 0.006 mmol,
3.0 mol%) and CH$_3$CN (3 mL) were added into a 10 mL Pyrex glass tube equipped with a magnetic stir bar. Then, the reaction mixture was stirred under irradiation of 6 W white LEDs at room temperature. After azobenzenes were consumed completely (monitored by TLC), the reaction mixture was concentrated under vacuum and the residue was purified by column chromatography on silica gel (PE/EtOAc = 200/1 to 50/1) to afford the desired product.

4. Procedure for the Gram-Scale Synthesis of 3

Azobenzene 1 (0.728 g, 4.0 mmol), N-phenyl glycine 2 (1.512 g, 10 mmol, 2.5 equiv.), MB (38.4 mg, 0.12 mmol, 3.0 mol%) and CH$_3$CN (50.0 mL) were added into a 100 mL round flask equipped with a magnetic stirring bar. The mixture was then irradiated by two 15 W white LEDs and stirred at room temperature for 24 hours. Upon completion (monitored by TLC), the reaction mixture was concentrated under vacuum and the residue was purified by column chromatography (petrol ether/EtOAc = 200:1) to give the desired product 3 (0.855 g, 71%) as a white solid.

5. Unsuccessful Substrates
6. Crystallographic Structure Determination

The colorless single crystal of product 15 was obtained by recrystallization using a CHCl₃/acetone solvent system at room temperature. The X-ray single-crystal diffraction was performed on an Agilent SuperNOVA instrument (Figure S2).

Figure S2. Thermal ellipsoid plot of compound 15 at the 30% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Table S1. Crystal data and structure refinement for songmh_1210.

<table>
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<th>Identification code</th>
<th>songmh_1210</th>
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<tbody>
<tr>
<td>Empirical formula</td>
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<td>Formula weight</td>
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</tr>
<tr>
<td>Temperature/K</td>
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<td>b/Å</td>
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<tr>
<td>c/Å</td>
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</tr>
<tr>
<td>α/°</td>
<td>90</td>
</tr>
<tr>
<td>β/°</td>
<td>104.1994(11)</td>
</tr>
<tr>
<td>Parameter</td>
<td>Value</td>
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<td>---------------------------------</td>
<td>------------------------------</td>
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<td>$\gamma^\circ$</td>
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<td>Volume/Å³</td>
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<td>$\rho_{\text{calc}}$ g/cm³</td>
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<td>$\mu$ mm⁻¹</td>
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<td>F(000)</td>
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<td>Crystal size/mm³</td>
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<td>Radiation</td>
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<td>Independent reflections</td>
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<tr>
<td>Data/restraints/parameters</td>
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<tr>
<td>Goodness-of-fit on $F^2$</td>
<td>1.037</td>
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<td>Final R indexes [I&gt;=2σ (I)]</td>
<td>$R_1 = 0.0458$, $wR_2 = 0.1208$</td>
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<tr>
<td>Final R indexes [all data]</td>
<td>$R_1 = 0.0481$, $wR_2 = 0.1229$</td>
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<tr>
<td>Largest diff. peak/hole / e Å⁻³</td>
<td>0.68/-0.84</td>
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Table S2. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\AA^2 \times 10^3$) for songmh_1210. $U_{eq}$ is defined as $1/3$ of of the trace of the orthogonalised $U_{ij}$ tensor.

<table>
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<th>Atom</th>
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<th>$y$</th>
<th>$z$</th>
<th>$U_{eq}$</th>
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Table S3. Anisotropic Displacement Parameters (Å$^2 \times 10^3$) for songmh_1210. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[a^2U_{11} + 2hka*b*U_{12} + ...].$

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<th>U$_{22}$</th>
<th>U$_{33}$</th>
<th>U$_{23}$</th>
<th>U$_{13}$</th>
<th>U$_{12}$</th>
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Table S4. Bond Lengths for songmh_1210.

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Table S6. Hydrogen Atom Coordinates ($\AA \times 10^4$) and Isotropic Displacement Parameters ($\AA^2 \times 10^3$) for songmh_1210.

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7. Mechanistic Studies

7.1 UV-Vis Absorption Spectra

The UV-visible absorption spectra of azobenzene 1, N-phenyl glycine 2, methylene blue (MB) in acetonitrile show that 1 especially MB has significant absorption in the visible-light region, while 2 has no obvious absorption in the visible-light region (Figure S3).

Figure S3. The UV-Vis absorption spectra in CH₃CN (2 mM)

7.2 Stern-Volmer Quenching Experiments

Stern-Volmer luminescence quenching experiments were run with freshly prepared solutions of 1.0 × 10⁻⁴ M MB and the appropriate amount of quencher in CH₃CN at room temperature. The solutions were irradiated at 425 nm and luminescence was measured at 702 nm. Then, appropriate amount of quencher was added to the measured solution, the emission spectrum of the sample was collected (Figures S4–S6).

Figure S4. Fluorescence quenching of MB with 1 (λₑₓ = 425 nm)
Figure S5. Fluorescence quenching of MB with 2 ($\lambda_{ex} = 425$ nm)

Figure S6. Stern-Volmer analysis

7.3 Radical Inhibiting Experiment with TEMPO

Azobenzene 1 (36.4 mg, 0.2 mmol), N-phenyl glycine 2 (75.6 mg, 0.5 mmol, 2.5 equiv.), MB (1.9 mg, 0.006 mmol, 3.0 mol%), TEMPO (62.5 mg, 0.4 mmol, 2 equiv.) and CH$_3$CN (3 mL) were added into a 10 mL Pyrex glass tube. The reaction mixture was continually stirred at room temperature under 6 W white LEDs irradiation for 4 hours. There, 3 was not detected by TLC and the reduced TEMPO-H and a dimerization product $N,N'$-diphenylethane-1,2-diamine were detected by HRMS (Figures S7–S8, data of [M+H]$^+$ are showed).
Figure S7

Figure S8

S13
7.4 Detecting the Reaction Mixture by HRMS

Azobenzene 1 (36.4 mg, 0.2 mmol), N-phenyl glycine 2 (75.6 mg, 0.5 mmol, 2.5 equiv.), MB (1.9 mg, 0.006 mmol, 3.0 mol%) and CH$_3$CN (3 mL) were added into a 10 mL Pyrex glass tube. The reaction mixture was continually stirred at room temperature under 6 W white LEDs irradiation for 1.5 hours. And then, without any treatment, the reaction mixture was detected by HRMS (Figures S9–S11, data of [M+H]$^+$ are showed).

![Figure S9](image)

**Figure S9**

![Figure S10](image)

**Figure S10**
8. Characterization Data of Products

1) 1,2,4-Triphenyl-1,2,4-triazolidine (3)\(^{[3]}\)

\[ \text{Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and } N\text{-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product 3.} \]

White solid, m.p. 140–141 ℃; yield: 57.2 mg (95%).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta 7.33–7.28\) (m, 4H), 7.27–7.19 (m, 6H), 6.98 (t, \(J = 7.4\) Hz, 2H), 6.80 (t, \(J = 7.4\) Hz, 1H), 6.61 (d, \(J = 8.0\) Hz, 2H), 4.89 (s, 2H), 4.71 (s, 2H).

