



Since January 2020 Elsevier has created a COVID-19 resource centre with free information in English and Mandarin on the novel coronavirus COVID-19. The COVID-19 resource centre is hosted on Elsevier Connect, the company's public news and information website.

Elsevier hereby grants permission to make all its COVID-19-related research that is available on the COVID-19 resource centre - including this research content - immediately available in PubMed Central and other publicly funded repositories, such as the WHO COVID database with rights for unrestricted research re-use and analyses in any form or by any means with acknowledgement of the original source. These permissions are granted for free by Elsevier for as long as the COVID-19 resource centre remains active.



# Lewis acid-promoted cascade reaction for the synthesis of Michael acceptors and its application in a dimerization reaction



Xiao-Jing Zhao <sup>a, b, 1</sup>, Jie Zhao <sup>a, b, 1</sup>, Xin Sun <sup>a, b</sup>, Ji-Kai Liu <sup>c, \*\*</sup>, Bin Wu <sup>a, c, \*</sup>

<sup>a</sup> State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences, Kunming 650201, China

<sup>b</sup> University of Chinese Academy of Sciences, Beijing 100049, China

<sup>c</sup> School of Pharmaceutical Sciences, South-Central University for Nationalities, Wuhan 430074, China

## ARTICLE INFO

### Article history:

Received 17 March 2017

Received in revised form

25 April 2017

Accepted 4 May 2017

Available online 6 May 2017

### Keywords:

Dimethyl sulfoxide

Michael acceptors

Lewis acid

Pummerer

Dimerization

## ABSTRACT

An efficient Lewis acid-promoted cascade reaction with dimethyl sulfoxide as a methylene source for the synthesis of Michael acceptors is reported. The key to developing this procedure is the selection of a mild base to modulate the equilibrium of various intermediates in order to drive the reaction forward to the formation of Michael acceptor and dimeric compound products. Extensive studies were performed to gain insight into a possible reaction mechanism.

© 2017 Elsevier Ltd. All rights reserved.

## 1. Introduction

Molecules containing various Michael acceptor scaffolds are common in pharmaceuticals, agrochemicals and natural products and have attracted considerable attention from organic chemists and medicinal chemists.<sup>1</sup> Michael acceptors have been found to have various different biological activities, such as antibacterial, anticancer and antiviral activities.<sup>2</sup> For example, the crystal structure of HCoV-NL63 M<sup>Pro</sup> complexed with a Michael acceptor inhibitor was obtained, providing insight into the rational development of wide spectrum antiviral therapeutics to treat infections caused by human coronaviruses.<sup>3</sup> Two Michael acceptors used as cysteine protease inhibitors, K-777 (CRA-3316) and rupintrivir (AG7088), and developed to treat Chagas disease and as an antirhinoviral agent, respectively, are already in clinical trials.<sup>4</sup> On the other hand, as basic building blocks Michael acceptors have

been extensively employed in the area of organic chemistry, such as in multicomponent cascade or Domino reactions,<sup>5</sup> transition-metal catalyzed reactions,<sup>6</sup> enantioselective organocatalysis<sup>7</sup> and complex natural product synthesis.<sup>8</sup>

In the textbook of graduate students, dimethyl sulfoxide (DMSO) is described as a high-boiling, protic and low-toxicity solvent, and a widely used oxidant<sup>9</sup> in several named reactions, such as the Swern oxidation<sup>10</sup> and Pfitzner-Moffatt oxidation.<sup>11</sup> In the past few decades, the nature of DMSO has been further exploited as a reagent that can act as an oxygen source, a carbon source and a sulfur source.<sup>12</sup> However, the reaction scope of DMSO is still limited and needs to be expanded. To date, a cascade reaction with DMSO as both a solvent and one-carbon source for the preparation of Michael acceptors has not been reported.

Herein, we report a Lewis acid-promoted cascade reaction for the synthesis of  $\alpha,\beta$ -unsaturated amides. The proper choice of base in the reaction results in the production of dimeric compounds. The Pummerer rearrangement with DMSO as a methylene source is believed to be an initial step for the reaction based on mechanistic studies.

\*\* Corresponding author.

\* Corresponding author. State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences, Kunming 650201, China.

E-mail addresses: [jkliu@mail.kib.ac.cn](mailto:jkliu@mail.kib.ac.cn) (J.-K. Liu), [wubin@mail.kib.ac.cn](mailto:wubin@mail.kib.ac.cn) (B. Wu).

<sup>1</sup> These authors contributed equally to this work.

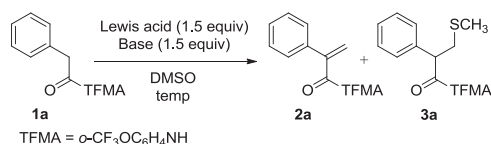
## 2. Results and discussion

We are focused on C(sp<sup>3</sup>)-H bond functionalization reactions.<sup>13</sup> In this project, the C(sp<sup>3</sup>)-H bond functionalization in amide substrate **1a** was tested under the following reaction conditions: CoCl<sub>2</sub> (1.5 equiv), KOAc (2.0 equiv) and AgOAc (2.0 equiv) dissolved in DMSO (2.0 mL) in a microwave and heated at 150 °C for 2 h. To our delight, functionalization at the benzylic position occurred as expected, affording  $\alpha$ ,  $\beta$ -unsaturated carboxylamide **2a** in a 23% yield (Table 1, entry 1). Control experiments showed that this reaction proceeded with similar results in the absence of AgOAc, while the reaction did not occur without KOAc (Table 1, entries 2–4). Then, various Lewis acids were screened in this reaction (Table 1, entries 5–9). When one equivalent of PbCl<sub>2</sub> was used as the Lewis acid, the yield of product **2a** increased to 55% along with some amount of sulfide product **3a** (Table 1, entry 9). The effect of the base on the reaction was screened with various bases. The results of this screening indicated that potassium acetate and sodium acetate are the most favorable bases for this reaction (Table 1, entries 9–11 and details in supporting information). When the reaction was carried out in air or under O<sub>2</sub> atmosphere, the yield of product decreased (Table 1, entries 12–13). In contrast, under N<sub>2</sub>, the reaction afforded product **2a** in a 65% yield (Table 1, entries 14). This indicated that the oxidizing reagents hindered the reaction from proceeding. The effect of temperature on the reaction was subsequently investigated, which showed that temperatures either above 160 °C or below 150 °C were not suitable for this reaction (Table 1, entries 14–17). Finally, optimized reaction conditions of PbCl<sub>2</sub> (1.5 equiv) and KOAc (1.5 equiv) in DMSO at 156 °C in a sealed tube under N<sub>2</sub> for 13 h gave the product **2a** in 77% yield and **3a** in 12% yields, respectively (Table 1, entry 17). Interestingly, when Ac<sub>2</sub>O

was used instead of PbCl<sub>2</sub>, sulfide product **3a** was obtained in 50% yield (Table 1, entry 18). The combination of Ac<sub>2</sub>O/AcOH, or Ac<sub>2</sub>O/TsOH, or TFAA/TFA in DMSO, commonly used in the Pummerer rearrangement reaction, did not afford any product.

The coexistence of products **2a** and **3a** throughout the process of optimizing the reaction conditions stimulated us to investigate the mechanism of this reaction. Several experiments concerning the study of the mechanism were conducted (eqs. (1)–(4)). Isolated sulfide **3a** was subjected to the standard reaction conditions, affording product **2a** in 75% yield with 22% recovery of **3a** (eq. (1)). When purified **2a** was put back under the standard conditions, it gave 17% yield of **3a** with 74% recovery of **2a**. These results indicated that there is an equilibrium between **2a** and **3a** in the reaction mixture. When the reaction was carried out in DMSO-d<sub>6</sub>, it provided the corresponding deuterated products (**d**<sub>2</sub>-**2a** and **d**<sub>5</sub>-**3a**) in 73% and 9% yields, respectively, which provided precise evidence for the methylene group originating from the solvent dimethylsulfoxide (eq. (3)). When six equivalents of TMPO was added to a reaction run under the reaction conditions, 53% of **2a** and 5% of **3a** were still obtained, which suggested that this reaction probably did not involve any radical species (eq. (4)). Moreover, two control experiments showed that both Lewis acid PbCl<sub>2</sub> and KOAc are necessary for the reaction to occur (see the supporting information).

**Table 1**  
Optimization of reaction conditions.<sup>a,b</sup>



| Entry           | Lewis acid        | Base              | Additive (equiv) | Temp (°C) | Product (%) |    |
|-----------------|-------------------|-------------------|------------------|-----------|-------------|----|
|                 |                   |                   |                  |           | 2a          | 3a |
| 1               | CoCl <sub>2</sub> | KOAc              | AgOAc/2          | 150       | 23          | —  |
| 2               | —                 | KOAc              | AgOAc/2          | 150       | 12          | 8  |
| 3               | CoCl <sub>2</sub> | —                 | AgOAc/2          | 150       | trace       | —  |
| 4               | CoCl <sub>2</sub> | KOAc              | —                | 150       | 24          | —  |
| 5               | CoF <sub>2</sub>  | KOAc              | —                | 150       | 26          | 6  |
| 6               | CoBr <sub>2</sub> | KOAc              | —                | 150       | 20          | 6  |
| 7               | ZnCl <sub>2</sub> | KOAc              | —                | 150       | 30          | 9  |
| 8               | MnCl <sub>2</sub> | KOAc              | —                | 150       | 23          | 7  |
| 9               | PbCl <sub>2</sub> | KOAc              | —                | 150       | 55          | 18 |
| 10              | PbCl <sub>2</sub> | NaOAc             | —                | 150       | 55          | 14 |
| 11              | PbCl <sub>2</sub> | KHCO <sub>3</sub> | —                | 150       | 10          | —  |
| 12 <sup>c</sup> | PbCl <sub>2</sub> | KOAc              | —                | 150       | 33          | 21 |
| 13 <sup>d</sup> | PbCl <sub>2</sub> | KOAc              | —                | 150       | trace       | —  |
| 14 <sup>e</sup> | PbCl <sub>2</sub> | KOAc              | —                | 150       | 65          | 18 |
| 15 <sup>e</sup> | PbCl <sub>2</sub> | KOAc              | —                | 160       | 48          | 26 |
| 16 <sup>e</sup> | PbCl <sub>2</sub> | KOAc              | —                | 156       | 66          | 14 |
| 17 <sup>e</sup> | PbCl <sub>2</sub> | KOAc              | —                | 156       | 77          | 12 |
| 18              | Ac <sub>2</sub> O | KOAc              | —                | 156       | —           | 50 |

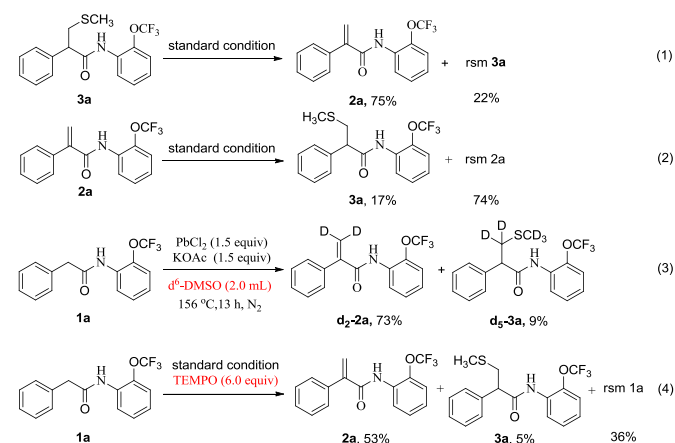
<sup>a</sup> Entry 1–11 was conducted in microwave for 2 h, entry 12–18 was conducted in sealed tube for 13 h. Isolated yields.

<sup>b</sup> Lewis acid (1.0 equiv) was used in entry 9–16.

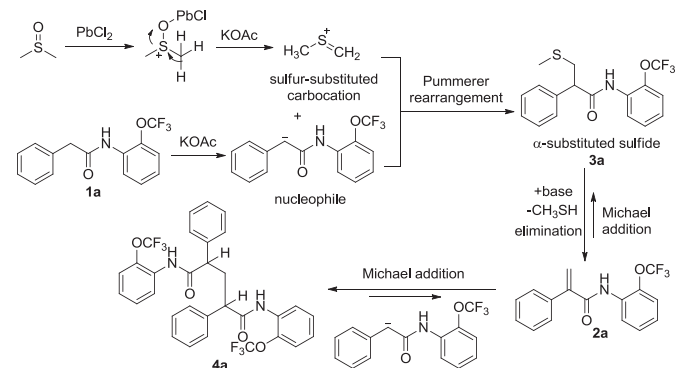
<sup>c</sup> The reaction was carried out in air.

<sup>d</sup> The reaction was carried out under O<sub>2</sub>.

<sup>e</sup> The reaction was carried out under N<sub>2</sub>.



Based on these preliminary results and some leading references on the Pummerer rearrangement<sup>14</sup> reaction, a rational mechanism for this reaction is proposed in Scheme 1. A sulfur-substituted carbocation is obtained through promotion by both the Lewis acid (PbCl<sub>2</sub>) and base (KOAc), and it then reacts with a carbon nucleophile to afford  $\alpha$ -substituted sulfide **3a**. There is an equilibrium between **3a** and **2a** in the reaction mixture. Michael acceptor



**Scheme 1.** Possible mechanism.

product **2a** is afforded by an elimination reaction of **3a** affected by the base (KOAc). Meanwhile, **2a** undergoes a Michael addition reaction to afford **3a**. Furthermore, another Michael addition reaction proceeds with **2a** through the attack of a carbon nucleophile to afford **4a**.

With the optimized conditions in hand, we next explored the effect on the reaction of substitution on the phenyl ring. The results are summarized in Table 2. Generally, electron-donating groups on the phenyl ring under our standard conditions led to good to excellent combined yields of products **2a-o** and **3a-o** (Table 2). This reaction tolerated mono-, di- or even tri-substituted patterns on the phenyl ring. Interestingly, a substrate with a heterocycle instead of a phenyl ring, such as thiofuran, afforded the corresponding product (**2o**) in a synthetically useful yield (Table 2). When substrates with electron-withdrawing groups on the phenyl rings were subjected to the standard reaction conditions, they gave low yields of the corresponding products. After an exploration of the types and amounts of bases, it was found that decreasing the amount of KOAc to 0.6 equivalent led to good yields of **2p-x** and **3p-x** with the concomitant production of dimeric compounds **4p-v** (Table 3). The reaction tolerated many electron-withdrawing groups on the phenyl rings, such as F, Cl, Br, I, OCF<sub>3</sub> and SCF<sub>3</sub>.

The substrate scope with different phenyl amine groups was subsequently investigated (Table 4). In general, electron-withdrawing groups on the phenyl amine rings promoted the reaction, probably due to the easy formation of a carbon anion at the benzylic position of these substrates in the reaction mixture. In the case of substrates bearing alkylamines being used as the amide component, the reaction did not work at all.

After an extensive screening of many different bases, KHCO<sub>3</sub> was identified to be the optimal one for producing a set of dimeric products (**4a**, **4l-m**, **4o-x**) in synthetically useful yields, probably because the mild basicity of KHCO<sub>3</sub> modulates the equilibrium between the intermediates, such as the sulfur-substituted

carbocation, carbon nucleophile,  $\alpha$ -substituted sulfide **3a** and Michael acceptor **2a**, all of which exist in the reaction mixture, to directly push the reaction forward to the formation of dimeric compounds (Table 5). Unfortunately, in the case of substrates with electron-donating groups on the phenyl ring, the reaction was unable to afford any of the corresponding dimeric products, regardless of the kind of base used.

### 3. Conclusion

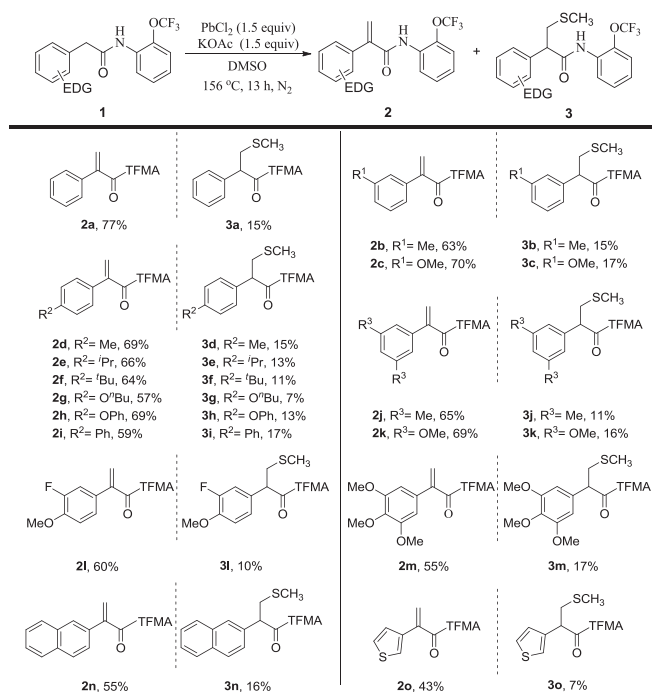
In conclusion, we have studied a Lewis acid-promoted cascade reaction with dimethyl sulfoxide as a methylene source for the synthesis of  $\alpha,\beta$ -unsaturated amides. The reaction proceeds with a wide substrate scope under mild conditions and with inexpensive reagents. The identity of both the Lewis acid and base were found to be critical for this reaction to proceed well. Extensive studies provide insight into a possible mechanism, leading to the belief that the Pummerer rearrangement with dimethyl sulfoxide as a methylene source is an initial step.

### 4. Experimental section

#### 4.1. General techniques

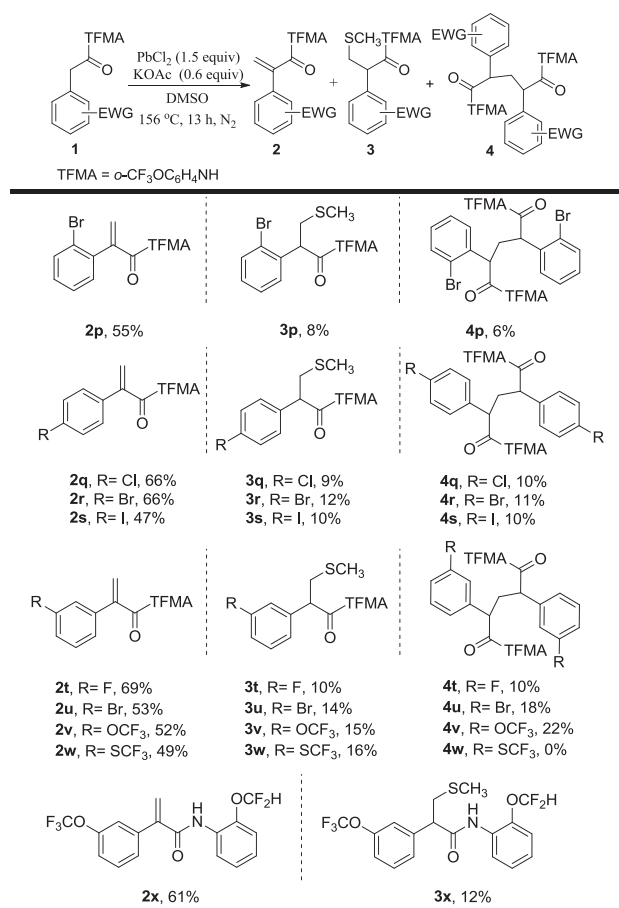
All melting points are uncorrected. Preparative chromatographic separations were performed on silica gel (300–400 mesh). Reactions were followed by TLC analysis using silica plates with a fluorescent indicator (254 nm) and visualized with a UV lamp. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded in Fourier transform mode at the field strength specified on a 400, or 600 MHz spectrometer. Spectra were obtained on CDCl<sub>3</sub> solution in 5 mm diameter tubes, and chemical shifts in ppm (part per million) are quoted relative to the residual signals of chloroform ( $\delta_{\text{H}}$  7.26 ppm, or  $\delta_{\text{C}}$  77.00 ppm). *J* values are given in hertz. IR spectra were measured for samples as

**Table 2**  
Substrate scope with the electron-donating groups on the phenyl ring.<sup>a</sup>



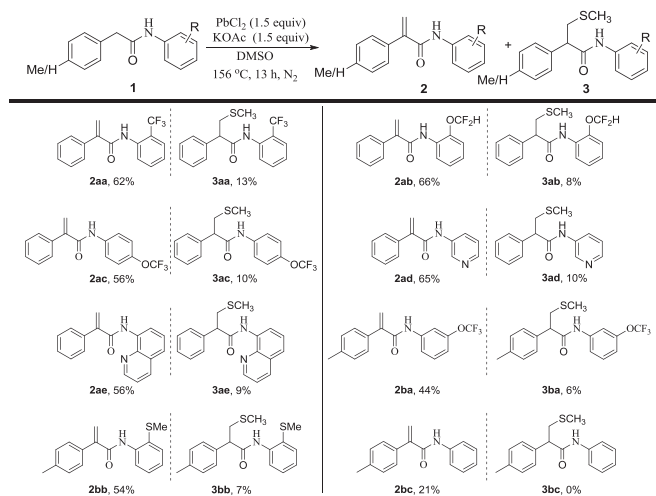
<sup>a</sup> Isolated yields.

