# Pd-Catalysed selective C(*sp*<sup>3</sup>)-H arylation and acetoxylation of alcohols

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## I. General

Unless otherwise stated, all experiments were carried out under air atmosphere. The reagents and solvents were purchased from commercial suppliers and used without further purification unless noted. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were obtained on a Bruker AVANCE III 500 instrument in CDCl<sub>3</sub> using TMS as an internal standard, operating at 500 MHz and 126 MHz, respectively. Chemical shifts ( $\delta$ ) are expressed in ppm and coupling constants *J* are given in Hz. For CDCl<sub>3</sub> solutions the chemical shifts are reported as parts per million (ppm) to residual protium or carbon of the solvents; CDCl<sub>3</sub>  $\delta$ H (7.28 ppm) and CDCl<sub>3</sub>  $\delta$ C (77.03 ppm). Multiplicities are reported using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, m = multiplet. GC experiments were carried out using Agilent 7890B GC. GC-MS experiments that used dodecane as an internal standard were performed with a Thermo DSQ II, Trace GC Ultra. High resolution mass spectra (HRMS (ESI-TOF)) were obtained on an Agilent 6545 Q-TOF LCMS spectrometer equipped with an ESI source.

## **II.** Preparation of substrates

The substrates 1 for  $C(sp^3)$ -H arylation and acetoxylation were prepared following the previous procedures. <sup>1-5</sup> Some new substrates are characterized as below:

Characterization data of new substrates:



(E)-2-((hexan-2-yloxy)imino)-N-(perfluorophenyl)propanamide: Colourless oil;  $R_f = 0.51$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.15$  (s, 1H), 4.38 (h, J = 6.3 Hz, 1H), 2.10 (s, 3H), 1.78-1.71 (m, 1H), 1.61-1.54 (m, 1H), 1.43-1.35 (m, 4H), 1.33 (d, J = 6.3 Hz, 3H), 0.94 (t, J = 7.0 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.5$ , 148.9, 144.2-144.0 (m), 142.2-142.0 (m), 141.0-140.9 (m), 139.0-138.8 (m), 137.0-136.9 (m), 111.9-111.7 (m), 81.9, 35.3, 27.6, 22.7, 19.9, 14.0, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 375.1102; found: 375.1105.



(E)-2-(((4-methylpentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide: White soild;  $R_f = 0.64$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.15$  (s, 1H), 4.50-4.43 (m, 1H), 2.09 (s, 3H), 1.82-1.74 (m, 1H), 1.73-1.64 (m, 1H), 1.42-1.35 (m, 1H), 1.32 (d, J = 6.3 Hz, 3H), 0.96 (dd,  $J_I = 8.1$  Hz,  $J_2 = 6.6$  Hz, 6H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.5$ , 148.9, 144.2-144.0 (m), 142.2-142.0 (m), 141.1-140.9 (m), 139.0-138.7 (m), 137.0-136.7 (m), 111.9-111.6 (m), 80.4, 44.7, 24.8, 22.9, 22.6, 20.5, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 375.1102; found: 375.1102.



(E)-2-(((5-chloropentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide: Yellow solid;  $R_f = 0.49$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.13$  (s, 1H), 4.43 (h, J = 6.4 Hz, 1H), 3.60 (t, J = 6.3 Hz, 2H), 2.10 (s, 3H), 1.98-1.92 (m, 1H), 1.91-1.85 (m, 2H), 1.84-1.76 (m, 1H), 1.36 (d, J = 6.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 149.4, 144.2-144.1 (m), 142.2-142.1 (m), 141.2-141.0 (m), 139.1-138.9 (m), 137.0-136.7 (m), 111.8-111.5 (m), 80.9, 44.8, 32.8, 28.5, 19.9, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 395.0556; found: 395.0556.



(E)-2-(((1-(4-methoxyphenyl)propan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide: Colourless oil;  $R_f = 0.44$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.97$  (s, 1H), 7.16-7.11 (m, 2H), 6.87-6.83 (m, 2H), 4.60 (h, J = 6.3 Hz, 1H), 3.78 (s, 3H), 2.99 (dd,  $J_1 = 14.0$  Hz,  $J_2 = 6.9$  Hz, 1H), 2.85 (dd,  $J_1 = 14.0$  Hz,  $J_2 = 5.8$  Hz, 1H), 2.09 (s, 3H), 1.35 (d, J = 6.4 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 158.2, 149.0, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 139.0-138.8 (m), 137.0-136.7 (m), 130.4 (2C), 130.0, 113.7 (2C) , 111.8-111.5 (m), 82.5, 55.1, 41.4, 19.4, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 439.1052; found: 439.1052.



(E)-N-(perfluorophenyl)-2-(((4-phenylbutan-2-yl)oxy)imino)propanamide: White soild;  $R_f = 0.54$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.12$  (s, 1H), 7.34-7.30 (m, 2H), 7.24-7.20 (m, 3H), 4.43 (h, J = 6.3 Hz, 1H), 2.82-2.70 (m, 2H), 2.15-2.07 (m, 4H), 1.98-1.90 (m, 1H), 1.39 (d, J = 6.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 149.1, 144.2-144.0 (m), 142.2-142.0 (m), 141.6, 141.0-140.9 (m), 139.0-138.7 (m), 137.0-136.7 (m), 128.4 (2C), 128.4 (2C), 126.0, 111.8-111.6 (m), 81.0, 37.1, 31.7, 20.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 423.1102; found: 423.1108.



(E)-N-(perfluorophenyl)-2-(((4-(*o*-tolyl)butan-2-yl)oxy)imino)propanamide: Yellowish oil;  $R_f = 0.58$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.16$  (s, 1H), 7.21-7.13 (m, 4H), 4.49 (h, J = 6.3 Hz, 1H), 2.84-2.77 (m, 1H), 2.76-2.69 (m, 1H), 2.36 (s, 3H), 2.16 (s, 3H), 2.09-2.01 (m, 1H), 1.95-1.87 (m, 1H), 1.43 (d, J = 6.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 149.2, 144.2-144.0 (m), 142.2-142.0 (m), 141.0-140.9 (m), 139.8, 139.0-138.7 (m), 137.0-136.7 (m), 135.8, 130.3, 128.8, 126.1, 126.0, 111.8-111.5 (m), 81.2, 36.0, 28.9, 19.9, 19.1, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 437.1259; found: 437.1265.



(E)-N-(perfluorophenyl)-2-(((4-(trifluoromethyl)phenyl)butan-2-yl)oxy)imino)propanamide:

Yellow oil;  $R_f = 0.49$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.11$  (s, 1H), 7.56 (d, J = 8.1 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 4.43 (h, J = 6.3 Hz, 1H), 2.89-2.75 (m, 2H), 2.15-2.06 (m, 4H), 2.00-1.91 (m, 1H), 1.39 (d, J = 6.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 149.4, 145.8, 144.1-144.0 (m), 142.2-142.0 (m), 141.1-140.9 (m), 139.0-138.8 (m), 137.0-136.7 (m), 128.7 (2C), 128.4 (q, J = 32.4 Hz), 125.3 (q, J = 3.5 Hz, 2C), 124.3 (q, J = 272.2 Hz), 111.8-111.5 (m), 80.7, 36.7, 31.5, 19.9, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 491.0976; found: 491.0976.



(E)-2-(((4-(2-fluorophenyl)butan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide: Colourless oil;  $R_f = 0.57$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.11$  (s, 1H), 7.20 (q, J = 7.2Hz, 2H), 7.10-6.99 (m, 2H), 4.42 (h, J = 6.3 Hz, 1H), 2.84-2.72 (m, 2H), 2.10 (s, 3H), 2.09-2.03 (m, 1H), 1.98-1.89 (m, 1H), 1.39 (d, J = 6.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 161.2 (d, J =244.5 Hz), 149.2, 144.2-144.1 (m), 142.2-142.0 (m), 141.1-140.9 (m), 138.9-138.7 (m), 137.0-136.8 (m), 130.6 (d, J = 5.2 Hz), 128.4 (d, J = 15.7 Hz), 127.8 (d, J = 8.3 Hz), 124.0 (d, J = 3.5 Hz), 115.3 (d, J =22.1 Hz), 111.7-111.6 (m), 81.0 35.7, 25.1 (d, J = 2.6 Hz), 19.9, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 441.1008; found: 441.1016.



(E)-2-(((4-(3-fluorophenyl)butan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide: Colourless oil;  $R_f = 0.49$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.10$  (s, 1H), 7.28-7.24 (m, 1H), 6.98 (d, J = 7.7 Hz, 1H), 6.91 (t, J = 8.6 Hz, 2H), 4.45-4.38 (m, 1H), 2.81-2.69 (m, 2H), 2.11 (s, 3H), 2.10-2.05 (m, 1H), 1.96-1.88 (m, 1H), 1.38 (d, J = 6.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta =$ 163.0 (d, J = 245.5 Hz), 161.3, 149.3, 144.2 (d, J = 6.9 Hz), 144.1-144.0 (m), 142.2-142.1 (m), 141.1-140.9 (m), 139.0-138.9 (m), 137.0-136.8 (m), 129.9 (d, J = 8.5 Hz), 124.0 (d, J = 2.6 Hz), 115.2 (d, J =20.9 Hz), 112.9 (d, J = 20.9 Hz), 111.8-111.6 (m), 80.8, 36.8, 31.4, 20.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 441.1008; found: 441.1010.



(E)-2-(((4-(4-fluorophenyl)butan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide: Colourless oil;  $R_f = 0.49$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.10$  (s, 1H), 7.18-7.14 (m, 2H), 7.01-6.96 (m, 2H), 4.44-4.37 (m, 1H), 2.78-2.66 (m, 2H), 2.12 (s, 3H), 2.09-2.03 (m, 1H), 1.93-1.86 (m, 1H), 1.38 (d, J = 6.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 161.3 (d, J = 243.7 Hz), 149.3, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 139.0-138.8 (m), 137.2 (d, J = 3.1 Hz), 137.0-136.8 (m), 129.7 (d, J = 7.8 Hz, 2C), 115.2 (d, J = 21.2 Hz, 2C), 111.7-111.5 (m), 80.8, 37.2, 30.8, 19.9, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 441.1008; found: 441.1011.



(E)-2-((4-methylphenethoxy)imino)-N-(perfluorophenyl)propanamide: Yellow oil;  $R_f = 0.56$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.00$  (s, 1H), 7.15 (s, 4H), 4.49 (t, J = 6.9 Hz, 2H), 3.04 (t, J = 6.9 Hz, 2H), 2.33 (s, 3H), 2.10 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 149.5, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 139.0-138.8 (m), 136.9-136.8 (m), 136.0, 134.9, 129.2 (2C), 128.8 (2C), 111.7-111.4 (m), 76.4, 35.4, 21.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 409.0946; found: 409.0948.



(E)-2-((4-methoxyphenethoxy)imino)-N-(perfluorophenyl)propanamide: Colourless oil;  $R_f = 0.33$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.01$  (s, 1H), 7.18-7.15 (m, 2H), 6.88-6.85 (m, 2H), 4.47 (t, J = 6.8 Hz, 2H), 3.79 (s, 3H), 3.01 (t, J = 6.8 Hz, 2H), 2.10 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 158.3, 149.6, 144.2-144.1 (m), 142.2-142.0 (m), 141.1-141.0 (m), 139.0-

138.8 (m), 136.9-136.7 (m), 130.0, 129.9 (2C), 113.9 (2C), 111.7-111.4 (m), 76.5, 55.2, 35.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 425.0895; found: 425.0898.



(E)-2-((2-(naphthalen-1-yl)ethoxy)imino)-N-(perfluorophenyl)propanamide: White soild;  $R_f = 0.47$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.09$  (d, J = 8.5 Hz, 1H), 7.92-7.85 (m, 2H), 7.78 (d, J = 8.0 Hz, 1H), 7.59-7.49 (m, 2H), 7.47-7.42 (m, 2H), 4.66 (t, J = 7.0 Hz, 2H), 3.55 (t, J = 7.0 Hz, 2H), 2.07 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.1$ , 149.7, 144.1-144.0 (m), 142.1-142.0 (m), 141.2-141.0 (m), 139.0-138.8 (m), 137.1-136.8 (m), 133.9, 133.9, 132.0, 128.9, 127.4, 127.1, 126.1, 125.7, 125.5, 123.5, 111.7-111.4 (m), 75.7, 32.9, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]+ 445.0946; found: 445.0953.



(E)-2-((2-bromophenethoxy)imino)-N-(perfluorophenyl)propanamide: Yellow oil;  $R_f = 0.50$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.00$  (s, 1H), 7.58 (d, J = 7.9 Hz, 1H), 7.28-7.26 (m, 2H), 7.14-7.09 (m, 1H), 4.53 (t, J = 6.7 Hz, 2H), 3.22 (t, J = 6.7 Hz, 2H), 2.09 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.1$ , 149.8, 144.2-144.0 (m), 142.1-142.0 (m), 141.2-141.0 (m), 138.9-138.8 (m), 137.31, 137.0-136.8 (m), 132.9, 131.2, 128.3, 127.5, 124.6, 111.8-111.5 (m), 74.6, 36.1, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 472.9895; found: 472.9895.