\(^{13}\)C NMR (150 MHz, CDCl\(_3\)): \(\delta 150.2, 145.2, 129.4, 129.2, 121.6, 118.4, 115.0, 113.3, 67.1\).

HRMS (ESI): \(m/z [M+H]^+ \text{ calcld for } C_{20}H_{20}N_3^+ : 302.1652; \text{ found: } 302.1651\).

2) 4-Phenyl-1,2-di-p-tolyl-1,2,4-triazolidine (4)
Prepared according to the general procedure from (E)-1,2-di-p-tolyl diazene (42.0 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product 4.

White solid, m.p. 134–136 °C; yield: 61.3 mg (93%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.27–7.20 (m, 2H), 7.10–7.08 (m, 8H), 6.76 (t, $J = 7.2$ Hz, 1H), 6.57 (d, $J = 7.6$ Hz, 2H), 4.82 (s, 2H), 4.65 (s, 2H), 2.28 (s, 6H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 148.0, 145.3, 130.9, 129.7, 129.3, 118.2, 115.2, 113.2, 67.2, 20.4.


3) 1,2-Bis(4-butyphenyl)-4-phenyl-1,2,4-triazolidine (5)

Prepared according to the general procedure from (E)-1,2-bis(4-butyphenyl)diazene (58.9 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product 5.

White solid, m.p. 104–105 °C; yield: 76.1 mg (92%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.26–7.20 (m, 2H), 7.15–7.07 (m, 8H), 6.78–6.74 (m, 1H), 6.61–6.54 (m, 2H), 4.84 (s, 2H), 4.66 (s, 2H), 2.54 (t, $J = 7.8$ Hz, 4H), 1.60–1.52 (m, 4H), 1.39–1.30 (m, 4H), 0.92 (t, $J = 7.2$ Hz, 6H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 148.1, 145.1, 136.0, 129.3, 129.0, 118.1, 115.0, 113.1, 67.1, 34.8, 33.8, 22.3, 13.9.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{28}$H$_{36}$N$_3$: 414.2904; found: 414.2907.

4) 1,2-Bis(4-(tert-butyphenyl)-4-phenyl-1,2,4-triazolidine (6)
Prepared according to the general procedure from (E)-1,2-bis(4-(tert-butyl)phenyl)diazene (58.9 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product 6.

White solid, m.p. 158–160 °C; yield: 73.6 mg (89%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.32–7.29 (m, 4H), 7.25–7.20 (m, 2H), 7.16–7.12 (m, 4H), 8.79–6.74 (m, 1H), 6.59–6.54 (m, 2H), 4.86 (s, 2H), 4.67 (s, 2H), 1.30 (s, 18H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 147.8, 145.1, 144.3, 129.3, 125.9, 118.0, 114.7, 113.0, 67.0, 34.1, 31.5.

HRMS (ESI): m/z [M+H]$^+$ calcd for C$_{28}$H$_{36}$N$_3$: 414.2904; found: 414.2906.

5) 1,2-Bis(4-fluorophenyl)-4-phenyl-1,2,4-triazolidine (7)

Prepared according to the general procedure from (E)-1,2-bis(4-fluorophenyl)diazene (43.6 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product 7.

White solid, m.p. 125–126 °C; yield: 60.0 mg (89%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.28–7.22 (m, 2H), 7.15–7.11 (m, 4H), 7.06–6.94 (m, 4H), 6.81 (t, $J = 7.2$ Hz, 1H), 6.64–6.56 (m, 2H), 4.77 (s, 2H), 4.70 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 147.8, 145.1, 144.3, 129.3, 125.9, 118.0, 114.7, 113.0, 67.0, 34.1, 31.5.

HRMS (ESI): m/z [M+H]$^+$ calcd for C$_{28}$H$_{36}$F$_2$N$_3$: 338.1463; found: 338.1464.

6) 1,2-Bis(4-chlorophenyl)-4-phenyl-1,2,4-triazolidine (8)
Prepared according to the general procedure from \((E)-1,2\text{-bis}(4\text{-chlorophenyl})\text{diazene (50.2 mg, 0.2 mmol) and }\) N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product 8.

White solid, m.p. 155–156 °C; yield: 64.4 mg (87%).

\(^1\text{H NMR (400 MHz, CDCl}_3\text{): }\delta 7.29–7.21 \text{ (m, 6H), 7.11–7.05 \text{ (m, 4H), 6.82 (t, } J = 7.4 \text{ Hz, 1H), 6.64–6.55 \text{ (m, 2H), 4.79 (s, 2H), 4.68 (s, 2H).}\n\)

\(^{13}\text{C NMR (150 MHz, CDCl}_3\text{): }\delta 148.5, 144.8, 129.4, 129.1, 126.7, 118.9, 116.3, 113.5, 67.4.\n\)

HRMS (ESI): \(m/\epsilon [\text{M+H}]^+\) calcd for C\(_{20}\)H\(_{18}\)Cl\(_2\)N\(_3\): 370.0872; found: 370.0876.

7) 1,2-Bis(4-bromophenyl)-4-phenyl-1,2,4-triazolidine (9)

Prepared according to the general procedure from \((E)-1,2\text{-bis}(4\text{-bromophenyl})\text{diazene (68.0 mg, 0.2 mmol) and }\) N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product 9.

White solid, m.p. 160–161 °C; yield: 80.8 mg (88%).

\(^1\text{H NMR (400 MHz, CDCl}_3\text{): }\delta 7.41–7.38 \text{ (m, 4H), 7.29–7.24 \text{ (m, 2H), 7.05–7.02 \text{ (m, 4H), 6.86–6.20 \text{ (m, 1H), 6.62–6.60 \text{ (m, 2H), 4.79 (s, 2H), 4.68 (s, 2H).}\n\)

\(^{13}\text{C NMR (150 MHz, CDCl}_3\text{): }\delta 148.9, 144.8, 132.0, 129.4, 118.9, 116.7, 114.1, 113.5, 67.2.\n\)

HRMS (ESI): \(m/\epsilon [\text{M+H}]^+\) calcd for C\(_{20}\)H\(_{18}\)Br\(_2\)N\(_3\): 459.9842; found: 459.9843.

8) 4-Phenyl-1,2-bis(4-(trifluoromethyl)phenyl)-1,2,4-triazolidine (10)
Prepared according to the general procedure from (E)-1,2-bis(4-(trifluoromethyl)phenyl)diazene (63.6 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 100:1) to afford the pure product 10.

White solid, m.p. 82–83 °C; yield: 66.5 mg (76%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.57–7.54 (m, 4H), 7.30–7.26 (m, 2H), 7.19 (d, $J = 8.8$ Hz, 4H), 6.85 (t, $J = 7.4$ Hz, 1H), 6.66–6.63 (m, 2H), 4.91 (s, 2H), 4.76 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 152.2, 144.7, 129.5, 126.6 (q, $J = 4.5$ Hz), 124.6 (d, $J = 190.5$ Hz), 123.6 (d, $J = 48.0$ Hz) 119.4, 114.5, 113.7, 67.1.