**Table 3**  
Substrate scope with the electron-withdrawing groups on the phenyl ring.<sup>a</sup>



<sup>a</sup> Isolated yields.

**Table 4**  
The effect on the reaction of substituents on the phenyl amine ring.<sup>a</sup>



<sup>a</sup> Isolated yields.

KBr pellets in a FT-IR spectrophotometer. High resolution mass spectra (HRMS) were measured at 70 eV using a double focusing

magnetic sector mass analyzer with an EI source or ESI source. 2-Phenyl-N-[2-(trifluoromethoxy) phenyl]acetamides (**1a-1x**, **1aa-**

**Table 5**The effect of base on the production of dimeric compounds **4**.<sup>a</sup>

| KHCO <sub>3</sub> (equiv) | Product (%)     | Rsm 1 (%)       | KHCO <sub>3</sub> (equiv) | Product (%)     | Rsm 1 (%)       |
|---------------------------|-----------------|-----------------|---------------------------|-----------------|-----------------|
| 3.0                       | <b>2a</b> , 30% | <b>4a</b> , 18% | 2.8                       | <b>2s</b> , 17% | <b>4s</b> , 52% |
| 2.2                       | <b>2i</b> , 39% | <b>4i</b> , 32% | 3.0                       | <b>2t</b> , 10% | <b>4t</b> , 61% |
| 2.5                       | <b>2n</b> , 22% | <b>4n</b> , 40% | 1.0                       | <b>2u</b> , 14% | <b>4u</b> , 56% |
| 5.0                       | <b>2o</b> , 18% | <b>4o</b> , 28% | 1.5                       | <b>2v</b> , 10% | <b>4v</b> , 66% |
| 2.0                       | <b>2q</b> , 21% | <b>4q</b> , 48% | 1.0                       | <b>2w</b> , 18% | <b>4w</b> , 61% |
| 3.0                       | <b>2r</b> , 14% | <b>4r</b> , 56% | 2.5                       | <b>2x</b> , 6%  | <b>4x</b> , 56% |

<sup>a</sup> Isolated yields.

**1ae** and **1ba-1bc**) were prepared according to the literature procedure.<sup>13a</sup> Among them, substrates **1aa**,<sup>15</sup> **1ad**<sup>16</sup> and **1ae**<sup>17</sup> were known.

#### 4.2. General procedure for the preparation of 2-phenyl-N-[2-(trifluoromethoxy)phenyl]

**Acetamides (1a-1x, 1ab, 1ac and 1ba-1bc).** To a solution of 2-phenylacetic acid (7.0 mmol), 2-(trifluoromethoxy)aniline (7.7 mmol) in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (25 mL) were added EDCI (1.745 g, 9.1 mmol) and DMAP (256.6 mg, 2.1 mmol). The reaction mixture was stirred at room temperature overnight, diluted with HCl (1 M) aqueous solution, and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 25 mL). The combined organic phase was washed with saturated NaHCO<sub>3</sub> aqueous solution and brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated under vacuum. Purification by flash chromatography (Silica gel, petroleum ether: ethyl acetate = 50: 1 as eluent) gave the corresponding 2-phenyl-N-[2-(trifluoromethoxy)phenyl]acetamide compound.

**2-Phenyl-N-[2-(trifluoromethoxy)phenyl]acetamide (1a):** White solid; mp 85–86 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.41 (d, *J* = 8.1 Hz, 1 H), 7.60–7.30 (m, 6 H), 7.26 (t, *J* = 7.8 Hz, 1 H), 7.16 (d, *J* = 8.2 Hz, 1 H), 7.10–7.01 (m, 1 H), 3.79 (s, 2 H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 169.0, 137.9, 133.7, 130.5, 129.6, 129.4, 128.0, 127.5, 124.2, 120.2 (q, *J*<sub>C-F</sub> = 257.8 Hz), 121.4, 120.5 (d, *J*<sub>C-F</sub> = 0.6 Hz), 45.1; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -58.11; HRMS (EI) Calculated for C<sub>15</sub>H<sub>12</sub>F<sub>3</sub>NO<sub>2</sub> [M<sup>+</sup>]: 295.0820, found 295.0819; IR (KBr) ν(cm<sup>-1</sup>): 3408, 3295, 1665, 1607, 1532, 1456, 1264, 1168, 1104.

**2-(*m*-Tolyl)-N-[2-(trifluoromethoxy)phenyl]acetamide (1b):** White solid; mp 65–66 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.42 (d, *J* = 8.1 Hz, 1 H), 7.51 (br, 1 H), 7.31 (t, *J* = 7.5 Hz, 1 H), 7.29–7.23 (m, 1 H), 7.21–7.10 (m, 4 H), 7.08–7.03 (m, 1 H), 3.75 (s, 2 H), 2.37 (s, 3 H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 169.2, 139.3, 137.8 (d, *J*<sub>C-F</sub> = 1.3 Hz), 133.6, 130.6, 130.2, 129.3, 128.7, 127.5, 126.6, 124.1, 121.4, 120.5 (d, *J*<sub>C-F</sub> = 0.8 Hz), 120.2 (q, *J*<sub>C-F</sub> = 257.8 Hz), 45.0, 21.2; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -58.22; HRMS (EI) Calculated for C<sub>16</sub>H<sub>14</sub>F<sub>3</sub>NO<sub>2</sub> [M<sup>+</sup>]: 309.0977, found 309.0971; IR (KBr) ν(cm<sup>-1</sup>): 3405, 3301, 1697, 1677, 1610, 1531, 1490, 1453, 1313, 1254, 1176, 1106.

**2-(3-Methoxyphenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1c):** White solid; mp 91–92 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.41 (d, *J* = 8.2 Hz, 1 H), 7.54 (br s, 1 H), 7.34 (t, *J* = 7.9 Hz, 1 H), 7.26 (t, *J* = 7.8 Hz, 1 H), 7.16 (d, *J* = 8.2 Hz, 1 H), 7.09–7.03 (m, 1 H), 6.94–6.88 (m, 2 H), 6.86 (s, 1 H), 3.82 (s, 3 H), 3.76 (s, 2 H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 168.9, 160.4, 137.9, 135.2, 130.6, 127.5, 124.2, 121.7, 121.4, 120.5 (d, *J*<sub>C-F</sub> = 0.7 Hz), 120.2 (q, *J*<sub>C-F</sub> = 257.9 Hz), 115.1, 113.5, 100.0, 55.2, 45.1; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -58.16; HRMS (EI) Calculated for C<sub>16</sub>H<sub>14</sub>F<sub>3</sub>NO<sub>3</sub> [M<sup>+</sup>]: 325.0926, found 325.0947; IR (KBr) ν(cm<sup>-1</sup>): 3405, 3267, 1666, 1608, 1587, 1533, 1491, 1454, 1302, 1248.

**2-(*p*-Tolyl)-N-[2-(trifluoromethoxy)phenyl]acetamide (1d):** White solid; mp 91–92 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.41 (d, *J* = 8.0 Hz, 1 H), 7.49 (br s, 1 H), 7.29–7.18 (m, 5 H), 7.15 (d, *J* = 8.2 Hz, 1 H), 7.08–7.01 (m, 1 H), 3.74 (s, 2 H), 2.37 (s, 3 H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 169.4, 137.84, 137.81, 130.62, 130.59, 130.1, 129.4, 127.5, 124.1, 121.3, 120.4 (d, *J*<sub>C-F</sub> = 1.0 Hz), 120.2 (q, *J*<sub>C-F</sub> = 257.9 Hz), 44.6, 21.0; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -58.19; HRMS (EI)

Calculated for  $C_{16}H_{14}F_3NO_2$  [ $M^+$ ]: 309.0977, found 309.0981; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3405, 3268, 1669, 1609, 1536, 1454, 1265, 1210, 1188, 1166.

**2-(4-Isopropylphenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1e)**: White solid; mp 57–58 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.44 (d,  $J = 8.0$  Hz, 1 H), 7.52 (br s, 1 H), 7.45–7.20 (m, 5 H), 7.15 (d,  $J = 8.2$  Hz, 1 H), 7.08–7.02 (m, 1 H), 3.76 (s, 2 H), 2.99–2.87 (m, 1 H), 1.26 (d,  $J = 6.9$  Hz, 6 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) 169.4, 148.8, 137.78, 137.77, 130.9, 130.6, 129.5, 127.5, 124.0, 121.2, 120.5 (d,  $J_{\text{C-F}} = 0.8$  Hz), 120.2 (q,  $J_{\text{C-F}} = 257.8$  Hz), 44.6, 33.8, 23.8;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.12; HRMS (EI) Calculated for  $C_{18}H_{18}F_3NO_2$  [ $M^+$ ]: 337.1290, found 337.1287; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3406, 2963, 2931, 1698, 1680, 1610, 1530, 1453, 1253, 1176.

**2-[4-(tert-Butyl)phenyl]-N-[2-(trifluoromethoxy)phenyl] acetamide (1f)**: Yellow oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.45 (d,  $J = 8.2$  Hz, 1 H), 7.52 (br s, 1 H), 7.46 (d,  $J = 8.2$  Hz, 2 H), 7.32–7.22 (m, 3 H), 7.19–7.12 (m, 1 H), 7.09–7.02 (m, 1 H), 3.77 (s, 2 H), 1.34 (s, 9 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) 169.4, 151.1, 137.8, 130.63, 130.58, 129.3, 127.5, 126.4, 124.0, 121.2, 120.5 (d,  $J_{\text{C-F}} = 1.0$  Hz), 120.2 (q,  $J_{\text{C-F}} = 258.1$  Hz), 44.5, 34.5, 31.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.10; HRMS (EI) Calculated for  $C_{19}H_{20}F_3NO_2$  [ $M^+$ ]: 351.1446, found 351.1450; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3405, 3325, 3268, 1686, 1668, 1610, 1532, 1453, 1254.

**2-(4-Butoxyphenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1g)**: Yellow solid; mp 86–88 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.41 (d,  $J = 8.1$  Hz, 1 H), 7.51 (br s, 1 H), 7.28–7.20 (m, 3 H), 7.16 (d,  $J = 8.2$  Hz, 1 H), 7.08–7.02 (m, 1 H), 6.94 (d,  $J = 8.5$  Hz, 2 H), 3.98 (t,  $J = 6.5$  Hz, 2 H), 3.72 (s, 2 H), 1.82–1.73 (m, 2 H), 1.57–1.45 (m, 2 H), 0.98 (t,  $J = 7.4$  Hz, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.6, 159.0, 137.9 (d,  $J_{\text{C-F}} = 1.1$  Hz), 130.7, 130.6, 127.5, 125.4, 124.1, 121.3, 120.4, 120.3 (q,  $J_{\text{C-F}} = 257.9$  Hz), 115.5, 67.8, 44.2, 31.2, 19.2, 13.8;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.08; HRMS (EI) Calculated for  $C_{19}H_{20}F_3NO_3$  [ $M^+$ ]: 367.1395, found 367.1390; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3440, 3404, 3306, 1667, 1609, 1529, 1514, 1455, 1262, 1212, 1167.

**2-(4-Phenoxyphenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1h)**: White solid; mp 56–58 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.40 (d,  $J = 8.2$  Hz, 1 H), 7.51 (br s, 1 H), 7.34 (t,  $J = 7.5$  Hz, 2 H), 7.30–7.23 (m, 3 H), 7.17 (d,  $J = 7.7$  Hz, 1 H), 7.11 (t,  $J = 7.4$  Hz, 1 H), 7.08–6.99 (m, 4 H), 3.74 (m, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.1, 157.3, 156.7, 137.9, 131.0, 130.5, 129.8, 128.2, 127.6, 124.2, 123.6, 121.4, 120.5, 120.3 (q,  $J_{\text{C-F}} = 257.9$  Hz), 119.4, 119.2, 44.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.95; HRMS (EI) Calculated for  $C_{21}H_{15}F_4NO_3$  [ $M^+$ ]: 387.1082, found 387.1076; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3312, 1672, 1609, 1592, 1531, 1507, 1489, 1453, 1433, 1356, 1249, 1196, 1165, 1106, 1073, 875, 845, 759, 692.

**2-[(1,1'-Biphenyl)-4-yl]-N-[2-(trifluoromethoxy)phenyl] acetamide (1i)**: Yellow solid; mp 99–100 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.43 (d,  $J = 8.2$  Hz, 1 H), 7.64 (d,  $J = 8.0$  Hz, 2 H), 7.59 (d,  $J = 7.5$  Hz, 2 H), 7.54 (br, 1 H), 7.45 (t,  $J = 7.6$  Hz, 2 H), 7.42–7.34 (m, 3 H), 7.26 (t,  $J = 7.3$  Hz, 1 H), 7.16 (d,  $J = 8.2$  Hz, 1 H), 7.09–7.02 (m, 1 H), 3.83 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.0, 141.1, 140.5, 137.9, 132.7, 130.5, 130.0, 128.8, 128.2, 127.6, 127.5, 127.1, 124.2, 121.4, 120.5, 120.2 (q,  $J_{\text{C-F}} = 257.9$  Hz), 44.6;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.05; HRMS (EI) Calculated for  $C_{21}H_{16}F_3NO_2$  [ $M^+$ ]: 371.1133, found 371.1131; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3405, 3293, 3283, 3197, 1665, 1609, 1534, 1489, 1454, 1259, 1188, 1170.

**2-(3,5-Dimethylphenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1j)**: White solid; mp 97–98 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.42 (d,  $J = 8.2$  Hz, 1 H), 7.53 (br s, 1 H), 7.26 (t,  $J = 7.8$  Hz, 1 H), 7.15 (d,  $J = 8.2$  Hz, 1 H), 7.08–7.02 (m, 1 H), 7.00 (s, 1 H), 6.94 (s, 2 H), 3.71 (s, 2 H), 2.33 (s, 6 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.3, 139.2, 137.8, 133.5, 130.7, 129.5, 127.5, 127.3, 124.0, 121.3, 120.5, 120.2 (q,  $J_{\text{C-F}} = 257.6$  Hz), 45.0, 21.1;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.30; HRMS (EI) Calculated for  $C_{17}H_{16}F_3NO_2$  [ $M^+$ ]: 323.1133, found 323.1140; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3402, 3295, 1668, 1608, 1532, 1450,

1356, 1290, 1261, 1209, 1174.

**2-(3,5-Dimethoxyphenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1k)**: White solid; mp 114–116 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.41 (d,  $J = 8.0$  Hz, 1 H), 7.61 (br s, 1 H), 7.26 (t,  $J = 7.8$  Hz, 1 H), 7.16 (d,  $J = 8.2$  Hz, 1 H), 7.09–7.01 (m, 1 H), 6.48–6.44 (m, 3 H), 3.80 (s, 6 H), 3.71 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.8, 161.7, 137.9, 135.8, 130.6, 127.6, 124.2, 121.3, 120.5, 120.3 (d,  $J_{\text{C-F}} = 258.0$  Hz), 107.5, 99.8, 55.3, 45.3;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.21; HRMS (EI) Calculated for  $C_{17}H_{16}F_3NO_4$  [ $M^+$ ]: 355.1031, found 355.1036; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3441, 3403, 3263, 1665, 1608, 1596, 1536, 1457, 1318, 1299, 1267, 1212, 1190, 1158.

**2-(3-Fluoro-4-methoxyphenyl)-N-[2-(trifluoromethoxy)phenyl]acetamide (1l)**: White solid; mp 91–92 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.39 (d,  $J = 8.2$  Hz, 1 H), 7.48 (br s, 1 H), 7.26 (t,  $J = 7.8$  Hz, 1 H), 7.17 (d,  $J = 8.1$  Hz, 1 H), 7.10–6.97 (m, 4 H), 3.90 (s, 3 H), 3.70 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) 168.7, 152.6 (d,  $J_{\text{C-F}} = 246.5$  Hz), 147.5 (d,  $J_{\text{C-F}} = 10.5$  Hz), 137.9, 130.4, 127.6, 126.5 (d,  $J_{\text{C-F}} = 6.3$  Hz), 125.4 (d,  $J_{\text{C-F}} = 3.6$  Hz), 124.3, 121.4, 120.5, 120.3 (q,  $J_{\text{C-F}} = 257.9$  Hz), 117.3 (d,  $J_{\text{C-F}} = 18.5$  Hz), 114.2 (d,  $J_{\text{C-F}} = 2.3$  Hz), 56.3, 43.9;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.09, -133.54; HRMS (EI) Calculated for  $C_{16}H_{13}F_4NO_3$  [ $M^+$ ]: 343.0832, found 343.0836; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3268, 1659, 1609, 1519, 1455, 1274, 1207, 1190, 1170.

**N-[2-(Trifluoromethoxy)phenyl]-2-(3,4,5-trimethoxyphenyl) acetamide (1m)**: White solid; mp 97–101 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.41 (d,  $J = 8.1$  Hz, 1 H), 7.64 (br s, 1 H), 7.26 (t,  $J = 8.0$  Hz, 1 H), 7.17 (d,  $J = 8.1$  Hz, 1 H), 7.06 (t,  $J = 7.6$  Hz, 1 H), 6.54 (s, 2 H), 3.86 (s, 6 H), 3.85 (s, 3 H), 3.72 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.9, 153.9, 137.7, 137.6, 130.5, 129.2, 127.5, 124.1, 121.3, 120.5, 120.2 (q,  $J_{\text{C-F}} = 257.7$  Hz), 106.3, 60.6, 55.9, 45.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.11; HRMS (EI) Calculated for  $C_{18}H_{18}F_3NO_5$  [ $M^+$ ]: 385.1137, found 385.1141; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3289, 1661, 1608, 1593, 1537, 1509, 1460, 1424, 1329, 1295, 1256, 1187, 1172, 1150, 1128.

**2-(Naphthalen-2-yl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1n)**: Yellow solid; mp 104–105 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.41 (d,  $J = 8.2$  Hz, 1 H), 7.91 (d,  $J = 8.4$  Hz, 1 H), 7.89–7.80 (m, 3 H), 7.57–7.48 (m, 3 H), 7.43 (dd,  $J = 8.4$  and 1.5 Hz, 1 H), 7.30–7.23 (m, 1 H), 7.12 (d,  $J = 8.2$  Hz, 1 H), 7.09–7.01 (m, 1 H), 3.95 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.0, 137.9, 133.6, 132.7, 131.2, 130.5, 129.3, 128.5, 127.7, 127.6, 127.5, 127.0, 126.6, 126.3, 124.2, 121.5, 120.4120.1 (q,  $J_{\text{C-F}} = 257.9$  Hz), 45.1;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.24; HRMS (EI) Calculated for  $C_{19}H_{14}F_3NO_2$  [ $M^+$ ]: 345.0977, found 345.0970; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3263, 1664, 1606, 1533, 1494, 1454, 1407, 1341, 1302, 1265, 1190, 1172.