(E)-2-((2-iodophenethoxy)imino)-N-(perfluorophenyl)propanamide: Yellow oil;  $R_f = 0.47$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.02$  (s, 1H), 7.86 (dd,  $J_I = 7.9$  Hz,  $J_2 = 1.1$  Hz, 1H), 7.31 (td,  $J_I = 7.4$  Hz,  $J_2 = 1.1$  Hz, 1H), 7.26 (dd,  $J_I = 7.6$  Hz,  $J_2 = 1.7$  Hz, 1H), 6.94 (td,  $J_I = 7.7$  Hz,  $J_2 = 1.8$  Hz, 1H), 4.50 (t, J = 6.8 Hz, 2H), 3.24-3.19 (m, 2H), 2.10 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.1$ , 149.8, 144.1-144.0 (m), 142.1-142.0 (m), 141.1-140.9 (m), 140.6, 139.7, 139.0-138.9 (m), 136.9-136.8 (m), 130.3, 128.5, 128.4, 111.7-111.3 (m), 100.7, 74.8, 40.5, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 520.9756; found: 520.9760.



(E)-N-(perfluorophenyl)-2-((4-phenylbutoxy)imino)propanamide: White soild;  $R_f = 0.53$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.17$  (s, 1H), 7.36-7.29 (m, 2H), 7.26-7.20 (m, 3H), 4.32 (t, J = 6.2 Hz, 2H), 2.73 (t, J = 1.4 Hz, 2H), 2.13 (s, 3H), 1.85-1.77 (m, 4H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 149.3, 144.1-144.0 (m), 142.1-142.0 (m), 142.0, 141.1-140.9 (m), 139.0-138.7 (m), 136.9-136.7 (m), 128.3, 128.3, 125.8, 111.7-111.5 (m), 75.7, 35.5, 28.5, 27.5, 9.6 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 423.1102; found: 423.1102.



(E)-2-((pentyloxy)imino)-N-(perfluorophenyl)propanamide: Colourless oil;  $R_f = 0.64$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.14$  (s, 1H), 4.27 (t, J = 6.7 Hz, 2H), 2.11 (s, 3H), 1.79-1.72 (m, 2H), 1.42-1.37 (m, 4H), 0.98-0.91 (m, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 149.2, 144.2-144.0 (m), 142.2-142.0 (m), 141.1-140.8 (m), 139.1-138.9 (m), 137.0-136.8 (m), 111.8-111.7 (m), 76.1, 28.8, 28.0, 22.5, 14.0, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 361.0946; found: 361.0947.



(E)-2-((2-cyclohexylethoxy)imino)-N-(perfluorophenyl)propanamide: White soild;  $R_f = 0.67$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.14$  (s, 1H), 4.31 (t, J = 6.8 Hz, 2H), 2.10 (s, 3H), 1.81-1.69 (m, 5H), 1.67-1.59 (m, 3H), 1.32-1.15 (m, 3H), 1.03-0.92 (m, 2H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 149.2, 144.2-144.0 (m), 142.2-142.0 (m), 141.1-141.0 (m), 139.0-138.8 (m), 137.0-136.8 (m), 111.8-111.6 (m), 74.3, 36.4, 34.6, 33.3, 26.5, 26.2, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 401.1259; found: 401.1262.



(E)-2-(((2-isopropyl-5-methylcyclohexyl)oxy)imino)-N-(perfluorophenyl)propanamide: White soild;  $R_f = 0.67$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.17$  (s, 1H), 4.67 (s, 1H), 2.15-2.09 (m, 4H), 1.81-1.75 (m, 2H), 1.68-1.60 (m, 2H), 1.44-1.35 (m, 1H), 1.13-1.05 (m, 2H), 0.97-0.89 (m, 7H), 0.90 (d, J = 6.6 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.5$ , 149.1, 144.1-144.0 (m), 142.2-142.0 (m), 141.0-140.8 (m), 139.0-138.8 (m), 137.0-136.7 (m), 111.9-111.6 (m), 81.9, 47.1, 39.5, 34.9, 29.3, 26.3, 25.2, 22.3, 21.1, 20.8, 9.6 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 429.1572; found: 429.1578.



(E)-2-((ethoxy-d<sub>5</sub>)imino)-N-(perfluorophenyl)propanamide: Yellow oil;  $R_f = 0.67$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.15$  (s, 1H), 2.11 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 149.2, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-140.9 (m), 139.1-138.7 (m), 137.0-136.7 (m), 111.8-111.5 (m), 71.0-70.3 (m), 13.9-13.0 (m), 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 324.0790; found: 324.0790.

**III. Screening of the conditions** 

A. C(*sp*<sup>3</sup>)-H Arylation of masked alcohols



<sup>a</sup> GC Yields.

Figure S1. Screening of directing groups

Table S1. Screening of the arylation conditions

$F + N = \begin{bmatrix} Pd \end{bmatrix} (10 \text{ mol}\%) \\ Phl (4.0 \text{ equiv.}) \\ \hline Additive (equiv.) \\ F + F \\ F + 1 (0.1 \text{ mmol}) \end{bmatrix} F + F \\ F + N = \begin{bmatrix} Ag \\ B \\ Solvent, 110 \ ^{\circ}C, 24 \text{ h} \\ F + F \\ Solvent, 110 \ ^{\circ}C, 24 \text{ h} \end{bmatrix} F + F \\ F + B \\ F + $
--

[Pd] (10 mol%)	[Ag] (equiv.)	Additive (equiv.)	Solvent (mL)	Yield (%) <sup>[a]</sup>
$Pd(OAc)_2$	$Ag_3PO_4(2.0)$	NaHCO <sub>3</sub> $(1.0)$	PhCl (1.0)	64 (58) <sup>[b]</sup>
Pd(OAc) <sub>2</sub>	$Ag_3PO_4(2.0)$	NaHCO <sub>3</sub> (1.0)	PhCl (0.1)	62
Pd(OAc) <sub>2</sub>	$Ag_{3}PO_{4}(1.5)$	NaHCO <sub>3</sub> (1.5)	PhCl (0.1)	61
Pd(OAc) <sub>2</sub>	Ag <sub>3</sub> PO <sub>4</sub> (1.5)	NaHCO <sub>3</sub> (1.0)	PhCl (0.1)	70
Pd(MeCN) <sub>2</sub> Cl <sub>2</sub>	$Ag_3PO_4(1.5)$	NaHCO <sub>3</sub> (1.0)	PhCl (0.1)	41
Pd(OTFA) <sub>2</sub>	$Ag_3PO_4(1.5)$	NaHCO <sub>3</sub> (1.0)	PhCl (0.1)	60
PdCl <sub>2</sub>	$Ag_3PO_4(1.5)$	NaHCO <sub>3</sub> (1.0)	PhCl (0.1)	33
Pd(OAc) <sub>2</sub>	$Ag_3PO_4(1.5)$	NaHCO <sub>3</sub> (1.0)	PhF (0.1)	50
Pd(OAc) <sub>2</sub>	$Ag_3PO_4(1.5)$	NaHCO <sub>3</sub> (1.0)	TBB (0.1)	55
Pd(OAc) <sub>2</sub>	$Ag_3PO_4(1.5)$	NaHCO <sub>3</sub> (1.0)	<i>o</i> -DCB (0.1)	49
Pd(OAc) <sub>2</sub>	Ag <sub>2</sub> CO <sub>3</sub> (1.5)	NaHCO <sub>3</sub> (1.0)	PhCl (0.1)	22
Pd(OAc) <sub>2</sub>	AgOAc (1.5)	NaHCO <sub>3</sub> (1.0)	PhCl (0.1)	44
Pd(OAc) <sub>2</sub>	AgNO <sub>3</sub> (1.5)	NaHCO <sub>3</sub> (1.0)	PhCl (0.1)	45
Pd(OAc) <sub>2</sub>	$Ag_{3}PO_{4}(1.5)$	H <sub>3</sub> BO <sub>3</sub> (1.0)	PhCl (0.1)	66
$Pd(OAc)_2$	$Ag_3PO_4(1.5)$	4-ClPhOH (1.0)	PhCl (0.1)	69
Pd(OAc) <sub>2</sub>	$Ag_{2}CO_{3}(2.0)$	-	PhCl (1.0)	47
Pd(OAc) <sub>2</sub>	$Ag_3PO_4(2.0)$	$Na_2CO_3(1.0)$	PhCl (1.0)	49
	[Pd]   (10 mol%)   Pd(OAc)2   <	[Pd]   [Ag]     (10 mol%)   (equiv.)     Pd(OAc)2   Ag3PO4(2.0)     Pd(OAc)2   Ag3PO4(2.0)     Pd(OAc)2   Ag3PO4(2.0)     Pd(OAc)2   Ag3PO4(2.0)     Pd(OAc)2   Ag3PO4(2.0)     Pd(OAc)2   Ag3PO4(2.0)     Pd(OAc)2   Ag3PO4(1.5)     Pd(OAc)2   Ag3PO4(1.5)     Pd(OTFA)2   Ag3PO4(1.5)     Pd(OAc)2   Ag0Ac (1.5)     Pd(OAc)2   Ag0Ac (1.5)     Pd(OAc)2   Ag0Ac (1.5)     Pd(OAc)2   Ag3PO4(1.5)     Pd(OAc)2   Ag3PO4(1.5)	[Pd]   [Ag]   Additive (equiv.)     Pd(OAc)2   Ag3PO4 (2.0)   NaHCO3 (1.0)     Pd(OAc)2   Ag3PO4 (2.0)   NaHCO3 (1.0)     Pd(OAc)2   Ag3PO4 (2.0)   NaHCO3 (1.0)     Pd(OAc)2   Ag3PO4 (1.5)   NaHCO3 (1.0)     Pd(OAc)2   Ag3PO4 (1.5)   NaHCO3 (1.0)     Pd(OAc)2   Ag3PO4 (1.5)   NaHCO3 (1.0)     Pd(MeCN)2Cl2   Ag3PO4 (1.5)   NaHCO3 (1.0)     Pd(OTFA)2   Ag3PO4 (1.5)   NaHCO3 (1.0)     Pd(OAc)2   Ag2CO3 (1.5)   NaHCO3 (1.0)     Pd(OAc)2   Ag0Ac (1.5)   NaHCO3 (1.0)     Pd(OAc)2   Ag3PO4 (1.5)   NaHCO3 (1.0)     Pd(OAc)2   Ag3PO4 (1.5)   NaHCO3 (1.0)     Pd(OAc)2   Ag3PO4 (1.5)   NaHCO3 (1.0)     Pd(OAc)2   Ag3PO	$\begin{array}{ c c c c c } [Ag] & Additive & Solvent \\ (10 mol%) & (equiv.) & (equiv.) & (mL) \\ \hline Pd(OAc)_2 & Ag_3PO_4(2.0) & NaHCO_3(1.0) & PhC1 (1.0) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(MeCN)_2Cl_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(OTFA)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhF (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & O-DCB (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_0Ac (1.5) & NaHCO_3(1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_0Ac (1.5) & NaHCO_3 (1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3 (1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & NaHCO_3 (1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4(1.5) & H_3BO_3 (1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4 (1.5) & 4-CIPhOH (1.0) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4 (2.0) & Na_2CO_3 (1.0) & PhC1 (1.0) \\ PhC1 (0.1) PhC1 (0.1) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4 (2.0) & Na_2CO_3 (1.0) & PhC1 (1.0) \\ PhC1 (0.1) PhC1 (0.1) & PhC1 (0.1) \\ Pd(OAc)_2 & Ag_3PO_4 (2.0) & Na_2CO_3 (1.0) & PhC1 (1.0) \\ PhC1 (0.1) PhC1 (0.1) & PhC1 (0.1) \\ PhC1 (0.1) PhC1 (0.1) & PhC1 (1.0) \\ PhC1 (0.1) PhC1 (0.1) & PhC1 (1.0) \\ PhC1 (0.1) PhC1 (0.1) PhC1 (0.1) \\ PhC1 (0.1) PhC1 (0.1) PhC1 (0.1) \\ PhC1 (0.1) PhC1 (0.1) PhC1 (0.1) PhC1 (0.1) \\ PhC1 (0.1) PhC1 (0.1) PhC1 (0.1) PhC1 (0.1) PhC1 (0.1)$

<sup>[a]</sup>GC yields. <sup>[b]</sup>100 °C. TBB = *tert*-butylbenzene, *o*-DCB = ortho dichlorobenzene

B. C(*sp*<sup>3</sup>)-H Acetoxylation of masked alcohols

*Table S2.* Screening of the methyl  $C(sp^3)$ -H acetoxylation conditions



Entry	Oxidant	AcOH/Ac <sub>2</sub> O	Tim	Temp.	Yield of <b>4'/4''</b>
	(equiv.)	(v/v)	e (h)	(°C)	(%) <sup>[a]</sup>
1	Oxone $(2.5)$	25:2	24	110	60/4
2	Oxone (2.5)	25:2	12	100	62/1
3	Oxone (2.5)	25:2	24	100	72/22
4	Oxone (2.5)	20:4	24	100	69/16
5	Oxone (2.5)	50:1	24	100	63/4
6	Oxone (2.5)	50:1	24	110	67/17
7	Oxone (2.0)	50:1	24	110	40/2
8	Oxone (3.0)	50:1	24	110	67/8
9	NaNO <sub>3</sub> (2.5)	50:1	16	110	8/trace
10	$K_2S_2O_8(2.5)$	50:1	16	110	69/15
11	$K_2S_2O_8(2.0)$	50:1	24	110	49/3
12	$K_2S_2O_8(3.0)$	50:1	24	110	51/46
13	$K_2S_2O_8(2.5)$	50:1	24	100	60/39
14	$K_2S_2O_8(2.5)$	50:1	24	90	80/10
15	$K_2S_2O_8(2.5)$	25:2	24	100	77/18
16	$K_2S_2O_8(2.5)$	20:4	24	100	33/2
17	$K_2S_2O_8(2.5)$	25:2	12	110	83/10
18	$K_2S_2O_8(2.5)$	25:2	12	100	72/4
19	$K_{2}S_{2}O_{8}(2.5)$	25:2	6	100	12/0
20	$K_2S_2O_8(2.5)$	50:1	12	100	43/1

<sup>[a]</sup>GC yields.