$^{19}$F NMR (376 MHz, CDCl$_3$): $\delta = -62.0$.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{22}$H$_{18}$F$_6$N$_3$: 438.1399; found: 438.1399.

9) 4-Phenyl-1,2-bis(4-(trifluoromethoxy)phenyl)-1,2,4-triazolidine (11)

Prepared according to the general procedure from (E)-1,2-bis(4-(trifluoromethoxy)phenyl)diazene (70.0 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 100:1) to afford the pure product 11.

Colorless oil; yield: 80.7 mg (86%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.30–7.25 (m, 2H), 7.14–7.20 (m, 8H), 6.84 (t, $J = 7.4$ Hz, 1H), 6.63 (d, $J = 8.0$ Hz, 2H), 4.83 (s, 2H), 4.73 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 148.6, 144.8, 143.8 (d, $J = 1.5$ Hz), 129.5, 122.2, 121.4 (d, $J = 255.0$ Hz), 119.0, 115.9, 113.5, 67.6.

$^{19}$F NMR (376 MHz, CDCl$_3$): $\delta = -58.7$.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{22}$H$_{18}$F$_6$O$_2$N$_3$: 470.1298; found: 470.1299.
10) 4-Phenyl-1,2-di-m-tolyl-1,2,4-triazolidine (12)

Prepared according to the general procedure from (E)-1,2-di-m-tolyldiazene (42.0 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product 12.

White solid, m.p. 58–59 °C; yield: 56.2 mg (85%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.29–7.19 (m, 4H), 7.06–7.00 (m, 4H), 6.83–6.78 (m, 3H), 6.65–6.60 (m, 2H), 4.90 (s, 2H), 4.70 (s, 2H), 2.37 (s, 6H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 150.2, 145.1, 139.0, 129.3, 129.0, 122.3, 118.2, 115.5, 113.1, 112.0, 67.0, 21.7.


11) 1,2-Bis(3-fluorophenyl)-4-phenyl-1,2,4-triazolidine (13)

Prepared according to the general procedure from (E)-1,2-bis(3-fluorophenyl)diazene (43.6 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 13.

White solid, m.p. 106–107 °C; yield: 56.0 mg (83%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.28–7.23 (m, 4H), 6.94–6.87 (m, 4H), 6. 84–6.80 (m, 1H), 6.70–6.64 (m, 2H), 6.63–6.59 (m, 2H), 4.83 (s, 2H), 4.69 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 164.6 (d, $J = 244.5$ Hz), 151.9 (d, $J = 9.0$ Hz), 144.8, 130.5 (d, $J = 9.0$ Hz), 129.4, 118.9, 113.5, 110.5 (d, $J = 1.5$ Hz), 108.5 (d, $J = 6.0$ Hz), 102.5 (d, $J = 27.0$ Hz), 67.3.

$^{19}$F NMR (376 MHz, CDCl$_3$): $\delta$ = -112.26– -112.33.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{20}$H$_{18}$F$_2$N$_3$: 338.1463; found: 338.1467.
12) 1,2-Bis(3-chlorophenyl)-4-phenyl-1,2,4-triazolidine (14)

[Chemical structure image]

Prepared according to the general procedure from (E)-1,2-bis(3-chlorophenyl)diazene (50.2 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 14. 

White solid, m.p. 90–92 °C; yield: 62.2 mg (84%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.29–7.20 (m, 6H), 7.03–6.94 (m, 4H), 6.84 (t, $J = 7.2$ Hz, 1H), 6.61 (d, $J = 8.0$ Hz, 2H), 4.82 (s, 2H), 4.69 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 151.1, 144.7, 135.1, 130.2, 129.4, 121.8, 119.0, 115.1, 113.5, 113.2, 67.3.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{20}$H$_{18}$Cl$_2$N$_3$: 370.0872; found: 370.0870.

13) 1,2-Bis(3-bromophenyl)-4-phenyl-1,2,4-triazolidine (15)

[Chemical structure image]

Prepared according to the general procedure from (E)-1,2-bis(3-bromophenyl)diazene (68.0 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 15.

White solid, m.p. 135–136 °C; yield: 74.4 mg (81%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.37–7.35 (m, 2H), 7.29–7.24 (m, 2H), 7.19–7.10 (m, 4H), 7.08–7.03 (m, 2H), 6.84 (t, $J = 7.2$ Hz, 1H), 6.61 (d, $J = 8.0$ Hz, 2H), 4.80 (s, 2H), 4.68 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 151.2, 144.7, 130.5, 129.4, 124.8, 123.3, 119.0, 118.0, 113.6, 113.5, 67.3.


14) 1,2-Bis(3,4-dimethylphenyl)-4-phenyl-1,2,4-triazolidine (16)
Prepared according to the general procedure from (E)-1,2-bis(3,4-dimethylphenyl)diazene (47.6 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product 16.

White solid, m.p. 145–147 °C; yield: 58.6 mg (82%).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): δ 7.26–7.21 (m, 2H), 7.07–7.01 (m, 4H), 6.96–6.93 (m, 2H), 6.77 (t, J = 7.2 Hz, 1H), 6.59 (d, J = 8.0 Hz, 2H), 4.84 (s, 2H), 4.65 (s, 2H), 2.26 (s, 6H), 2.21 (s, 6H).

\(^{13}\)C NMR (150 MHz, CDCl\(_3\)): δ 148.4, 145.3, 137.3, 130.2, 129.5, 129.3, 118.1, 116.5, 113.2, 112.5, 67.0, 20.1, 18.8.

HRMS (ESI): m/z [M+H]\(^+\) calcd for C\(_{24}\)H\(_{28}\)N\(_3\): 358.2278; found: 358.2279.

Prepared according to the general procedure from (E)-1,2-bis(3,5-bis(trifluoromethyl)phenyl)diazene (90.8 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 100:1) to afford the pure product 17.

White solid, m.p. 136–137 °C; yield: 58.5 mg (51%).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): δ 7.60–7.56 (m, 4H), 7.56–7.51 (m, 2H), 7.33–7.27 (m, 2H), 6.90 (t, J = 7.6 Hz, 1H), 6.8 (d, J = 7.6 Hz, 2H), 4.90 (s, 2H), 4.87 (s, 2H).

\(^{13}\)C NMR (150 MHz, CDCl\(_3\)): δ 150.9, 145.3, 137.3, 130.2, 129.5, 129.3, 118.1, 116.5, 113.2, 112.5, 120.2, 116.1 (quint, J = 3.0 Hz), 115.0 (d, J = 4.5 Hz), 114.0, 68.3.

\(^{19}\)F NMR (376 MHz, CDCl\(_3\)): δ = -63.3.

HRMS (ESI): m/z [M+H]\(^+\) calcd for C\(_{34}\)H\(_{16}\)F\(_{12}\)N\(_3\): 574.1147; found: 574.1150.

Prepared according to the general procedure from (E)-1,2-bis(3,5-bis(trifluoromethyl)phenyl)diazene (90.8 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product 16.