**2-(Thiophen-3-yl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1o)**: White solid; mp 50–51 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.42 (d,  $J = 8.1$  Hz, 1 H), 7.62 (br s, 1 H), 7.43 (dd,  $J = 4.7$  and 3.0 Hz, 1 H), 7.26 (t,  $J = 7.5$  Hz, 2 H), 7.17 (d,  $J = 8.2$  Hz, 1 H), 7.10–7.03 (m, 2 H), 3.82 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.6, 137.9, 133.7, 130.5, 128.2, 127.6, 127.5, 124.3, 124.2, 121.4, 120.4, 120.3 (q,  $J_{\text{C-F}} = 257.9$  Hz), 39.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.05; HRMS (EI) Calculated for  $C_{13}H_{10}F_3NO_2S$  [ $M^+$ ]: 301.0384, found 301.0381; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3401, 3280, 1675, 1610, 1532, 1454, 1256, 1174.

**2-(2-Bromophenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1p)**: White solid; mp 141–142 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.42 (d,  $J = 8.2$  Hz, 1 H), 7.65 (d,  $J = 8.0$  Hz, 1 H), 7.54 (br s, 1 H), 7.45–7.40 (m, 1 H), 7.37 (t,  $J = 7.4$  Hz, 1 H), 7.31–7.21 (m, 2 H), 7.21–7.16 (m, 1 H), 7.10–7.04 (m, 1 H), 3.94 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.7, 137.9, 133.9, 133.4, 131.7, 130.5, 129.6, 128.3, 127.5, 125.0, 124.2, 121.5, 120.3, 120.28 (q,  $J_{\text{C-F}} = 257.9$  Hz), 45.3;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.92; HRMS (EI) Calculated for  $C_{15}H_{11}BrF_3NO_2$  [ $M^+$ ]: 372.9925, found 372.9932; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3416, 3256, 1667, 1607, 1535, 1454, 1409, 1336, 1297, 1260, 1178.

**2-(4-Chlorophenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1q)**: White solid; mp 90–91 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.16 (d,  $J = 8.0$  Hz, 1 H), 7.26 (br s, 1 H), 7.17 (d,  $J = 8.1$  Hz, 2 H),

7.09–7.02 (m, 3 H), 6.97 (d,  $J = 7.9$  Hz, 1 H), 6.86 (t,  $J = 7.6$  Hz, 1 H), 3.54 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.4, 138.0, 134.0, 132.2, 130.8, 130.3, 129.4, 127.5, 124.4, 121.5, 120.4, 120.3 (q,  $J_{\text{C-F}} = 258.0$  Hz), 44.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.03; HRMS (EI) Calculated for  $\text{C}_{15}\text{H}_{11}\text{ClF}_3\text{NO}_2$  [ $\text{M}^+$ ]: 329.0430, found 329.0437; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3265, 1668, 1610, 1600, 1540, 1493, 1455, 1410, 1343, 1302, 1268, 1212, 1193.

**2-(3-Bromophenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1r)**: White solid; mp 111–112 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.29 (d,  $J = 8.1$  Hz, 1 H), 7.54–7.31 (m, 3 H), 7.26–7.14 (m, 3 H), 7.10 (d,  $J = 8.2$  Hz, 1 H), 6.99 (t,  $J = 7.4$  Hz, 1 H), 3.66 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.2, 138.0, 136.0, 132.5, 131.0, 130.7, 130.3, 128.0, 127.5, 124.4, 123.3, 121.6, 120.5, 120.3 (q,  $J_{\text{C-F}} = 258.0$  Hz), 44.3;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.00; HRMS (EI) Calculated for  $\text{C}_{15}\text{H}_{11}\text{BrF}_3\text{NO}_2$  [ $\text{M}^+$ ]: 372.9925, found 372.9926; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3295, 3279, 1671, 1610, 1541, 1454, 1345, 1303, 1257, 1192, 1164.

**2-(4-Iodophenyl)-N-[2-(trifluoromethoxy)phenyl]acetamide (1s)**: White solid; mp 134–135 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.35 (d,  $J = 8.1$  Hz, 1 H), 7.72 (d,  $J = 8.2$  Hz, 2 H), 7.51 (br s, 1 H), 7.24 (t,  $J = 7.8$  Hz, 1 H), 7.17 (d,  $J = 8.2$  Hz, 1 H), 7.07 (d,  $J = 8.1$  Hz, 3 H), 3.70 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.3, 138.3, 138.0, 133.4, 131.3, 130.3, 127.5, 124.4, 121.6, 120.4, 120.2 (q,  $J_{\text{C-F}} = 257.9$  Hz), 93.3, 44.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.98; HRMS (EI) Calculated for  $\text{C}_{15}\text{H}_{11}\text{F}_3\text{INO}_2$  [ $\text{M}^+$ ]: 420.9787, found 420.9795; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3261, 1675, 1662, 1609, 1535, 1487, 1455, 1257, 1214, 1194.

**2-(3-Fluorophenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1t)**: White solid; mp 96–97 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.36 (d,  $J = 8.1$  Hz, 1 H), 7.42 (br s, 1 H), 7.40–7.33 (m, 1 H), 7.25–7.21 (m, 1 H), 7.15 (d,  $J = 8.2$  Hz, 1 H), 7.10 (d,  $J = 7.6$  Hz, 1 H), 7.08–6.99 (m, 3 H), 3.75 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) 168.2, 163.2 (d,  $J_{\text{C-F}} = 246.6$  Hz), 137.9, 136.1 (d,  $J_{\text{C-F}} = 7.4$  Hz), 131.0 (d,  $J_{\text{C-F}} = 8.4$  Hz), 130.4, 127.6, 125.2 (d,  $J_{\text{C-F}} = 3.0$  Hz), 124.4, 120.3 (q,  $J_{\text{C-F}} = 257.9$  Hz), 121.5, 120.5, 116.6 (d,  $J_{\text{C-F}} = 21.5$  Hz), 115.0 (d,  $J_{\text{C-F}} = 20.9$  Hz), 44.6;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.10, –111.71; HRMS (EI) Calculated for  $\text{C}_{15}\text{H}_{11}\text{F}_4\text{NO}_2$  [ $\text{M}^+$ ]: 313.0726, found 313.0732; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3413, 3277, 1669, 1644, 1611, 1593, 1535, 1490, 1454, 1347, 1304, 1261, 1215, 1188, 1172, 1144, 1106, 763  $\text{cm}^{-1}$ .

**2-(3-Bromophenyl)-N-[2-(trifluoromethoxy)phenyl] acetamide (1u)**: White solid; mp 111–112 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.29 (d,  $J = 8.1$  Hz, 1 H), 7.54–7.31 (m, 3 H), 7.26–7.14 (m, 3 H), 7.10 (d,  $J = 8.2$  Hz, 1 H), 6.99 (t,  $J = 7.4$  Hz, 1 H), 3.66 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.2, 138.0, 136.0, 132.5, 131.0, 130.7, 130.3, 128.0, 127.5, 124.4, 123.3, 121.6, 120.5, 120.3 (q,  $J_{\text{C-F}} = 258.0$  Hz), 44.3;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.00; HRMS (EI) Calculated for  $\text{C}_{15}\text{H}_{11}\text{BrF}_3\text{NO}_2$  [ $\text{M}^+$ ]: 372.9925, found 372.9926; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3295, 3279, 1671, 1610, 1541, 1454, 1345, 1303, 1257, 1192, 1164.

**N-[2-(Trifluoromethoxy)phenyl]-2-[4-(trifluoromethoxy)phenyl]acetamide (1v)**: White solid; mp 104–105 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.36 (d,  $J = 8.1$  Hz, 1 H), 7.52 (br s, 1 H), 7.43 (t,  $J = 8.3$  Hz, 1 H), 7.26 (t,  $J = 7.3$  Hz, 2 H), 7.23–7.15 (m, 3 H), 7.06 (t,  $J = 7.3$  Hz, 1 H), 3.78 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.1, 149.8 (d,  $J_{\text{C-F}} = 1.8$  Hz), 138.1, 136.0, 130.6, 130.3, 127.8, 127.5, 124.5, 122.0, 121.7, 120.5, 120.1, 120.38 (q,  $J_{\text{C-F}} = 256.0$  Hz), 120.27 (q,  $J_{\text{C-F}} = 257.8$  Hz), 44.4;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.89, –58.17; HRMS (EI) Calculated for  $\text{C}_{16}\text{H}_{11}\text{F}_6\text{NO}_3$  [ $\text{M}^+$ ]: 379.0643, found 379.0634; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3295, 3281, 1670, 1611, 1542, 1491, 1456, 1281.

**N-[2-(Trifluoromethoxy)phenyl]-2-[3-((trifluoromethyl)thio)phenyl]acetamide (1w)**: White solid; mp 90–91 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.39 (d,  $J = 8.1$  Hz, 1 H), 7.74–7.58 (m, 2 H), 7.49 (d,  $J = 4.8$  Hz, 2 H), 7.39 (br s, 1 H), 7.28 (t,  $J = 7.5$  Hz, 1 H), 7.19 (d,  $J = 8.2$  Hz, 1 H), 7.09 (t,  $J = 7.5$  Hz, 1 H), 3.82 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.0, 137.2, 135.6, 135.4, 131.9, 130.3, 130.2, 129.4 (q,  $J_{\text{C-F}} = 306.3$  Hz), 127.6, 124.5, 121.6, 120.5, 120.3 (q,  $J_{\text{C-F}} = 258.6$  Hz), 44.4;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –42.55, –58.07;

HRMS (EI) Calculated for  $\text{C}_{16}\text{H}_{11}\text{F}_6\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 395.0415, found 395.0404; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3416, 3295, 1669, 1610, 1534, 1456, 1357, 1300, 1263, 1211, 1185, 1167, 1149.

**N-(2-(difluoromethoxy)phenyl)-2-(3-(trifluoromethoxy)phenyl)acetamide (1x)**: White solid; mp 84–85 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.37 (d,  $J = 8.1$  Hz, 1H), 7.56 (s, 1H), 7.44 (t,  $J = 8.3$  Hz, 1H), 7.29 (d,  $J = 7.6$  Hz, 1H), 7.24–7.15 (m, 3H), 7.09–7.01 (m, 2H), 6.33 (t,  $J = 73.3$  Hz, 1H), 3.79 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.0, 149.7, 140.0, 136.2, 130.6, 129.8, 127.9, 126.3, 124.4, 122.1, 121.3, 120.4 (q,  $J_{\text{C-F}} = 255.9$  Hz), 120.1, 118.6, 116.1 (t,  $J_{\text{C-F}} = 258.9$  Hz), 44.5;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.81, –80.26; HRMS (EI) Calculated for  $\text{C}_{16}\text{H}_{12}\text{F}_5\text{NO}_3$  [ $\text{M}^+$ ]: 361.0737, found 361.0742; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3262, 1670, 1607, 1540, 1454, 1381, 1348, 1280, 1216, 1151, 1123.

**N-(2-(difluoromethoxy)phenyl)-2-phenylacetamide (1ab)**: White solid; mp 60–61 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.38 (d,  $J = 7.7$  Hz, 1 H), 7.60 (br s, 1 H), 7.52–7.28 (m, 5 H), 7.22–7.16 (m, 1 H), 7.04 (d,  $J = 3.6$  Hz, 2 H), 6.27 (t,  $J = 73.4$  Hz, 1 H), 3.78 (s, 2 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.1, 139.9, 134.0, 130.0, 129.6, 129.3, 127.8, 126.2, 124.2, 121.1, 118.7, 116.0 (t,  $J_{\text{C-F}} = 259.9$  Hz), 45.1;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –80.24; HRMS (EI) Calculated for  $\text{C}_{15}\text{H}_{13}\text{F}_2\text{NO}_2$  [ $\text{M}^+$ ]: 277.0914, found 277.0906; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3399, 3294, 1673, 1608, 1530, 1496, 1455, 1383, 1349, 1321, 1295, 1260, 1191, 1131, 1100, 1054, 1041, 757, 727, 706.

**2-Phenyl-N-(4-(trifluoromethoxy)phenyl)acetamide (1ac)**: White solid; mp 136–138 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49–7.41 (m, 3H), 7.40–7.28 (m, 5H), 7.12 (d,  $J = 8.6$  Hz, 2H), 3.72 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.3, 145.3, 136.2, 134.1, 129.4, 129.2, 127.8, 121.6, 121.0, 120.4 (q,  $J_{\text{C-F}} = 255.2$  Hz), 44.6;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.16; HRMS (EI) Calculated for  $\text{C}_{15}\text{H}_{12}\text{F}_3\text{NO}_2$  [ $\text{M}^+$ ]: 295.0820, found 295.0821; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3265, 1657, 1611, 1556, 1509, 1408, 1294, 1204, 1164.

**2-(p-Tolyl)-N-[3-(trifluoromethoxy)phenyl]acetamide (1ba)**: White solid; mp 112–113 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) 7.64 (s, 1 H), 7.53 (s, 1 H), 7.35–7.18 (m, 6 H), 6.97 (d,  $J = 6.9$  Hz, 1 H), 3.70 (s, 2 H), 2.39 (s, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.8, 149.4, 139.1, 137.5, 130.9, 129.9, 129.8, 129.3, 120.4 (q,  $J_{\text{C-F}} = 255.9$  Hz), 117.8, 116.4, 112.6, 44.2, 21.0;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.17; HRMS (EI) Calculated for  $\text{C}_{16}\text{H}_{14}\text{F}_3\text{NO}_2$  [ $\text{M}^+$ ]: 309.0977, found 309.0973; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3429, 1664, 1639, 1609, 1539, 1493, 1441, 1262, 1221, 1168.

**N-[2-(Methylthio)phenyl]-2-(p-tolyl)acetamide (1bb)**: White solid; mp 85–86 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.35 (d,  $J = 7.6$  Hz, 2 H), 7.48–7.33 (m, 1 H), 7.29–7.17 (m, 5 H), 6.99 (t,  $J = 7.4$  Hz, 1 H), 3.74 (s, 2 H), 2.35 (s, 3 H), 2.02 (s, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.4, 138.4, 137.2, 133.3, 131.1, 129.8, 129.5, 129.0, 124.9, 124.1, 120.0, 44.8, 21.0, 18.5; HRMS (EI) Calculated for  $\text{C}_{16}\text{H}_{17}\text{NOS}$  [ $\text{M}^+$ ]: 271.1031, found 271.1025; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3431, 3247, 1661, 1577, 1527, 1517, 1437, 1341, 1296, 1275, 1186.

**N-Phenyl-2-(p-tolyl)acetamide (1bc)**: White solid; mp 145–146 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) 7.41 (d,  $J = 7.7$  Hz, 2 H), 7.31–7.18 (m, 6 H), 7.08 (t,  $J = 7.3$  Hz, 2 H), 3.70 (s, 2 H), 2.37 (s, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.3, 137.6, 137.4, 131.3, 129.9, 129.4, 128.9, 124.4, 119.7, 44.4, 21.1; HRMS (EI) Calculated for  $\text{C}_{15}\text{H}_{15}\text{NO}$  [ $\text{M}^+$ ]: 225.1154, found 225.1148; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3440, 3432, 3298, 1670, 1598, 1530, 1499, 1442, 1410, 1343, 1307, 1298, 1254, 1188.

#### 4.3. General procedure for the synthesis of $\alpha,\beta$ -unsaturated amide compounds 2 and sulfide compounds 3

A mixture of substrate **1** (0.1 mmol),  $\text{PbCl}_2$  (41.7 mg, 0.15 mmol) and KOAc (14.7 mg, 0.15 mmol) or (5.9 mg, 0.06 mmol) in DMSO (2.0 mL) was put into a 15 mL seal tube. The air in the seal tube was exchanged with  $\text{N}_2$ . The reaction mixture was stirred at 156 °C for



13 h under N<sub>2</sub>, then cooled down to room temperature, diluted with H<sub>2</sub>O, and extracted with EtOAc (3 × 5 mL). The combined organic phase was washed with brine and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated under vacuum. The residue was purified by preparative TLC plate (petroleum ether: ethyl acetate = 10: 1 as eluent) to get the product **2** and **3**.

**2-Phenyl-N-(2-(trifluoromethoxy)phenyl)acrylamide (2a), 3-(Methylthio)-2-phenyl-N-(2-(trifluoromethoxy)phenyl) propanamide (3a).** Data of **2a**: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.55 (d, *J* = 7.8 Hz, 1H), 7.83 (s, 1H), 7.51–7.38 (m, 5H), 7.34–7.28 (m, 1H), 7.24–7.16 (m, 1H), 7.13–7.06 (m, 1H), 6.43 (s, 1H), 5.75 (s, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.4, 144.5, 138.1 (d, *J* = 1.3 Hz), 136.3, 130.5, 128.9, 128.8, 128.3, 127.5, 124.7, 124.3, 121.5, 120.2 (q, *J*<sub>C-F</sub> = 257.8 Hz), 120.3 (d, *J* = 1.0 Hz); <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –57.99; HRMS (EI) Calcd for C<sub>16</sub>H<sub>12</sub>F<sub>3</sub>NO<sub>2</sub> [M<sup>+</sup>]: 307.0820, found 307.0828; IR (KBr) ν(cm<sup>-1</sup>): 3416, 1691, 1611, 1528, 1452, 1257, 1216, 1180. Data of **3a**: White solid; mp 74–75 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.39 (d, *J* = 8.2 Hz, 1H), 7.54 (s, 1H), 7.44–7.32 (m, 5H), 7.29–7.23 (m, 1H), 7.20–7.15 (m, 1H), 7.10–7.04 (m, 1H), 3.82 (t, *J* = 7.4 Hz, 1H), 3.44 (dd, *J* = 13.3 and 7.1 Hz, 1H), 2.99 (dd, *J* = 13.3 and 7.7 Hz, 1H), 2.10 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.3, 138.0, 137.6, 130.4, 129.3, 128.3, 128.0, 127.5, 121.6, 120.4 (d, *J* = 1.0 Hz), 120.3 (q, *J*<sub>C-F</sub> = 258.1 Hz), 54.7, 36.9, 16.3; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –57.92; HRMS (EI) Calcd for C<sub>17</sub>H<sub>16</sub>F<sub>3</sub>NO<sub>2</sub>S [M<sup>+</sup>]: 355.0854, found 355.0848; IR (KBr) ν(cm<sup>-1</sup>): 3420, 1692, 1670, 1610, 1528, 1452, 1258, 1217, 1180.

**2-(*m*-Tolyl)-N-(2-(trifluoromethoxy)phenyl)acrylamide (2b), 3-(Methylthio)-2-(*m*-tolyl)-N-(2-(trifluoromethoxy) phenyl) propanamide (3b).** Data of **2b**: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.52 (d, *J* = 8.1 Hz, 1H), 7.83 (s, 1H), 7.34–7.25 (m, 2H), 7.25–7.11 (m, 4H), 7.09–7.03 (m, 1H), 6.40 (s, 1H), 5.69 (d, *J* = 0.6 Hz, 1H), 2.36 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.5, 144.6, 138.9, 138.1 (d, *J* = 1.2 Hz), 136.3, 130.7, 129.7, 129.01, 128.95, 127.6, 125.5, 124.8, 124.3, 121.6, 120.4 (d, *J* = 1.0 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.9 Hz), 21.3; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –58.07; HRMS (EI) Calcd for C<sub>17</sub>H<sub>14</sub>F<sub>3</sub>NO<sub>2</sub> [M<sup>+</sup>]: 321.0977, found 321.0982; IR (KBr) ν(cm<sup>-1</sup>): 3414, 1691, 1611, 1527, 1452, 1255, 1216, 1178. Data of **3b**: White solid; mp 81–82 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.39 (d, *J* = 8.1 Hz, 1H), 7.54 (s, 1H), 7.33–7.22 (m, 2H), 7.21–7.13 (m, 4H), 7.10–7.03 (m, 1H), 3.79 (t, *J* = 7.4 Hz, 1H), 3.43 (dd, *J* = 13.3 and 7.0 Hz, 1H), 2.98 (dd, *J* = 13.2 and 7.9 Hz, 1H), 2.36 (s, 3H), 2.11 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.4, 139.2, 137.9, 137.5, 130.5, 129.2, 129.1, 128.7, 127.5, 125.1, 124.2, 121.6, 120.4 (d, *J* = 0.9 Hz), 120.3 (q, *J*<sub>C-F</sub> = 258.7 Hz), 54.6, 36.8, 21.4, 16.3; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –58.00; HRMS (EI) Calcd for C<sub>18</sub>H<sub>18</sub>F<sub>3</sub>NO<sub>2</sub>S [M<sup>+</sup>]: 369.1010, found 369.1014; IR (KBr) ν(cm<sup>-1</sup>): 3409, 1692, 1609, 1528, 1257, 1217, 1180.

