



## Supporting Information

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### ***N*-Aryl Isoleucine Derivatives as Angiotensin II AT<sub>2</sub> Receptor Ligands**

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open\_201300040\_sm\_miscellaneous\_information.pdf

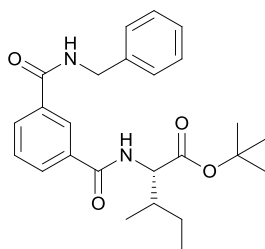
## Supporting information

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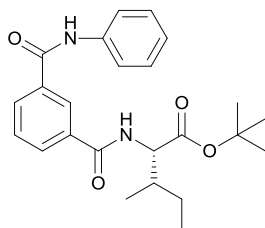
## 1. Experimental data for compounds 9–36 and 65–67.

### (2S,3R)-tert-Butyl-2-(3-(benzylcarbamoyl)benzamido)-3-methylpentanoate (9).



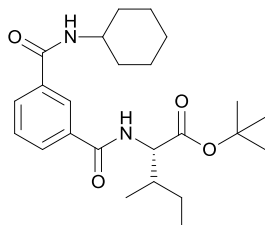
Yield: 165 mg (78%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.91 – 1.00 (m, 6H), 1.20 – 1.29 (m, 1H), 1.44 – 1.57 (m, 1H), 1.48 (s, 9H), 1.91 – 2.05 (m, 1H), 4.63 (d,  $J$  = 5.6 Hz, 2H), 4.68 (dd,  $J$  = 8.3 Hz, 4.5 Hz, 1H), 6.71 (s, 1H), 6.83 (d,  $J$  = 7.8 Hz, 1H), 7.24 – 7.36 (m, 5H), 7.49 (dt,  $J$  = 7.8 Hz, 1.4 Hz, 1H), 7.90 (dd,  $J$  = 7.7 Hz, 0.9 Hz, 1H), 7.97 (dd,  $J$  = 7.8 Hz, 0.6 Hz, 1H), 8.20 (t,  $J$  = 1.6 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 15.4, 25.5, 28.1, 38.4, 44.2, 57.2, 82.4, 125.4, 127.6, 127.9, 128.8, 128.9, 129.7, 130.4, 134.5, 134.8, 137.9, 166.1, 166.4, 171.0.

### (2S,3R)-tert-Butyl-3-methyl-2-(3-(phenylcarbamoyl)benzamido)pentanoate (10).



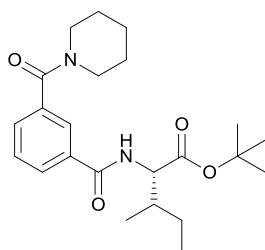
Yield: 133 mg (66%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.96 (t,  $J$  = 7.5 Hz, 3H), 0.97 (d,  $J$  = 6.9 Hz, 3H), 1.24 – 1.33 (m, 1H), 1.45 – 1.57 (m, 1H), 1.47 (s, 9H), 1.97 – 2.03 (m, 1H), 4.70 (dd,  $J$  = 8.3 Hz, 4.7 Hz, 1H), 7.05 (d,  $J$  = 8.2 Hz, 1H), 7.15 (ddt,  $J$  = 7.9 Hz, 4.7 Hz, 1.1 Hz, 1H), 7.34 – 7.39 (m, 2H), 7.50 (dt,  $J$  = 7.8 Hz, 3.9 Hz, 1H), 7.69 (dd,  $J$  = 8.5 Hz, 1.0 Hz, 2H), 7.88 (ddd,  $J$  = 7.8 Hz, 1.7 Hz, 1.2 Hz, 1H), 8.00 – 8.03 (m, 1H), 8.23 (t,  $J$  = 1.6 Hz, 1H), 8.34 (s br, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 15.5, 25.6, 28.0, 38.3, 57.4, 82.6, 120.3, 124.6, 125.2, 129.0, 129.1, 130.0, 130.7, 134.4, 135.7, 138.0, 165.2, 166.2, 171.1.

### (2S,3R)-tert-Butyl-2-(3-(cyclohexylcarbamoyl)benzamido)-3-methylpentanoate (11).



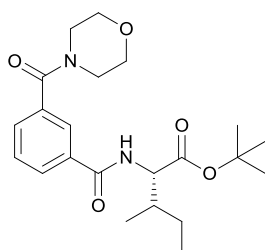
Yield: 145 mg (70%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.94 – 1.00 (m, 6H), 1.18 – 1.33 (m, 5H), 1.47 – 1.60 (m, 1H), 1.49 (s, 9H), 1.64 (s, 2H), 1.72 – 1.79 (m, 2H), 2.01 (m,  $J$  = 8.0 Hz, 3.0 Hz, 3H), 3.96 (dt,  $J$  = 10.8 Hz, 9.4 Hz, 1H), 4.69 (dd,  $J$  = 8.3 Hz, 4.5 Hz, 1H), 6.16 (s br, 1H), 6.81 (d,  $J$  = 7.7 Hz, 1H), 7.49 (t,  $J$  = 7.7 Hz, 1H), 7.86 – 7.89 (m, 1H), 7.93 – 7.97 (m, 1H), 8.17 (t,  $J$  = 1.7 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 15.4, 24.9, 25.5, 25.5, 25.6, 28.1, 33.2, 33.9, 38.4, 49.0, 57.2, 82.4, 125.3, 128.9, 129.3, 130.4, 134.4, 135.5, 165.6, 166.2, 171.0.

**(2S,3R)-tert-Butyl-3-methyl-2-(3-(piperidine-1-carbonyl)benzamido)pentanoate (12).**



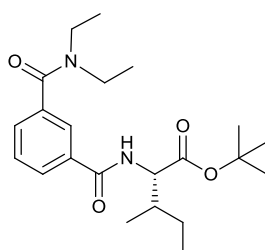
Yield: 153 mg (76%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.93 – 0.99 (m, 6H), 1.21 – 1.33 (m, 1H), 1.48 (s, 9H), 1.49 – 1.58 (m, 3H), 1.63 – 1.71 (m, 4H), 1.93 – 2.01 (m, 1H), 3.39 – 3.22 (m, 2H), 3.62 – 3.79 (m, 2H), 4.68 (dd,  $J$  = 8.3 Hz, 4.5 Hz, 1H), 6.71 (d,  $J$  = 8.1 Hz, 1H), 7.43 – 7.53 (m, 2H), 7.80 (s, 1H), 7.83 (dt,  $J$  = 7.4 Hz, 1.4 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 15.4, 24.5, 25.5, 26.5, 28.1, 38.4, 43.2, 48.8, 57.1, 82.3, 125.4, 128.0, 128.7, 129.7, 134.8, 136.9, 166.2, 169.2, 171.1.

**(2S,3R)-tert-Butyl-3-methyl-2-(3-(morpholine-4-carbonyl)benzamido)pentanoate (13).**



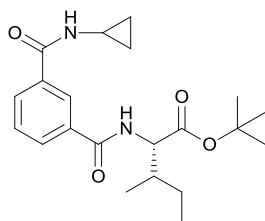
Yield: 115 mg (57%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.99 – 0.92 (m, 6H), 1.32 – 1.18 (m, 1H), 1.47 (s, 9H), 1.54 – 1.49 (m, 1H), 1.96 (dd,  $J$  = 8.2 Hz, 3.3 Hz, 1H), 3.43 (s br, 2H), 3.63 (s br, 2H), 3.75 (s br, 4H), 4.67 (dd,  $J$  = 8.2 Hz, 4.5 Hz, 1H), 6.77 (d,  $J$  = 8.1 Hz, 1H), 7.53 – 7.44 (m, 2H), 7.82 – 7.86 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 15.3, 25.5, 28.0, 38.4, 42.6, 48.2, 57.1, 66.8, 82.4, 125.8, 128.4, 128.9, 130.0, 134.9, 135.7, 166.0, 169.4, 171.0.

**(2S,3R)-tert-Butyl-2-(3-(diethylcarbamoyl)benzamido)-3-methylpentanoate (14).**



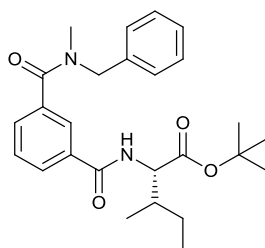
Yield: 45 mg (24%, colourless oil);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.93 (d,  $J$  = 6.9 Hz, 3H), 0.94 (t,  $J$  = 7.4 Hz, 3H), 1.02 – 1.15 (m, 3H), 1.16 – 1.30 (m, 4H), 1.45 (s, 9H), 1.43 – 1.56 (m, 1H), 1.95 (ddt,  $J$  = 11.4 Hz, 6.8 Hz, 2.2 Hz, 1H), 3.20 (s, 2H), 3.51 (s, 2H), 4.65 (dd,  $J$  = 8.2 Hz, 4.5 Hz, 1H), 6.73 (d,  $J$  = 8.2 Hz, 1H), 7.43 (t,  $J$  = 7.5 Hz, 1H), 7.47 (ddd,  $J$  = 7.6 Hz, 1.6 Hz, 1.1 Hz, 1H), 7.76 (t,  $J$  = 1.6 Hz, 1H), 7.80 (dt,  $J$  = 7.3 Hz, 1.6 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 12.7, 14.1, 15.3, 25.5, 28.0, 38.3, 39.3, 43.3, 57.1, 82.2, 124.9, 127.7, 128.7, 129.2, 134.6, 137.6, 166.2, 170.2, 170.9.

**(2S,3R)-tert-Butyl-2-(3-(cyclopropylcarbamoyl)benzamido)-3-methylpentanoate (15).**



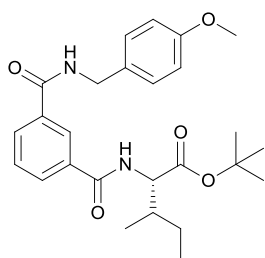
Yield: 78 mg (42%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.58 – 0.63 (m, 2H), 0.77 – 0.85 (m, 2H), 0.90 – 0.96 (m, 6H), 1.17 – 1.31 (m, 1H), 1.46 (s, 9H), 1.44 – 1.56 (m, 1H), 1.90 – 2.02 (m, 1H), 2.83 – 2.91 (m, 1H), 4.65 (dd,  $J = 8.3$  Hz, 4.6 Hz, 1H), 6.79 (s, 1H), 6.91 (d,  $J = 8.2$  Hz, 1H), 7.42 (t,  $J = 7.4$  Hz, 1H), 7.85 (d,  $J = 7.8$  Hz, 1H), 7.91 (d,  $J = 7.8$  Hz, 1H), 8.12 (t,  $J = 1.6$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 6.5, 6.6, 11.7, 15.4, 23.2, 25.5, 28.0, 38.3, 57.2, 82.3, 125.2, 128.8, 129.6, 130.4, 134.3, 134.8, 166.2, 168.1, 171.0.

**(2S,3R)-tert-Butyl-2-(3-(benzyl(methyl)carbamoyl)benzamido)-3-methylpentanoate (16).**



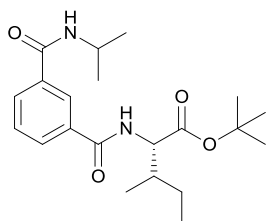
Yield: 165 mg (75%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.90 – 1.00 (m, 6H), 1.18 – 1.32 (m, 1H), 1.48 (s, 9H), 1.46 – 1.58 (m, 1H), 1.93 – 2.04 (m, 1H), 2.84 – 3.08 (m, 3H), 4.43 – 4.73 (m, 2H), 4.76 (s br, 1H), 6.63 – 6.78 (m, 1H), 7.13 – 7.22 (m, 1H), 7.27 – 7.32 (m, 1H), 7.35 (s br, 3H), 7.41 – 7.53 (m, 1H), 7.58 (d,  $J = 7.5$ , 1H), 7.82 – 7.87 (m, 1H), 7.89 (s br, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 15.4, 25.5, 28.1, 33.3, 37.0, 38.4, 50.8, 55.1, 57.1, 82.3, 125.7, 126.6, 127.6, 128.0, 128.2, 128.4, 128.7, 128.9, 130.0, 134.8, 136.8, 138.8, 166.1, 168.3, 171.0.

**(2S,3R)-tert-Butyl-2-(3-((4-methoxybenzyl)carbamoyl)benzamido)-3-methylpentanoate (17).**



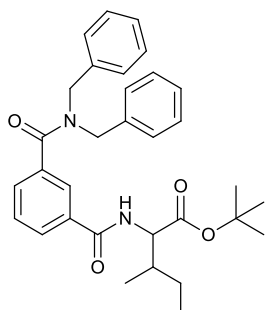
Yield: 193 mg (85%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.93 – 1.00 (m, 6H), 1.22 – 1.33 (m, 1H), 1.49 (s, 9H), 1.49 – 1.58 (m, 1H), 1.98 (td,  $J = 8.6, 4.3$ , 1H), 3.79 (s, 3H), 4.57 (t,  $J = 5.4$ , 2H), 4.66 – 4.71 (m, 1H), 6.51 – 6.64 (m, 1H), 6.81 (t,  $J = 9.2$ , 1H), 6.85 – 6.90 (m, 2H), 7.25 – 7.30 (m, 2H), 7.46 – 7.53 (m, 1H), 7.88 – 7.92 (m, 1H), 7.94 – 7.99 (m, 1H), 8.19 (s br, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 15.4, 25.5, 28.1, 38.4, 43.8, 55.3, 57.2, 82.4, 114.2, 125.4, 128.9, 129.4, 129.7, 129.9, 130.4, 134.5, 134.9, 159.1, 166.1, 166.3, 171.0.

**(2S,3R)-tert-Butyl-2-(3-(isopropylcarbamoyl)benzamido)-3-methylpentanoate (18).**



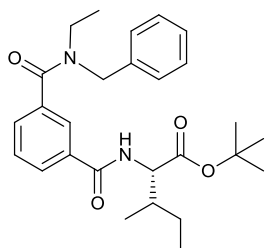
Yield: 103 mg (55%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.87 – 0.96 (m, 6H), 1.18 – 1.22 (m, 6H), 1.23 – 1.30 (m, 1H), 1.45 (s, 9H), 1.47 – 1.53 (m, 1H), 1.89 – 1.99 (m, 1H), 4.18 – 4.29 (m, 1H), 4.61 – 4.67 (m, 1H), 6.34 – 6.44 (m, 1H), 6.86 – 6.93 (m, 1H), 7.37 – 7.45 (m, 1H), 7.84 (dd,  $J = 7.7$  Hz, 1.0 Hz, 1H), 7.90 (dd,  $J = 7.7$  Hz, 1.0 Hz, 1H), 8.15 (s br, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.6, 15.3, 22.6, 25.4, 28.0, 38.3, 42.0, 57.2, 82.2, 125.3, 128.7, 129.4, 130.3, 134.3, 135.3, 165.7, 166.2, 170.9.

**(2S,3R)-tert-Butyl-2-(3-(dibenzylcarbamoyl)benzamido)-3-methylpentanoate (19).**



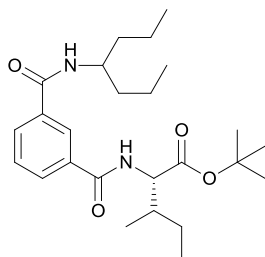
Yield: 72 mg (14%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.94 (d,  $J = 7.0$  Hz, 3H), 0.95 – 1.00 (m, 3H), 1.17 – 1.30 (m, 1H), 1.46 – 1.58 (m, 1H), 1.49 (s, 9H), 1.92 – 2.02 (m, 1H), 4.39 (s br, 2H), 4.67 (dd,  $J = 8.3$  Hz, 4.5 Hz, 1H), 4.68 – 4.82 (m, 2H), 6.67 (d,  $J = 8.2$  Hz, 1H), 7.08 – 7.18 (m, 2H), 7.26 – 7.39 (m, 8H), 7.45 (t,  $J = 7.8$  Hz, 1H), 7.59 – 7.64 (m, 1H), 7.83 (ddd,  $J = 7.8$  Hz, 1.7 Hz, 1.2 Hz, 1H), 7.92 (t,  $J = 1.5$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 15.4, 25.5, 28.1, 38.4, 47.1, 51.5, 57.1, 82.3, 125.4, 126.9, 127.7, 128.2, 128.4, 128.7, 128.9, 129.6, 134.8, 136.1, 136.7, 166.0, 170.9, 171.3.

**(2S,3R)-tert-Butyl-2-(3-(benzyl(ethyl)carbamoyl)benzamido)-3-methylpentanoate (20).**



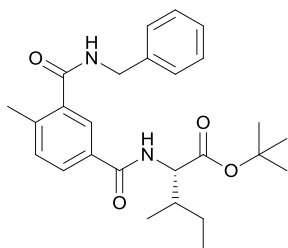
Yield: 97 mg (43%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.92 – 1.00 (m, 6H), 1.02 – 1.15 (m, 1H), 1.15 – 1.27 (m, 3H), 1.44 – 1.56 (m, 1H), 1.47 (s, 9H), 1.91 – 2.01 (m, 1H), 3.18 (s br, 1H), 3.51 (s br, 1H), 4.46 (s, 1H), 4.66 (s br, 1H), 4.76 (s, 1H), 6.70 (d,  $J = 33.1$  Hz, 1H), 7.10 – 7.38 (m, 5H), 7.50 – 7.38 (m, 1H), 7.54 (s, 1H), 7.79 – 7.89 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 12.1, 13.6, 15.3, 25.5, 28.0, 38.4, 40.0, 42.9, 46.9, 52.1, 57.1, 82.2, 125.1, 126.6, 127.4, 127.9, 128.1, 128.4, 128.6, 128.8, 129.4, 133.1, 134.7, 137.1, 162.5, 166.1, 170.9.

**(2S,3R)-tert-Butyl-2-(3-(heptan-4-ylcarbamoyl)benzamido)-3-methylpentanoate (21).**



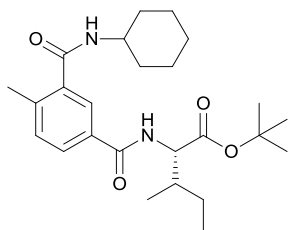
Yield: 139 mg (70%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.86 (t,  $J=7.2$  Hz, 6H), 0.89 – 0.95 (m, 6H), 1.19 – 1.27 (m, 1H), 1.17 – 1.55 (m, 9H), 1.44 (s, 9H), 1.93 (ddt,  $J = 9.2$  Hz, 6.8 Hz, 4.8 Hz, 1H), 4.04 – 4.16 (m, 1H), 4.64 (dd,  $J = 8.2$  Hz, 4.6 Hz, 1H), 6.22 (s, 1H), 6.86 (d,  $J = 8.2$  Hz, 1H), 7.41 (t,  $J = 7.7$  Hz, 1H), 7.83 (d,  $J = 7.7$  Hz, 1H), 7.91 (d,  $J = 7.7$  Hz, 1H), 8.17 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.6, 13.9, 15.3, 19.1, 25.4, 28.0, 37.4, 38.3, 49.5, 57.1, 82.2, 125.3, 128.7, 129.3, 130.2, 134.3, 135.4, 166.0, 166.2, 170.8.

**(2S,3R)-tert-Butyl-2-(3-(benzylcarbamoyl)-4-methylbenzamido)-3-methylpentanoate (22).**



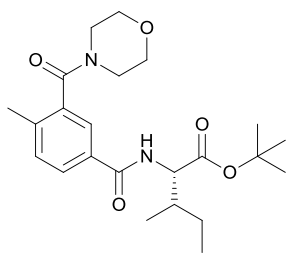
Yield: 185 mg (85%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.93 (d,  $J = 6.9$ , 3H), 0.95 (t,  $J = 7.4$ , 3H), 1.18 – 1.33 (m, 1H), 1.45 – 1.57 (m, 1H), 1.49 (s, 9H), 1.90 – 2.01 (m, 1H), 2.45 (s, 3H), 4.69 – 4.51 (m, 2H), 4.69 – 4.63 (m, 1H), 6.60 (t,  $J = 5.3$  Hz, 1H), 6.97 (d,  $J = 8.5$  Hz, 1H), 7.19 (d,  $J = 8.0$  Hz, 1H), 7.23 – 7.30 (m, 1H), 7.32 – 7.39 (m, 4H), 7.60 (dd,  $J = 7.9$  Hz, 1.9 Hz, 1H), 7.75 (d,  $J = 1.9$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 15.4, 19.8, 25.5, 28.1, 38.4, 43.9, 57.1, 82.4, 125.3, 127.5, 127.9, 128.0, 128.7, 131.0, 131.4, 136.5, 138.0, 140.3, 166.2, 169.0, 171.4.

**(2S,3R)-tert-Butyl-2-(3-(cyclohexylcarbamoyl)-4-methylbenzamido)-3-methylpentanoate (23).**



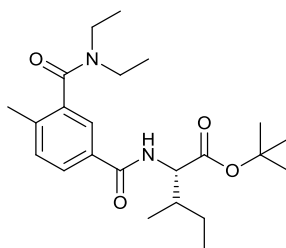
Yield: 162 mg (75%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.90 (t,  $J = 7.3$  Hz, 3H), 0.90 (d,  $J = 7.0$  Hz, 3H), 1.10 – 1.40 (m, 7H), 1.45 (s, 9H), 1.46 – 1.51 (m, 1H), 1.68 – 1.77 (m, 2H), 1.94 (ddd,  $J = 11.5$  Hz, 5.8 Hz, 3.5 Hz, 1H), 1.96 – 2.04 (m, 2H), 2.35 (s, 3H), 3.81 – 3.92 (m, 1H), 4.69 (dd,  $J = 8.7$  Hz, 4.7 Hz, 1H), 6.43 (d,  $J = 8.1$  Hz, 1H), 7.05 (d,  $J = 8.0$  Hz, 1H), 7.24 (d,  $J = 8.7$  Hz, 1H), 7.44 (dd,  $J = 8.0$  Hz, 1.9 Hz, 1H), 7.57 (d,  $J = 1.8$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.6, 15.4, 19.4, 24.8, 24.9, 25.4, 25.4, 27.9, 32.6, 32.9, 38.2, 48.7, 57.1, 82.3, 124.5, 127.8, 130.6, 130.7, 137.2, 139.4, 166.2, 168.4, 171.6.

**(2S,3R)-tert-Butyl-3-methyl-2-(4-methyl-3-(morpholine-4-carbonyl)benzamido)pentanoate (24).**



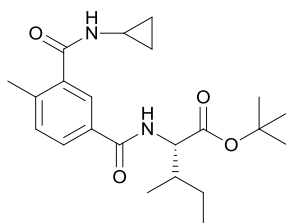
Yield: 111 mg (53%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.92 – 0.98 (m, 6H), 1.17 – 1.32 (m, 1H), 1.47 (s, 9H), 1.49 – 1.56 (m, 1H), 1.96 (ddd,  $J = 13.7$  Hz, 6.9 Hz, 4.5 Hz, 1H), 2.34 (s, 3H), 3.21 (s, 2H), 3.56 (s, 2H), 3.76 (s, 2H), 3.80 (s, 2H), 4.66 (dd,  $J = 7.6$  Hz, 4.3 Hz, 1H), 6.67 (d,  $J = 7.7$  Hz, 1H), 7.28 (s, 1H), 7.60 (s, 1H), 7.69 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 15.3, 19.0, 25.5, 28.0, 38.4, 41.9, 47.2, 57.0, 66.8, 66.9, 82.3, 124.5, 127.7, 130.7, 132.4, 136.0, 138.1, 166.0, 169.0, 171.1.

**(2S,3R)-tert-Butyl-2-(3-(diethylcarbamoyl)-4-methylbenzamido)-3-methylpentanoate (25).**



Yield: 43 mg (22%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.94 (d,  $J = 6.8$  Hz, 3H), 0.95 (t,  $J = 7.2$  Hz, 3H), 1.02 (t,  $J = 7.1$  Hz, 3H), 1.17 – 1.23 (m, 1H), 1.25 (t,  $J = 7.1$  Hz, 3H), 1.47 (s, 9H), 1.49 – 1.56 (m, 1H), 1.96 (ddt,  $J = 9.2$  Hz, 6.8 Hz, 4.6 Hz, 1H), 2.32 (s, 3H), 3.06 – 3.14 (m, 2H), 3.22 – 3.81 (m, 2H), 4.66 (dd,  $J = 8.3$  Hz, 4.5 Hz, 1H), 6.65 (d,  $J = 8.2$  Hz, 1H), 7.26 (dd,  $J = 8.0$  Hz, 0.4 Hz, 1H), 7.59 (d,  $J = 1.7$  Hz, 1H), 7.68 (dd,  $J = 8.0$  Hz, 1.1 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 12.8, 14.0, 15.3, 18.9, 25.5, 28.0, 38.4, 38.8, 42.7, 57.0, 82.2, 124.2, 127.1, 130.6, 132.1, 137.4, 138.0, 166.2, 169.8, 171.0.

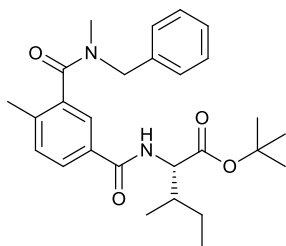
**(2S,3R)-tert-Butyl-2-(3-(cyclopropylcarbamoyl)-4-methylbenzamido)-3-methylpentanoate (26).**



Yield: 58 mg (30%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.61 – 0.73 (m, 2H), 0.83 – 0.88 (m, 2H), 0.94 (t,  $J = 7.1$  Hz, 3H), 0.94 (d,  $J = 6.8$  Hz, 3H), 1.21 – 1.33 (m, 1H), 1.49 (s, 9H), 1.52 – 1.56 (m, 1H), 1.95 – 2.01 (m, 1H), 2.41 (s, 3H), 2.90 (ddt,  $J = 7.5$  Hz, 3.8 Hz, 3.7 Hz, 1H), 4.70 (dd,  $J = 8.6$  Hz, 4.6 Hz, 1H), 6.52 – 6.59 (m, 1H), 7.12 (d,  $J = 8.0$  Hz, 1H), 7.14 – 7.18 (m, 1H), 7.51 (d,  $J = 7.9$  Hz, 1H), 7.61 (s br, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 6.3, 6.7, 11.7, 15.5, 19.6, 23.0, 25.5, 28.1, 38.4, 57.2, 82.5, 124.8, 128.1, 130.9, 131.0, 136.6, 140.1, 166.3, 170.6, 171.7.

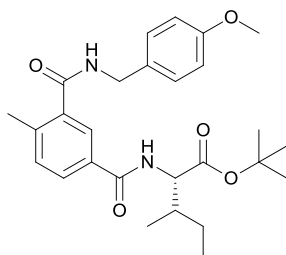


**(2S,3R)-tert-Butyl-2-(3-(benzyl(methyl)carbamoyl)-4-methylbenzamido)-3-methylpentanoate (27).**



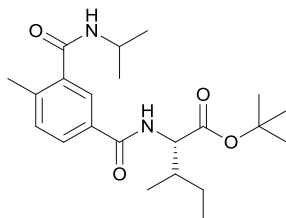
Yield: 102 mg (45%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.91 – 1.00 (m, 6H), 1.18 – 1.34 (m, 1H), 1.49 (s, 9H), 1.51 – 1.58 (m, 1H), 1.92 – 2.03 (m, 1H), 2.36 (s, 3H), 2.70 (s, 2H), 3.08 (s, 1H), 4.35 (s, 1H), 4.68 (dd,  $J = 8.3$  Hz, 4.5 Hz, 1H), 4.78 (s, 1H), 6.67 (d,  $J = 8.3$  Hz, 1H), 7.09 (d,  $J = 7.6$  Hz, 1H), 7.25 – 7.36 (m, 3H), 7.36 – 7.39 (m, 2H), 7.66 (d,  $J = 1.7$  Hz, 1H), 7.71 (t,  $J = 7.2$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 15.4, 19.0, 25.5, 28.1, 32.6, 35.7, 38.5, 50.2, 54.6, 57.0, 82.3, 124.6, 127.0, 127.6, 128.4, 128.7, 128.8, 130.9, 132.3, 136.0, 136.7, 138.0, 166.2, 170.5, 171.1.

**(2S,3R)-tert-Butyl-2-(3-((4-methoxybenzyl)carbamoyl)-4-methylbenzamido)-3-methylpentanoate (28).**



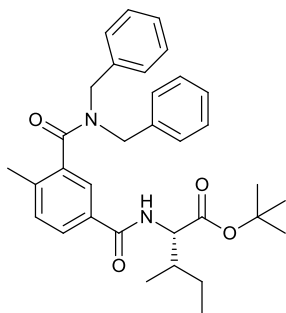
Yield: 195 mg (83%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.94 (d,  $J = 7.0$  Hz, 3H), 0.96 (t,  $J = 7.4$  Hz, 3H), 1.20 – 1.32 (m, 1H), 1.49 (s, 9H), 1.51 – 1.55 (m, 1H), 1.97 (ddt,  $J = 9.3$  Hz, 6.9 Hz, 4.7 Hz, 1H), 2.47 (s, 3H), 3.80 (s, 3H), 4.46 – 4.61 (m, 2H), 4.67 (dd,  $J = 8.3$  Hz, 4.5 Hz, 1H), 6.25 – 6.37 (m, 1H), 6.79 – 6.86 (m, 1H), 6.86 – 6.90 (m, 2H), 7.20 – 7.25 (m, 1H), 7.27 – 7.23 (m, 2H), 7.59 – 7.66 (m, 1H), 7.79 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 15.4, 19.9, 25.5, 28.1, 38.4, 43.5, 55.3, 57.1, 82.4, 114.2, 125.5, 127.9, 129.3, 130.0, 131.1, 131.6, 136.6, 140.3, 159.1, 166.1, 168.9, 171.3.

**(2S,3R)-tert-Butyl-2-(3-(isopropylcarbamoyl)-4-methylbenzamido)-3-methylpentanoate (29).**



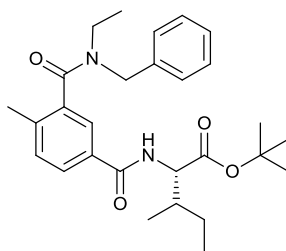
Yield: 145 mg (74%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.88 – 0.95 (m, 6H), 0.98 – 1.04 (m, 1H), 1.24 (t,  $J = 6.9$ , 6H), 1.21 – 1.31 (m, 1H), 1.46 (s, 9H), 1.47 – 1.54 (m, 1H), 1.95 (ddd,  $J = 9.2$ , 6.8, 4.7, 1H), 2.37 (s, 3H), 4.14 – 4.26 (m, 1H), 4.70 (dd,  $J = 8.6$ , 4.7, 1H), 6.32 (s, 1H), 7.08 (d,  $J = 7.8$ , 1H), 7.16 (s, 1H), 7.47 (d,  $J = 6.0$ , 1H), 7.60 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.6, 15.4, 19.4, 22.4, 22.6, 25.5, 28.0, 38.2, 41.8, 57.1, 82.3, 124.7, 127.7, 130.7, 130.9, 137.2, 139.5, 166.2, 168.5, 171.6.

**(2S,3R)-tert-Butyl-2-(3-(dibenzylcarbamoyl)-4-methylbenzamido)-3-methylpentanoate (30).**



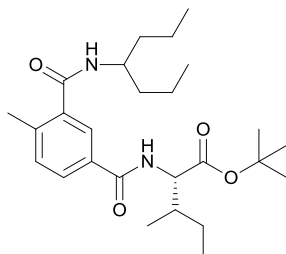
Yield: 115 mg (22%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.93 (d,  $J = 6.9$  Hz, 3H), 0.97 (t,  $J = 7.4$  Hz, 3H), 1.16 – 1.29 (m, 1H), 1.49 (s, 9H), 1.50 – 1.56 (m, 1H), 1.91 – 1.99 (m, 1H), 2.36 (s, 3H), 4.22 (s, 2H), 4.29 – 4.57 (m, 1H), 4.64 (dd,  $J = 8.3$  Hz, 4.5 Hz, 1H), 4.87 – 5.34 (m, 1H), 6.59 (d,  $J = 8.2$  Hz, 1H), 7.05 – 7.10 (m, 2H), 7.26 – 7.39 (m, 9H), 7.67 – 7.71 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 15.3, 19.2, 25.5, 28.1, 38.4, 46.7, 50.9, 57.0, 82.2, 124.6, 127.2, 127.5, 127.7, 127.8, 128.7, 128.8, 128.8, 130.9, 132.1, 135.8, 136.4, 136.7, 138.5, 166.0, 170.96, 170.99.

