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## **Supporting Information**

### **Alternating Current Enabled of para-Selective**

### C(sp<sup>2</sup>)–H/N–H Cross-Coupling of Aniline

Zhaoliang Yang<sup>a</sup><sup>§</sup>\*, Haiyan Du<sup>a</sup><sup>§</sup>, Yuan Zhou<sup>a</sup>, Mingming Yu<sup>c</sup> and Jianye Zhang<sup>b\*</sup>

<sup>a</sup> National Engineering Research Center for Carbohydrate Synthesis, Jiangxi Province Key Laboratory of Natural and Biomimetic Drugs Research, College of Chemistry and Materials, Jiangxi Normal University, Nanchang, 330022, P. R. China;
<sup>b</sup> Jiangxi Provincial Key Laboratory of Drug Design and Evaluation, School of Pharmacy, Jiangxi Science & Technology Normal University, Nanchang 330013, P. R. China;
<sup>c</sup> School of Chemistry and Chemical Engineering, Zhejiang Sci-Tech University, Xiasha West Higher Education District, Hangzhou, 310018, China
<sup>§</sup> These authors contributed equally to this work
\* Corresponding author: zhaoliangyang@jxnu.edu.cn; jianyezhang1222@qq.com

### **General Information**

All manipulations were carried out by standard Schlenk techniques. Unless otherwise stated, analytical grade solvents and commercially available reagents were used to conduct the reactions. Most substrates were derived from commercially available reagents, such as N-Boc-aniline was purchased from LeYan, shanghai, China.; the drying acetonitrile used was from Adamas-beta®; 1,1,1,3,3,3-Hexafluoro-2-propanol was purchased from Energy Chemical®; 4-(trifluoromethyl) pyridine was purchased from Macklin®. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in dichloromethane (bp. 39.8 °C). Gradient flash chromatography was conducted and eluted with a continuous gradient from petroleum to ethyl acetate. All the new compounds were characterized by <sup>1</sup>H NMR, <sup>13</sup>C NMR, <sup>19</sup>F NMR and HRMS. The known compounds were characterized by <sup>1</sup>H NMR, <sup>13</sup>C NMR and <sup>19</sup>F NMR. The <sup>1</sup>H NMR, <sup>13</sup>C NMR and <sup>19</sup>F NMR spectra were recorded on a Bruker 400 MHz NMR spectrometer. The chemical shifts ( $\delta$ ) were given in part per million relative to Chloroform-d (7.26 ppm for <sup>1</sup>H NMR), Chloroform-d (77.16 ppm for <sup>13</sup>C NMR), DMSO-d<sub>6</sub> (2.50 ppm for <sup>1</sup>H NMR), DMSO-*d*<sub>6</sub> (39.52 ppm for <sup>13</sup>C NMR). High resolution mass spectra (HRMS) were measured with a Waters Micromass GCT Premier. Electrolysis experiments were performed using a dual display potentiostat (DJS-292B) or galvanostat (made in China). The electrode was graphite rod ( $\phi$  6 mm, hard). Cyclic voltammograms (CV) were obtained on a CHI 660E potentiostat.

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## **1. Experimental Section**

#### 1.1 Optimization of reaction conditions

#### Scheme S1 Optimization of reaction conditions

(A) Attempts at reactions under different electrolytic conditions



#### (B) Different electrolyte



#### (C) Different bases

Ĺ	NHBoc -	$\begin{array}{c} \text{AC: CR   CR} \\ \hline \text{TBAOAc (0.15 mmol)} \\ \text{CH}_3\text{CN (6.0 mL)} \\ 10 \text{ mA, 5 h} \\ f = 1/4 \text{ Hz} \\ \end{array} \qquad \textbf{4}$	HBoc
	Entry	Additive	Yield (%) <sup>[a]</sup>
	1	4-CF <sub>3</sub> Py (0.6 mmol)	40
	2	4-(dimethylamino)pyridine (0.6 mmol)	26
	3	4-cyanopyridine (0.6 mmol)	36
	4	Pyridine (0.6 mmol)	25
	6	KOAc (0.6 mmol)	42
	7	NaOAc (0.6 mmol)	38
	8	K <sub>2</sub> CO <sub>3</sub> (0.6 mmol)	30
	9	Cs <sub>2</sub> CO <sub>3</sub> (0.6 mmol)	32
	10	4-CF <sub>3</sub> Py (0.6 mmol) and KOAc (0.6 mmol)	53

<sup>[a]</sup> Yield of NMR, with 1,3,5-trimethylbenzene as internal standard.

#### (D) Different solvent

NHBoc 1	AC: CR   CR 4-CF <sub>3</sub> Py( (0.6 mmol)), KOAc (0.6 mmol)) TBAOAc (0.15 mmol) CH <sub>3</sub> CN (6.0 mL) 10 mA, 5 h f = 1/4 Hz 4	NHBoc
Entry	Solvent	Yield (%) <sup>[a]</sup>
1	CH <sub>3</sub> CN (6.0 mL)	53
2	DMF (6.0 mL)	25
3	Acetone (6.0 mL)	39
4	CH <sub>3</sub> CN: HFIP (6.0 : 0.1 mL)	68
5	CH <sub>3</sub> CN: HFIP (6.0 : 0.2 mL)	55
6	CH <sub>3</sub> CN: HFIP (6.0 mL : 63 µL)	71

<sup>[a]</sup> Yield of NMR, with 1,3,5-trimethylbenzene as internal standard.