**2-(3-Methoxyphenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2c) and 2-(3-Methoxyphenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3c).** Data of **2c**: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.55 (d, *J* = 8.1 Hz, 1H), 7.89 (s, 1H), 7.37 (t, *J* = 7.9 Hz, 1H), 7.34–7.28 (m, 1H), 7.20 (d, *J* = 8.2 Hz, 1H), 7.12–7.06 (m, 1H), 7.03–6.92 (m, 3H), 6.44 (s, 1H), 5.75 (d, *J* = 0.6 Hz, 1H), 3.83 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.3, 160.0, 144.4, 138.1 (d, *J* = 1.4 Hz), 137.7, 130.6, 130.2, 127.6, 125.0, 124.3, 121.5, 120.6, 120.4 (d, *J* = 1.1 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.8 Hz), 114.5, 114.0, 55.2; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –58.03; HRMS (EI) Calcd for C<sub>17</sub>H<sub>14</sub>F<sub>3</sub>NO<sub>3</sub> [M<sup>+</sup>]: 337.0926, found 337.0929; IR (KBr) ν(cm<sup>-1</sup>): 3411, 1691, 1606, 1527, 1453, 1255, 1217, 1180. Data of **3c**: White solid; mp 81–82 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.38 (d, *J* = 8.0 Hz, 1H), 7.57 (s, 1H), 7.32 (t, *J* = 7.8 Hz, 1H), 7.29–7.22 (m, 1H), 7.21–7.14 (m, 1H), 7.10–7.03 (m, 1H), 6.95 (d, *J* = 7.5 Hz, 1H), 6.92–6.84 (m, 2H), 3.83–3.77 (m, 4H), 3.42 (dd, *J* = 13.3 and 7.0 Hz, 1H), 2.98 (dd, *J* = 13.3 and 7.8 Hz, 1H), 2.11 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.2, 160.3, 139.1, 138.0, 130.44, 130.38, 127.5, 124.3, 121.6, 120.4 (d, *J* = 1.1 Hz), 120.3 (q, *J*<sub>C-F</sub> = 258.3 Hz), 120.2,

113.7, 113.6, 55.2, 54.7, 36.7, 16.3; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –57.95; HRMS (EI) Calcd for C<sub>18</sub>H<sub>18</sub>F<sub>3</sub>NO<sub>3</sub>S [M<sup>+</sup>]: 385.0960, found 385.0959; IR (KBr) ν(cm<sup>-1</sup>): 3352, 1670, 1608, 1529, 1255, 1215, 1185.

**2-(*p*-Tolyl)-N-(2-(trifluoromethoxy)phenyl)acrylamide (2d) and 3-(Methylthio)-2-(*p*-tolyl)-N-(2-(trifluoromethoxy)phenyl)propanamide (3d).** Data of **2d**: White solid; mp 61–62 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.54 (d, *J* = 8.1 Hz, 1H), 7.86 (s, 1H), 7.35–7.28 (m, 3H), 7.25 (d, *J* = 7.8 Hz, 2H), 7.22–7.16 (m, 1H), 7.12–7.06 (m, 1H), 6.38 (s, 1H), 5.72 (s, 1H), 2.40 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.7, 144.5, 139.0, 138.2, 133.4, 130.6, 129.7, 128.3, 127.6, 124.29, 124.25, 121.6, 120.4 (d, *J* = 1.1 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.4 Hz), 21.2; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –57.99; HRMS (EI) Calcd for C<sub>17</sub>H<sub>14</sub>F<sub>3</sub>NO<sub>2</sub> [M<sup>+</sup>]: 321.0977, found 321.0968; IR (KBr) ν(cm<sup>-1</sup>): 3419, 1689, 1614, 1527, 1256, 1217, 1180. Data of **3d**: White solid; mp 80–81 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.38 (d, *J* = 8.1 Hz, 1H), 7.54 (s, 1H), 7.31–7.14 (m, 6H), 7.10–7.02 (m, 1H), 3.79 (t, *J* = 7.4 Hz, 1H), 3.42 (dd, *J* = 13.2 and 6.8 Hz, 1H), 2.98 (dd, *J* = 13.2 and 8.1 Hz, 1H), 2.35 (s, 3H), 2.10 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.6, 138.2, 134.5, 130.5, 130.0, 128.7, 127.9, 127.5, 124.2, 121.5, 120.4, 120.3 (q, *J*<sub>C-F</sub> = 257.4 Hz), 54.2, 36.8, 21.1, 16.3; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –57.96; HRMS (EI) Calcd for C<sub>18</sub>H<sub>18</sub>F<sub>3</sub>NO<sub>2</sub>S [M<sup>+</sup>]: 369.1010, found 369.1020; IR (KBr) ν(cm<sup>-1</sup>): 3409, 1660, 1609, 1529, 1455, 1253, 1216, 1186.

**2-(4-Isopropylphenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2e) and 2-(4-Isopropylphenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3e).** Data of **2e**: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.57 (d, *J* = 7.6 Hz, 1H), 7.88 (s, 1H), 7.38–7.28 (m, 5H), 7.22–7.16 (m, 1H), 7.12–7.06 (m, 1H), 6.41 (d, *J* = 0.8 Hz, 1H), 5.72 (d, *J* = 0.9 Hz, 1H), 3.01–2.91 (m, 1H), 1.28 (d, *J* = 6.9 Hz, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.7, 149.9, 144.5, 138.1 (d, *J* = 1.5 Hz), 133.8, 130.7, 128.5, 127.6, 127.1, 124.5, 124.2, 121.4, 120.4 (d, *J* = 1.0 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.9 Hz), 34.0, 23.8; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –57.96; HRMS (EI) Calcd for C<sub>19</sub>H<sub>18</sub>F<sub>3</sub>NO<sub>2</sub> [M<sup>+</sup>]: 349.1290, found 349.1293; IR (KBr) ν(cm<sup>-1</sup>): 3401, 1699, 1609, 1531, 1455, 1255, 1217, 1180. Data of **3e**: White solid; mp 85–86 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.40 (d, *J* = 8.0 Hz, 1H), 7.53 (s, 1H), 7.31–7.22 (m, 5H), 7.20–7.13 (m, 1H), 7.10–7.02 (m, 1H), 3.80 (t, *J* = 7.4 Hz, 1H), 3.43 (dd, *J* = 13.2 and 6.9 Hz, 1H), 2.98 (dd, *J* = 13.2 and 7.9 Hz, 1H), 2.95–2.85 (m, 1H), 2.11 (s, 3H), 1.24 (d, *J* = 6.9 Hz, 6H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ 170.6, 149.0, 137.9, 134.7, 130.5, 128.0, 127.5, 127.4, 124.2, 121.5, 120.4, 120.2 (q, *J*<sub>C-F</sub> = 258.0 Hz), 54.2, 36.8, 33.8, 23.8, 23.8, 16.4. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –57.92; HRMS (EI) Calcd for C<sub>20</sub>H<sub>22</sub>F<sub>3</sub>NO<sub>2</sub>S [M<sup>+</sup>]: 397.1323, found 397.1324; IR (KBr) ν(cm<sup>-1</sup>): 3432, 1681, 1634, 1528, 1252, 1215, 1175.

**2-(4-(*Tert*-butyl)phenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2f) and 2-(4-(*Tert*-butyl)phenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3f).** Data of **2f**: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.58 (d, *J* = 7.9 Hz, 1H), 7.89 (s, 1H), 7.48 (d, *J* = 8.2 Hz, 2H), 7.36 (d, *J* = 8.3 Hz, 2H), 7.34–7.28 (m, 1H), 7.19 (d, *J* = 8.2 Hz, 1H), 7.14–7.05 (m, 1H), 6.42 (s, 1H), 5.73 (s, 1H), 1.35 (s, 9H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.6, 152.1, 144.4, 138.1 (d, *J* = 1.2 Hz), 133.4, 130.7, 128.2, 127.6, 126.0, 124.5, 124.2, 121.4, 120.3 (d, *J* = 1.0 Hz), 120.2 (q, *J*<sub>C-F</sub> = 258.0 Hz), 34.7, 31.2; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –57.94; HRMS (EI) Calcd for C<sub>20</sub>H<sub>20</sub>F<sub>3</sub>NO<sub>2</sub> [M<sup>+</sup>]: 363.1446, found 363.1442; IR (KBr) ν(cm<sup>-1</sup>): 3411, 1695, 1610, 1529, 1454, 1255, 1216, 1179. Data of **3f**: White solid; mp 84–85 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.40 (d, *J* = 8.1 Hz, 1H), 7.54 (s, 1H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.31–7.23 (m, 3H), 7.20–7.13 (m, 1H), 7.10–7.01 (m, 1H), 3.81 (t, *J* = 7.4 Hz, 1H), 3.43 (dd, *J* = 13.2 and 6.9 Hz, 1H), 2.99 (dd, *J* = 13.2 and 7.9 Hz, 1H), 2.11 (s, 3H), 1.31 (s, 9H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ 170.6, 151.3, 137.9, 134.4, 130.5, 127.7, 127.5, 126.3, 124.2, 121.5, 120.4, 120.2 (q, *J*<sub>C-F</sub> = 257.8 Hz), 54.2, 36.8, 34.6, 31.2, 16.4; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ –57.90; HRMS (EI) Calcd for C<sub>21</sub>H<sub>24</sub>F<sub>3</sub>NO<sub>2</sub>S [M<sup>+</sup>]: 411.1480, found 411.1479; IR (KBr) ν(cm<sup>-1</sup>):

3426, 1686, 1617, 1528, 1257, 1217, 1178.

**2-(4-Butoxyphenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2g) and 2-(4-Butoxyphenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3g).** Data of **2g**: White solid; mp 64–66 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.54 (d, *J* = 8.1 Hz, 1H), 7.89 (s, 1H), 7.38–7.28 (m, 3H), 7.21 (d, *J* = 8.1 Hz, 1H), 7.13–7.05 (m, 1H), 6.95 (d, *J* = 8.5 Hz, 2H), 6.32 (s, 1H), 5.69 (s, 1H), 4.00 (t, *J* = 7.4 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.1, 159.7, 144.2, 138.2 (d, *J* = 1.3 Hz), 130.7, 129.6, 128.3, 127.6, 124.3, 123.5, 121.6, 120.4 (d, *J* = 1.0 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.8 Hz), 115.0, 67.8, 31.2, 19.2, 13.8; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.87; HRMS (EI) Calcd for C<sub>20</sub>H<sub>20</sub>F<sub>3</sub>NO<sub>3</sub> [M<sup>+</sup>]: 379.1395, found 379.1401; IR (KBr) ν(cm<sup>-1</sup>): 3415, 1690, 1611, 1525, 1453, 1255, 1217, 1178. Data of **3g**: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.38 (d, *J* = 8.1 Hz, 1H), 7.54 (s, 1H), 7.29–7.23 (m, 3H), 7.22–7.14 (m, 1H), 7.10–7.02 (m, 1H), 6.92 (d, *J* = 8.6 Hz, 2H), 3.96 (t, *J* = 6.4 Hz, 2H), 3.76 (t, *J* = 7.4 Hz, 1H), 3.41 (dd, *J* = 13.2 and 6.8 Hz, 1H), 2.96 (dd, *J* = 13.2 and 8.2 Hz, 1H), 2.10 (s, 3H), 1.81–1.72 (m, 2H), 1.54–1.44 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ 170.8, 159.1, 137.9, 130.5, 129.1, 128.9, 127.5, 124.2, 121.5, 120.4, 120.3 (q, *J*<sub>C-F</sub> = 257.9 Hz), 115.2, 67.7, 53.8, 37.0, 31.2, 19.2, 16.3, 13.8; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.89; HRMS (EI) Calcd for C<sub>21</sub>H<sub>24</sub>F<sub>3</sub>NO<sub>3</sub> [M<sup>+</sup>]: 427.1429, found 427.1441; IR (KBr) ν(cm<sup>-1</sup>): 3426, 1688, 1614, 1521, 1255, 1217, 1180.

**2-(4-Phenoxyphenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2h) and 3-(Methylthio)-2-(4-phenoxyphenyl)-N-(2-(trifluoromethoxy)phenyl)propanamide (3h).** Data of **2h**: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.56 (d, *J* = 8.1 Hz, 1H), 7.88 (s, 1H), 7.44–7.35 (m, 4H), 7.35–7.29 (m, 1H), 7.25–7.20 (m, 1H), 7.17 (d, *J* = 7.4 Hz, 1H), 7.15–7.10 (m, 1H), 7.10–7.02 (m, 4H), 6.37 (s, 1H), 5.75 (s, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.7, 158.2, 156.5, 144.0, 138.1 (d, *J* = 1.3 Hz), 130.9, 130.6, 129.90, 129.87, 127.7, 124.4, 124.1, 123.8, 121.6, 120.4 (d, *J* = 1.1 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.9 Hz), 119.4, 118.8; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.82; HRMS (EI) Calcd for C<sub>22</sub>H<sub>16</sub>F<sub>3</sub>NO<sub>3</sub> [M<sup>+</sup>]: 399.1082, found 399.1086; IR (KBr) ν(cm<sup>-1</sup>): 3415, 1692, 1610, 1526, 1490, 1454, 1250, 1217, 1172. Data of **3h**: White solid; mp 83–84 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.39 (d, *J* = 8.0 Hz, 1H), 7.56 (s, 1H), 7.39–7.30 (m, 4H), 7.30–7.24 (m, 2H), 7.23–7.17 (m, 1H), 7.15–7.05 (m, 2H), 7.02 (d, *J* = 8.4 Hz, 3H), 3.80 (t, *J* = 7.4 Hz, 1H), 3.41 (dd, *J* = 13.3 and 7.1 Hz, 1H), 2.97 (dd, *J* = 13.2 and 7.7 Hz, 1H), 2.12 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.4, 158.6, 157.5, 156.6, 132.1, 130.4, 129.8, 129.4, 127.6, 124.3, 123.7, 121.7, 120.5, 120.4 (q, *J*<sub>C-F</sub> = 259.1 Hz), 119.3, 119.2, 54.0, 37.1, 16.4; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.80; HRMS (EI) Calcd for C<sub>23</sub>H<sub>20</sub>F<sub>3</sub>NO<sub>3</sub>S [M<sup>+</sup>]: 447.1116, found 447.1115; IR (KBr) ν(cm<sup>-1</sup>): 3426, 1633, 1616, 1529, 1252, 1220, 1176.

**2-([1,1'-Biphenyl]-4-yl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2i) and 2-([1,1'-Biphenyl]-4-yl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3i).** Data of **2i**: White solid; mp 116–118 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.57 (d, *J* = 8.2 Hz, 1H), 7.91 (s, 1H), 7.68 (d, *J* = 8.0 Hz, 2H), 7.62 (d, *J* = 7.5 Hz, 2H), 7.55–7.45 (m, 4H), 7.43–7.36 (m, 1H), 7.36–7.29 (m, 1H), 7.22 (d, *J* = 8.1 Hz, 1H), 7.15–7.08 (m, 1H), 6.44 (s, 1H), 5.82 (s, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.6, 144.3, 141.9, 140.3, 138.2 (d, *J* = 1.4 Hz), 135.1, 130.6, 128.9, 128.8, 127.74, 127.69, 127.6, 127.1, 124.5, 124.4, 121.6, 120.4 (d, *J* = 1.1 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.6 Hz); <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.86; HRMS (EI) Calcd for C<sub>22</sub>H<sub>16</sub>F<sub>3</sub>NO<sub>2</sub> [M<sup>+</sup>]: 383.1133 found 383.1138; IR (KBr) ν(cm<sup>-1</sup>): 3416, 1665, 1609, 1527, 1453, 1254, 1218, 1177. Data of **3i**: White solid; mp 79–80 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.40 (d, *J* = 8.1 Hz, 1H), 7.63 (d, *J* = 8.1 Hz, 2H), 7.58 (d, *J* = 7.4 Hz, 3H), 7.49–7.42 (m, 4H), 7.39–7.33 (m, 1H), 7.31–7.24 (m, 1H), 7.21–7.15 (m, 1H), 7.11–7.04 (m, 1H), 3.87 (t, *J* = 7.4 Hz, 1H), 3.47 (dd, *J* = 13.3 and 7.1 Hz, 1H), 3.04 (dd, *J* = 13.3 and 7.8 Hz, 1H), 2.14 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.3, 140.4, 139.9, 136.5, 130.4, 128.8, 128.5, 128.0, 127.5, 127.1, 124.3,

121.7, 120.5 (q, *J*<sub>C-F</sub> = 259.0 Hz), 120.4, 117.7, 117.5, 54.3, 36.9, 16.4; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.86; HRMS (EI) Calcd for C<sub>23</sub>H<sub>20</sub>F<sub>3</sub>NO<sub>2</sub>S [M<sup>+</sup>]: 431.1167 found 431.1154; IR (KBr) ν(cm<sup>-1</sup>): 3406, 1673, 1608, 1529, 1255, 1217, 1180.

**2-(3,5-Dimethylphenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2j) and 2-(3,5-Dimethylphenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3j).** Data of **2j**: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.56 (d, *J* = 8.0 Hz, 1H), 7.89 (s, 1H), 7.35–7.27 (m, 1H), 7.25–7.15 (m, 1H), 7.14–7.04 (m, 2H), 7.02 (s, 2H), 6.42 (d, *J* = 0.6 Hz, 1H), 5.70 (d, *J* = 0.8 Hz, 1H), 2.35 (s, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.6, 144.7, 138.8, 138.1 (d, *J* = 1.5 Hz), 136.3, 130.7, 130.5, 127.6, 126.2, 124.7, 124.2, 121.6, 120.4 (d, *J* = 1.0 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.7 Hz), 21.2; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -58.15; HRMS (EI) Calcd for C<sub>18</sub>H<sub>16</sub>F<sub>3</sub>NO<sub>2</sub> [M<sup>+</sup>]: 335.1133, found 335.1138; IR (KBr) ν(cm<sup>-1</sup>): 3408, 1693, 1610, 1526, 1454, 1254, 1216, 1175. Data of **3j**: White solid; mp 81–82 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.39 (d, *J* = 8.2 Hz, 1H), 7.54 (s, 1H), 7.29–7.22 (m, 1H), 7.20–7.13 (m, 1H), 7.10–7.02 (m, 1H), 6.97 (d, *J* = 6.7 Hz, 3H), 3.75 (t, *J* = 7.4 Hz, 1H), 3.42 (dd, *J* = 13.2 and 6.9 Hz, 1H), 2.98 (dd, *J* = 13.2 and 8.0 Hz, 1H), 2.32 (s, 6H), 2.12 (s, 3H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ 170.5, 139.1, 137.9, 137.3, 130.5, 130.0, 127.5, 125.7, 124.2, 121.5, 120.4, 120.3 (q, *J*<sub>C-F</sub> = 258.0 Hz), 54.4, 36.6, 21.2, 16.3; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -58.08; HRMS (EI) Calcd for C<sub>19</sub>H<sub>20</sub>F<sub>3</sub>NO<sub>2</sub>S [M<sup>+</sup>]: 383.1167, found 383.1161; IR (KBr) ν(cm<sup>-1</sup>): 3404, 1691, 1607, 1528, 1255, 1217, 1179.