**(2S,3R)-tert-Butyl-2-(3-(benzyl(ethyl)carbamoyl)-4-methylbenzamido)-3-methylpentanoate (31).**



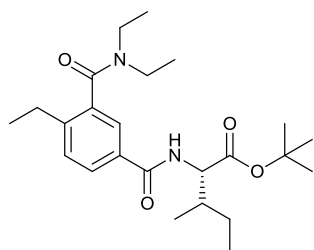
Yield: 101 mg (45%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.90 – 1.00 (m, 7H), 1.12 – 1.33 (m, 3H), 1.48 (s, 9H), 1.50 – 1.58 (m, 1H), 1.92 – 2.02 (m, 1H), 2.36 (s, 3H), 3.01 – 3.10 (m,  $J = 7.0$  Hz, 2H), 4.31 – 4.55 (m, 2H), 4.68 (dd,  $J = 8.2$  Hz, 4.4 Hz, 1H), 6.67 (d,  $J = 8.2$  Hz, 1H), 7.10 (d,  $J = 7.9$  Hz, 1H), 7.21 – 7.41 (m, 5H), 7.60 – 7.70 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 12.2, 13.4, 15.3, 19.1, 25.5, 28.0, 38.4, 42.1, 51.5, 57.0, 82.2, 124.3, 127.0, 127.4, 127.7, 128.1, 128.6, 128.7, 130.6, 132.2, 136.3, 137.2, 138.1, 162.5, 166.0, 170.4.

**(2S,3R)-tert-Butyl-2-(3-(heptan-4-ylcarbamoyl)-4-methylbenzamido)-3-methylpentanoate (32).**



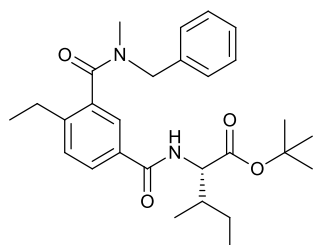
Yield: 165 mg (75%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.88 – 0.96 (m, 12H), 1.19 – 1.27 (m, 1H), 1.33 – 1.54 (m, 9H), 1.47 (s, 9H), 1.89 – 1.98 (m, 1H), 2.41 (s, 3H), 4.07 (d,  $J = 7.2$  Hz, 1H), 4.62 (ddd,  $J = 8.2$  Hz, 4.6 Hz, 1.6 Hz, 1H), 5.90 (s br, 1H), 6.83 (d,  $J = 7.8$  Hz, 1H), 7.14 – 7.19 (m, 1H), 7.56 (dd,  $J = 7.9$  Hz, 1.7 Hz, 1H), 7.71 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.6, 14.0, 15.3, 19.15, 19.20, 19.7, 25.5, 28.0, 37.3, 37.4, 38.3, 49.2, 57.1, 82.2, 125.4, 127.3, 130.8, 131.5, 137.4, 139.8, 166.2, 168.8, 171.1.

**(2S,3R)-tert-Butyl-2-(3-(diethylcarbamoyl)-4-ethylbenzamido)-3-methylpentanoate (33).**



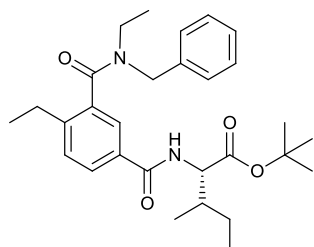
Yield: 100 mg (48%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.99 – 0.93 (m, 6H), 1.04 (t,  $J = 7.1$  Hz, 3H), 1.29 – 1.20 (m, 7H), 1.48 (s, 9H), 1.59 – 1.49 (m, 1H), 2.02 – 1.92 (m, 1H), 2.65 (q,  $J = 7.6$  Hz, 2H), 3.11 (q,  $J = 6.7$  Hz, 2H), 3.83 – 3.29 (m, 2H), 4.67 (dd,  $J = 8.3$  Hz, 4.5 Hz, 1H), 6.64 (d,  $J = 8.2$  Hz, 1H), 7.33 (d,  $J = 8.1$  Hz, 1H), 7.58 (s, 1H), 7.73 (d,  $J = 6.7$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 12.8, 14.0, 14.8, 15.4, 25.5, 25.9, 28.1, 38.5, 38.8, 42.9, 57.0, 82.2, 124.0, 127.0, 129.0, 132.1, 136.9, 144.1, 166.2, 169.8, 171.0.

**(2S,3R)-tert-Butyl-2-(3-(benzyl(methyl)carbamoyl)-4-ethylbenzamido)-3-methylpentanoate (34).**



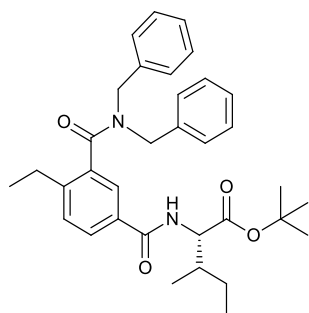
Yield: 155 mg (66%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.89 – 0.99 (m, 6H), 1.16 – 1.28 (m, 4H), 1.47 (s, 9H), 1.49 – 1.56 (m, 1H), 1.89 – 2.02 (m, 1H), 2.65 (q,  $J = 7.5$  Hz, 3H), 2.68 (s, 2H), 3.06 (s, 1H), 4.20 – 4.53 (m, 1H), 4.67 (dd,  $J = 8.2$  Hz, 4.5 Hz, 1H), 4.71 – 4.88 (m, 1H), 6.69 (d,  $J = 7.8$  Hz, 1H), 7.09 (d,  $J = 7.9$  Hz, 1H), 7.32 (m, 5H), 7.63 (d,  $J = 1.8$  Hz, 1H), 7.70 – 7.78 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 14.8, 15.3, 25.5, 25.9, 28.0, 32.6, 35.9, 38.4, 50.2, 54.7, 57.0, 82.2, 124.4, 126.9, 127.6, 127.7, 128.3, 128.6, 128.8, 129.0, 132.2, 136.0, 136.7, 144.2, 166.0, 170.4, 171.0.

**(2S,3R)-tert-Butyl-2-(3-(benzyl(ethyl)carbamoyl)-4-ethylbenzamido)-3-methylpentanoate (35).**



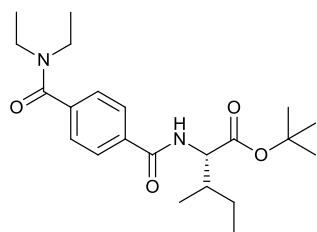
Yield: 120 mg (50%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.93 – 1.04 (m, 9H), 1.19 – 1.33 (m, 4H), 1.49 (s, 9H), 1.54 (ddd,  $J = 13.0$  Hz, 7.5 Hz, 5.1 Hz, 1H), 1.93 – 2.02 (m, 1H), 2.68 (q,  $J = 7.5$  Hz, 2H), 3.05 (q,  $J = 7.1$  Hz, 2H), 4.32 (d,  $J = 7.9$  Hz, 2H), 4.68 (dd,  $J = 8.3$  Hz, 4.5 Hz, 1H), 6.67 (d,  $J = 8.3$  Hz, 1H), 7.11 (d,  $J = 7.3$  Hz, 1H), 7.21 – 7.41 (m, 7H), 7.59 – 7.67 (m, 1H), 7.75 (d,  $J = 7.8$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 13.3, 15.0, 15.4, 25.5, 26.0, 28.1, 38.4, 42.2, 51.7, 57.0, 82.2, 124.2, 127.0, 127.5, 127.7, 128.3, 128.6, 128.8, 129.1, 132.1, 136.3, 137.3, 144.2, 166.0, 170.4, 171.0.

**(2S,3R)-tert-Butyl-2-(3-(dibenzylcarbamoyl)-4-ethylbenzamido)-3-methylpentanoate (36).**



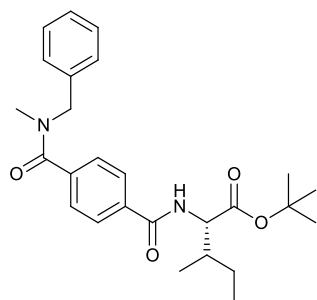
Yield: 339 mg (62%, white semi-solid);  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$ : 0.89 – 0.94 (m, 3H), 0.96 (t,  $J = 7.4$  Hz, 3H), 1.15 – 1.27 (m, 1H), 1.23 (t,  $J = 7.6$  Hz, 3H), 1.47 – 1.55 (m, 1H), 1.49 (s, 9H), 1.94 (s, 1H), 2.61 – 2.75 (m, 2H), 4.10 – 4.27 (m, 2H), 4.27 – 4.45 (m, 1H), 4.64 (dd,  $J = 8.3$  Hz, 4.5 Hz, 1H), 5.04 – 5.27 (m, 1H), 6.57 (d,  $J = 8.0$  Hz, 1H), 7.07 – 7.11 (m, 2H), 7.24 – 7.39 (m, 9H), 7.68 (d,  $J = 1.9$  Hz, 1H), 7.75 (dd,  $J = 8.0$  Hz, 1.9 Hz, 1H);  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 15.0, 15.4, 25.5, 26.1, 28.1, 38.4, 46.6, 51.0, 57.0, 82.2, 124.5, 127.1, 127.7, 127.8, 128.7, 128.8, 128.9, 129.2, 132.1, 135.8, 136.0, 136.7, 144.5, 166.0, 170.9, 171.0.

**(2S,3R)-tert-Butyl-2-(4-(diethylcarbamoyl)benzamido)-3-methylpentanoate (65).**



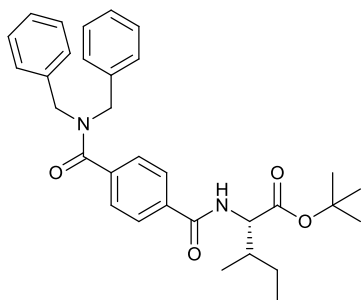
Yield: 133 mg (68%, white semi-solid);  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$ : 0.94 – 0.99 (m, 6H), 1.05 – 1.14 (m, 3H), 1.22 – 1.27 (m, 3H), 1.28 – 1.35 (m, 1H), 1.48 (s, 9H), 1.49 – 1.61 (m, 1H), 1.94 – 2.02 (m, 1H), 3.14 – 3.26 (m, 2H), 3.49 – 3.58 (m, 2H), 4.69 (dd,  $J = 8.2$  Hz, 4.4 Hz, 1H), 6.71 (d,  $J = 7.6$  Hz, 1H), 7.40 – 7.46 (m, 2H), 7.80 – 7.85 (m, 2H);  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 14.2, 15.4, 25.5, 28.1, 38.5, 39.4, 43.2, 57.1, 82.4, 126.5, 127.2, 134.9, 140.4, 166.3, 170.3, 171.1.

**(2S,3R)-tert-Butyl-2-(4-(benzyl(methyl)carbamoyl)benzamido)-3-methylpentanoate (66).**



Yield: 160 mg (73%, white semi-solid);  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$ : 0.94 (s br, 6H), 1.18 – 1.32 (m, 1H), 1.46 (s, 9H), 1.49 – 1.63 (m, 1H), 1.91 – 2.02 (m, 1H), 2.80 (s, 2H), 3.02 (s, 1H), 4.44 (s, 1H), 4.68 (s br, 1H), 4.73 (s, 1H), 6.78 (s br, 1H), 7.12 (d,  $J = 6.6$  Hz, 1H), 7.37 – 7.25 (m, 4H), 7.48 (t,  $J = 6.4$  Hz, 2H), 7.74 – 7.85 (m, 2H);  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ )  $\delta$ : 11.7, 15.3, 25.5, 28.0, 33.2, 36.8, 38.4, 50.7, 54.9, 57.1, 82.3, 126.5, 126.9, 127.2, 127.7, 128.2, 128.7, 128.8, 135.3, 136.1, 139.1, 166.1, 170.5, 171.0.

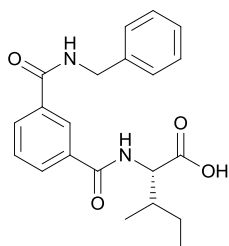
**(2S,3R)-tert-Butyl-2-(4-(dibenzylcarbamoyl)benzamido)-3-methylpentanoate (67).**



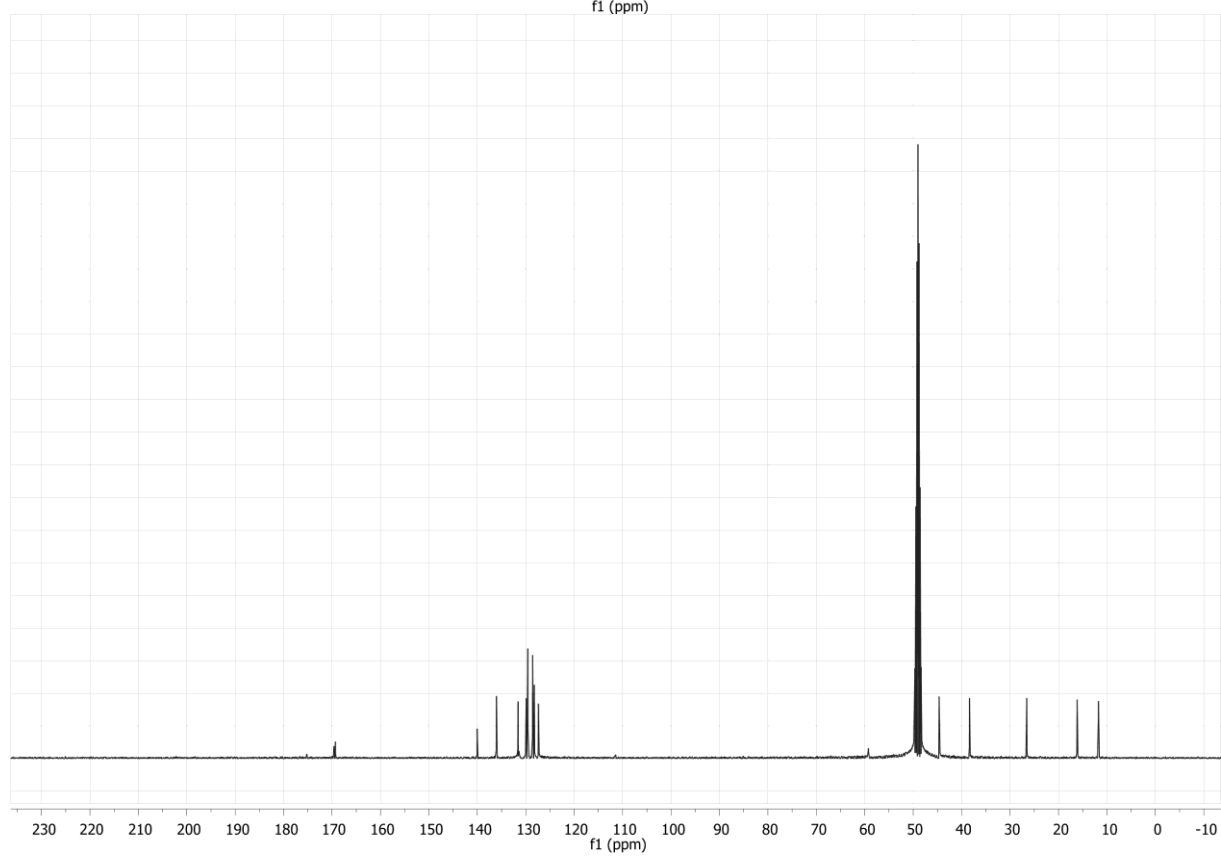
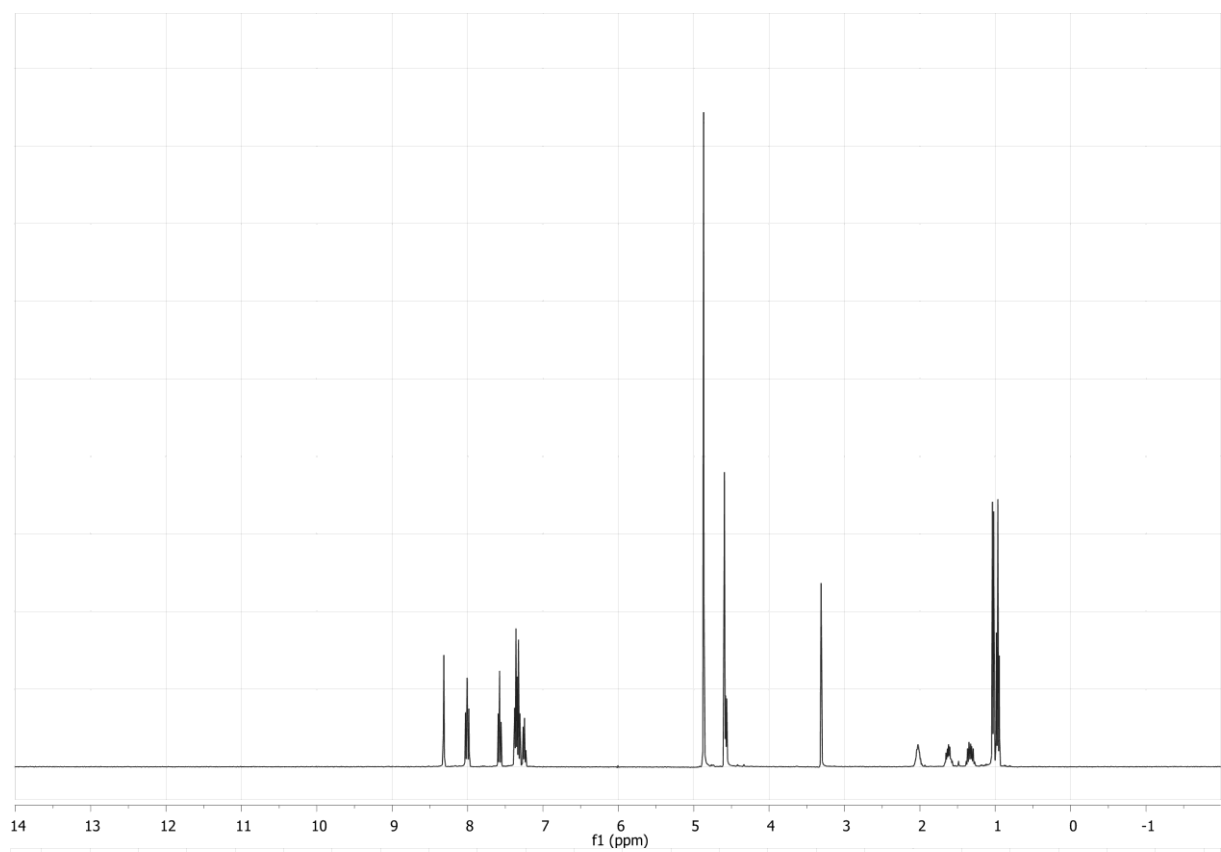
Yield: 40 mg (16%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.96 (d,  $J = 7.0$  Hz, 3H), 0.97 (t,  $J = 7.6$  Hz, 3H), 1.21 – 1.30 (m, 1H), 1.48 (s, 9H), 1.50 – 1.58 (m, 1H), 1.98 (ddt,  $J = 9.2$  Hz, 4.7 Hz, 2.4 Hz, 1H), 4.72 (s, 2H), 4.36 (s, 2H), 4.68 (dd,  $J = 8.2$  Hz, 4.4 Hz, 1H), 6.71 (d,  $J = 8.2$  Hz, 1H), 7.12 (d,  $J = 6.6$  Hz, 2H), 7.27 – 7.39 (m, 8H), 7.53 – 7.57 (m, 2H), 7.79 – 7.83 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 15.4, 25.5, 28.1, 38.5, 47.0, 51.4, 57.1, 82.4, 126.8, 126.9, 127.3, 127.7, 127.8, 128.5, 128.7, 128.9, 135.4, 136.0, 136.7, 139.2, 166.0, 171.1, 171.3.

**2. Experimental data for all synthesized final products (compounds 37–64 and 68–70), including  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra and HPLC chromatograms for all final products with determined  $K_i$  value for the human  $\text{AT}_2\text{R}$  (compounds 42, 44, 47–48, 53, 55, 58–59, 61–64 and 68–70).**

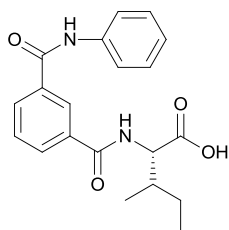
**(2S,3R)-2-(3-(Benzylcarbamoyl)benzamido)-3-methylpentanoic acid (37).**



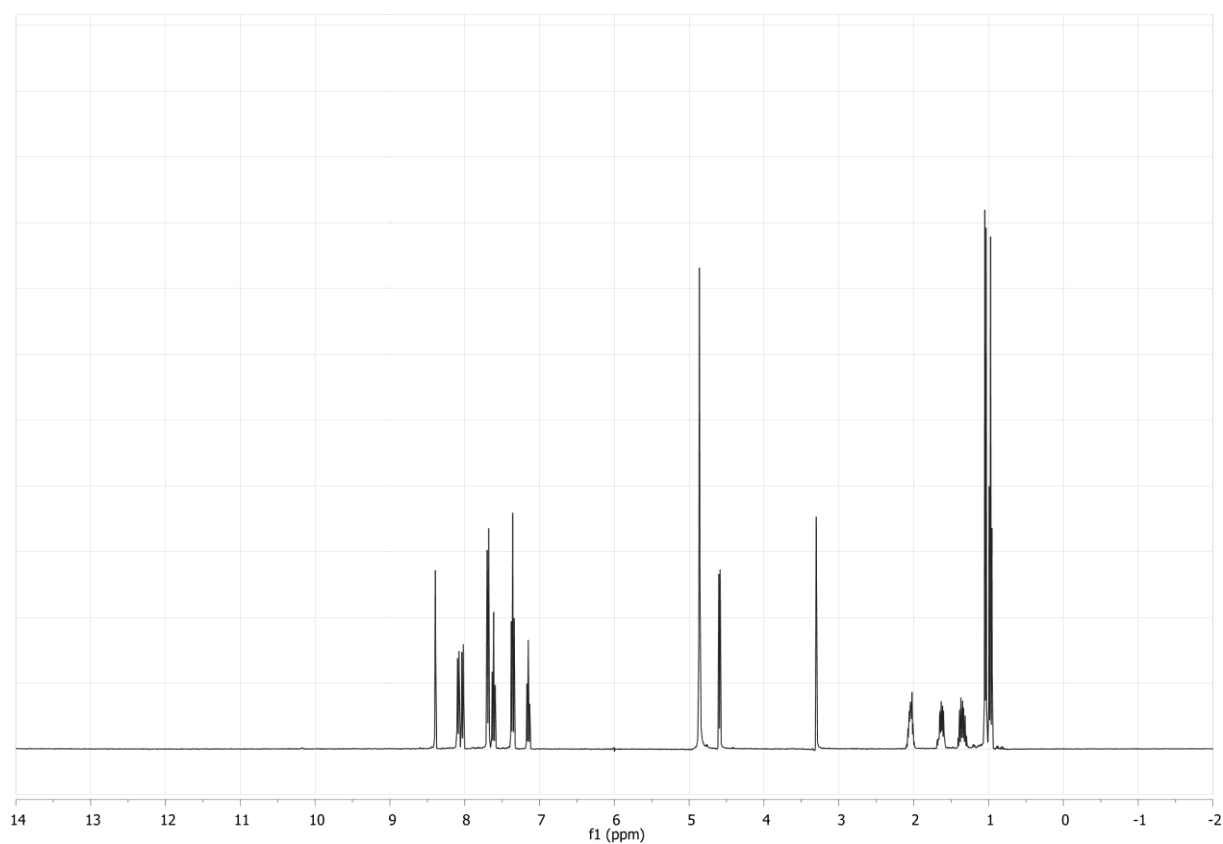
Yield: 10 mg (21%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.97 (t,  $J = 7.4$  Hz, 3H), 1.03 (d,  $J = 6.9$  Hz, 3H), 1.26 – 1.41 (m, 1H), 1.56 – 1.68 (m, 1H), 1.96 – 2.08 (m, 1H), 4.57 (d,  $J = 6.2$  Hz, 1H), 4.59 (s, 2H), 7.24 (t,  $J = 7.1$  Hz, 1H), 7.29 – 7.39 (m, 4H), 7.57 (t,  $J = 7.8$  Hz, 1H), 8.00 (td,  $J = 8.5$  Hz, 1.6 Hz, 2H), 8.32 (t,  $J = 1.6$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.9, 16.3, 26.7, 38.5, 44.8, 59.4, 127.5, 128.4, 128.7, 129.7, 130.1, 131.6, 131.7, 136.2, 140.2, 169.5, 169.8, 175.4; HRMS ( $\text{M}+\text{H}$ ) calcd. for  $\text{C}_{21}\text{H}_{24}\text{N}_2\text{O}_4$  369.1814; found: 369.1811.

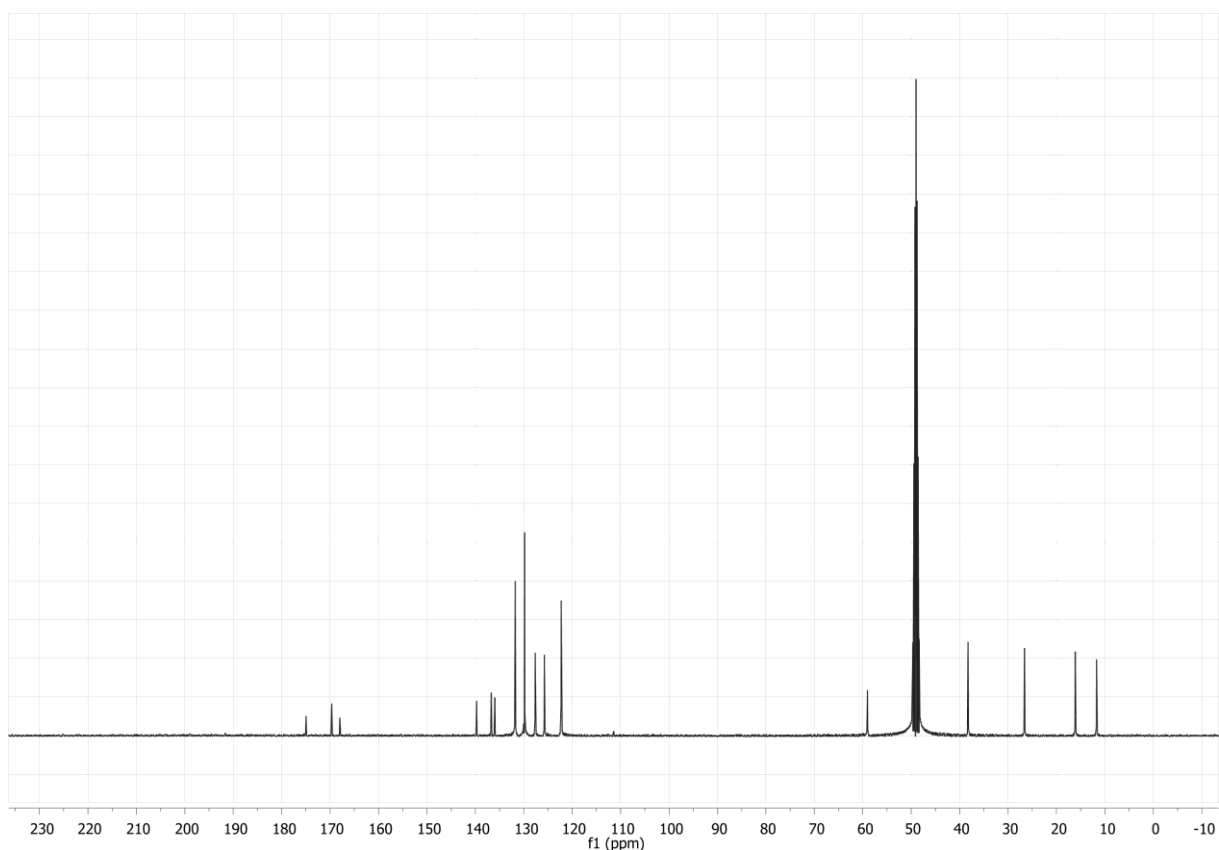


**(2S,3R)-3-Methyl-2-(3-(phenylcarbamoyl)benzamido)pentanoic acid (38).**

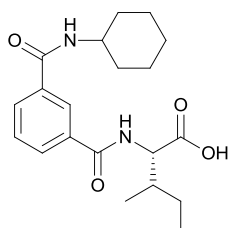


Yield: 30 mg (56%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.98 (t,  $J = 7.4$  Hz, 3H), 1.05 (d,  $J = 6.9$  Hz, 3H), 1.29 – 1.43 (m, 1H), 1.58 – 1.70 (m, 1H), 2.00 – 2.11 (m, 1H), 4.60 (d,  $J = 6.3$  Hz, 1H), 7.16 (t,  $J = 7.4$  Hz, 1H), 7.37 (t,  $J = 7.8$  Hz, 2H), 7.62 (t,  $J = 7.8$  Hz, 1H), 7.70 (d,  $J = 8.3$  Hz, 2H), 8.04 (d,  $J = 7.6$  Hz, 1H), 8.09 (d,  $J = 7.8$  Hz, 1H), 8.40 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.9, 16.3, 26.8, 38.4, 59.2, 122.4, 125.9, 127.8, 130.0, 130.1, 131.9, 136.1, 136.9, 139.9, 168.1, 169.8, 175.1; HRMS (M+H) calcd. for  $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_4$  355.1658; found: 355.1660.



