

# Metal-free $\alpha$ -arylation of $\alpha$ -fluoro- $\alpha$ -nitroacetamides employing diaryliodonium salts

## SUPPORTING INFORMATION

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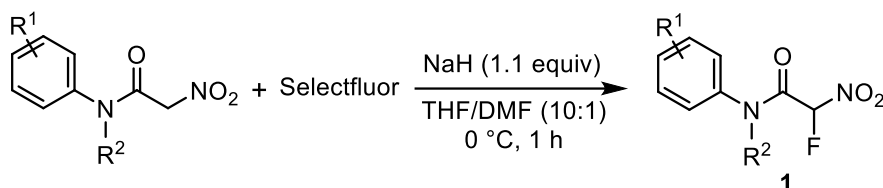
## General experimental information

Unless otherwise specified, all reactions were performed under air atmosphere in oven dried round-bottom flasks. The reactions were monitored by TLC visualized by UV (254 nm) and/or with iodine. Column chromatography was performed on 100-200 mesh silica gel using the gradient system ethyl acetate-hexane. NMR data were recorded at Bruker AV 400 MHz in CDCl<sub>3</sub> using as internal standards the residual CHCl<sub>3</sub> signal for <sup>1</sup>H NMR ( $\delta$  = 7.26 ppm) and the deuterated solvent signal for <sup>13</sup>C NMR ( $\delta$  = 77.16 ppm). Coupling constants are given in Hertz (Hz) and the classical abbreviations are used to describe the signal multiplicities. Melting points were measured with a Büchi B-540 apparatus and are uncorrected. High resolution mass spectra were obtained using Q-TOF mass spectrometer. All commercially available reagents were used as received.  $\alpha$ -nitroacetamides<sup>1</sup> (**1a-1x**),  $\alpha$ -cyanoacetamides<sup>2</sup> (**4a-4p**) and diaryliodonium salts (**2a-2w**) were prepared by following a literature procedure.<sup>3</sup>

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1. S. Mo, P. Huang and J. Xu, *Org. Biomol. Chem.*, 2014, **12**, 4192–4200.
  2. H. Hao, J. Mingyan, X. Lijun, H. Gang, Z. Cuirong, Z. Lixia, Z. Shunguang, Z. Meihui and G. Ping, *Chem. Res. Chin. Univ.* 2015, **31**, 746–755.
  3. S. K. Sundalam, A. Nilova, T. L. Seidl and D. R. Stuart, *Angew. Chem., Int. Ed.* 2016, **55**, 8431–8434.

## Procedures

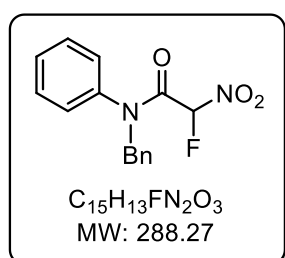
### General procedure for the synthesis of $\alpha$ -fluoro- $\alpha$ -nitroacetamides **1**



To a solution of  $\alpha$ -nitroacetamide (1.50 mmol) in THF (20 mL) was added NaH (1.65 mmol) at 0 °C. The resulting mixture was stirred at the same temperature for 30 min. Then selectfluor (1.5 mmol) solution in DMF (2 mL) was added dropwise to the mixture at 0 °C, and the reaction mixture was stirred to room temperature for 1 h. The reaction mixture was quenched by addition of water (10 mL) and the mixture was extracted with ethyl acetate (25 mL  $\times$  3). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and evaporated under reduced pressure. The residue was purified using column chromatography (100-200 mesh silica gel) using ethyl acetate /hexane as the eluent to afford the desired  $\alpha$ -fluoro- $\alpha$ -nitroacetamide **1** (40-76% yield) and the side product  $\alpha,\alpha$ -difluoronitroacetamide in traces. The same strategy was followed for the synthesis of  $\alpha$ -cyano- $\alpha$ -fluoroacetamides **4**.

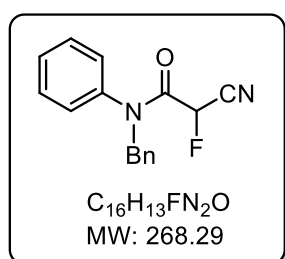
### Characterization data of compounds **1a** and **4h**

#### Compound **1a**: *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide



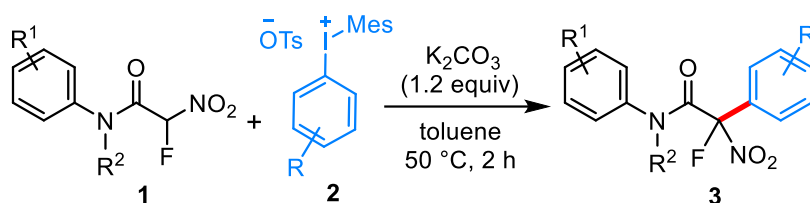
White solid, 259 mg, 60%; **Mp** 72-74 °C. **<sup>13</sup>C NMR** (100 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 159.1 (d,  $J_{\text{C-F}}$  = 22.3 Hz, C), 138.8 (C), 135.2 (C), 130.4 (CH), 130.4 (CH), 129.9 (CH), 129.1 (CH), 129.1 (CH), 128.8 (CH), 128.8 (CH), 128.6 (CH), 128.6 (CH), 128.3 (CH), 100.7 (d,  $J_{\text{C-F}}$  = 243.5 Hz, CH), 54.3 (CH<sub>2</sub>). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 7.44-7.40 (m, 3H), 7.31-7.28 (m, 3H), 7.19-7.17 (m, 2H), 7.13-7.11 (m, 2H), 5.86 (d,  $J$  = 48.8 Hz, 1H), 4.96 (s, 2H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): -145.5 (s). **HRMS** for C<sub>15</sub>H<sub>14</sub>FN<sub>2</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+H]<sup>+</sup>: 289.0983, found: 289.0982.

#### Compound **4h**: *N*-benzyl-2-cyano-2-fluoro-*N*-phenylacetamide



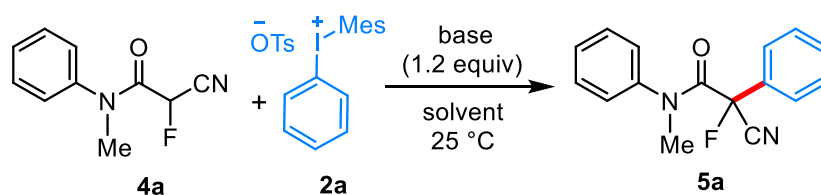
Colorless liquid, 185 mg, 46%; **<sup>13</sup>C NMR** (100 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 159.8 (d,  $J_{\text{C-F}}$  = 22.6 Hz, C), 138.2 (C), 135.4 (C), 130.4 (CH), 130.4 (CH), 130.0 (CH), 129.3 (CH), 129.3 (CH), 128.8 (CH), 128.8 (CH), 128.8 (CH), 128.8 (CH), 128.3 (CH), 112.6 (d,  $J_{\text{C-F}}$  = 30.8 Hz, C), 74.5 (d,  $J_{\text{C-F}}$  = 189.8 Hz, CH), 54.5 (CH<sub>2</sub>). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 7.44-7.38 (m, 3H), 7.30-7.27 (m, 3H), 7.19-7.17 (m, 2H), 7.06-7.04 (m, 2H), 5.36 (d,  $J$  = 46.8 Hz, 1H), 4.97 (d,  $J$  = 14.4 Hz, 1H), 4.88 (d,  $J$  = 14.0 Hz, 1H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): -189.0 (s). **HRMS** for C<sub>16</sub>H<sub>14</sub>FN<sub>2</sub>O<sup>+</sup>: calcd. [M+H]<sup>+</sup>: 269.1085, found: 269.1085.

### General procedure for the $\alpha$ -arylation of $\alpha$ -fluoro- $\alpha$ -nitroacetamides



To an oven-dried round bottom flask were added  $\alpha$ -fluoro- $\alpha$ -nitroacetamide **1** (0.20 mmol), aryl(mesityl)iodonium salt **2** (0.22 mmol),  $K_2CO_3$  (0.24 mmol) under open air respectively and dissolved in toluene (2.0 mL). The reaction mixture was allowed to stir at 50 °C for 2 h. After the completion of reaction, as indicated by TLC, the reaction mixture was extracted using ethyl acetate (3  $\times$  10 mL) and washed with saturated brine solution. The organic layer was dried over anhydrous  $Na_2SO_4$  and evaporated under reduced pressure. The residue was purified using column chromatography (100-200 mesh silica gel) using ethylacetate/hexane as the eluent to afford the product **3**.

### Optimization of the Reaction Conditions for $\alpha$ -arylation of $\alpha$ -cyano- $\alpha$ -fluoroacetamides **4**



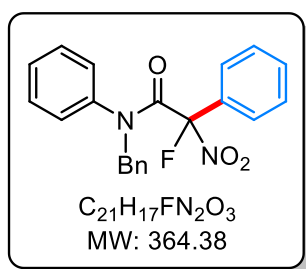
| entry    | base (1.2 equiv)     | solvent    | temperature (°C) | time (h)   | yield (%) |
|----------|----------------------|------------|------------------|------------|-----------|
| 1        | $K_2CO_3$            | toluene    | 50               | 6          | 37        |
| 2        | <i>t</i> -BuOK       | toluene    | 50               | 6          | 40        |
| 3        | <i>t</i> -BuOK       | DCM        | 25               | 6          | 54        |
| <b>4</b> | <b><i>t</i>-BuOK</b> | <b>THF</b> | <b>25</b>        | <b>0.5</b> | <b>70</b> |
| 5        | <i>t</i> -BuOK       | dioxane    | 25               | 6          | NR        |
| 6        | <i>t</i> -BuOK       | TBME       | 25               | 6          | 42        |
| 7        | $K_2CO_3$            | THF        | 25               | 6          | 36        |
| 8        | $Na_2CO_3$           | THF        | 25               | 6          | NR        |
| 9        | KOH                  | THF        | 25               | 6          | 50        |
| 10       | NaH                  | THF        | 25               | 6          | 47        |

### General procedure for the $\alpha$ -arylation of $\alpha$ -cyano- $\alpha$ -fluoroacetamides

To an oven-dried round bottom flask were added  $\alpha$ -cyano- $\alpha$ -fluoroacetamide **4** (0.20 mmol) and aryl(mesityl)iodonium salt **2** (0.22 mmol), which were then dissolved in THF (2.0 mL). Subsequently, *t*-BuOK (0.24 mmol) was added portionwise. The reaction mixture was allowed

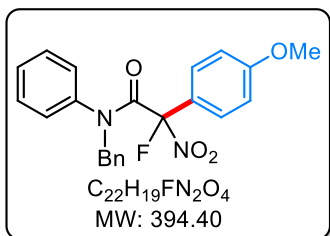
to stir at 25 °C for 0.5 h. After the completion of reaction, as indicated by TLC, the reaction mixture was extracted using ethyl acetate (3 × 10 mL) and washed with saturated brine solution. Finally, the organic layer dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and evaporated under reduced pressure. The residue was purified using column chromatography (100-200 mesh silica gel) using ethylacetate/hexane as the eluent to afford the product **5**.

### Compound 3a: *N*-benzyl-2-fluoro-2-nitro-*N*,2-diphenylacetamide



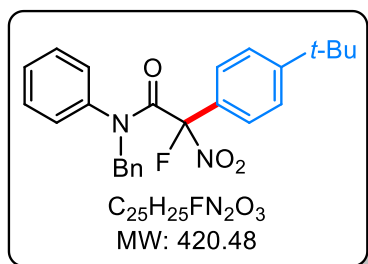
Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3a** (62 mg, 85%) as white solid. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.40. *Mp* 97-99 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.8 (d, *J*<sub>C-F</sub> = 21.5 Hz, C), 138.5 (C), 135.7 (C), 131.2 (CH), 130.4 (d, *J*<sub>C-F</sub> = 21.6 Hz, C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 128.8 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.4 (CH), 128.4 (CH), 128.2 (CH), 126.3 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 126.3 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 116.2 (d, *J*<sub>C-F</sub> = 255.3 Hz, C), 56.1 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.42-6.27 (m, 15H), 4.95 (d, *J* = 14.0 Hz, 1H), 4.89 (d, *J* = 14.4 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.6 (s). HRMS for C<sub>21</sub>H<sub>21</sub>FN<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 382.1561, found: 382.1552.

### Compound 3b: *N*-benzyl-2-fluoro-2-(4-methoxyphenyl)-2-nitro-*N*-phenylacetamide



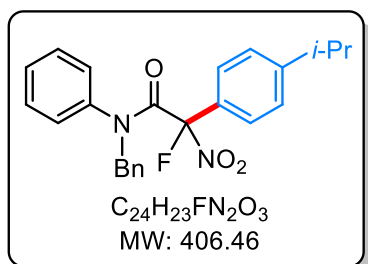
Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(4-methoxyphenyl)iodonium 4-methylbenzenesulfonate **2b** (115 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 3 h followed by column chromatography afforded the product **3b** (55 mg, 70%) as white solid. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.30. *Mp* 95-97 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 161.8 (C), 161.0 (d, *J*<sub>C-F</sub> = 16.8 Hz, C), 138.7 (C), 135.7 (C), 129.3 (CH), 129.3 (CH), 129.2 (CH), 129.2 (CH), 128.8 (CH), 128.8 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.1 (CH), 128.0 (d, *J*<sub>C-F</sub> = 7.1 Hz, CH), 128.0 (d, *J*<sub>C-F</sub> = 7.1 Hz, CH), 122.4 (d, *J*<sub>C-F</sub> = 17.4 Hz, C), 116.5 (d, *J*<sub>C-F</sub> = 204.8 Hz, C), 113.9 (CH), 113.9 (CH), 56.1 (CH<sub>2</sub>), 55.5 (CH<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.31-7.15 (m, 11H), 6.82-6.40 (m, 3H), 4.94 (d, *J* = 13.6 Hz, 1H), 4.83 (d, *J* = 14.0 Hz, 1H), 3.80 (s, 3H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.2 (s). HRMS for C<sub>22</sub>H<sub>23</sub>FN<sub>3</sub>O<sub>4</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 412.1667, found: 412.1669.

### Compound 3c: *N*-benzyl-2-(4-(*t*-butyl)phenyl)-2-fluoro-2-nitro-*N*-phenylacetamide



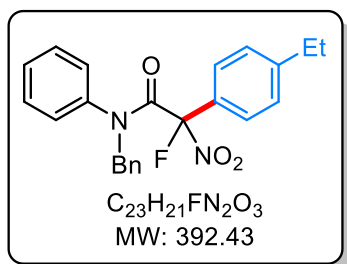
Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (4-(*t*-butyl)phenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2c** (121 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3c** (67 mg, 80%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.40. **mp** 120-122 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 161.0 (d,  $J_{C-F}$  = 21.5 Hz, C), 154.6 (C), 138.6 (C), 135.8 (C), 129.4 (CH), 129.4 (CH), 129.3 (CH), 129.3 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.6 (CH), 128.6 (CH), 128.1 (CH), 127.5 (d,  $J_{C-F}$  = 22.0 Hz, C), 126.1 (d,  $J_{C-F}$  = 8.6 Hz, CH), 126.1 (d,  $J_{C-F}$  = 8.6 Hz, CH), 125.4 (CH), 125.4 (CH), 116.2 (d,  $J_{C-F}$  = 253.5 Hz, C), 56.1 ( $CH_2$ ), 35.0 (C), 31.2 ( $CH_3$ ), 31.2 ( $CH_3$ ), 31.2 ( $CH_3$ ).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.30 (s, 4H), 7.26-7.24 (m, 3H), 7.17-7.03 (m, 7H), 4.90 (d,  $J$  = 11.2 Hz, 1H), 4.85 (d,  $J$  = 11.2 Hz, 1H), 1.29 (s, 9H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -126.8 (s). **HRMS** for  $C_{25}H_{29}FN_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 438.2187, found: 438.2178.

### Compound 3d: *N*-benzyl-2-fluoro-2-(4-*i*-propylphenyl)-2-nitro-*N*-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (4-*i*-propylphenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2d** (118 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3d** (53 mg, 65%) as pale yellow solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.45. **mp** 108-110 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 161.0 (d,  $J_{C-F}$  = 21.6 Hz, C), 152.4 (C), 138.6 (C), 135.8 (C), 129.4 (CH), 129.4 (CH), 129.3 (CH), 129.3 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.6 (CH), 128.1 (CH), 127.8 (d,  $J_{C-F}$  = 21.5 Hz, C), 126.5 (CH), 126.5 (CH), 126.3 (d,  $J_{C-F}$  = 8.7 Hz, CH), 126.3 (d,  $J_{C-F}$  = 8.7 Hz, CH), 116.3 (d,  $J_{C-F}$  = 254.8 Hz, C), 56.1 ( $CH_2$ ), 34.1 (CH), 23.9 ( $CH_3$ ), 23.8 ( $CH_3$ ).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.28-7.24 (m, 5H), 7.15-6.40 (m, 9H), 4.88 (s, 2H), 2.89 (s, 1H), 1.22 (s, 6H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.1 (s). **HRMS** for  $C_{24}H_{23}FN_2NaO_3^+$ : calcd.  $[M+Na]^+$ : 429.1585, found: 429.1577.