**2-(3,5-Dimethoxyphenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2k) and 2-(3,5-Dimethoxyphenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3k).** Data of **2k**: White solid; mp 57–59 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.54 (d, *J* = 7.9 Hz, 1H), 7.95 (s, 1H), 7.33–7.27 (m, 1H), 7.20 (d, *J* = 8.2 Hz, 1H), 7.12–7.05 (m, 1H), 6.58–6.48 (m, 3H), 6.45 (d, *J* = 0.6 Hz, 1H), 5.73 (d, *J* = 0.8 Hz, 1H), 3.81 (s, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.1, 161.2, 144.4, 138.3, 138.1 (d, *J* = 1.3 Hz), 130.6, 127.6, 125.1, 124.3, 121.6, 120.4 (d, *J* = 1.1 Hz), 120.3 (q, *J*<sub>C-F</sub> = 258.0 Hz), 106.5, 100.7, 55.4; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -58.10; HRMS (EI) Calcd for C<sub>18</sub>H<sub>16</sub>F<sub>3</sub>NO<sub>4</sub> [M<sup>+</sup>]: 367.1031, found 367.1025; IR (KBr) ν(cm<sup>-1</sup>): 3409, 1689, 1600, 1528, 1454, 1255, 1213, 1161. Data of **3k**: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.38 (d, *J* = 8.1 Hz, 1H), 7.60 (s, 1H), 7.29–7.23 (m, 1H), 7.21–7.15 (m, 1H), 7.10–7.01 (m, 1H), 6.55–6.47 (m, 2H), 6.47–7.39 (m, 1H), 3.79 (s, 6H), 3.75 (t, *J* = 7.4 Hz, 1H), 3.40 (dd, *J* = 13.3 and 6.9 Hz, 1H), 2.97 (dd, *J* = 13.3 and 7.9 Hz, 1H), 2.12 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.0, 161.5, 142.9, 139.8, 130.5, 127.5, 124.2, 121.6, 120.45, 120.38 (q, *J*<sub>C-F</sub> = 256.6 Hz), 106.1, 99.9, 55.3, 54.9, 36.5, 16.4; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -58.00; HRMS (EI) Calcd for C<sub>19</sub>H<sub>20</sub>F<sub>3</sub>NO<sub>4</sub>S [M<sup>+</sup>]: 415.1065, found 415.1071; IR (KBr) ν(cm<sup>-1</sup>): 3404, 1696, 1608, 1527, 1255, 1215, 1184.

**2-(3-Fluoro-4-methoxyphenyl)-N-(2-(trifluoromethoxy)phenyl)acrylamide (2l) and 2-(3-Fluoro-4-methoxyphenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3l).** Data of **2l**: White solid; mp 69–70 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.52 (d, *J* = 8.2 Hz, 1H), 7.84 (s, 1H), 7.35–7.29 (m, 1H), 7.25–7.15 (m, 3H), 7.14–7.08 (m, 1H), 7.05–6.98 (m, 1H), 6.30 (s, 1H), 5.73 (s, 1H), 3.93 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.6, 152.3 (d, *J*<sub>C-F</sub> = 246.5 Hz), 148.3 (d, *J* = 10.6 Hz), 143.4 (d, *J* = 15.7 Hz), 138.2 (d, *J* = 1.5 Hz), 130.5, 129.0 (d, *J* = 6.5 Hz), 127.7, 124.5, 124.4 (d, *J* = 3.6 Hz), 123.8, 121.6, 120.4 (d, *J* = 1.2 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.9 Hz), 116.2 (d, *J* = 19.1 Hz), 113.6 (d, *J* = 2.3 Hz), 56.3; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -58.09, -133.54; HRMS (EI) Calcd for C<sub>17</sub>H<sub>13</sub>F<sub>4</sub>NO<sub>3</sub> [M<sup>+</sup>]: 355.0832 found 355.0848; IR (KBr) ν(cm<sup>-1</sup>): 3416, 1666, 1615, 1522, 1450, 1252, 1217, 1181. Data of **3l**: White solid; mp 81–82 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.36 (d, *J* = 8.1 Hz, 1H), 7.54 (s, 1H), 7.30–7.25 (m, 1H), 7.23–7.16 (m, 1H), 7.16–7.05 (m, 3H), 7.02–6.94 (m, 1H), 3.89 (s, 3H), 3.74 (t, *J* = 7.4 Hz, 1H), 3.37 (dd, *J* = 13.3 and 7.0 Hz, 1H), 2.94 (dd, *J* = 13.3 and 7.9 Hz, 1H), 2.11 (s, 3H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ 170.0, 152.6 (d, *J*<sub>C-F</sub> = 246.5 Hz),

147.6 (d,  $J_{C-F} = 18.9$  Hz), 138.1, 130.4 (d,  $J_{C-F} = 5.9$  Hz), 130.3, 127.6, 124.4, 124.0 (d,  $J_{C-F} = 3.5$  Hz), 121.7, 120.5, 120.3 (q,  $J_{C-F} = 257.9$  Hz), 115.7 (d,  $J_{C-F} = 18.9$  Hz), 113.9 (d,  $J_{C-F} = 2.0$  Hz), 56.3, 53.7, 37.0, 16.4;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.09, -133.54; HRMS (EI) Calcd for  $\text{C}_{18}\text{H}_{17}\text{F}_4\text{NO}_3\text{S}$  [ $\text{M}^+$ ]: 403.0865 found 403.0875; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3435, 1632, 1522, 1259, 1219, 1177.

***N*-(2-(Trifluoromethoxy)phenyl)-2-(3,4,5-trimethoxyphenyl) acrylamide (2m) and 3-(Methylthio)-*N*-(2-(trifluoromethoxy)phenyl)-2-(3,4,5-trimethoxyphenyl) propanamide (3m).** Data of **2m**: White solid; mp 62–63 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.55 (d,  $J = 8.0$  Hz, 1H), 7.92 (s, 1H), 7.35–7.27 (m, 1H), 7.24–7.15 (m, 1H), 7.14–7.05 (m, 1H), 6.61 (s, 2H), 6.42 (s, 1H), 5.72 (d,  $J = 0.7$  Hz, 1H), 3.87 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.3, 153.6, 144.5, 138.6, 138.0 (d,  $J = 1.1$  Hz), 131.8, 130.6, 127.7, 124.7, 124.3, 121.4, 120.4, 120.3 (q,  $J_{C-F} = 257.9$  Hz), 105.6, 60.8, 56.1;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.00; HRMS (EI) Calcd for  $\text{C}_{19}\text{H}_{18}\text{F}_3\text{NO}_5$  [ $\text{M}^+$ ]: 397.1137, found 397.1133; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3411, 1690, 1611, 1585, 1528, 1454, 1254, 1219, 1180, 1127. Data of **3m**: White solid; mp 95–96 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.38 (d,  $J = 8.2$  Hz, 1H), 7.59 (s, 1H), 7.28 (d,  $J = 7.6$  Hz, 1H), 7.19 (d,  $J = 8.2$  Hz, 1H), 7.12–7.05 (m, 1H), 6.57 (s, 2H), 3.87 (s, 6H), 3.84 (s, 3H), 3.74 (t,  $J = 7.3$  Hz, 1H), 3.41 (dd,  $J = 13.3$  and 7.0 Hz, 1H), 2.98 (dd,  $J = 13.3$  and 7.7 Hz, 1H), 2.13 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 153.9, 137.9, 133.2, 130.4, 127.6, 124.3, 121.6, 120.5 (d,  $J = 1.0$  Hz), 120.3 (q,  $J_{C-F} = 258.9$  Hz), 104.9, 100.0, 60.8, 56.1, 55.0, 36.8, 16.5;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.92; HRMS (EI) Calcd for  $\text{C}_{20}\text{H}_{22}\text{F}_3\text{NO}_5\text{S}$  [ $\text{M}^+$ ]: 445.1171, found 445.1190; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3427, 1634, 1615, 1529, 1250, 1217, 1182.

**2-(Naphthalen-2-yl)-*N*-(2-(trifluoromethoxy)phenyl) acrylamide (2n) and 3-(Methylthio)-2-(naphthalen-2-yl)-*N*-(2-(trifluoromethoxy)phenyl)propanamide (3n).** Data of **2n**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.56 (d,  $J = 8.0$  Hz, 1H), 7.96–7.82 (m, 5H), 7.58–7.49 (m, 3H), 7.36–7.29 (m, 1H), 7.22–7.15 (m, 1H), 7.14–7.07 (m, 1H), 6.47 (s, 1H), 5.88 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.8, 144.7, 138.3 (d,  $J = 1.4$  Hz), 133.6, 133.2, 130.6, 128.9, 128.2, 127.8, 127.7, 127.6, 126.8, 126.7, 125.6, 124.7, 124.5, 121.8, 120.4 (d,  $J = 1.0$  Hz), 120.2 (q,  $J_{C-F} = 258.1$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.98; HRMS (EI) Calcd for  $\text{C}_{20}\text{H}_{14}\text{F}_3\text{NO}_2$  [ $\text{M}^+$ ]: 357.0977 found 357.0986; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3401, 1693, 1609, 1529, 1454, 1255, 1216, 1180. Data of **3n**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.39 (d,  $J = 8.1$  Hz, 1H), 7.92–7.81 (m, 4H), 7.60 (s, 1H), 7.54–7.44 (m, 3H), 7.28–7.23 (m, 1H), 7.19–7.11 (m, 1H), 7.09–7.01 (m, 1H), 3.99 (t,  $J = 7.4$  Hz, 1H), 3.52 (dd,  $J = 13.3$  and 7.1 Hz, 1H), 3.09 (dd,  $J = 13.3$  and 7.7 Hz, 1H), 2.12 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 138.0, 135.0, 133.5, 133.0, 130.4, 129.3, 127.9, 127.7, 127.5, 127.4, 126.6, 126.4, 125.2, 124.3, 121.7, 120.4, 120.2 (q,  $J_{C-F} = 258.3$  Hz), 54.8, 36.9, 16.4;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.99; HRMS (EI) Calcd for  $\text{C}_{21}\text{H}_{18}\text{F}_3\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 405.1010 found 405.1003; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3406, 1688, 1608, 1528, 1256, 1217, 1180.

**2-(Thiophen-3-yl)-*N*-(2-(trifluoromethoxy)phenyl) acrylamide (2o) and 3-(Methylthio)-2-(thiophen-3-yl)-*N*-(2-(trifluoromethoxy)phenyl)propanamide (3o).** Data of **2o**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.55 (d,  $J = 8.1$  Hz, 1H), 7.99 (s, 1H), 7.50–7.45 (m, 1H), 7.45–7.39 (m, 1H), 7.35–7.29 (m, 1H), 7.25–7.19 (m, 2H), 7.15–7.09 (m, 1H), 6.27 (s, 1H), 5.81 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.7, 139.5, 138.2 (d,  $J = 1.4$  Hz), 136.7, 130.5, 127.6, 127.04, 126.99, 124.6, 124.4, 123.0, 121.7, 120.38 (d,  $J = 1.0$  Hz), 120.37 (q,  $J_{C-F} = 257.7$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.78; HRMS (EI) Calcd for  $\text{C}_{14}\text{H}_{10}\text{F}_3\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 313.0384 found 313.0378; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3397, 1688, 1610, 1529, 1453, 1258, 1216, 1181. Data of **3o**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.40 (d,  $J = 8.1$  Hz, 1H), 7.65 (s, 1H), 7.45–7.39 (m, 1H), 7.36–7.27 (m, 2H), 7.23–7.17 (m, 1H), 7.13–7.04 (m, 2H), 4.00 (t,  $J = 7.2$  Hz, 1H), 3.37 (dd,  $J = 13.3$  and 6.7 Hz, 1H), 3.00 (dd,  $J = 13.3$  and 7.8 Hz, 1H), 2.11 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.0, 137.9, 130.4, 127.6, 127.5, 127.4, 126.6, 124.4, 123.4, 121.6, 120.4, 120.3 (q,  $J_{C-F} = 257.9$  Hz), 50.0, 37.0, 16.4;

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.87; HRMS (EI) Calcd for  $\text{C}_{15}\text{H}_{14}\text{F}_3\text{NO}_2\text{S}_2$  [ $\text{M}^+$ ]: 361.0418 found 361.0412; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3422, 1688, 1613, 1529, 1256, 1217, 1180.

**2-(2-Bromophenyl)-*N*-(2-(trifluoromethoxy)phenyl) acrylamide (2p) and 2-(2-Bromophenyl)-3-(methylthio)-*N*-(2-(trifluoromethoxy)phenyl)propanamide (3p).** Data of **2p**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.63–8.50 (m, 1H), 7.69 (d,  $J = 8.0$  Hz, 1H), 7.52 (s, 1H), 7.46–7.36 (m, 2H), 7.35–7.27 (m, 2H), 7.20–7.13 (m, 1H), 7.11–7.04 (m, 1H), 6.64 (d,  $J = 0.9$  Hz, 1H), 5.71 (d,  $J = 0.8$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  162.9, 143.9, 138.0 (d,  $J = 1.5$  Hz), 137.5, 133.3, 131.4, 130.65, 130.59, 128.1, 127.6, 127.3, 124.2, 123.7, 121.3, 120.3 (d,  $J = 1.0$  Hz), 120.2 (q,  $J_{C-F} = 258.0$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.12; HRMS (EI) Calcd for  $\text{C}_{16}\text{H}_{11}\text{BrF}_3\text{NO}_2$  [ $\text{M}^+$ ]: 384.9925 found 384.9922; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3419, 1696, 1611, 1526, 1453, 1254, 1216, 1183. Data of **3p**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.39 (d,  $J = 8.1$  Hz, 1H), 7.76 (s, 1H), 7.63 (d,  $J = 7.9$  Hz, 1H), 7.49–7.42 (m, 1H), 7.35 (t,  $J = 7.3$  Hz, 1H), 7.29–7.23 (m, 1H), 7.23–7.14 (m, 2H), 7.12–7.07 (m, 1H), 4.52–4.42 (m, 1H), 3.43 (dd,  $J = 13.5$  and 8.1 Hz, 1H), 2.95 (dd,  $J = 13.5$  and 6.6 Hz, 1H), 2.17 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  169.4, 138.0, 137.1, 133.2, 130.4, 129.5, 128.7, 128.4, 127.5, 124.7, 124.3, 121.7, 120.4 (q,  $J_{C-F} = 257.8$  Hz), 120.3, 52.3, 36.0, 16.3;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.70; HRMS (EI) Calcd for  $\text{C}_{17}\text{H}_{15}\text{BrF}_3\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 432.9959 found 432.9931; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3420, 1699, 1611, 1527, 1259, 1217, 1182.

**2-(4-Chlorophenyl)-*N*-(2-(trifluoromethoxy)phenyl) acrylamide (2q) and 2-(4-Chlorophenyl)-3-(methylthio)-*N*-(2-(trifluoromethoxy)phenyl)propanamide (3q).** Data of **2q**: White solid; mp 88–89 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.51 (d,  $J = 8.1$  Hz, 1H), 7.77 (s, 1H), 7.45–7.36 (m, 4H), 7.35–7.28 (m, 1H), 7.25–7.19 (m, 1H), 7.15–7.08 (m, 1H), 6.35 (s, 1H), 5.77 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.4, 143.7, 138.2 (d,  $J = 1.4$  Hz), 135.1, 134.7, 130.4, 129.6, 129.2, 127.6, 124.6, 124.5, 121.7, 120.4 (d,  $J = 1.1$  Hz), 120.3 (q,  $J_{C-F} = 257.9$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.89; HRMS (EI) Calcd for  $\text{C}_{16}\text{H}_{11}\text{ClF}_3\text{NO}_2$  [ $\text{M}^+$ ]: 341.0430 found 341.0439; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3420, 1689, 1611, 1527, 1452, 1256, 1216, 1180.

Data of **3q**: White solid; mp 88–89 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.35 (d,  $J = 7.9$  Hz, 1H), 7.52 (s, 1H), 7.39–7.24 (m, 5H), 7.22–7.17 (m, 1H), 7.12–7.06 (m, 1H), 3.77 (t,  $J = 7.4$  Hz, 1H), 3.38 (dd,  $J = 13.3$  and 7.3 Hz, 1H), 2.94 (dd,  $J = 13.3$  and 7.5 Hz, 1H), 2.11 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  169.8, 138.1, 136.1, 134.2, 129.4, 129.3, 129.0, 127.6, 124.5, 121.7, 120.6 (q,  $J_{C-F} = 256.9$  Hz), 120.5, 54.0, 37.0, 16.4;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.85; HRMS (EI) Calcd for  $\text{C}_{17}\text{H}_{15}\text{ClF}_3\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 389.0464 found 389.0471; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3426, 1634, 1529, 1255, 1217, 1179.

**2-(4-Bromophenyl)-*N*-(2-(trifluoromethoxy)phenyl) acrylamide (2r) and 2-(4-Bromophenyl)-3-(methylthio)-*N*-(2-(trifluoromethoxy)phenyl)propanamide (3r).** Data of **2r**: White solid; mp 90–91 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.50 (d,  $J = 8.1$  Hz, 1H), 7.76 (s, 1H), 7.58 (d,  $J = 8.3$  Hz, 2H), 7.37–7.28 (m, 3H), 7.22 (d,  $J = 8.2$  Hz, 1H), 7.16–7.08 (m, 1H), 6.36 (s, 1H), 5.78 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.3, 143.8, 138.2 (d,  $J = 1.1$  Hz), 135.1, 132.2, 130.4, 129.9, 127.7, 124.6, 124.5, 123.3, 121.7, 120.5 (d,  $J = 1.2$  Hz), 120.3 (q,  $J_{C-F} = 257.7$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.87; HRMS (EI) Calcd for  $\text{C}_{16}\text{H}_{11}\text{BrF}_3\text{NO}_2$  [ $\text{M}^+$ ]: 384.9925, found 384.9921; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3420, 1689, 1611, 1527, 1453, 1256, 1216, 1181. Data of **3r**: White solid; mp 97–98 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.35 (d,  $J = 7.9$  Hz, 1H), 7.57–7.47 (m, 3H), 7.30–7.24 (m, 3H), 7.23–7.17 (m, 1H), 7.13–7.05 (m, 1H), 3.76 (t,  $J = 7.4$  Hz, 1H), 3.37 (dd,  $J = 13.3$  and 7.4 Hz, 1H), 2.93 (dd,  $J = 13.3$  and 7.4 Hz, 1H), 2.11 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.8, 138.2, 136.7, 132.1, 130.1, 129.5, 129.2, 127.4, 124.5, 122.0, 120.4, 120.2 (q,  $J_{C-F} = 257.8$  Hz), 53.7, 36.9, 16.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.79; HRMS (EI) Calcd for  $\text{C}_{17}\text{H}_{15}\text{BrF}_3\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 432.9959, found 432.9956; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3274, 1666, 1608, 1528, 1267, 1216, 1171.