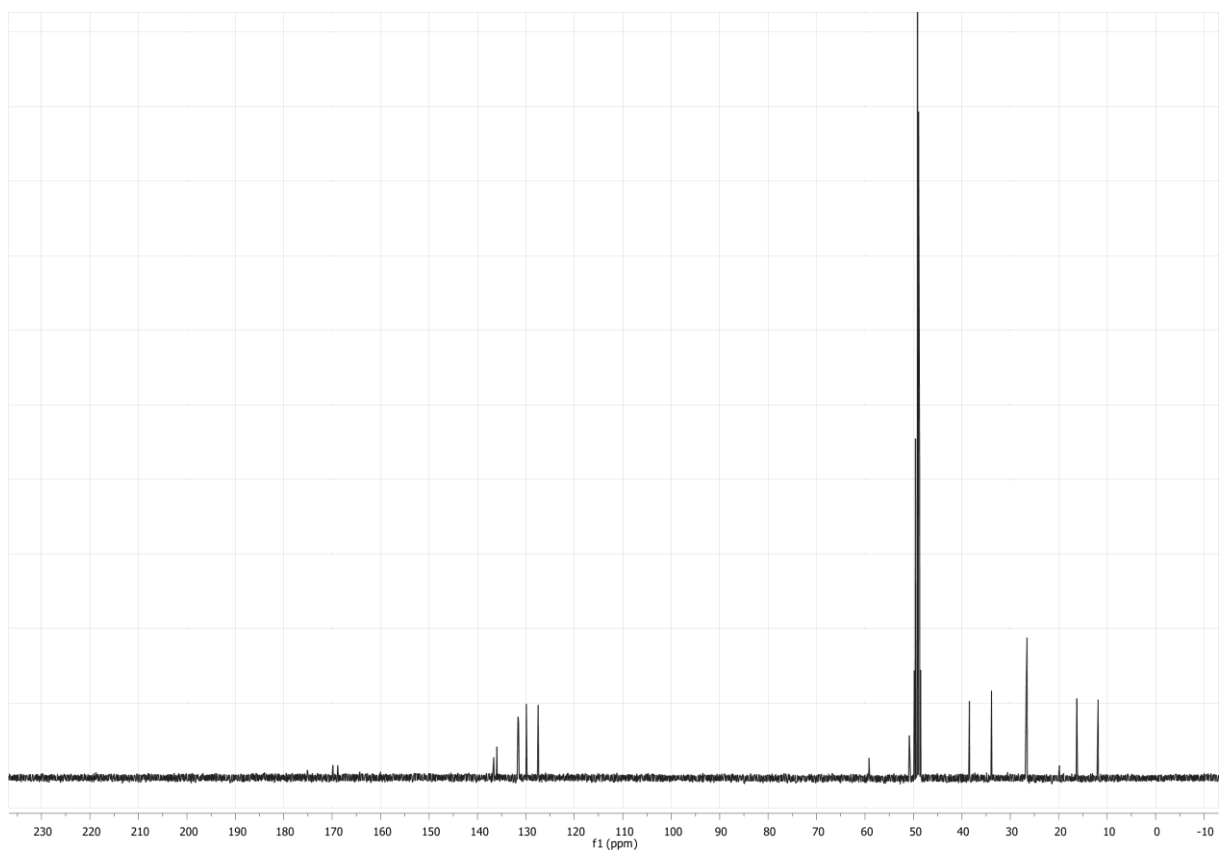
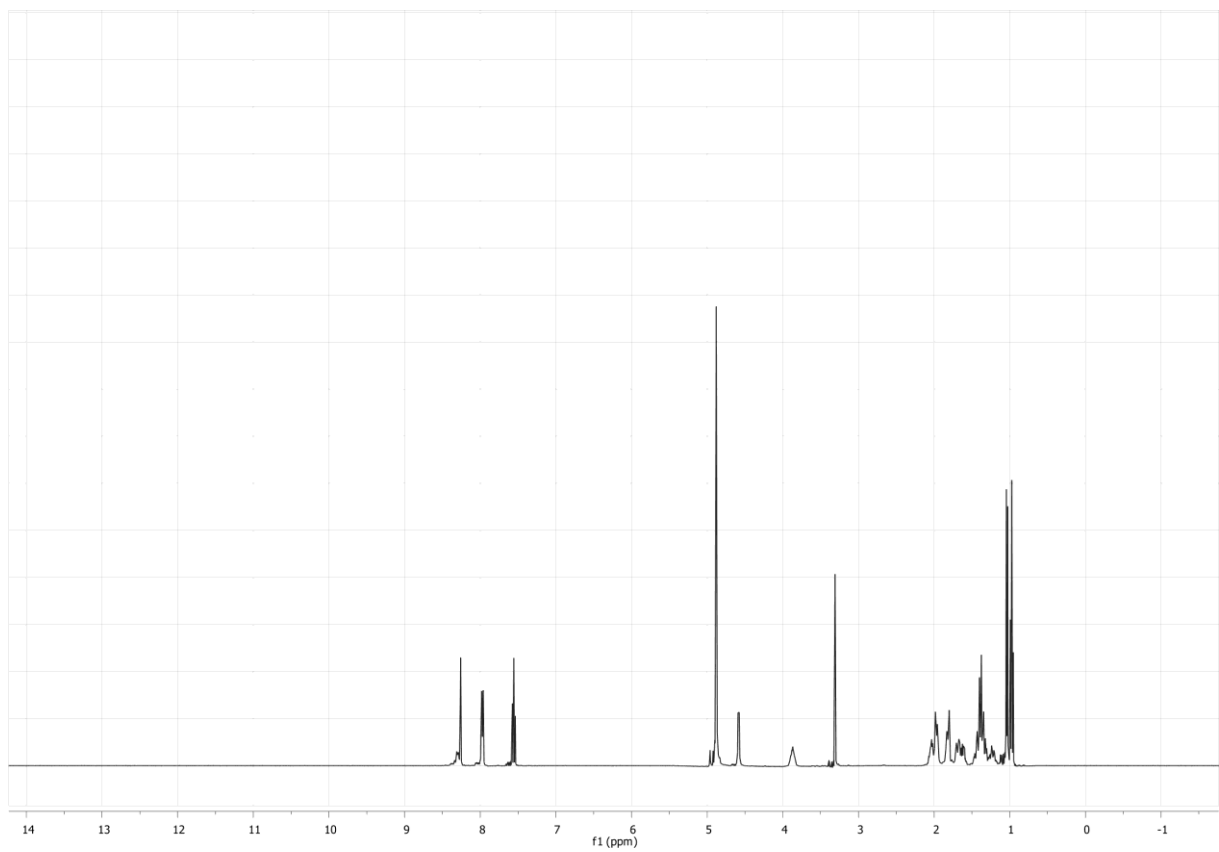


**(2S,3R)-2-(3-(Cyclohexylcarbamoyl)benzamido)-3-methylpentanoic acid (39).**

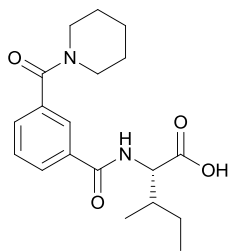


Yield: 37 mg (75%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.97 (t,  $J$  = 7.4 Hz, 3H), 1.04 (d,  $J$  = 6.9 Hz, 3H), 1.15 – 1.30 (m, 1H), 1.29 – 1.49 (m, 5H), 1.57 – 1.65 (m, 1H), 2.04 (ddt,  $J$  = 9.0 Hz, 6.7 Hz, 4.5 Hz, 1H), 1.65 – 1.72 (m, 1H), 1.78 – 1.87 (m, 2H), 1.92 – 1.99 (m, 2H), 3.82 – 3.92 (m, 1H), 4.58 (d,  $J$  = 6.2 Hz, 1H), 7.56 (t,  $J$  = 7.8 Hz, 1H), 7.94 – 8.00 (m, 2H), 8.26 (t,  $J$  = 1.7 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.9, 16.3, 26.6, 26.7, 26.8, 33.9, 33.9, 38.4, 50.8, 51.0, 59.2, 127.5, 129.9, 131.5, 131.6, 136.0, 136.65, 136.69, 168.9, 169.8, 175.1; HRMS (M+H) calcd. for  $\text{C}_{20}\text{H}_{28}\text{N}_2\text{O}_4$  361.2127; found: 361.2122.

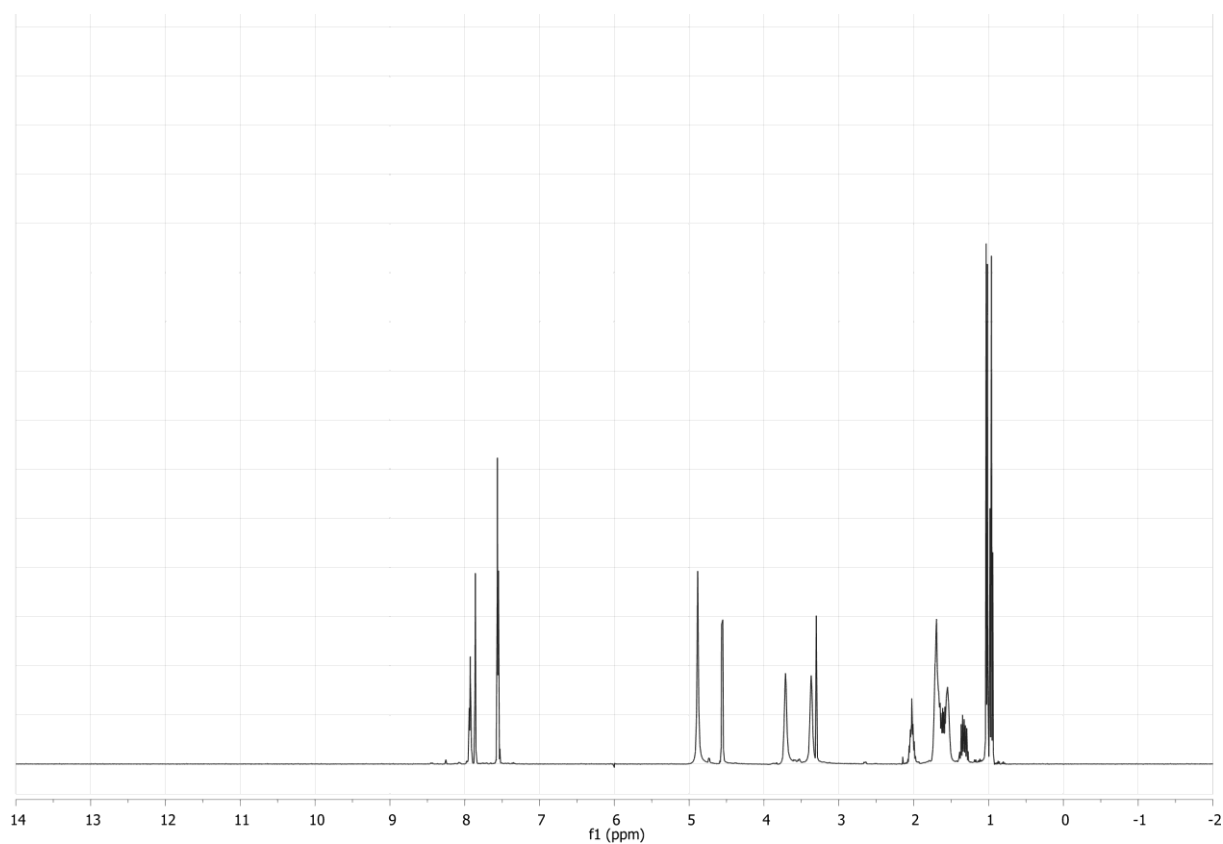


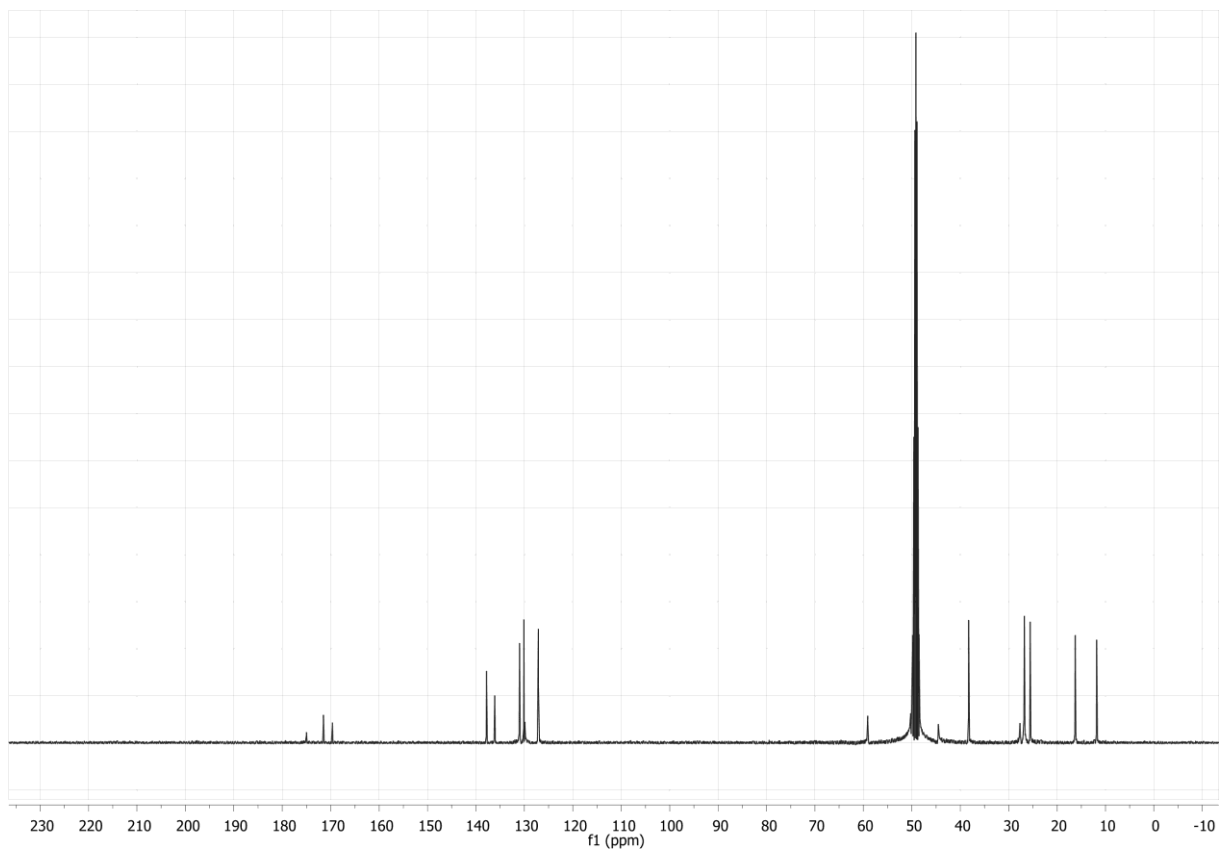


**(2*S*,3*R*)-3-Methyl-2-(3-(piperidine-1-carbonyl)benzamido)pentanoic acid (40).**

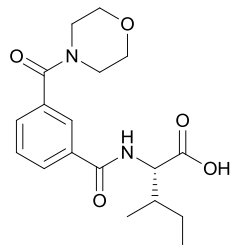


Yield: 137 mg (82%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J = 7.4$  Hz, 3H), 1.02 (d,  $J = 6.9$  Hz, 3H), 1.27 – 1.40 (m, 1H), 1.50 – 1.58 (m, 2H), 1.58 – 1.65 (m, 1H), 1.65 – 1.75 (m, 4H), 1.97 – 2.08 (m, 1H), 3.37 (s br, 2H), 3.71 (s br, 2H), 4.56 (d,  $J = 6.4$  Hz, 1H), 7.56 (d,  $J = 5.1$  Hz, 2H), 7.86 (s, 1H), 7.90 – 7.96 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 25.6, 26.8, 26.9, 27.7, 38.3, 44.5, 50.2, 59.1, 127.1, 130.0, 130.1, 131.0, 136.1, 137.8, 169.6, 171.5, 175.0; HRMS ( $\text{M}+\text{H}$ ) calcd. for  $\text{C}_{19}\text{H}_{26}\text{N}_2\text{O}_4$  347.1971; found: 347.1976.

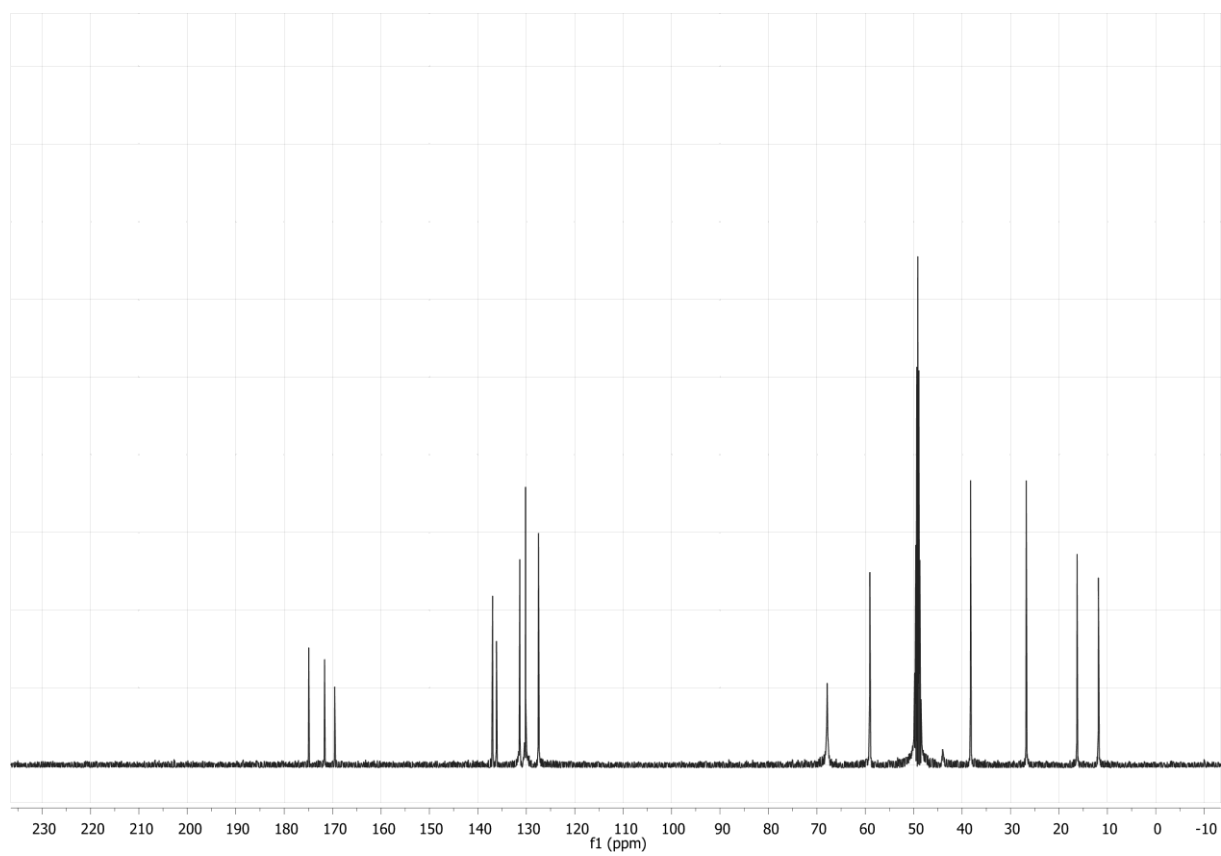
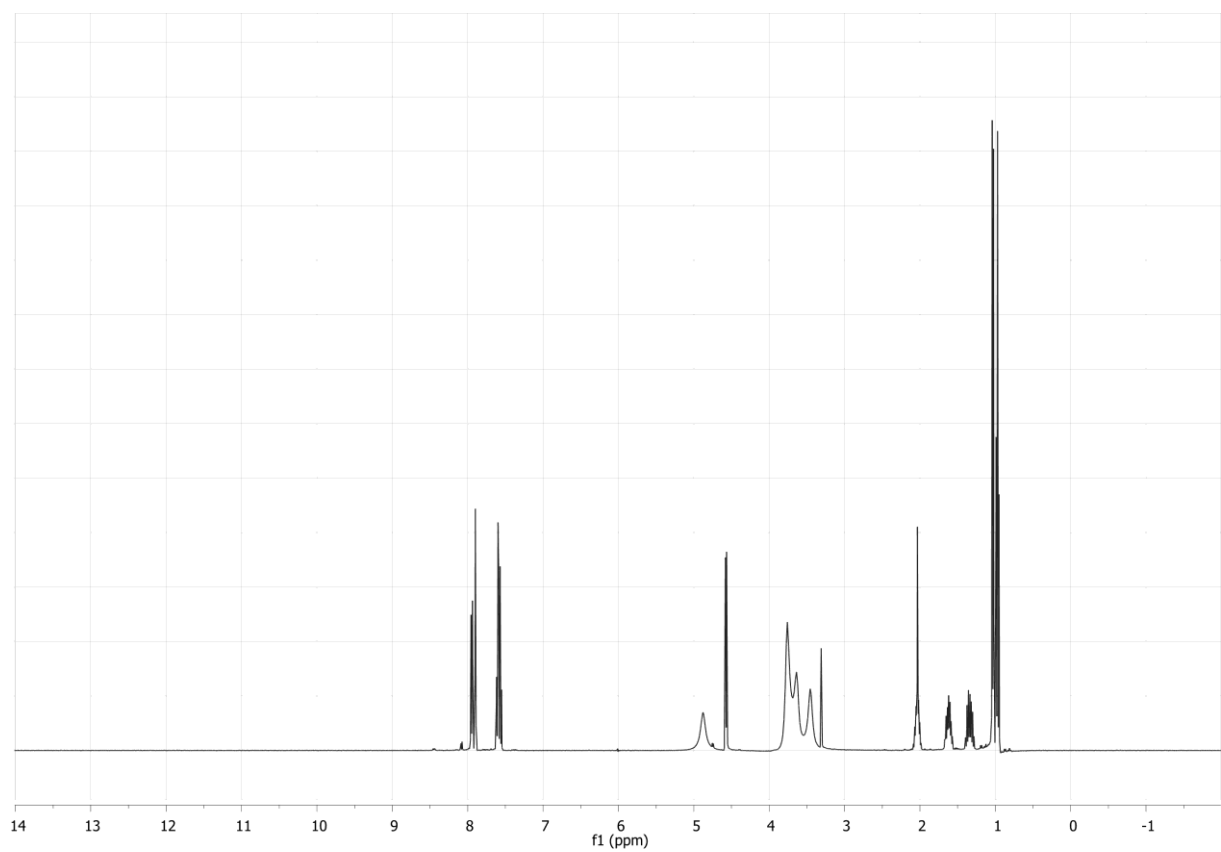




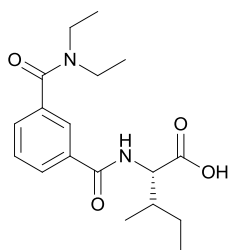
**(2*S*,3*R*)-3-Methyl-2-(3-(morpholine-4-carbonyl)benzamido)pentanoic acid (41).**



Yield: 32 mg (70%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.97 (t,  $J = 7.4$  Hz, 3H), 1.03 (d,  $J = 6.9$  Hz, 3H), 1.27 – 1.40 (m, 1H), 1.62 (ddq,  $J = 14.9$  Hz, 7.5 Hz, 4.4 Hz, 1H), 1.98 – 2.09 (m, 1H), 3.37 – 3.84 (m, 8H), 4.57 (d,  $J = 6.4$  Hz, 1H), 7.54 – 7.63 (m, 2H), 7.88 – 7.92 (m, 1H), 7.95 (dt,  $J = 7.3$  Hz, 1.6 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 26.7, 38.3, 44.0, 49.7, 59.1, 67.9, 127.5, 130.2, 130.3, 131.3, 136.2, 137.0, 169.6, 171.7, 174.9; HRMS (M+H) calcd. for  $\text{C}_{18}\text{H}_{24}\text{N}_2\text{O}_5$  349.1763; found: 349.1769.



**(2S,3R)-2-(3-(Diethylcarbamoyl)benzamido)-3-methylpentanoic acid (42).**



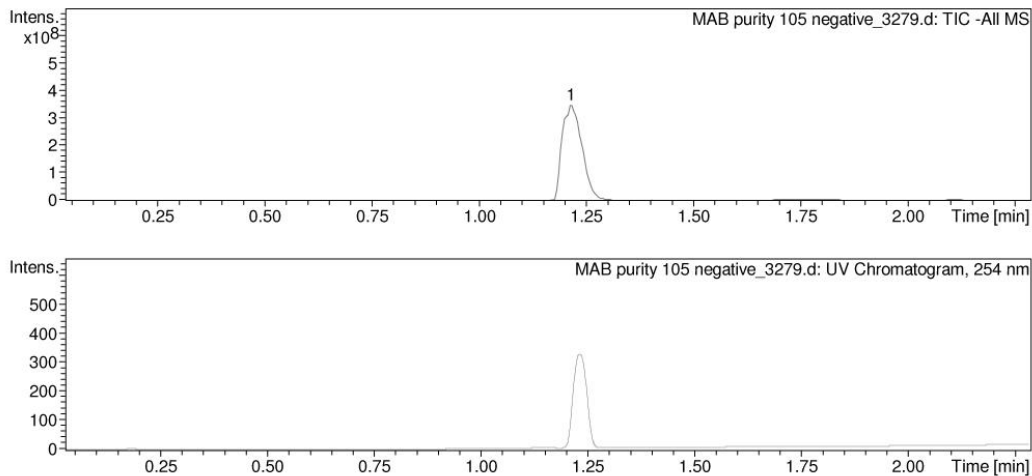
Yield 31 mg (73%, white semi-solid).  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.97 (t,  $J = 7.4$  Hz, 3H), 1.03 (d,  $J = 6.8$  Hz, 3H), 1.14 (t,  $J = 6.4$  Hz, 3H), 1.27 (t,  $J = 6.4$  Hz, 3H), 1.30 – 1.41 (m, 1H), 1.56 – 1.68 (m, 1H), 1.98 – 2.10 (m, 1H), 3.25 – 3.37 (m, 2H), 3.57 (d,  $J = 6.8$  Hz, 2H), 4.57 (d,  $J = 6.3$  Hz, 1H), 7.56 (dt,  $J = 5.8$  Hz, 3.2 Hz, 2H), 7.86 (s, 1H), 7.91 – 7.97 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.7, 13.1, 14.4, 16.1, 26.6, 38.1, 41.0, 45.0, 59.0, 126.5, 129.6, 130.0, 130.4, 135.9, 138.3, 169.4, 172.6, 174.9; HRMS (M+H): calcd. for  $\text{C}_{18}\text{H}_{26}\text{N}_2\text{O}_4$  335.1971, found 335.1972;  $[\alpha]_{589}^{25} = +10.3$ .

## LC-MS Analysis Report

### General Information

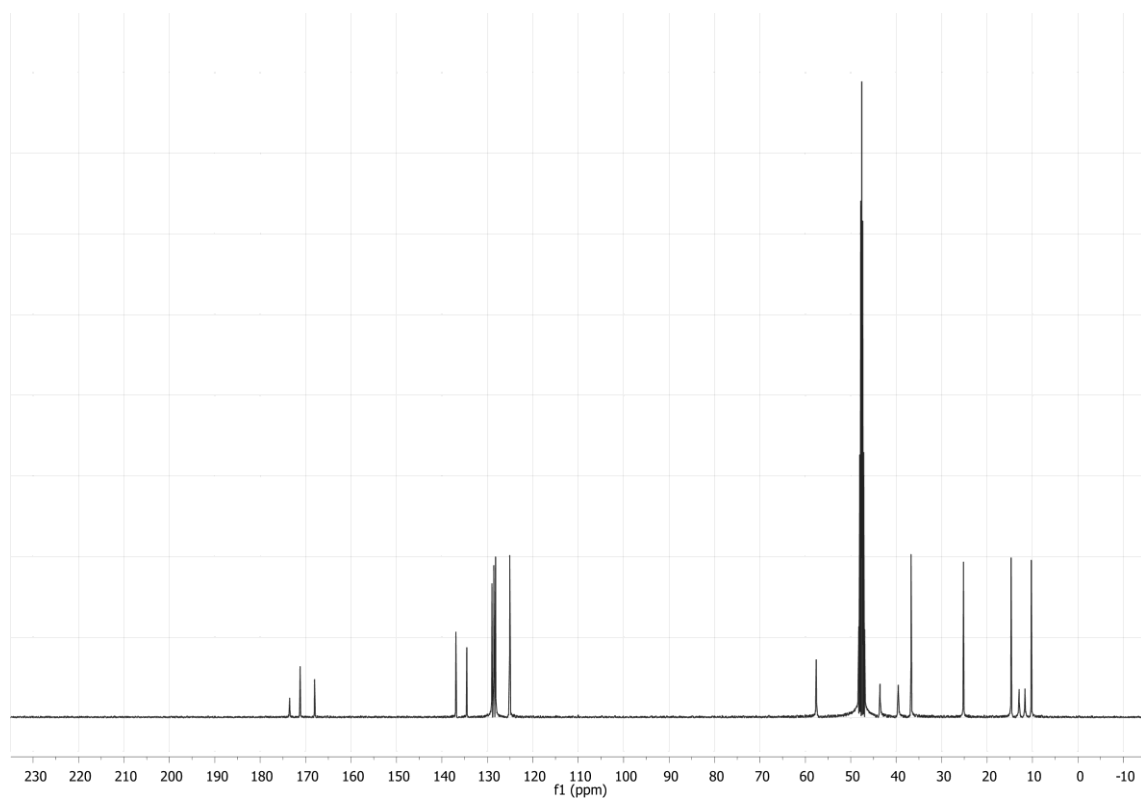
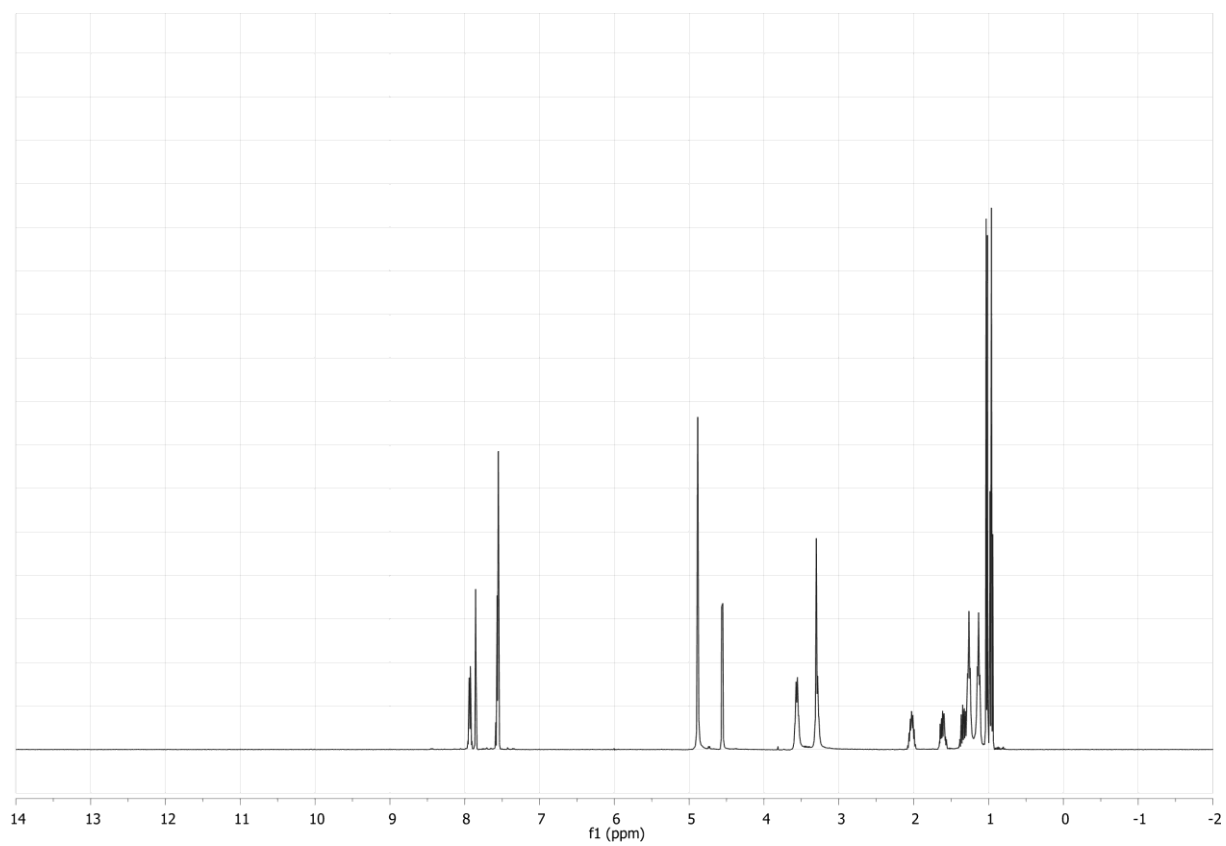
Sample ID: MAB purity 105 negative  
Date & Time: 10/7/2010 11:49:26 AM  
Data File: B:\Malte\Results\Purity\_check\MAB purity 105 negative\_3279.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram