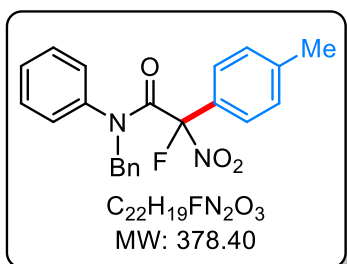
### Compound 3e: *N*-benzyl-2-(4-ethylphenyl)-2-fluoro-2-nitro-*N*-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(4-ethylphenyl)iodonium 4-methylbenzenesulfonate **2e** (115 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product (4-ethylphenyl)(mesityl)iodonium 4-methylbenzenesulfonate **3e**

(72 mg, 92%) as white solid. *R<sub>f</sub>*(Ethyl acetate/Hexane: 10/90) = 0.45. *Mp* 101-103 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 161.0 (d, *J*<sub>C-F</sub> = 21.4 Hz, C), 147.8 (C), 138.6 (C), 135.7 (C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.1 (CH), 127.9 (CH), 127.9 (CH), 127.7 (d, *J*<sub>C-F</sub> = 21.3 Hz, C), 126.3 (d, *J*<sub>C-F</sub> = 8.5 Hz, CH), 126.3 (d, *J*<sub>C-F</sub> = 8.5 Hz, CH), 116.4 (d, *J*<sub>C-F</sub> = 254.6 Hz, C), 56.1 (CH<sub>2</sub>), 28.8 (CH<sub>2</sub>), 15.4 (CH<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.28-7.22 (m, 5H), 7.17-6.31 (m, 9H), 4.89 (d, *J* = 11.2 Hz, 1H), 4.84 (d, *J* = 11.2 Hz, 1H), 2.62 (q, *J* = 6.0 Hz, 2H), 1.20 (t, *J* = 6.4 Hz, 3H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -127.1 (s). HRMS for C<sub>23</sub>H<sub>25</sub>FN<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 410.1874, found: 410.1863.

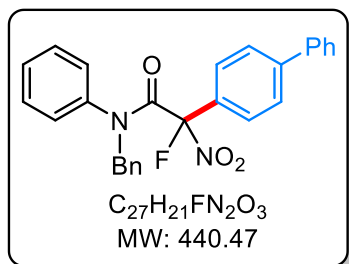
### Compound 3f: *N*-benzyl-2-fluoro-2-nitro-*N*-phenyl-2-(*p*-tolyl)acetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(*p*-tolyl)iodonium 4-methylbenzenesulfonate **2f** (112 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3f** (72 mg, 95%) as white solid. *R<sub>f</sub>*(Ethyl acetate/Hexane: 10/90) = 0.45. *Mp* 99-101 °C. <sup>13</sup>C

NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.9 (d, *J*<sub>C-F</sub> = 17.0 Hz, C), 141.6 (C), 138.7 (C), 135.8 (C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.1 (CH), 129.1 (CH), 128.8 (CH), 128.8 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.1 (CH), 127.6 (d, *J*<sub>C-F</sub> = 17.3 Hz, C), 126.3 (d, *J*<sub>C-F</sub> = 6.8 Hz, CH), 126.3 (d, *J*<sub>C-F</sub> = 6.8 Hz, CH), 116.5 (d, *J*<sub>C-F</sub> = 204.0 Hz, C), 56.1 (CH<sub>2</sub>), 21.4 (CH<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.28-7.24 (m, 5H), 7.20-7.10 (m, 9H), 4.94 (d, *J* = 11.2 Hz, 1H), 4.83 (d, *J* = 11.6 Hz, 1H), 2.35 (s, 3H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.6 (s). HRMS for C<sub>22</sub>H<sub>23</sub>FN<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 396.1718, found: 396.1707.

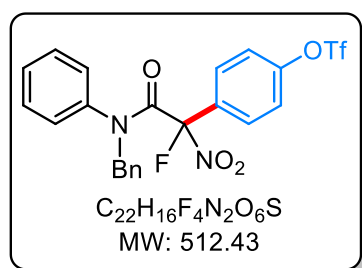
### Compound 3g: 2-([1,1'-biphenyl]-4-yl)-*N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with [1,1'-biphenyl]-4-yl(mesityl)iodonium 4-methylbenzenesulfonate **2g** (126 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3g** (83 mg, 94%) as white solid. *R*<sub>f</sub>(Ethyl acetate/Hexane:

10/90) = 0.40. **Mp** 132-134 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.8 (d, *J*<sub>C-F</sub> = 21.3 Hz, C), 144.0 (C), 139.7 (C), 138.5 (d, *J*<sub>C-F</sub> = 2.5 Hz, C), 135.7 (C), 129.4 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.1 (CH), 129.1 (CH), 129.1 (CH), 128.8 (C), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.3 (CH), 128.2 (CH), 127.3 (CH), 127.3 (CH), 127.0 (CH), 127.0 (CH), 126.8 (d, *J*<sub>C-F</sub> = 8.5 Hz, CH), 126.8 (d, *J*<sub>C-F</sub> = 8.5 Hz, CH), 116.2 (d, *J*<sub>C-F</sub> = 255.7 Hz, C), 56.1 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.55-7.50 (m, 4H), 7.45-7.42 (m, 4H), 7.36 (t, *J* = 1.5 Hz, 1H), 7.26-6.33 (m, 10H), 4.92 (d, *J* = 11.2 Hz, 1H), 4.86 (d, *J* = 11.2 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.3 (s). **HRMS** for C<sub>27</sub>H<sub>25</sub>FN<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 458.1874, found: 458.1874.

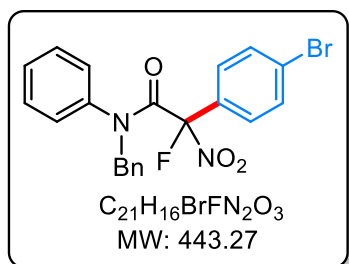
### Compound 3h: 4-(2-(benzyl(phenyl)amino)-1-fluoro-1-nitro-2-oxoethyl)phenyl trifluoromethanesulfonate



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(4-(((trifluoromethyl)sulfonyl)oxy)phenyl)iodonium 4-methylbenzenesulfonate **2h** (141 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3h** (93 mg, 91%) as white solid. *R*<sub>f</sub> (Ethyl

acetate/Hexane: 10/90) = 0.40. **Mp** 79-81 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.2 (d, *J*<sub>C-F</sub> = 21.4 Hz, C), 151.1 (C), 138.0 (d, *J*<sub>C-F</sub> = 2.1 Hz, C), 135.4 (C), 130.7 (d, *J*<sub>C-F</sub> = 22.2 Hz, C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.1 (CH), 129.1 (CH), 129.1 (CH), 128.8 (CH), 128.8 (CH), 128.7 (CH), 128.6 (CH), 128.4 (CH), 121.5 (CH), 121.5 (CH), 118.8 (q, *J*<sub>C-F</sub> = 318.9 Hz, CH), 114.8 (d, *J*<sub>C-F</sub> = 256.6 Hz, C), 56.3 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.53 (d, *J* = 2.2 Hz, 2H), 7.31-7.02 (m, 11H), 6.26 (s, 1H), 4.97 (d, *J* = 14.0 Hz, 1H), 4.86 (d, *J* = 13.6 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -72.7 (s), -121.6 (s). **HRMS** for C<sub>22</sub>H<sub>20</sub>F<sub>4</sub>N<sub>3</sub>O<sub>6</sub>S<sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 530.1003, found: 530.0989.

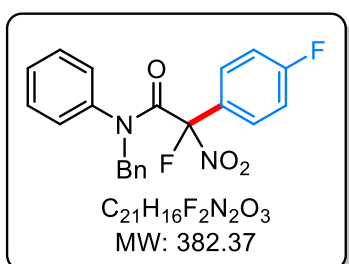
### Compound 3i: *N*-benzyl-2-(4-bromophenyl)-2-fluoro-2-nitro-*N*-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (4-bromophenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2i** (126 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3i** (58 mg, 65%) as white solid.  $R_f$ (Ethyl acetate/Hexane: 10/90) = 0.45. **Mp** 101-103 °C.  $^{13}C$

**NMR** (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.4 (d,  $J_{C-F}$  = 17.0 Hz, C), 138.3 (d,  $J_{C-F}$  = 2.0 Hz, C), 135.5 (C), 131.7 (CH), 131.7 (CH), 129.5 (C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 128.9 (CH), 128.9 (CH), 128.9 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.3 (CH), 127.9 (d,  $J_{C-F}$  = 7.0 Hz, CH), 127.9 (d,  $J_{C-F}$  = 7.0 Hz, CH), 126.0 (C), 115.6 (d,  $J_{C-F}$  = 205.2 Hz, C), 56.2 (CH<sub>2</sub>).  $^1H$  **NMR** (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.47 (d,  $J$  = 7.6 Hz, 2H), 7.29-7.22 (m, 7H), 7.18-7.14 (m, 5H), 4.93 (d,  $J$  = 14.0 Hz, 1H), 4.85 (d,  $J$  = 14.0 Hz, 1H).  $^{19}F$  **NMR** (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.6 (s). **HRMS** for  $C_{21}H_{20}BrFN_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 460.0667, found: 460.0667.

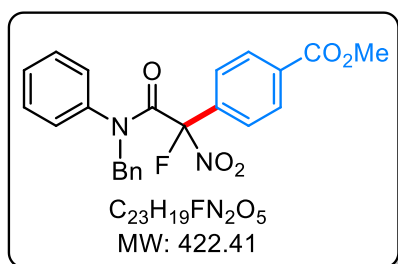
### Compound 3j: *N*-benzyl-2-fluoro-2-(4-fluorophenyl)-2-nitro-*N*-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (4-fluorophenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2j** (113 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3j** (45 mg, 59%) as white solid.  $R_f$ (Ethyl acetate/Hexane: 10/90) = 0.50. **Mp** 119-121 °C.  $^{13}C$

**NMR** (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 164.3 (d,  $J_{C-F}$  = 250.9 Hz, C), 160.6 (d,  $J_{C-F}$  = 21.3 Hz, C), 138.4 (d,  $J_{C-F}$  = 2.6 Hz, C), 135.5 (C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 128.9 (CH), 128.9 (CH), 128.8 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.6 (CH), 128.2 (CH), 126.4 (dd,  $J_{C-F}$  = 21.8 Hz, 3.2 Hz, C), 115.6 (d,  $J_{C-F}$  = 2.2 Hz, CH), 115.6 (d,  $J_{C-F}$  = 2.2 Hz, CH), 115.6 (d,  $J_{C-F}$  = 255.9 Hz, C), 56.1 (CH<sub>2</sub>).  $^1H$  **NMR** (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.44-7.41 (m, 2H), 7.31-7.17 (m, 7H), 7.03 (t,  $J$  = 8.4 Hz, 3H), 6.23 (s, 2H), 4.95 (d,  $J$  = 14.0 Hz, 1H), 4.95 (d,  $J$  = 14.0 Hz, 1H).  $^{19}F$  **NMR** (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -108.3 (s), -121.9 (s). **HRMS** for  $C_{21}H_{20}F_2N_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 400.1467, found: 400.1464.

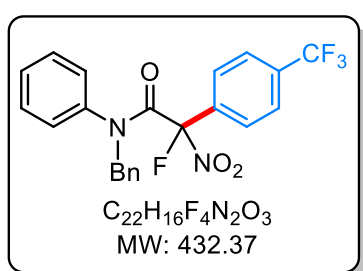
### Compound 3k: methyl 4-(2-(benzyl(phenyl)amino)-1-fluoro-1-nitro-2-oxoethyl)benzoate



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(4-(methoxycarbonyl)phenyl)iodonium 4-methylbenzenesulfonate **2k** (122 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded

the product **3k** (58 mg, 69%) as yellow solid.  $R_f$ (Ethyl acetate/Hexane: 10/90) = 0.50. **Mp** 77-79 °C.  $^{13}\text{C}$  NMR (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 166.1 (C), 160.3 (d,  $J_{\text{C-F}}$  = 16.9 Hz, C), 138.2 (C), 135.5 (C), 134.6 (d,  $J_{\text{C-F}}$  = 17.6 Hz, C), 132.7 (C), 129.5 (CH), 129.5 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.0 (CH), 129.0 (CH), 128.9 (CH), 128.7 (CH), 128.7 (CH), 128.3 (CH), 126.5 (d,  $J_{\text{C-F}}$  = 7.0 Hz, CH), 126.5 (d,  $J_{\text{C-F}}$  = 7.0 Hz, CH), 115.6 (d,  $J_{\text{C-F}}$  = 205.1 Hz, C), 56.2 ( $\text{CH}_2$ ), 52.6 ( $\text{CH}_3$ ).  $^1\text{H}$  NMR (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 7.97 (d,  $J$  = 2.1 Hz, 2H), 7.47 (d,  $J$  = 2.2 Hz, 2H), 7.28-7.18 (m, 5H), 7.17-6.99 (m, 5H), 4.93 (d,  $J$  = 14.0 Hz, 1H), 4.84 (d,  $J$  = 14.0 Hz, 1H), 3.93 (s, 3H).  $^{19}\text{F}$  NMR (376 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): -122.3 (s). **HRMS** for  $\text{C}_{23}\text{H}_{23}\text{FN}_3\text{O}_5^+$ : calcd.  $[\text{M}+\text{NH}_4]^+$ : 440.1616, found: 440.1614.

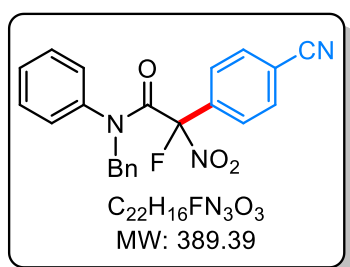
**Compound 3l: *N*-benzyl-2-fluoro-2-nitro-*N*-phenyl-2-(4-(trifluoromethyl)phenyl)acetamide**



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(4-(trifluoromethyl)phenyl)iodonium 4-methylbenzenesulfonate **2l** (124 mg, 0.22 mmol) in the presence of  $\text{K}_2\text{CO}_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3l** (46 mg, 53%) as white solid.  $R_f$ (Ethyl acetate/Hexane:

10/90) = 0.45. **Mp** 105-107 °C.  $^{13}\text{C}$  NMR (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 160.2 (d,  $J_{\text{C-F}}$  = 21.2 Hz, C), 138.1 (d,  $J_{\text{C-F}}$  = 2.4 Hz, C), 135.4 (C), 134.0 (d,  $J_{\text{C-F}}$  = 21.4 Hz, C), 133.2 (q,  $J_{\text{C-F}}$  = 32.8 Hz, C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.0 (CH), 129.0 (CH), 129.0 (CH), 128.8 (CH), 128.8 (CH), 128.3 (CH), 126.9 (d,  $J_{\text{C-F}}$  = 9.0 Hz, CH), 126.9 (d,  $J_{\text{C-F}}$  = 9.0 Hz, CH), 125.4 (d,  $J_{\text{C-F}}$  = 5.5 Hz, CH), 125.4 (d,  $J_{\text{C-F}}$  = 5.5 Hz, CH), 123.5 (q,  $J_{\text{C-F}}$  = 271.0 Hz, C), 115.0 (d,  $J_{\text{C-F}}$  = 257.1 Hz, C), 56.2 ( $\text{CH}_2$ ).  $^1\text{H}$  NMR (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 7.63-7.58 (m, 4H), 7.33-7.01 (m, 9H), 6.31 (s, 1H), 4.94 (s, 2H).  $^{19}\text{F}$  NMR (376 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): -63.1 (s), -122.1 (s). **HRMS** for  $\text{C}_{22}\text{H}_{20}\text{F}_4\text{N}_3\text{O}_3^+$ : calcd.  $[\text{M}+\text{NH}_4]^+$ : 450.1435, found: 450.1434.

**Compound 3m: *N*-benzyl-2-(4-cyanophenyl)-2-fluoro-2-nitro-*N*-phenylacetamide**

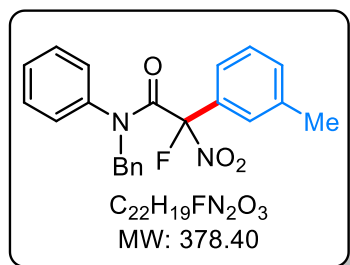


Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (4-cyanophenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2m** (114 mg, 0.22 mmol) in the presence of  $\text{K}_2\text{CO}_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3m** (67 mg, 86%) as white solid.  $R_f$ (Ethyl acetate/Hexane: 10/90) = 0.25. **Mp** 120-122 °C.  $^{13}\text{C}$

NMR (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 159.9 (d,  $J_{\text{C-F}}$  = 16.9 Hz, C), 138.0 (C), 135.3 (C), 134.7 (d,  $J_{\text{C-F}}$  = 18.0 Hz, C), 132.0 (CH), 132.0 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.2 (CH), 129.2 (CH), 129.2 (CH), 128.8 (CH), 128.8 (CH), 128.4 (CH), 127.2 (d,  $J_{\text{C-F}}$  = 7.2 Hz, CH), 127.2 (d,  $J_{\text{C-F}}$  = 7.2 Hz, CH), 117.6 (C), 115.1 (C), 114.7 (d,  $J_{\text{C-F}}$  = 207.7 Hz, C), 56.3 ( $\text{CH}_2$ ).  $^1\text{H}$  NMR (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 7.63 (d,  $J$  = 8.4 Hz, 1H), 7.56 (d,  $J$  = 8.4 Hz, 1H), 7.43-7.02 (m, 9H), 6.28 (s,

1H), 4.90 (s, 2H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.4 (s). HRMS for C<sub>22</sub>H<sub>20</sub>FN<sub>4</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 407.1514, found: 407.1511.