**2-(4-Iodophenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2s) and 2-(4-Iodophenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3s).** Data of **2s**: White solid; mp 95–97 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.50 (d,  $J = 8.1$  Hz, 1H), 7.97–7.67 (m, 3H), 7.35–7.28 (m, 1H), 7.25–7.20 (m, 1H), 7.18 (d,  $J = 8.3$  Hz, 2H), 7.15–7.09 (m, 1H), 6.35 (s, 1H), 5.77 (s, 1H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.2, 143.9, 138.3 (d,  $J = 1.3$  Hz), 138.1, 135.7, 130.4, 130.0, 127.7, 124.6, 124.4, 121.7, 120.5 (d,  $J = 1.0$  Hz), 120.3 (q,  $J_{\text{C-F}} = 257.9$  Hz), 94.9;  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.85; HRMS (EI) Calcd for  $\text{C}_{16}\text{H}_{11}\text{IF}_3\text{NO}_2$  [ $\text{M}^+$ ]: 432.9787 found 432.9780; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3418, 1664, 1610, 1525, 1452, 1258, 1215, 1161. Data of **3s**: White solid; mp 114–115 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.34 (d,  $J = 7.8$  Hz, 1H), 7.73 (d,  $J = 8.2$  Hz, 2H), 7.53 (s, 1H), 7.31–7.23 (m, 1H), 7.22–7.17 (m, 1H), 7.16–7.05 (m, 3H), 3.74 (t,  $J = 7.4$  Hz, 1H), 3.37 (dd,  $J = 13.3$  and 7.4 Hz, 1H), 2.93 (dd,  $J = 13.3$  and 7.4 Hz, 1H), 2.11 (s, 3H);  $^{13}\text{C NMR}$  (150 MHz,  $\text{CDCl}_3$ )  $\delta$  169.7, 138.3, 137.3, 130.2, 129.8, 129.6, 127.6, 124.5, 121.7, 120.5, 120.3 (q,  $J_{\text{C-F}} = 258.2$  Hz), 93.9, 54.1, 36.9, 16.4;  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.80; HRMS (EI) Calcd for  $\text{C}_{17}\text{H}_{15}\text{F}_3\text{INO}_2\text{S}$  [ $\text{M}^+$ ]: 480.9820 found 480.9810; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3428, 1634, 1528, 1254, 1216, 1178.

**2-(3-Fluorophenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2t) and 2-(3-Fluorophenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3t).** Data of **2t**: Brown oil;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.52 (d,  $J = 8.1$  Hz, 1H), 7.80 (s, 1H), 7.48–7.38 (m, 1H), 7.35–7.29 (m, 1H), 7.25–7.19 (m, 2H), 7.19–7.08 (m, 3H), 6.41 (s, 1H), 5.79 (s, 1H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.1, 164.0, 161.7, 143.7, 138.3 (d,  $J = 22.0$  Hz), 130.7 (d,  $J = 8.4$  Hz), 130.4, 127.7, 125.1, 124.6, 124.0 (d,  $J = 3.1$  Hz), 121.6, 120.5 (d,  $J = 1.1$  Hz), 120.3 (q,  $J_{\text{C-F}} = 257.8$  Hz), 116.0 (d,  $J = 20.9$  Hz), 115.5 (d,  $J = 22.1$  Hz);  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.10, –111.71; HRMS (EI) Calcd for  $\text{C}_{16}\text{H}_{11}\text{F}_4\text{NO}_2$  [ $\text{M}^+$ ]: 325.0726 found 325.0724; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3419, 1691, 1610, 1528, 1451, 1256, 1217, 1181. Data of **3t**: White solid; mp 73–74 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.36 (d,  $J = 8.0$  Hz, 1H), 7.54 (s, 1H), 7.41–7.33 (m, 1H), 7.31–7.24 (m, 1H), 7.22–7.14 (m, 2H), 7.13–6.98 (m, 3H), 3.80 (t,  $J = 7.4$  Hz, 1H), 3.39 (dd,  $J = 13.3$  and 7.4 Hz, 1H), 2.95 (dd,  $J = 13.3$  and 7.4 Hz, 1H), 2.12 (s, 3H);  $^{13}\text{C NMR}$  (150 MHz,  $\text{CDCl}_3$ )  $\delta$  169.6, 163.2 (d,  $J_{\text{C-F}} = 246.3$  Hz), 140.1 (d,  $J = 7.1$  Hz), 138.1, 130.8 (d,  $J = 8.3$  Hz), 130.3, 127.6, 124.5, 123.8 (d,  $J = 2.9$  Hz), 121.8, 120.5, 120.3 (q,  $J_{\text{C-F}} = 258.0$  Hz), 115.3 (d,  $J = 20.9$  Hz), 115.0 (d,  $J = 21.9$  Hz), 54.3, 36.9, 16.4;  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.10, –111.71; HRMS (EI) Calcd for  $\text{C}_{17}\text{H}_{15}\text{F}_4\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 373.0760 found 373.0766; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3417, 1682, 1612, 1529, 1257, 1218, 1181.

**2-(3-Bromophenyl)-N-(2-(trifluoromethoxy)phenyl) acrylamide (2u) and 2-(3-Bromophenyl)-3-(methylthio)-N-(2-(trifluoromethoxy)phenyl)propanamide (3u).** Data of **2u**: White solid; mp 66–67 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.51 (d,  $J = 8.1$  Hz, 1H), 7.76 (s, 1H), 7.63–7.54 (m, 2H), 7.38 (d,  $J = 7.7$  Hz, 1H), 7.35–7.29 (m, 2H), 7.25–7.19 (m, 1H), 7.15–7.08 (m, 1H), 6.38 (s, 1H), 5.78 (s, 1H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.1, 143.5, 138.3, 138.2 (d,  $J = 1.5$  Hz), 132.0, 131.4, 130.42, 130.37, 127.6, 126.8, 125.1, 124.6, 123.1, 121.7, 120.5 (d,  $J = 1.1$  Hz), 120.3 (q,  $J_{\text{C-F}} = 258.0$  Hz);  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.91; HRMS (EI) Calcd for  $\text{C}_{16}\text{H}_{11}\text{BrF}_3\text{NO}_2$  [ $\text{M}^+$ ]: 384.9925 found 384.9926; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3303, 1661, 1608, 1526, 1453, 1257, 1213, 1170. Data of **3u**: White solid; mp 72–73 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.36 (d,  $J = 7.9$  Hz, 1H), 7.54 (s, 2H), 7.51–7.44 (m, 1H), 7.35–7.24 (m, 3H), 7.23–7.18 (m, 1H), 7.14–7.05 (m, 1H), 3.75 (t,  $J = 7.4$  Hz, 1H), 3.38 (dd,  $J = 13.3$  and 7.6 Hz, 1H), 2.94 (dd,  $J = 13.3$  and 7.2 Hz, 1H), 2.12 (s, 3H);  $^{13}\text{C NMR}$  (150 MHz,  $\text{CDCl}_3$ )  $\delta$  169.5, 139.9, 138.1, 131.4, 131.0, 130.8, 130.7, 130.2, 127.6, 126.6, 124.6, 123.3, 121.8, 120.5, 120.3 (q,  $J_{\text{C-F}} = 258.1$  Hz), 54.2, 37.0, 16.4;  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.83; HRMS (EI) Calcd for  $\text{C}_{17}\text{H}_{15}\text{BrF}_3\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 432.9959 found 432.9945; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3423, 1668, 1634, 1529, 1256, 1217, 1179.

**N-(2-(trifluoromethoxy)phenyl)-2-(3-(trifluoromethoxy)**

**phenyl)acrylamide (2v) and 3-(Methylthio)-N-(2-(trifluoromethoxy)phenyl)-2-(3-(trifluoromethoxy) phenyl) propanamide (3v).** Data of **2v**: Brown oil;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.52 (d,  $J = 8.1$  Hz, 1H), 7.75 (s, 1H), 7.48 (t,  $J = 7.8$  Hz, 1H), 7.38 (d,  $J = 7.7$  Hz, 1H), 7.35–7.27 (m, 3H), 7.25–7.19 (m, 1H), 7.15–7.09 (m, 1H), 6.40 (s, 1H), 5.81 (s, 1H);  $^{13}\text{C NMR}$  (150 MHz,  $\text{CDCl}_3$ )  $\delta$  164.0, 149.6, 143.5, 138.23, 138.17, 130.4, 130.3, 127.7, 126.6, 125.2, 124.6, 121.6, 121.2, 121.0, 120.5, 120.4 (q,  $J_{\text{C-F}} = 255.7$  Hz), 120.3 (q,  $J_{\text{C-F}} = 257.8$  Hz);  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.90, –58.08; HRMS (EI) Calcd for  $\text{C}_{17}\text{H}_{11}\text{F}_6\text{NO}_3$  [ $\text{M}^+$ ]: 391.0643 found 391.0638; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3422, 1691, 1610, 1528, 1453, 1260, 1216, 1173. Data of **3v**: White solid; mp 114–115 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.36 (d,  $J = 7.9$  Hz, 1H), 7.54 (s, 1H), 7.43 (t,  $J = 7.9$  Hz, 1H), 7.33 (d,  $J = 7.6$  Hz, 1H), 7.31–7.25 (m, 2H), 7.24–7.16 (m, 2H), 7.13–7.06 (m, 1H), 3.81 (t,  $J = 7.3$  Hz, 1H), 3.39 (dd,  $J = 13.4$  and 7.6 Hz, 1H), 2.95 (dd,  $J = 13.4$  and 7.1 Hz, 1H), 2.11 (s, 3H);  $^{13}\text{C NMR}$  (150 MHz,  $\text{CDCl}_3$ )  $\delta$  169.5, 149.7, 140.0, 138.1, 130.6, 130.2, 127.6, 126.3, 126.0, 124.6, 121.9, 120.7, 120.5, 120.4 (q,  $J_{\text{C-F}} = 257.9$  Hz), 120.3 (q,  $J_{\text{C-F}} = 257.7$  Hz), 54.4, 37.1, 16.4;  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.85, –57.98; HRMS (EI) Calcd for  $\text{C}_{18}\text{H}_{15}\text{F}_6\text{NO}_3\text{S}$  [ $\text{M}^+$ ]: 439.0677 found 439.0673; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3423, 1662, 1612, 1529, 1263, 1218, 1176.

**N-(2-(trifluoromethoxy)phenyl)-2-(3-(trifluoromethoxy)phenyl)acrylamide (2w) and 3-(Methylthio)-N-(2-(trifluoromethoxy)phenyl)-2-(3-(trifluoromethoxy) phenyl) propanamide (3w).** Data of **2w**: White solid; mp 62–63 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.52 (d,  $J = 8.0$  Hz, 1H), 7.82–7.68 (m, 3H), 7.60–7.55 (m, 1H), 7.51 (t,  $J = 7.7$  Hz, 1H), 7.35–7.29 (m, 1H), 7.25–7.19 (m, 1H), 7.15–7.09 (m, 1H), 6.39 (s, 1H), 5.83 (s, 1H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.2, 143.6, 138.2 (d,  $J = 1.4$  Hz), 137.8, 136.6, 136.0, 130.6, 130.3, 130.0, 129.4 (q,  $J_{\text{C-F}} = 306.1$  Hz), 127.7, 125.5 (d,  $J = 2.2$  Hz), 125.0, 124.7, 121.6, 120.4 (d,  $J = 1.1$  Hz), 120.3 (q,  $J_{\text{C-F}} = 258.5$  Hz);  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –42.52, –58.01; HRMS (EI) Calcd for  $\text{C}_{17}\text{H}_{11}\text{F}_6\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 407.0415 found 407.0422; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3423, 1693, 1611, 1526, 1454, 1256, 1217, 1168, 1119. Data of **3w**: White solid; mp 79–80 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.35 (d,  $J = 7.9$  Hz, 1H), 7.68 (s, 1H), 7.64 (d,  $J = 7.8$  Hz, 1H), 7.54 (d,  $J = 8.1$  Hz, 2H), 7.46 (t,  $J = 7.7$  Hz, 1H), 7.31–7.24 (m, 1H), 7.24–7.17 (m, 1H), 7.14–7.07 (m, 1H), 3.81 (t,  $J = 7.4$  Hz, 1H), 3.40 (dd,  $J = 13.4$  and 7.7 Hz, 1H), 2.95 (dd,  $J = 13.4$  and 7.0 Hz, 1H), 2.10 (s, 3H);  $^{13}\text{C NMR}$  (150 MHz,  $\text{CDCl}_3$ )  $\delta$  169.5, 139.4, 138.1, 135.94, 135.86, 130.3, 130.2, 130.1, 129.4 (q,  $J_{\text{C-F}} = 306.0$  Hz), 127.6, 125.4, 124.6, 121.9, 120.5, 120.3 (q,  $J_{\text{C-F}} = 257.8$  Hz), 54.3, 37.2, 16.4;  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –42.56, –57.92; HRMS (EI) Calcd for  $\text{C}_{18}\text{H}_{15}\text{F}_6\text{NO}_2\text{S}_2$  [ $\text{M}^+$ ]: 455.0448 found 455.0445; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3432, 1632, 1529, 1258, 1217, 1166.

**N-(2-(difluoromethoxy)phenyl)-2-(3-(trifluoromethoxy) phenyl)acrylamide (2x) and N-(2-(difluoromethoxy)phenyl)-3-(methylthio)-2-(3-(trifluoromethoxy) phenyl)propanamide (3x).** Data of **2x**: Brown oil;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.49 (d,  $J = 8.2$  Hz, 1H), 7.90 (s, 1H), 7.48 (t,  $J = 7.9$  Hz, 1H), 7.40 (d,  $J = 7.7$  Hz, 1H), 7.33 (s, 1H), 7.31–7.22 (m, 2H), 7.20–7.04 (m, 2H), 6.38 (t,  $J = 73.3$  Hz, 1H), 6.35 (s, 1H), 5.81 (s, 1H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.3, 149.5, 143.8, 140.3 (t,  $J = 2.4$  Hz), 138.3, 130.3, 129.8, 126.6, 126.3, 124.6, 124.5, 121.4, 121.2, 120.9, 120.4 (q,  $J_{\text{C-F}} = 260.0$  Hz), 118.6, 116.1 (t,  $J_{\text{C-F}} = 260.0$  Hz);  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –80.31; HRMS (EI) Calcd for  $\text{C}_{17}\text{H}_{12}\text{F}_5\text{NO}_3$  [ $\text{M}^+$ ]: 373.0737 found 373.0741; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3413, 1686, 1608, 1527, 1455, 1260, 1214, 1168. Data of **3x**: Brown oil;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.36 (d,  $J = 8.2$  Hz, 1H), 7.66 (s, 1H), 7.45–7.39 (m, 1H), 7.34 (d,  $J = 7.7$  Hz, 1H), 7.28–7.25 (m, 1H), 7.24–7.16 (m, 2H), 7.07 (d,  $J = 4.1$  Hz, 2H), 6.37 (t,  $J = 73.3$  Hz, 1H), 3.79 (t,  $J = 7.4$  Hz, 1H), 3.38 (dd,  $J = 13.3$  and 7.9 Hz, 1H), 2.93 (dd,  $J = 13.3$  and 6.9 Hz, 1H), 2.12 (s, 3H);  $^{13}\text{C NMR}$  (150 MHz,  $\text{CDCl}_3$ )  $\delta$  169.5, 149.7, 140.2, 130.9, 130.5, 129.7, 126.3, 126.2, 124.6, 121.5, 120.7, 120.40, 120.39 (q,  $J_{\text{C-F}} = 255.8$  Hz), 118.7, 116.3 (t,  $J_{\text{C-F}} = 259.7$  Hz), 54.4, 37.3, 16.4;  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )

$\delta$  –57.78, –80.07; HRMS (EI) Calcd for  $C_{18}H_{16}F_5NO_3S$  [ $M^+$ ]: 421.0771 found 421.0776; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3423, 1683, 1609, 1529, 1265, 1220, 1175.

**2-Phenyl-N-(2-(trifluoromethyl)phenyl)acrylamide (2aa) and 3-(Methylthio)-2-phenyl-N-(2-(trifluoromethyl)phenyl) propanamide (3aa).** Data of **2aa**: White solid; mp 66–67 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.40 (d,  $J = 8.3$  Hz, 1H), 7.79 (s, 1H), 7.62–7.52 (m, 2H), 7.49–7.37 (m, 5H), 7.25–7.18 (m, 1H), 6.40 (s, 1H), 5.77 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.0, 144.7, 136.0, 135.2 (d,  $J = 1.4$  Hz), 132.8 (d,  $J = 0.8$  Hz), 128.96, 128.94, 128.5, 126.0 (q,  $J_{\text{C-F}} = 5.3$  Hz), 124.53, 124.46, 123.84, 123.8 (q,  $J_{\text{C-F}} = 271.4$  Hz), 119.9 (d,  $J_{\text{C-F}} = 29.7$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –61.07; HRMS (EI) Calcd for  $C_{16}H_{12}F_3NO$  [ $M^+$ ]: 291.0871 found 291.0869; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3428, 1692, 1594, 1530, 1454, 1321, 1290, 1171, 1114. Data of **3aa**: White solid; mp 85–86 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.20 (d,  $J = 8.0$  Hz, 1H), 7.57–7.49 (m, 2H), 7.46 (s, 1H), 7.43–7.30 (m, 5H), 7.19 (t,  $J = 7.6$  Hz, 1H), 3.80 (t,  $J = 7.4$  Hz, 1H), 3.44 (dd,  $J = 13.3$  and 7.1 Hz, 1H), 2.98 (dd,  $J = 13.3$  and 7.8 Hz, 1H), 2.10 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 137.4, 135.0, 132.8, 129.3, 128.3, 128.1, 126.0 (q,  $J_{\text{C-F}} = 5.3$  Hz), 124.5, 124.1, 123.7 (q,  $J_{\text{C-F}} = 271.4$  Hz), 54.7, 36.9, 16.4;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –60.93; HRMS (EI) Calcd for  $C_{17}H_{16}F_3NOS$  [ $M^+$ ]: 339.0905 found 339.0915; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3422, 1665, 1616, 1527, 1283, 1208, 1169.

**N-(2-(difluoromethoxy)phenyl)-2-phenylacrylamide (2ab) and N-(2-(difluoromethoxy)phenyl)-3-(methylthio)-2-phenylpropanamide (3ab).** Data of **2ab**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.51 (d,  $J = 8.2$  Hz, 1H), 7.95 (s, 1H), 7.49–7.41 (m, 5H), 7.29–7.18 (m, 1H), 7.14–6.99 (m, 2H), 6.38 (s, 1H), 6.33 (t,  $J = 73.3$  Hz, 1H), 5.75 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.7, 144.8, 140.2 (t,  $J = 2.3$  Hz), 136.4, 130.0, 128.9, 128.8, 128.4, 126.3, 124.4, 124.2, 121.4, 118.6, 116.0 (t,  $J_{\text{C-F}} = 260.6$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –80.28; HRMS (EI) Calcd for  $C_{16}H_{13}F_2NO_2$  [ $M^+$ ]: 289.0914 found 289.0907; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3406, 1684, 1608, 1527, 1453, 1128, 1055. Data of **3ab**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.37 (d,  $J = 8.2$  Hz, 1H), 7.65 (s, 1H), 7.46–7.36 (m, 4H), 7.36–7.30 (m, 1H), 7.22–7.16 (m, 1H), 7.10–7.02 (m, 2H), 6.32 (t,  $J = 73.4$  Hz, 1H), 3.80 (t,  $J = 7.4$  Hz, 1H), 3.42 (dd,  $J = 13.3$  and 7.5 Hz, 1H), 2.96 (dd,  $J = 13.3$  and 7.3 Hz, 1H), 2.11 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 140.1, 137.9, 129.9, 129.2, 128.1, 128.0, 126.3, 124.3, 121.3, 118.7, 116.2 (t,  $J_{\text{C-F}} = 259.8$  Hz) 54.8, 37.1, 16.4;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –80.28; HRMS (EI) Calcd for  $C_{17}H_{17}F_2NO_2S$  [ $M^+$ ]: 337.0948 found 337.0941; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3420, 1686, 1608, 1528, 1256, 1196, 1127.