*Table S3.* Screening of the methylene  $C(sp^3)$ -H acetoxylation conditions



Entry	[Pd] (10 mol%)	Oxidant (equiv.)	AcOH/Ac <sub>2</sub> O	Time (h)	Temp. (°C)	Yield of 4x <sup>[a]</sup>
1	$Pd(OAc)_2$	$K_2S_2O_8(3.0)$	25:2	24	110	52
2	Pd(OAc) <sub>2</sub>	$K_2S_2O_8(3.0)$	50:1	24	100	77
3	Pd(OAc) <sub>2</sub>	$K_2S_2O_8$ (3.0)	50:1	24	110	83
4	Pd(dba) <sub>2</sub>	$K_2S_2O_8(3.0)$	50:1	24	110	57
5	Pd(TFA) <sub>2</sub>	$K_2S_2O_8(3.0)$	50:1	24	110	56
6	Pd(OAc) <sub>2</sub>	$K_2S_2O_8(3.0)$	25:1	24	110	46
7	Pd(OAc) <sub>2</sub>	$K_2S_2O_8(2.5)$	50:1	24	110	46

<sup>[a]</sup>GC yields.

# **IV. General procedures**

#### a) Typical procedure for the arylation of alcohols

In a 10 mL test tube equipped with a stir bar, alkyl alcohol derivatives **1** (0.3 mmol),  $Pd(OAc)_2$  (6.6 mg, 0.03 mmol),  $Ag_3PO_4$  (188.4 mg, 0.45 mmol), PhI (244.8 mg, 1.2 mmol), NaHCO<sub>3</sub> (25.2 mg, 0.3 mmol) and PhCl (0.3 mL) were added successively. Then the tube was heated at 110 °C for 24 h under stirring. Upon completion, the resulting mixture was allowed to cool to room temperature and diluted with EtOAc. The solvent was then removed in *vacuo* and the residue was purified through silica gel chromatography (petroleum ether/EtOAc = 50:1) to give the corresponding products **3a-3q**.

#### b) Typical procedure for the arylation with diverse aromatic iodides

In a 10 mL test tube equipped with a stir bar, (E)-N-methyl-2-((pentan-2-yloxy)imino)-N-(perfluorophenyl)propanamide (101.4 mg, 0.3 mmol),  $Pd(OAc)_2$  (6.6 mg, 0.03 mmol),  $Ag_3PO_4$  (188.4 mg, 0.45 mmol), ArI (1.2 mmol), NaHCO<sub>3</sub> (25.2 mg, 0.3 mmol) and PhCl (0.3 mL) were added successively. Then the tube was sealed and stirred at the indicated temperature for 24 h. Upon completion, the resulting mixture was cooled to room temperature, diluted with EtOAc and concentrated under reduced pressure. Then the residue was purified by silica gel chromatography (petroleum ether/EtOAc = 50:1-10:1) to afford the desired products **3r-3ai**.

# c) Typical procedure for the acetoxylation of methyl C(sp<sup>3</sup>)-H bond

In a 10 mL test tube equipped with a stir bar, alkyl alcohol derivatives 1 (0.3 mmol), Pd(OAc)<sub>2</sub> (6.6mg,

0.03 mmol),  $K_2S_2O_8$  (202.8 mg, 0.75 mmol), AcOH/Ac<sub>2</sub>O (75:6, v/v, 1.5 mL) were added successively. Then the tube was sealed and stirred at 110 °C for 12-48 h. Upon completion, the resulting mixture was allowed to cool to room temperature, diluted with EtOAc. The solvent was then removed in *vacuo* and the residue was purified through silica gel chromatography (petroleum ether/EtOAc = 10:1-5:1) to give the corresponding products **4'**, **4b**-4g.

#### d) Typical procedure for the acetoxylation of methylene $C(sp^3)$ -H bond

In a 10 mL test tube equipped with a stir bar, phenethanol derivatives **1** (0.3 mmol),  $Pd(OAc)_2$  (6.6mg, 0.03 mmol),  $K_2S_2O_8$  (243.9 mg, 0.9 mmol),  $AcOH/Ac_2O$  (150:3, v/v, 3 mL) were added successively. Then the tube was sealed and stirred at 100-110 °C for 24 h. Upon completion, the resulting mixture was allowed to cool to room temperature, diluted with EtOAc. The solvent was then removed in *vacuo* and the residue was purified through silica gel chromatography (petroleum ether/EtOAc = 10:1-5:1) to afford the desired products **4h-4x**.

#### V. Removal of the directing groups



Following the literature procedure<sup>3, 5</sup>: A 25 mL Schlenk tube was charged with arylated product **3v** (58 mg, 0.12 mmol, 1.0 equiv.),  $Mo(CO)_6$  (38 mg, 0.144 mmol, 1.2 equiv.),  $CH_3CN$  (2.0 mL),  $H_2O$  (5 drops). The tube was sealed with a Teflon-lined screw cap, refrigerated with liquid nitrogen, evacuated the air and filled with nitrogen by the schlenk line for 3 times. Then the tube was heated at 90 °C for 12 h under stirring. Then the tube was allowed to cool to room temperature. The solvent was then removed in vacuo and the residue was purified through flash column chromatography on silica gel (eluent: petroleum ether/EtOAc = 5:1) to give the corresponding product **5** as a white soild (20 mg, 83% yield).

A 25 mL Schlenk tube was charged with acetoxylated product **4m** (84 mg, 0.18 mmol, 1.0 equiv.),  $Mo(CO)_6$  (57 mg, 0.216 mmol, 1.2 equiv.),  $CH_3CN$  (3.0 mL),  $H_2O$  (7 drops). The tube was sealed with a Teflon-lined screw cap, refrigerated with liquid nitrogen, evacuated the air and filled with nitrogen by the schlenk line for 3 times. Then the tube was heated at 90 °C for 12 h under stirring. Then the tube was allowed to cool to room temperature. The solvent was then removed in vacuo and the residue was purified through flash column chromatography on silica gel (petroleum ether/EtOAc = 5:1) to give the corresponding product **6** as a white soild (25 mg, 72% yield).

# VI. Hydrolysis of 4i



In a 10 mL test tube equipped with a stir bar, acetoxylated product **4i** (0.06 mmol),  $K_2CO_3$  (20.7 mg, 0.15 mmol), MeOH (1 mL) were added successively. Then the solution was allowed to stir at room temperature. After 24 h, the resulting solution was diluted with MeOH. The solvent was then removed in *vacuo* and the residue was purified through silica gel chromatography (petroleum ether/EtOAc = 5:1) to give the corresponding products **7** as a colorless oil (18 mg, 75% yield).

# **VII.** Competing experiments

Intermolecular competing experiment:



In a 10 mL test tube equipped with a stir bar, 2-(4-methoxyphenyl)ethan-1-ol derivatives (20.1 mg, 0.05 mmol), pentan-1-ol derivatives (16.9 mg, 0.05 mmol),  $Pd(OAc)_2$  (2.2 mg, 0.01 mmol),  $K_2S_2O_8$  (81.3 mg, 0.3 mmol), AcOH/Ac<sub>2</sub>O (50:1, v/v, 1 mL) were added successively. Then the tube was sealed and stirred at 110 °C for 4 h. Upon completion, the resulting mixture was allowed to cool to room temperature, diluted with EtOAc. The ratio of the mixture [4j (62%) + 4u (25%)] was determined by GC-MS.

Intramolecular competing experiment:



In a 10 mL test tube equipped with a stir bar, alkyl alcohol derivatives **3r** (0.05 mmol),  $Pd(OAc)_2$  (1.1 mg, 0.005 mmol),  $K_2S_2O_8$  (40.7 mg, 0.15 mmol),  $AcOH/Ac_2O$  (50:1, v/v, 0.5 mL) were added successively. Then the tube was sealed and stirred at 110 °C for 24 h. Upon completion, the resulting mixture was allowed to cool to room temperature, diluted with EtOAc. The solvent was then removed in *vacuo* and the residue was purified through silica gel chromatography (petroleum ether/EtOAc = 5:1) to give the

corresponding benzylic C-H bond acetoxylated product **8** as the major product (10 mg,  $\sim$  42% yield). The minor regioisomer could not be isolated due to its low content.

(1S)-2-((((E)-1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)-1-(*p*-tolyl)pentyl acetate (8): Colourless oil;  $R_f = 0.38$  (petroleum ether- EtOAc = 5:1); d.r. = 1.4 / 1; (major): <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.02$  (s, 1H), 7.27-7.23 (m, 4H), 6.08-6.01 (m, 1H), 4.65-4.56 (m, 1H), 2.35 (s, 3H), 2.12 (s, 3H), 2.10 (s, 3H), 1.64-1.43 (m, 4H), 0.92-0.88 (m, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.0$ , 161.3, 149.6, 144.2-144.0 (m), 142.2-142.0 (m), 141.0-140.9 (m), 139.1-139.0 (m), 138.3, 137.0-136.9 (m), 134.0, 129.2 (2C), 127.3 (2C), 111.7-111.6 (m), 86.6, 76.1, 32.2, 31.5, 21.1, 18.6, 13.8, 9.9 ppm; (minor): <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.90$  (s, 1H), 7.22-7.10 (m, 4H), 6.01-5.95 (m, 1H), 4.71-4.53 (m, 1H), 2.35 (s, 3H), 2.14 (s, 3H), 2.07 (s, 3H), 1.41-1.24 (m, 4H), 0.99-0.92 (m, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 169.9$ , 161.2, 149.5, 144.2-144.0 (m), 142.2-142.0 (m), 141.0-140.9 (m), 139.1-139.0 (m), 139.1-139.0 (m), 138.0, 137.0-136.9 (m), 133.9, 129.0 (2C), 127.2 (2C), 111.7-111.6 (m), 86.8, 76.1, 32.2, 31.5, 21.1, 18.9, 13.9, 10.0 ppm.







In a 25 mL test tube equipped with a stir bar, ethanol derivatives (59.2 mg, 0.2 mmol), ethan- $d_5$ -1-ol derivatives (60.2 mg, 0.2 mmol), Pd(OAc)<sub>2</sub> (8.8 mg, 0.04 mmol), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (325.2 mg, 1.2 mmol), AcOH/Ac<sub>2</sub>O (200:4, v/v, 4 mL) were added successively. Then the tube was sealed and stirred at 110 °C for 6 h. Upon completion, the resulting mixture was allowed to cool to room temperature, diluted with EtOAc. The solvent was then removed in *vacuo* and the residue was purified through silica gel chromatography (petroleum ether/EtOAc = 3:1) to give a mixture of **4a** and **4a-d<sub>4</sub>** (50 mg, ~36% yield).

May07-2019



# **IX. References**

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#### X. Characterization data of products



(E)-N-(perfluorophenyl)-2-(((1-phenylpropan-2-yl)oxy)imino)propanamide (3a): Follow the typical process an isolated as colourless oil (66 mg, 57% yield);  $R_f = 0.55$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.93$  (s, 1H), 7.34-7.29 (m, 2H), 7.23 (d, J = 7.3 Hz, 3H), 4.65 (h, J = 6.4 Hz, 1H), 3.06 (dd,  $J_I = 13.9$  Hz,  $J_2 = 7.0$  Hz, 1H), 2.92 (dd,  $J_I = 13.9$  Hz,  $J_2 = 5.8$  Hz, 1H), 2.09 (s, 3H), 1.38 (d, J = 6.4 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 149.1, 144.2-144.0 (m), 142.2-142.0 (m), 141.1-141.0 (m), 139.0-138.8 (m), 138.0, 136.9-136.7 (m), 129.5 (2C), 128.3 (2C), 126.4, 111.8-111.6 (m), 82.3, 42.4, 19.5, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 409.0946; found: 409.0955.