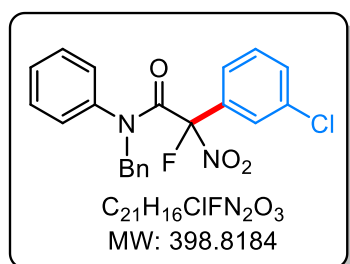
### Compound 3n: *N*-benzyl-2-fluoro-2-nitro-*N*-phenyl-2-(*m*-tolyl)acetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(*m*-tolyl)iodonium 4-methylbenzenesulfonate **2n** (112 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3n** (72 mg, 95%) as pale yellow viscous liquid. *R<sub>f</sub>*(Ethyl acetate/Hexane: 10/90) = 0.40. <sup>13</sup>C

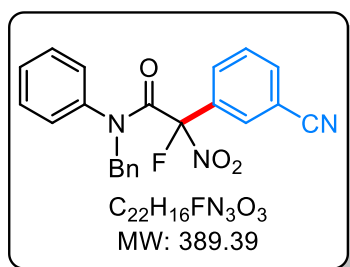
NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.9 (d, *J*<sub>C-F</sub> = 21.4 Hz, C), 138.5 (d, *J*<sub>C-F</sub> = 2.6 Hz, C), 138.3 (d, *J*<sub>C-F</sub> = 1.9 Hz, C), 135.7 (C), 131.9 (CH), 130.3 (d, *J*<sub>C-F</sub> = 21.4 Hz, C), 129.4 (CH), 129.4 (CH), 129.3 (CH), 129.3 (CH), 128.6 (CH), 128.6 (CH), 128.6 (CH), 128.6 (CH), 128.6 (CH), 128.3 (CH), 128.1 (CH), 126.8 (d, *J*<sub>C-F</sub> = 8.6 Hz, CH), 123.4 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 116.3 (d, *J*<sub>C-F</sub> = 255.3 Hz, C), 56.1 (CH<sub>2</sub>), 21.4 (CH<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.29-7.28 (m, 3H), 7.24-6.17 (m, 11H), 4.97 (d, *J* = 11.2 Hz, 1H), 4.84 (d, *J* = 10.8 Hz, 1H), 2.30 (s, 3H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.3 (s). HRMS for C<sub>22</sub>H<sub>23</sub>FN<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 396.1718, found: 396.1718.

### Compound 3o: *N*-benzyl-2-(3-chlorophenyl)-2-fluoro-2-nitro-*N*-phenylacetamide



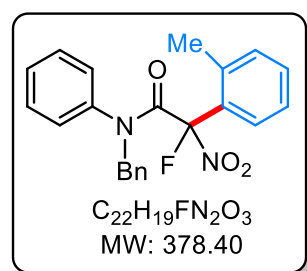
Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (3-chlorophenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2o** (116 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3o** (72 mg, 90%) as pale yellow viscous liquid. *R<sub>f</sub>*(Ethyl acetate/Hexane: 10/90) = 0.40. <sup>13</sup>C

NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.3 (d, *J*<sub>C-F</sub> = 21.2 Hz, C), 138.1 (d, *J*<sub>C-F</sub> = 2.5 Hz, C), 135.5 (C), 134.6 (d, *J*<sub>C-F</sub> = 2.2 Hz, C), 132.1 (d, *J*<sub>C-F</sub> = 21.9 Hz, C), 131.3 (CH), 129.7 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 128.9 (CH), 128.9 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.2 (CH), 126.5 (d, *J*<sub>C-F</sub> = 9.5 Hz, CH), 124.6 (d, *J*<sub>C-F</sub> = 8.4 Hz, CH), 115.0 (d, *J*<sub>C-F</sub> = 256.8 Hz, C), 56.1 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.40-7.38 (m, 1H), 7.34-7.33 (m, 2H), 7.29-7.21 (m, 5H), 7.17-7.00 (m, 5H), 6.23 (s, 1H), 4.91 (d, *J* = 11.2 Hz, 1H), 4.88 (d, *J* = 11.2 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.0 (s). HRMS for C<sub>21</sub>H<sub>20</sub>ClFN<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 416.1172, found: 416.1168.

**Compound 3p: *N*-benzyl-2-(3-cyanophenyl)-2-fluoro-2-nitro-*N*-phenylacetamide**

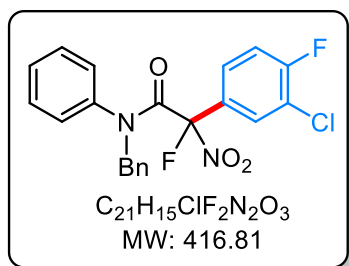
Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (3-cyanophenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2p** (114 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3p** (56 mg, 72%) as white solid.  $R_f$ (Ethyl acetate/Hexane: 10/90) = 0.25. **Mp** 98-100 °C.  $^{13}C$

**NMR** (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 159.9 (d,  $J_{C-F}$  = 16.9 Hz, C), 138.0 (C), 135.3 (C), 134.5 (CH), 132.0 (d,  $J_{C-F}$  = 18.0 Hz, C), 130.8 (d,  $J_{C-F}$  = 6.8 Hz, CH), 129.9 (d,  $J_{C-F}$  = 7.9 Hz, CH), 130.0 (CH), 130.0 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.2 (CH), 129.2 (CH), 128.8 (CH), 128.8 (CH), 128.8 (CH), 128.4 (CH), 117.5 (C), 114.3 (d,  $J_{C-F}$  = 205.0 Hz, C), 113.0 (d,  $J_{C-F}$  = 1.6 Hz, C), 56.3 (CH<sub>2</sub>).  $^1H$  **NMR** (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.71-7.46 (m, 4H), 7.26-6.99 (m, 9H), 6.26 (s, 1H), 4.87 (s, 2H).  $^{19}F$  **NMR** (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.6 (s). **HRMS** for  $C_{22}H_{20}FN_4O_3^+$ : calcd.  $[M+NH_4]^+$ : 407.1514, found: 407.1515.

**Compound 3q: *N*-benzyl-2-fluoro-2-nitro-*N*-phenyl-2-(*o*-tolyl)acetamide**

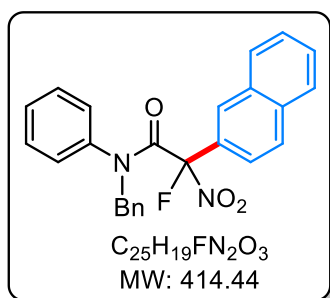
Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(*o*-tolyl)iodonium 4-methylbenzenesulfonate **2q** (112 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3q** (26 mg, 35%) as colorless liquid.  $R_f$ (Ethyl acetate/Hexane: 10/90) = 0.40. **Mp** 66-68 °C.  $^{13}C$  **NMR** (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.9 (d,  $J_{C-F}$

$J_{C-F}$  = 22.1 Hz, C), 138.7 (d,  $J_{C-F}$  = 2.7 Hz, C), 137.3 (C), 135.6 (C), 132.4 (CH), 131.1 (CH), 129.6 (CH), 129.6 (CH), 129.3 (d,  $J_{C-F}$  = 20.0 Hz, C), 129.1 (CH), 129.1 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.6 (CH), 128.6 (CH), 128.3 (CH), 127.9 (d,  $J_{C-F}$  = 9.4 Hz, CH), 125.8 (CH), 118.4 (d,  $J_{C-F}$  = 254.7 Hz, C), 56.2 (CH<sub>2</sub>), 20.5 (d,  $J_{C-F}$  = 6.1 Hz, CH<sub>3</sub>).  $^1H$  **NMR** (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.34-7.26 (m, 4H), 7.23-7.20 (m, 3H), 7.16 (t,  $J$  = 7.2 Hz, 1H), 7.11-7.04 (m, 4H), 6.76 (s, 2H), 5.02 (d,  $J$  = 14.0 Hz, 1H), 4.86 (d,  $J$  = 14.0 Hz, 1H), 2.16 (d,  $J$  = 4.0 Hz, 3H).  $^{19}F$  **NMR** (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -113.6 (s). **HRMS** for  $C_{22}H_{23}FN_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 396.1718, found: 396.1709.

**Compound 3r: *N*-benzyl-2-(3-chloro-4-fluorophenyl)-2-fluoro-2-nitro-*N*-phenylacetamide**

Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (3-chloro-4-fluorophenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2r** (120 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3r** (52 mg, 62%) as white solid.  $R_f$ (Ethyl acetate/Hexane: 10/90) = 0.25.

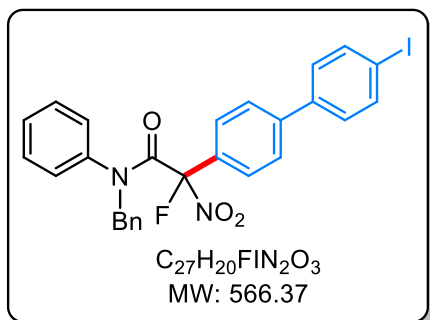
**Mp** 95-97 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.2 (d,  $J_{C-F}$  = 16.7 Hz, C), 159.8 (d,  $J_{C-F}$  = 202.7 Hz, C), 138.2 (C), 135.4 (C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.2 (CH), 129.2 (CH), 129.1 (CH), 129.1 (CH), 128.8 (CH), 128.8 (CH), 128.8 (CH), 128.3 (CH), 127.4 (dd,  $J_{C-F}$  = 18.2 Hz, 3.1 Hz, C), 126.8 (d,  $J_{C-F}$  = 6.7 Hz, CH), 121.8 (d,  $J_{C-F}$  = 14.8 Hz, C), 116.8 (d,  $J_{C-F}$  = 17.6 Hz, CH), 114.5 (d,  $J_{C-F}$  = 205.8 Hz, C), 56.2 ( $CH_2$ ).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.45-7.42 (m, 1H), 7.37-7.34 (m, 1H), 7.29-7.24 (m, 5H), 7.17-7.08 (m, 5H), 6.28 (s, 1H), 4.91 (d,  $J$  = 14.0 Hz, 1H), 4.87 (d,  $J$  = 14.0 Hz, 1H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -110.3 (s), -121.5 (s). **HRMS** for  $C_{21}H_{19}ClF_2N_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 434.1078, found: 434.1077.

**Compound 3t: *N*-benzyl-2-fluoro-2-(naphthalen-2-yl)-2-nitro-*N*-phenylacetamide**

Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with mesityl(naphthalen-2-yl)iodonium 4-methylbenzenesulfonate **2t** (120 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3t** (62 mg, 75%) as white solid.  $R_f$ (Ethyl acetate/Hexane: 10/90) = 0.35. **Mp** 129-131 °C.  $^{13}C$

**NMR** (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.8 (d,  $J_{C-F}$  = 17.0 Hz, C), 138.6 (C), 135.7 (C), 134.2 (C), 132.2 (C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 128.9 (CH), 128.9 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.7 (CH), 128.4 (CH), 128.2 (CH), 128.1 (CH), 127.8 (CH), 127.6 (C), 127.1 (CH), 126.8 (d,  $J_{C-F}$  = 7.7 Hz, CH), 122.7 (d,  $J_{C-F}$  = 6.1 Hz, CH), 116.4 (d,  $J_{C-F}$  = 204.7 Hz, C), 56.1 ( $CH_2$ ).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.84-7.72 (m, 4H), 7.57-7.50 (m, 3H), 7.26-6.08 (m, 10H), 4.97 (d,  $J$  = 14.0 Hz, 1H), 4.85 (d,  $J$  = 14.4 Hz, 1H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.0 (s). **HRMS** for  $C_{25}H_{23}FN_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 432.1718, found: 432.1715.

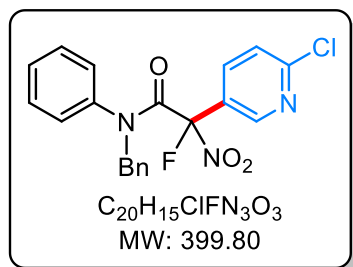
**Compound 3u:** *N*-benzyl-2-fluoro-2-(4'-iodo-[1,1'-biphenyl]-4-yl)-2-nitro-*N*-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (4'-iodo-[1,1'-biphenyl]-4-yl)(mesityl)iodonium 4-methylbenzenesulfonate **2u** (153 mg, 0.22 mmol) in the presence of  $\text{K}_2\text{CO}_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3u** (68 mg, 60%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.45. **Mp**

125-127 °C.  $^{13}\text{C}$  NMR (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 160.7 (d,  $J_{\text{C-F}}$  = 21.1 Hz, C), 142.9 (C), 139.3 (C), 138.5 (d,  $J_{\text{C-F}}$  = 2.7 Hz, C), 138.2 (CH), 138.2 (CH), 135.6 (C), 129.7 (d,  $J_{\text{C-F}}$  = 21.8 Hz, C), 129.4 (CH), 129.4 (CH), 129.4 (CH), 129.4 (CH), 129.1 (CH), 129.1 (CH), 128.9 (CH), 128.9 (CH), 128.8 (CH), 128.7 (CH), 128.7 (CH), 128.2 (CH), 127.0 (d,  $J_{\text{C-F}}$  = 8.6 Hz, CH), 127.0 (d,  $J_{\text{C-F}}$  = 8.6 Hz, CH), 126.8 (CH), 126.8 (CH), 116.0 (d,  $J_{\text{C-F}}$  = 255.5 Hz, C), 94.3 (C), 56.2 ( $\text{CH}_2$ ).  $^1\text{H}$  NMR (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 7.77 (d,  $J$  = 1.6 Hz, 2H), 7.48-7.44 (m, 4H), 7.28-7.23 (m, 5H), 7.21-6.32 (m, 7H), 4.92 (d,  $J$  = 11.2 Hz, 1H), 4.85 (d,  $J$  = 11.2 Hz, 1H).  $^{19}\text{F}$  NMR (376 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): -122.3 (s). **HRMS** for  $\text{C}_{27}\text{H}_{20}\text{FIN}_2\text{NaO}_3^+$ : calcd.  $[\text{M}+\text{Na}]^+$ : 589.0395, found: 589.0386.

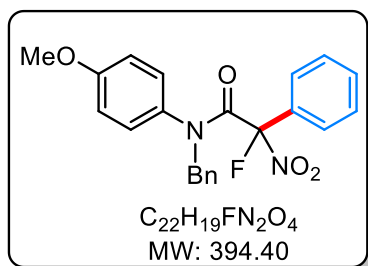
**Compound 3v:** *N*-benzyl-2-(6-chloropyridin-3-yl)-2-fluoro-2-nitro-*N*-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-phenylacetamide **1a** (58 mg, 0.20 mmol) with (6-chloropyridin-3-yl)(mesityl)iodonium 4-methylbenzenesulfonate **2v** (117 mg, 0.22 mmol) in the presence of  $\text{K}_2\text{CO}_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3v** (70 mg, 87%) as white solid.  $R_f$  (Ethyl acetate/Hexane:

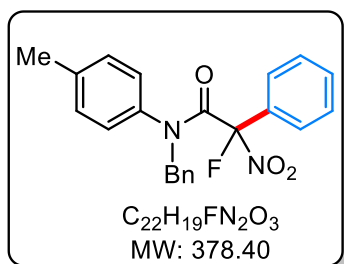
10/90) = 0.30. **Mp** 102-104 °C.  $^{13}\text{C}$  NMR (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 159.7 (d,  $J_{\text{C-F}}$  = 21.1 Hz, C), 154.4 (C), 147.5 (d,  $J_{\text{C-F}}$  = 10.2 Hz, CH), 137.9 (C), 136.9 (d,  $J_{\text{C-F}}$  = 8.0 Hz, CH), 135.2 (C), 129.4 (CH), 129.4 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 128.8 (CH), 128.8 (CH), 128.4 (CH), 125.8 (d,  $J_{\text{C-F}}$  = 22.1 Hz, C), 123.9 (CH), 113.9 (d,  $J_{\text{C-F}}$  = 258.1 Hz, C), 56.3 ( $\text{CH}_2$ ).  $^1\text{H}$  NMR (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 8.40 (s, 1H), 7.72 (d,  $J$  = 2.1 Hz, 1H), 7.29-7.12 (m, 10H), 6.38 (s, 1H), 4.89 (d,  $J$  = 14.0 Hz, 1H), 4.84 (d,  $J$  = 14.0 Hz, 1H).  $^{19}\text{F}$  NMR (376 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): -123.9 (s). **HRMS** for  $\text{C}_{20}\text{H}_{16}\text{ClFN}_3\text{O}_3^+$ : calcd.  $[\text{M}+\text{H}]^+$ : 400.0859, found: 400.0861.