**2-Phenyl-N-(4-(trifluoromethoxy)phenyl)acrylamide (2ac) and 3-(Methylthio)-2-phenyl-N-(4-(trifluoromethoxy)phenyl) propanamide (3ac).** Data of **2ac**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.55 (d,  $J = 9.0$  Hz, 2H), 7.51–7.38 (m, 6H), 7.17 (d,  $J = 8.6$  Hz, 2H), 6.31 (s, 1H), 5.75 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.2, 145.5 (t,  $J = 1.9$  Hz), 144.7, 136.4, 136.3, 129.04, 129.00, 128.3, 123.9, 121.7, 121.1, 120.4 (q,  $J_{\text{C-F}} = 257.8$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.13; HRMS (EI) Calcd for  $C_{16}H_{12}F_3NO_2$  [ $M^+$ ]: 307.0820 found 307.0809; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3294, 1657, 1610, 1554, 1507, 1408, 1297, 1208, 1165. Data of **3ac**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 (d,  $J = 8.9$  Hz, 2H), 7.44 (s, 1H), 7.42–7.36 (m, 4H), 7.35–7.31 (m, 1H), 7.14 (d,  $J = 8.6$  Hz, 2H), 3.73 (t,  $J = 7.3$  Hz, 1H), 3.40 (dd,  $J = 13.2$  and 8.0 Hz, 1H), 2.91 (dd,  $J = 13.2$  and 6.6 Hz, 1H), 2.10 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 145.3, 138.1, 136.2, 129.2, 128.1, 127.9, 121.7, 120.9, 120.4 (q,  $J_{\text{C-F}} = 255.3$  Hz), 54.6, 37.6, 16.5;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –58.17; HRMS (EI) Calcd for  $C_{17}H_{16}F_3NO_2S$  [ $M^+$ ]: 355.0854 found 355.0851; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3421, 1662, 1610, 1511, 1411, 1267, 1223, 1167.

**2-Phenyl-N-(pyridin-3-yl)acrylamide (2ad) and 3-(Methylthio)-2-phenyl-N-(pyridin-3-yl)propanamide (3ad).** Data of **2ad**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.48 (d,  $J = 2.4$  Hz, 1H), 8.34 (d,  $J = 4.5$  Hz, 1H), 8.22 (d,  $J = 8.2$  Hz, 1H), 7.62 (s, 1H), 7.51–7.38 (m,

5H), 7.30–7.25 (m, 1H), 6.31 (s, 1H), 5.77 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.7, 145.5, 144.5, 141.2, 136.3, 134.5, 129.1, 129.0, 128.2, 127.3, 124.1, 123.6; HRMS (EI) Calcd for  $C_{14}H_{12}N_2O$  [ $M^+$ ]: 224.0950 found 224.0953; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3430, 1677, 1592, 1537, 1482, 1422. Data of **3ad**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.46 (d,  $J = 2.2$  Hz, 1H), 8.32 (d,  $J = 4.5$  Hz, 1H), 8.15 (d,  $J = 8.2$  Hz, 1H), 7.58 (s, 1H), 7.42–7.28 (m, 5H), 7.25–7.21 (m, 1H), 3.81–3.75 (m, 1H), 3.40 (dd,  $J = 13.2$  and 8.2 Hz, 1H), 2.90 (dd,  $J = 13.3$  and 6.4 Hz, 1H), 2.10 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 145.3, 141.0, 138.0, 134.6, 129.2, 128.2, 127.9, 127.2, 123.7, 54.5, 37.6, 16.5; HRMS (EI) Calcd for  $C_{15}H_{16}N_2OS$  [ $M^+$ ]: 272.0983 found 272.0980; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3429, 1690, 1667, 1540, 1422, 1280, 1027.

**2-Phenyl-N-(quinolin-8-yl)acrylamide (2ae)<sup>18</sup> and 3-(Methylthio)-2-phenyl-N-(quinolin-8-yl)propanamide (3ae).** Data of **2ae**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  10.26 (s, 8.90 (d,  $J = 7.4$  Hz, 1H), 8.64 (dd,  $J = 4.1$  and 1.5 Hz, 1H), 8.13 (dd,  $J = 8.3$  and 1.4 Hz, 1H), 7.60–7.49 (m, 4H), 7.49–7.42 (m, 3H), 7.39 (dd,  $J = 8.3$  and 4.2 Hz, 1H), 6.33 (s, 1H), 5.84 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.8, 148.2, 145.8, 138.7, 136.7, 136.2, 134.4, 128.7, 128.6, 128.3, 127.8, 127.3, 122.3, 121.8, 121.5, 116.6; Data of **3ae**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.96 (s, 1H), 8.82–8.71 (m, 2H), 8.12 (dd,  $J = 8.2$  and 1.3 Hz, 1H), 7.55–7.46 (m, 4H), 7.44–7.35 (m, 3H), 7.33–7.27 (m, 1H), 4.01–3.95 (m, 1H), 3.48 (dd,  $J = 13.2$  and 8.3 Hz, 1H), 3.00 (dd,  $J = 13.2$  and 6.6 Hz, 1H), 2.15 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 148.2, 138.6, 138.3, 136.3, 134.3, 129.0, 127.9, 127.84, 127.79, 127.3, 121.7, 121.6, 116.5, 55.0, 37.6, 16.4; HRMS (EI) Calcd for  $C_{19}H_{18}N_2OS$  [ $M^+$ ]: 322.1140 found 322.1124; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3428, 1683, 1604, 1528, 1485, 1426.

**2-(p-Tolyl)-N-(4-(trifluoromethoxy)phenyl)acrylamide (2ba) and 3-(Methylthio)-2-(p-tolyl)-N-(3-(trifluoromethoxy)phenyl) propanamide (3ba).** Data of **2ba**: White solid; mp 84–85 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 (s, 1H), 7.40 (s, 1H), 7.30–7.20 (m, 4H), 7.17 (d,  $J = 8.2$  Hz, 2H), 6.90 (d,  $J = 7.5$  Hz, 1H), 6.21 (s, 1H), 5.64 (s, 1H), 2.33 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.4, 149.6 (d,  $J = 1.7$  Hz), 144.5, 139.1, 133.4, 130.0, 129.7, 128.2, 123.6, 121.7, 120.4 (q,  $J_{\text{C-F}} = 255.9$  Hz), 117.8, 116.6, 112.7, 21.2;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.75; HRMS (EI) Calcd for  $C_{17}H_{14}F_3NO_2$  [ $M^+$ ]: 321.0977 found 321.0980; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3412, 1657, 1605, 1537, 1439, 1266, 1211, 1159. Data of **3ba**: White solid; mp 64–65 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (s, 1H), 7.30–7.23 (m, 5H), 7.22–7.17 (m, 2H), 6.94 (d,  $J = 3.5$  Hz, 1H), 3.74–3.67 (m, 1H), 3.38 (dd,  $J = 13.2$  and 7.7 Hz, 1H), 2.90 (dd,  $J = 13.2$  and 7.0 Hz, 1H), 2.35 (s, 3H), 2.10 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 149.5, 139.0, 138.0, 134.9, 129.93, 129.92, 127.9, 120.4 (q,  $J_{\text{C-F}} = 255.8$  Hz), 117.7, 116.4, 112.6, 54.2, 37.5, 21.1, 16.5;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  –57.75; HRMS (EI) Calcd for  $C_{18}H_{18}F_3NO_2S$  [ $M^+$ ]: 369.1010 found 369.1018; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3426, 1660, 1610, 1544, 1261, 1220, 1167.

**N-(2-(methylthio)phenyl)-2-(p-tolyl)acrylamide (2bb) and 3-(Methylthio)-N-(2-(methylthio)phenyl)-2-(p-tolyl) propanamide (3bb).** Data of **2bb**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.67 (s, 1H), 8.49 (d,  $J = 8.2$  Hz, 1H), 7.47–7.41 (m, 1H), 7.36 (d,  $J = 8.0$  Hz, 2H), 7.31 (d,  $J = 7.6$  Hz, 1H), 7.24 (d,  $J = 7.9$  Hz, 2H), 7.09–7.01 (m, 1H), 6.32 (s, 1H), 5.71 (s, 1H), 2.40 (s, 3H), 2.16 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.2, 145.2, 138.6, 138.5, 133.7, 133.2, 129.4, 129.0, 128.4, 125.5, 124.5, 123.1, 120.4, 21.3, 18.8; HRMS (EI) Calcd for  $C_{17}H_{17}NOS$  [ $M^+$ ]: 283.1031 found 283.1020; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3423, 1682, 1616, 1579, 1513, 1435. Data of **3bb**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.43 (s, 1H), 8.32 (d,  $J = 8.2$  Hz, 1H), 7.43 (d,  $J = 7.6$  Hz, 1H), 7.33–7.27 (m, 3H), 7.19 (d,  $J = 7.8$  Hz, 2H), 7.05–6.98 (m, 1H) 3.79 (t,  $J = 7.5$  Hz, 1H), 3.42 (dd,  $J = 13.2$  and 7.4 Hz, 1H), 2.96 (dd,  $J = 13.2$  and 7.6 Hz, 1H), 2.34 (s, 3H), 2.12 (s, 3H), 2.06 (s, 3H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 138.6, 137.8, 135.2, 133.7, 129.8, 129.3, 127.9, 124.9, 124.3, 120.2, 54.4, 37.0, 21.1, 18.8, 16.4; HRMS (EI) Calcd for  $C_{18}H_{21}NOS_2$  [ $M^+$ ]: 331.1065 found 331.1066; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3437, 1638, 1582, 1516, 1435, 1298, 1161.

**N-phenyl-2-(p-tolyl)acrylamide (2bc).** Data of **2bc**: White solid; mp 132–134 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (d,  $J = 7.9$  Hz, 2H), 7.38 (s, 1H), 7.37–7.28 (m, 4H), 7.26–7.21 (m, 2H), 7.11 (t,  $J = 7.4$  Hz, 1H), 6.27 (s, 1H), 5.69 (s, 1H), 2.40 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.3, 144.9, 138.9, 137.7, 133.7, 129.6, 129.0, 128.2, 124.5, 123.0, 119.8, 21.2; HRMS (EI) Calcd for  $\text{C}_{16}\text{H}_{15}\text{NO}$  [ $\text{M}^+$ ]: 237.1154 found 237.1158; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3423, 1652, 1598, 1533, 1441.

#### 4.4. General procedure for the preparation of $d^2$ -2a and $d^5$ -3a

A mixture of substrate **1a** (29.5 mg, 0.1 mmol),  $\text{PbCl}_2$  (41.7 mg, 0.15 mmol) and  $\text{KOAc}$  (14.7 mg, 0.15 mmol) in  $\text{DMSO}-d_6$  (2.0 mL) was put into a 15 mL seal tube. The air in the seal tube was exchanged with  $\text{N}_2$ . The reaction mixture was stirred at 156 °C for 13 h under  $\text{N}_2$ , then cooled down to room temperature, diluted with  $\text{H}_2\text{O}$ , and extracted with  $\text{EtOAc}$  ( $3 \times 5$  mL). The combined organic phase was washed with brine and dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and concentrated under vacuum. The residue was purified by preparative TLC plate (petroleum ether: ethyl acetate = 10: 1 as eluent) to get the product **d<sub>2</sub>-2a** (22.8 mg, 73% yield), **d<sub>5</sub>-3a** (3.9 mg, 9% yield). Data of **d<sub>2</sub>-2a**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.55 (d,  $J = 8.1$  Hz, 1H), 7.84 (s, 1H), 7.52–7.35 (m, 5H), 7.34–7.28 (m, 1H), 7.23–7.15 (m, 1H), 7.13–7.06 (m, 1H), 6.43 (s, 0.02H), 5.75 (s, 0.02H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.5, 144.4, 138.2 (d,  $J = 1.3$  Hz), 136.3, 130.6, 129.0, 128.9, 128.4, 127.6, 124.4, 121.6, 120.4 (d,  $J = 1.1$  Hz), 120.3 (q,  $J_{\text{C-F}} = 257.8$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.98; HRMS (EI) Calcd for  $\text{C}_{16}\text{H}_{10}\text{D}_2\text{F}_3\text{NO}$  [ $\text{M}^+$ ]: 309.0946 found 309.0942; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3400, 1697, 1608, 1531, 1453, 1256, 1216, 1181. Data of **d<sub>5</sub>-3a**: Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.39 (d,  $J = 8.1$  Hz, 1H), 7.53 (s, 1H), 7.44–7.33 (m, 5H), 7.29–7.24 (m, 1H), 7.21–7.15 (m, 1H), 7.10–7.04 (m, 1H), 3.80 (s, 1H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.3, 137.9, 137.6, 130.4, 129.3, 128.3, 128.04, 127.5, 124.3, 121.6, 120.4, 120.3 (q,  $J_{\text{C-F}} = 258.3$  Hz), 62.12;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.92; HRMS (EI) Calcd for  $\text{C}_{16}\text{H}_{11}\text{D}_5\text{F}_3\text{NO}_2\text{S}$  [ $\text{M}^+$ ]: 348.1168 found 348.1157; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3403, 1692, 1608, 1529, 1453, 1255, 1218, 1180.

#### 4.5. General procedure for the synthesis of the dimeric compounds 4

A mixture of substrate **1** (0.1 mmol),  $\text{PbCl}_2$  (41.7 mg, 0.15 mmol) and  $\text{KHCO}_3$  ( $x$  mmol) in  $\text{DMSO}$  (2.0 mL) was put into a 15 mL seal tube. The air in the seal tube was exchanged with  $\text{N}_2$ . The reaction mixture was stirred at 156 °C for 13 h under  $\text{N}_2$ , then cooled down to room temperature, diluted with  $\text{H}_2\text{O}$ , and extracted with  $\text{EtOAc}$  ( $3 \times 5$  mL). The combined organic phase was washed with brine and dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and concentrated under vacuum. The residue was purified by preparative TLC plate (dichloromethane: petroleum ether = 2: 1 as eluent) to get the product **2** and **4**.

**meso-2,4-Diphenyl- $N^1,N^5$ -bis(2-(trifluoromethoxy)phenyl)pentanediamide (meso-4a) and ( $\pm$ )-2,4-Diphenyl- $N^1,N^5$ -bis(2-(trifluoromethoxy)phenyl)pentanediamide ( $\pm$ )-4a.** Data of (**meso-4a**): White solid; mp 115–117 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.38 (d,  $J = 8.1$  Hz, 2H), 7.43 (s, 2H), 7.42–7.36 (m, 4H), 7.36–7.29 (m, 6H), 7.29–7.23 (m, 2H), 7.20–7.15 (m, 2H), 7.10–7.03 (m, 2H), 3.67 (t,  $J = 7.5$  Hz, 2H), 3.25–3.15 (m, 1H), 2.42–2.32 (m, 1H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  171.2, 138.4, 138.0, 130.5, 129.4, 128.1, 127.5, 124.2, 121.6, 120.4, 120.2 (q,  $J_{\text{C-F}} = 257.9$  Hz), 51.5, 35.9;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.92; HRMS (EI) Calcd for  $\text{C}_{31}\text{H}_{24}\text{F}_6\text{N}_2\text{O}_4$  [ $\text{M}^+$ ]: 602.1640, found 602.1629; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3412, 1674, 1608, 1531, 1452, 1260, 1216, 1177. Data of ( $\pm$ )-**4a**: White solid; mp 135–137 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.36 (d,  $J = 8.1$  Hz, 2H), 7.47 (s, 2H), 7.44–7.39 (m, 4H), 7.39–7.34 (m, 2H), 7.34–7.28 (m,

4H), 7.25–7.21 (m, 2H), 7.18–7.10 (m, 2H), 7.08–7.01 (m, 2H), 3.59 (t,  $J = 7.8$  Hz, 2H), 2.88 (t,  $J = 7.8$  Hz, 2H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  171.3, 137.9, 137.6, 130.5, 129.6, 128.4, 128.3, 127.5, 124.1, 121.5, 120.5, 120.2 (q,  $J_{\text{C-F}} = 258.1$  Hz), 51.6, 35.0;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.08; HRMS (EI) Calcd for  $\text{C}_{31}\text{H}_{24}\text{F}_6\text{N}_2\text{O}_4$  [ $\text{M}^+$ ]: 602.1640, found 602.1636; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3419, 1665, 1608, 1528, 1452, 1260, 1218, 1173.

**2,4-Di([1,1'-biphenyl]-4-yl)- $N^1,N^5$ -bis(2-(trifluoromethoxy)phenyl)pentanediamide 4i.** Data of **4i**: White solid; mp 160–161 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.39 (t,  $J = 7.4$  Hz, 3.27H), 7.65 (d,  $J = 8.1$  Hz, 3.55H), 7.63–7.52 (m, 12.80H), 7.51–7.39 (m, 16.12H), 7.39–7.31 (m, 3.64H), 7.30–7.21 (m, 4.59H), 7.20–7.10 (m, 3.44H), 7.10–7.00 (m, 3.59H), 3.78 (t,  $J = 7.5$  Hz, 1.79H), 3.71 (t,  $J = 7.8$  Hz, 2H), 3.34–3.21 (m, 1H), 2.94 (t,  $J = 7.8$  Hz, 1.79H), 2.54–2.40 (m, 1H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  171.3, 171.2, 141.4, 141.1, 140.45, 140.42, 138.04, 137.96, 137.3, 136.5, 130.5, 128.9, 128.83, 128.81, 128.5, 128.3, 128.1, 127.52, 127.47, 127.09, 127.06, 124.24, 124.20, 121.7, 121.5, 120.5, 120.3 (q,  $J_{\text{C-F}} = 258.2$  Hz), 120.2 (q,  $J_{\text{C-F}} = 257.4$  Hz), 51.3, 35.9, 35.0;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.85, -57.99; HRMS (ESI) Calcd for  $\text{C}_{43}\text{H}_{32}\text{F}_6\text{N}_2\text{O}_4$  [ $\text{M}^+$ ]: 755.2345 found 755.2338; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3414, 1671, 1608, 1528, 1451, 1257, 1217, 1178.

**2,4-Di(naphthalen-2-yl)- $N^1,N^5$ -bis(2-(trifluoromethoxy)phenyl)pentanediamide 4n.** Data of **4n**: White solid; mp 128–129 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.44–8.31 (m, 4H), 7.92 (d,  $J = 8.5$  Hz, 2H), 7.90–7.77 (m, 12H), 7.75 (s, 2H), 7.59–7.40 (m, 16H), 7.30–7.18 (m, 4H), 7.17–6.97 (m, 8H), 3.91 (t,  $J = 6.8$  Hz, 2H), 3.78 (t,  $J = 7.3$  Hz, 2H), 3.46–3.33 (m, 1H), 3.07 (t,  $J = 7.4$  Hz, 2H), 2.66–2.53 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.2, 138.1, 138.0, 135.9, 135.0, 133.61, 133.57, 133.1, 132.9, 130.5, 130.4, 129.6, 129.4, 127.84, 127.78, 127.74, 127.67, 127.4, 127.3, 126.6, 126.5, 126.4, 126.3, 125.5, 125.4, 124.25, 124.20, 121.8, 121.6, 121.4, 120.4, 120.3 (q,  $J_{\text{C-F}} = 258.2$  Hz), 120.2 (q,  $J_{\text{C-F}} = 257.4$  Hz), 51.7, 35.9, 34.8;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -58.03, -58.21; HRMS (ESI) Calcd for  $\text{C}_{39}\text{H}_{28}\text{F}_6\text{N}_2\text{O}_4$  [ $\text{M}^+$ ]: 703.2032 found 703.2028; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3262, 1669, 1607, 1530, 1452, 1258, 1216, 1179.