White solid, m.p. 145–147 °C; yield: 58.6 mg (82%).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): δ 7.26–7.21 (m, 2H), 7.07–7.01 (m, 4H), 6.96–6.93 (m, 2H), 6.77 (t, J = 7.2 Hz, 1H), 6.59 (d, J = 8.0 Hz, 2H), 4.84 (s, 2H), 4.65 (s, 2H), 2.26 (s, 6H), 2.21 (s, 6H).

\(^{13}\)C NMR (150 MHz, CDCl\(_3\)): δ 148.4, 145.3, 137.3, 130.2, 129.5, 129.3, 118.1, 116.5, 113.2, 112.5, 67.0, 20.1, 18.8.

HRMS (ESI): m/z [M+H]\(^+\) calcd for C\(_{24}\)H\(_{28}\)N\(_3\): 358.2278; found: 358.2279.

\(^{15}\) 1,2-Bis(3,5-bis(trifluoromethyl)phenyl)-4-phenyl-1,2,4-triazolidine (17)

Prepared according to the general procedure from (E)-1,2-bis(3,5-bis(trifluoromethyl)phenyl)diazene (90.8 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 100:1) to afford the pure product 17.

White solid, m.p. 136–137 °C; yield: 58.5 mg (51%).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): δ 7.60–7.56 (m, 4H), 7.56–7.51 (m, 2H), 7.33–7.27 (m, 2H), 6.90 (t, J = 7.6 Hz, 1H), 6.8 (d, J = 7.6 Hz, 2H), 4.90 (s, 2H), 4.87 (s, 2H).

\(^{13}\)C NMR (150 MHz, CDCl\(_3\)): δ 150.9, 145.3, 137.3, 130.2, 129.5, 129.3, 118.1, 116.5, 113.2, 112.5, 120.2, 116.1 (quint, J = 3.0 Hz), 115.0 (d, J = 4.5 Hz), 114.0, 68.3.

\(^{19}\)F NMR (376 MHz, CDCl\(_3\)): δ = -63.3.

HRMS (ESI): m/z [M+H]\(^+\) calcd for C\(_{34}\)H\(_{16}\)F\(_{12}\)N\(_3\): 574.1147; found: 574.1150.
Prepared according to the general procedure from (E)-1,2-di-o-tolylidiazene (42.0 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product 18.

White solid, m.p. 80–81 °C; yield: 19.8 mg (30%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.58–7.55 (m, 2H), 7.30–7.25 (m, 2H), 7.24–7.16 (m, 4H), 7.08–7.00 (m, 2H), 6.83–6.78 (m, 1H), 6.59–6.55 (m, 2H), 4.96 (s, 2H), 4.57 (s, 2H), 2.42 (s, 6H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 150.1, 144.7, 131.2, 129.8, 129.4, 126.3, 123.6, 117.9, 117.8, 112.8, 69.1, 19.2.


17) 1,2-Di-o-tolylhydrazine (19)$^{[4]}$

Prepared according to the general procedure from (E)-1,2-di-o-tolylidiazene (42.0 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product 19.

White solid; yield: 22.5 mg (53%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.15–7.10 (m, 4H), 6.96–6.89 (m, 2H), 6.87–6.75 (m, 2H), 5.54 (s, 2H), 2.29 (s, 6H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 146.2, 130.4, 127.2, 121.1, 119.4, 111.0, 17.1.

18) 1,2-Bis(2-chlorophenyl)hydrazine (20)$^{[4]}$

Prepared according to the general procedure from (E)-1,2-bis(2-chlorophenyl)diazene (50.0 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product 20.
White solid; yield: 21.8 mg (43%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.34–7.31 (m, 2H), 7.19–7.12 (m, 2H), 7.00–6.96 (m, 2H), 6.86–6.77 (m, 2H), 6.22 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 143.8, 129.3, 127.9, 120.2, 117.9, 112.8.

19) 1,2-Bis(2-bromophenyl)hydrazine (21)$^{[4]}$

![Diagram of 1,2-Bis(2-bromophenyl)hydrazine](image)

Prepared according to the general procedure from (E)-1,2-bis(2-bromophenyl)diazene (67.6 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product 21.

White solid; yield: 26.7 mg (39%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.48–7.45 (m, 2H), 7.20–7.15 (m, 2H), 6.96–6.91 (m, 2H), 6.77–6.70 (m, 2H), 6.23 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 144.7, 132.5, 128.6, 120.8, 113.1, 107.5.

20) 1,4-Diphenyl-2-(p-toly)-1,2,4-triazolidine (22)$^{[3]}$

![Diagram of 1,4-Diphenyl-2-(p-toly)-1,2,4-triazolidine](image)

Prepared according to the general procedure from (E)-1-phenyl-2-(p-toly)diazene (39.2 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 22.

White solid, m.p. 106–107 °C; yield: 58.7 mg (93%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.32–7.18 (m, 6H), 7.11–7.10 (m, 4H), 6.99–6.94 (m, 1H), 6.79 (t, $J =$ 7.2 Hz, 1H), 6.61–6.56 (m, 2H), 4.89–4.63 (m, 4H), 2.30 (s, 3H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 150.1, 147.9, 145.1, 131.1, 129.7, 129.3, 129.1, 121.3, 118.2, 115.2, 114.8, 113.2, 67.3, 66.9, 20.5.

HRMS (ESI): $m/z$ [M+H]$^+$ calcld for C$_{21}$H$_{22}$N$_3$: 316.1808; found: 316.1808.

21) 1-(4-Isopropylphenyl)-2,4-diphenyl-1,2,4-triazolidine (23)

S24
Prepared according to the general procedure from (E)-1-(4-isopropylphenyl)-2-phenyldiazene (44.8 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 23.

White solid, m.p. 100–101 °C; yield: 61.8 mg (90%).

\[ \text{1H NMR (400 MHz, CDCl}_3\text{): } \delta \text{ 7.31–7.12 (m, 10H), 6.97–6.92 (m, 1H), 6.80–6.75 (m, 1H), 6.61–6.56 (m, 2H), 4.94–4.58 (m, 4H), 2.90–2.81 (m, 1H), 1.22 (d, } J = 6.8 \text{ Hz, 6H).} \]

\[ \text{13C NMR (150 MHz, CDCl}_3\text{): } \delta \text{ 150.2, 148.2, 145.1, 142.2, 129.3, 129.1, 127.0, 121.3, 118.2, 115.2, 114.9, 113.1, 67.3, 66.9, 33.3, 24.1.} \]

HRMS (ESI): \text{m/z [M+H]+ calcld for C}_{23}\text{H}_{26}\text{N}_{3}^+: 344.2121; found: 344.2121.}

22) 1-(4-(Tert-butyl)phenyl)-2,4-diphenyl-1,2,4-triazolidine (24)

Prepared according to the general procedure from (E)-1-(4-(tert-butyl)phenyl)-2-phenyldiazene (47.6 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 24.