### Compound 3w: *N*-benzyl-2-fluoro-*N*-(4-methoxyphenyl)-2-nitro-2-phenylacetamide

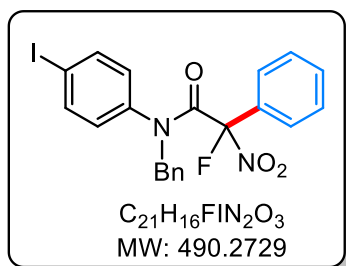


Following the general procedure, treatment of *N*-benzyl-2-fluoro-*N*-(4-methoxyphenyl)-2-nitroacetamide **1b** (64 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3w** (75 mg, 95%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.30. **Mp** 64–66 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 161.0 (d,  $J_{C-F}$  = 20.7 Hz, C), 159.4 (C), 135.8 (C), 131.1 (CH), 130.8 (C), 130.5 (CH), 130.5 (CH), 130.5 (d,  $J_{C-F}$  = 21.6 Hz, C), 129.3 (CH), 129.3 (CH), 128.6 (CH), 128.6 (CH), 128.3 (CH), 128.3 (CH), 128.1 (CH), 126.2 (d,  $J_{C-F}$  = 8.7 Hz, CH), 126.2 (d,  $J_{C-F}$  = 8.7 Hz, CH), 116.2 (d,  $J_{C-F}$  = 255.6 Hz, C), 113.8 (CH), 113.8 (CH), 56.1 ( $CH_2$ ), 55.4 ( $CH_3$ ).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.44–7.40 (m, 3H), 7.35–7.31 (m, 2H), 7.29–7.27 (m, 3H), 7.19–7.17 (m, 2H), 6.75–6.04 (m, 4H), 4.89 (d,  $J$  = 14.0 Hz, 1H), 4.84 (d,  $J$  = 13.6 Hz, 1H), 3.72 (s, 3H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.5 (s). **HRMS** for  $C_{22}H_{23}FN_3O_4^+$ : calcd.  $[M+NH_4]^+$ : 412.1667, found: 412.1663.

### Compound 3x: *N*-benzyl-2-fluoro-2-nitro-2-phenyl-*N*-(*p*-tolyl)acetamide

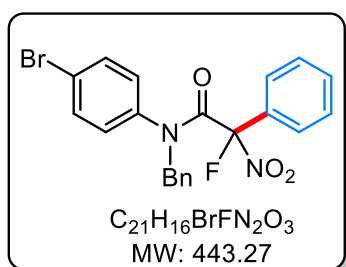


Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-(*p*-tolyl)acetamide **1c** (60 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3x** (61 mg, 80%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.35. **Mp** 58–60 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.9 (d,  $J_{C-F}$  = 21.1 Hz, C), 138.7 (C), 135.8 (C), 135.8 (C), 131.1 (CH), 130.5 (d,  $J_{C-F}$  = 21.8 Hz, C), 129.4 (CH), 129.4 (CH), 129.3 (CH), 129.3 (CH), 129.0 (CH), 129.0 (CH), 128.6 (CH), 128.6 (CH), 128.3 (CH), 128.3 (CH), 128.1 (CH), 126.3 (d,  $J_{C-F}$  = 8.8 Hz, CH), 126.3 (d,  $J_{C-F}$  = 8.8 Hz, CH), 116.2 (d,  $J_{C-F}$  = 255.8 Hz, C), 56.1 ( $CH_2$ ), 21.1 ( $CH_3$ ).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.37 (d,  $J$  = 6.0 Hz, 3H), 7.29–7.23 (m, 5H), 7.14–6.10 (m, 6H), 4.87 (d,  $J$  = 11.2 Hz, 1H), 4.81 (d,  $J$  = 11.2 Hz, 1H), 2.22 (s, 3H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.5 (s). **HRMS** for  $C_{22}H_{23}FN_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 396.1718, found: 396.1717.

**Compound 3y: *N*-benzyl-2-fluoro-*N*-(4-iodophenyl)-2-nitro-2-phenylacetamide**

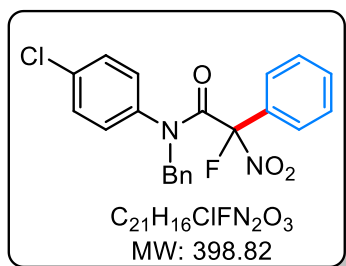
Following the general procedure, treatment of *N*-benzyl-2-fluoro-*N*-(4-iodophenyl)-2-nitroacetamide **1d** (83 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3y** (93 mg, 95%) as white solid. *R<sub>f</sub>* (Ethyl acetate/Hexane:

10/90) = 0.30. **Mp** 100-102 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.6 (d, *J*<sub>C-F</sub> = 21.4 Hz, C), 138.2 (C), 138.1 (CH), 138.1 (CH), 135.3 (C), 131.4 (CH), 131.2 (CH), 131.2 (CH), 130.2 (d, *J*<sub>C-F</sub> = 21.6 Hz, C), 129.3 (CH), 129.3 (CH), 128.8 (CH), 128.8 (CH), 128.6 (CH), 128.6 (CH), 128.4 (CH), 126.3 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 126.3 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 116.0 (d, *J*<sub>C-F</sub> = 254.9 Hz, C), 94.6 (C), 56.0 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.46-7.38 (m, 5H), 7.36-7.25 (m, 5H), 7.15 (s, 2H), 6.38 (s, 2H), 4.88 (d, *J* = 11.2 Hz, 1H), 4.83 (d, *J* = 11.2 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.7 (s). **HRMS** for C<sub>21</sub>H<sub>20</sub>FIN<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 508.0528, found: 508.0518.

**Compound 3z: *N*-benzyl-*N*-(4-bromophenyl)-2-fluoro-2-nitro-2-phenylacetamide**

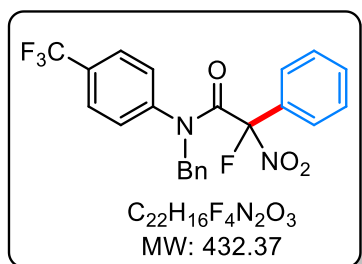
Following the general procedure, treatment of *N*-benzyl-*N*-(4-bromophenyl)-2-fluoro-2-nitroacetamide **1e** (53 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3z** (76 mg, 86%) as white solid. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.35. **Mp** 65-67 °C. <sup>13</sup>C

NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.6 (d, *J*<sub>C-F</sub> = 21.4 Hz, C), 137.4 (C), 135.3 (C), 132.0 (CH), 132.0 (CH), 131.4 (CH), 131.0 (CH), 131.0 (CH), 130.2 (d, *J*<sub>C-F</sub> = 21.6 Hz, C), 129.3 (CH), 129.3 (CH), 128.8 (CH), 128.8 (CH), 128.6 (CH), 128.6 (CH), 128.4 (CH), 126.2 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 126.2 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 122.9 (C), 116.0 (d, *J*<sub>C-F</sub> = 254.8 Hz, C), 55.9 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.47-7.43 (m, 1H), 7.41-7.39 (m, 2H), 7.37-7.33 (m, 2H), 7.29-7.28 (m, 3H), 7.17-7.11 (m, 6H), 4.90 (d, *J* = 14.0 Hz, 1H), 4.84 (d, *J* = 14.0 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.8 (s). **HRMS** for C<sub>21</sub>H<sub>17</sub>BrFN<sub>2</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+H]<sup>+</sup>: 443.0401, found: 443.0395.

**Compound 3aa: *N*-benzyl-*N*-(4-chlorophenyl)-2-fluoro-2-nitro-2-phenylacetamide**

Following the general procedure, treatment of *N*-benzyl-*N*-(4-chlorophenyl)-2-fluoro-2-nitroacetamide **1f** (53 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3aa** (56 mg, 70%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.30. **Mp** 72-74 °C.  $^{13}C$

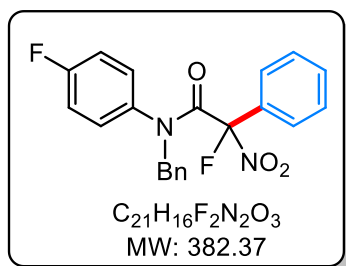
**NMR** (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.7 (d,  $J_{C-F}$  = 21.4 Hz, C), 136.9 (C), 135.3 (C), 134.8 (C), 131.4 (CH), 130.7 (CH), 130.7 (CH), 130.2 (d,  $J_{C-F}$  = 21.6 Hz, C), 129.3 (CH), 129.3 (CH), 129.0 (CH), 129.0 (CH), 128.8 (CH), 128.8 (CH), 128.5 (CH), 128.5 (CH), 128.4 (CH), 126.2 (d,  $J_{C-F}$  = 8.8 Hz, CH), 126.2 (d,  $J_{C-F}$  = 8.8 Hz, CH), 116.0 (d,  $J_{C-F}$  = 254.9 Hz, C), 56.0 ( $CH_2$ ).  $^1H$  **NMR** (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.46-7.39 (m, 3H), 7.36-7.32 (m, 2H), 7.29-6.78 (m, 8H), 6.20 (s, 1H), 4.90 (d,  $J$  = 14.0 Hz, 1H), 4.84 (d,  $J$  = 13.6 Hz, 1H).  $^{19}F$  **NMR** (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.8 (s). **HRMS** for  $C_{21}H_{17}ClFN_2O_3^+$ : calcd.  $[M+H]^+$ : 399.0906, found: 399.0908.

**Compound 3ab: *N*-benzyl-2-fluoro-2-nitro-2-phenyl-*N*-(4-(trifluoromethyl) phenyl) acetamide**

Following the general procedure, treatment of *N*-benzyl-2-fluoro-2-nitro-*N*-(4-(trifluoromethyl)phenyl)acetamide **1g** (71 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ab** (86 mg, 75%) as white solid.  $R_f$  (Ethyl

acetate/Hexane: 10/90) = 0.40. **Mp** 67-69 °C.  $^{13}C$  **NMR** (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.6 (d,  $J_{C-F}$  = 21.8 Hz, C), 141.7 (C), 135.1 (C), 131.5 (CH), 130.9 (d,  $J_{C-F}$  = 32.6 Hz, C), 130.2 (C), 130.0 (CH), 130.0 (CH), 129.3 (CH), 129.3 (CH), 128.9 (CH), 128.9 (CH), 128.7 (CH), 128.7 (CH), 128.5 (CH), 126.2 (d,  $J_{C-F}$  = 8.7 Hz, CH), 126.2 (d,  $J_{C-F}$  = 8.7 Hz, CH), 126.0 (CH), 126.0 (CH), 123.5 (d,  $J_{C-F}$  = 270.9 Hz, C), 115.9 (d,  $J_{C-F}$  = 254.1 Hz, C), 56.0 ( $CH_2$ ).  $^1H$  **NMR** (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.45 (t,  $J$  = 7.0 Hz, 2H), 7.38-7.33 (m, 5H), 7.31-7.28 (m, 3H), 7.16-7.14 (m, 2H), 6.73 (s, 2H), 4.91 (s, 2H).  $^{19}F$  **NMR** (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -62.8 (s), -122.8 (s). **HRMS** for  $C_{22}H_{20}F_4N_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 450.1435, found: 450.1447.

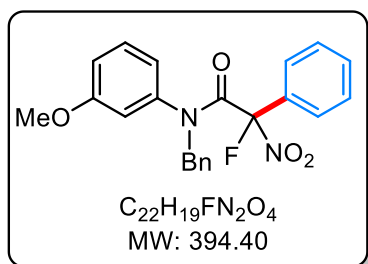
### Compound 3ac: *N*-benzyl-2-fluoro-*N*-(4-fluorophenyl)-2-nitro-2-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-*N*-(4-fluorophenyl)-2-nitroacetamide **1h** (61 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ac** (68 mg, 89%) as yellow viscous liquid. *R*<sub>f</sub> (Ethyl acetate/Hexane:

10/90) = 0.40. **Mp** 102-104 °C. **<sup>13</sup>C NMR** (100 MHz, δ ppm/CDCl<sub>3</sub>): 162.6 (d, *J*<sub>C-F</sub> = 248.4 Hz, C), 160.8 (d, *J*<sub>C-F</sub> = 21.4 Hz, C), 135.4 (C), 134.3 (C), 131.3 (CH), 131.3 (CH), 131.3 (CH), 130.3 (d, *J*<sub>C-F</sub> = 21.5 Hz, C), 129.4 (CH), 129.4 (CH), 128.8 (CH), 128.8 (CH), 128.5 (CH), 128.5 (CH), 128.3 (CH), 126.2 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 126.2 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 115.7 (CH), 115.7 (CH), 116.1 (d, *J*<sub>C-F</sub> = 254.6 Hz, C), 56.1 (CH<sub>2</sub>). **<sup>1</sup>H NMR** (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.46-7.38 (m, 3H), 7.36-7.32 (m, 2H), 7.29-7.27 (m, 3H), 7.17-7.14 (m, 2H), 6.73-6.08 (m, 4H), 4.91 (d, *J* = 14.0 Hz, 1H), 4.84 (d, *J* = 14.0 Hz, 1H). **<sup>19</sup>F NMR** (376 MHz, δ ppm/CDCl<sub>3</sub>): -111.6 (s), -122.8 (s). **HRMS** for C<sub>21</sub>H<sub>16</sub>F<sub>2</sub>N<sub>2</sub>NaO<sub>3</sub><sup>+</sup>: calcd. [M+Na]<sup>+</sup>: 405.1021, found: 405.1016.

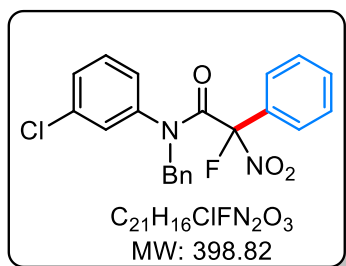
### Compound 3ad: *N*-benzyl-2-fluoro-*N*-(3-methoxyphenyl)-2-nitro-2-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-fluoro-*N*-(3-methoxyphenyl)-2-nitroacetamide **1i** (64 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ad** (53 mg, 67%) as yellow viscous liquid. *R*<sub>f</sub> (Ethyl

acetate/Hexane: 10/90) = 0.25. **<sup>13</sup>C NMR** (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.7 (d, *J*<sub>C-F</sub> = 21.5 Hz, C), 159.6 (C), 139.5 (C), 135.8 (C), 131.2 (CH), 130.5 (d, *J*<sub>C-F</sub> = 21.5 Hz, C), 129.4 (CH), 129.4 (CH), 129.4 (CH), 128.7 (CH), 128.7 (CH), 128.4 (CH), 128.4 (CH), 128.2 (CH), 126.4 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 126.4 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 121.5 (CH), 116.2 (d, *J*<sub>C-F</sub> = 256.0 Hz, C), 115.2 (CH), 114.3 (CH), 56.0 (CH<sub>2</sub>), 55.2 (CH<sub>3</sub>). **<sup>1</sup>H NMR** (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.45-7.41 (m, 3H), 7.34 (d, *J* = 7.6 Hz, 2H), 7.29-7.26 (m, 4H), 7.20-7.18 (m, 2H), 6.99 (s, 1H), 6.74 (dd, *J* = 2.1 Hz, 0.6 Hz, 2H), 7.93 (d, *J* = 14.0 Hz, 1H), 4.85 (d, *J* = 14.0 Hz, 1H), 3.45 (s, 3H). **<sup>19</sup>F NMR** (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.8 (s). **HRMS** for C<sub>22</sub>H<sub>23</sub>FN<sub>3</sub>O<sub>4</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 412.1667, found: 412.1661.

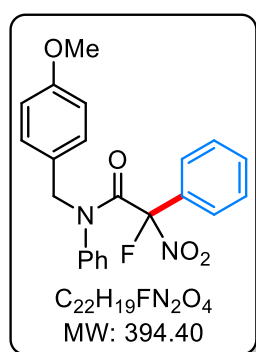
### Compound 3ae: *N*-benzyl-*N*-(3-chlorophenyl)-2-fluoro-2-nitro-2-phenylacetamide



Following the general procedure, treatment of *N*-benzyl-*N*-(3-chlorophenyl)-2-fluoro-2-nitroacetamide **1j** (65 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ae** (48 mg, 60%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.35. **Mp** 89-91 °C.  $^{13}C$

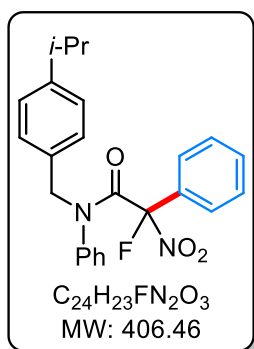
**NMR** (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.7 (d,  $J_{C-F}$  = 18.6 Hz, C), 139.6 (C), 135.2 (C), 134.2 (C), 131.5 (CH), 130.2 (d,  $J_{C-F}$  = 27.4 Hz, C), 129.7 (CH), 129.7 (CH), 129.1 (CH), 129.1 (CH), 128.8 (CH), 128.8 (CH), 128.8 (CH), 128.6 (CH), 128.6 (CH), 128.4 (CH), 127.8 (CH), 126.2 (d,  $J_{C-F}$  = 8.7 Hz, CH), 126.2 (d,  $J_{C-F}$  = 8.7 Hz, CH), 116.1 (d,  $J_{C-F}$  = 255.0 Hz, C), 56.0 ( $CH_2$ ).  $^1H$  **NMR** (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.47-7.33 (m, 5H), 7.29-7.26 (m, 3H), 7.19-6.17 (m, 6H), 4.88 (s, 2H).  $^{19}F$  **NMR** (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -123.3 (s). **HRMS** for  $C_{21}H_{20}ClFN_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 416.1172, found: 416.1162.