#### (E) Different frequency and current

NHBoc 1	AC : CR   CR 4-CF <sub>3</sub> Py (0.6 mmol), KOAc (0.6 mmol) <u>TBAOAc ((0.15 mmol))</u> HFIP (63 µL) CH <sub>3</sub> CN (6.0 mL) 10 mA, 5 h	NHBoc
	ſ	
Entry	Frequency and Current	Yield (%) <sup>[a]</sup>
1	<i>f</i> = 1/ 2 Hz	70
2	<i>f</i> = 1/ 4 Hz	71
3	<i>f</i> = 1/ 8 Hz	82
4	<i>f</i> = 1/ 12 Hz	45
5	3 V, f = 50 Hz, square wave	70
6	3 V, $f$ = 50 Hz, sine wave	69
7	15 mA, 3 h 15 min	48
8	12 mA, 4 h	62
9	8 mA, 6 h	75
10	6 mA, 8 h	78
11	DC	trace

<sup>[a]</sup> Yield of NMR, with 1,3,5-trimethylbenzene as internal standard.

#### (E) Different additives.

	AC: CR   CR 4-CF <sub>3</sub> Py(0.6 mmol) KOAc (0.6 mmol) TBAOAc (0.15 mmol) HFIP (63 µL), CH <sub>3</sub> CN (6.0 mL) 10 mA.5 h		**N-Boc
·	f = 1/4 Hz	-	
En	ry Variation from standard of	conditions yield (%	o) <sup>[a]</sup>
1	none	82	
2	KOAc (0.3 mmc	ol) 65	
3	KOAc (0.9 mmc	ol) 50	
4	CH <sub>3</sub> CN: MeOH (6.0	: 63 µL) trace	e
5	CH <sub>3</sub> CN: EtOH (6.0 :	63 μL) trace	e
6	CH <sub>3</sub> CN: H <sub>2</sub> O (6.0 :	63 μL) 10	
[a] Yield	l of NMR, with 1,3,5-trimethylbenzene as ir	nternal standard.	

Standard conditions: 1 (0.3 mmol), 4-CF<sub>3</sub>Py (0.6 mmol), TBAOAc (0.15 mmol), HFIP (0.6 mmol), KOAc (0.6 mmol), CH<sub>3</sub>CN (6.0 mL), carbon rods as anode and cathode, undivided cell, I =

10.0 mA, f = 1/8 Hz under N<sub>2</sub> at room temperature for 5 h. r.t = room temperature, adjust the room temperature at about 25 °C.

#### 1.2 General procedure for cyclic voltammetry experiments

Scheme S2 Cyclic voltammetry experiments General procedure for cyclic voltammetry (CV) experiment



An undivided three-electrode cell equipped with a stir bar was charged with a degassed solution of **1** or **4** (0.15 mmol), TBABF<sub>4</sub> (1 mmol), KOAc (0.3 mmol), and HFIP (0.3 mmol) in MeCN (6.0 mL), which was purged with  $N_2$  at 50 mL/min for 5 min prior to electrolysis using a glassy carbon working electrode, platinum plate counter electrode, and Ag/AgCl (1 M KCl aqueous) reference electrode, with cyclic voltammetry performed at 0.1 V/s under static (non-stirred) conditions.

#### 1.3 General procedure for kinetic experiments

Scheme S3 General procedure for Kinetic experiments

(A) The Kinetic curve of Electricity



An oven-dried, undivided three-necked flask (10 mL) equipped with a stir bar and two carbon rod electrodes ( $\phi = 6$  mm) was charged with a solution of **1** (0.3 mmol, 62.1 mg), KOAc (0.6 mmol, 30 mg), TBAOAc (0.15 mmol, 45 mg), 4-(trifluoromethyl)pyridine (0.6 mmol, 70 µL) and HFIP (0.6 mmol, 63 µL) in CH<sub>3</sub>CN (6 mL). The mixture was stirred under a nitrogen atmosphere and 6

electrolyzed at a frequency (*f*) of 1/8 Hz with different currents at room temperature. The reaction was stopped after one hour and the product was quantitatively analyzed by NMR. (**B**) The Kinetic curve of Substrate 1



An oven-dried, undivided three-necked flask (10 mL) equipped with a stir bar and two carbon rod electrodes ( $\phi = 6$  mm) was charged with a solution of KOAc (0.6 mmol, 30 mg), TBAOAc (0.15 mmol, 45 mg), 4-(trifluoromethyl)pyridine (0.6 mmol, 70 µL), HFIP (0.6 mmol, 63 µL) and substrates **1** with different concentrations in CH<sub>3</sub>CN (6 mL). The mixture was stirred under a nitrogen atmosphere and electrolyzed at a frequency (*f*) of 1/8 Hz with a constant current of 10.0 mA at room temperature. The reaction was stopped after one hour and the product was quantitatively analyzed by NMR.

#### 1.4 General procedure for concentration-Time experiments

Scheme S4 Concentration-Time experiments between AC condition and DC conditionA) Concentration-Time experiments of the AC condition



General procedure for Concentration-Time experiments of the AC condition:

An oven-dried, undivided three-necked flask (10 mL) equipped with a stir bar and two carbon rod electrodes ( $\phi = 6$  mm) was charged with a solution of 1 (0.3 mmol, 62.1 mg), KOAc (0.6 mmol, 30 mg), TBAOAc (0.15 mmol, 45 mg), 4-(trifluoromethyl)pyridine (0.6 mmol, 70 µL), HFIP (0.6 mmol, 63 µL), and mesitylene (0.15 mmol, 18 mg) in CH<sub>3</sub>CN (6 mL). The mixture was stirred under a nitrogen atmosphere and electrolyzed at a frequency (*f*) of 1/8 Hz with a constant current of 10.0 mA at room temperature. Aliquots were quantitatively sampled via NMR at specified time intervals. **B**) Concentration-Time experiments of the DC condition 7



General procedure for Concentration-Time experiments of the DC condition:

An oven-dried, undivided three-necked flask (10 mL) equipped with a stir bar and two carbon rod electrodes ( $\phi = 6$  mm) was charged with a solution of 1 (0.3 mmol, 62.1 mg), KOAc (0.6 mmol, 30 mg), TBAOAc (0.15 mmol, 45 mg), 4-(trifluoromethyl)pyridine (0.6 mmol, 70 µL), HFIP (0.6 mmol, 63 µL), and mesitylene (0.15 mmol, 18 mg) in CH<sub>3</sub>CN (6 mL). The mixture was stirred under a nitrogen atmosphere and electrolyzed at a constant current of 10.0 mA at room temperature. Aliquots were quantitatively sampled via NMR at specified time intervals.