White solid, m.p. 121–122 °C; yield: 65.1 mg (91%).

\[ \text{1H NMR (400 MHz, CDCl}_3\text{): } \delta \text{ 7.37–7.20 (m, 8H), 7.18–7.13 (m, 2H), 7.00–6.95 (m, 1H), 6.81–6.77 (m, 1H), 6.63–6.56 (m, 2H), 4.97–4.58 (m, 4H), 1.32 (s, 9H).} \]

\[ \text{13C NMR (150 MHz, CDCl}_3\text{): } \delta \text{ 150.2, 147.8, 145.1, 144.4, 129.3, 129.1, 126.0, 121.4, 118.2, 114.9, 114.8, 113.1, 67.2, 66.9, 34.1, 31.5.} \]

HRMS (ESI): \text{m/z [M+H]+ calcld for C}_{24}\text{H}_{28}\text{N}_{3}^+: 358.2278; found: 358.2284.}

23) 1-(4-Fluorophenyl)-2,4-diphenyl-1,2,4-triazolidine (25)
Prepared according to the general procedure from (E)-1-(4-fluorophenyl)-2-phenyldiazene (40.0 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 25.

White solid, m.p. 96–97 °C; yield: 54.3 mg (85%).

$^1$H NMR (400 MHz, CDCl$_3$): δ 7.37–7.24 (m, 4H), 7.23–7.13 (m, 4H), 7.03–6.98 (m, 3H), 6.86–6.80 (m, 1H), 6.64–6.60 (m, 2H), 4.89–4.61 (m, 4H).

$^{13}$C NMR (150 MHz, CDCl$_3$): δ 158.3 (d, $J = 238.5$ Hz), 149.9, 146.5 (d, $J = 1.5$ Hz), 145.0, 129.3, 129.2, 121.6, 118.5, 116.6 (d, $J = 7.5$ Hz), 115.6 (d, $J = 22.5$ Hz), 114.8, 113.3, 67.7, 67.2.

$^{19}$F NMR (376 MHz, CDCl$_3$): δ = -123.3 – -123.4.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{20}$H$_{19}$FN$_3$: 320.1558; found: 320.1560.

24) 1-(4-Chlorophenyl)-2,4-diphenyl-1,2,4-triazolidine (26)$^{[3]}$

Prepared according to the general procedure from (E)-1-(4-chlorophenyl)-2-phenyldiazene (43.3 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 26.

White solid, m.p. 120–121 °C; yield: 58.4 mg (87%).

$^1$H NMR (400 MHz, CDCl$_3$): δ 7.36–7.30 (m, 2H), 7.30–7.24 (m, 4H), 7.21–7.16 (m, 2H), 7.16–7.11 (m, 2H), 7.04–6.97 (m, 1H), 6.86–6.80 (t, $J = 7.4$ Hz, 1H), 6.64–6.60 (m, 2H), 4.89–4.68 (m, 4H).

$^{13}$C NMR (150 MHz, CDCl$_3$): δ 149.8, 148.7, 144.9, 129.4, 129.2, 129.0, 126.4, 121.8, 118.6, 116.2, 115.0, 113.3, 67.3, 67.1.


25) 1-(4-Bromophenyl)-2,4-diphenyl-1,2,4-triazolidine (27)
Prepared according to the general procedure from (E)-1-(4-bromophenyl)-2-phenyldiazene (52.2 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 27.

White solid, m.p. 155–156 °C; yield: 63.1 mg (83%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.42–7.35 (m, 2H), 7.33–7.22 (m, 4H), 7.17–7.13 (m, 2H), 7.09–7.03 (m, 2H), 7.02–6.95 (m, 1H), 6.84–6.79 (m, 1H), 6.63–6.59 (m, 2H), 4.75 (s, 4H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 149.8, 149.2, 144.9, 131.9, 129.4, 129.2, 121.8, 118.7, 116.6, 115.0, 113.7, 113.3, 67.3, 67.0.


26) 1-(4-Iodophenyl)-2,4-diphenyl-1,2,4-triazolidine (28)

Prepared according to the general procedure from (E)-1-(4-iodophenyl)-2-phenyldiazene (61.6 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 28.

White solid, m.p. 178–179 °C; yield: 70.1 mg (82%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.61–7.50 (m, 2H), 7.33–7.22 (m, 4H), 7.14 (d, $J$ = 8.0 Hz, 2H), 7.00–6.93 (m, 3H), 6.80 (t, $J$ = 7.2 Hz, 1H), 6.60 (d, $J$ = 8.0 Hz, 2H), 4.87–4.65 (m, 4H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 149.9, 149.8, 144.9, 131.9, 129.4, 129.2, 121.9, 118.7, 116.7, 115.1, 113.4, 83.6, 67.3, 66.9.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{20}$H$_{19}$IN$_3$: 428.0618; found: 420.0619.

27) 1-(4-Methoxyphenyl)-2,4-diphenyl-1,2,4-triazolidine (29)
Prepared according to the general procedure from (E)-1-(4-methoxyphenyl)-2-phenyldiazene (42.4 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 50:1) to afford the pure product 29.

White solid, m.p. 92–93 °C; yield: 58.3 mg (88%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.33–7.28 (m, 2H), 7.25–7.14 (m, 6H), 6.98–6.93 (m, 1H), 6.88–6.83 (m, 2H), 6.81–6.76 (m, 1H), 6.62–6.57 (m, 2H), 4.86–4.77 (m, 3H), 4.59 (s, 1H), 3.78 (s, 3H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 155.1, 150.1, 145.1, 144.1, 129.3, 129.1, 121.2, 118.2, 117.0, 114.7, 114.5, 113.2, 67.8, 66.9, 55.6.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{21}$H$_{22}$NO$_3$: 332.1757; found: 332.1761.

28) 1,4-Diphenyl-2-(4-(trifluoromethoxy)phenyl)-1,2,4-triazolidine (30)

Prepared according to the general procedure from (E)-1-phenyl-2-(4-(trifluoromethoxy)phenyl)diazene (53.2 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 30.

Colorless oil; yield: 62.4 mg (81%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.34–7.29 (m, 2H), 7.28–7.23 (m, 2H), 7.21–7.15 (m, 6H), 7.02–6.97 (m, 1H), 7.85–6.79 (m, 1H), 6.64–6.59 (m, 2H), 4.89–4.72 (m, 4H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 149.9, 148.8, 144.9, 143.5 (d, $J = 3.0$ Hz), 129.3 (d, $J = 12.0$ Hz), 122.1, 121.9, 120.5 (d, $J = 255.0$ Hz), 118.7, 115.8, 115.0, 113.4, 67.4, 67.3.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{21}$H$_{19}$F$_3$ON$_3$: 386.1475; found: 386.1474.

29) Ethyl 4-(2,4-diphenyl-1,2,4-triazolidin-1-yl)benzoate (31)
Prepared according to the general procedure from (E)-4-(phenyldiazenyl)phenyl propionate (50.8 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 31.

Colorless oil; yield: 59.8 mg (80%).

1H NMR (400 MHz, CDCl₃): δ 7.99 (d, J = 8.8 Hz, 2H), 7.35–7.25 (m, 4H), 7.18–7.12 (m, 4H), 7.02 (t, J = 7.2 Hz, 1H), 6.83 (t, J = 7.2 Hz, 1H), 6.63–6.60 (d, J = 7.6 Hz, 2H), 4.97–4.85 (s, 3H), 4.60 (s, 1H), 4.36 (q, J = 7.2 Hz, 2H), 1.39 (t, J = 7.2 Hz, 3H).