### Compound 3af: 2-fluoro-*N*-(4-methoxybenzyl)-2-nitro-*N*,2-diphenylacetamide



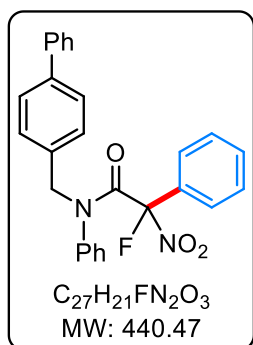
Following the general procedure, treatment of 2-fluoro-*N*-(4-methoxybenzyl)-2-nitro-*N*-phenylacetamide **1k** (64 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3af** (64 mg, 81%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.25. **Mp** 96-98 °C.  $^{13}C$  **NMR** (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.6 (d,  $J_{C-F}$  = 16.9 Hz, C), 159.5 (C), 138.4 (C), 131.1 (CH), 130.8 (CH), 130.8 (CH), 130.4 (d,  $J_{C-F}$  = 17.2 Hz, C), 129.4 (CH), 129.4 (CH), 128.7 (CH), 128.6 (CH), 128.6 (CH), 128.4 (CH), 128.4 (CH), 127.8 (C), 126.2 (d,  $J_{C-F}$  = 6.9 Hz, CH), 126.2 (d,  $J_{C-F}$  = 6.9 Hz, CH), 116.2 (d,  $J_{C-F}$  = 204.2 Hz, C), 114.0 (CH), 114.0 (CH), 55.5 ( $CH_2$ ), 55.3 ( $CH_3$ ).  $^1H$  **NMR** (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.43-7.36 (m, 3H), 7.33-7.29 (m, 2H), 7.19 (t,  $J$  = 7.2 Hz, 2H), 7.09-6.98 (m, 4H), 6.79 (d,  $J$  = 8.4 Hz, 2H), 6.18 (s, 1H), 4.88 (d,  $J$  = 13.6 Hz, 1H), 4.79 (d,  $J$  = 14.0 Hz, 1H), 3.78 (s, 3H).  $^{19}F$  **NMR** (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.6 (s). **HRMS** for  $C_{22}H_{23}FN_3O_4^+$ : calcd.  $[M+NH_4]^+$ : 412.1667, found: 412.1664.

### Compound 3ag: 2-fluoro-*N*-(4-isopropylbenzyl)-2-nitro-*N*,2-diphenylacetamide



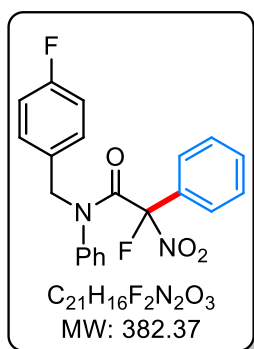
Following the general procedure, treatment of 2-fluoro-*N*-(4-*i*-propylbenzyl)-2-nitro-*N*-phenylacetamide **1l** (66 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ag** (72 mg, 89%) as yellow liquid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.40.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.7 (d,  $J_{C-F}$  = 21.1 Hz, C), 148.8 (C), 138.6 (C), 132.9 (C), 131.1 (CH), 130.5 (d,  $J_{C-F}$  = 21.4 Hz, C), 129.4 (CH), 129.4 (CH), 129.3 (CH), 129.3 (CH), 128.8 (CH), 128.6 (CH), 128.6 (CH), 128.4 (CH), 128.4 (CH), 126.7 (CH), 126.7 (CH), 126.3 (d,  $J_{C-F}$  = 8.7 Hz, CH), 126.3 (d,  $J_{C-F}$  = 8.7 Hz, CH), 116.2 (d,  $J_{C-F}$  = 255.3 Hz, C), 55.9 (CH<sub>2</sub>), 33.9 (CH), 24.0 (CH<sub>3</sub>), 24.0 (CH<sub>3</sub>).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.45-7.39 (m, 4H), 7.32 (t,  $J$  = 7.8 Hz, 2H), 7.20 (t,  $J$  = 7.6 Hz, 2H), 7.16-6.11 (m, 6H), 4.89 (d,  $J$  = 14.0 Hz, 1H), 4.85 (d,  $J$  = 14.0 Hz, 1H), 2.93-2.86 (m, 1H), 1.24 (d,  $J$  = 6.8 Hz, 6H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.5 (s). HRMS for  $C_{24}H_{27}FN_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 424.2031, found: 424.2025.

### Compound 3ah: *N*-([1,1'-biphenyl]-4-ylmethyl)-2-fluoro-2-nitro-*N*,2-diphenylacetamide



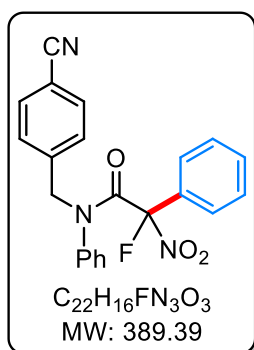
Following the general procedure, treatment of *N*-([1,1'-biphenyl]-4-ylmethyl)-2-fluoro-2-nitro-*N*-phenylacetamide **1m** (73 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ah** (79 mg, 90%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.40.  $Mp$  92-94 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.8 (d,  $J_{C-F}$  = 21.1 Hz, C), 141.0 (C), 140.6 (C), 138.6 (C), 134.7 (C), 131.2 (CH), 130.4 (d,  $J_{C-F}$  = 21.3 Hz, C), 129.8 (CH), 129.8 (CH), 129.4 (CH), 129.4 (CH), 128.9 (CH), 128.9 (CH), 128.9 (CH), 128.9 (CH), 128.7 (CH), 128.4 (CH), 128.4 (CH), 127.6 (CH), 127.3 (CH), 127.3 (CH), 127.1 (CH), 127.1 (CH), 126.3 (d,  $J_{C-F}$  = 8.8 Hz, CH), 126.3 (d,  $J_{C-F}$  = 8.8 Hz, CH), 116.1 (d,  $J_{C-F}$  = 255.6 Hz, C), 55.9 (CH<sub>2</sub>).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.63 (d,  $J$  = 7.6 Hz, 2H), 7.57 (d,  $J$  = 7.6 Hz, 2H), 7.51-7.45 (m, 5H), 7.42-7.36 (m, 3H), 7.30-7.13 (m, 6H), 6.39 (s, 1H), 5.02 (d,  $J$  = 14.0 Hz, 1H), 4.96 (d,  $J$  = 14.0 Hz, 1H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.6 (s). HRMS for  $C_{27}H_{25}FN_3O_3^+$ : calcd.  $[M+NH_4]^+$ : 458.1874, found: 458.1875.

### Compound 3ai: 2-fluoro-*N*-(4-fluorobenzyl)-2-nitro-*N*,2-diphenylacetamide



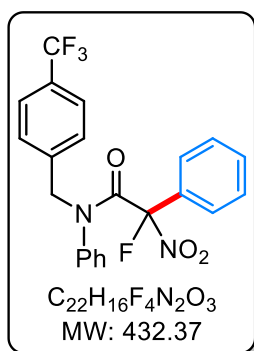
Following the general procedure, treatment of 2-fluoro-*N*-(4-fluorobenzyl)-2-nitro-*N*-phenylacetamide **1n** (61 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ai** (57 mg, 75%) as white solid. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.45. *Mp* 102-104 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 162.7 (d, *J*<sub>C-F</sub> = 245.5 Hz, C), 160.8 (d, *J*<sub>C-F</sub> = 21.4 Hz, C), 138.3 (C), 131.6 (d, *J*<sub>C-F</sub> = 3.3 Hz, C), 131.3 (CH), 131.3 (CH), 131.2 (CH), 130.3 (d, *J*<sub>C-F</sub> = 21.8 Hz, C), 129.4 (CH), 129.4 (CH), 128.9 (CH), 128.8 (CH), 128.8 (CH), 128.5 (CH), 128.5 (CH), 126.3 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 126.3 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 116.1 (d, *J*<sub>C-F</sub> = 255.0 Hz, C), 115.7 (CH), 115.5 (CH), 55.4 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.45-7.40 (m, 1H), 7.38-7.36 (m, 2H), 7.34-7.19 (m, 4H), 7.16-7.13 (m, 3H), 6.99-6.93 (m, 3H), 6.18 (s, 1H), 4.89 (d, *J* = 14.0 Hz, 1H), 4.82 (d, *J* = 14.0 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -113.7 (s), -122.8 (s). HRMS for C<sub>21</sub>H<sub>20</sub>F<sub>2</sub>N<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 400.1467, found: 400.1466.

### Compound 3aj: *N*-(4-cyanobenzyl)-2-fluoro-2-nitro-*N*,2-diphenylacetamide



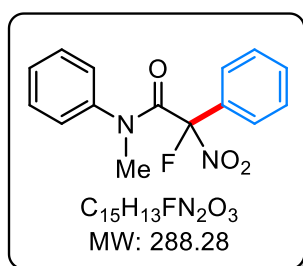
Following the general procedure, treatment of *N*-(4-cyanobenzyl)-2-fluoro-2-nitro-*N*-phenylacetamide **1o** (63 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3aj** (65 mg, 84%) as colorless liquid. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.20. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 161.1 (d, *J*<sub>C-F</sub> = 21.5 Hz, C), 140.9 (C), 138.3 (C), 132.5 (CH), 132.5 (CH), 131.4 (CH), 130.0 (d, *J*<sub>C-F</sub> = 21.6 Hz, C), 129.8 (CH), 129.8 (CH), 129.2 (CH), 129.1 (CH), 129.1 (CH), 129.0 (CH), 129.0 (CH), 128.5 (CH), 128.5 (CH), 126.2 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 126.2 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 118.6 (C), 115.8 (d, *J*<sub>C-F</sub> = 255.2 Hz, C), 112.2 (C), 55.8 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.77 (d, *J* = 8.0 Hz, 2H), 7.46-7.42 (m, 1H), 7.39-7.35 (m, 3H), 7.33-7.30 (m, 3H), 7.23 (t, *J* = 8.4 Hz, 1H), 7.11-6.34 (m, 4H), 4.95 (d, *J* = 14.4 Hz, 1H), 4.90 (d, *J* = 14.4 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.9 (s). HRMS for C<sub>22</sub>H<sub>20</sub>FN<sub>4</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 407.1514, found: 407.1508.

### Compound 3ak: 2-fluoro-2-nitro-*N*,2-diphenyl-*N*-(4-(trifluoromethyl)benzyl)acetamide



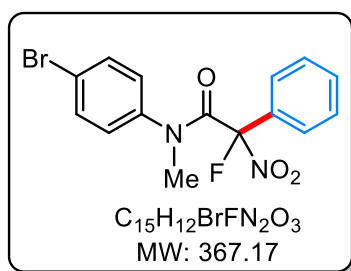
Following the general procedure, treatment of 2-fluoro-2-nitro-*N*-phenyl-*N*-(4-(trifluoromethyl)benzyl)acetamide **1p** (71 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ak** (68 mg, 79%) as white solid. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.40. *Mp* 67-69 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 161.1 (d, *J*<sub>C-F</sub> = 21.6 Hz, C), 139.6 (C), 138.4 (C), 131.3 (CH), 130.4 (d, *J*<sub>C-F</sub> = 27.4 Hz, C), 130.2 (d, *J*<sub>C-F</sub> = 16.6 Hz, C), 129.5 (CH), 129.5 (CH), 129.1 (CH), 129.1 (CH), 129.1 (CH), 129.0 (CH), 129.0 (CH), 128.5 (CH), 128.5 (CH), 126.2 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 126.2 (d, *J*<sub>C-F</sub> = 8.8 Hz, CH), 125.7 (d, *J*<sub>C-F</sub> = 3.8 Hz, CH), 125.7 (d, *J*<sub>C-F</sub> = 3.8 Hz, CH), 124.1 (q, *J*<sub>C-F</sub> = 270.6 Hz, C), 116.0 (d, *J*<sub>C-F</sub> = 255.2 Hz, C), 55.7 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.55 (d, *J* = 7.6 Hz, 2H), 7.46-7.38 (m, 3H), 7.33 (t, *J* = 7.8 Hz, 4H), 7.23 (t, *J* = 6.8 Hz, 1H), 7.18-7.31 (m, 4H), 4.95 (s, 2H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -62.6 (s), -122.8 (s). HRMS for C<sub>22</sub>H<sub>20</sub>F<sub>4</sub>N<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+Na]<sup>+</sup>: 455.0989, found: 450.0987.

### Compound 3al: 2-fluoro-*N*-methyl-2-nitro-*N*,2-diphenylacetamide



Following the general procedure, treatment of 2-fluoro-*N*-methyl-2-nitro-*N*-phenylacetamide **1q** (42 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3al** (50 mg, 87%) as brown oil. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.35. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.8 (d, *J*<sub>C-F</sub> = 21.5 Hz, C), 140.5 (C), 131.2 (CH), 130.5 (d, *J*<sub>C-F</sub> = 21.6 Hz, C), 129.2 (CH), 129.2 (CH), 128.7 (CH), 128.5 (CH), 128.5 (CH), 128.2 (CH), 128.2 (CH), 126.3 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 126.3 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 116.1 (d, *J*<sub>C-F</sub> = 255.1 Hz, C), 40.4 (CH<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.73-7.39 (m, 4H), 7.33 (t, *J* = 7.2 Hz, 2H), 7.26-7.17 (m, 2H), 6.85 (s, 2H), 3.34 (s, 3H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.4 (s). HRMS for C<sub>15</sub>H<sub>13</sub>FN<sub>2</sub>NaO<sub>3</sub><sup>+</sup>: calcd. [M+Na]<sup>+</sup>: 311.0802, found: 311.0794.

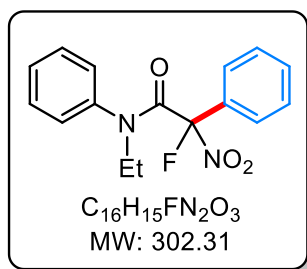
### Compound 3am: 2-(4-bromophenyl)-2-fluoro-*N*-methyl-2-nitro-*N*-phenylacetamide



Following the general procedure, treatment of *N*-(4-bromophenyl)-2-fluoro-*N*-methyl-2-nitroacetamide **1r** (58 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3am** (59 mg, 81%) as white solid. *R<sub>f</sub>* (Ethyl

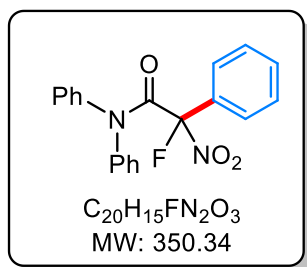
acetate/Hexane: 10/90) = 0.35. **Mp** 76-78 °C. **<sup>13</sup>C NMR** (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 160.6 (d,  $J_{\text{C-F}}$  = 21.5 Hz, C), 139.5 (C), 132.4 (CH), 132.4 (CH), 131.4 (CH), 130.2 (d,  $J_{\text{C-F}}$  = 20.9 Hz, C), 129.9 (CH), 129.9 (CH), 128.6 (CH), 128.6 (CH), 126.3 (d,  $J_{\text{C-F}}$  = 8.7 Hz, CH), 126.3 (d,  $J_{\text{C-F}}$  = 8.7 Hz, CH), 122.7 (C), 116.0 (d,  $J_{\text{C-F}}$  = 254.1 Hz, C), 40.3 ( $\text{CH}_3$ ). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 7.55-7.45 (m, 2H), 7.41-7.29 (m, 5H), 6.72 (s, 2H), 3.31 (s, 3H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): -122.6 (s). **HRMS** for  $\text{C}_{15}\text{H}_{13}\text{BrFN}_2\text{O}_3^+$ : calcd.  $[\text{M}+\text{H}]^+$ : 367.0088, found: 367.0077.

#### Compound 3an: *N*-ethyl-2-fluoro-2-nitro-*N*,2-diphenylacetamide



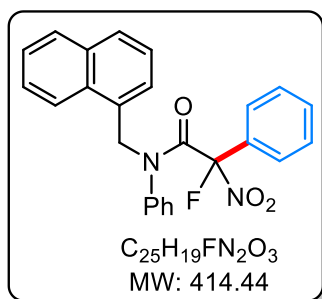
Following the general procedure, treatment of *N*-ethyl-2-fluoro-2-nitro-*N*-phenylacetamide **1s** (45 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $\text{K}_2\text{CO}_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3an** (56 mg, 92%) as white solid. **R<sub>f</sub>** (Ethyl acetate/Hexane: 10/90) = 0.35. **Mp** 80-82 °C. **<sup>13</sup>C NMR** (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 160.2 (d,  $J_{\text{C-F}}$  = 21.2 Hz, C), 138.6 (d,  $J_{\text{C-F}}$  = 2.5 Hz, C), 131.1 (CH), 130.5 (d,  $J_{\text{C-F}}$  = 21.6 Hz, C), 129.2 (CH), 129.2 (CH), 128.9 (CH), 128.9 (CH), 128.6 (CH), 128.4 (CH), 128.4 (CH), 126.2 (d,  $J_{\text{C-F}}$  = 8.7 Hz, CH), 126.2 (d,  $J_{\text{C-F}}$  = 8.7 Hz, CH), 116.2 (d,  $J_{\text{C-F}}$  = 255.1 Hz, C), 47.5 ( $\text{CH}_2$ ), 12.3 ( $\text{CH}_3$ ). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 7.73-7.31 (m, 6H), 7.24-6.49 (m, 4H), 3.82-3.73 (m, 2H), 1.16 (t,  $J$  = 7.2 Hz, 3H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): -122.6 (s). **HRMS** for  $\text{C}_{16}\text{H}_{15}\text{FN}_2\text{NaO}_3^+$ : calcd.  $[\text{M}+\text{Na}]^+$ : 325.0959, found: 325.0959.