(E)-N-(perfluorophenyl)-2-(((1-phenylbutan-2-yl)oxy)imino)propanamide (3b): Follow the typical process an isolated as colourless oil (90 mg, 75% yield);  $R_f = 0.56$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.83$  (s, 1H), 7.32-7.28 (m, 2H), 7.23-7.18 (m, 3H), 4.49-4.43 (m, 1H), 3.07-2.92 (m, 2H), 2.08 (s, 3H), 1.77-1.71 (m, 2H), 1.03 (t, J = 7.4 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 149.0, 144.2-144.0 (m), 142.2-142.0 (m), 141.1-141.0 (m), 139.0-138.8 (m), 138.3, 136.9-136.7 (m), 129.4 (2C), 128.3 (2C), 126.3, 111.8-111.6 (m), 87.5, 40.4, 26.5, 9.8, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 423.1102; found: 423.1107.



(E)-N-(perfluorophenyl)-2-(((1-phenylpentan-2-yl)oxy)imino)propanamide (3c): Follow the typical process an isolated as colourless oil (83 mg, 67% yield);  $R_f = 0.61$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.81$  (s, 1H), 7.32-7.28 (m, 2H), 7.21 (d, J = 7.6 Hz, 3H), 4.56-4.51 (m, 1H), 3.05-2.92 (m, 2H), 2.08 (s, 3H), 1.78-1.68 (m, 1H), 1.69-1.59 (m, 1H), 1.59-1.48 (m, 1H), 1.47-1.42 (m, 1H), 0.97 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 148.9, 144.2-144.1 (m), 142.0-142.0 (m), 141.2-141.1 (m), 139.0-138.8 (m), 138.3, 137.0-136.7(m), 129.5 (2C), 128.3 (2C), 126.3, 111.8-111.6 (m), 86.2, 41.0, 35.7, 18.8, 14.1, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 437.1259; found: 437.1261.



(E)-N-(perfluorophenyl)-2-(((1-phenylhexan-2-yl)oxy)imino)propanamide (3d): Follow the typical process an isolated as colourless oil (72 mg, 56% yield);  $R_f = 0.62$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.83$  (s, 1H), 7.33-7.28 (m, 2H), 7.23-7.18 (m, 3H), 4.55-4.50 (m, 1H), 3.04-2.94 (m, 2H), 2.08 (s, 3H), 1.80-1.71 (m, 1H), 1.70-1.62 (m, 1H), 1.52-1.46 (m, 1H), 1.44-1.35 (m, 3H), 0.94 (t, J = 7.2 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 148.9, 144.1-144.0 (m), 142.1-142.0 (m), 141.0-140.9 (m), 139.0-138.8 (m), 138.3, 136.9-136.7 (m), 129.4 (2C), 128.3 (2C), 126.3, 111.8-111.5 (m), 86.4, 40.9, 33.2, 27.6, 22.6, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]+ 451.1415; found: 451.1418.



(E)-2-(((4-methyl-1-phenylpentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3e): Follow the typical process an isolated as colourless oil (81 mg, 63% yield);  $R_f = 0.61$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.79$  (s, 1H), 7.33-7.28 (m, 2H), 7.23-7.18 (m, 3H), 4.65-4.60 (m, 1H), 3.03-2.93 (m, 2H), 2.08 (s, 3H), 1.85-1.79 (m, 1H), 1.75-1.70 (m, 1H), 1.49-1.41 (m, 1H), 0.96 (dd,  $J_I = 7.5$  Hz,  $J_2 = 6.8$  Hz, 6H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 148.8, 144.1-144.0 (m), 142.2-142.0 (m), 141.1-141.0 (m), 139.0-138.8 (m), 138.3, 137.0-136.8 (m), 129.4 (2C), 128.3 (2C), 126.3, 111.8-111.5 (m), 84.8, 42.6, 41.6, 24.8, 23.2, 22.2, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]+ 451.1415; found: 451.1417.



(E)-2-(((3-methyl-1-phenylbutan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3f): Follow the typical process an isolated as colourless oil (79 mg, 64% yield);  $R_f$ = 0.60 (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.68 (s, 1H), 7.30-7.22 (m, 4H), 7.18 (t, *J* = 7.3 Hz, 1H), 4.35 (dt, *J<sub>I</sub>* = 9.0 Hz, *J<sub>2</sub>* = 4.6 Hz, 1H), 2.99 (dd, *J<sub>I</sub>* = 14.3 Hz, *J<sub>2</sub>* = 4.2 Hz, 1H), 2.93 (dd, *J<sub>I</sub>* = 14.3 Hz, *J<sub>2</sub>* = 8.7 Hz, 1H), 2.07 (s, 3H), 1.08-1.06 (m, 7H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$  = 161.4, 148.6, 144.2-144.0 (m), 142.2-142.0 (m), 141.1-141.0 (m), 139.0, 138.9-138.8 (m), 136.9-136.8 (m), 129.4 (2C), 128.3 (2C), 126.2, 111.8-111.5 (m), 91.3, 37.7, 31.3, 18.6, 17.9, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 437.1259; found: 437,1262.



(E)-2-((1-cyclohexyl-2-phenylethoxy)imino)-N-(perfluorophenyl)propanamide (3g): Follow the typical process an isolated as colourless oil (84 mg, 62% yield);  $R_f$ = 0.61 (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.68 (s, 1H), 7.31-7.27 (m, 2H), 7.23-7.16 (m, 3H), 4.38-4.31 (m, 1H), 3.02 (dd,  $J_I$  = 14.3 Hz,  $J_2$  = 4.0 Hz, 1H), 2.93 (dd,  $J_I$  = 14.3 Hz,  $J_2$  = 8.8 Hz, 1H), 2.06 (s, 3H), 1.92 (d, J = 12.4 Hz, 1H), 1.83-1.81 (m, 3H), 1.74 (d, J = 11.2 Hz, 2H), 1.34-1.12 (m, 5H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$  = 161.4, 148.4, 144.1-144.0 (m), 142.1-142.0 (m), 141.0-140.9 (m), 139.0, 138.8-138.7 (m), 136.9-136.7 (m), 129.4 (2C), 128.3 (2C), 126.1, 111.8-111.6 (m), 90.9, 41.3, 38.0, 29.2, 28.3, 26.5, 26.2, 26.2, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 477.1572; found: 477.1573.



(E)-2-(((5-chloro-1-phenylpentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3h): Follow the typical process an isolated as yellow oil (103 mg, 77% yield);  $R_f = 0.40$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.79$  (s, 1H), 7.33-7.29 (m, 2H), 7.25-7.18 (m, 3H), 4.56 (p, J = 6.0 Hz, 1H), 3.59 (t, J = 6.2 Hz, 2H), 3.04 (dd,  $J_I = 14.1$  Hz,  $J_2 = 7.2$  Hz, 1H), 2.97 (dd,  $J_I = 14.1$  Hz,  $J_2 = 5.5$  Hz, 1H), 2.08 (s, 3H), 2.03-1.96 (m, 1H), 1.96-1.84 (m, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 149.5, 144.2-144.0 (m), 142.2-142.1 (m), 141.2-141.0 (m), 139.1-138.9 (m), 137.8, 136.9-136.8 (m), 129.4 (2C), 128.4 (2C), 126.5, 111.6-111.4 (m), 85.5, 44.8, 41.0, 30.8, 28.6, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 471.0869; found: 471.0870.



(E)-2-(((1,3-diphenylpropan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3i): Follow the typical process an isolated as colourless oil (58 mg, 42% yield);  $R_f = 0.52$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.57$  (s, 1H), 7.36-7.18 (m, 8H), 7.14 (d, J = 8.4 Hz, 2H), 4.74 (p, J = 5.9 Hz, 1H), 3.07-2.95 (m, 4H), 2.04 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 149.1, 144.1-144.0 (m), 142.1-142.0 (m), 141.0-140.8 (m), 138.9-138.8 (m), 138.0 (2C), 136.9-136.8 (m), 129.5 (4C), 128.4 (4C), 126.5 (2C), 111.7-111.4 (m), 86.9, 40.5 (2C), 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 485.1259; found: 485.1265.



(E)-2-(((1-(4-fluorophenyl)-3-phenylpropan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3j): Follow the typical process an isolated as colourless oil (72 mg, 50% yield);  $R_f = 0.51$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.63$  (s, 1H), 7.32 (t, J = 7.5 Hz, 2H), 7.25-7.20 (m, 3H), 7.17 (dd,  $J_I = 8.5$  Hz,  $J_2 = 5.4$  Hz, 2H), 7.00 (t, J = 8.7 Hz, 2H), 4.74 (p, J = 6.4 Hz, 1H), 3.11-2.88 (m, 4H), 2.05 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.7$  (d, J = 244.3 Hz), 161.2, 149.4, 144.2-144.1 (m), 142.1-142.0 (m), 141.2-141.1 (m), 138.9-138.8 (m), 137.8, 137.0-136.7 (m), 133.5 (d, J = 3.4 Hz), 130.8 (d, J = 7.2 Hz, 2C), 129.4 (2C), 128.4 (2C), 126.5, 115.2 (d, J = 21.3 Hz, 2C), 111.6-111.3 (m), 86.8, 40.4, 39.5, 10.0 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 503.1165; found: 503.1166.



#### (E)-2-(((1-(4-methoxyphenyl)-3-phenylpropan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3k), Follow the tunical process on isolated as valley, ail (61 mg, 41% yield); P = 0.40 (perfective at the tunical process) of the tunical process of tunical proces

(3k): Follow the typical process an isolated as yellow oil (61 mg, 41% yield);  $R_f = 0.40$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.61$  (s, 1H), 7.33-7.28 (m, 2H), 7.24-7.19 (m, 3H), 7.13 (d, J = 8.6 Hz, 2H), 6.85 (d, J = 8.6 Hz, 2H), 4.74 (p, J = 6.4 Hz, 1H), 3.78 (s, 3H), 3.00 (d, J = 6.4Hz, 2H), 2.97 (dd,  $J_1 = 6.3$  Hz,  $J_2 = 2.8$  Hz, 2H), 2.05 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta =$ 161.3, 158.3, 149.1, 144.3-144.1 (m), 142.2-142.1 (m), 141.1-140.9 (m), 139.0-138.9 (m), 138.1, 136.9-136.8 (m), 130.4 (2C), 129.9, 129.5 (2C), 128.4 (2C), 126.4, 113.8 (2C), 111.7-111.5 (m), 87.1, 55.2, 40.4, 39.5, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 515.1365; found: 515.1365.



(E)-2-(((1,4-diphenylbutan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (31): Follow the typical process an isolated as yellowish oil (79 mg, 55% yield);  $R_f = 0.54$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H

NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.79 (s, 1H), 7.31 (t, *J* = 7.5 Hz, 4H), 7.21 (q, *J*<sub>1</sub> = 7.6 Hz, *J*<sub>2</sub> = 6.8 Hz, 6H), 4.59-4.54 (m, 1H), 3.03 (qd, *J*<sub>1</sub> = 14.1 Hz, *J*<sub>2</sub> = 6.3 Hz, 2H), 2.86-2.81 (m, 1H), 2.78-2.71 (m, 1H), 2.11-1.99 (m, 5H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$  = 161.3, 149.2, 144.2-144.0 (m), 142.2-142.0 (m), 141.4, 141.2-141.0 (m), 139.0-138.8 (m), 137.9, 137.0-136.8 (m), 129.4 (2C), 128.5 (2C), 128.4 (2C), 128.4 (2C), 128.4 (2C), 126.4, 126.0, 111.7-111.5 (m), 85.5, 40.9, 35.0, 31.7, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 499.1415; found: 499.1424.



**(E)-N-(perfluorophenyl)-2-(((1-phenyl-4-(o-tolyl)butan-2-yl)oxy)imino)propanamide (3m):** Follow the typical process an isolated as yellowish oil (96 mg, 65% yield);  $R_f = 0.53$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.81$  (s, 1H), 7.34-7.30 (m, 2H), 7.25-7.20 (m, 3H), 7.18-7.11 (m, 4H), 4.64-4.58 (m, 1H), 3.09 (dd,  $J_I = 14.0$  Hz,  $J_2 = 7.2$  Hz, 1H), 3.01 (dd,  $J_I = 14.1$  Hz,  $J_2 = 5.6$  Hz, 1H), 2.86-2.80 (m, 1H), 2.75-2.67 (m, 1H), 2.31 (s, 3H), 2.12 (s, 3H), 2.04-1.96 (m, 2H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 149.3, 144.2-144.1 (m), 142.2-142.0 (m), 141.2-141.1 (m), 139.7, 138.9-138.8 (m), 137.9, 136.9-136.8 (m), 135.8, 130.3, 129.4 (2C), 128.8, 128.4 (2C), 126.4, 126.2, 126.1, 111.7-111.4 (m), 85.8, 40.9, 33.9, 29.1, 19.2, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 513.1572; found: 513.1579.