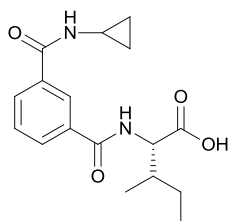


### Compound List (Area Frac. % of UV 254 nm)

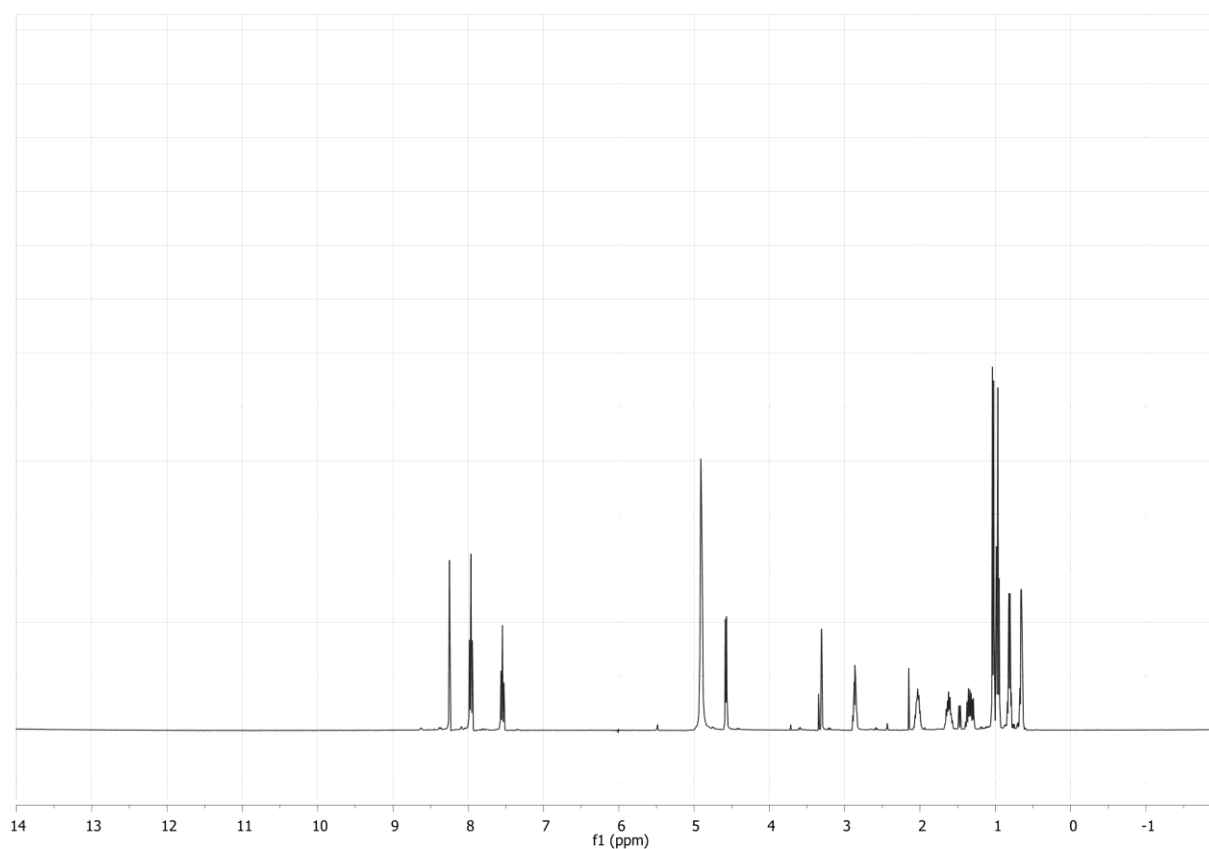
Cmpd. Label	Area Frac. %
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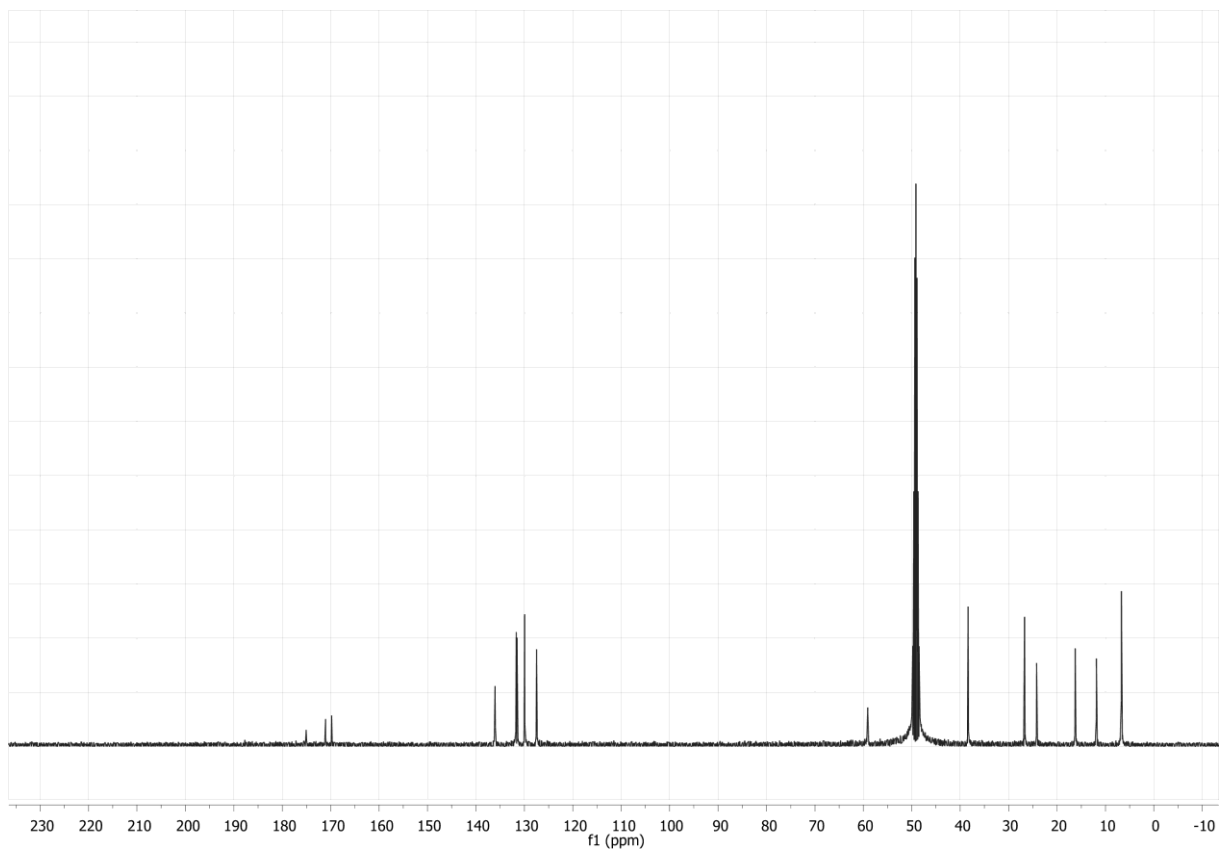


**(2*S*,3*R*)-2-(3-(Cyclopropylcarbamoyl)benzamido)-3-methylpentanoic acid (43).**

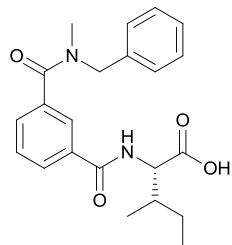


Yield: 32 mg (69%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.62 – 0.69 (m, 2H), 0.81 (dt,  $J = 6.3$  Hz, 6.2 Hz, 2H), 0.97 (t,  $J = 7.4$  Hz, 3H), 1.03 (d,  $J = 6.8$  Hz, 3H), 1.27 – 1.40 (m, 1H), 1.56 – 1.68 (m, 1H), 1.98 – 2.09 (m, 1H), 2.83 – 2.90 (m, 1H), 4.58 (d,  $J = 6.2$  Hz, 1H), 7.55 (t,  $J = 7.7$  Hz, 1H), 7.96 (t,  $J = 7.4$  Hz, 2H), 8.25 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 6.7, 11.9, 16.2, 24.2, 26.7, 38.4, 59.1, 127.5, 129.9, 131.5, 131.7, 136.0, 136.1, 169.8, 171.1, 175.1; HRMS (M+H) calcd. for  $\text{C}_{17}\text{H}_{22}\text{N}_2\text{O}_4$  319.1658; found: 319.1656.





**(2S,3R)-2-(3-(Benzyl(methyl)carbamoyl)benzamido)-3-methylpentanoic acid (44).**



Yield: 51 mg (65%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J = 7.3$  Hz, 3H), 1.03 (d,  $J = 6.9$  Hz, 3H), 1.25 – 1.40 (m, 1H), 1.54 – 1.68 (m, 1H), 1.97 – 2.08 (m, 1H), 2.89 (s, 2H), 3.02 (s, 1H), 4.52 (s br, 1H), 4.57 (d,  $J = 6.0$  Hz, 1H), 4.76 (s br, 1H), 7.17 (d,  $J = 6.8$  Hz, 1H), 7.25 – 7.42 (m, 4H), 7.49 – 7.59 (m, 1H), 7.62 (d,  $J = 6.5$  Hz, 1H), 7.89 – 7.98 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 26.7, 33.9, 37.7, 38.3, 52.0, 56.3, 59.1, 127.2, 128.1, 128.8, 129.3, 129.8, 130.0, 130.1, 131.1, 136.1, 137.8, 138.2, 169.6, 173.0, 175.0; HRMS (M+H) calcd. for  $\text{C}_{22}\text{H}_{26}\text{N}_2\text{O}_4$  383.1971, found: 383.1974;  $[\alpha]_{589}^{25} = +8.0$ .

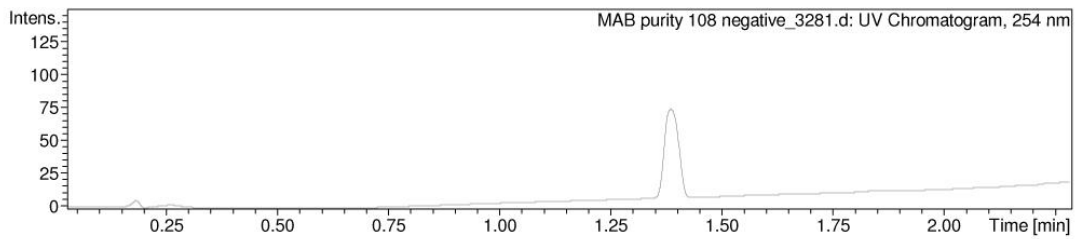
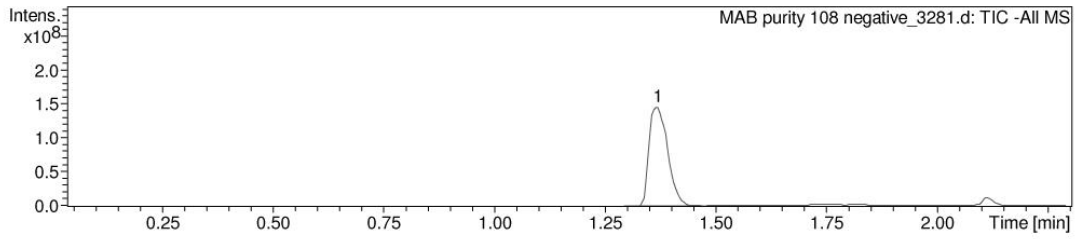


# LC-MS Analysis Report

## General Information

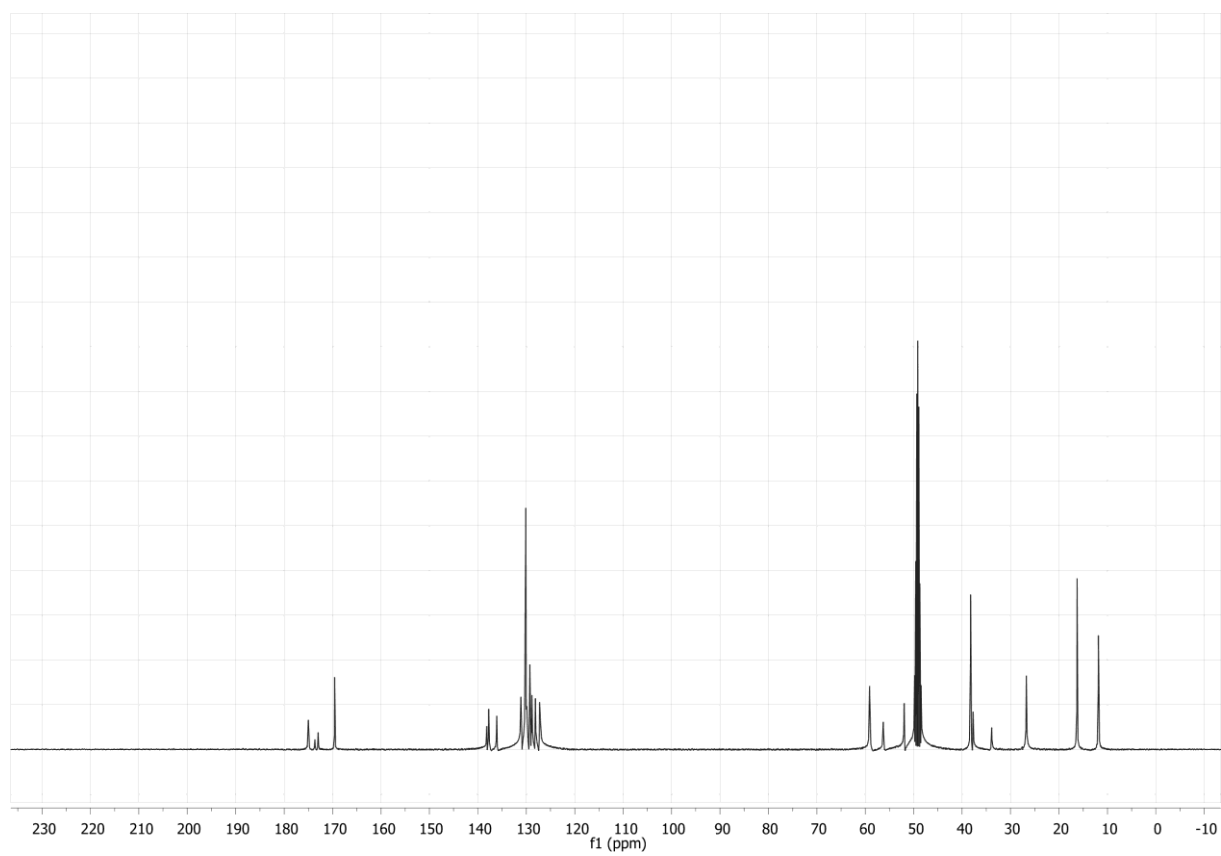
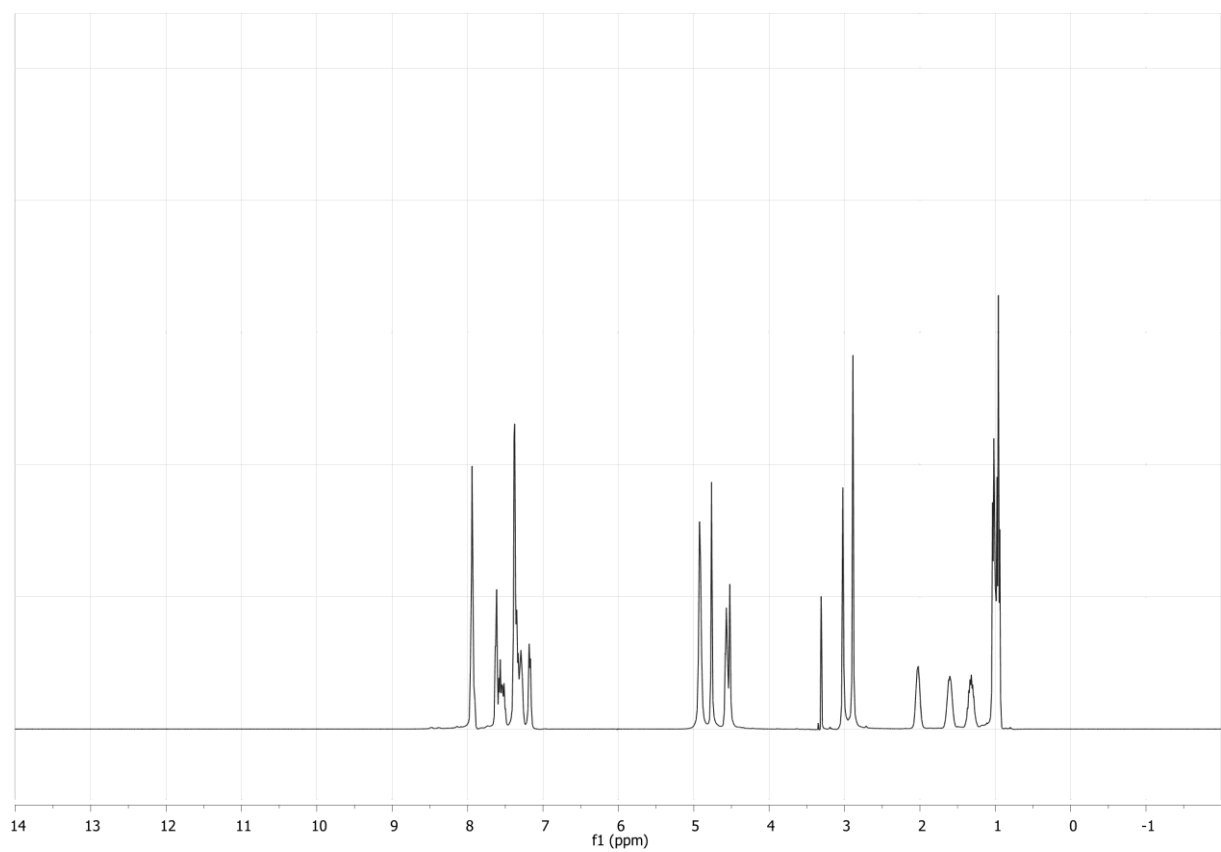
Sample ID: MAB purity 108 negative  
Date & Time: 10/7/2010 11:56:38 AM  
Data File: B:\Malte\Results\Purity\_check\MAB purity 108 negative\_3281.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

## Chromatogram

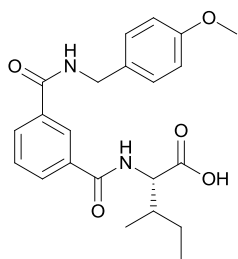


## Compound List (Area Frac. % of UV 254 nm)

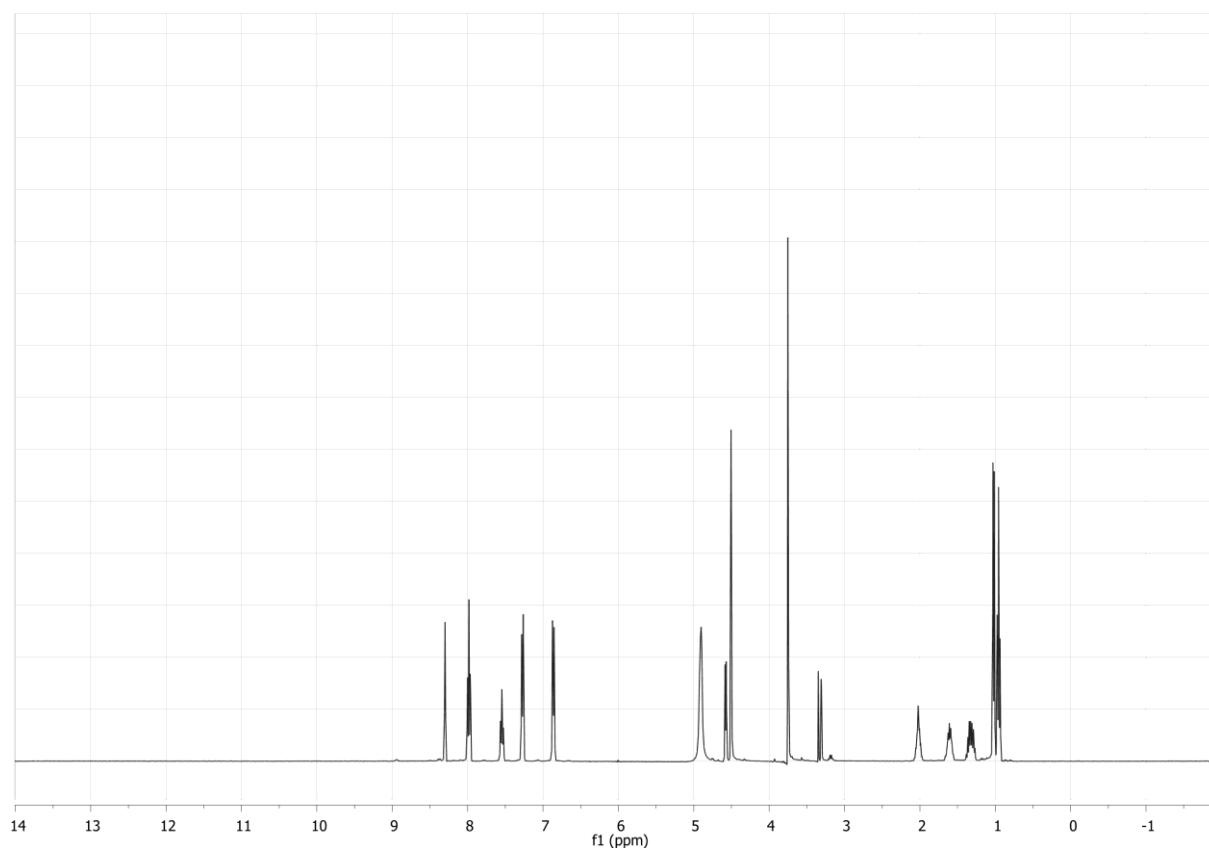
Cmpd. Label	Area Frac. %
Cmpd 1, 1.4 min	100.0

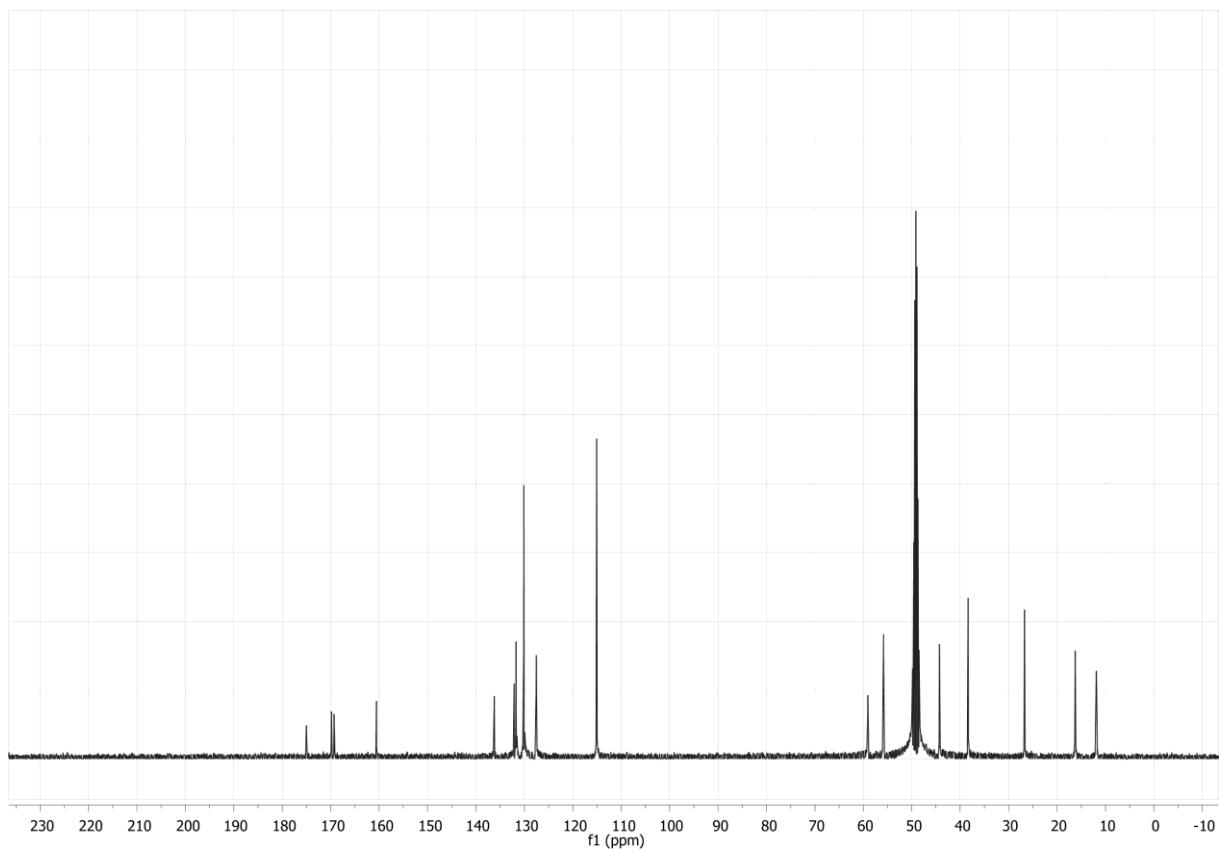


**(2S,3R)-2-(3-((4-Methoxybenzyl)carbamoyl)benzamido)-3-methylpentanoic acid (45).**

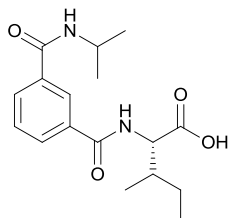


Yield: 46 mg (62%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J = 7.4$  Hz, 3H), 1.02 (d,  $J = 6.8$  Hz, 3H), 1.26 – 1.40 (m, 1H), 1.55 – 1.67 (m, 1H), 1.97 – 2.09 (m, 1H), 3.75 (s, 3H), 4.50 (s, 2H), 4.58 (d,  $J = 6.2$  Hz, 1H), 6.86 (d,  $J = 8.4$  Hz, 2H), 7.27 (d,  $J = 8.3$  Hz, 2H), 7.55 (t,  $J = 7.7$  Hz, 1H), 7.98 (t,  $J = 7.3$  Hz, 2H), 8.30 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.9, 16.2, 26.7, 38.4, 44.3, 55.8, 59.1, 115.1, 127.6, 130.0, 130.1, 131.6, 131.7, 132.1, 136.0, 136.2, 160.6, 169.2, 169.8, 175.0; HRMS (M+H) calcd. for  $\text{C}_{22}\text{H}_{26}\text{N}_2\text{O}_5$  399.1920; found: 399.1918.

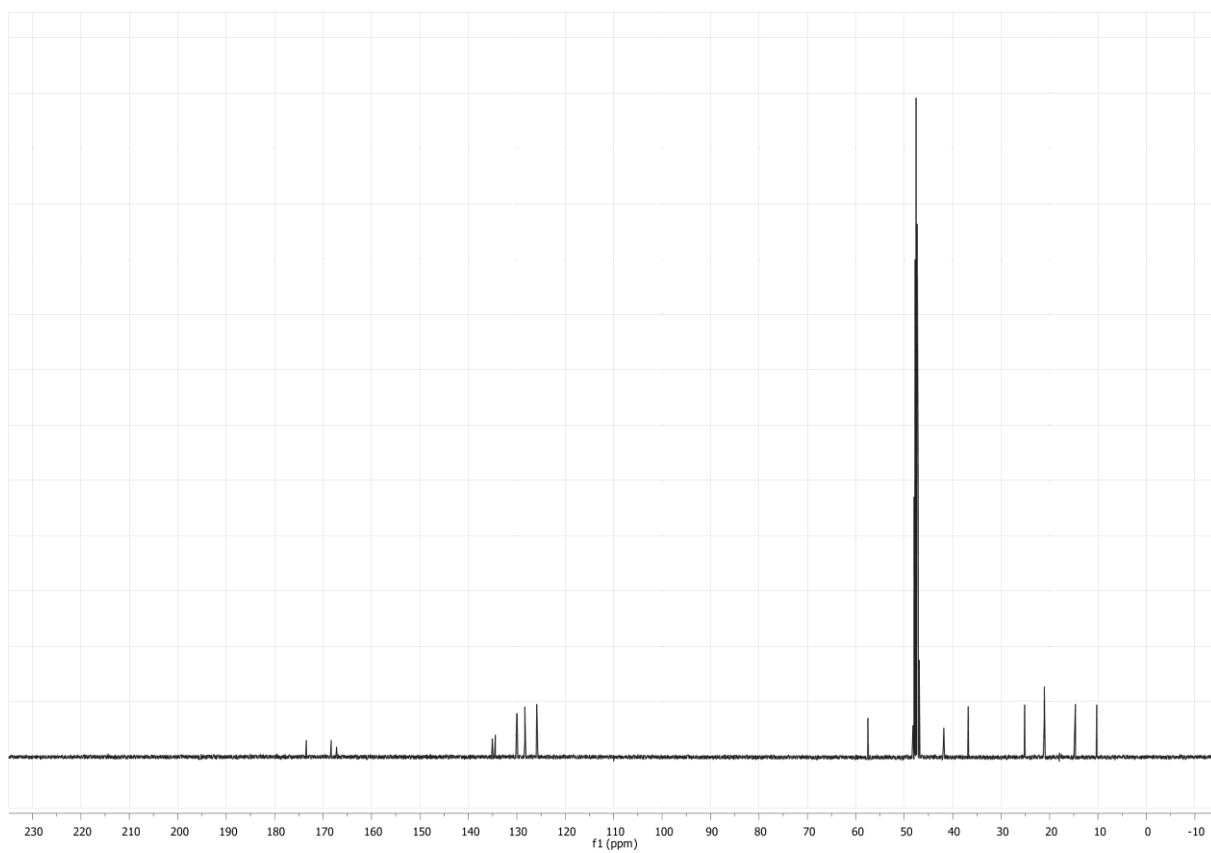
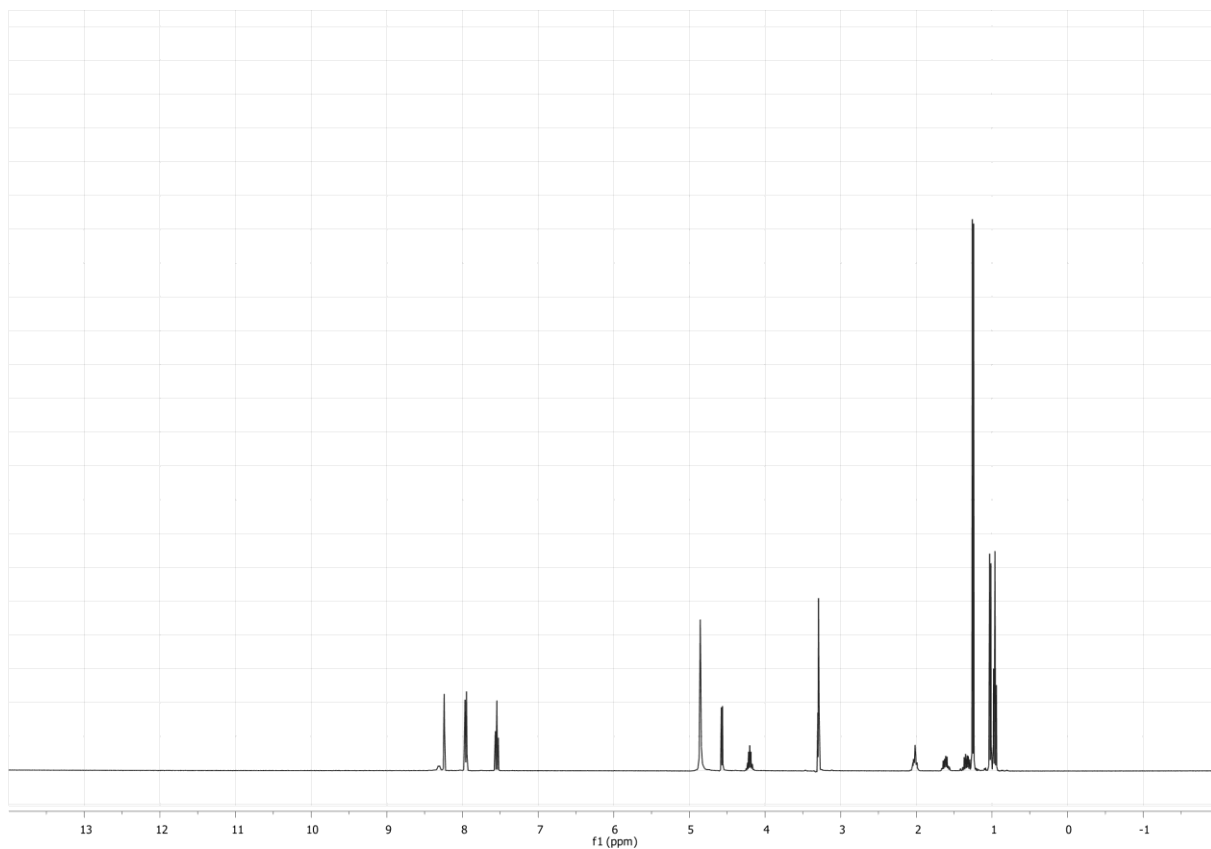




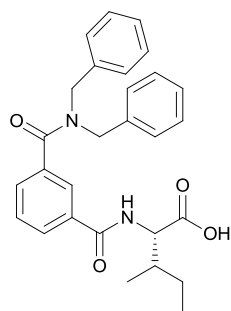
**(2S,3R)-2-(3-(Isopropylcarbamoyl)benzamido)-3-methylpentanoic acid (46).**



Yield: 55 mg (95%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.97 (t,  $J = 7.4$  Hz, 3H), 1.04 (d,  $J = 6.9$  Hz, 3H), 1.26 (d,  $J = 6.6$  Hz, 6H), 1.29 – 1.41 (m, 1H), 1.63 (ddq,  $J = 15.0$  Hz, 7.5 Hz, 4.3 Hz, 1H), 1.99 – 2.07 (m, 1H), 4.22 (hept,  $J = 6.6$  Hz, 1H), 4.59 (d,  $J = 6.3$  Hz, 1H), 7.55 (td,  $J = 7.0$  Hz, 0.4 Hz, 1H), 7.95 – 7.97 (m, 1H), 7.97 – 7.99 (m, 1H), 8.25 – 8.27 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.7, 16.1, 22.5, 26.6, 38.2, 43.3, 58.9, 127.3, 129.8, 131.4, 131.4, 135.8, 136.4, 168.6, 169.7, 174.9; HRMS (M+H) calcd. for  $\text{C}_{17}\text{H}_{24}\text{N}_2\text{O}_4$  321.1814; found: 321.1816.