#### Compound 3ao: *N*-ethyl-2-fluoro-2-nitro-*N*,2-diphenylacetamide



Following the general procedure, treatment of 2-fluoro-2-nitro-*N,N*-diphenylacetamide **1t** (55 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $\text{K}_2\text{CO}_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ao** (53 mg, 75%) as white solid. **R<sub>f</sub>** (Ethyl acetate/Hexane: 10/90) = 0.25. **Mp** 140-142 °C. **<sup>13</sup>C NMR** (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 160.1 (d,  $J_{\text{C-F}}$  = 21.9 Hz, C), 131.4 (C), 131.4 (C), 130.4 (d,  $J_{\text{C-F}}$  = 21.3 Hz, C), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 128.6 (CH), 128.6 (CH), 128.6 (CH), 128.6 (CH), 126.4 (d,  $J_{\text{C-F}}$  = 8.8 Hz, CH), 126.4 (d,  $J_{\text{C-F}}$  = 8.8 Hz, CH), 126.4 (d,  $J_{\text{C-F}}$  = 8.8 Hz, CH), 126.4 (d,  $J_{\text{C-F}}$  = 8.8 Hz, CH), 116.2 (d,  $J_{\text{C-F}}$  = 255.6 Hz, C). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 7.52-7.46 (m, 3H), 7.39 (t,  $J$  = 7.6 Hz, 2H), 7.33-7.01 (m, 10H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): -122.7 (s). **HRMS** for  $\text{C}_{20}\text{H}_{19}\text{FN}_3\text{O}_3^+$ : calcd.  $[\text{M}+\text{NH}_4]^+$ : 368.1405, found: 368.1398.

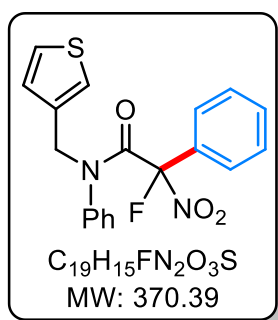
### Compound 3ap: 2-fluoro-*N*-(naphthalen-1-ylmethyl)-2-nitro-*N*,2-diphenylacetamide



Following the general procedure, treatment of 2-fluoro-*N*-(naphthalen-1-ylmethyl)-2-nitro-*N*-phenylacetamide **1u** (68 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ap** (47 mg, 57%) as white solid. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.30.

**Mp** 104-106 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.5 (d, *J*<sub>C-F</sub> = 21.1 Hz, C), 137.5 (C), 133.8 (C), 131.8 (C), 131.2 (CH), 130.8 (C), 130.4 (d, *J*<sub>C-F</sub> = 21.7 Hz, C), 129.5 (CH), 129.5 (CH), 129.3 (CH), 129.2 (CH), 128.9 (CH), 128.6 (CH), 128.5 (CH), 128.4 (CH), 128.4 (CH), 126.9 (CH), 126.3 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 126.3 (d, *J*<sub>C-F</sub> = 8.7 Hz, CH), 126.2 (CH), 125.0 (CH), 123.9 (CH), 116.3 (d, *J*<sub>C-F</sub> = 256.1 Hz, C), 53.2 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 8.07 (d, *J* = 8.4 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.58-7.50 (m, 2H), 7.39-7.36 (m, 3H), 7.28-7.24 (m, 2H), 7.18 (t, *J* = 7.6 Hz, 1H), 7.08 (t, *J* = 7.2 Hz, 1H), 6.94-5.92 (m, 4H), 5.92 (s, 1H), 5.56 (d, *J* = 14.0 Hz, 1H), 5.28 (d, *J* = 14.0 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.8 (s). HRMS for C<sub>25</sub>H<sub>23</sub>FN<sub>3</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 432.1718, found: 432.1717.

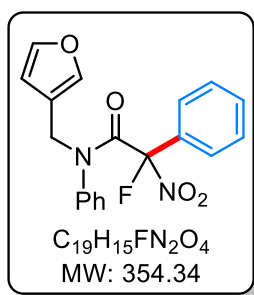
### Compound 3aq: 2-fluoro-2-nitro-*N*,2-diphenyl-*N*-(thiophen-3-ylmethyl)acetamide



Following the general procedure, treatment of 2-fluoro-2-nitro-*N*-phenyl-*N*-(thiophen-3-ylmethyl)acetamide **1v** (59 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of K<sub>2</sub>CO<sub>3</sub> (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3aq** (47 mg, 63%) as white solid. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.50. **Mp** 113-115 °C. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 160.6 (d, *J*<sub>C-F</sub> = 17.2 Hz, C), 138.5 (C), 136.0 (C), 131.2 (CH), 130.4 (d, *J*<sub>C-F</sub> = 17.2 Hz, C), 129.2 (CH), 129.2 (CH), 128.8 (CH), 128.8 (CH), 128.7 (CH), 128.4 (CH), 128.4 (CH), 128.3 (CH), 126.3 (CH), 126.3 (CH), 126.2 (CH), 125.0 (CH), 116.1 (d, *J*<sub>C-F</sub> = 204.1 Hz, C), 50.9 (CH<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.40 (t, *J* = 7.2 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.31-7.24 (m, 3H), 7.20 (t, *J* = 7.2 Hz, 1H), 7.01-6.61 (m, 5H), 6.18 (s, 1H), 4.91 (d, *J* = 14.0 Hz, 1H), 4.80 (d, *J* = 14.0 Hz, 1H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -122.7 (s).

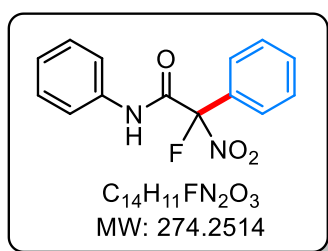
HRMS for C<sub>19</sub>H<sub>19</sub>FN<sub>3</sub>O<sub>3</sub>S<sup>+</sup>: calcd. [M+NH<sub>4</sub>]<sup>+</sup>: 388.1126, found: 388.1122.

### Compound 3ar: 2-fluoro-*N*-(furan-3-ylmethyl)-2-nitro-*N*,2-diphenylacetamide



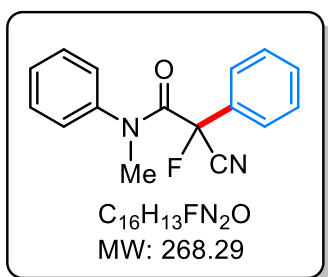
Following the general procedure, treatment of 2-fluoro-*N*-(furan-3-ylmethyl)-2-nitro-*N*-phenylacetamide **1w** (56 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3ar** (48 mg, 68%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.30. **Mp** 81-83 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.6 (d,  $J_{C-F}$  = 17.0 Hz, C), 143.5 (CH), 141.9 (CH), 138.5 (C), 131.2 (CH), 130.4 (d,  $J_{C-F}$  = 17.1 Hz, C), 129.2 (CH), 129.2 (CH), 128.9 (CH), 128.9 (CH), 128.8 (CH), 128.4 (CH), 128.4 (CH), 126.3 (d,  $J_{C-F}$  = 7.0 Hz, CH), 126.3 (d,  $J_{C-F}$  = 7.0 Hz, CH), 119.6 (C), 116.1 (d,  $J_{C-F}$  = 204.3 Hz, C), 111.1 (CH), 47.0 (CH<sub>2</sub>).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.45-7.36 (m, 5H), 7.32 (t,  $J$  = 7.6 Hz, 2H), 7.22 (t,  $J$  = 7.2 Hz, 2H), 7.08 (s, 2H), 6.36 (s, 2H), 4.77 (d,  $J$  = 14.4 Hz, 1H), 4.64 (d,  $J$  = 14.4 Hz, 1H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -122.8 (s). **HRMS** for  $C_{19}H_{15}FN_2O_4^+$ : calcd.  $[M+NH_4]^+$ : 372.1354, found: 372.1346.

### Compound 3as: 2-fluoro-2-nitro-*N*,2-diphenylacetamide



Following the general procedure, treatment of 2-fluoro-2-nitro-*N*-phenylacetamide **1x** (40 mg, 0.20 mmol) with **2a** (109 mg, 0.22 mmol) in the presence of  $K_2CO_3$  (33 mg, 0.24 mmol) in toluene (2 mL) at 50 °C for 2 h followed by column chromatography afforded the product **3as** (13 mg, 23%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.40. **Mp** 111-113 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 158.2 (d,  $J_{C-F}$  = 22.6 Hz, C), 135.7 (C), 132.1 (CH), 129.5 (CH), 129.5 (CH), 129.3 (C), 129.1 (CH), 129.1 (CH), 126.9 (d,  $J_{C-F}$  = 8.4 Hz, CH), 126.9 (d,  $J_{C-F}$  = 8.4 Hz, CH), 126.3 (CH), 120.5 (CH), 120.5 (CH), 113.8 (d,  $J_{C-F}$  = 248.4 Hz, C).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 8.06 (s, 1H), 7.84 (d,  $J$  = 7.2 Hz, 2H), 7.59-7.50 (m, 5H), 7.38 (d,  $J$  = 7.6 Hz, 2H), 7.22 (d,  $J$  = 7.6 Hz, 1H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -124.3 (s). **HRMS** for  $C_{14}H_{11}FN_2O_3^+$ : calcd.  $[M+NH_4]^+$ : 292.1092, found: 292.1089.

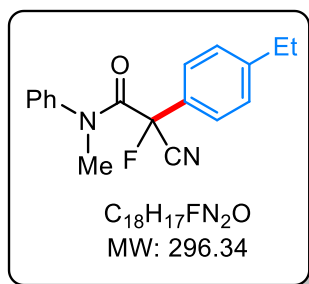
### Compound 5a: 2-cyano-2-fluoro-*N*-methyl-*N*,2-diphenylacetamide



Following the general procedure, treatment of 2-cyano-2-fluoro-*N*-methyl-*N*-phenylacetamide **4a** (38 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5a** (38 mg, 70%) as yellow oil.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.25.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 162.1 (d,  $J_{C-F}$  = 23.7 Hz, C), 140.4 (C), 133.0 (d,  $J_{C-F}$  = 23.2 Hz, C), 130.6 (CH), 129.3 (CH), 129.3 (CH), 129.0 (CH), 129.0 (CH), 128.8 (CH), 128.5 (CH), 128.5 (CH), 125.2 (d,  $J_{C-F}$  = 5.5 Hz, CH),

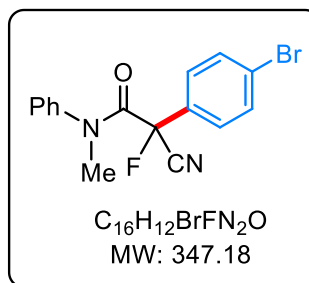
125.2 (d,  $J_{C-F}$  = 5.5 Hz, CH), 114.9 (d,  $J_{C-F}$  = 34.6 Hz, C), 89.9 (d,  $J_{C-F}$  = 197.8 Hz, C), 40.7 (CH<sub>3</sub>). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 7.41 (t,  $J$  = 7.2 Hz, 1H), 7.33 (t,  $J$  = 7.2 Hz, 2H), 7.30-7.25 (m, 3H), 7.20 (t,  $J$  = 7.6 Hz, 2H), 6.81 (s, 2H), 3.30 (s, 3H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): -137.5 (s). **HRMS** for C<sub>16</sub>H<sub>14</sub>FN<sub>2</sub>O<sup>+</sup>: calcd. [M+H]<sup>+</sup>: 269.1085, found: 269.1083.

#### Compound 5b: 2-cyano-2-(4-ethylphenyl)-2-fluoro-*N*-methyl-*N*-phenylacetamide



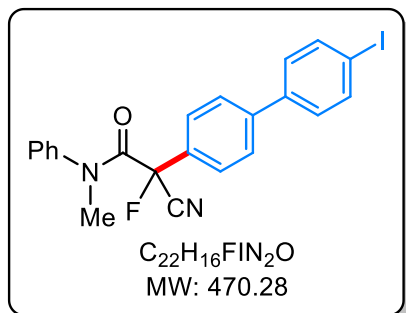
Following the general procedure, treatment of 2-cyano-*N*-methyl-2-fluoro-*N*-phenylacetamide **4a** (38 mg, 0.20 mmol) with (4-ethylphenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2e** (115 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5b** (36 mg, 61%) as colorless oil. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.40. **<sup>13</sup>C NMR** (100 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 162.3 (d,  $J_{C-F}$  = 24.2 Hz, C), 147.3 (C), 140.5 (C), 130.4 (d,  $J_{C-F}$  = 18.7 Hz, CH), 129.3 (CH), 129.3 (CH), 128.7 (CH), 128.6 (CH), 128.6 (CH), 128.6 (CH), 128.6 (CH), 125.5 (d,  $J_{C-F}$  = 5.3 Hz, CH), 125.5 (d,  $J_{C-F}$  = 5.3 Hz, CH), 115.0 (d,  $J_{C-F}$  = 34.5 Hz, C), 89.8 (d,  $J_{C-F}$  = 196.7 Hz, C), 40.7 (CH<sub>2</sub>), 28.7 (CH<sub>3</sub>), 15.5 (CH<sub>3</sub>). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 7.31-7.26 (m, 1H), 7.22-7.17 (m, 6H), 6.83 (s, 2H), 3.31 (s, 3H), 2.67 (q,  $J$  = 7.6 Hz, 2H), 1.24 (t,  $J$  = 7.6 Hz, 3H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): -135.5 (s). **HRMS** for C<sub>18</sub>H<sub>18</sub>FN<sub>2</sub>O<sup>+</sup>: calcd. [M+H]<sup>+</sup>: 297.1398, found: 297.1400.

#### Compound 5c: 2-(4-bromophenyl)-2-cyano-2-fluoro-*N*-methyl-*N*-phenylacetamide



Following the general procedure, treatment of 2-cyano-*N*-methyl-2-fluoro-*N*-phenylacetamide **4a** (38 mg, 0.20 mmol) with (4-bromophenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2i** (126 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5c** (51 mg, 73%) as colorless oil. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.40. **<sup>13</sup>C NMR** (100 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 161.7 (d,  $J_{C-F}$  = 23.5 Hz, C), 140.3 (C), 132.1 (C), 132.3 (CH), 132.3 (CH), 129.5 (CH), 129.5 (CH), 129.0 (CH), 128.5 (CH), 128.5 (CH), 126.9 (d,  $J_{C-F}$  = 5.8 Hz, CH), 126.9 (d,  $J_{C-F}$  = 5.8 Hz, CH), 125.2 (C), 114.5 (d,  $J_{C-F}$  = 34.4 Hz, C), 89.5 (d,  $J_{C-F}$  = 200.1 Hz, C), 40.8 (CH<sub>3</sub>). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 7.48 (d,  $J$  = 8.0 Hz, 2H), 7.35-7.30 (m, 1H), 7.25 (t,  $J$  = 7.2 Hz, 2H), 7.01 (d,  $J$  = 8.0 Hz, 2H), 6.86 (d,  $J$  = 7.2 Hz, 2H), 3.31 (s, 3H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): -138.8 (s). **HRMS** for C<sub>16</sub>H<sub>13</sub>BrFN<sub>2</sub>O<sup>+</sup>: calcd. [M+H]<sup>+</sup>: 347.0190, found: 347.0188.

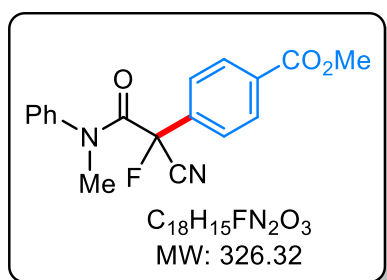
**Compound 5d: 2-cyano-2-fluoro-2-(4'-iodo-[1,1'-biphenyl]-4-yl)-*N*-methyl-*N*-phenylacetamide**



Following the general procedure, treatment of 2-cyano-2-fluoro-*N*-methyl-*N*-phenylacetamide **4a** (38 mg, 0.20 mmol) with (4'-iodo-[1,1'-biphenyl]-4-yl)(mesityl)iodonium 4-methylbenzenesulfonate **2u** (153 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5d** (48 mg, 51%) as colorless solid. *R<sub>f</sub>* (Ethyl acetate/Hexane: 10/90) = 0.20. <sup>13</sup>C NMR (100 MHz, δ

ppm/CDCl<sub>3</sub>): 162.0 (d, *J*<sub>C-F</sub> = 23.7 Hz, C), 142.4 (C), 140.5 (C), 139.2 (C), 138.2 (CH), 138.2 (CH), 132.3 (d, *J*<sub>C-F</sub> = 22.2 Hz, C), 129.4 (CH), 129.4 (CH), 129.0 (CH), 129.0 (CH), 128.9 (CH), 128.5 (CH), 128.5 (CH), 127.4 (CH), 127.4 (CH), 125.9 (d, *J*<sub>C-F</sub> = 5.6 Hz, CH), 125.9 (d, *J*<sub>C-F</sub> = 5.6 Hz, CH), 114.9 (d, *J*<sub>C-F</sub> = 34.6 Hz, C), 94.2 (C), 89.7 (d, *J*<sub>C-F</sub> = 199.0 Hz, C), 40.8 (CH<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.80 (d, *J* = 7.6 Hz, 2H), 7.59-7.46 (m, 3H), 7.36-7.24 (m, 6H), 6.90 (s, 2H), 3.34 (s, 3H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -137.6 (s). HRMS for C<sub>22</sub>H<sub>17</sub>FIN<sub>2</sub>O<sup>+</sup>: calcd. [M+H]<sup>+</sup>: 471.0364, found: 471.0361.