# (E)-N-(perfluorophenyl)-2-(((1-phenyl-4-(4-(trifluoromethyl)phenyl)butan-2-yl)oxy)imino)

**propanamide (3n):** Follow the typical process an isolated as yellowish oil (122 mg, 75% yield);  $R_f = 0.26$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.80$  (s, 1H), 7.56 (d, J = 8.1 Hz, 2H), 7.31 (q, J = 7.6 Hz, 4H), 7.25-7.19 (m, 3H), 4.59-4.54 (m, 1H), 3.08 (dd,  $J_I = 14.0$  Hz,  $J_2 = 7.1$  Hz, 1H), 3.00 (dd,  $J_I = 14.0$  Hz,  $J_2 = 5.7$  Hz, 1H), 2.92-2.87 (m, 1H), 2.83-2.77 (m, 1H), 2.11-2.01 (m, 5H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 149.5, 145.6, 144.2-144.1 (m), 142.2-142.1 (m), 141.2-141.0 (m), 139.1-138.9 (m), 137.7, 137.0-136.8 (m), 129.4 (2C), 128.7 (2C), 128.5 (q, J = 32.3 Hz), 128.4 (2C), 126.5, 125.4 (q, J = 3.8 Hz, 2C), 124.3 (q, J = 272.2 Hz), 111.7-111.4 (m), 85.3, 41.0, 34.6, 31.6, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 567.1289; found: 567.1297.



(E)-2-(((4-(2-fluorophenyl)-1-phenylbutan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3o): Follow the typical process an isolated as yellow oil (92 mg, 62% yield);  $R_f = 0.52$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.82$  (s, 1H), 7.31 (t, J = 7.6 Hz, 2H), 7.23-7.16 (m, 5H), 7.10-7.00 (m, 2H), 4.56 (ddd,  $J_I = 12.5$  Hz,  $J_2 = 7.4$  Hz,  $J_3 = 5.3$  Hz, 1H), 3.09-2.98 (m, 2H), 2.86 (ddd,  $J_I$ = 14.7 Hz,  $J_2 = 9.1$  Hz,  $J_3 = 6.1$  Hz, 1H), 2.78 (dt,  $J_I = 14.3$  Hz,  $J_2 = 7.9$  Hz, 1H), 2.12-1.99 (m, 5H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 161.1 (d, J = 244.5 Hz), 149.3, 144.1-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 139.0-138.8 (m), 137.9, 136.9-136.7 (m), 130.6 (d, J = 4.8 Hz), 129.4 (2C), 128.3 (2C), 128.2, 127.8 (d, J = 8.1 Hz), 126.4, 124.0 (d, J = 3.5 Hz), 115.3 (d, J = 22.0 Hz), 111.7-111.5(m), 85.5, 40.8, 33.7, 25.1 (d, J = 2.4 Hz), 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 517.1321; found: 517.1323.



(E)-2-(((4-(3-fluorophenyl)-1-phenylbutan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3p): Follow the typical process an isolated as yellow oil (98 mg, 66% yield);  $R_f = 0.51$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.82$  (s, 1H), 7.31 (t, J = 7.4 Hz, 2H), 7.27-7.19 (m, 4H), 6.99-6.86 (m, 3H), 4.59-4.53 (m, 1H), 3.07 (dd,  $J_I = 14.0$  Hz,  $J_2 = 7.1$  Hz, 1H), 3.00 (dd,  $J_I = 14.0$  Hz,  $J_2 = 5.6$  Hz, 1H), 2.87-2.81 (m, 1H), 2.77-2.70 (m, 1H), 2.11-1.98 (m, 5H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 162.9$  (d, J = 245.6 Hz), 161.2, 149.3, 144.2-144.1 (m), 144.0 (d, J = 7.4 Hz), 142.2-142.0 (m), 141.1-141.0 (m), 139.0-138.8 (m), 137.7, 136.9-136.7 (m), 129.8 (d, J = 8.5 Hz), 129.4 (2C), 128.4 (2C), 126.5, 124.0 (d, J = 2.7 Hz), 115.2 (d, J = 20.9 Hz), 112.9 (d, J = 20.9 Hz), 111.7-111.4 (m), 85.3, 40.9, 34.6, 31.5 (d, J = 1.7 Hz), 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]+ 517.1321; found: 517.1324.



(E)-2-(((4-(4-fluorophenyl)-1-phenylbutan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3q): Follow the typical process an isolated as colourless oil (93 mg, 63% yield);  $R_f = 0.50$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.79$  (s, 1H), 7.31 (t, J = 7.5 Hz, 2H), 7.24-7.18 (m, 3H), 7.13 (dd,  $J_I = 8.4$  Hz,  $J_2 = 5.5$  Hz, 2H), 6.98 (t, J = 8.7 Hz, 2H), 4.57-4.51 (m, 1H), 3.05 (dd,  $J_I = 14.0$  Hz,  $J_2 = 7.1$  Hz, 1H), 2.99 (dd,  $J_I = 14.0$  Hz,  $J_2 = 5.6$  Hz, 1H), 2.80 (ddd,  $J_I = 14.7$  Hz,  $J_2 = 9.5$  Hz,  $J_3 = 5.7$ 

Hz, 1H), 2.74-2.66 (m, 1H), 2.10 (s, 3H), 2.08-1.94 (m, 2H) ppm;  $^{13}$ C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$  = 161.3 (d, *J* = 243.9 Hz), 161.2, 149.3, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-141.1 (m), 139.0-138.8 (m), 137.8, 137.0 (d, *J* = 3.4 Hz), 136.9-136.7 (m), 129.7 (d, *J* = 8.0 Hz, 2C), 129.4 (2C), 128.4 (2C), 126.4, 115.2 (d, *J* = 21.2 Hz, 2C), 111.6-111.4 (m), 85.3, 41.0, 35.1, 30.9, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]+ 517.1321; found: 517.1323.



**(E)-N-(perfluorophenyl)-2-(((1-(p-tolyl)pentan-2-yl)oxy)imino)propanamide (3r):** Follow the typical process an isolated as colourless oil (77 mg, 60% yield);  $R_f = 0.64$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.84$  (s, 1H), 7.10 (s, 4H), 4.54-4.48 (m, 1H), 3.01-2.88 (m, 2H), 2.29 (s, 3H), 2.08 (s, 3H), 1.76-1.68 (m, 1H), 1.66-1.59 (m, 1H), 1.56-1.50 (m, 1H), 1.48-1.40 (m, 1H), 0.97 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 148.8, 144.1-144.0 (m), 142.1-142.0 (m), 141.2-141.1 (m), 139.0-138.8 (m), 136.9-136.7 (m), 135.8, 135.2, 129.3 (2C), 129.0 (2C), 111.8-111.7 (m), 86.3, 40.5, 35.6, 20.9, 18.8, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 451.1415; found: 451.1419.



**(E)-N-(perfluorophenyl)-2-(((1-(m-tolyl)pentan-2-yl)oxy)imino)propanamide (3s):** Follow the typical process an isolated as colourless oil (69 mg, 54% yield);  $R_f = 0.64$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.84$  (s, 1H), 7.19 (t, J = 7.5 Hz, 1H), 7.02 (d, J = 7.9 Hz, 3H), 4.56-4.50 (m, 1H), 3.00-2.89 (m, 2H), 2.34 (s, 3H), 2.09 (s, 3H), 1.76-1.70 (m, 1H), 1.67-1.61 (m, 1H), 1.57-1.51 (m, 1H), 1.48-1.41 (m, 1H), 0.97 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 148.8, 144.2-144.0 (m), 142.1-142.0 (m), 141.1-141.0 (m), 139.0-138.8 (m), 138.2, 137.9, 136.9-136.7 (m), 130.2, 128.2, 127.0, 126.4, 111.8-111.5 (m), 86.2, 40.9, 35.7, 21.3, 18.7, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 451.1415; found: 451.1424.



(E)-2-(((1-(3,5-dimethylphenyl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3t): Follow the typical process an isolated as colourless oil (74 mg, 56% yield);  $R_f = 0.67$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.83$  (s, 1H), 6.83 (s, 3H), 4.51 (ddd,  $J_I = 7.3$  Hz,  $J_2 =$ 5.1 Hz,  $J_3 = 2.2$  Hz, 1H), 2.94 (dd,  $J_I = 14.0$  Hz,  $J_2 = 7.2$  Hz, 1H), 2.86 (dd,  $J_I = 13.9$  Hz,  $J_2 = 5.5$  Hz, 1H), 2.29 (s, 6H), 2.09 (s, 3H), 1.76-1.68 (m, 1H), 1.67-1.59 (m, 1H), 1.57-1.50 (m, 1H), 1.49-1.42 (m, 1H), 0.97 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.5$ , 148.7, 144.1-144.0 (m), 142.2142.0 (m), 141.2-141.0 (m), 139.1-138.8 (m), 138.2, 137.8 (2C), 137.0-136.9 (m), 127.8, 127.3 (2C), 111.7-111.5 (m), 86.3, 40.8, 35.7, 21.2 (2C), 18.8, 14.1, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 465.1572; found: 465.1581.



(E)-2-(((1-(4-(*tert*-butyl)phenyl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3u): Follow the typical process an isolated as yellow solid (80 mg, 57% yield);  $R_f = 0.64$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.96$  (s, 1H), 7.32 (d, J = 8.3 Hz, 2H), 7.14 (d, J = 8.2Hz, 2H), 4.54-4.50 (m, 1H), 2.99 (dd,  $J_I = 14.2$  Hz,  $J_2 = 7.0$  Hz, 1H), 2.91 (dd,  $J_I = 14.2$  Hz,  $J_I = 5.5$  Hz, 1H), 2.09 (s, 3H), 1.74-1.69 (m, 1H), 1.66-1.59 (m, 1H), 1.56-1.50 (m, 1H), 1.46-1.42 (m, 1H), 1.30 (s, 9H), 0.96 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.4$ , 149.2, 149.0, 144.3-144.1 (m), 142.2-142.0 (m), 141.1-141.0 (m), 139.0-138.9 (m), 136.9-136.8 (m), 135.1, 129.1 (2C), 125.2 (2C), 111.7-111.6 (m), 86.2, 40.2, 35.7, 34.4, 31.3 (3C), 18.7, 14.1, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 493.1885; found: 493.1889.



(E)-2-(((1-([1,1'-biphenyl]-4-yl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3v): Follow the typical process an isolated as white soild (82 mg, 56% yield);  $R_f = 0.33$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.60$  (s, 1H), 7.59-7.50 (m, 4H), 7.44-7.37 (m, 2H), 7.37-7.27 (m, 3H), 4.63-4.57 (m, 1H), 3.07-2.98 (m, 2H), 2.09 (s, 3H), 1.84-1.75 (m, 1H), 1.73-1.66 (m, 1H), 1.60-1.54 (m, 1H), 1.52-1.46 (m, 1H), 1.00 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$ = 161.4, 148.7, 144.0-143.9 (m), 142.0-141.9 (m), 140.9-140.8 (m), 140.3, 139.0-138.8 (m), 138.7, 137.9, 136.9-136.7 (m), 129.9 (2C), 128.7 (2C), 127.2, 126.7 (2C), 126.5 (2C), 111.7-111.4 (m), 86.0, 41.0, 35.8, 18.8, 14.1, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]+ 513.1572; found: 513.1573.



(E)-2-(((1-(4-methoxyphenyl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3w): Follow the typical process an isolated as colourless oil (61 mg, 46% yield);  $R_f = 0.48$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.87$  (s, 1H), 7.15-7.08 (m, 2H), 6.87-6.80 (m, 2H), 4.51-4.46 (m, 1H), 3.77 (s, 3H), 2.97-2.86 (m, 2H), 2.08 (s, 3H), 1.73-1.67 (m, 1H), 1.66-1.57 (m, 1H), 1.55-1.49 (m, 1H), 1.46-1.40 (m, 1H), 0.96 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.5$ , 158.2, 148.9, 144.2-144.1 (m), 142.2-142.0 (m), 141.1-141.0 (m), 139.0-138.9(m), 136.9-136.8 (m), 130.4 (2C),

130.3, 113.7 (2C), 111.8-111.6 (m), 86.4, 55.2, 40.0, 35.6, 18.8, 14.1, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 467.1365; found: 467.1373.



(E)-N-(perfluorophenyl)-2-(((1-(4-(trifluoromethoxy)phenyl)pentan-2-yl)oxy)imino)propanamide (3x): Follow the typical process an isolated as yellow oil (81 mg, 54% yield);  $R_f = 0.31$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.91$  (s, 1H), 7.22 (d, J = 8.6 Hz, 2H), 7.15 (d, J = 8.2Hz, 2H), 4.59-4.45 (m, 1H), 3.07-2.90 (m, 2H), 2.08 (s, 3H), 1.78-1.68 (m, 1H), 1.65-1.60 (m, 1H), 1.57-1.49 (m, 1H), 1.48-1.41 (m, 1H), 0.97 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 149.4, 147.9, 144.1-144.0 (m), 142.2-141.9 (m), 141.2-140.9 (m), 139.0-138.9 (m), 137.0-136.8 (m), 136.9, 130.7 (2C), 120.8 (2C), 120.5 (q, J = 257.4 Hz), 111.7-111.5 (m), 85.8, 40.0, 35.6, 18.7, 14.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 521.1082; found: 521.1083.