13C NMR (150 MHz, CDCl₃): δ 166.4, 153.0, 149.8, 144.9, 131.2, 129.4, 129.3, 122.8, 122.3, 118.8, 115.4, 113.4, 67.8, 66.0, 60.5, 14.4.


Prepared according to the general procedure from (E)-4-(phenyldiazenyl)benzonitrile (41.4 mg, 0.2 mmol) and N-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 100:1) to afford the pure product 32.

White solid, m.p. 143–144 °C; yield: 45.0 mg (69%).

1H NMR (400 MHz, CDCl₃): δ 7.58–7.53 (m, 2H), 7.35–7.30 (m, 2H), 7.29–7.25 (m, 2H), 7.16–7.11 (m, 4H), 7.07–7.02 (m, 1H), 6.89–6.82 (m, 1H), 6.66–6.62 (m, 2H), 5.02–4.94 (m, 1H), 4.89–4.81 (m, 2H), 4.57 (s, 1H).

13C NMR (150 MHz, CDCl₃): δ 152.4, 149.4, 144.7, 133.5, 129.4, 129.3, 122.7, 119.5, 119.2, 115.5, 113.9, 113.6, 103.1, 68.3, 65.8.


31) 1-(4-Nitrophenyl)-2,4-diphenyl-1,2,4-triazolidine (33)
Prepared according to the general procedure from \((E)-1\)-(4-nitrophenyl)-2-phenyl diazene (45.4 mg, 0.2 mmol) and \(N\)-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 50:1) to afford the pure product 33.

Yellow solid, m.p. 130–131°C; yield: 53.3 mg (77%).

\[ ^1H \text{NMR (400 MHz, CDCl}_3\):} \delta 8.20–8.16 (m, 2H), 7.36–7.26 (m, 4H), 7.15–7.11 (m, 2H), 7.10–7.04 (m, 3H), 6.87 (t, \(J = 7.4 \text{ Hz}, 1H\)), 6.66 (d, \(J = 8.0 \text{ Hz}, 2H\)), 4.99 (s, 2H), 4.84 (s, 1H), 4.57 (s, 1H). \]

\[ ^{13}C \text{NMR (150 MHz, CDCl}_3\):} \delta 153.6, 149.3, 144.6, 140.9, 129.5, 129.4, 125.8, 123.1, 119.4, 115.8, 113.7, 112.7, 68.8, 65.6. \]

HRMS (ESI): \(m/z [M+H]^+\) calcd for C\(_{20}\)H\(_{19}\)O\(_2\)N\(_4\): 347.1503; found: 347.1505.

32) \(1\)-(4-(Methylsulfonyl)phenyl)-2,4-diphenyl-1,2,4-triazolidine (34)

Prepared according to the general procedure from \((E)-1\)-(4-(methylsulfonyl)phenyl)-2-phenyl diazene (52.0 mg, 0.2 mmol) and \(N\)-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 50:1) to afford the pure product 34.

White solid, m.p. 183–184 °C; yield: 39.7 mg (76%).

\[ ^1H \text{NMR (400 MHz, CDCl}_3\):} \delta 7.75 (d, \(J = 8.8 \text{ Hz}, 2H\)), 7.26–7.22 (m, 2H)), 7.21–7.15 (m, 2H), 7.14–7.10 (m, 2H), 7.05 (d, \(J = 8.0 \text{ Hz}, 2H\)), 6.96 (t, \(J = 7.4 \text{ Hz}, 1H\)), 6.76 (t, \(J = 7.2 \text{ Hz}, 1H\)), 6.55 (d, \(J = 8.0 \text{ Hz}, 2H\)), 4.91–4.76 (m, 3H), 4.50 (s, 1H), 2.94 (s, 3H). \]

\[ ^{13}C \text{NMR (150 MHz, CDCl}_3\):} \delta 153.4, 149.4, 144.7, 131.7, 129.5, 129.3, 129.1, 122.7, 119.1, 115.6, 113.7, 113.6, 68.3, 66.0, 44.9. \]

HRMS (ESI): \(m/z [M+H]^+\) calcd for C\(_{21}\)H\(_{22}\)O\(_2\)N\(_3\): 380.1427; found: 380.1434.

33) \(1\)-(4-(2,4-Diphenyl-1,2,4-triazolidin-1-yl)phenyl)ethan-1-one (35)
Prepared according to the general procedure from \((E)\)-1-(4-(phenyldiazenyl)phenyl)ethan-1-one (44.8 mg, 0.2 mmol) and \(N\)-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 100:1) to afford the pure product 35.

White solid, m.p. 117–118 °C; yield: 43.27 mg (63%).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.95–7.90 (m, 2H), 7.36–7.29 (m, 2H), 7.28–7.23 (m, 2H), 7.18–7.11 (m, 4H), 7.02 (s, 1H), 6.83 (t, \(J = 7.2\) Hz, 1H), 6.63 (d, \(J = 8.0\) Hz, 2H), 4.98 (s, 1H), 4.87 (s, 2H), 4.59 (s, 1H), 2.55 (s, 3H).

\(^{13}\)C NMR (150 MHz, CDCl\(_3\)): \(\delta\) 196.6, 153.1, 149.7, 144.8, 130.3, 130.1, 129.4, 129.3, 122.4, 118.9, 115.5, 113.5, 113.2, 68.0, 65.9, 26.2.

HRMS (ESI): \(m/z\) [M+H]\(^+\) calcd for C\(_{20}\)H\(_{19}\)FN\(_3\): 344.1757; found: 344.1754.

34) 1-(3-Chlorophenyl)-2,4-diphenyl-1,2,4-triazolidine (36)

Prepared according to the general procedure from \((E)\)-1-(3-chlorophenyl)-2-phenyldiazenene (43.3 mg, 0.2 mmol) and \(N\)-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 36.

White solid, m.p. 95–96 °C; yield: 54.4 mg (81%).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 7.35–7.22 (m, 6H), 7.21–7.17 (m, 2H), 7.05–6.99 (m, 2H), 6.97–6.92 (m, 1H), 6.83 (t, \(J = 7.2\) Hz, 1H), 6.62 (d, \(J = 8.0\) Hz, 2H), 4.90–4.67 (m, 4H).

\(^{13}\)C NMR (150 MHz, CDCl\(_3\)): \(\delta\) 151.3, 149.8, 144.9, 130.3, 130.1, 129.4, 129.2, 122.0, 121.4, 118.7, 115.1, 114.9, 113.3, 113.0, 67.4, 66.9.

HRMS (ESI): \(m/z\) [M+H]\(^+\) calcd for C\(_{20}\)H\(_{19}\)ClN\(_3\): 336.1262; found: 336.1263.

35) 1,2-Diphenyl-4-(p-tolyl)-1,2,4-triazolidine (37)
Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and p-tolylglycine (82.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 37.