**(2S,3R)-2-(3-(Dibenzylcarbamoyl)benzamido)-3-methylpentanoic acid (47).**



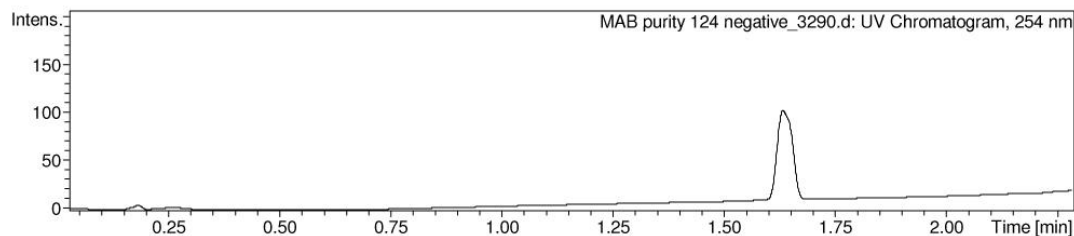
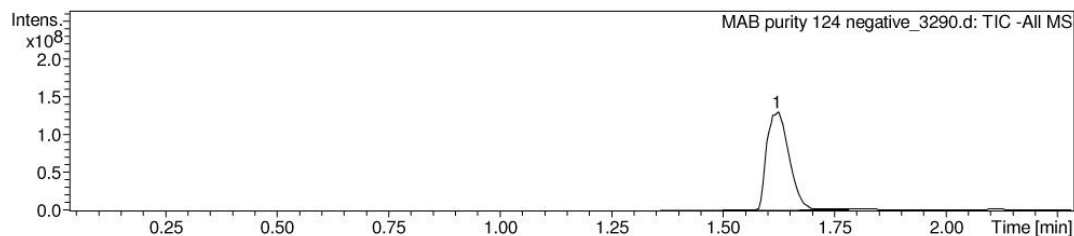
Yield: 37 mg (96%, white semi-solid):  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.95 (t,  $J$  = 7.4 Hz, 3H), 1.00 (d,  $J$  = 6.9 Hz, 3H), 1.23 – 1.37 (m, 1H), 1.59 (dq,  $J$  = 14.9 Hz, 7.5 Hz, 4.3 Hz, 1H), 1.95– 2.08 (m, 1H), 4.44 (s br, 2H), 4.55 (d,  $J$  = 6.3 Hz, 1H), 4.63 – 4.77 (m, 2H), 7.14 (s, 2H), 7.29 – 7.44 (m, 8H), 7.52 (t,  $J$  = 7.7 Hz, 1H), 7.64 (dt,  $J$  = 7.7 Hz, 1.4 Hz, 1H), 7.89 – 7.93 (m, 1H), 7.94 (t,  $J$  = 1.5 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 26.7, 38.3, 48.7, 53.3, 59.1, 127.0, 128.3, 128.9, 129.0, 129.5, 130.0, 130.1, 130.16, 130.21, 130.8, 136.2, 137.5, 137.7, 138.1, 169.6, 173.8, 175.0; HRMS (M+H) calcd. for  $\text{C}_{28}\text{H}_{30}\text{N}_2\text{O}_4$  459.2284; found: 459.2287;  $[\alpha]_{589}^{25} = +5.5$ .

## LC-MS Analysis Report

### General Information

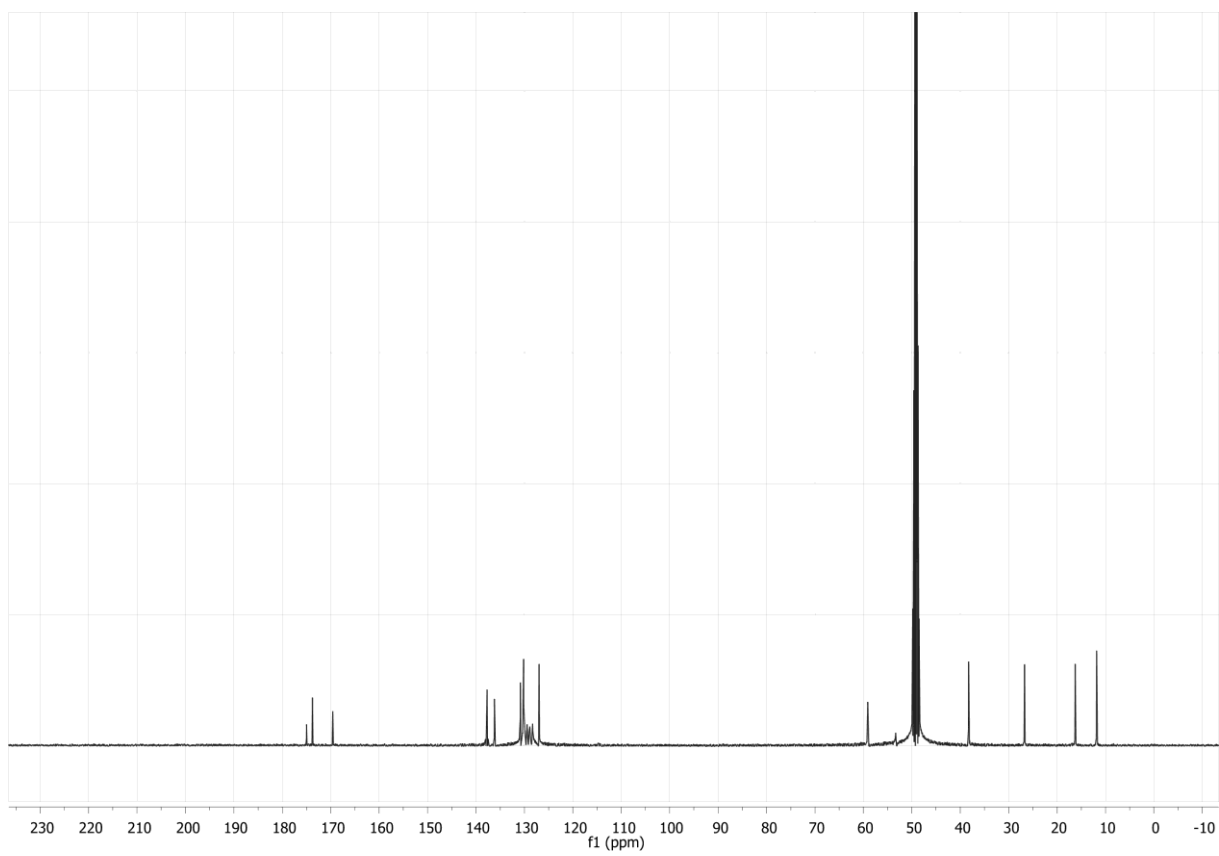
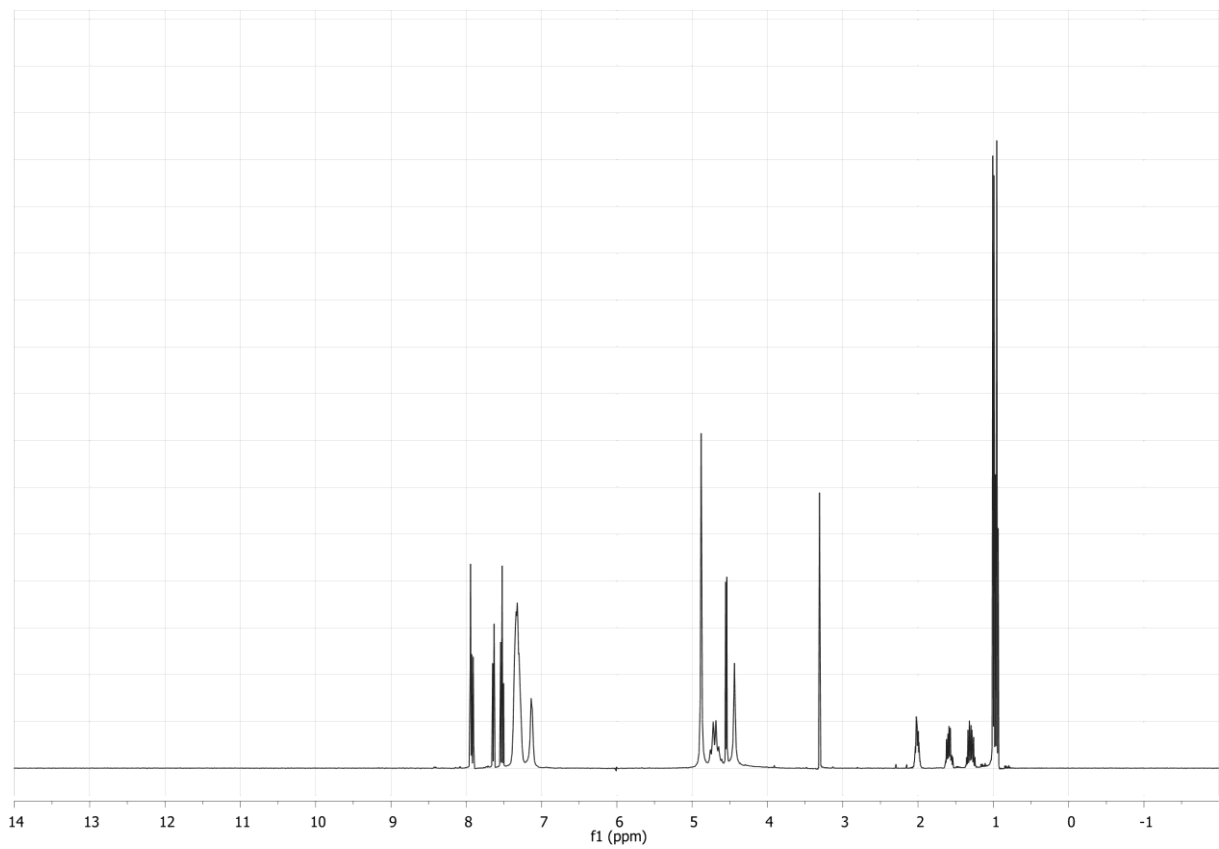
Sample ID: MAB purity 124 negative  
Date & Time: 10/7/2010 12:29:04 PM  
Data File: B:\Malte\Results\Purity\_check\MAB purity 124 negative\_3290.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram

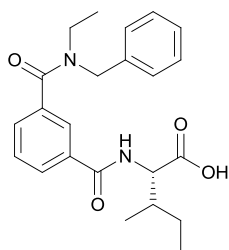


### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 1.6 min	100.0



**(2S,3R)-2-(3-(Benzyl(ethyl)carbamoyl)benzamido)-3-methylpentanoic acid (48).**



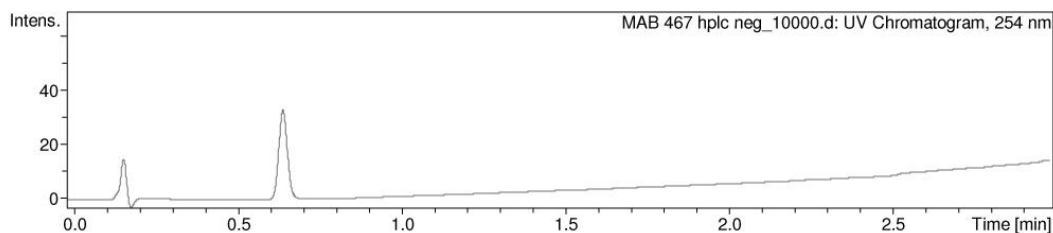
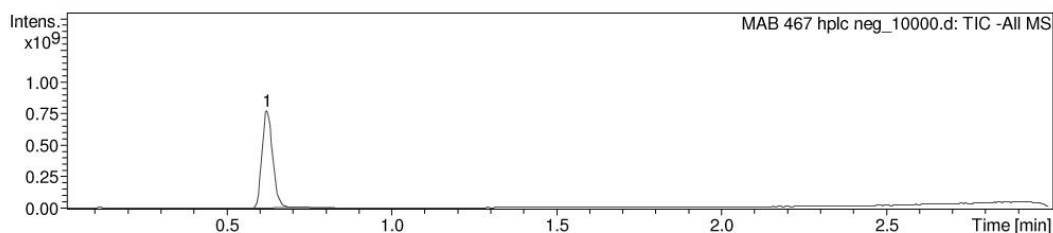
Yield: 47 mg (64%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.97 (t,  $J = 7.4$  Hz, 3H), 1.03 (d,  $J = 6.7$  Hz, 3H), 1.06 – 1.25 (m, 2H), 1.27 – 1.39 (m, 1H), 1.54 – 1.69 (m, 1H), 1.97 – 2.09 (m, 1H), 3.22 – 3.29 (m, 1H), 3.45 – 3.60 (m, 1H), 4.54 (s br, 1H), 4.56 (d,  $J = 6.4$  Hz, 1H), 4.80 (s, 1H), 7.16 – 7.43 (m, 5H), 7.49 – 7.65 (m, 2H), 7.88 – 7.98 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 12.7, 14.1, 16.3, 26.8, 38.3, 41.8, 44.9, 48.9, 53.7, 59.2, 126.7, 128.2, 128.7, 128.9, 129.2, 129.9, 130.1, 130.2, 130.6, 136.2, 138.1, 138.7, 169.6, 173.4, 175.1; HRMS (M+H) calcd. for  $\text{C}_{23}\text{H}_{28}\text{N}_2\text{O}_4$  397.2122, found: 397.2122;  $[\alpha]_{589}^{25} = +6.4$ .

## LC-MS Analysis Report

### General Information

Sample ID: MAB 467 hplc neg  
Date & Time: 5/18/2011 10:20:02 AM  
Data File: D:\BMC\Users\Malte\Results\benzamides\Acids purity\MAB 467 hplc neg\_10000.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

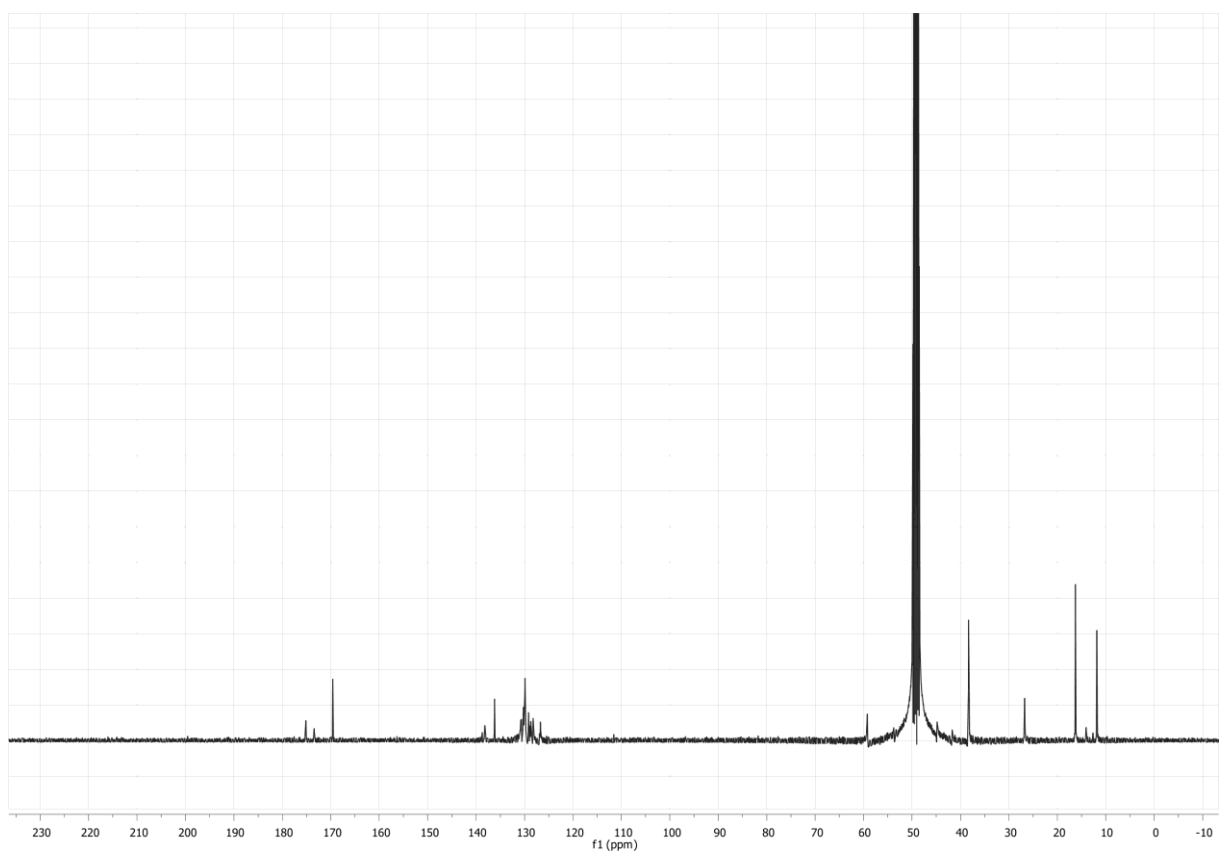
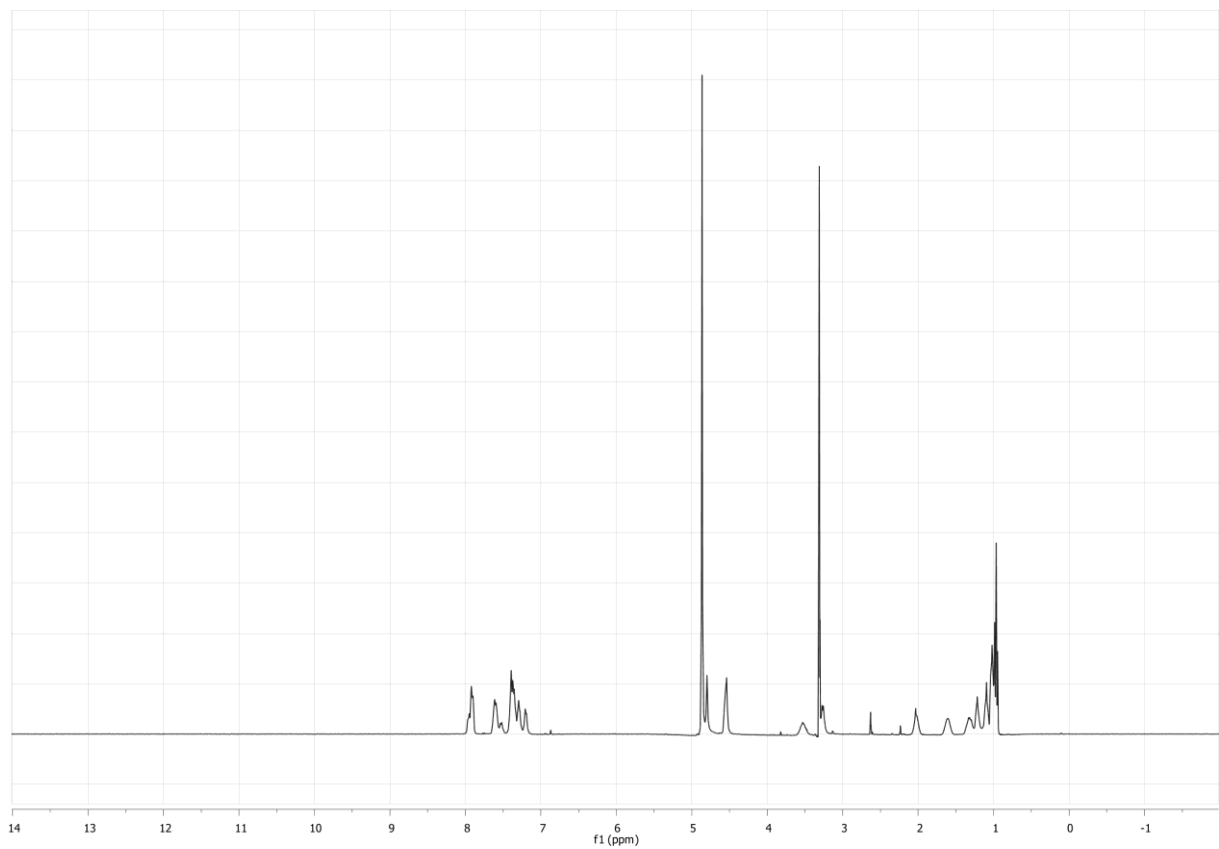
### Chromatogram



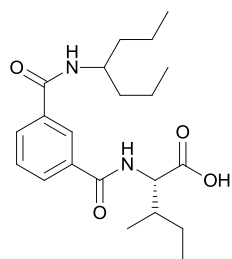
### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 0.62 min	100.0

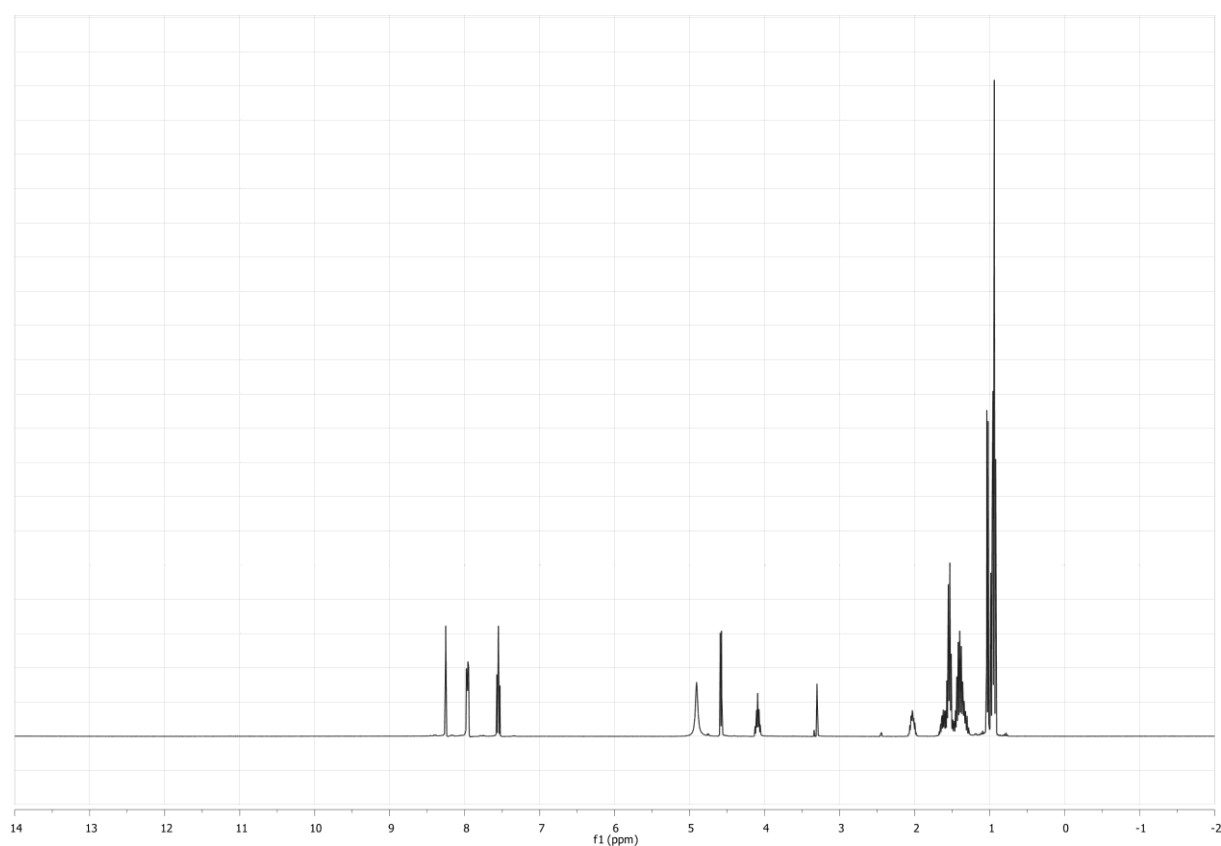


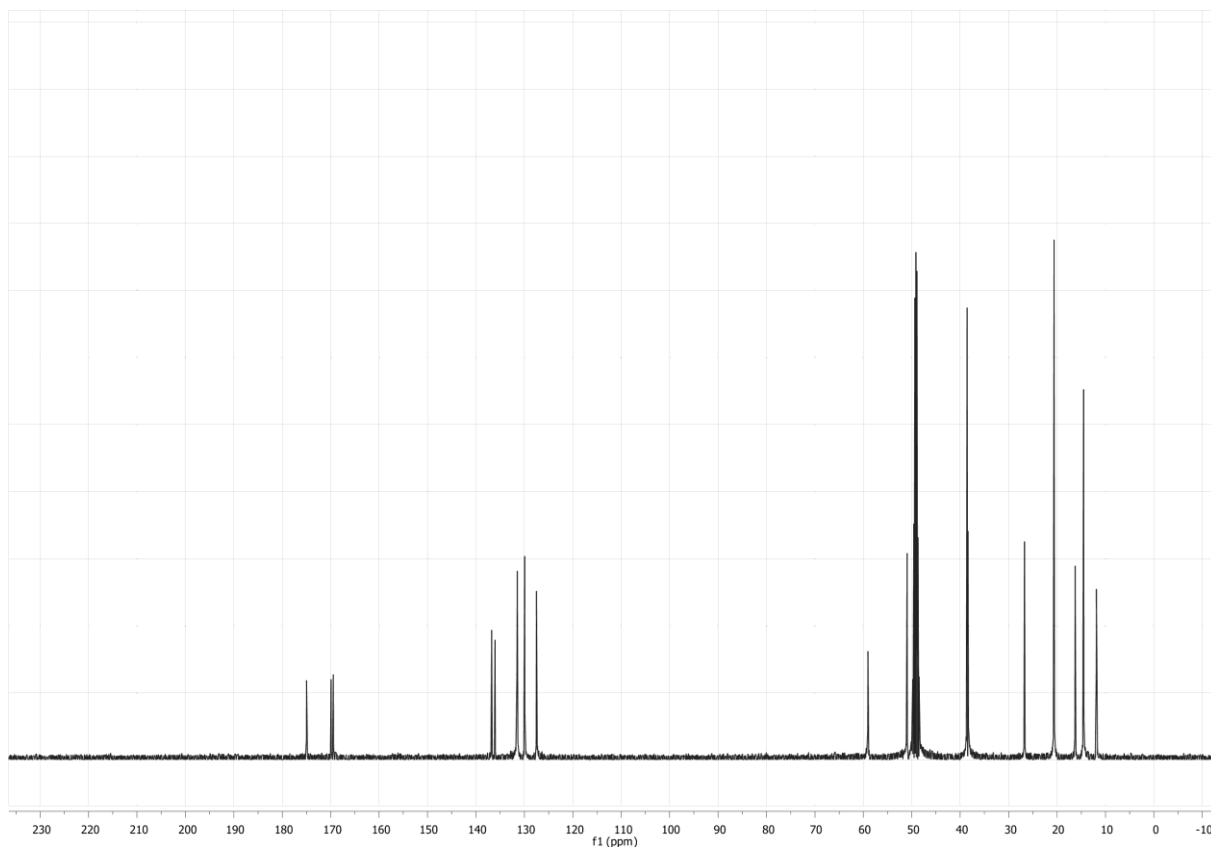


**(2S,3R)-2-(3-(Heptan-4-ylcarbamoyl)benzamido)-3-methylpentanoic acid (49).**

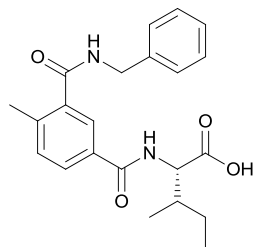


Yield: 49 mg (89%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.94 (t,  $J = 7.3$  Hz, 6H), 0.96 (t,  $J = 7.4$  Hz, 3H), 1.03 (d,  $J = 6.9$  Hz, 3H), 1.28 – 1.35 (m, 1H), 1.37 – 1.49 (m, 4H), 1.51 – 1.57 (m, 4H), 1.58 – 1.68 (m, 1H), 2.03 (ddt,  $J = 9.0$  Hz, 6.6 Hz, 4.4 Hz, 1H), 4.05 – 4.14 (m, 1H), 4.58 (d,  $J = 6.3$  Hz, 1H), 7.55 (t,  $J = 7.8$  Hz, 1H), 7.94 – 7.98 (m, 2H), 8.24 – 8.26 (m, 1H),  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.9, 14.5, 16.3, 20.6, 26.7, 38.4, 38.6, 51.0, 59.1, 127.5, 129.9, 131.5, 131.5, 136.1, 136.8, 169.5, 169.9, 175.0; HRMS (M+H) calcd. for  $\text{C}_{21}\text{H}_{32}\text{N}_2\text{O}_4$  377.2435, found: 377.2446;  $[\alpha]_{589}^{25} = +12.3$ .