(E)-2-(((1-(4-fluorophenyl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3y): Follow the typical process an isolated as colourless oil (53 mg, 41% yield);  $R_f = 0.60$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.90$  (s, 1H), 7.18-7.13 (m, 2H), 7.02-6.95 (m, 2H), 4.53-4.45 (m, 1H), 3.02-2.87 (m, 2H), 2.08 (s, 3H), 1.78-1.66 (m, 1H), 1.65-1.57 (m, 1H), 1.56-1.47 (m, 1H), 1.46-1.39 (m, 1H), 0.96 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.6$  (d, J = 245.0 Hz), 161.3, 149.1, 144.2-144.0 (m), 142.3-142.0 (m), 141.1-140.9 (m), 139.1-138.6 (m), 137.0-136.7 (m), 133.8 (d, J = 3.4 Hz), 130.8 (d, J = 7.1 Hz, 2C), 115.1 (d, J = 20.9 Hz, 2C), 111.8-111.4 (m), 86.1, 39.9, 35.6, 18.7, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 455.1165; found: 455.1167.



(E)-2-(((1-(4-chlorophenyl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3z): Follow the typical process an isolated as colourless oil (56 mg, 42% yield);  $R_f = 0.42$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.83$  (s, 1H), 7.26 (dt,  $J_I = 8.9$  Hz,  $J_2 = 2.2$  Hz, 2H), 7.16-7.11 (m, 2H), 4.59-4.39 (m, 1H), 2.96-2.93 (m, 2H), 2.07 (s, 3H), 1.77-1.66 (m, 1H), 1.65-1.57 (m, 1H), 1.55-1.48 (m, 1H), 1.47-1.40 (m, 1H), 0.96 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 149.2, 144.1-144.0 (m), 142.2-142.0 (m), 141.2-141.1 (m), 139.1-138.8 (m), 137.0-136.9 (m), 136.8, 132.2, 130.8 (2C), 128.4 (2C), 111.6-111.5 (m), 85.8, 40.2, 35.6, 18.7, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 471.0869; found: 471.0870.



(E)-2-(((1-(4-bromophenyl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3aa): Follow the typical process an isolated as white soild (86 mg, 58% yield);  $R_f = 0.44$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.79$  (s, 1H), 7.41 (d, J = 8.4 Hz, 2H), 7.08 (d, J = 8.3 Hz, 2H), 4.50 (dt,  $J_I = 12.4$  Hz,  $J_2 = 6.2$  Hz, 1H), 2.93 (d, J = 6.2 Hz, 2H), 2.07 (s, 3H), 1.76-1.67 (m, 1H), 1.64-1.57 (m, 1H), 1.55-1.48 (m, 1H), 1.47-1.39 (m, 1H), 0.96 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 149.2, 144.1-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 139.1-138.8 (m), 137.0-136.9 (m), 137.3, 131.4 (2C), 131.2 (2C), 120.2, 111.7-111.5 (m), 85.7, 40.3, 35.6, 18.7, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]+ 515.0364; found: 515.0369.



(E)-2-(((1-(3-chloro-4-methylphenyl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3ab): Follow the typical process an isolated as colourless oil (82 mg, 59% yield);  $R_f = 0.60$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.88$  (s, 1H), 7.20 (d, J = 1.6 Hz, 1H), 7.15 (d, J =7.8 Hz, 1H), 6.98 (dd,  $J_I = 7.8$  Hz,  $J_2 = 1.8$  Hz, 1H), 4.49 (ddd,  $J_I = 7.3$  Hz,  $J_2 = 5.2$  Hz,  $J_3 = 2.1$  Hz, 1H), 2.97-2.86 (m, 2H), 2.32 (s, 3H), 2.08 (s, 3H), 1.76-1.67 (m, 1H), 1.63-1.59 (m, 1H), 1.55-1.48 (m, 1H), 1.47-1.40 (m, 1H), 0.96 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 149.2, 144.1-144.0 (m), 142.2-142.0 (m), 141.0-140.9 (m), 139.1-138.7 (m), 137.4, 136.8-136.7 (m), 134.1, 133.9, 130.8, 129.9, 127.7, 111.8-111.5 (m), 85.9, 40.0, 35.6, 19.5, 18.7, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 485.1026; found: 485.1026.



(E)-2-(((1-(3-nitrophenyl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3ac): Follow the typical process an isolated as yellow oil (59 mg, 43% yield);  $R_f = 0.57$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.10$  (d, J = 7.6 Hz, 2H), 7.93 (s, 1H), 7.57-7.43 (m, 2H), 4.56 (p, J = 6.1 Hz, 1H), 3.10 (d, J = 6.3 Hz, 2H), 2.09 (s, 3H), 1.79-1.71 (m, 1H), 1.66-1.58 (m, 1H), 1.56-1.50 (m, 1H), 1.49-1.43 (m, 1H), 0.97 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.0$ , 149.7, 148.3, 144.1-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 140.1, 139.0-138.7 (m), 137.0-136.7 (m), 135.7, 129.2, 124.2, 121.6, 111.5-111.2 (m), 85.4, 40.1, 35.7, 18.7, 14.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 482.1110; found: 482.1113.



(E)-2-(((1-(4-nitrophenyl)pentan-2-yl)oxy)imino)-N-(perfluorophenyl)propanamide (3ad): Follow the typical process an isolated as yellow oil (66 mg, 66% yield);  $R_f = 0.31$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.20$ -8.13 (m, 2H), 7.88 (s, 1H), 7.37 (d, J = 8.7 Hz, 2H), 4.56 (ddd,  $J_I = 12.4$  Hz,  $J_2 = 7.0$  Hz,  $J_3 = 5.4$  Hz, 1H), 3.10 (dd,  $J_I = 6.1$  Hz,  $J_2 = 2.1$  Hz, 2H), 2.08 (s, 3H), 1.79-1.70 (m, 1H), 1.64-1.60 (m, 1H), 1.56-1.49 (m, 1H), 1.47-1.41 (m, 1H), 0.97 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.0$ , 149.8, 146.8, 146.0, 144.2-144.0 (m), 142.1-142.0 (m), 141.1-141.0 (m), 139.2-138.9 (m), 137.0-136.8 (m), 130.2 (2C), 123.5 (2C), 111.5-111.2 (m), 85.3, 40.5, 35.7, 18.7, 14.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 482.1110; found: 482.1110.



(E)-N-(perfluorophenyl)-2-(((1-(4-(trifluoromethyl)phenyl)pentan-2-yl)oxy)imino)propanamide (3ae): Follow the typical process an isolated as colourless oil (80 mg, 55% yield);  $R_f = 0.33$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.80$  (s, 1H), 7.56 (d, J = 8.0 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 4.55 (p,  $J_I = 6.8$  Hz,  $J_2 = 6.2$  Hz, 1H), 3.07-3.02 (m, 2H), 2.08 (s, 3H), 1.80-1.71 (m, 1H), 1.67-1.59 (m, 1H), 1.57-1.51 (m, 1H), 1.49-1.41 (m, 1H), 0.97 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.2$ , 149.3, 144.1-144.0 (m), 142.5, 142.1-142.0 (m), 141.2-141.0 (m), 139.0-138.9 (m), 137.0-136.8 (m), 129.7 (2C), 128.8 (q, J = 32.3 Hz), 125.2 (q, J = 3.9 Hz, 2C), 124.2 (q, J = 272.5 Hz), 111.6-111.4 (m), 85.6, 40.7, 35.7, 18.7, 14.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 505.1133; found: 505.1133.



Methyl(E)-3-(2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)pentyl)benzoate (3af): Follow the typical process an isolated as colourless oil (82 mg, 58% yield);  $R_f = 0.21$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.91 (s, 1H), 7.89-7.83 (m, 2H), 7.41-7.34 (m, 2H), 4.59-4.51 (m, 1H), 3.91 (s, 3H), 3.02 (dd,  $J_I = 6.2$  Hz,  $J_2 = 3.2$  Hz, 2H), 2.07 (s, 3H), 1.77-1.69 (m, 1H), 1.66-1.58 (m, 1H), 1.56-1.48 (m, 1H), 1.48-1.38 (m, 1H), 0.96 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ = 167.0, 161.2, 149.2, 144.2-144.1 (m), 142.2-142.0 (m), 141.0-140.9 (m), 139.0-138.8 (m), 138.6, 136.9-136.8 (m), 134.0, 130.5, 130.3, 128.4, 127.6, 111.8-111.5 (m), 85.8, 52.1, 40.6, 35.7, 18.7, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 495.1314; found: 495.1314.



**Methyl(E)-4-(2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)pentyl)benzoate** (**3ag):** Follow the typical process an isolated as colourless oil (57 mg, 40% yield);  $R_f = 0.20$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.98-7.94$  (m, 2H), 7.71 (s, 1H), 7.28 (d, J = 8.3 Hz, 2H), 4.58-4.52 (m, 1H), 3.88 (s, 3H), 3.03 (d, J = 6.3 Hz, 2H), 2.05 (s, 3H), 1.78-1.69 (m, 1H), 1.66-1.58 (m, 1H), 1.56-1.50 (m, 1H), 1.48-1.41 (m, 1H), 0.96 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 166.8$ , 161.2, 149.1, 144.1-144.0 (m), 143.9, 142.1-141.9 (m), 141.1-140.9 (m), 139.0-138.8 (m), 137.0-136.8 (m), 129.6 (2C), 129.4 (2C), 128.3, 111.8-111.5 (m), 85.7, 52.0, 41.0, 35.7, 18.7, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 495.1314; found: 495.1317.



(E)-2-(((1-(4-(1-((1,3-dioxoisoindo lin-2-yl)oxy)ethyl)phenyl)pentan-2-yl)oxy)imino)-N-

(perfluorophenyl)propanamide (3ah): Follow the typical process an isolated as yellow oil (118 mg, 65% yield);  $R_f = 0.36$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.94$  (d, J = 8.7 Hz, 1H), 7.79-7.73 (m, 2H), 7.70 (dt,  $J_I = 6.0$  Hz,  $J_2 = 3.0$  Hz, 2H), 7.45 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 5.49-5.14 (m, 1H), 4.50-4.47 (m, 1H), 3.02-2.88 (m, 2H), 2.04 (d, J = 7.6 Hz, 2H), 1.70 (dd,  $J_I = 6.5$  Hz,  $J_2 = 2.2$  Hz, 3H), 1.68-1.63 (m, 2H), 1.61-1.55 (m, 1H), 1.52-1.45 (m, 1H), 1.43-1.38 (m, 1H), 0.93 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz CDCl<sub>3</sub>) (Major):  $\delta = 163.8$  (2C), 161.3, 149.2, 144.1-144.0 (m), 142.1-142.0 (m), 141.2-141.0 (m), 139.1, 139.0-138.7(m), 137.0, 136.9-136.8 (m), 134.3, 129.4 (2C), 128.8 (2C), 127.7 (2C), 123.3 (2C), 111.8-111.5 (m), 85.9, 84.9, 40.4, 35.5, 20.0, 18.6, 14.0, 9.8 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 626.1685; found: 626.1690.



(E)-N-(perfluorophenyl)-2-(((1-(thiophen-2-yl)pentan-2-yl)oxy)imino)propanamide (3ai): Follow the typical process an isolated as yellow oil (84 mg, 67% yield);  $R_f = 0.60$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.98$  (s, 1H), 7.17 (dd,  $J_I = 5.1$  Hz,  $J_2 = 1.1$  Hz, 1H), 6.95 (dd,  $J_I = 5.1$  Hz,  $J_2 = 3.4$  Hz, 1H), 6.85 (d, J = 3.5 Hz, 1H), 4.52 (dd,  $J_I = 7.6$  Hz,  $J_2 = 5.4$  Hz, 1H), 3.21 (d, J = 5.9 Hz, 2H), 2.13 (s, 3H), 1.78-1.70 (m, 1H), 1.69-1.63 (m, 1H), 1.57-1.49 (m, 1H), 1.49-1.41 (m, 1H), 0.97 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.3$ , 149.5, 144.1-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 139.9, 139.1-138.8 (m), 137.0-136.9 (m), 126.7, 126.1, 124.1, 111.8-111.7 (m), 85.6, 35.3, 34.7, 18.7, 14.0, 10.0 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 443.0823; found: 443.0826.



(E)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)pentyl acetate (4'): Follow the typical process an isolated as yellowish oil (124 mg, 83% yield);  $R_f = 0.56$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.13$  (s, 1H), 4.49 (m, 1H), 4.28 (d, J = 5.1 Hz, 2H), 2.12 (s, 3H), 2.09 (s, 3H), 1.81-1.69 (m, 1H), 1.67-1.59 (m, 1H), 1.54-1.40 (m, 2H), 0.98 (t, J = 7.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.9$ , 161.2, 150.0, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 139.0-138.8 (m), 137.1-136.8 (m), 111.7-111.4 (m), 82.8, 65.1, 32.7, 20.8, 18.5, 14.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 419.1001; found: 419.1005.



(E)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4a): Follow the typical process an isolated as yellow oil (101 mg, 95% yield);  $R_f = 0.40$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.14$  (s, 1H), 4.45 (dd,  $J_I = 5.8$  Hz,  $J_2 = 3.5$  Hz, 2H), 4.38 (dd,  $J_I = 5.8$  Hz,  $J_2 = 3.5$  Hz, 2H), 2.11 (s, 3H), 2.09 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.9$ , 161.0, 150.6, 144.2-144.1 (m), 142.2-142.1 (m), 141.2-141.0 (m), 139.0-138.7 (m), 136.9-136.7 (m), 111.6-111.3 (m), 73.4, 62.4, 20.8, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 377.0531; found: 377.0534.