White solid, m.p. 94–95 °C; yield: 56.1 mg (89%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.32–7.27 (m, 4H), 7.20–7.16 (m, 4H), 7.07–7.03 (m, 2H), 6.99–6.94 (m, 2H), 6.56–6.52 (m, 2H), 4.85 (s, 2H), 4.68 (s, 2H), 2.25 (s, 3H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 150.2, 143.1, 129.8, 129.1, 127.8, 121.4, 114.9, 113.5, 67.5, 20.4.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{21}$H$_{22}$N$_3$: 316.1808; found: 316.1806.

36) 4-(4-Isopropylphenyl)-1,2-diphenyl-1,2,4-triazolidine (38)

Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (4-isopropylphenyl)glycine (96.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 38.

White solid, m.p. 88–90 °C; yield: 57.7 mg (84%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.36–7.29 (m, 4H), 7.24–7.19 (m, 4H), 7.16–7.12 (m, 2H), 7.01–6.96 (m, 2H), 6.62–6.57 (m, 2H), 4.89 (s, 2H), 4.71 (s, 2H), 2.90–280 (m, 1H), 1.24 (d, $J = 7.2$ Hz, 6H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 150.2, 143.3, 139.0, 129.2, 127.3, 121.5, 115.0, 113.4, 67.5, 33.2, 24.2.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{23}$H$_{26}$N$_3$: 344.2121; found: 344.2122.

37) 4-(4-(Tert-butyl)phenyl)-1,2-diphenyl-1,2,4-triazolidine (39)

Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and
(4-(tert-butyl)phenyl)glycine (103.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 39. Colorless oil; yield: 59.3 mg (83%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.30–7.25 (m, 6H), 7.18–7.15 (m, 4H), 6.97–6.92 (m, 2H), 6.57–6.54 (m, 2H), 4.85 (s, 2H), 4.68 (s, 2H), 1.26 (s, 9H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 150.2, 142.8, 141.2, 129.1, 126.1, 115.0, 113.0, 67.3, 33.9, 31.4.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{24}$H$_{28}$N$_3$: 358.2278; found: 358.2277.

38) 4-(4-Fluorophenyl)-1,2-diphenyl-1,2,4-triazolidine (40)

![4-(4-Fluorophenyl)-1,2-diphenyl-1,2,4-triazolidine (40)](image)

Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (4-fluorophenyl)glycine (84.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 40. White solid, m.p. 114–115 °C; yield: 53.7 mg (84%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.33–7.27 (m, 4H), 7.20–7.16 (m, 4H), 7.01–6.92 (m, 4H), 6.57–6.52 (m, 2H), 4.82 (s, 2H), 4.68 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 156.5 (d, $J = 235.5$ Hz), 150.1, 141.9 (d, $J = 1.5$ Hz), 129.2, 121.6, 115.8 (d, $J = 22.5$ Hz), 114.5 (d, $J = 7.5$ Hz), 114.4, 67.9.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{20}$H$_{19}$FN$_3$: 320.1558; found: 320.1555.

39) 4-(4-Chlorophenyl)-1,2-diphenyl-1,2,4-triazolidine (41)

![4-(4-Chlorophenyl)-1,2-diphenyl-1,2,4-triazolidine (41)](image)

Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and
(4-chlorophenyl)glycine (92.8 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 41.

White solid, m.p. 132–133 °C; yield: 57.1 mg (85%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.30 (t, $J = 7.8$ Hz, 4H), 7.22–7.15 (m, 6H), 7.00–6.95 (m, 2H), 6.52–6.48 (m, 2H), 4.83 (s, 2H), 4.66 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 149.9, 143.6, 129.21, 129.19, 123.2, 121.7, 115.0, 114.3, 67.1.


40) 4-(4-Bromophenyl)-1,2-diphenyl-1,2,4-triazolidine (42)

Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (4-bromophenyl)glycine (115.0 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 42.

White solid, m.p. 137–138 °C; yield: 62.4 mg (82%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.33–7.28 (m, 6H), 7.20–7.15 (m, 4H), 7.01–6.95 (m, 2H), 6.49–6.42 (m, 2H), 4.83 (s, 2H), 4.65 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 149.9, 143.9, 132.1, 129.2, 121.7, 115.0, 114.7, 110.3, 67.0.


41) 4-(4-Methoxyphenyl)-1,2-diphenyl-1,2,4-triazolidine (43)

Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (4-methoxyphenyl)glycine (90.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on
silica gel (PE: EtOAc = 200:1) to afford the pure product 43.

White solid, m.p. 70–71 °C; yield: 47.1 mg (71%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.30–7.26 (m, 4H), 7.17–7.11 (m, 4H), 6.98–6.91 (m, 2H), 6.83–6.78 (m, 2H), 6.63–6.58 (m, 2H), 4.79 (s, 2H), 4.67 (s, 2H), 3.74 (s, 3H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 152.9, 150.3, 140.0, 129.1, 121.3, 115.1, 114.9, 114.9, 68.4, 55.7.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{21}$H$_{22}$ON$_3$: 332.1757; found: 332.1758.

42) 1,2-Diphenyl-4-(m-tolyl)-1,2,4-triazolidine (44)

Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and m-tolylglycine (82.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 44.

White solid, m.p. 118–119 °C; yield: 47.9 mg (76%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.32–7.26 (m, 4H), 7.20–7.16 (m, 4H), 7.15–7.08 (m, 1H), 6.98–6.92 (m, 2H), 6.63–6.58 (m, 1H), 6.43–6.39 (m, 2H), 4.86 (s, 2H), 4.68 (s, 2H), 2.30 (s, 3H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 150.1, 145.1, 139.2, 129.2, 129.1, 121.5, 119.3, 115.0, 114.0, 110.4, 67.1, 21.7.

HRMS (ESI): $m/z$ [M+H]$^+$ calcd for C$_{21}$H$_{22}$N$_3$: 316.1808; found: 316.1810.

43) 4-(3-Chlorophenyl)-1,2-diphenyl-1,2,4-triazolidine (45)
Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (3-chlorophenyl)glycine (92.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 45.

White solid, m.p. 99–101 °C; yield: 49.0 mg (73%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.33–7.28 (m, 4H), 7.20–7.17 (m, 4H), 7.13 (t, $J = 8.0$ Hz, 1H), 7.02–6.94 (m, 2H), 6.77–6.73 (m, 1H), 6.58–6.55 (m, 1H), 6.47–6.42 (m, 1H), 4.88–6.63 (m, 4H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 149.8, 145.8, 135.1, 130.3, 129.2, 121.8, 118.1, 115.0, 112.9, 111.2, 66.7.


44) 1,2-Diphenylhydrazine (46)$^{[4]}$

\[
\text{\begin{center}\includegraphics[scale=0.5]{12-dihydrazone.png}\end{center}}
\]

Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (2-chlorophenyl)glycine (92.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product 46.

White solid; yield: 12.9 mg (35%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.22–7.13 (m, 4H), 7.02–6.95 (m, 4H), 6.84–6.76 (m, 2H), 5.39 (s, 2H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 147.7, 129.1, 120.0, 114.1.