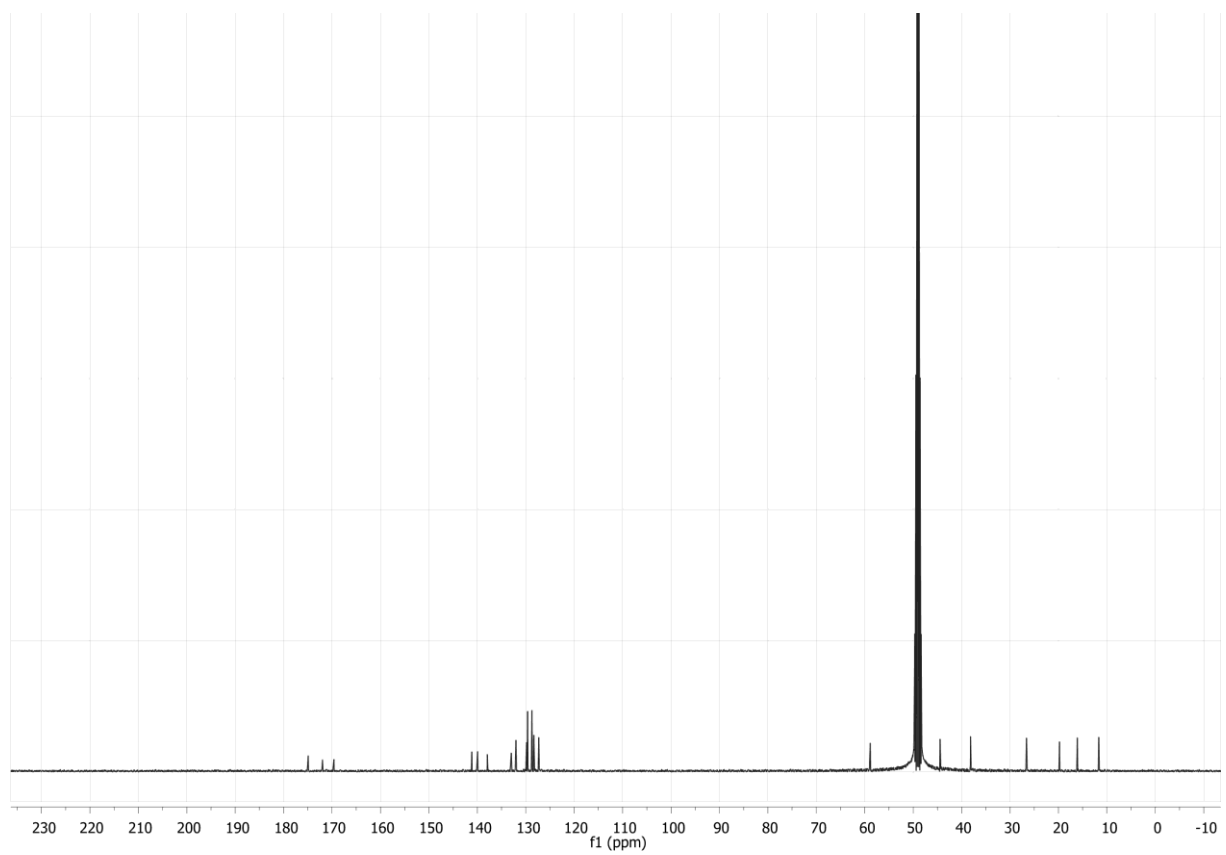
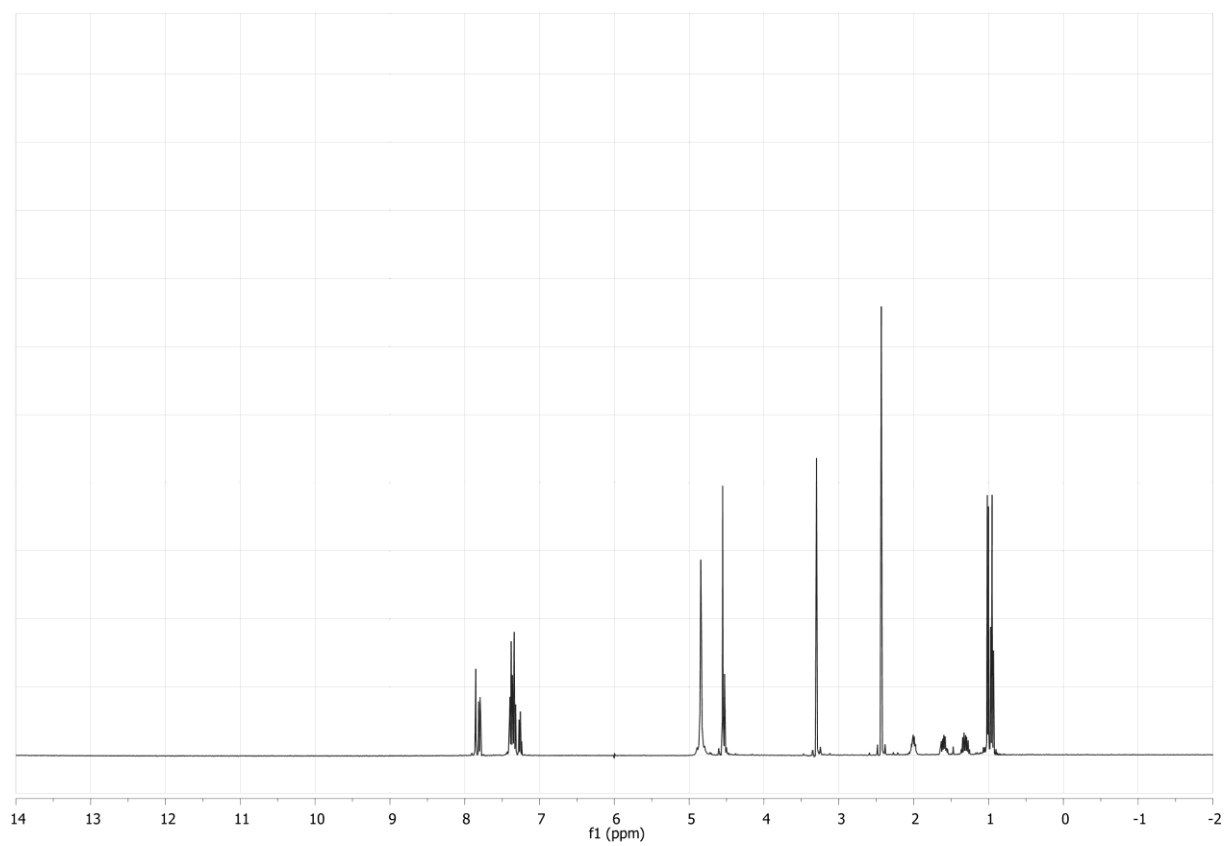




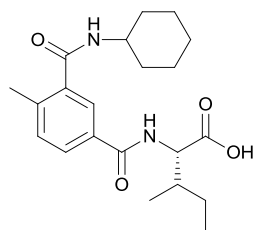
**(2S,3R)-2-(3-(Benzylcarbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (50).**



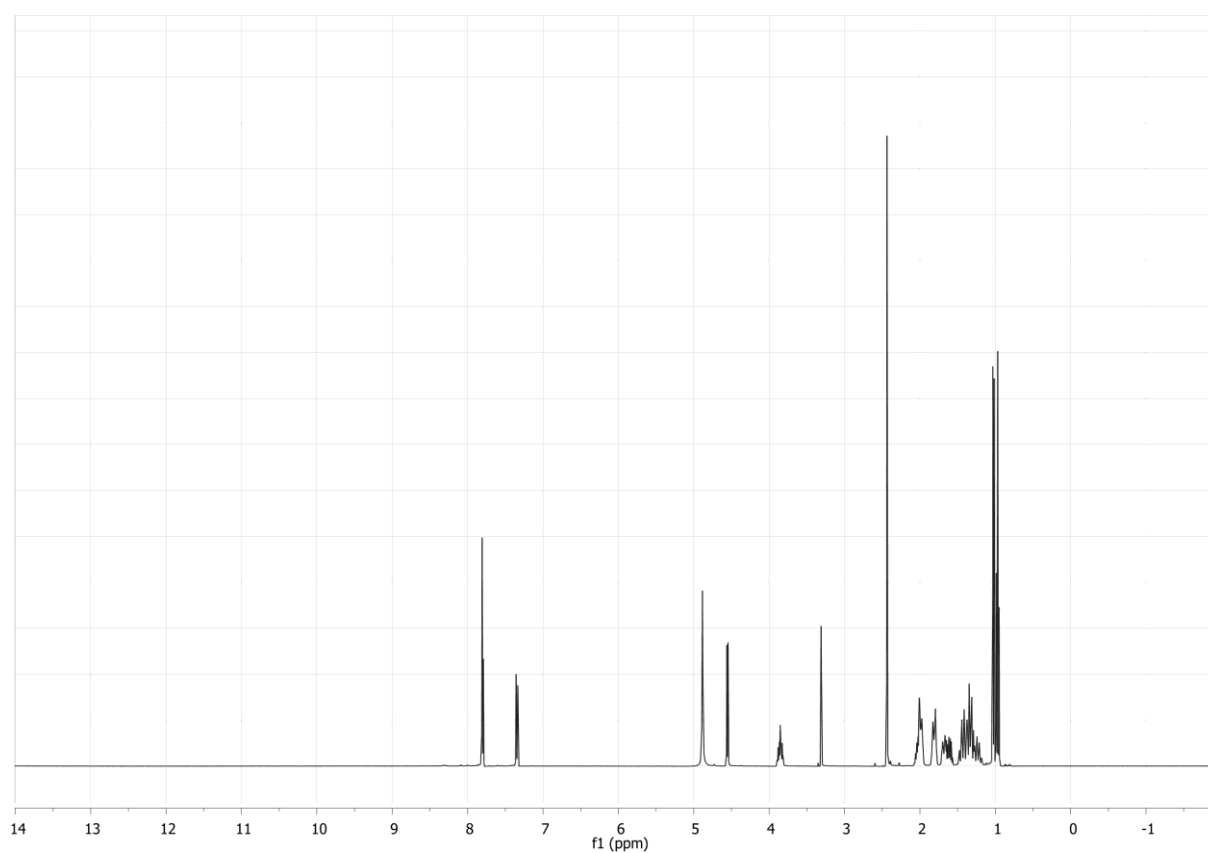
Yield: 26 mg (47%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.95 (t,  $J$  = 7.4 Hz, 3H), 1.01 (d,  $J$  = 6.9 Hz, 3H), 1.24 – 1.37 (m, 1H), 1.60 (ddq,  $J$  = 14.9 Hz, 7.5 Hz, 4.3 Hz, 1H), 2.01 (ddt,  $J$  = 8.9 Hz, 6.7 Hz, 4.3 Hz, 1H), 2.43 (s, 3H), 4.54 (d,  $J$  = 6.4 Hz, 1H), 4.55 (s, 2H), 7.23 – 7.28 (m, 1H), 7.32 – 7.37 (m, 3H), 7.37 – 7.40 (m, 2H), 7.80 (dd,  $J$  = 8.0 Hz, 2.0 Hz), 7.85 (d,  $J$  = 2.0 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.7, 16.1, 19.8, 26.6, 38.2, 44.5, 58.9, 127.3, 128.3, 128.7, 129.6, 129.8, 132.0, 133.0, 138.0, 139.9, 141.2, 169.6, 172.0, 174.9; HRMS (M+H) calcd. for  $\text{C}_{22}\text{H}_{26}\text{N}_2\text{O}_4$  383.1971; found: 383.1967.

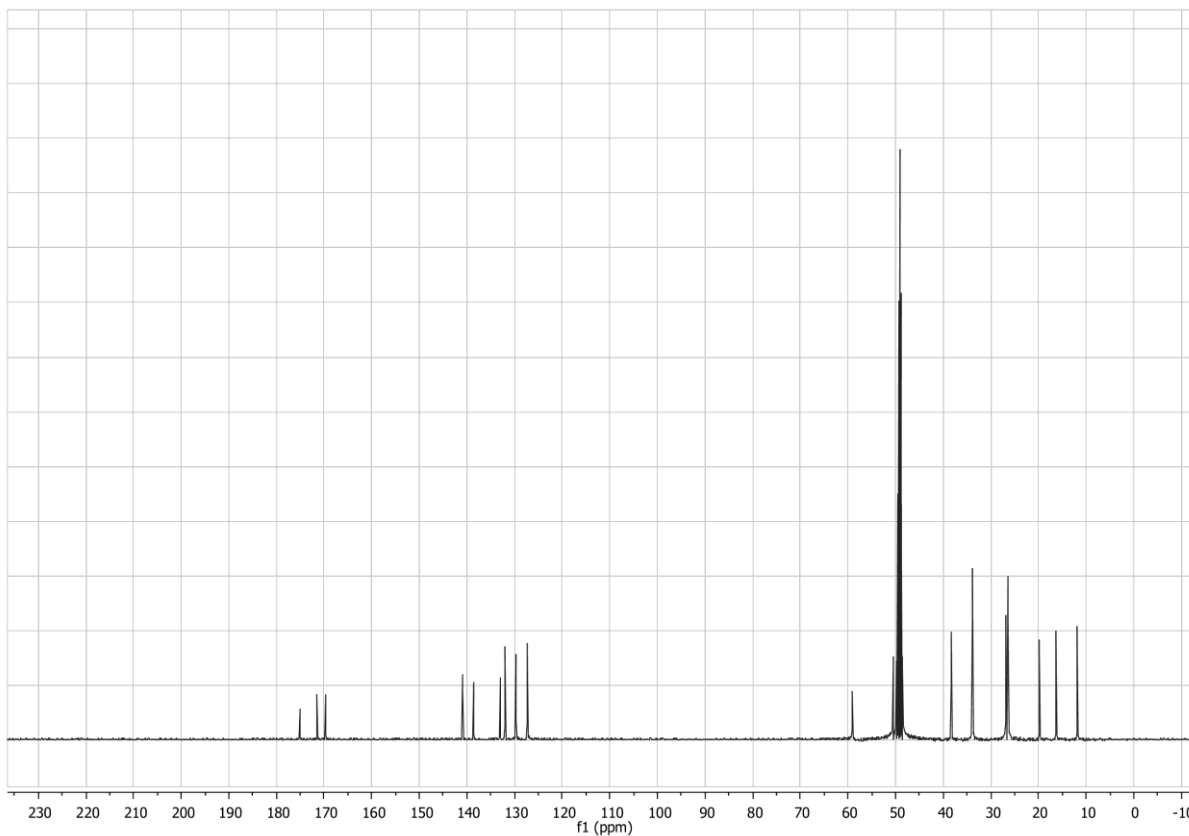


**(2S,3R)-2-(3-(Cyclohexylcarbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (51).**

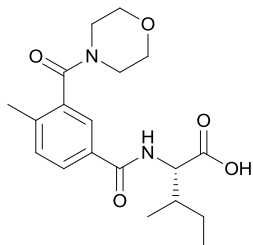


Yield: 63 mg (77%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.97 (t,  $J = 7.4$  Hz, 3H), 1.02 (d,  $J = 6.9$  Hz, 3H), 1.17 – 1.29 (m, 1H), 1.29 – 1.49 (m, 5H), 1.56 – 1.64 (m, 1H), 1.64 – 1.72 (m, 1H), 1.77 – 1.85 (m, 2H), 1.96 – 2.01 (m, 2H), 2.04 (ddd,  $J = 9.0$  Hz, 4.5 Hz, 2.3 Hz, 1H), 2.44 (s, 3H), 3.85 (tt,  $J = 10.7$  Hz, 3.9 Hz, 1H), 4.55 (d,  $J = 6.4$  Hz, 1H), 7.32 – 7.36 (m, 1H), 7.77 – 7.82 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 19.8, 26.4, 26.77, 26.79, 33.9, 38.3, 50.5, 59.0, 127.3, 129.7, 132.0, 133.0, 138.6, 140.9, 169.7, 171.4, 175.1; HRMS ( $\text{M}+\text{H}$ ) calcd. for  $\text{C}_{21}\text{H}_{30}\text{N}_2\text{O}_4$  375.2284; found: 375.2281.

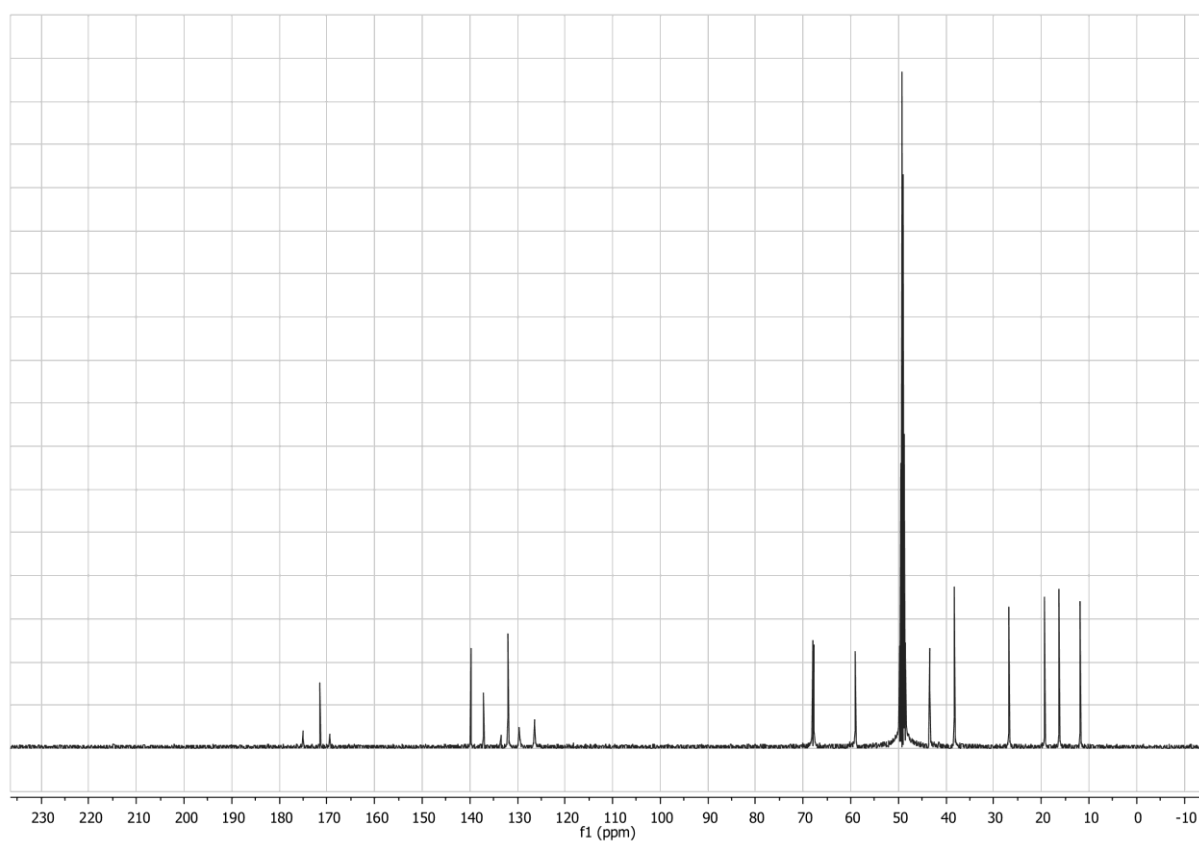
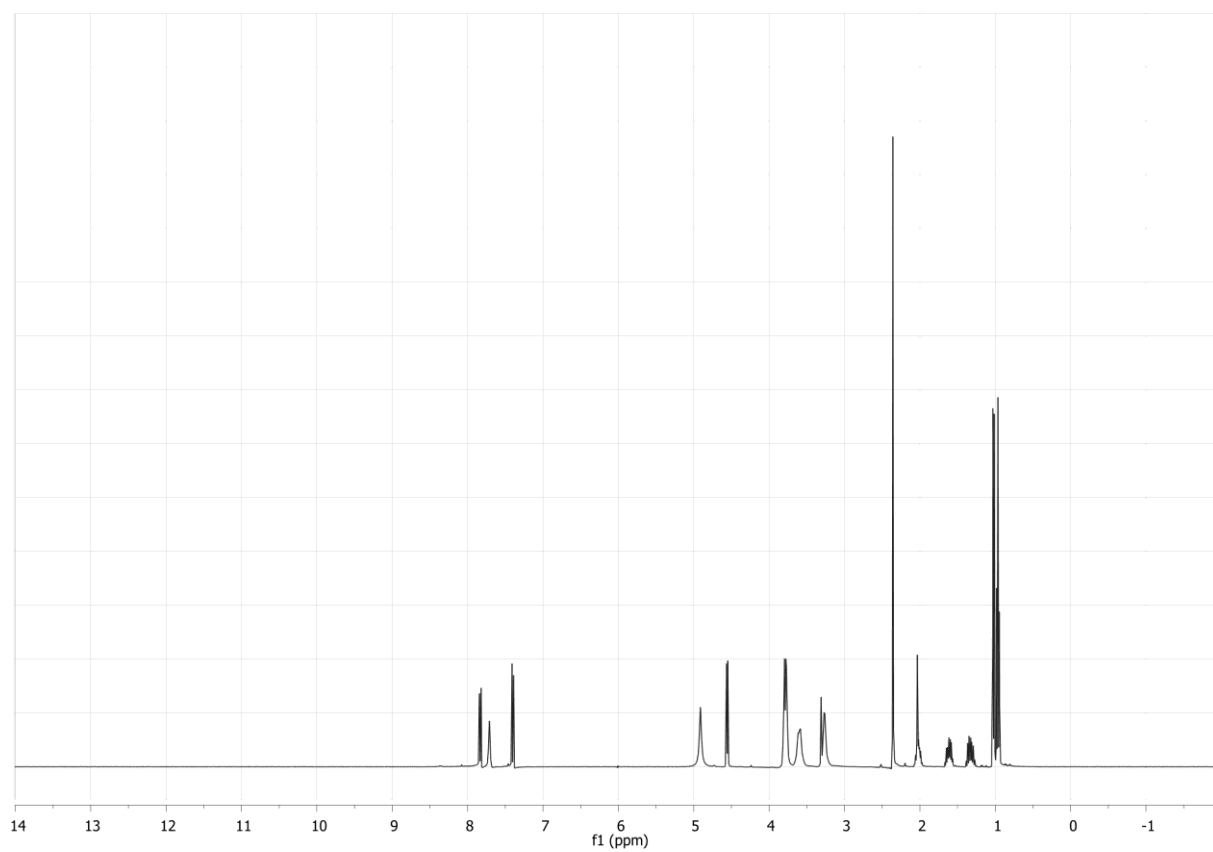




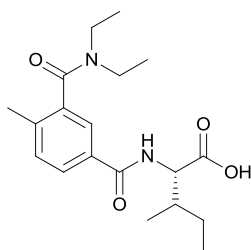
**(2S,3R)-3-Methyl-2-(4-methyl-3-(morpholine-4-carbonyl)benzamido)pentanoic acid (52).**



Yield: 62 mg (75%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J$  = 7.4 Hz, 3H), 1.02 (d,  $J$  = 6.9 Hz, 3H), 1.26 – 1.40 (m, 1H), 1.55 – 1.67 (m, 1H), 1.98 – 2.07 (m, 1H), 2.36 (s, 3H), 3.27 (d,  $J$  = 3.7 Hz, 2H), 3.55 – 3.66 (m, 2H), 3.78 (tt,  $J$  = 7.3 Hz, 3.5 Hz, 4H), 4.56 (d,  $J$  = 6.4 Hz, 1H), 7.40 (d,  $J$  = 8.1 Hz, 1H), 7.71 (s, 1H), 7.83 (dd,  $J$  = 8.0 Hz, 1.9 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 19.3, 26.8, 38.3, 43.4, 48.8, 59.0, 67.8, 68.0, 126.4, 129.6, 132.0, 133.5, 137.1, 139.8, 169.4, 171.4, 175.0; HRMS (M+H) calcd. for  $\text{C}_{19}\text{H}_{26}\text{N}_2\text{O}_5$  363.192; found: 363.1913.



**(2S,3R)-2-(3-(Diethylcarbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (53).**



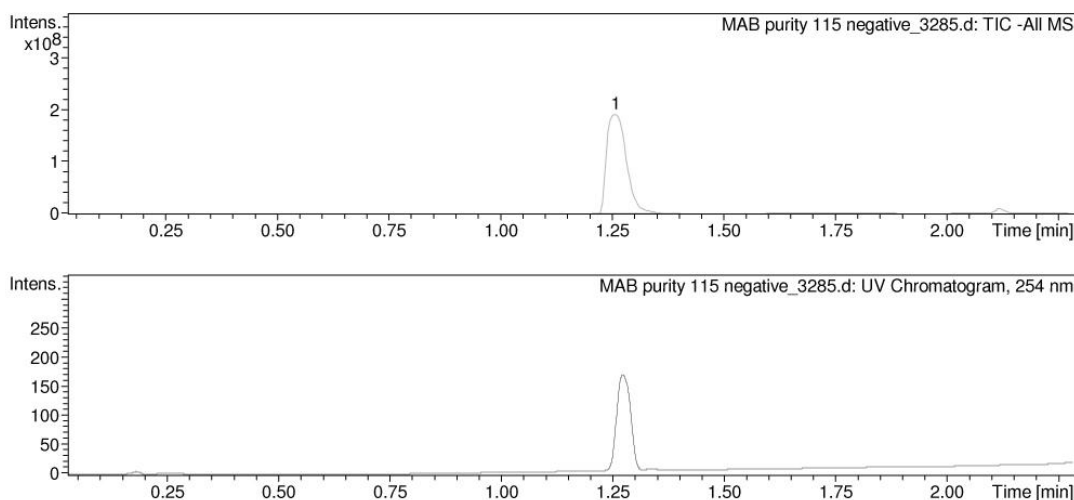
Yield: 30 mg (72%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J$  = 7.4 Hz, 3H), 1.02 (d,  $J$  = 6.8 Hz, 3H), 1.07 (t,  $J$  = 7.1 Hz, 3H), 1.29 (t,  $J$  = 7.1 Hz, 3H), 1.31 – 1.39 (m, 1H), 1.56 – 1.68 (m, 1H), 1.08 – 2.08 (m, 1H), 2.34 (s, 3H), 3.08 – 3.28 (m, 2H), 3.44 – 3.77 (m, 2H), 4.55 (d,  $J$  = 6.5 Hz, 1H), 7.39 (d,  $J$  = 8.0 Hz, 1H), 7.70 (s, 1H), 7.83 (dd,  $J$  = 8.0 Hz, 1.8 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 13.2, 14.3, 16.3, 19.1, 26.8, 38.2, 40.7, 44.7, 59.0, 126.0, 129.3, 131.9, 133.3, 138.2, 139.5, 169.5, 172.4, 175.1; HRMS (M+H) calcd. for  $\text{C}_{19}\text{H}_{28}\text{N}_2\text{O}_4$  349.2127, found: 349.2136;  $[\alpha]_{589}^{25} = +9.7$ .

## LC-MS Analysis Report

### General Information

Sample ID: MAB purity 115 negative  
Date & Time: 10/7/2010 12:11:04 PM  
Data File: D:\BMC\Users\Malte\Results\benzamides\Acids purity\MAB purity 115 negative\_3285.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

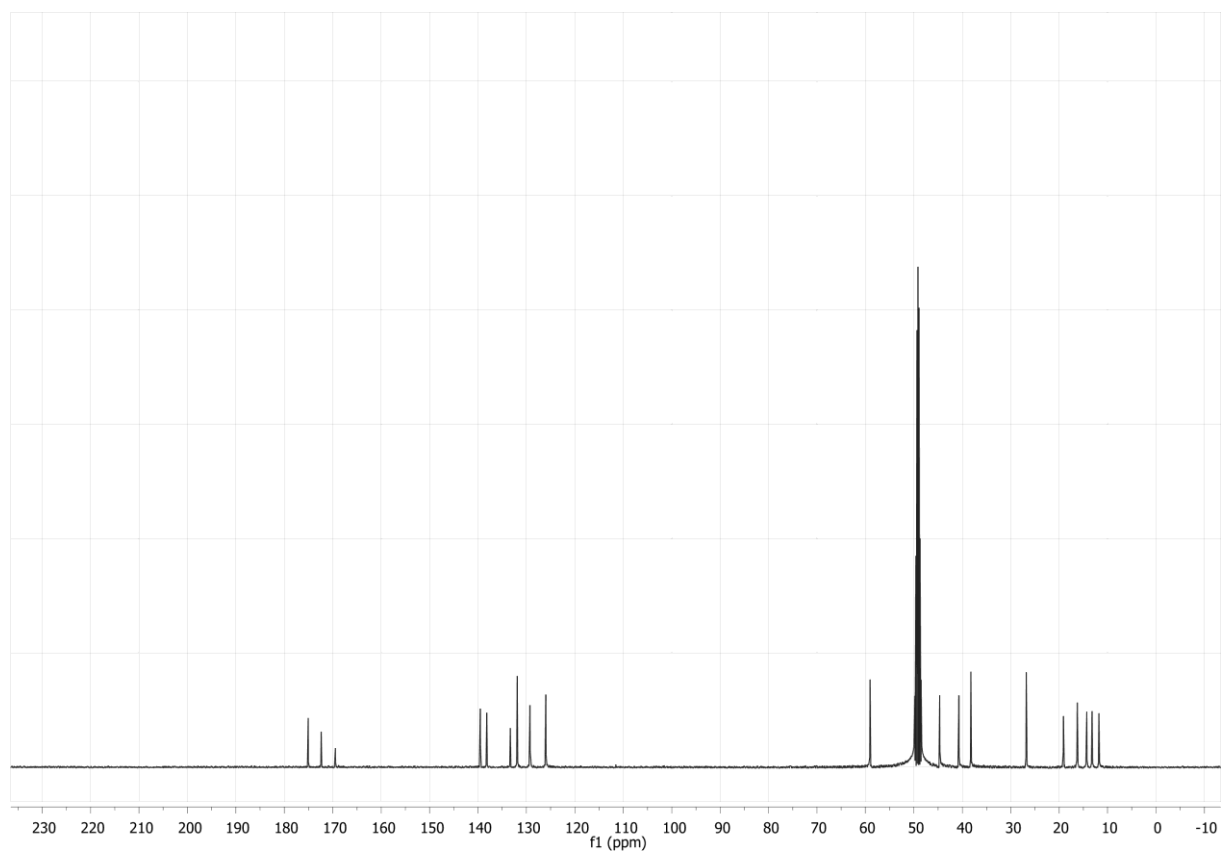
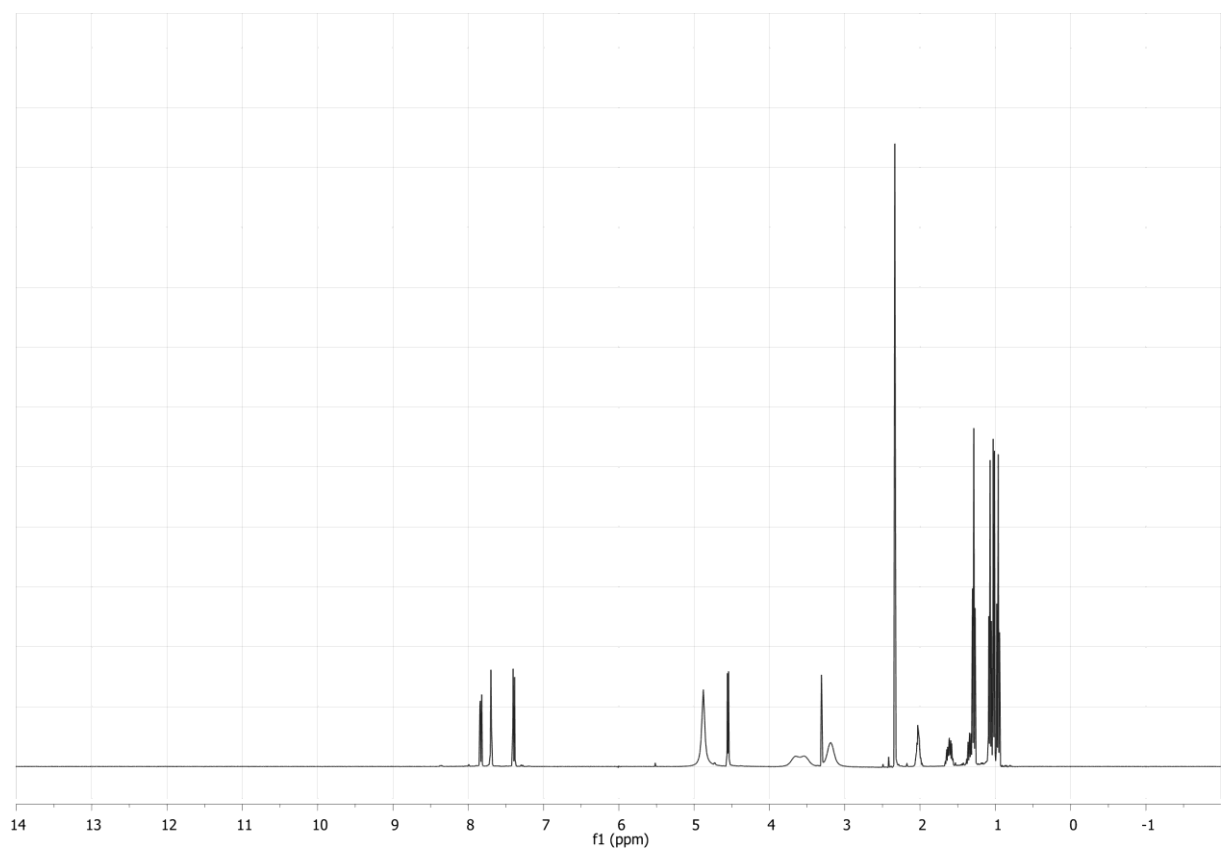
### Chromatogram



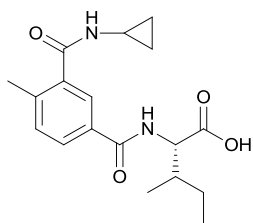
### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 1.3 min	100.0