(E)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)propyl acetate (4b): Follow the typical process an isolated as yellow oil (65 mg, 59% yield);  $R_f = 0.46$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.14$  (s, 1H), 4.65-4.58 (m, 1H), 4.29-4.21 (m, 2H), 2.10 (d, J = 6.2 Hz, 6H), 1.37 (d, J = 6.6 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.8$ , 161.2, 150.1, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 139.0-138.8 (m), 137.0-136.7 (m), 111.7-111.4 (m), 79.0, 65.9, 20.7, 16.4, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 391.0688; found: 391.0688.



(E)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)propane-1,3-diyl diacetate (4b'): Follow the typical process an isolated as yellow oil (99 mg, 78% yield);  $R_f$ = 0.28 (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.18 (s, 1H), 4.67 (p, *J* = 5.2 Hz, 1H), 4.35 (d, *J* = 5.2 Hz, 4H), 2.11 (s, 3H), 2.09 (s, 6H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$  = 170.6 (2C), 160.9, 151.3, 144.2-144.1 (m), 142.2-142.1 (m), 141.2-141.1 (m), 138.9-138.7 (m), 136.9-136.7 (m), 111.5-111.3 (m), 80.3, 62.2 (2C), 20.6 (2C), 10.1 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 449.0742; found: 449.0744.



(E)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)hexyl acetate (4c): Follow the typical process an isolated as yellow oil (81 mg, 66% yield);  $R_f = 0.21$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.13$  (s, 1H), 4.50-4.45 (m, 1H), 4.28 (d, J = 5.1 Hz, 2H), 2.12 (s, 3H), 2.09 (s, 3H), 1.79-1.72 (m, 1H), 1.69-1.63 (m, 2H), 1.48-1.43 (m, 1H), 1.41-1.36 (m, 2H), 0.94 (t, J = 7.1 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.9$ , 161.2, 150.0, 144.2-144.0 (m), 142.2-142.1 (m), 141.1-140.9 (m), 139.1-138.9 (m), 136.9-136.7 (m), 111.7-111.4 (m), 83.0, 65.1, 30.4, 27.4, 22.6, 20.8, 13.9, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 433.1157; found: 433.1165.



(E)-3-methyl-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)butyl acetate (4d): Follow the typical process an isolated as yellow oil (114 mg, 96% yield);  $R_f = 0.21$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.18$  (s, 1H), 4.34-4.26 (m, 2H), 4.25-4.21 (m, 1H), 2.12-2.03 (m, 7H), 1.01 (dd,  $J_I = 9.9$  Hz,  $J_2 = 6.9$  Hz, 6H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.8$ , 161.2, 149.8, 144.2-144.0 (m), 142.2-142.0 (m), 141.1-140.9 (m), 139.1-138.6 (m), 136.9-136.6 (m), 111.8-111.5 (m), 87.7, 63.5, 29.3, 20.6, 18.3, 17.9, 9.7 ppm; HRMS (ESI-TOF): calcd. [M+Na]+ 419.1001; found: 419.1005.



(E)-2-cyclohexyl-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4e): Follow the typical process an isolated as colourless oil (129 mg, 93% yield);  $R_f = 0.37$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.13$  (s, 1H), 4.38-4.25 (m, 3H), 2.12 (s, 3H), 2.09 (s, 3H), 1.88-1.69 (m, 6H), 1.31-1.26 (m, 2H), 1.23-1.09 (m, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 171.0$ , 161.2, 149.7, 144.2-144.0 (m), 142.2-142.0 (m), 141.1-141.0 (m), 139.1-138.8 (m), 137.0-136.8

(m), 111.7-111.5 (m), 87.3, 63.6, 39.1, 28.8, 28.6, 26.3, 26.0, 25.9, 20.9, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 459.1314; found: 459.1321.



(E)-5-chloro-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)pentyl acetate (4f): Follow the typical process an isolated as yellow oil (108 mg, 84% yield);  $R_f = 0.42$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.10$  (s, 1H), 4.52 (p, J = 5.2 Hz, 1H), 4.33-4.26 (m, 2H), 3.62 (t, J = 5.6 Hz, 2H), 2.13 (s, 3H), 2.10 (s, 3H), 2.00-1.87 (m, 4H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.8$ , 161.0, 150.6, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-140.9 (m), 139.1-139.0 (m), 136.8-136.7 (m), 111.5-111.3 (m), 82.1, 64.8, 44.5, 28.3, 28.0, 20.8, 10.0 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 453.0611; found: 453.0611.



(E)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)propane-1,3-diyl diacetate (4g): Follow the typical process an isolated as yellow oil (112 mg, 88% yield);  $R_f = 0.28$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.18$  (s, 1H), 4.67 (p, J = 5.2 Hz, 1H), 4.35 (d, J = 5.2 Hz, 4H), 2.11 (s, 3H), 2.09 (s, 6H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.6$  (2C), 160.9, 151.3, 144.2-144.1 (m), 142.2-142.1 (m), 141.2-141.1 (m), 138.9-138.7 (m), 136.9-136.7 (m), 111.5-111.3 (m), 80.3, 62.2 (2C), 20.6 (2C), 10.1 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 449.0742; found: 449.0744.



(E)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)-1-phenylethyl acetate (4h): Follow the typical process an isolated as yellowish oil (126 mg, 98% yield);  $R_f = 0.51$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.07$  (s, 1H), 7.43-7.32 (m, 5H), 6.17 (dd,  $J_I = 7.8$  Hz,  $J_2 = 4.1$  Hz, 1H), 4.56 (dd,  $J_I = 12.0$  Hz,  $J_2 = 7.9$  Hz, 1H), 4.47 (dd,  $J_I = 11.9$  Hz,  $J_2 = 4.1$  Hz, 1H), 2.14 (s, 3H), 2.09 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.0$ , 161.0, 150.6, 144.2-144.1 (m), 142.2-142.1 (m), 141.2-141.0 (m), 138.9-138.7 (m), 136.9-136.8 (m), 136.6, 128.6, 128.6 (2C), 126.7 (2C), 111.6-111.4 (m), 77.5, 73.6, 21.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 453.0844; found: 453.0847.



(E)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)-1-(*p*-tolyl)ethyl acetate (4i): Follow the typical process an isolated as yellow oil (123 mg, 92% yield);  $R_f = 0.22$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.03$  (s, 1H), 7.31-7.26 (m, 2H), 7.22-7.18 (m, 2H), 6.13 (dd,  $J_I = 8.0$  Hz,  $J_2 = 4.0$  Hz, 1H), 4.55 (dd,  $J_I = 11.9$  Hz,  $J_2 = 8.0$  Hz, 1H), 4.45 (dd,  $J_I = 11.9$  Hz,  $J_2$ = 4.1 Hz, 1H), 2.36 (d, J = 3.6 Hz, 3H), 2.13 (s, 3H), 2.09 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta =$ 170.1, 161.0, 150.5, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-141.1 (m), 139.2-139.1 (m), 138.5, 136.9-136.8 (m), 133.6, 129.3, 129.3, 126.8, 126.7, 111.6-111.3 (m), 77.5, 73.5, 21.1, 21.1, 10.0 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 467.1001; found: 467.1001.



(E)-1-(4-methoxyphenyl)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4j): Follow the typical process an isolated as yellow oil (132 mg, 96% yield);  $R_f = 0.41$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.04$  (s, 1H), 7.34-7.31 (m, 2H), 6.93-6.90 (m, 2H), 6.12 (dd,  $J_I = 8.0 \text{ Hz}, J_2 = 4.2 \text{ Hz}, 1\text{ H})$ , 4.55 (dd,  $J_I = 11.9 \text{ Hz}, J_2 = 8.0 \text{ Hz}, 1\text{ H})$ , 4.44 (dd,  $J_I = 11.9 \text{ Hz}, J_2 = 4.2 \text{ Hz}, 1\text{ H})$ , 3.81 (s, 3H), 2.12 (s, 3H), 2.09 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.1$ , 161.0, 159.9, 150.5, 144.2-144.1 (m), 142.2-142.1 (m), 141.2-141.1 (m), 139.2-139.0 (m), 137.0-136.8 (m), 128.6, 128.3 (2C), 114.1 (2C), 111.6-111.3 (m), 77.4, 73.2, 55.3, 21.1, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 483.0950; found: 483.0953.



(E)-1-(naphthalen-1-yl)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4k): Follow the typical process an isolated as red soild (134 mg, 93% yield);  $R_f = 0.46$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.20$  (d, J = 8.5 Hz, 1H), 7.98 (s, 1H), 7.88 (dd,  $J_I = 17.8$  Hz,  $J_2 = 8.1$  Hz, 2H), 7.68-7.46 (m, 4H), 6.99 (dd,  $J_I = 7.8$  Hz,  $J_2 = 3.7$  Hz, 1H), 4.71 (dd,  $J_I = 12.2$  Hz,  $J_2 = 7.8$  Hz, 1H), 4.65 (dd,  $J_I = 12.2$  Hz,  $J_2 = 3.7$  Hz, 1H), 2.21 (s, 3H), 2.09 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.0$ , 160.9, 150.7, 144.2-143.9 (m), 142.1-142.0 (m), 141.1-141.0 (m), 138.9-138.7 (m), 136.9-136.7 (m), 133.8, 132.4, 130.4, 129.1, 129.0, 126.7, 125.9, 125.3, 124.5, 122.7, 111.6-111.3 (m), 77.4, 71.0, 21.1, 10.0 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 503.1001; found: 503.1010.



(E)-1-(4-fluorophenyl)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4l): Follow the typical process an isolated as yellow oil (132 mg, 98% yield);  $R_f = 0.25$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.07$  (s, 1H), 7.39-7.36 (m, 2H), 7.10-7.05 (m, 2H), 6.13 (dd,  $J_I = 7.8$  Hz,  $J_2 = 4.2$  Hz, 1H), 4.53 (dd,  $J_I = 11.9$  Hz,  $J_2 = 7.8$  Hz, 1H), 4.44 (dd,  $J_I = 11.9$  Hz,  $J_2 = 4.2$  Hz, 1H), 2.13 (s, 3H), 2.08 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.0$ , 162.8 (d, J = 247.6 Hz), 160.9, 150.8, 144.3-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 138.8-138.7 (m), 136.9-136.7 (m), 132.5 (d, J = 3.0 Hz), 128.6 (d, J = 8.2 Hz, 2C), 115.7 (d, J = 21.7 Hz, 2C), 111.6-111.3 (m), 77.3, 72.9, 21.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 471.0750; found: 471.0754.



(E)-1-(4-chlorophenyl)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4m): Follow the typical process an isolated as yellow solid (135 mg, 97% yield);  $R_f = 0.27$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.02$  (s, 1H), 7.38-7.31 (m, 4H), 6.12 (dd,  $J_I = 7.6$  Hz,  $J_2 = 4.2$  Hz, 1H), 4.52 (dd,  $J_I = 11.9$  Hz,  $J_2 = 7.6$  Hz, 1H), 4.44 (dd,  $J_I = 11.9$  Hz,  $J_2 = 4.2$  Hz, 1H), 2.14 (s, 3H), 2.08 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 169.9$ , 160.8, 150.8, 144.2-144.0 (m), 142.2-142.0 (m), 141.2-141.0 (m), 138.9-138.7 (m), 136.9-136.6 (m), 135.2, 134.5, 128.8 (2C), 128.1 (2C), 111.5-111.3 (m), 77.1, 72.9, 20.9, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 487.0454; found: 487.0454.



(E)-1-(3-chlorophenyl)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4n): Follow the typical process an isolated as yellow oil (135 mg, 97% yield);  $R_f = 0.23$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.03$  (s, 1H), 7.39 (s, 1H), 7.35-7.30 (m, 2H), 7.28-7.25 (m, 1H), 6.11 (dd,  $J_I = 7.5$  Hz,  $J_2 = 4.1$  Hz, 1H), 4.53 (dd,  $J_I = 12.0$  Hz,  $J_2 = 7.6$  Hz, 1H), 4.46 (dd,  $J_I = 12.0$  Hz,  $J_2 = 4.2$  Hz, 1H), 2.16 (s, 3H), 2.09 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 169.9$ , 160.8, 150.9, 144.2-144.0 (m), 142.2-142.1 (m), 141.2-141.1 (m), 139.2-139.0 (m), 138.6, 136.9-136.7 (m), 134.7, 130.0, 128.8, 126.9, 124.9, 111.5-111.2 (m), 77.2, 72.9, 21.0, 10.0 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 487.0454; found: 487.0458.



(E)-1-(4-bromophenyl)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (40): Follow the typical process an isolated as yellow oil (148 mg, 97% yield);  $R_f = 0.46$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.00$  (s, 1H), 7.54-7.51 (m, 2H), 7.29-7.26 (m, 2H), 6.10 (dd,  $J_1 = 7.5$  Hz,  $J_2 = 4.3$  Hz, 1H), 4.52 (dd,  $J_1 = 11.9$  Hz,  $J_2 = 7.5$  Hz, 1H), 4.44 (dd,  $J_1 = 11.9$  Hz,  $J_2 = 4.3$  Hz, 1H), 2.14 (s, 3H), 2.08 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 169.9$ , 160.8, 150.8, 144.2-144.0 (m), 142.2-142.1 (m), 141.2-141.1 (m), 138.9-138.7 (m), 136.9-136.7 (m), 135.7, 131.9 (2C), 128.5 (2C), 122.6, 111.5-111.3 (m), 77.1, 72.9, 21.0, 10.0 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 530.9949; found: 530.9954.