45) N$^1$N$^2$-Diphenylethane-1,2-diamine(47)$^{[5]}$

\[
\text{\begin{center}\includegraphics[scale=0.5]{n1n2_diamine.png}\end{center}}
\]

Prepared according to the general procedure from N-aryl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 50:1) to afford the pure product 47.

White solid; yield: 7.9 mg (15%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.34–7.28 (m, 4H), 6.87–6.74 (m, 2H), 6.72–6.65 (m, 4H), 4.67 (s, 2H), 3.66 (s, 4H).

$^{13}$C NMR (150 MHz, CDCl$_3$): $\delta$ 146.4, 129.3, 117.6, 112.4, 46.5.
9. References


10. Copy of NMR Spectra

$^1$H NMR spectrum of 3 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 3 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 4 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 4 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 5 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 5 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 6 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 6 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 7 (400 MHz, CDCl$_3$):

13C NMR spectrum of 7 (150 MHz, CDCl$_3$):
$^{19}$F NMR spectrum of 7 (376 MHz, CDCl$_3$):

$^1$H NMR spectrum of 8 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 8 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 9 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 9 (150 MHz, CDCl$_3$):

![Spectrum 9]

$^1$H NMR spectrum of 10 (400 MHz, CDCl$_3$):

![Spectrum 10]
$^{13}$C NMR spectrum of 10 (150 MHz, CDCl$_3$):

![13C NMR spectrum of 10](image)

$^{19}$F NMR spectrum of 10 (376 MHz, CDCl$_3$):

![19F NMR spectrum of 10](image)
$^1$H NMR spectrum of 11 (400 MHz, CDCl$_3$):

![H NMR spectrum of 11 (400 MHz, CDCl$_3$)](image)

$^{13}$C NMR spectrum of 11 (150 MHz, CDCl$_3$):

![$^{13}$C NMR spectrum of 11 (150 MHz, CDCl$_3$)](image)
$^{19}$F NMR spectrum of 11 (376 MHz, CDCl$_3$):

$^1$H NMR spectrum of 12 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 12 (100 MHz, CDCl$_3$):

![C NMR spectrum of 12](image)

$^1$H NMR spectrum of 13 (400 MHz, CDCl$_3$):

![H NMR spectrum of 13](image)
$^{13}$C NMR spectrum of 13 (150 MHz, CDCl$_3$):

![13C NMR spectrum of 13](image)

$^{19}$F NMR spectrum of 13 (376 MHz, CDCl$_3$):

![$^{19}$F NMR spectrum of 13](image)
$^1$H NMR spectrum of 14 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 14 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 15 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 15 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 16 (400 MHz, CDCl$_3$): 

![H NMR spectrum of 16](image)

$^{13}$C NMR spectrum of 16 (150 MHz, CDCl$_3$): 

![C NMR spectrum of 16](image)
$^1$H NMR spectrum of 17 (400 MHz, CDCl$_3$):

![H NMR spectrum of 17](image)

$^{13}$C NMR spectrum of 17 (150 MHz, CDCl$_3$):

![C NMR spectrum of 17](image)
$^{19}$F NMR spectrum of 17 (376 MHz, CDCl$_3$):

![F NMR spectrum of 17](image)

$^1$H NMR spectrum of 18 (400 MHz, CDCl$_3$):

![H NMR spectrum of 18](image)
$^{13}$C NMR spectrum of 18 (150 MHz, CDCl$_3$):

![Carbon NMR spectrum of 18](image1)

$^1$H NMR spectrum of 19 (400 MHz, CDCl$_3$):

![Hydrogen NMR spectrum of 19](image2)
$^{13}$C NMR spectrum of 19 (150 MHz, CDCl$_3$): 

1H NMR spectrum of 20 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 20 (150 MHz, CDCl$_3$):

![NMR spectrum of 20](image)

$^1$H NMR spectrum of 21 (400 MHz, CDCl$_3$):

![NMR spectrum of 21](image)
$^{13}$C NMR spectrum of 21 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 22 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 22 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 23 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 23 (150 MHz, CDCl$_3$):

![Carbon NMR spectrum of 23](image)

$^1$H NMR spectrum of 24 (400 MHz, CDCl$_3$):

![Hydrogen NMR spectrum of 24](image)
$^{13}$C NMR spectrum of 24 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 25 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 25 (150 MHz, CDCl$_3$):

![Carbon NMR spectrum](image)

$^{19}$F NMR spectrum of 25 (376 MHz, CDCl$_3$):

![Fluorine NMR spectrum](image)
$^1$H NMR spectrum of 26 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 26 (150 MHz, CDCl$_3$):
\(^{1}\)H NMR spectrum of 27 (400 MHz, CDCl\(_3\)):

\[ \begin{array}{c}
\text{27} \\
\end{array} \]

\(^{13}\)C NMR spectrum of 27 (150 MHz, CDCl\(_3\)):
$^1$H NMR spectrum of 28 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 28 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 29 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 29 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 30 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 30 (150 MHz, CDCl$_3$):
$^{19}$F NMR spectrum of **30** (376 MHz, CDCl$_3$):

![Chemical structure of 30 with NMR spectrum]

$^1$H NMR spectrum of **31** (400 MHz, CDCl$_3$):

![Chemical structure of 31 with NMR spectrum]
$^{13}$C NMR spectrum of 31 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 32 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 32 (150 MHz, CDCl$_3$):

![C NMR spectrum of 32](image)

$^1$H NMR spectrum of 33 (400 MHz, CDCl$_3$):

![H NMR spectrum of 33](image)
\(^{13}\)C NMR spectrum of 33 (150 MHz, CDCl\(_3\)):

\(^{1}\)H NMR spectrum of 34 (400 MHz, CDCl\(_3\)):
$^{13}$C NMR spectrum of 34 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 35 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of **35** (150 MHz, CDCl$_3$):

![13C NMR spectrum of 35](image)

$^1$H NMR spectrum of **36** (400 MHz, CDCl$_3$):

![1H NMR spectrum of 36](image)
$^{13}$C NMR spectrum of 36 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 37 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 37 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 38 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 38 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 39 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 39 (150 MHz, CDCl$_3$):

$^1$H NMR spectrum of 40 (400 MHz, CDCl$_3$):
$^{13}$C NMR spectrum of 40 (150 MHz, CDCl$_3$):

$^{19}$F NMR spectrum of 40 (376 MHz, CDCl$_3$):
$^1$H NMR spectrum of 41 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 41 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of \textbf{42} (400 MHz, CDCl$_3$):

\begin{center}
\includegraphics[width=0.5\textwidth]{hnmr_spectrum.png}
\end{center}

$^{13}$C NMR spectrum of \textbf{42} (150 MHz, CDCl$_3$):

\begin{center}
\includegraphics[width=0.5\textwidth]{cnmr_spectrum.png}
\end{center}
$^1$H NMR spectrum of 43 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 43 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 44 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 44 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 45 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 45 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 46 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 46 (150 MHz, CDCl$_3$):
$^1$H NMR spectrum of 47 (400 MHz, CDCl$_3$):

$^{13}$C NMR spectrum of 47 (150 MHz, CDCl$_3$):