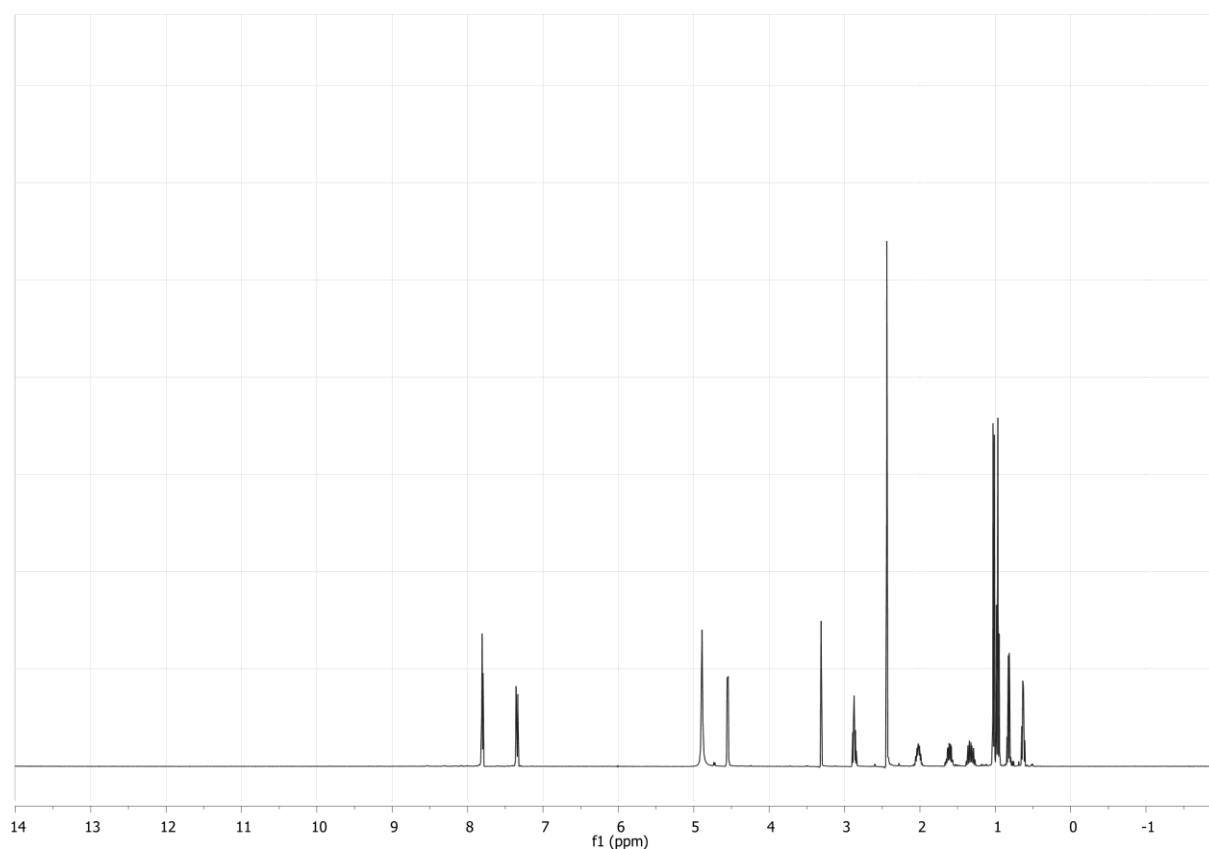


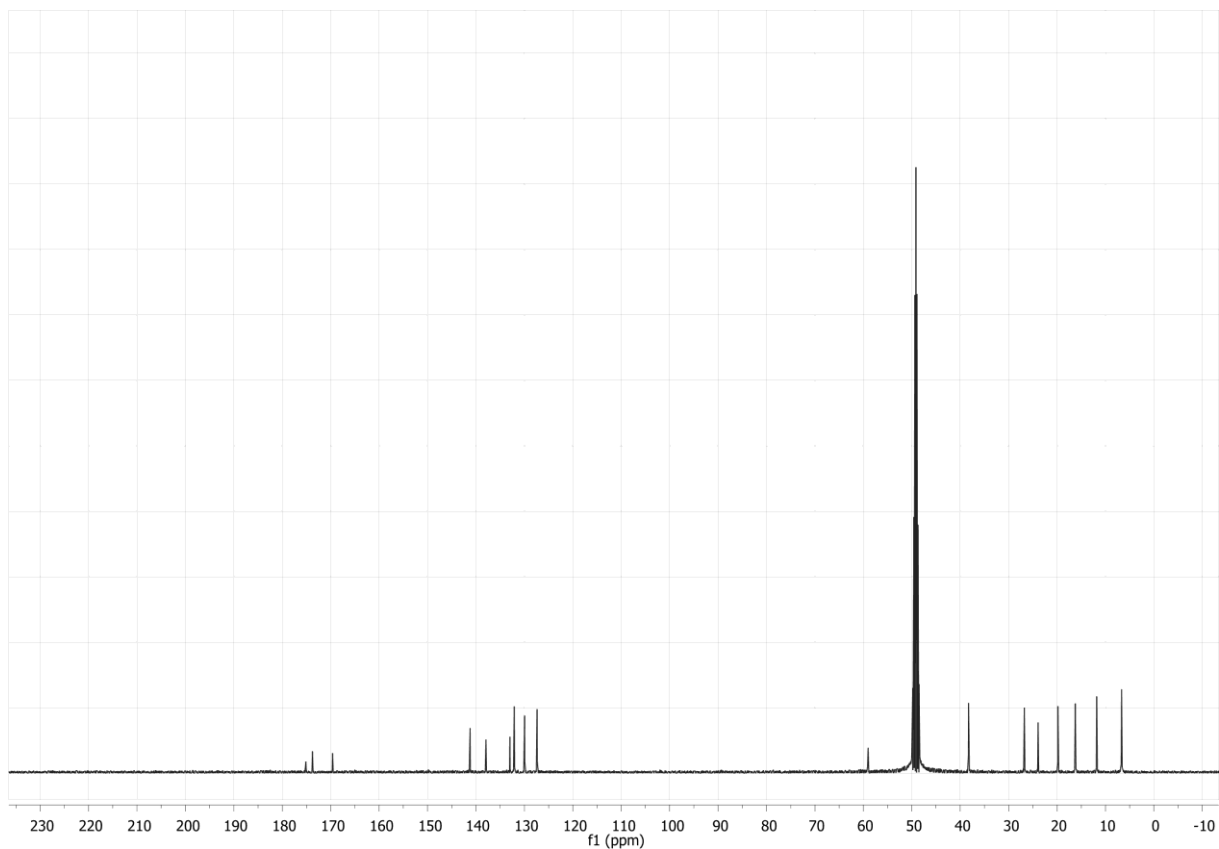


**(2S,3R)-2-(3-(Cyclopropylcarbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (54).**

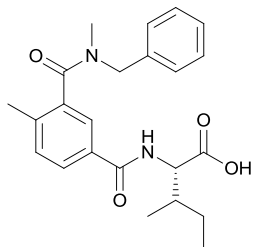


Yield: 29 mg (84%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.63 (dt,  $J = 7.0$  Hz, 4.4 Hz, 2H), 0.82 (dt,  $J = 7.1$  Hz, 5.1 Hz, 2H), 0.97 (t,  $J = 7.4$  Hz, 3H), 1.02 (d,  $J = 6.9$  Hz, 3H), 1.26 – 1.39 (m, 1H), 1.61 (dq,  $J = 15.0$  Hz, 7.5 Hz, 4.3 Hz, 1H), 2.02 (dtd,  $J = 9.0$  Hz, 6.7 Hz, 4.3 Hz, 1H), 2.44 (s, 3H), 2.84 – 2.91 (m, 1H), 4.55 (d,  $J = 6.4$  Hz, 1H), 7.32 – 7.37 (m, 1H), 7.78 – 7.83 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 6.67, 6.68, 11.8, 16.2, 19.8, 23.9, 26.8, 38.3, 59.0, 127.4, 130.0, 132.1, 133.0, 137.9, 141.2, 169.6, 173.8, 175.1; HRMS (M+H) calcd. for  $\text{C}_{18}\text{H}_{24}\text{N}_2\text{O}_4$  333.1809, found: 333.1808.





**(2S,3R)-2-(3-(Benzyl(methyl)carbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (55).**



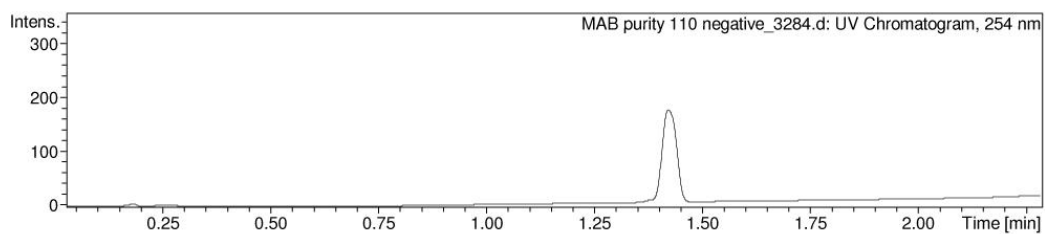
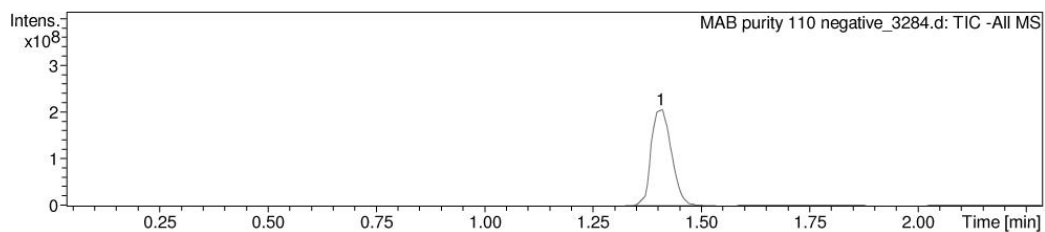
Yield: 46 mg (68%, white semi-solid);  $^1\text{H NMR}$  ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J = 7.4$  Hz, 3H), 1.02 (d,  $J = 6.9$  Hz, 3H), 1.24 – 1.39 (m, 1H), 1.54 – 1.67 (m, 1H), 1.97 – 2.07 (m, 1H), 2.32 (s, 3H), 2.75 (s, 2H), 3.08 (s, 1H), 4.39 (s br, 1H), 4.56 (d,  $J = 6.7$  Hz, 1H), 4.66 – 4.87 (m, 1H), 7.12 (d,  $J = 7.1$  Hz, 1H), 7.23 – 7.35 (m, 2H), 7.35 – 7.44 (m, 3H), 7.71 – 7.78 (m, 1H), 7.80 – 7.86 (m, 1H);  $^{13}\text{C NMR}$  ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 19.2, 26.8, 33.5, 36.6, 38.2, 51.4, 55.8, 59.1, 126.2, 128.5, 129.0, 129.4, 129.5, 130.0, 130.1, 131.9, 133.6, 137.6, 138.2, 139.5, 169.5, 173.0, 175.1; HRMS (M+H) calcd. for  $\text{C}_{23}\text{H}_{28}\text{N}_2\text{O}_4$  397.2127; found: 397.2117;  $[\alpha]_{589}^{25} = +7.3$ .

# LC-MS Analysis Report

## General Information

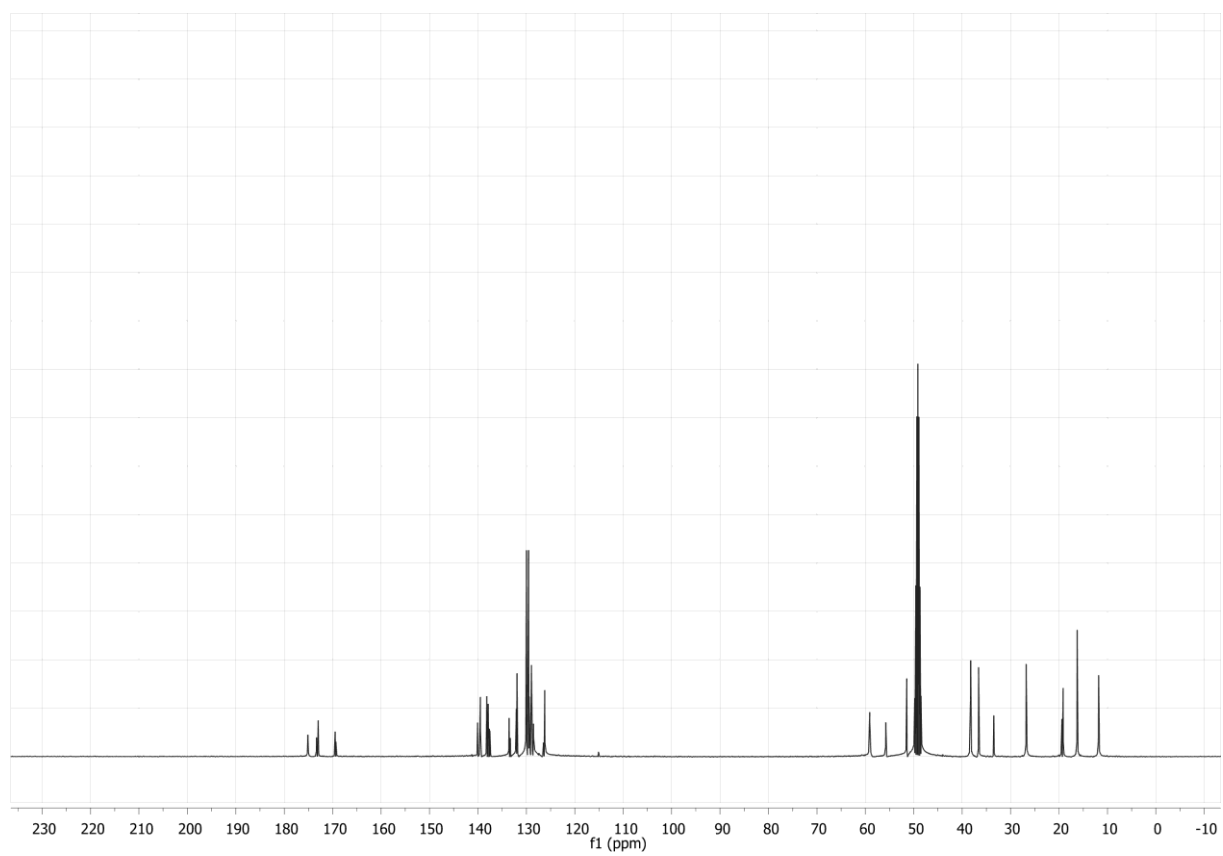
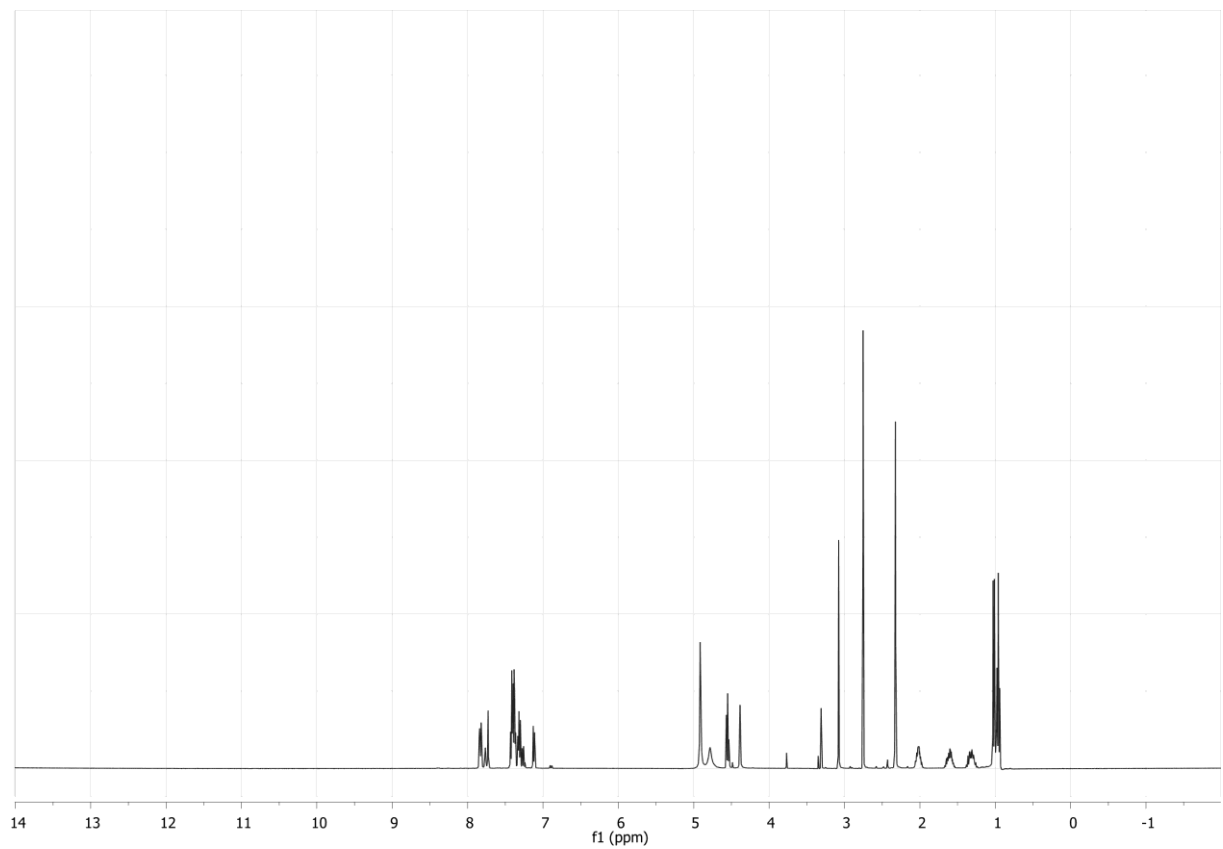
Sample ID: MAB purity 110 negative  
Date & Time: 10/7/2010 12:07:29 PM  
Data File: B:\Malte\Results\Purity\_check\MAB purity 110 negative\_3284.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

## Chromatogram

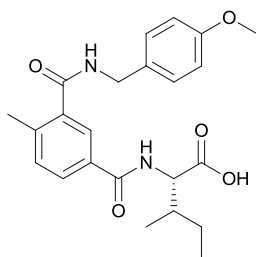


## Compound List (Area Frac. % of UV 254 nm)

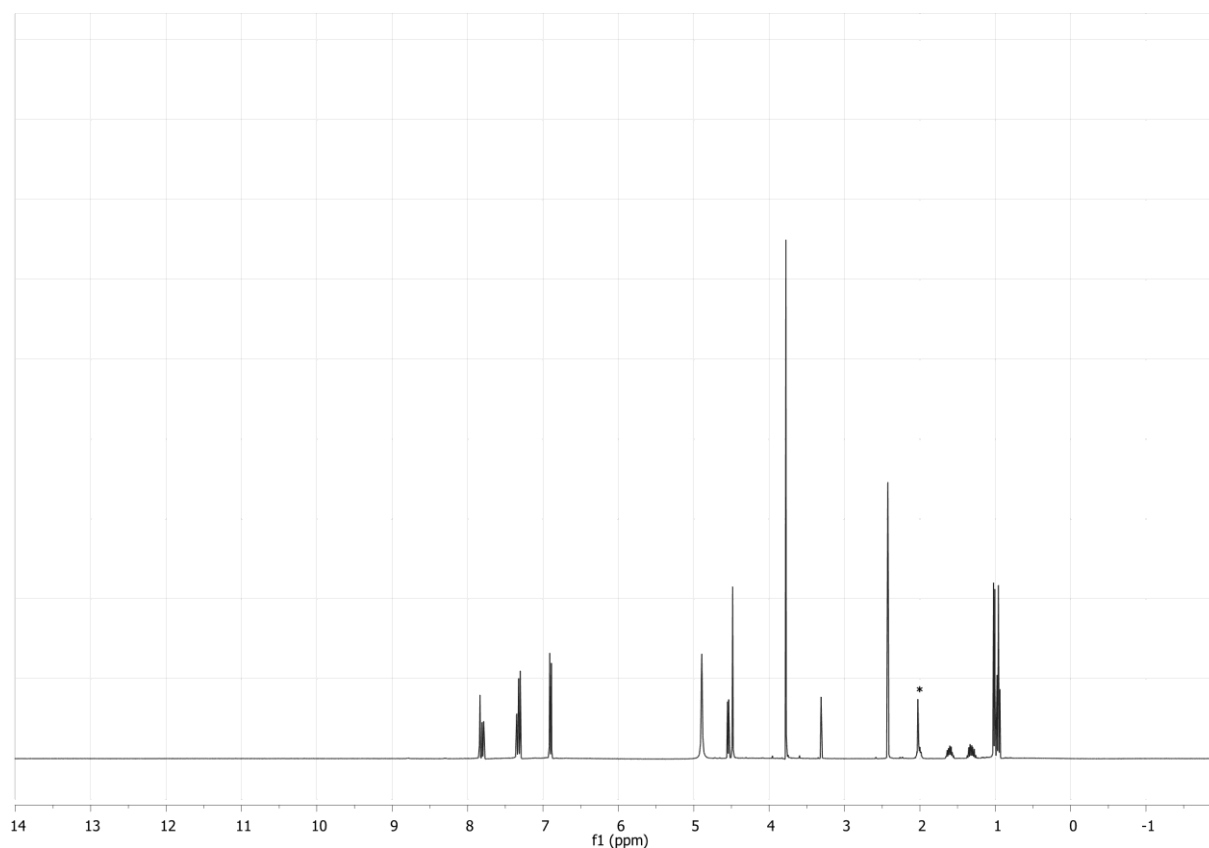
Cmpd. Label	Area Frac. %
Cmpd 1, 1.4 min	100.0

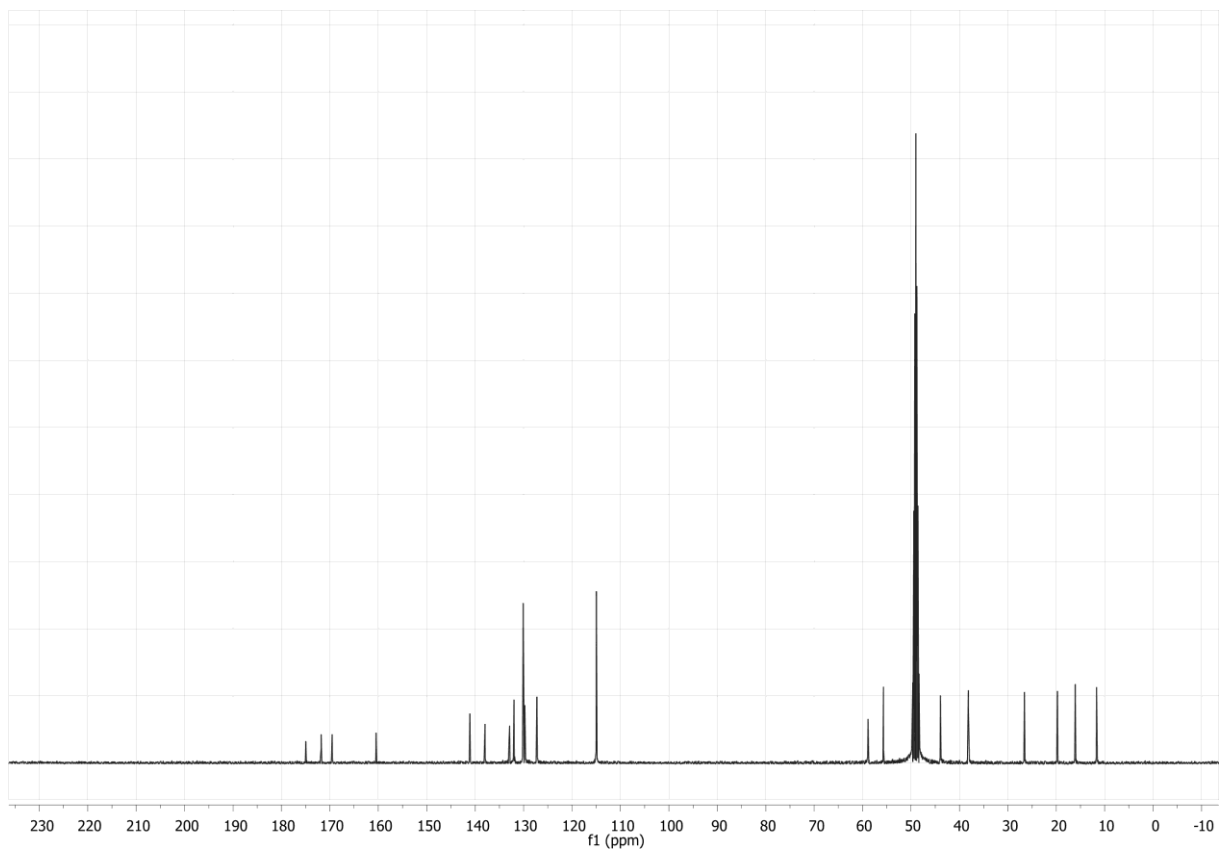


**(2*S*,3*R*)-2-(3-((4-Methoxybenzyl)carbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (56).**

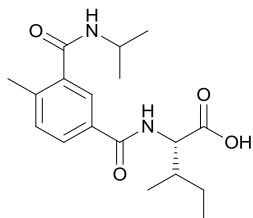


Yield: 54 mg (65%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J = 7.4$  Hz, 3H), 1.01 (d,  $J = 6.9$  Hz, 3H), 1.25 – 1.38 (m, 1H), 1.60 (ddq,  $J = 14.9$  Hz, 7.5 Hz, 4.3 Hz, 1H), 2.06 – 1.97 (m, 1H), 2.42 (s, 3H), 3.78 (s, 3H), 4.48 (s, 2H), 4.54 (d,  $J = 6.4$  Hz, 1H), 6.88 – 6.92 (m, 2H), 7.29 – 7.32 (m, 2H), 7.34 (d,  $J = 8.0$  Hz, 1H), 7.80 (dd,  $J = 7.9$  Hz, 2.0 Hz, 1H), 7.84 (d,  $J = 1.9$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.7, 16.1, 19.8, 26.6, 38.2, 43.9, 55.7, 58.9, 115.0, 127.3, 129.8, 130.1, 131.9, 132.0, 132.9, 138.0, 141.1, 160.5, 169.5, 171.8, 175.0; HRMS (M+H) calcd. for  $\text{C}_{23}\text{H}_{28}\text{N}_2\text{O}_5$  413.2076; found: 413.2073.

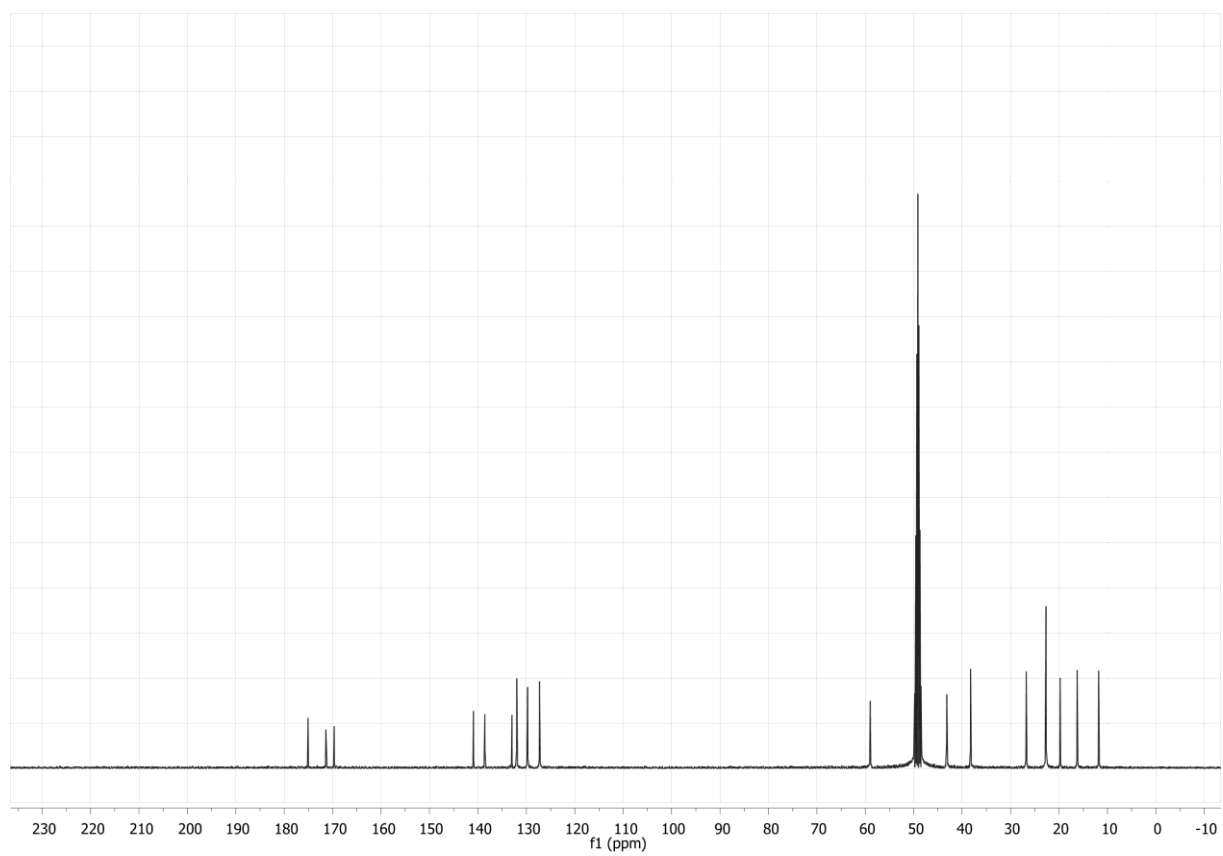
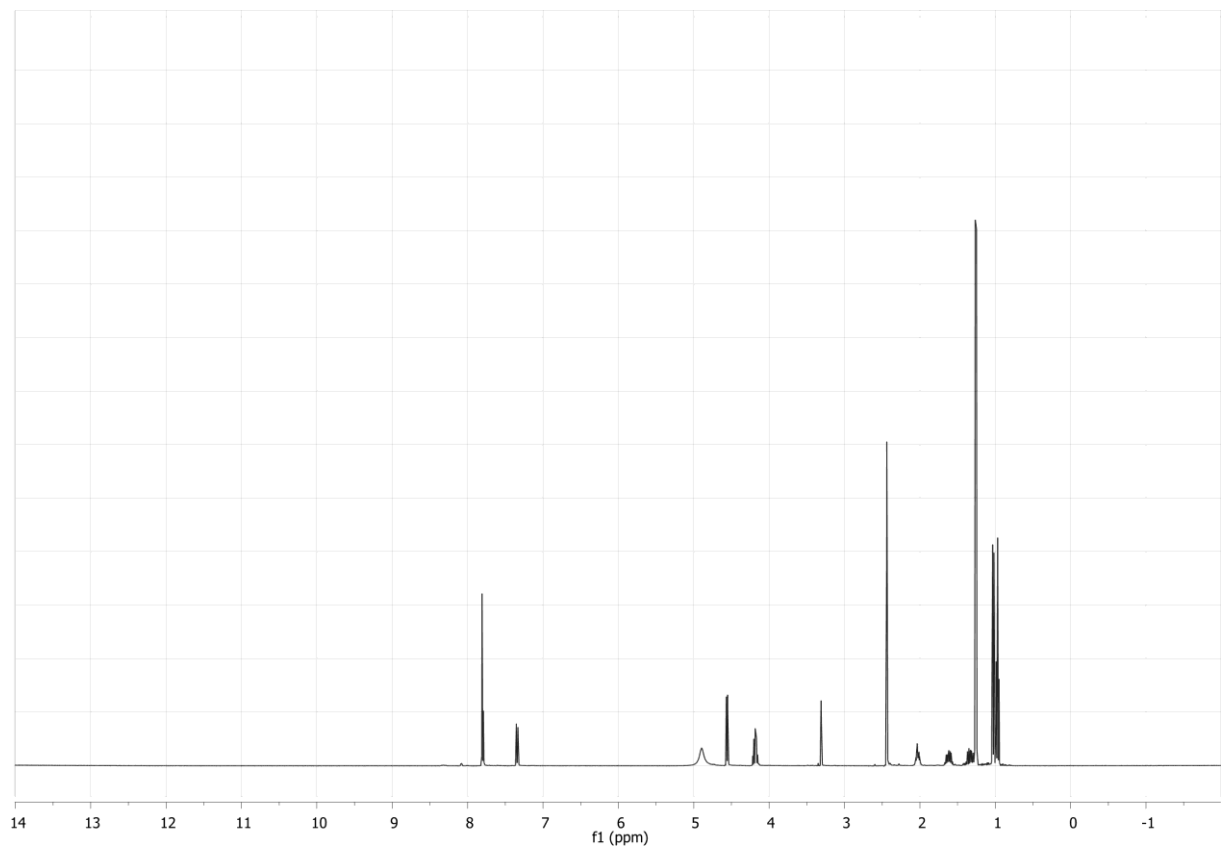




**(2S,3R)-2-(3-(Isopropylcarbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (57).**

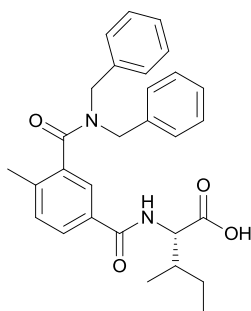


Yield: 52 mg (75%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.97 (t,  $J = 7.4$  Hz, 3H), 1.03 (d,  $J = 6.9$  Hz, 3H), 1.26 (d,  $J = 6.6$  Hz, 6H), 1.28 – 1.39 (m, 1H), 1.62 (ddq,  $J = 14.9$  Hz, 7.5 Hz, 4.3 Hz, 1H), 2.03 (ddt,  $J = 6.7$  Hz, 5.5 Hz, 3.2 Hz, 1H), 2.44 (s, 3H), 4.18 (hept,  $J = 6.6$  Hz, 1H), 4.56 (d,  $J = 6.5$  Hz, 1H), 7.33 – 7.36 (m, 1H), 7.79 (d,  $J = 2.0$  Hz, 1H), 7.81 (d,  $J = 1.6$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 19.8, 22.7, 26.8, 38.3, 43.2, 59.0, 127.3, 129.7, 132.0, 133.0, 138.6, 140.9, 169.7, 171.4, 175.1; HRMS (M+H) calcd. for  $\text{C}_{18}\text{H}_{26}\text{N}_2\text{O}_4$  335.1971; found: 335.1969.