#### 1.5 General procedure for control experiments



Control experiments were conducted to evaluate the reaction under different electrolysis modes. Prior to electrolysis, the electrode surface showed no deposits and the reaction solution remained clear. Under AC conditions, post-electrolysis analysis revealed: (1) the electrode surface maintained its cleanliness without observable deposits, (2) the reaction solution turned light yellow with slight turbidity. In contrast, DC conditions resulted in: (1) significant anode surface deposition, (2) a dark brown, highly turbid solution. TLC monitoring demonstrated distinct reaction outcomes: the AC system primarily contained residual substrate **1a** and product **2**, whereas the DC system showed

Scheme S5 Control experiments between AC condition and DC condition

complete consumption of **1a** with only trace amounts of **2**. These results suggest that excessive oxidation under DC conditions likely prevents target product formation.

#### 1.6 General procedure for free radical inhibition experiments

Scheme S6 Free radical inhibition experiments

(A) TEMPO as free radical inhibitor



An oven-dried, undivided three-necked flask (10 mL) equipped with a stir bar and two carbon rod electrodes ( $\phi = 6$  mm) was charged with a solution of **1** (0.3 mmol), KOAc (0.6 mmol), TBAOAc (0.15 mmol), 4-(trifluoromethyl)pyridine (0.6 mmol), HFIP (0.6 mmol) and TEMPO (0.3 mmol) in CH<sub>3</sub>CN (6 mL). The mixture was stirred under a nitrogen atmosphere and electrolyzed at a constant current of 10.0 mA for 5 hours at room temperature.

(B) BHT as free radical inhibitor



An oven-dried, undivided three-necked flask (10 mL) equipped with a stir bar and two carbon rod electrodes ( $\phi = 6$  mm) was charged with a solution of **1** (0.3 mmol), KOAc (0.6 mmol), TBAOAc (0.15 mmol), 4-(trifluoromethyl)pyridine (0.6 mmol), HFIP (0.6 mmol) and BHT (0.3 mmol) in CH<sub>3</sub>CN (6 mL). The mixture was stirred under a nitrogen atmosphere and electrolyzed at a constant current of 10.0 mA for 5 hours at room temperature.

#### 1.7 General procedure for electron paramagnetic resonance (EPR) experiments

Scheme S7 Electron Paramagnetic Resonance (EPR) experiments





In dry three-necked flasks (10 ml), equipped with stirrers with two carbon rods ( $\phi$ =6 mm). *N*-Boc aniline (0.3 mmol), 4-trifluoromethyl pyridine (0.6 mmol), potassium acetate (0.6 mmol), tetrabutyl ammonium acetate (0.15 mmol) acetonitrile (6 mL) and hexafluoroisopropanol (63 µL) were added under nitrogen atmosphere, the solution was electrolyzated at a constant current of 10.0 mA at 1/8Hz for 1 h, then DMPO (30 µL) was added to the reaction tube for 2 min, and the mixture was put into a simple melting point tube for EPR testing. EPR spectra were recorded at room temperature on a Bruker A200 spectrometer operating at 9.823 GHz. Typical spectrometer parameters are as follows: scan width: 100.00G; Center group :3505.08 G; Conversion time :30.00 ms; Time constant :163.84 ms; Scanning time :30.72 s; Modulation amplitude :1.00 G; Modulation frequency :9.81 GHz; Receiver gain :10×104; Microwave power :2.14 mW; Attenuator :10db; Number of X-scans :10. DMPO captured carbon-centric radicals were detected g= 2.0073, A<sub>N</sub> = 14.6 G, A<sub>H</sub> = 20.9 G.

#### 1.8 General procedure for electrochemical ESI-MS experiments.

 Image: NHBoc
 -e<sup>-</sup>
 Image: NHBoc
 -H<sup>+</sup>
 Image: NHBoc
 N

*N*-Boc-aniline (0.006 mmol), 4-(trifluoromethyl)pyridine (0.024 mmol), potassium acetate (0.024 mmol), and lithium acetate (0.06 mmol) were weighed into a polyethylene (PE) tube and dissolved in acetonitrile (6 mL) with ultrasonic mixing. A 30  $\mu$ L aliquot of the solution was transferred into a capillary using a pipette, followed by insertion of two platinum wire electrodes. The electrostatic spray conditions were set to: frequency = 385 Hz, amplitude = 7.4 Vpp, offset = 3.9 Vdc, generating an electric field of ~7 kV at 60,000 resolution. Initial detection was conducted under zero-current conditions for 2 min, followed by continuous electrolysis at 1 mA (5 Hz AC) for an additional 2 min.

#### 1.9 General procedure for scale-up reaction



An oven-dried, undivided three-necked flask (20 mL) equipped with a stir bar and two carbon rod electrodes ( $\phi = 6$  mm) was charged with a solution of **1** (0.6 mmol), KOAc (1.2 mmol), TBAOAc

Scheme S8 Electrochemical ESI-MS experiments.