**2,4-Di(thiophen-3-yl)- $N^1,N^5$ -bis(2-(trifluoromethoxy)phenyl)pentanediamide 4o.** Data of **4o**: White solid; mp 118–119 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.38 (d,  $J = 8.2$  Hz, 3.11H), 7.68 (s, 1.80H), 7.53 (s, 1.73H), 7.45 (dd,  $J = 4.8$  and 3.0 Hz, 1.57H), 7.40 (dd,  $J = 4.8$  and 3.0 Hz, 1.48H), 7.31–7.22 (m, 7.25H), 7.22–7.14 (m, 3.24H), 7.12 (d,  $J = 4.8$  Hz, 1.70H), 7.10–7.01 (m, 5.10H), 3.90 (t,  $J = 7.5$  Hz, 1.87H), 3.82 (t,  $J = 7.9$  Hz, 2H), 3.21–3.08 (m, 1H), 2.82 (t,  $J = 7.9$  Hz, 1.88H), 2.43–2.31 (m, 1H);  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )  $\delta$  170.9, 170.8, 138.6, 137.9, 130.5, 130.4, 127.9, 127.6, 127.52, 127.50, 126.7, 126.6, 124.3, 123.6, 123.1, 121.6, 121.5, 120.50, 120.45, 120.29 (q,  $J_{\text{C-F}} = 258.2$  Hz), 120.26 (q,  $J_{\text{C-F}} = 257.4$  Hz), 46.9, 46.8, 35.7, 35.4;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.88, -57.99; HRMS (ESI) Calcd for  $\text{C}_{27}\text{H}_{20}\text{F}_6\text{N}_2\text{O}_4\text{S}_2$  [ $\text{M}^+$ ]: 615.0847 found 615.0843; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3408, 1671, 1609, 1529, 1452, 1258, 1217, 1181.

**2,4-Bis(4-chlorophenyl)- $N^1,N^5$ -bis(2-(trifluoromethoxy)phenyl)pentanediamide 4q.** Data of **4q**: White solid; mp 132–134 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.32 (d,  $J = 8.1$  Hz, 4.03H), 7.46 (s, 2.69H), 7.44–7.33 (m, 10.66H), 7.27 (d,  $J = 6.3$  Hz, 14.29H), 7.22–7.14 (m, 4.54H), 7.13–7.03 (m, 4.67H), 3.62 (dd,  $J = 13.7$  and 7.4 Hz, 4.72H), 3.16–3.04 (m, 1H), 2.75 (t,  $J = 7.7$  Hz, 2.71H), 2.35–2.25 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.6, 170.5, 138.2 (d,  $J = 2.5$  Hz), 138.1 (d,  $J = 1.3$  Hz), 136.7, 136.2, 134.3, 134.1, 130.8, 130.2, 129.7, 129.53, 129.52, 129.3, 127.52, 127.50, 124.5, 123.6, 121.9, 121.7, 120.5, 120.3 (q,  $J_{\text{C-F}} = 258.1$  Hz), 120.2 (q,  $J_{\text{C-F}} = 257.9$  Hz), 50.9, 50.8, 36.4, 35.7;  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.86, -58.01; HRMS (ESI) Calcd for  $\text{C}_{31}\text{H}_{22}\text{Cl}_2\text{F}_6\text{N}_2\text{O}_4$  [ $\text{M}^+$ ]: 671.0939 found 671.0936; IR (KBr)  $\nu(\text{cm}^{-1})$ : 3424, 1636, 1616, 1529, 1493, 1257, 1217, 1180.

**meso-2,4-Bis(4-bromophenyl)- $N^1,N^5$ -bis(2-(trifluoromethoxy)phenyl)pentanediamide (meso-4r) and ( $\pm$ )-2,4-**

**Bis(4-bromophenyl)-*N*<sup>1</sup>,*N*<sup>5</sup>-bis(2-(trifluoromethoxy) phenyl) pentanediamide (±)-4r.** Data of (*meso*-4r): White solid; mp 167–171 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.32 (d, *J* = 8.1 Hz, 2H), 7.58–7.46 (m, 4H), 7.41 (s, 2H), 7.30–7.24 (m, 3H), 7.20 (d, *J* = 8.3 Hz, 5H), 7.14–7.05 (m, 2H), 3.60 (t, *J* = 7.5 Hz, 2H), 3.15–3.03 (m, 1H), 2.35–2.25 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.5, 138.2, 137.2136.8, 132.7, 132.5, 130.2, 129.9, 129.7, 127.5, 124.6, 122.3, 121.9, 120.5 (d, *J* = 1.0 Hz), 120.3 (q, *J*<sub>C-F</sub> = 258.2 Hz), 50.9, 36.4; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.84; HRMS (ESI) Calcd for C<sub>31</sub>H<sub>22</sub>Br<sub>2</sub>F<sub>6</sub>N<sub>2</sub>O<sub>4</sub>Na [M + Na<sup>+</sup>]: 780.9748 found 780.9740; IR (KBr) ν(cm<sup>-1</sup>): 3422, 1674, 1613, 1530, 1452, 1254, 1218, 1179. Data of (±)-4r: White solid; mp 152–155 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.31 (d, *J* = 8.0 Hz, 2H), 7.60–7.49 (m, 4H), 7.45 (s, 2H), 7.32–7.24 (m, 3H), 7.23–7.16 (m, 5H), 7.13–7.04 (m, 2H), 3.62 (t, *J* = 7.7 Hz, 2H), 2.74 (t, *J* = 7.7 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.5, 138.2, 136.8, 132.6, 130.2, 129.8, 127.5, 124.6, 122.4, 121.7, 120.5, 120.2 (q, *J*<sub>C-F</sub> = 257.6 Hz), 51.0, 35.6; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.99; HRMS (EI) Calcd for C<sub>31</sub>H<sub>22</sub>Br<sub>2</sub>F<sub>6</sub>N<sub>2</sub>O<sub>4</sub> [M<sup>+</sup>]: 757.9850 found 757.9849; IR (KBr) ν(cm<sup>-1</sup>): 3418, 1665, 1609, 1535, 1453, 1257, 1217, 1171.

**2,4-Bis(4-iodophenyl)-*N*<sup>1</sup>,*N*<sup>5</sup>-bis(2-(trifluoromethoxy) phenyl)pentanediamide 4s.** Data of 4s: White solid; mp 164–165 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.30 (d, *J* = 7.9 Hz, 3.79H), 7.79–7.66 (m, 7.81H), 7.44 (d, *J* = 9.1 Hz, 4.32H), 7.31–7.22 (m, 5.06H), 7.22–7.14 (m, 4.07H), 7.13–7.02 (m, 12.47H), 3.64–3.53 (m, 4.17H), 3.16–3.01 (m, 1H), 2.74 (t, *J* = 7.7 Hz, 2.16H), 2.33–2.24 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.43, 170.39, 138.6, 138.4, 138.2, 138.1, 137.9, 137.4, 130.2, 130.1, 129.9, 127.52, 127.50, 124.6, 121.9, 121.7, 120.5, 120.33 (q, *J*<sub>C-F</sub> = 258.9 Hz), 120.29 (q, *J*<sub>C-F</sub> = 258.4 Hz), 93.9, 93.7, 51.1, 51.0, 36.2, 35.5; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.81, -57.97; HRMS (ESI) Calcd for C<sub>31</sub>H<sub>22</sub>F<sub>6</sub>I<sub>2</sub>N<sub>2</sub>O<sub>4</sub> [M<sup>+</sup>]: 854.9652 found 854.9644; IR (KBr) ν(cm<sup>-1</sup>): 3415, 1670, 1608, 1530, 1452, 1256, 1217, 1179.

**2,4-Bis(3-fluorophenyl)-*N*<sup>1</sup>,*N*<sup>5</sup>-bis(2-(trifluoromethoxy) phenyl) pentanediamide 4t.** Data of 4t: White solid; mp 130–131 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.34 (d, *J* = 8.2 Hz, 2.53H), 7.49 (s, 1.40H), 7.45–7.32 (m, 4.46H), 7.31–7.23 (m, 5.42H), 7.22–7.15 (m, 2.64H), 7.14–6.99 (m, 10.61H), 3.74–3.62 (m, 3.02H), 3.18–3.08 (m, 1H), 2.77 (t, *J* = 7.7 Hz, 1.41H), 2.39–2.29 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.4, 170.3, 164.6, 164.5, 162.1, 162.0, 140.7, 140.6, 140.3, 140.2, 138.1, 131.15, 131.07, 131.0, 130.9, 130.3, 127.5, 124.5, 123.93, 123.90, 123.81, 123.78, 121.8, 121.7, 120.5, 120.3 (q, *J*<sub>C-F</sub> = 258.0 Hz), 120.2 (q, *J*<sub>C-F</sub> = 257.7 Hz), 115.4, 115.3, 115.2, 115.1, 114.9, 51.2, 36.1, 35.6; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.96, -58.08, -111.06, -111.25; HRMS (ESI) Calcd for C<sub>31</sub>H<sub>22</sub>F<sub>8</sub>N<sub>2</sub>O<sub>4</sub> [M<sup>+</sup>]: 639.1530 found 639.1528; IR (KBr) ν(cm<sup>-1</sup>): 3421, 1668, 1611, 1530, 1451, 1261, 1217, 1173.

**2,4-Bis(3-bromophenyl)-*N*<sup>1</sup>,*N*<sup>5</sup>-bis(2-(trifluoromethoxy) phenyl)pentanediamide 4u.** Data of 4u: Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.33 (d, *J* = 8.2 Hz, 3.54H), 7.62–7.39 (m, 11.71H), 7.36–7.23 (m, 12.50H), 7.23–7.15 (m, 3.95H), 7.14–7.03 (m, 4.12H), 3.70 (t, *J* = 7.7 Hz, 2H), 3.62 (t, *J* = 7.4 Hz, 2.04H), 3.19–3.05 (m, 1H), 2.72 (t, *J* = 7.7 Hz, 2.04H), 2.39–2.28 (m, 1H); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 170.34, 170.26, 140.4, 140.2, 138.2, 131.4, 131.3, 131.2, 131.0, 130.9, 130.8, 130.20, 130.16, 127.5, 126.7, 124.6, 123.5, 123.4, 121.9, 121.8, 120.5, 120.3 (q, *J*<sub>C-F</sub> = 258.1 Hz), 120.2 (q, *J*<sub>C-F</sub> = 258.0 Hz), 51.1, 36.2, 35.9; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.87, -57.99. HRMS (ESI) Calcd for C<sub>31</sub>H<sub>22</sub>F<sub>6</sub>Br<sub>2</sub>N<sub>2</sub>O<sub>4</sub> [M<sup>+</sup>]: 758.9929 found 758.9924; IR (KBr) ν(cm<sup>-1</sup>): 3417, 1677, 1609, 1527, 1453, 1256, 1215, 1181.

***N*<sup>1</sup>,*N*<sup>5</sup>-bis(2-(trifluoromethoxy)phenyl)-2,4-bis(3-(trifluoromethoxy)phenyl)pentanediamide 4v.** Data of 4v: White solid; mp 100–101 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.33 (d, *J* = 8.2 Hz, 3.98H), 7.55–7.38 (m, 8.78H), 7.35–7.23 (m, 10.71H), 7.23–7.14 (m, 10.77H), 7.14–7.04 (m, 4.40H), 3.73 (t, *J* = 7.7 Hz, 2.36H), 3.67 (t, *J* = 7.4 Hz, 2H), 3.21–3.07 (m, 1H), 2.77 (t, *J* = 7.7 Hz,

2.36H), 2.42–2.30 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.2, 170.1, 149.9, 140.4, 140.2, 138.2, 130.9, 130.8, 130.1, 127.5, 126.4, 126.3, 124.7, 121.9, 121.8, 120.9, 120.6, 120.5, 120.36 (q, *J*<sub>C-F</sub> = 256.1 Hz), 120.35 (q, *J*<sub>C-F</sub> = 256.1 Hz), 120.3 (q, *J*<sub>C-F</sub> = 257.8 Hz), 120.2 (q, *J*<sub>C-F</sub> = 257.8 Hz), 51.2, 36.4, 36.0; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.90, -57.94, -58.08, -58.19; HRMS (ESI) Calcd for C<sub>33</sub>H<sub>22</sub>F<sub>12</sub>N<sub>2</sub>O<sub>6</sub> [M<sup>+</sup>]: 771.1365 found 771.1369; IR (KBr) ν(cm<sup>-1</sup>): 3420, 1665, 1610, 1532, 1454, 1265, 1217, 1172.

***N*<sup>1</sup>,*N*<sup>5</sup>-bis(2-(trifluoromethoxy)phenyl)-2,4-bis(3-(trifluoromethylthio)phenyl)pentanediamide 4w.** Data of 4w: White solid; mp 90–91 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.32 (d, *J* = 7.8 Hz, 4.26H), 7.73–7.56 (m, 9.14H), 7.55–7.39 (m, 14.37H), 7.32–7.23 (m, 5.25H), 7.23–7.15 (m, 4.52H), 7.14–7.04 (m, 4.55H), 3.75 (t, *J* = 7.6 Hz, 2.58H), 3.68 (t, *J* = 7.4 Hz, 2H), 3.22–3.10 (m, 1H), 2.78 (t, *J* = 7.6 Hz, 2.54H), 2.44–2.32 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.2, 170.1, 139.8, 139.6, 138.2, 137.3, 136.0, 135.9, 135.7, 130.54, 130.50, 130.43, 130.37, 130.12, 130.08, 129.4 (q, *J*<sub>C-F</sub> = 306.0 Hz), 127.5, 125.8, 124.7, 122.0, 121.9, 120.5, 120.3 (q, *J*<sub>C-F</sub> = 258.2 Hz), 120.2 (q, *J*<sub>C-F</sub> = 257.9 Hz), 51.1, 36.7, 36.2; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -42.54, -42.63, -58.01, -58.13; HRMS (ESI) Calcd for C<sub>33</sub>H<sub>22</sub>F<sub>12</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub> [M<sup>+</sup>]: 803.0908 found 803.0913; IR (KBr) ν(cm<sup>-1</sup>): 3272, 1672, 1611, 1534, 1454, 1257, 1220, 1168.

***N*<sup>1</sup>,*N*<sup>5</sup>-bis(2-(difluoromethoxy)phenyl)-2,4-bis(3-(trifluoromethoxy)phenyl)pentanediamide 4x.** Data of 4x: White solid; mp 86–87 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.29 (d, *J* = 8.2 Hz, 3.01H), 7.57 (s, 1.70H), 7.51 (s, 1.51H), 7.48–7.37 (m, 3.61H), 7.35–7.25 (m, 6.05H), 7.24–7.15 (m, 9.08H), 7.13–7.03 (m, 6.09H), 6.37 (t, *J* = 73.3 Hz, 1.78H), 6.30 (t, *J* = 73.2 Hz, 2H), 3.73 (t, *J* = 7.7 Hz, 2H), 3.62 (t, *J* = 7.5 Hz, 1.79H), 3.15–3.05 (m, 1H), 2.73 (t, *J* = 7.7 Hz, 1.78H), 2.42–2.31 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.2, 170.1, 149.8, 140.6, 140.5, 140.4, 130.74, 130.67, 129.6, 126.4, 126.3, 126.21, 126.19, 124.72, 124.67, 121.85, 121.7, 120.8, 120.58, 120.57 (q, *J*<sub>C-F</sub> = 257.3 Hz), 120.55 (q, *J*<sub>C-F</sub> = 257.8 Hz), 120.4, 119.1, 118.9, 118.8, 116.11 (t, *J*<sub>C-F</sub> = 260.0 Hz), 116.06 (t, *J*<sub>C-F</sub> = 260.1 Hz), 51.24, 51.18, 36.6, 36.5; <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -57.80, -57.85, -80.27, -80.30, -80.32, -80.35; HRMS (ESI) Calcd for C<sub>33</sub>H<sub>24</sub>F<sub>10</sub>N<sub>2</sub>O<sub>6</sub> [M<sup>+</sup>]: 735.1553 found 735.1552; IR (KBr) ν(cm<sup>-1</sup>): 3287, 1660, 1609, 1530, 1455, 1262, 1217, 1135.

## Acknowledgements

Financial support from “Hundred Talents Project” of Chinese Academy of Sciences, “High-end Science and Technology Talents Program” of Yunnan Province (2011HA008), the National Natural Science Foundation of China (No. 21472198), Grant (2014FA039) from Yunnan Province of China and Key Projects of Technological Innovation of Hubei Province of China (No. 2016ACA138) is gratefully acknowledged.

## Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.tet.2017.05.017>.

## References

- Gersch M, Kreuzer J, Sieber SA. *Nat Prod Rep.* 2012;29:659–682.
- (a) Rando R. *Nat New Biol.* 1972;237:53; (b) Johansson MH. *Mini-Rev Med Chem.* 2012;12:1330; (c) Van DWR, Strauss E. *J Am Chem Soc.* 2010;132:12853–12855; (d) Furman JL, Kang M, Choi S, et al. *J Am Chem Soc.* 2014;136:8411–8417.
- Wang F, Chen C, Tan W, Yang K, Yang H. *Sci Rep.* 2016;6:22677.
- Santos MM, Moreira R. *Mini-Rev Med Chem.* 2007;7:1040.
- Volla CM, Atodiresel I, Rueping M. *Chem Rev.* 2014;114:2390–2431.
- (a) Jusseau X, Chabaud L, Guillou C. *Tetrahedron.* 2014;70:2595–2615; (b) Streuff J, Gansauer A. *Angew Chem Int Ed.* 2015;54:14232–14242; (c) Lowe MA, Ostovar M, Ferrini S, et al. *Angew Chem Int Ed.* 2011;50:

- 6370–6374.
7. (a) Almaši D, Alonso DA, Nájera C. *Tetrahedron Asymmetry*. 2007;18:299–365;  
(b) Kaur J, Chauhan P, Chimni SS. *Org Biomol Chem*. 2016;14:7832–7847;  
(c) Enders D, Narine AA, Toulgoat F, Bisschops T. *Angew Chem Int Ed*. 2008;47:5661–5665;  
(d) Cheng J, Huang Z, Chi YR. *Angew Chem Int Ed*. 2013;52:8592–8596;  
(e) Kano T, Shirozu F, Akakura M, Maruoka K. *J Am Chem Soc*. 2012;134:16068–16073;  
(f) Alaniz JRD, Rovis T. *J Am Chem Soc*. 2005;127:6284.
  8. (a) Nicolaou KC, Edmonds DJ, Bulger PG. *Angew Chem Int Ed*. 2006;45:7134–7186;  
(b) Brimble M, Rathwell K. *Synthesis*. 2007:643–662;  
(c) Trost BM, Dong G, Vance JA. *J Am Chem Soc*. 2007;129:4540–4541.
  9. (a) Epstein WW, Sweat FW. *Chem Rev*. 1967;67:247–260;  
(b) Martin D, Weise A, Niclas HJ. *Angew Chem Int Ed*. 1967;6:318–334;  
(c) Mancuso AJ, Swern D. *Synthesis*. 1981:165–185;  
(d) Tidwell TT. *Synthesis*. 1990:857–870.
  10. Omura K, Sharma AK, Swern D. *J Org Chem*. 1976;41:957–962.
  11. Pfitzner KE, Moffatt JG. *J Am Chem Soc*. 1963;85:3027–3028.
  12. (a) Wu X-F, Natte K. *Adv Synth Catal*. 2016;358:336–352;  
(b) Magolan J, Jones-Mensah E, Karki M. *Synthesis*. 2016;48:1421–1436.
  13. (a) Sun WW, Cao P, Mei RQ, Li Y, Ma YL, Wu B. *Org Lett*. 2014;16:480–483;  
(b) Zhang SJ, Sun WW, Cao P, Dong XP, Liu JK, Wu B. *J Org Chem*. 2016;81:956–968.
  14. (a) Pummerer R. *Ber.Dtsch Chem Gas*. 1909;42:2282;  
(b) Pummerer R. *Ber Dtsch Chem Gas*. 1910;43:1401;  
(c) Bur SK, Padwa A. *Chem Rev*. 2004;104:2401–2432;  
(d) Smith LH, Coote SC, Sneddon HF, Procter DJ. *Angew Chem Int Ed*. 2010;49:5832–5844.
  15. Kiselyov AS, Piatnitski EL, Doody J. *Org Lett*. 2004;6:4061.
  16. Lanigan RM, Starkov P, Sheppard TD. *J Org Chem*. 2013;78:4512–4523.
  17. Ni J, Li J, Fan Z, Zhang A. *Org Lett*. 2016;18:5960–5963.
  18. Ilies L, Matsubara T, Ichikawa S, Asako S, Nakamura E. *J Am Chem Soc*. 2014;136:13126–13129.