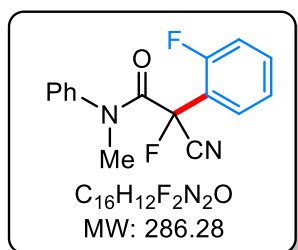
**Compound 5e: methyl 4-(1-cyano-1-fluoro-2-(methyl(phenyl)amino)-2-oxoethyl)benzoate**



Following the general procedure, treatment of 2-cyano-2-fluoro-*N*-methyl-*N*-phenylacetamide **4a** (38 mg, 0.20 mmol) with mesityl(4-(methoxycarbonyl)phenyl)iodonium 4-methylbenzenesulfonate **2k** (122 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5e** (42 mg, 64%) as yellow oil. *R<sub>f</sub>* (Ethyl

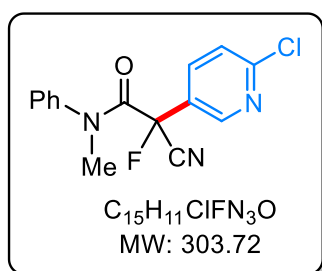
acetate/Hexane: 10/90) = 0.20. <sup>13</sup>C NMR (100 MHz, δ ppm/CDCl<sub>3</sub>): 166.0 (C), 161.6 (d, *J*<sub>C-F</sub> = 22.9 Hz, C), 140.1 (C), 137.3 (d, *J*<sub>C-F</sub> = 22.4 Hz, C), 132.1 (C), 130.1 (CH), 130.1 (CH), 129.4 (CH), 129.4 (CH), 129.0 (CH), 128.4 (CH), 128.4 (CH), 125.2 (d, *J*<sub>C-F</sub> = 6.0 Hz, CH), 125.2 (d, *J*<sub>C-F</sub> = 6.0 Hz, CH), 114.5 (d, *J*<sub>C-F</sub> = 34.2 Hz, C), 89.8 (d, *J*<sub>C-F</sub> = 199.7 Hz, C), 52.5 (CH<sub>3</sub>), 40.7 (CH<sub>3</sub>). <sup>1</sup>H NMR (400 MHz, δ ppm/CDCl<sub>3</sub>): 7.93 (d, *J* = 8.0 Hz, 2H), 7.27-7.21 (m, 3H), 7.15 (t, *J* = 8.0 Hz, 2H), 6.76 (s, 2H), 3.87 (s, 3H), 3.24 (s, 3H). <sup>19</sup>F NMR (376 MHz, δ ppm/CDCl<sub>3</sub>): -140.6 (s). HRMS for C<sub>18</sub>H<sub>16</sub>FN<sub>2</sub>O<sub>3</sub><sup>+</sup>: calcd. [M+H]<sup>+</sup>: 327.1139, found: 327.1142.

### Compound 5f: 2-cyano-2-fluoro-2-(2-fluorophenyl)-*N*-methyl-*N*-phenylacetamide



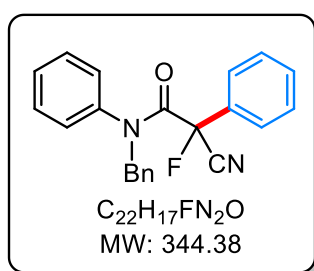
Following the general procedure, treatment of 2-cyano-2-fluoro-*N*-methyl-*N*-phenylacetamide **4a** (38 mg, 0.20 mmol) with (2-fluorophenyl)(mesityl)iodonium 4-methylbenzenesulfonate **2w** (113 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5f** (37 mg, 65%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.25. **Mp** 64–66 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 160.9 (d,  $J_{C-F}$  = 24.0 Hz, C), 159.5 (d,  $J_{C-F}$  = 24.0 Hz, C), 140.6 (C), 132.6 (d,  $J_{C-F}$  = 8.2 Hz, C), 129.5 (CH), 129.5 (CH), 128.8 (CH), 128.2 (CH), 128.2 (CH), 127.8 (CH), 124.6 (CH), 116.4 (d,  $J_{C-F}$  = 19.8 Hz, CH), 114.0 (d,  $J_{C-F}$  = 33.5 Hz, C), 87.3 (d,  $J_{C-F}$  = 196.3 Hz, C), 40.8 (CH<sub>3</sub>).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.40–7.34 (m, 1H), 7.25–7.18 (m, 3H), 7.11–7.0 (m, 5H), 3.37 (s, 3H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -112.5 (s), -137.6 (s). **HRMS** for  $C_{16}H_{13}F_2N_2O^+$ : calcd.  $[M+H]^+$ : 287.0990, found: 287.0987.

### Compound 5g: 2-(6-chloropyridin-3-yl)-2-cyano-2-fluoro-*N*-methyl-*N*-phenylacetamide



Following the general procedure, treatment of 2-cyano-2-fluoro-*N*-methyl-*N*-phenylacetamide **4a** (38 mg, 0.20 mmol) with (6-chloropyridin-3-yl)(mesityl)iodonium 4-methylbenzenesulfonate **2v** (117 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5g** (33 mg, 55%) as colorless oil.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.20.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 161.0 (d,  $J_{C-F}$  = 18.4 Hz, C), 153.9 (C), 146.6 (d,  $J_{C-F}$  = 5.4 Hz, CH), 140.1 (C), 135.9 (C), 129.8 (CH), 129.8 (CH), 129.8 (CH), 129.5 (CH), 128.3 (CH), 128.3 (CH), 124.5 (CH), 114.0 (d,  $J_{C-F}$  = 27.1 Hz, C), 88.0 (d,  $J_{C-F}$  = 161.2 Hz, C), 40.9 (CH<sub>3</sub>).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 8.22 (s, 1H), 7.62 (d,  $J$  = 7.6 Hz, 1H), 7.40–7.30 (m, 4H), 6.95 (d,  $J$  = 7.6 Hz, 2H), 3.33 (s, 3H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -141.5 (s). **HRMS** for  $C_{15}H_{12}ClFN_3O^+$ : calcd.  $[M+H]^+$ : 304.0647, found: 304.0644.

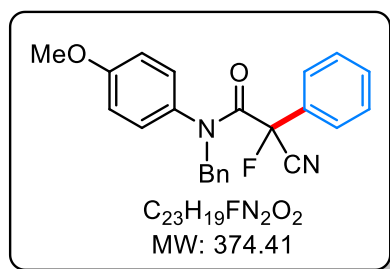
### Compound 5h: *N*-benzyl-2-cyano-2-fluoro-*N*,2-diphenylacetamide



Following the general procedure, treatment of *N*-benzyl-2-cyano-2-fluoro-*N*-phenylacetamide **4b** (53 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5h** (43 mg, 62%) as yellow oil.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.35.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 162.1 (d,  $J_{C-F}$  = 23.7 Hz, C), 138.2 (C), 135.7 (C), 132.9 (d,  $J_{C-F}$  = 22.6 Hz, C), 130.6 (CH), 129.7 (CH), 129.7 (CH), 129.2 (CH), 129.2 (CH), 129.0 (CH), 129.0 (CH), 128.8 (CH), 128.8 (CH), 128.8

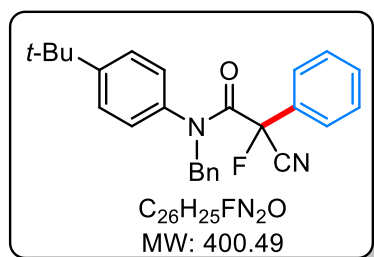
(CH), 128.6 (CH), 128.6 (CH), 128.1 (CH), 125.2 (d,  $J_{C-F}$  = 5.6 Hz, CH), 125.2 (d,  $J_{C-F}$  = 5.6 Hz, CH), 114.9 (d,  $J_{C-F}$  = 34.9 Hz, C), 89.9 (d,  $J_{C-F}$  = 197.3 Hz, C), 56.2 (CH<sub>2</sub>). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 7.36 (t,  $J$  = 7.2 Hz, 1H), 7.28 (t,  $J$  = 7.6 Hz, 2H), 7.24-7.18 (m, 6H), 7.13-7.10 (m, 2H), 7.05 (t,  $J$  = 8.0 Hz, 2H), 6.51 (d,  $J$  = 7.6 Hz, 2H), 4.88 (d,  $J$  = 14.0 Hz, 1H), 4.83 (d,  $J$  = 13.6 Hz, 1H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): -136.7 (s). **HRMS** for C<sub>22</sub>H<sub>18</sub>FN<sub>2</sub>O<sup>+</sup>: calcd. [M+H]<sup>+</sup>: 345.1398, found: 345.1398.

**Compound 5i: *N*-benzyl-2-cyano-2-fluoro-*N*-(4-methoxyphenyl)-2-phenylacetamide**

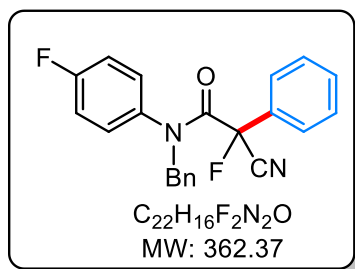


Following the general procedure, treatment of *N*-benzyl-2-cyano-2-fluoro-*N*-(4-methoxyphenyl)acetamide **4c** (60 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5i** (54 mg, 72%) as yellow oil. **R<sub>f</sub>** (Ethyl acetate/Hexane: 10/90) = 0.20. **<sup>13</sup>C NMR** (100 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 162.3 (d,  $J_{C-F}$  = 24.4 Hz, C), 159.7 (C), 135.9 (C), 133.0 (d,  $J_{C-F}$  = 22.7 Hz, C), 131.2 (CH), 131.2 (CH), 130.6 (C), 130.6 (CH), 129.3 (CH), 129.3 (CH), 129.0 (CH), 129.0 (CH), 128.6 (CH), 128.6 (CH), 128.1 (CH), 125.4 (d,  $J_{C-F}$  = 5.2 Hz, CH), 125.4 (d,  $J_{C-F}$  = 5.2 Hz, CH), 114.9 (d,  $J_{C-F}$  = 35.0 Hz, C), 113.9 (CH), 113.9 (CH), 89.6 (d,  $J_{C-F}$  = 195.5 Hz, C), 56.3 (CH<sub>2</sub>), 56.3 (CH<sub>2</sub>), 55.5 (CH<sub>3</sub>). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 7.36 (t,  $J$  = 6.8 Hz, 1H), 7.28 (t,  $J$  = 7.6 Hz, 2H), 7.23-7.20 (m, 5H), 7.12-7.09 (m, 2H), 6.51-6.23 (m, 4H), 4.83 (d,  $J$  = 14.0 Hz, 1H), 4.77 (d,  $J$  = 14.0 Hz, 1H), 3.71 (s, 3H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): -134.4 (s). **HRMS** for C<sub>23</sub>H<sub>20</sub>FN<sub>2</sub>O<sub>2</sub><sup>+</sup>: calcd. [M+H]<sup>+</sup>: 375.1503, found: 375.1505.

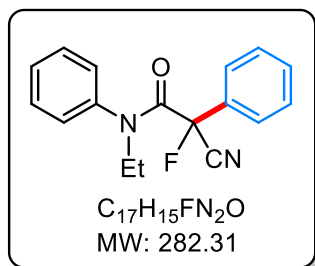
**Compound 5j: *N*-benzyl-*N*-(4-(*tert*-butyl)phenyl)-2-cyano-2-fluoro-2-phenylacetamide**



Following the general procedure, treatment of *N*-benzyl-*N*-(4-(*tert*-butyl)phenyl)-2-cyano-2-fluoroacetamide **4d** (65 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5j** (66 mg, 83%) as yellow oil. **R<sub>f</sub>** (Ethyl acetate/Hexane: 10/90) = 0.35. **<sup>13</sup>C NMR** (100 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 162.3 (d,  $J_{C-F}$  = 24.4 Hz, C), 152.1 (C), 136.0 (C), 135.5 (C), 133.0 (d,  $J_{C-F}$  = 22.7 Hz, C), 130.4 (CH), 129.3 (CH), 129.3 (CH), 129.2 (CH), 129.2 (CH), 128.9 (CH), 128.9 (CH), 128.6 (CH), 128.6 (CH), 128.0 (CH), 125.7 (CH), 125.7 (CH), 125.2 (d,  $J_{C-F}$  = 5.4 Hz, CH), 125.2 (d,  $J_{C-F}$  = 5.4 Hz, CH), 114.9 (d,  $J_{C-F}$  = 34.6 Hz, C), 89.8 (d,  $J_{C-F}$  = 195.7 Hz, C), 56.2 (CH<sub>2</sub>), 34.7 (C), 31.3 (CH<sub>3</sub>), 31.3 (CH<sub>3</sub>), 31.3 (CH<sub>3</sub>). **<sup>1</sup>H NMR** (400 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): 7.28 (t,  $J$  = 7.2 Hz, 1H), 7.19-7.16 (m, 5H), 7.11-7.07 (m, 4H), 6.97 (d,  $J$  = 8.4 Hz, 2H), 6.40 (s, 2H), 4.78 (s, 2H), 1.18 (s, 9H). **<sup>19</sup>F NMR** (376 MHz,  $\delta$  ppm/CDCl<sub>3</sub>): -135.0 (s). **HRMS** for C<sub>26</sub>H<sub>26</sub>FN<sub>2</sub>O<sup>+</sup>: calcd. [M+H]<sup>+</sup>: 401.2024, found: 401.2023.

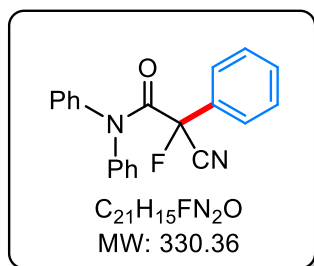
**Compound 5k: *N*-benzyl-2-cyano-2-fluoro-*N*-(4-fluorophenyl)-2-phenylacetamide**

Following the general procedure, treatment of *N*-benzyl-2-cyano-2-fluoro-*N*-(4-fluorophenyl)acetamide **4e** (53 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5k** (54 mg, 74%) as colorless oil.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.30.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 162.4 (d,  $J_{C-F}$  = 248.5 Hz, C), 162.1 (d,  $J_{C-F}$  = 23.8 Hz, C), 135.5 (C), 134.1 (C), 132.8 (d,  $J_{C-F}$  = 22.8 Hz, C), 131.8 (d,  $J_{C-F}$  = 8.8 Hz, CH), 131.8 (d,  $J_{C-F}$  = 8.8 Hz, CH), 130.7 (d,  $J_{C-F}$  = 2.1 Hz, CH), 129.3 (CH), 129.3 (CH), 129.2 (CH), 129.2 (CH), 128.7 (CH), 128.7 (CH), 128.3 (CH), 125.1 (d,  $J_{C-F}$  = 5.5 Hz, CH), 125.1 (d,  $J_{C-F}$  = 5.5 Hz, CH), 115.8 (d,  $J_{C-F}$  = 22.7 Hz, CH), 115.8 (d,  $J_{C-F}$  = 22.7 Hz, CH), 114.8 (d,  $J_{C-F}$  = 35.0 Hz, C), 89.9 (d,  $J_{C-F}$  = 197.1 Hz, C), 56.3 ( $CH_2$ ).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.36 (t,  $J$  = 7.2 Hz, 1H), 7.28 (t,  $J$  = 7.6 Hz, 2H), 7.24-7.18 (m, 5H), 7.13-7.10 (m, 2H), 7.05 (t,  $J$  = 8.0 Hz, 2H), 6.46 (d,  $J$  = 7.6 Hz, 2H), 4.88 (d,  $J$  = 14.0 Hz, 1H), 4.83 (d,  $J$  = 13.6 Hz, 1H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -111.3 (s), -137.0 (s). HRMS for  $C_{22}H_{17}F_2N_2O^+$ : calcd.  $[M+H]^+$ : 363.1303, found: 363.1316.

**Compound 5l: 2-cyano-*N*-ethyl-2-fluoro-*N*,2-diphenylacetamide**

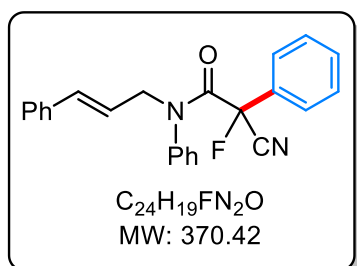
Following the general procedure, treatment of 2-cyano-2-fluoro-*N*-ethyl-*N*-phenylacetamide **4f** (53 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5l** (37 mg, 66%) as brown oil.  $R_f$  (Ethyl acetate/Hexane: 10/96) = 0.30.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 161.5 (d,  $J_{C-F}$  = 23.1 Hz, C), 138.6 (C), 133.2 (d,  $J_{C-F}$  = 22.8 Hz, C), 130.5 (CH), 129.6 (CH), 129.6 (CH), 129.1 (CH), 129.1 (CH), 129.0 (CH), 129.0 (CH), 128.8 (CH), 125.2 (d,  $J_{C-F}$  = 5.5 Hz, CH), 125.2 (d,  $J_{C-F}$  = 5.5 Hz, CH), 115.0 (d,  $J_{C-F}$  = 34.8 Hz, C), 89.9 (d,  $J_{C-F}$  = 197.6 Hz, C), 47.7 ( $CH_2$ ), 12.4 ( $CH_3$ ).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.39 (t,  $J$  = 7.2 Hz, 1H), 7.33-7.21 (m, 5H), 7.60 (t,  $J$  = 7.6 Hz, 2H), 6.73 (d,  $J$  = 8.0 Hz, 2H), 3.77-3.68 (m, 2H), 1.10 (t,  $J$  = 7.2 Hz, 3H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -137.3 (s). HRMS for  $C_{17}H_{16}FN_2O^+$ : calcd.  $[M+H]^+$ : 283.1241, found: 283.1243.