(0.3 mmol), 4-(trifluoromethyl)pyridine (1.2 mmol), HFIP (1.2 mmol) in CH<sub>3</sub>CN (12 mL). The mixture was stirred under a nitrogen atmosphere and electrolyzed at a constant current of 10.0 mA for 10 hours at room temperature. After completion of the reaction, it was quenched by H<sub>2</sub>O. The aqueous solution was extracted with EA ( $3 \times 5$  mL) and the combined extracts were dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>. The solvent was removed under reduced pressure by rotary evaporation. Then, the crude product was purified by column chromatography on silica gel (200–300 mesh) using PE/EA (15/1, v/v) as the eluent, affording **4** (260 mg, 63% yield).

#### 1.10 Incompatible substrate



### 2. Characterization of Products



*Tert-butyl (4-((tert-butoxycarbonyl)amino)phenyl)(phenyl)carbamate* (2). The product 2 was obtained as a white solid in 57% (33 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.33–7.26 (m, 4H), 7.22–7.18 (m, 2H), 7.15–7.10 (m, 3H), 6.55 (s, 1H), 1.51 (s, 9H), 1.44 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  153.9, 152.7, 143.1, 138.0, 136.1, 128.6, 127.8, 126.6, 125.4, 118.8, 81.1, 80.6, 28.4, 28.3. HRMS (ESI, m/z): calcd for C<sub>22</sub>H<sub>29</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 385.2122; Found: 385.2123.



*Tert-butyl (4-((tert-butoxycarbonyl)amino)phenyl-2,3,5,6-d4)(phenyl-d5)carbamate* (3). The product **3** was obtained as a white solid in 46% (27 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  6.52 (s, 1H), 1.51 (s, 9H), 1.44 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  154.02, 152.83, 143.07, 138.00, 136.03, 128.23 (m), 127.45 (m), 126.30 (m), 124.99 (m), 118.48 (m), 81.20, 80.72, 28.46, 28.38. HRMS (ESI, m/z): calcd for C<sub>22</sub>H<sub>20</sub>D<sub>9</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 394.2687; Found: 394.2682.



*Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-methylphenyl)(m-tolyl)carbamate* (4). The product 4 was obtained as a white solid in 74% (46 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.34–7.30 (m, 1H), 7.15–7.10 (m, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 7.00–6.97 (m, 1H), 6.89 (d, *J* = 7.5 Hz, 1H), 6.57 (s, 1H), 2.28 (s, 3H), 2.19 (s, 3H), 1.52 (s, 9H), 1.42 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  154.0, 152.9, 142.5, 138.3, 137.4, 136.9, 136.4, 129.5, 128.3, 125.5, 125.3, 121.9, 120.5, 116.8, 81.0, 80.7, 28.4, 28.3, 21.5, 18.2. HRMS (ESI, m/z): calcd for C<sub>24</sub>H<sub>33</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 413.2435; Found: 413.2440.



*Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-ethylphenyl)(3-ethylphenyl)carbamate* (5). The product 5 was obtained as a colorless oil in 51% (34 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.30 (d, J = 2.5 Hz, 1H), 7.21–7.17 (m, 1H), 7.16–7.07 (m, 3H), 7.01–6.97 (m, 1H), 6.92–6.88 (m, 1H), 6.55 (s, 1H), 2.61–2.46 (m, 4H), 1.52 (s, 9H), 1.42 (s, 9H), 1.18 (t, J = 7.6 Hz, 3H), 1.11 (t, J = 7.5 Hz, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  154.2, 152.8, 144.6, 142.8,

142.5, 137.7, 135.7, 130.1, 128.3, 124.1, 123.9, 121.9, 118.8, 116.6, 80.8, 80.6, 29.0, 28.5, 28.4, 24.2, 15.7, 14.0. HRMS (ESI, m/z): calcd for C<sub>26</sub>H<sub>37</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 441.2748; Found: 413.2452.

Tert-butyl

#### (4-((tert-butoxycarbonyl)amino)-2-(methoxymethyl)phenyl)(3

(*methoxymethyl*)*phenyl*)*carbamate* (6). The product 6 was obtained as a colorless oil in 70% (49 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.44–7.40 (m, 1H), 7.40–7.35 (m, 1H), 7.25–7.19 (m, 2H), 7.12–7.04 (m, 3H), 6.64 (s, 1H), 4.41–4.21 (m, 4H), 3.33 (d, *J* = 4.9 Hz, 6H), 1.51 (s, 9H), 1.41 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  153.7, 152.8, 142.6, 138.7, 137.8, 137.0, 134.9, 129.9, 128.6, 124.0, 118.2, 118.1, 81.2, 80.8, 74.5, 70.5, 58.6, 58.2, 28.4, 28.3. HRMS (ESI, m/z): calcd for C<sub>26</sub>H<sub>36</sub>N<sub>2</sub>NaO<sub>6</sub><sup>+</sup>, [M+Na]<sup>+</sup>: 495.2466; Found: 495.2475.