(E)-1-(2-bromophenyl)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4p): Follow the typical process an isolated as yellow oil (98 mg, 84% yield);  $R_f = 0.50$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.05$  (s, 1H), 7.59 (dd,  $J_I = 8.0$  Hz,  $J_2 = 1.0$  Hz, 1H), 7.46 (dd,  $J_I = 7.8$  Hz,  $J_2 = 1.6$  Hz, 1H), 7.36 (td,  $J_I = 7.5$  Hz,  $J_2 = 1.0$  Hz, 1H), 7.21 (td,  $J_I = 7.8$  Hz,  $J_2 = 1.7$  Hz, 1H), 6.51 (dd,  $J_I = 7.0$  Hz,  $J_2 = 3.5$  Hz, 1H), 4.57-4.47 (m, 2H), 2.17 (s, 3H), 2.09 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 169.6$ , 160.8, 150.5, 144.1-143.9 (m), 142.1-141.9 (m), 141.1-141.0 (m), 139.1-138.9 (m), 136.9-136.8 (m), 136.0, 133.0, 129.9, 128.0, 127.7, 122.3, 111.7-111.4 (m), 76.2, 73.0, 20.9, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 530.9949; found: 530.9954.



(E)-1-(2-iodophenyl)-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4q): Follow the typical process an isolated as yellow solid (100 mg, 60% yield);  $R_f = 0.50$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.10$  (s, 1H), 7.90-7.84 (m, 1H), 7.44-7.37 (m, 2H), 7.06-7.02 (m, 1H), 6.36 (dd,  $J_I = 7.3$  Hz,  $J_2 = 3.5$  Hz, 1H), 4.52-4.43 (m, 2H), 2.17 (s, 3H), 2.11 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 169.7$ , 160.8, 150.6, 144.1-143.9 (m), 142.1-141.9 (m), 141.1-141.0 (m), 139.7, 139.0, 138.9-138.8 (m), 137.0-136.8 (m), 130.2, 128.6, 127.7, 111.6-111.4 (m), 97.5, 77.3, 76.4, 21.0, 10.0 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 578.9811; found: 578.9814.



Methyl(E)-3-acetoxy-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)-3phenylpropanoate (4r): Follow the typical process an isolated as white soild (138 mg, 96% yield);  $R_{f}$ = 0.29 (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.85 (s, 1H), 7.42-7.34 (m, 5H), 6.36 (d, J = 5.4 Hz, 1H), 5.07 (d, J = 5.4 Hz, 1H), 3.70 (s, 3H), 2.18 (s, 3H), 2.16 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ = 169.6, 168.2, 160.5, 152.1, 144.1-144.0 (m), 142.2-142.0 (m), 141.2-141.1 (m), 139.1-139.0 (m), 136.9-136.7 (m), 135.3, 129.0, 128.6 (2C), 127.1 (2C), 111.3-111.1 (m), 85.1, 73.9, 52.5, 20.9, 10.3 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 511.0899; found: 511.0906.



(E)-1-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)-3-phenylpropan-2-yl

**acetate (4s):** Follow the typical process an isolated as white soild (109 mg, 82% yield);  $R_f = 0.31$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.09$  (s, 1H), 7.33 (t, J = 7.3 Hz, 2H), 7.26-7.23 (m, 3H), 5.41 (qd,  $J_I = 6.8$  Hz,  $J_2 = 3.7$  Hz, 1H), 4.37 (dd,  $J_I = 11.8$  Hz,  $J_2 = 3.7$  Hz, 1H), 4.30 (dd,  $J_I = 11.8$  Hz,  $J_2 = 6.2$  Hz, 1H), 3.02-2.92 (m, 2H), 2.12 (s, 3H), 2.06 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.3$ , 161.0, 150.5, 144.2-144.1 (m), 142.2-141.0 (m), 141.2-141.1 (m), 139.0-138.8 (m), 137.0-136.8 (m), 136.3, 129.4 (2C), 128.6 (2C), 126.9, 111.7-111.3 (m), 75.7, 72.5, 37.2, 21.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 467.1001; found: 467.1001.



(E)-1-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)-4-phenylbutan-2-yl acetate (4t): Follow the typical process an isolated as yellow oil (96 mg, 70% yield);  $R_f = 0.53$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.09$  (s, 1H), 7.31 (t, J = 7.5 Hz, 2H), 7.21 (t, J = 8.6 Hz, 3H), 5.28-5.23 (m, 1H), 4.35 (qd,  $J_I = 11.8$  Hz,  $J_2 = 5.1$  Hz, 2H), 2.81-2.64 (m, 2H), 2.10 (d, J = 5.7 Hz, 6H), 2.06-1.91 (m, 2H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.6$ , 161.0, 150.4, 144.2-144.0 (m), 142.3-141.0 (m), 141.3-141.1 (m), 140.9, 139.0-138.8 (m), 137.0-136.8 (m), 128.5 (2C), 128.3 (2C), 126.2, 111.7-111.4 (m), 76.5, 71.4, 32.4, 31.5, 21.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 481.1157; found: 481.1159.


(E)-1-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)pentan-2-yl acetate (4u): Follow the typical process an isolated as colourless oil (77 mg, 65% yield);  $R_f = 0.20$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.14$  (s, 1H), 5.27-5.21 (m, 1H), 4.32 (qd,  $J_I = 11.8$  Hz,  $J_2 = 5.2$  Hz, 2H), 2.10 (s, 3H), 2.08 (s, 3H), 1.65-1.58 (m, 2H), 1.46-1.35 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.7$ , 161.1, 150.4, 144.2-144.1 (m), 142.2-141.0 (m), 141.2-141.0 (m), 139.1-139.0 (m), 137.2-137.0 (m), 111.7-111.6 (m), 76.7, 71.7, 32.9, 21.0, 18.5, 13.9, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 419.1001; found: 419.1004.



(E)-1-cyclohexyl-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)ethyl acetate (4v): Follow the typical process an isolated as colourless oil (60 mg, 46% yield);  $R_f = 0.42$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.13$  (s, 1H), 5.07 (td,  $J_I = 6.9$  Hz,  $J_2 = 3.3$  Hz, 1H), 4.42 (dd,  $J_I = 11.8$  Hz,  $J_2 = 3.3$  Hz, 1H), 4.34 (dd,  $J_I = 11.8$  Hz,  $J_2 = 7.0$  Hz, 1H), 2.09 (s, 6H), 1.80-1.63 (m, 5H), 1.29-1.06 (m, 6H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 170.7$ , 161.1, 150.3, 144.2-143.9 (m), 142.2-141.0 (m), 141.2-141.0 (m), 138.9-138.8 (m), 136.9-136.7 (m), 111.6-111.4 (m), 75.4, 75.3, 38.9, 29.0, 28.3, 26.2, 25.9, 25.8, 21.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 459.1314; found: 459.1317.



(E)-3-isopropyl-6-methyl-2-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)
cyclohexyl acetate (4w): Follow the typical process an isolated as yellow solid (71 mg, 51% yield (与正 文不一致)); R<sub>f</sub>= 0.32 (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 8.45 (s, 1H),
5.12-5.05 (m, 1H), 4.71-4.68 (m, 1H), 2.21-2.13 (m, 1H), 2.09 (s, 3H), 2.03 (s, 3H), 2.02-1.95 (m, 1H),
1.83-1.76 (m, 1H), 1.68-1.60 (m, 2H), 1.57-1.47 (m, 2H), 1.19 (d, J = 6.3 Hz, 3H), 0.96 (d, J = 6.1 Hz,
3H), 0.92 (d, J = 6.3 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ = 171.7, 162.0, 150.6, 144.3-144.2
(m), 142.4-142.2 (m), 141.1-141.0 (m), 139.1-138.9 (m), 136.9-136.8 (m), 112.0-111.8 (m), 86.2, 70.6, 52.4, 51.0, 28.9, 27.4, 24.5, 21.8, 21.7, 21.5, 19.3, 10.3 ppm; HRMS (ESI-TOF): calcd.  $[M+Na]^+$ 487.1627; found: 487.1628. Follow the typical process an isolated as yellow solid (52 mg, 37% yield); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.09$  (s, 1H), 4.92 (s, 1H), 4.42 (dd,  $J_I = 11.3$  Hz,  $J_2 = 2.5$  Hz, 1H), 2.14 (s, 3H), 2.05 (s, 3H), 1.97-1.90 (m, 1H), 1.89-1.83 (m, 1H), 1.76-1.70 (m, 1H), 1.65-1.57 (m, 1H), 1.40-1.29 (m, 1H), 1.27-1.19 (m, 1H), 1.14-1.05 (m, 1H), 0.94 (dd,  $J_I = 8.7$  Hz,  $J_2 = 6.6$  Hz, 9H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 171.1$ , 161.4, 148.8, 144.3-144.2 (m), 142.3-142.1 (m), 141.2-141.1 (m), 138.9-138.7 (m), 137.0-136.7 (m), 111.8-111.4 (m), 82.7, 80.1, 47.0, 32.6, 31.1, 29.0, 24.3, 20.9, 20.8, 20.7, 18.0, 9.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 487.1627; found: 487.1630.



(E)-1-(((1-oxo-1-((perfluorophenyl)amino)propan-2-ylidene)amino)oxy)butan-2-yl acetate (4x): Follow the typical process an isolated as yellowish oil (95 mg, 83% yield);  $R_f = 0.20$  (petroleum ether-EtOAc = 10:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.11$  (s, 1H), 5.18-5.13 (m, 1H), 4.38-4.29 (m, 2H), 2.10 (d, J = 5.2 Hz, 6H), 1.74-1.61 (m, 2H), 0.99 (t, J = 7.5 Hz, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$ = 170.7, 161.0, 150.4, 144.2-144.0 (m), 142.2-142.0 (m), 141.3-141.1 (m), 138.9-138.7 (m), 137.1-136.8 (m), 111.6-111.4 (m), 76.3, 73.0, 23.9, 21.0, 9.9, 9.5 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 405.0844; found: 405.0844.



**1-([1,1'-biphenyl]-4-yl)pentan-2-ol (5):** White soild;  $R_f = 0.49$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.63-7.56$  (m, 4H), 7.48-7.44 (m, 2H), 7.38-7.30 (m, 3H), 3.90 (s, 1H), 2.90 (dd,  $J_I = 13.6$  Hz,  $J_2 = 4.2$  Hz, 1H), 2.72 (dd,  $J_I = 13.6$  Hz,  $J_2 = 8.4$  Hz, 1H), 1.65-1.51 (m, 4H), 1.49-1.39 (m, 1H), 1.02-0.95 (m, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 140.9$ , 139.4, 137.8, 129.8 (2C), 128.8 (2C), 127.3 (2C), 127.1 (2C), 127.0, 72.4, 43.7, 39.1, 19.0, 14.1 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 263.1406; found: 263.1409.



**1-(4-chlorophenyl)-2-hydroxyethyl acetate (6):** White soild;  $R_f = 0.20$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 7.37-7.32$  (m, 4H), 4.95 (dd,  $J_I = 8.3$  Hz,  $J_2 = 3.3$  Hz, 1H), 4.27 (dd,  $J_I = 11.6$  Hz,  $J_2 = 3.3$  Hz, 1H), 4.13 (dd,  $J_I = 11.6$  Hz,  $J_2 = 8.3$  Hz, 1H), 2.12 (s, 3H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 171.2$ , 138.2, 134.0, 128.8 (2C), 127.5 (2C), 71.8, 69.1, 20.9 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 273.0289; found: 273.0289.



(S,E)-2-((2-hydroxy-2-(*p*-tolyl)ethoxy)imino)-N-(perfluorophenyl)propanamide (7): Colourless oil;  $R_f = 0.20$  (petroleum ether-EtOAc = 5:1); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta = 8.03$  (s, 1H), 7.31 (d, J = 8.0Hz, 2H), 7.21 (d, J = 7.9 Hz, 2H), 5.06 (t, J = 5.5 Hz, 1H), 4.41 (d, J = 5.9 Hz, 2H), 2.36 (s, 3H), 2.15 (s, 3H), 1.65 (s, 1H) ppm; <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta = 161.0$ , 150.6, 144.2-144.1 (m), 142.2-142.0 (m), 141.2-141.1 (m), 139.0-138.8 (m), 138.1, 137.1-137.0 (m), 136.9, 129.3 (2C), 126.2 (2C), 111.5-111.3 (m), 80.5, 72.6, 21.1, 10.1 ppm; HRMS (ESI-TOF): calcd. [M+Na]<sup>+</sup> 425.0895; found: 425.0895.

## XI. NMR Spectra













































-0.020

180607.18080732.fid wbx20180607-2 CDCl3 0607









180410.18043421.fid wbx20180410 CDCl3 0410






























ppm 





-0.029


























































































-0.017





















150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 ppm