### Compound 5m: 2-cyano-2-fluoro-*N,N*,2-triphenylacetamide



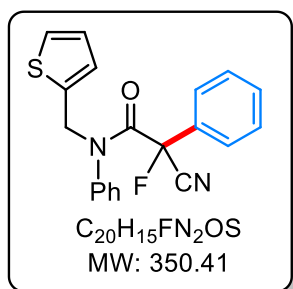
Following the general procedure, treatment of 2-cyano-2-fluoro-*N,N*-diphenylacetamide **4g** (51 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5m** (44 mg, 66%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.25. **Mp** 114-116 °C.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 162.3 (d,  $J_{C-F}$  = 24.1 Hz, C), 140.4 (C), 133.0 (d,  $J_{C-F}$  = 22.7 Hz, C), 130.7 (d,  $J_{C-F}$  = 2.1 Hz, CH), 130.7 (d,  $J_{C-F}$  = 2.1 Hz, CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.3 (CH), 129.2 (CH), 129.2 (CH), 129.2 (CH), 128.2 (C), 128.2 (C), 125.3 (d,  $J_{C-F}$  = 5.5 Hz, CH), 125.3 (d,  $J_{C-F}$  = 5.5 Hz, CH), 125.3 (d,  $J_{C-F}$  = 5.5 Hz, CH), 114.9 (d,  $J_{C-F}$  = 34.6 Hz, C), 90.0 (d,  $J_{C-F}$  = 198.1 Hz, C).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.42-7.04 (m, 15H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -136.2 (s). **HRMS** for  $C_{21}H_{16}FN_2O^+$ : calcd.  $[M+H]^+$ : 331.1241, found: 331.1230.

### Compound 5n: *N*-cinnamyl-2-cyano-2-fluoro-*N*,2-diphenylacetamide



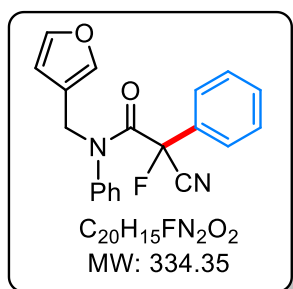
Following the general procedure, treatment of *N*-cinnamyl-2-cyano-2-fluoro-*N*-phenylacetamide **4h** (59 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5n** (54 mg, 73%) as yellow oil.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.25.  $^{13}C$  NMR (100 MHz,  $\delta$  ppm/ $CDCl_3$ ): 161.9 (d,  $J_{C-F}$  = 23.8 Hz, C), 138.6 (C), 136.4 (C), 135.2 (CH), 133.0 (d,  $J_{C-F}$  = 22.4 Hz, C), 130.6 (CH), 129.7 (CH), 129.7 (CH), 129.1 (CH), 129.1 (CH), 129.1 (CH), 129.1 (CH), 129.0 (CH), 128.7 (CH), 128.7 (CH), 128.2 (CH), 126.6 (CH), 126.6 (CH), 125.3 (d,  $J_{C-F}$  = 5.6 Hz, CH), 125.3 (d,  $J_{C-F}$  = 5.6 Hz, CH), 122.1 (CH), 114.9 (d,  $J_{C-F}$  = 34.7 Hz, C), 89.9 (d,  $J_{C-F}$  = 197.7 Hz, C), 55.1 (CH<sub>2</sub>).  $^1H$  NMR (400 MHz,  $\delta$  ppm/ $CDCl_3$ ): 7.39 (t,  $J$  = 7.2 Hz, 1H), 7.33-7.20 (m, 10H), 7.15 (t,  $J$  = 7.6 Hz, 2H), 6.76 (d,  $J$  = 8.0 Hz, 2H), 6.35 (d,  $J$  = 16.0 Hz, 1H), 6.23-6.16 (m, 1H), 4.41 (d,  $J$  = 6.8 Hz, 2H).  $^{19}F$  NMR (376 MHz,  $\delta$  ppm/ $CDCl_3$ ): -137.5 (s). **HRMS** for  $C_{24}H_{20}FN_2O^+$ : calcd.  $[M+H]^+$ : 371.1554, found: 371.1550.

### Compound 5o: 2-cyano-2-fluoro-*N*,2-diphenyl-*N*-(thiophen-2-ylmethyl)acetamide



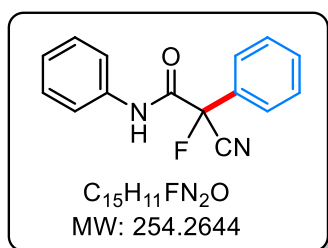
Following the general procedure, treatment of 2-cyano-2-fluoro-*N*-phenyl-*N*-(thiophen-2-ylmethyl)acetamide **4i** (55 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5o** (50 mg, 72%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.30. **Mp** 99-101 °C.  $^{13}\text{C}$  NMR (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 162.0 (d,  $J_{\text{C-F}}$  = 19.3 Hz, C), 138.2 (C), 137.4 (C), 132.9 (d,  $J_{\text{C-F}}$  = 18.1 Hz, C), 130.6 (CH), 129.7 (CH), 129.7 (CH), 129.1 (CH), 129.0 (CH), 129.0 (CH), 129.0 (CH), 129.0 (CH), 128.5 (CH), 126.6 (d,  $J_{\text{C-F}}$  = 6.5 Hz, CH), 126.6 (d,  $J_{\text{C-F}}$  = 6.5 Hz, CH), 125.3 (d,  $J_{\text{C-F}}$  = 4.4 Hz, CH), 125.3 (d,  $J_{\text{C-F}}$  = 4.4 Hz, CH), 114.8 (d,  $J_{\text{C-F}}$  = 27.9 Hz, C), 89.7 (d,  $J_{\text{C-F}}$  = 158.6 Hz, C), 50.9 ( $\text{CH}_2$ ).  $^1\text{H}$  NMR (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 7.35 (t,  $J$  = 7.2 Hz, 1H), 7.26 (t,  $J$  = 7.6 Hz, 2H), 7.22-7.18 (m, 4H), 7.08 (t,  $J$  = 7.6 Hz, 2H), 6.83-6.81 (m, 1H), 6.71 (d,  $J$  = 3.6 Hz, 1H), 6.54 (d,  $J$  = 8.0 Hz, 2H), 4.96 (s, 2H).  $^{19}\text{F}$  NMR (376 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): -137.0 (s). **HRMS** for  $\text{C}_{20}\text{H}_{16}\text{FN}_2\text{OS}^+$ : calcd.  $[\text{M}+\text{H}]^+$ : 351.0962, found: 351.0959.

### Compound 5p: 2-cyano-2-fluoro-*N*-(furan-3-ylmethyl)-*N*,2-diphenylacetamide



Following the general procedure, treatment of 2-cyano-2-fluoro-*N*-(furan-3-ylmethyl)-*N*-phenylacetamide **4j** (53 mg, 0.20 mmol) with mesityl(phenyl)iodonium 4-methylbenzenesulfonate **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5p** (51 mg, 76%) as yellow oil.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.25.  $^{13}\text{C}$  NMR (100 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 162.0 (d,  $J_{\text{C-F}}$  = 24.0 Hz, C), 143.4 (CH), 141.8 (CH), 138.3 (C), 132.9 (d,  $J_{\text{C-F}}$  = 22.8 Hz, C), 130.6 (CH), 129.6 (CH), 129.6 (CH), 129.0 (CH), 129.0 (CH), 128.9 (CH), 128.9 (CH), 128.9 (CH), 125.2 (d,  $J_{\text{C-F}}$  = 5.5 Hz, CH), 125.2 (d,  $J_{\text{C-F}}$  = 5.5 Hz, CH), 119.6 (C), 114.9 (d,  $J_{\text{C-F}}$  = 34.9 Hz, C), 111.0 (CH), 89.8 (d,  $J_{\text{C-F}}$  = 197.4 Hz, C), 47.2 ( $\text{CH}_2$ ).  $^1\text{H}$  NMR (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 7.37 (t,  $J$  = 7.2 Hz, 1H), 7.32-7.17 (m, 7H), 7.10 (t,  $J$  = 8.0 Hz, 2H), 6.58 (d,  $J$  = 8.0 Hz, 2H), 6.30 (s, 1H), 4.65 (s, 2H).  $^{19}\text{F}$  NMR (376 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): -137.1 (s). **HRMS** for  $\text{C}_{20}\text{H}_{16}\text{FN}_2\text{O}_2^+$ : calcd.  $[\text{M}+\text{H}]^+$ : 335.1190, found: 335.1195.

### Compound 5q: 2-cyano-2-fluoro-*N*,2-diphenylacetamide

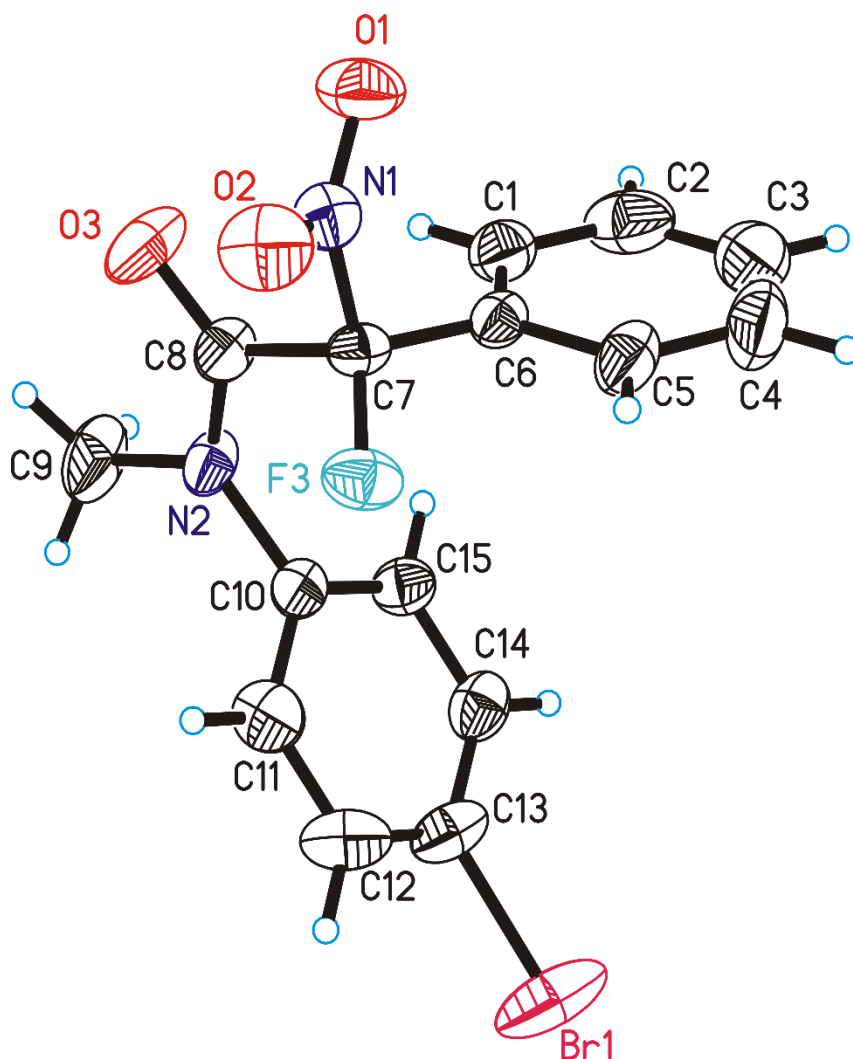


Following the general procedure, treatment of 2-cyano-2-fluoro-*N*-phenylacetamide **4k** (36 mg, 0.20 mmol) with **2a** (109 mg, 0.22 mmol) in the presence of *t*-BuOK (27 mg, 0.24 mmol) in THF (2 mL) at 25 °C for 30 min followed by column chromatography afforded the product **5q** (15 mg, 30%) as white solid.  $R_f$  (Ethyl acetate/Hexane: 10/90) = 0.30. **Mp** 124-126 °C.  $^{13}\text{C}$  NMR (100

MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 160.6 (d,  $J_{\text{C-F}} = 22.2$  Hz, C), 135.8 (C), 132.0 (d,  $J_{\text{C-F}} = 22.2$  Hz, C), 131.4 (CH), 129.5 (CH), 129.5 (CH), 129.5 (CH), 129.5 (CH), 126.2 (CH), 125.8 (d,  $J_{\text{C-F}} = 5.7$  Hz, CH), 125.8 (d,  $J_{\text{C-F}} = 5.7$  Hz, CH), 120.4 (CH), 120.4 (CH), 114.4 (d,  $J_{\text{C-F}} = 33.4$  Hz, C), 89.6 (d,  $J_{\text{C-F}} = 200.0$  Hz, C).  **$^1\text{H}$  NMR** (400 MHz,  $\delta$  ppm/ $\text{CDCl}_3$ ): 8.10 (s, 1H), 7.71-7.68 (m, 2H), 7.57 (d,  $J = 7.6$  Hz, 2H), 7.53-7.48 (m, 3H), 7.38 (d,  $J = 7.6$  Hz, 2H), 7.22 (t,  $J = 7.6$  Hz, 1H).  **$^{19}\text{F}$  NMR** (376 MHz  $\delta$  ppm/ $\text{CDCl}_3$ ): -142.8 (s). **HRMS** for  $\text{C}_{15}\text{H}_{12}\text{FN}_2\text{O}^+$ : calcd.  $[\text{M}+\text{H}]^+$ : 255.0928, found: 255.0925.

### X-Ray Data Collection and Structure Refinement Details for compound **3am**:

A good quality colorless single crystal of size 0.38 x 0.21 x 0.10 mm, was selected under a polarizing microscope and was mounted on a glass fiber for data collection. Single crystal X-ray data for compound **3am** were collected on the Rigaku Kappa 3 circle diffractometer equipped with the AFC12 goniometer and enhanced sensitivity (HG) Saturn724+ CCD detector in the 4x4 bin mode using the monochromated Mo-K $\alpha$  radiation generated from the microfocus sealed tube MicroMax-003 X-ray generator equipped with specially designed confocal multilayer optics. Data collection was performed using  $\omega$ -scans of 0.5° steps at 293(2) K. Cell determination, data collection and data reduction was performed using the Rigaku CrystalClear-SM Expert 2.1 b24 software.<sup>1</sup> Asymmetric unit contains four molecules of compound **3am** along with two water molecules. Structure solution and refinement were performed by using SHELXTL-NT.<sup>2</sup> Refinement of coordinates and anisotropic thermal parameters of non-hydrogen atoms were carried out by the full-matrix least-squares method. The hydrogen atoms attached to carbon atoms were generated with idealized geometries and isotropically refined using a riding model.



**Figure S1.** ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the crystal structure of compound **3am** determined at 293 K.

**Table S1** Crystal data and structure refinement details for **3am**.

| Compound                                                                 | <b>3am</b>                                                         |
|--------------------------------------------------------------------------|--------------------------------------------------------------------|
| Empirical formula                                                        | C <sub>15</sub> H <sub>12</sub> Br F N <sub>2</sub> O <sub>3</sub> |
| Formula weight                                                           | 367.18                                                             |
| Crystal System                                                           | Monoclinic                                                         |
| Space group                                                              | <i>P</i> 2 <sub>1</sub> /c                                         |
| <i>a</i> (Å)                                                             | 15.548(5)                                                          |
| <i>b</i> (Å)                                                             | 24.427(7)                                                          |
| <i>c</i> (Å)                                                             | 8.266(2)                                                           |
| $\alpha$ (°)                                                             | 90.00                                                              |
| $\beta$ (°)                                                              | 91.708(5)                                                          |
| $\gamma$ (°)                                                             | 90.00                                                              |
| <i>V</i> (Å <sup>3</sup> )                                               | 3138.0(16)                                                         |
| <i>Z</i>                                                                 | 8                                                                  |
| <i>D</i> <sub>c</sub> (g/cm <sup>3</sup> )                               | 1.554                                                              |
| <i>F</i> <sub>000</sub>                                                  | 1472                                                               |
| $\mu$ (mm <sup>-1</sup> )                                                | 2.643                                                              |
| $\theta_{\max}$ (°)                                                      | 25.36                                                              |
| Total reflections                                                        | 20516                                                              |
| Unique reflections                                                       | 5599                                                               |
| Reflections [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]                        | 2836                                                               |
| Parameters                                                               | 399                                                                |
| <i>R</i> <sub>int</sub>                                                  | 0.0655                                                             |
| Goodness-of-fit                                                          | 1.004                                                              |
| <i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )] | 0.0562                                                             |
| <i>wR</i> ( <i>F</i> <sup>2</sup> , all data)                            | 0.1405                                                             |
| CCDC No.                                                                 | 1940141                                                            |

1. CrystalClear 2.1, Rigaku Corporation, Tokyo, Japan
2. Sheldrick, G. M. *Acta Crystallogr., Sect. A* **2008**, *64*, 112–122.