#### Tert-butyl

#### (4-((tert-butoxycarbonyl)amino)-2-(methylthio)phenyl)(3-

*(methylthio)phenyl)carbamate* (7). The product 7 was obtained as a yellow oil in 51% (36.5 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.40 (s, 1H), 7.24 (t, *J* = 2.0 Hz, 1H), 7.15 (t, *J* = 7.9 Hz, 1H), 7.09–7.03 (m, 3H), 7.00–6.96 (m, 1H), 6.59 (s, 1H), 2.42 (s, 6H), 1.52 (s, 9H), 1.43 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  153.5, 152.7, 142.7, 139.7, 138.6, 138.4, 134.6, 129.9, 128.7, 123.3, 123.1, 121.9, 115.4, 115.3, 81.4, 80.9, 28.5, 28.3, 16.1, 15.3. HRMS (ESI, m/z): calcd for C<sub>24</sub>H<sub>33</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub><sup>+</sup>, [M+H]<sup>+</sup>: 477.1876; Found: 477.1872



*Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-ethynylphenyl)(3-ethynylphenyl)carbamate* (8). The product **8** was obtained as a colorless oil in 58% (38 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.59 (d, J = 2.6 Hz, 1H), 7.40–7.37 (m, 1H), 7.34 (dd, J = 8.7, 2.6 Hz, 1H), 7.30–7.26 (m, 1H), 7.24–7.19 (m, 2H), 7.07 (d, J = 8.7 Hz, 1H), 6.60 (s, 1H), 3.15 (s, 1H), 3.02 (s, 1H), 1.51 (s, 9H), 1.42 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  153.49, 152.59, 142.65, 139.10, 137.53, 129.60, 129.02, 128.82, 128.55, 126.25, 123.04, 122.81, 122.44, 119.80, 83.44, 81.75, 81.58, 81.16, 80.46, 77.37, 28.42, 28.23. HRMS (ESI, m/z): calcd for C<sub>26</sub>H<sub>29</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 433.2122; Found: 433.2129



*Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-fluorophenyl)(3-fluorophenyl)carbamate* (9). The product 9 was obtained as a white solid in 43% (27 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.43 (dd, J = 12.2, 2.4 Hz, 1H), 7.24–7.17 (m, 1H), 7.05 (d, J = 8.5 Hz, 1H), 7.03–6.93 (m, 3H), 6.85–6.79 (m, 1H), 6.66 (s, 1H), 1.52 (s, 9H), 1.43 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  162.6 (d, J = 245.4 Hz), 158.5 (d, J = 248.8 Hz), 153.3, 152.3, 143.9 (d, J = 9.9 Hz), 139.1 (d, J = 10.8 Hz), 129.9, 129.5 (d, J = 9.3 Hz), 124.5 (d, J = 13.2 Hz), 120.8, 113.9, 112.7 (d, J = 24.2 Hz), 112.1 (d, J = 21.1 Hz), 106.5 (d, J = 25.6 Hz), 81.8, 81.2, 28.3, 28.1. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*)  $\delta$  -112.49, -119.09. HRMS (ESI, m/z): calcd for C<sub>22</sub>H<sub>27</sub>F<sub>2</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 421.1933; Found: 421.1931.



**Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-chlorophenyl)(3-chlorophenyl)carbamate (10)**. The product **10** was obtained as a colorless oil in 32% (22 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.65 (d, J = 2.4 Hz, 1H), 7.28 (t, J = 2.1 Hz, 1H), 7.21–7.05 (m, 6H), 6.66 (s, 1H), 1.52 (s, 9H), 1.42 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 153.1, 152.5, 143.3, 138.9, 134.2, 134.1, 130.6, 129.5, 125.1, 124.9, 122.9, 119.6, 117.5, 82.0, 81.4, 28.4, 28.2. HRMS (ESI, m/z): calcd for C<sub>22</sub>H<sub>27</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 453.1342; Found: 453.1342



Methyl2-((tert-butoxycarbonyl)(3-(methoxycarbonyl)phenyl)amino)-5-((tert-<br/>butoxycarbonyl)amino)benzoate (11). The product 11 was obtained as a colorless oil in 36% (27<br/>mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-d)  $\delta$  7.94 (t, J = 1.9 Hz, 1H), 7.87 (d, J = 2.7<br/>Hz, 1H), 7.81–7.77 (m, 1H), 7.61–7.55 (m, 1H), 7.45 (d, J = 7.7 Hz, 1H), 7.33 (t, J = 7.9 Hz, 1H),<br/>7.04 (d, J = 8.6 Hz, 1H), 6.66 (s, 1H), 3.87 (s, 3H), 3.84 (s, 3H), 1.51 (s, 9H), 1.39 (s, 9H). <sup>13</sup>C NMR<br/>(101 MHz, Chloroform-d)  $\delta$  166.9, 166.3, 153.4, 152.6, 143.2, 137.3, 136.7, 130.7, 130.5, 130.4,<br/>129.9, 128.6, 127.0, 126.3, 122.6, 120.7, 81.7, 81.3, 52.5, 52.3, 28.4, 28.2. HRMS (ESI, m/z): calcd<br/>for C<sub>26</sub>H<sub>33</sub>N<sub>2</sub>O<sub>8</sub><sup>+</sup>, [M+H]<sup>+</sup>: 501.2231; Found: 501.2233

*Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-cyanophenyl)(3-cyanophenyl)carbamate* (12). The Product 12 was obtained as a colorless oil in 51% (34 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.86 (d, J = 2.5 Hz, 1H), 7.57–7.51 (m, 3H), 7.46–7.38 (m, 2H), 7.20 (d, J = 8.7 Hz, 1H), 6.87 (s, 1H), 1.52 (s, 9H), 1.46 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  152.57, 152.3, 142.7, 138.6, 138.4, 130.4, 130.3, 129.8, 129.2, 128.9, 123.3, 122.5, 118.3, 116.2, 113.9,

113.0, 83.5, 81.9, 28.3, 28.1. HRMS (ESI, m/z): calcd for  $C_{24}H_{27}N_4O_4^+$ , [M+H]<sup>+</sup>: 435.2027; Found: 435.2026.

*Di-tert-butyl* (4-((tert-butoxycarbonyl)(2-((tert-butoxycarbonyl)amino)phenyl)amino)-1,2phenylene)dicarbamate (13). The Product 13 was obtained as a white solid in 44% (40 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.97 (d, J = 8.2 Hz, 1H), 7.40 (s, 2H), 7.27–7.22 (m, 1H), 7.05–6.98 (m, 3H), 6.94–6.89 (m, 1H), 6.84 (s, 1H), 6.71 (s, 1H), 6.68 (s, 1H), 1.48 (s, 27H), 1.42 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 153.8, 153.7, 153.4, 153.0, 139.2, 135.3, 132.3, 130.4, 128.8, 128.2, 127.9, 124.5, 124.0, 121.9, 121.6, 120.7, 82.2, 81.2, 80.9, 80.8, 28.4, 28.4, 28.3, 28.2. HRMS (ESI, m/z): calcd for C<sub>32</sub>H<sub>47</sub>N<sub>4</sub>O<sub>8</sub><sup>+</sup>, [M+H]<sup>+</sup>: 615.3388; Found: 615.3386

*Tert-butyl* (4-((tert-butoxycarbonyl)amino)-3-isopropylphenyl)(2-isopropylphenyl)carbamate (14). The Product 14 was obtained as a colorless oil in 80% (56 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.29 (d, J = 2.5 Hz, 1H), 7.25–7.19 (m, 1H), 7.17–7.06 (m, 3H), 7.0 - 7.00 (m, 1H), 6.94–6.90 (m, 1H), 6.61 (s, 1H), 3.12–3.03 (m, 1H), 2.86–2.77 (m, 1H), 1.52 (s, 9H), 1.42 (s, 9H), 1.23–1.16 (m, 9H), 0.96 (d, J = 6.8 Hz, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  154.4, 152.8, 149.2, 147.2, 143.0, 138.0, 134.8, 130.1, 128.3, 122.7, 122.3, 121.8, 116.6, 116.5, 80.9, 80.6, 34.2, 28.5, 28.4, 28.1, 24.1, 24.0, 23.7, 23.5. HRMS (ESI, m/z): calcd for C<sub>28</sub>H<sub>41</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 469.3061; Found: 469.3060

*Tert-butyl (4-((tert-butoxycarbonyl)amino)-3-methoxyphenyl)(2-methoxyphenyl)carbamate* (15). The Product **15** was obtained as a colorless oil in 34% (23 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.89 (d, *J* = 8.6 Hz, 1H), 7.26–7.20 (m, 1H), 7.17–7.12 (m, 1H), 6.99–6.87 (m, 4H), 6.71 (dd, *J* = 8.7, 2.3 Hz, 1H), 3.83 (s, 3H), 3.81 (s, 3H), 1.50 (s, 9H), 1.41 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  155.6, 154.3, 152.9, 147.4, 137.8, 132.1, 129.6, 128.4, 125.5, 120.9, 118.3, 117.8, 111.9, 108.6, 80.6, 80.3, 55.8, 55.7, 28.5, 28.4. HRMS (ESI, m/z): calcd for C<sub>24</sub>H<sub>33</sub>N<sub>2</sub>O<sub>6</sub><sup>+</sup>, [M+H]<sup>+</sup>: 445.2333; Found: 445.2342

*tert*-butyl [1,1'-biphenyl]-2-yl(6-((*tert*-butoxycarbonyl)amino)-[1,1'-biphenyl]-3yl)carbamate(16). The product 16 (33 mg, 42%) was obtained. Colorless oil.<sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.84 (d, *J* = 8.8 Hz, 1H), 7.46 – 7.40 (m, 3H), 7.39 – 7.29 (m, 10H), 7.24 – 7.19 (m, 2H), 6.83 (d, J = 9.2 Hz, 1H), 6.36 (s, 1H), 1.44 (s, 9H), 1.30 (s, 9H).<sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  153.89, 152.98, 140.23, 139.68, 138.09, 137.92, 131.87, 131.02, 130.30, 129.84, 129.39, 129.34, 129.01, 128.64, 128.36, 128.33, 127.84, 127.58, 127.35, 126.68, 125.14, 81.06, 80.56, 28.39, 28.15. HRMS (ESI, m/z): calcd for C<sub>34</sub>H<sub>37</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 537.2748; Found: 537.2752

Methyl5-((tert-butoxycarbonyl)(2-(methoxycarbonyl)phenyl)amino)-2-((tert-<br/>butoxycarbonyl)amino)benzoate (17). The Product 17 was obtain (12 mg, 16%) was obtained.Colorless oil. <sup>1</sup>H NMR (400 MHz, Chloroform-d)  $\delta$  10.19 (s, 1H), 8.35 (d, J = 9.0 Hz, 1H), 8.04–<br/>7.87 (m, 3H), 7.48–7.35 (m, 2H), 7.34–7.25 (m, 1H), 7.09 (d, J = 8.0 Hz, 1H), 3.87 (s, 6H), 1.51 (s,<br/>9H), 1.39 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-d)  $\delta$  168.1, 166.8, 152.8, 141.9, 139.8, 136.1,<br/>132.7, 130.9, 130.8, 129.3, 129.3, 128.4, 126.6, 119.1, 114.5, 81.5, 80.6, 52.3, 28.3, 28.1. HRMS<br/>(ESI, m/z): calcd for C<sub>26</sub>H<sub>33</sub>N<sub>2</sub>O<sub>8</sub><sup>+</sup>, [M+H]<sup>+</sup>: 501.2231; Found: 501.2231



*Tert-butyl (4-((tert-butoxycarbonyl)amino)-3-cyanophenyl)(2-cyanophenyl)carbamate* (18). The Product 18 was obtained as a colorless oil in 36% (23 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  8.22–8.17 (m, 1H), 7.71 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.64–7.58 (m, 1H), 7.48–7.38 (m, 3H), 7.31–7.27 (m, 1H), 7.01 (s, 1H), 1.52 (s, 9H), 1.46 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  152.5, 151.9, 144.5, 139.4, 136.4, 133.9, 133.8, 132.4, 129.7, 129.6, 127.9, 119.9, 116.6, 115.9, 113.3, 101.1, 83.4, 82.3, 28.3, 28.2. HRMS (ESI, m/z): calcd for C<sub>24</sub>H<sub>27</sub>N<sub>4</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 435.2027; Found: 435.2028.



*Tert-butyl (4-((tert-butoxycarbonyl)amino)-2,5-dimethylphenyl)(2,5-dimethylphenyl)carbamate* (19). The Product 19 was obtained as a colorless oil in 33% (22 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.74 (s, 1H), 7.09 (d, *J* = 7.7 Hz, 1H), 6.92–6.89 (m, 1H), 6.74 (s, 2H), 6.20 (s, 1H), 2.35–2.30 (m, 6H), 2.20 (s, 3H), 2.10 (s, 3H), 1.52 (s, 9H), 1.42 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  153.6, 153.2, 141.9, 137.5, 136.2, 134.6, 130.8, 130.7, 129.1, 127.9, 127.4, 125.5, 123.0, 122.3, 80.6, 80.6, 28.5, 28.4, 21.0, 18.5, 18.3, 17.4. HRMS (ESI, m/z): calcd for C<sub>26</sub>H<sub>37</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 441.2748; Found: 441.2752

Tert-butyl $(4-((tert-butoxycarbonyl)amino)-2-chloro-5-methylphenyl)(5-chloro-2-<br/>methylphenyl)carbamate (20). The Product 20 was obtained as a colorless oil in 39% (28 mg)<br/>isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-d) <math>\delta$  8.11 (s, 1H), 7.17–7.09 (m, 3H), 6.82 (s, 1H),<br/>6.27 (s, 1H), 2.33 (s, 3H), 2.13 (s, 3H), 1.53 (s, 9H), 1.43 (s, 9H). <sup>13</sup>C NMR (101 MHz,<br/>Chloroform-d)  $\delta$  152.7, 152.6, 142.5, 137.0, 136.2, 132.8, 131.9, 131.6, 130.6, 130.1, 127.5, 127.0,<br/>124.7, 120.6, 81.6, 81.3, 28.4, 28.2, 18.1, 17.4. HRMS (ESI, m/z): calcd for C<sub>24</sub>H<sub>31</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>,<br/>[M+H]<sup>+</sup>: 481.1655; Found: 481.1663



*Tert-butyl (4-((tert-butoxycarbonyl)amino)-2,6-dimethylphenyl)(3,5-dimethylphenyl)carbamate* (21). The Product 21 was obtained as a colorless oil in 67% (44 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.11 (s, 2H), 6.82 (s, 2H), 6.67 (s, 1H), 6.53 (s, 1H), 2.22 (s, 6H), 2.11 (s, 6H), 1.52 (s, 9H), 1.40 (s, 9H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  153.5, 152.9, 141.4, 137.9, 137.3, 137.2, 135.1, 125.6, 120.5, 118.0, 80.6, 28.5, 28.3, 21.6, 21.6 18.4, 18.4. HRMS (ESI, m/z): calcd for C<sub>26</sub>H<sub>37</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 441.2748; Found: 441.2753



*Ethyl (4-((ethoxycarbonyl)amino)phenyl)(phenyl)carbamate* (22). The Product 22 was obtained as a colorless oil in 61% (29 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.36–7.28 (m, 4H), 7.25–7.14 (m, 5H), 6.70 (s, 1H), 4.21 (q, *J* = 7.1 Hz, 4H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.23 (t, *J* = 7.1 Hz, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  155.1, 153.7, 142.8, 138.0, 136.2, 129.0, 127.9, 126.8, 126.0, 119.2, 62.2, 61.4, 14.7, 14.6. HRMS (ESI, m/z): calcd for C<sub>18</sub>H<sub>21</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 329.1496; Found: 329.1496



*Butyl (4-((butoxycarbonyl)amino)phenyl)(phenyl)carbamate* (23). The Product 23 was obtained as a colorless oil in 50% (29 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.37–7.28 (m, 4H), 7.24–7.13 (m, 5H), 6.70 (s, 1H), 4.17 - 4.13 (m, 4H), 1.69–1.62 (m, 3H), 1.60–1.52 (m, 2H), 1.46–1.36 (m, 2H), 1.33–1.24 (m, 2H), 0.95 (t, J = 7.4 Hz, 3H), 0.87 (t, J = 7.4 Hz, 3H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 155.2, 153.8, 142.8, 137.9, 136.2, 128.9, 127.8, 126.8, 126.0, 119.1, 66.1, 65.3, 31.1, 30.9, 19.2, 19.2, 13.8, 13.8. HRMS (ESI, m/z): calcd for C<sub>22</sub>H<sub>29</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 385.2122; Found: 385.2130



*Benzyl (4-(((benzyloxy)carbonyl)amino)phenyl)(phenyl)carbamate* (24). The Product 24 was obtained as a colorless oil in 60% (40 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.41–7.28 (m, 13H), 7.25–7.15 (m, 6H), 6.69 (s, 1H), 5.20 (d, *J* = 1.9 Hz, 4H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  154.8, 154.1, 142.6, 138.0, 136.4, 136.1, 129.0, 128.8, 128.6, 128.5, 128.1, 127.9, 126.8, 126.2, 119.2, 119.2, 67.7, 67.3. HRMS (ESI, m/z): calcd for C<sub>28</sub>H<sub>25</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 453.1809; Found: 453.1813.



*Allyl (4-(((allyloxy)carbonyl)amino)phenyl)(phenyl)carbamate* (25). The Product 25 was obtained as a colorless oil in 60% (32 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.39–7.29 (m, 4H), 7.26–7.15 (m, 5H), 6.82 (s, 1H), 6.01–5.83 (m, 2H), 5.40–5.12 (m, 4H), 4.69–4.62 (m, 4H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*) δ 154.7, 153.3, 142.6, 137.9, 136.1, 132.5, 132.4, 129.0, 127.8, 126.8, 126.2, 119.2, 118.5, 117.7, 66.7, 66.0. HRMS (ESI, m/z): calcd for C<sub>20</sub>H<sub>21</sub>N<sub>2</sub>O<sub>4</sub><sup>+</sup>, [M+H]<sup>+</sup>: 353.1496; Found: 353.1502



2,2,2-trichloroethyl phenyl(4-(((2,2,2-trichloroethoxy)carbonyl)amino)phenyl)carbamate (26). The Product 26 was obtained as a colorless oil in 50% (40 mg) isolated yield. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.36–7.25 (m, 4H), 7.24–7.15 (m, 5H), 6.99 (s, 1H), 4.74 (s, 4H). <sup>13</sup>C NMR (101 MHz, Chloroform-*d*)  $\delta$  153.1, 151.6, 141.8, 137.9, 135.7, 129.2, 127.8, 126.9, 126.8, 119.4, 95.3, 75.4, 74.7. HRMS (ESI, m/z): calcd for C<sub>18</sub>H<sub>14</sub>Cl<sub>6</sub>N<sub>2</sub>NaO<sub>4</sub><sup>+</sup>, [M+Na]<sup>+</sup>: 554.8977; Found: 554.8981.

# 3. NMR spectra of products



Tert-butyl (4-((tert-butoxycarbonyl)amino)phenyl-2,3,5,6-d4)(phenyl-d5)carbamate (3)

![](_page_19_Figure_1.jpeg)

Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-methylphenyl)(m-tolyl)carbamate (4).

![](_page_20_Figure_1.jpeg)

21

![](_page_21_Figure_0.jpeg)

Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-ethylphenyl)(3-ethylphenyl)carbamate (5)

22

Tert-butyl

(methoxymethyl)phenyl)carbamate (6)

![](_page_22_Figure_3.jpeg)

 $Tert-butyl \ (4-((tert-butoxycarbonyl)amino)-2-(methylthio)phenyl)(3-(methylthio)phenyl)carbamate \ (methylthio)phenyl)(3-(methylthio)phenyl)carbamate \ (methylthio)phenyl)(3-(methylthio)phenyl)carbamate \ (methylthio)phenyl)(3-(methylthio)phenyl)(3-(methylthio)phenyl)carbamate \ (methylthio)phenyl)(3-(me$ 

![](_page_23_Figure_1.jpeg)

24

Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-ethynylphenyl)(3-ethynylphenyl)carbamate (8)

![](_page_24_Figure_1.jpeg)

![](_page_25_Figure_1.jpeg)

![](_page_25_Figure_2.jpeg)

![](_page_26_Figure_0.jpeg)

-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 fl (ppm)

![](_page_27_Figure_1.jpeg)

Methyl

butoxycarbonyl)amino)benzoate (11)

![](_page_28_Figure_3.jpeg)

![](_page_29_Figure_0.jpeg)

Tert-butyl (4-((tert-butoxycarbonyl)amino)-2-cyanophenyl)(3-cyanophenyl)carbamate (12)

Di-tert-butyl (4-((tert-butoxycarbonyl)(2-((tert-butoxycarbonyl)amino)phenyl)amino)-1,2phenylene)dicarbamate (13)

![](_page_30_Figure_1.jpeg)

![](_page_31_Figure_0.jpeg)

(4-((tert-butoxycarbonyl)amino)-3-isopropylphenyl)(2-isopropylphenyl)carbamate (14)

![](_page_32_Figure_1.jpeg)

*Tert-butyl* [1,1'-biphenyl]-2-yl(6-((tert-butoxycarbonyl)amino)-[1,1'-biphenyl]-3-yl)carbamate (16)

![](_page_33_Figure_1.jpeg)

butoxycarbonyl)amino)benzoate (17)

Methyl

![](_page_34_Figure_2.jpeg)

![](_page_35_Figure_0.jpeg)

*Tert-butyl (4-((tert-butoxycarbonyl)amino)-2,5-dimethylphenyl)(2,5-dimethylphenyl)carbamate (19)* 

![](_page_36_Figure_1.jpeg)

methylphenyl)carbamate (20)

Tert-butyl

![](_page_37_Figure_2.jpeg)

![](_page_38_Figure_1.jpeg)

![](_page_39_Figure_1.jpeg)

90 80 f1 (ppm) 

![](_page_40_Figure_1.jpeg)

20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2 f1 (ppm) ![](_page_41_Figure_2.jpeg)

7.7.7.3 7.7.3.3 7.7.3.3 7.7.3.3 7.7.3.3 7.7.3.3 7.7.3.3 7.7.3.3 7.7.3.3 7.7.3.3 7.7.3 7.7.3 7.7.3 7.7.3 7.7.3 7.7.3 7.7.3 7.7.3 7.7.3 7.7.3 7.7.2 7.5.5 7.5

![](_page_42_Figure_2.jpeg)

43

![](_page_43_Figure_1.jpeg)

![](_page_43_Figure_2.jpeg)

35 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 f1 (ppm)