**(2S,3R)-2-(3-(Dibenzylcarbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (58).**



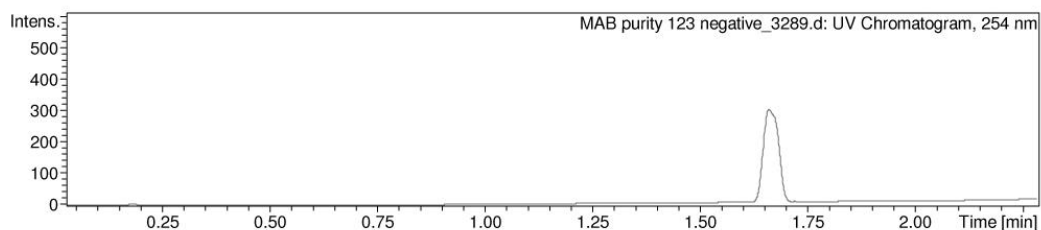
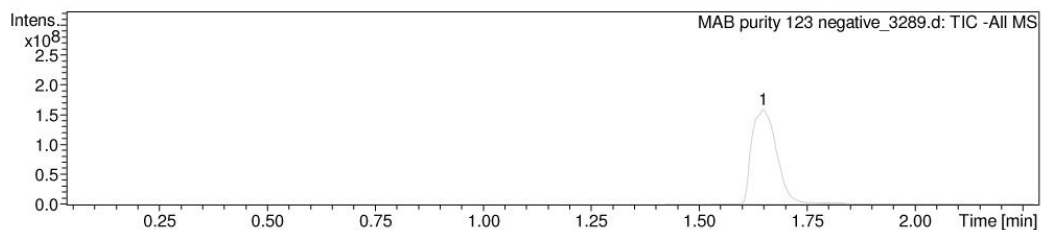
Yield: 67 mg (82%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.95 (t,  $J$  = 7.4 Hz, 3H), 0.99 (d,  $J$  = 6.8 Hz, 3H), 1.23 – 1.35 (m, 1H), 1.58 (ddt,  $J$  = 11.7 Hz, 7.5 Hz, 4.3 Hz, 1H), 1.96 – 2.05 (m, 1H), 2.29 (s, 3H), 4.28 (s, 2H), 4.54 (d,  $J$  = 6.4 Hz, 1H), 4.92 (s br, 2H), 7.04 – 7.07 (m, 2H), 7.20 – 7.44 (m, 9H), 7.78 (s, 1H), 7.81 (dd,  $J$  = 7.9 Hz, 1.9 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 19.4, 26.7, 38.3, 48.8, 52.8, 59.0, 126.4, 128.7, 129.0, 129.1, 129.5, 129.8, 129.95, 130.02, 132.1, 133.3, 137.2, 137.5, 138.1, 134.0, 169.3, 173.5, 175.0; HRMS (M+H) calcd. for  $\text{C}_{29}\text{H}_{32}\text{N}_2\text{O}_4$  473.2440; found: 473.2445;  $[\alpha]_{589}^{25} = +5.5$ .

## LC-MS Analysis Report

### General Information

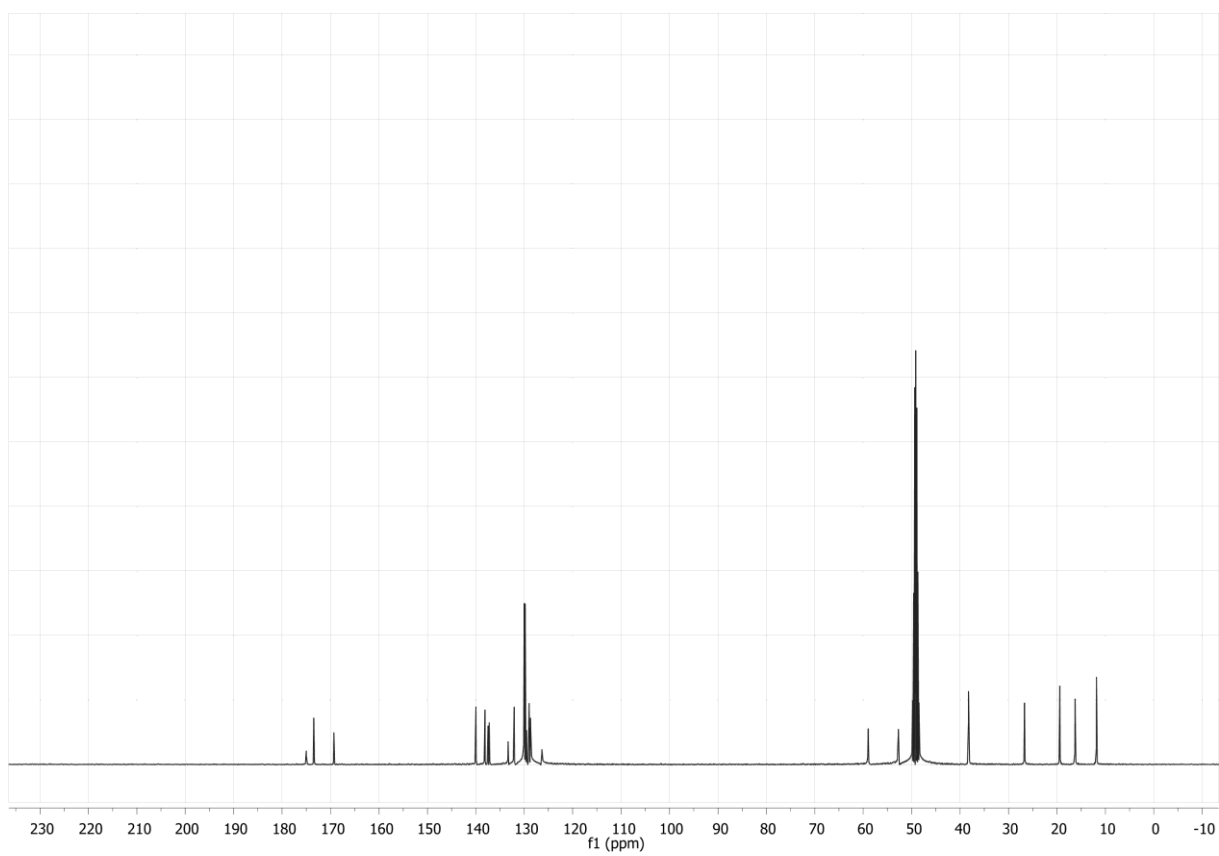
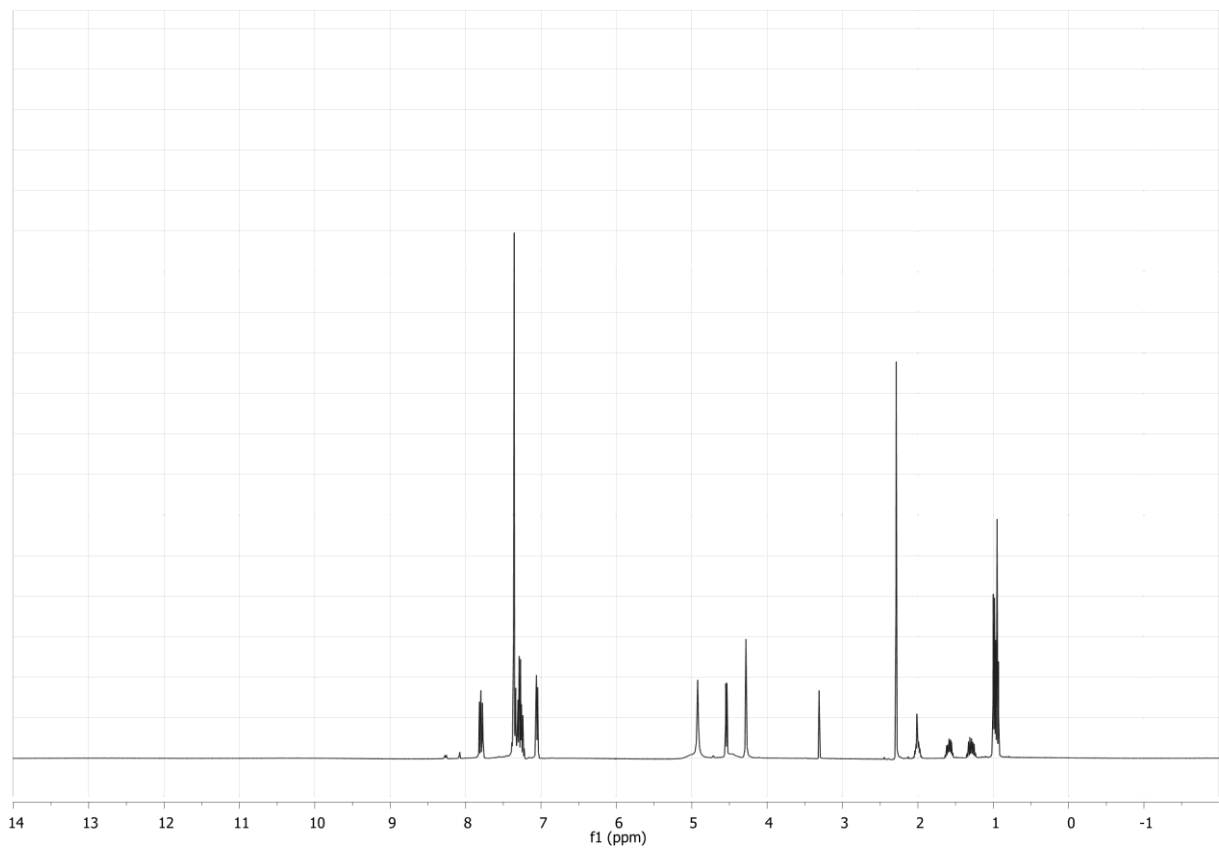
Sample ID: MAB purity 123 negative  
Date & Time: 10/7/2010 12:25:29 PM  
Data File: B:\Malte\Results\Purity\_check\MAB purity 123 negative\_3289.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram

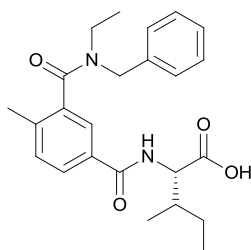


### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 1.6 min	100.0



**(2S,3R)-2-(3-(Benzyl(ethyl)carbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (59).**



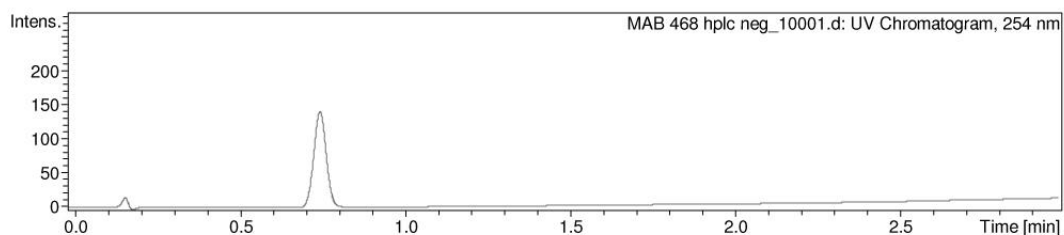
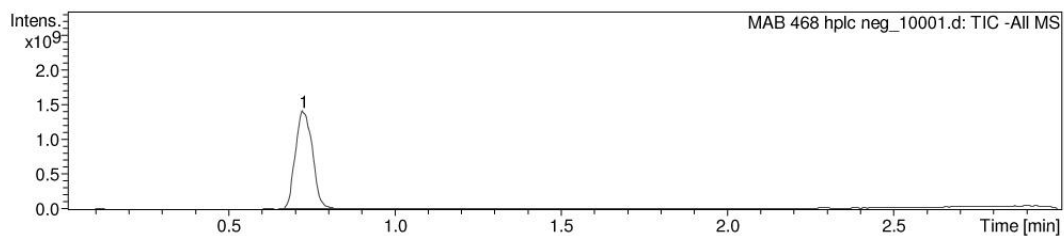
Yield: 48 mg (71%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J = 7.4$  Hz, 3H), 0.99 (d,  $J = 6.9$  Hz, 3H), 1.01 – 1.26 (m, 3H), 1.26 – 1.39 (m, 1H), 1.54 – 1.67 (m, 1H), 1.95 – 2.07 (m, 1H), 4.40 (s br, 1H), 4.55 (d,  $J = 6.5$  Hz, 1H), 2.34 (s, 3H), 3.07 – 3.40 (m, 2H), 4.69 – 4.96 (m, 1H), 7.11 – 7.16 (m, 1H), 7.25 – 7.46 (m, 5H), 7.69 – 7.77 (m, 1H), 7.82 (ddd,  $J = 13.8$  Hz, 8.0 Hz, 1.9 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 12.7, 13.8, 16.3, 19.3, 26.8, 38.4, 41.4, 44.3, 48.3, 53.2, 59.3, 126.1, 128.6, 128.9, 129.0, 129.3, 129.5, 129.9, 130.0, 132.0, 133.5, 137.9, 138.8, 139.7, 169.3, 173.0, 175.3; HRMS (M+H) calcd. for  $\text{C}_{24}\text{H}_{30}\text{N}_2\text{O}_4$  411.2278, found: 411.2289;  $[\alpha]_{589}^{25} = +6.4$ .

## LC-MS Analysis Report

### General Information

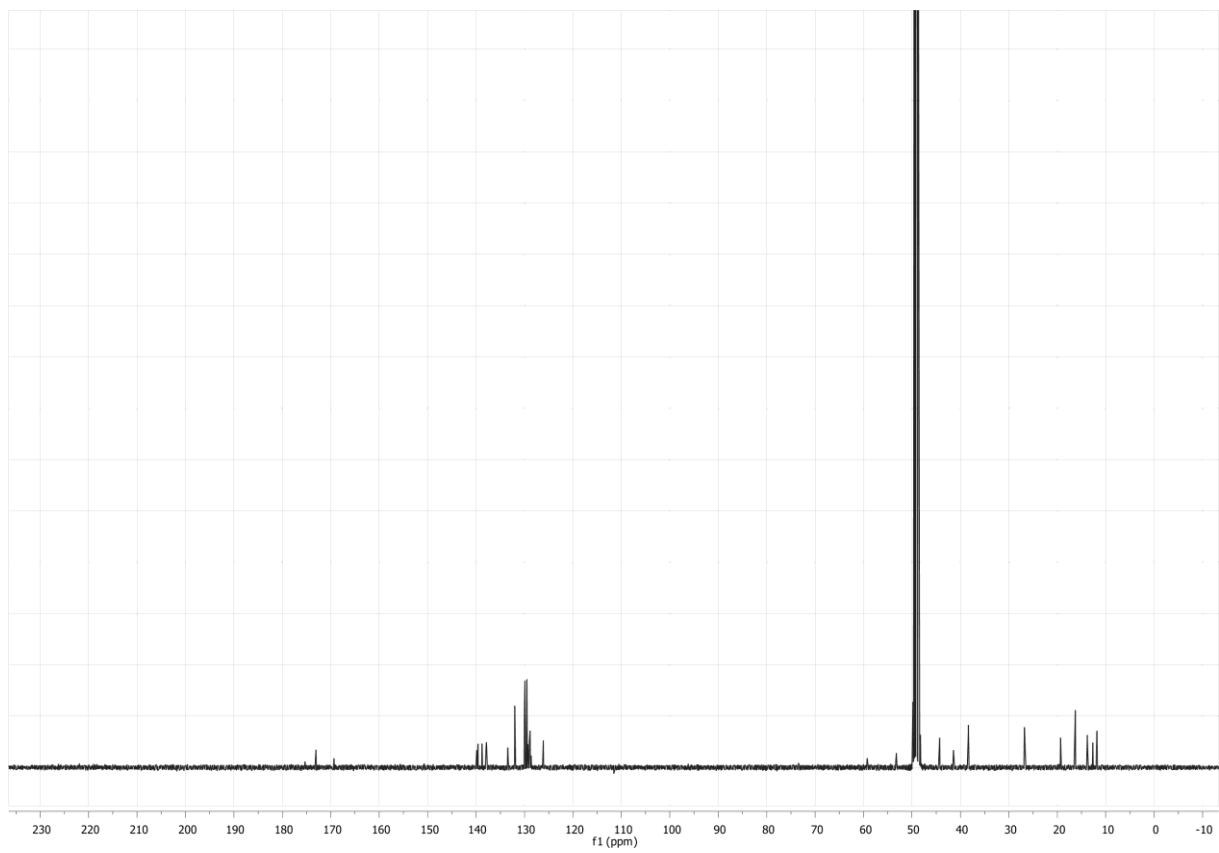
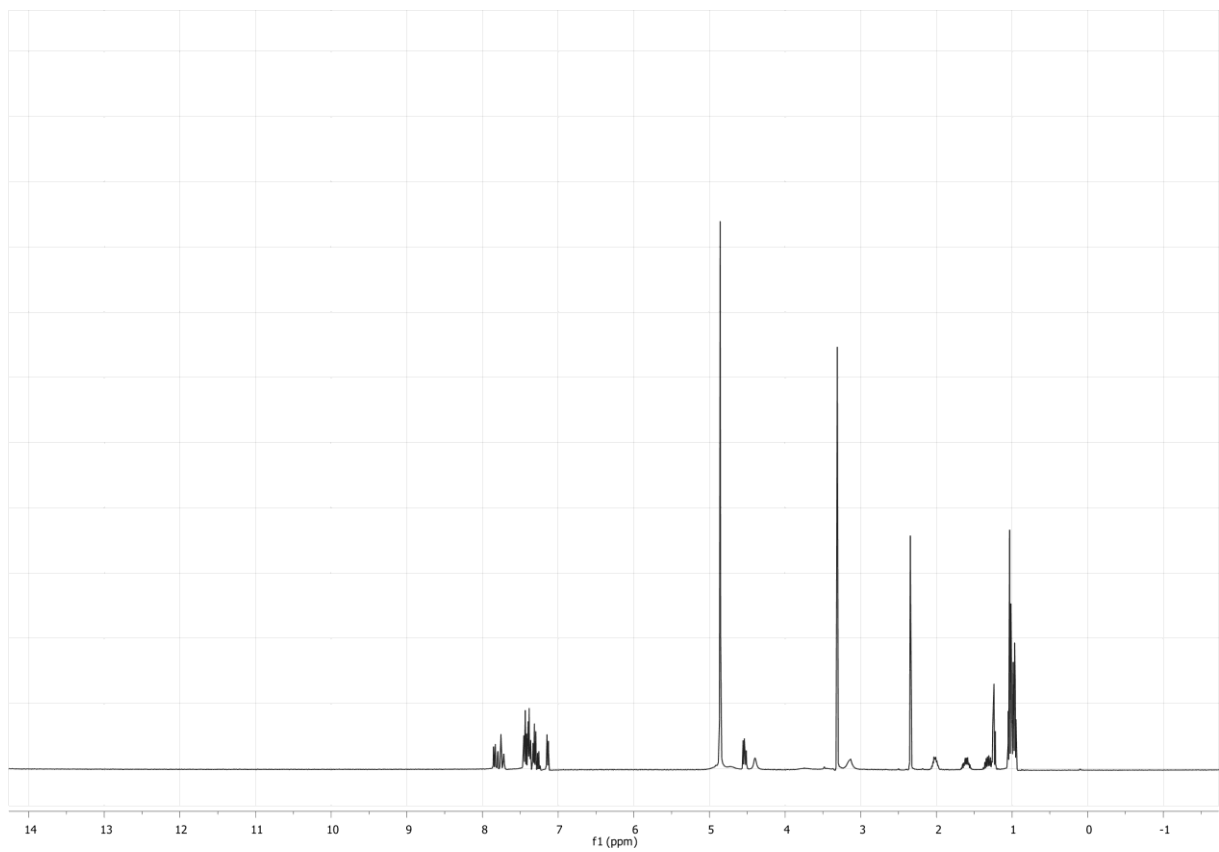
Sample ID: MAB 468 hplc neg  
Date & Time: 5/18/2011 10:24:25 AM  
Data File: B:\Malte\Results\benzamides\Acids purity\MAB 468 hplc neg\_10001.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram

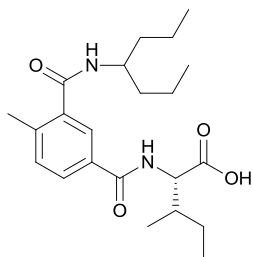


### Compound List (Area Frac. % of UV 254 nm)

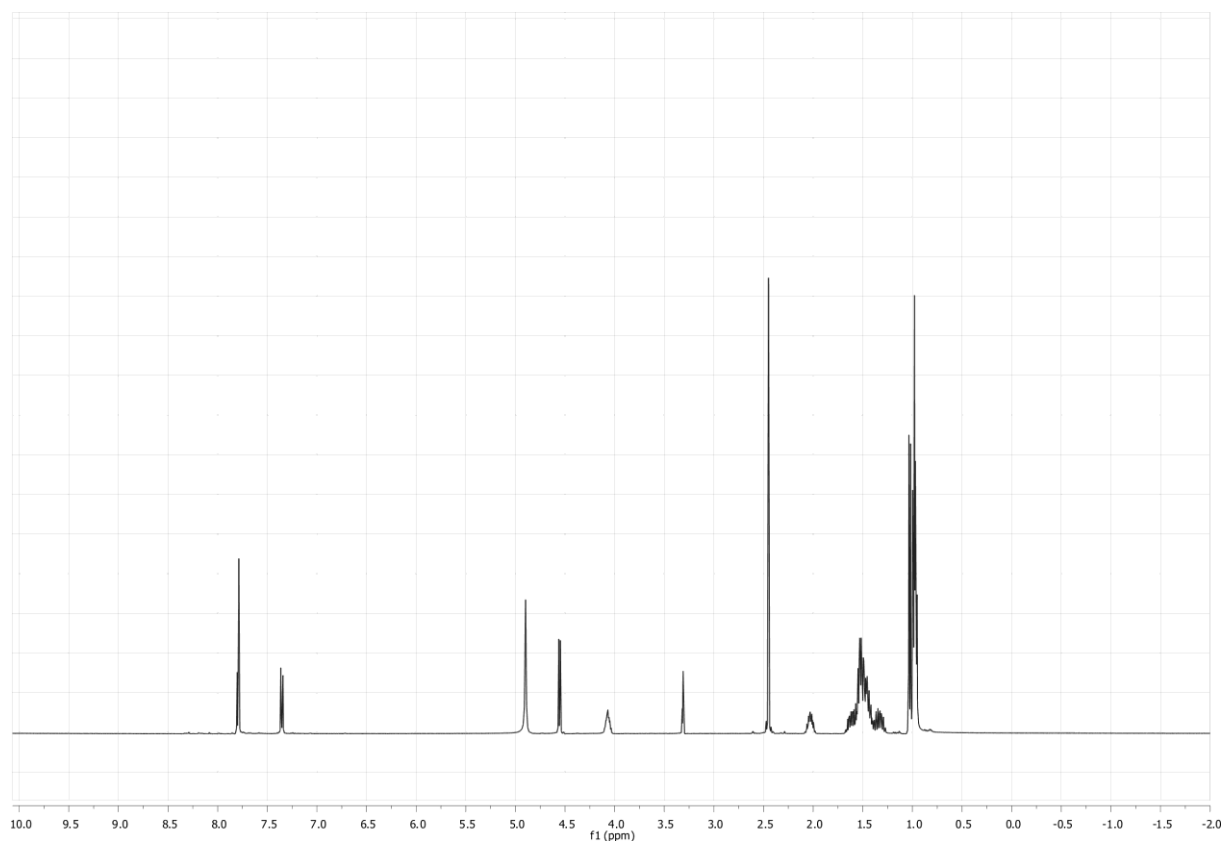
Cmpd. Label	Area Frac. %
Cmpd 1, 0.73 min	100.0

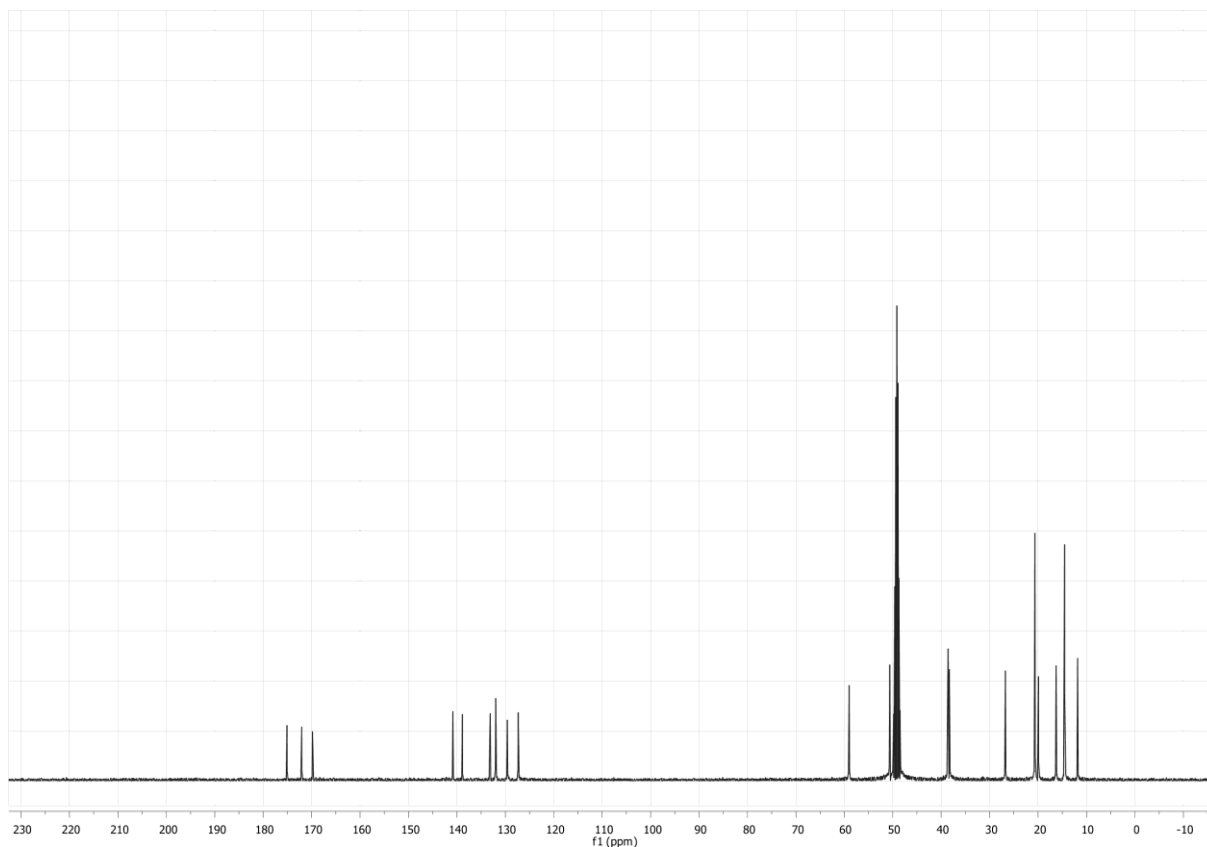


**(2*S*,3*R*)-2-(3-(Heptan-4-ylcarbamoyl)-4-methylbenzamido)-3-methylpentanoic acid (60).**

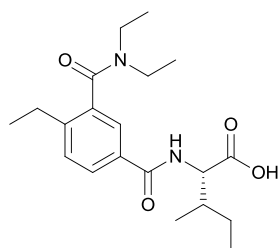


Yield: 54 mg (76%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 0.97 (t,  $J = 7.4$  Hz, 3H), 0.98 (t,  $J = 7.1$  Hz, 6H), 1.03 (d,  $J = 6.9$  Hz, 3H), 1.27 – 1.38 (m, 1H), 1.40 – 1.49 (m, 4H), 1.50 – 1.56 (m, 4H), 1.62 (ddt,  $J = 9.4$  Hz, 7.6 Hz, 3.8 Hz, 1H), 2.03 (ddt,  $J = 8.9$  Hz, 6.7 Hz, 4.3 Hz, 1H), 2.45 (s, 3H), 4.03 – 4.11 (m, 1H), 4.56 (d,  $J = 6.4$  Hz, 1H), 7.33 – 7.37 (m, 1H), 7.78 – 7.79 (m, 1H), 7.79 – 7.81 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ )  $\delta$ : 11.8, 14.5, 16.3, 19.9, 20.6, 26.7, 38.3, 38.55, 38.57, 50.6, 59.0, 127.3, 129.6, 132.0, 133.1, 138.9, 140.8, 169.8, 172.1, 175.1; HRMS ( $\text{M}+\text{H}$ ) calcd. for  $\text{C}_{22}\text{H}_{34}\text{N}_2\text{O}_4$  391.2591, found: 391.2600;  $[\alpha]_{589}^{25} = +11.5$ .





**(2S,3R)-2-(3-(Diethylcarbamoyl)-4-ethylbenzamido)-3-methylpentanoic acid (61).**



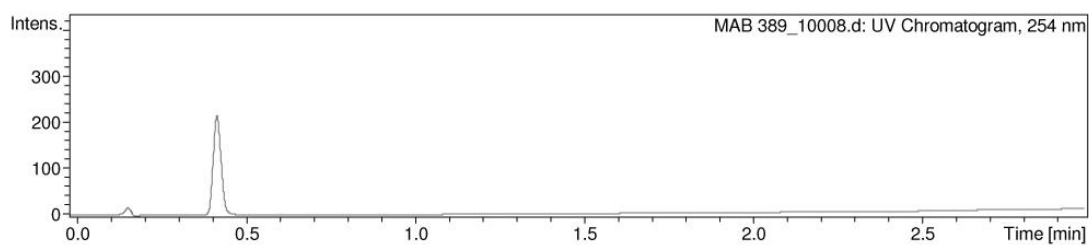
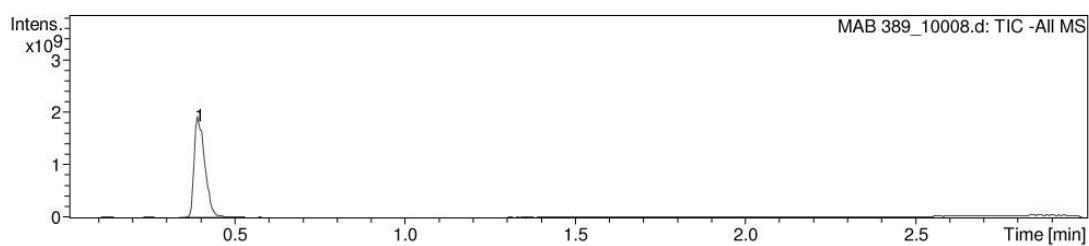
Yield: 31 mg (58%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J = 7.4$  Hz, 3H), 1.02 (d,  $J = 6.9$  Hz, 3H), 1.08 (t,  $J = 7.1$  Hz, 3H), 1.25 (t,  $J = 7.6$  Hz, 3H), 1.28 (t,  $J = 7.1$  Hz, 3H), 1.31 – 1.39 (m, 1H), 1.62 (ddq,  $J = 14.9$  Hz, 7.5 Hz, 4.3 Hz, 1H), 1.97 – 2.08 (m, 1H), 2.61 – 2.72 (m, 2H), 3.09 – 3.27 (m, 2H), 3.39 – 3.56 (m, 1H), 3.71 (s br, 1H), 4.56 (d,  $J = 6.5$  Hz, 1H), 7.45 (d,  $J = 8.1$  Hz, 1H), 7.69 (s, 1H), 7.88 (dd,  $J = 8.1$  Hz, 2.0 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 13.1, 14.3, 15.4, 16.3, 26.8, 27.1, 38.3, 40.6, 44.8, 59.1, 126.1, 129.5, 130.4, 133.3, 137.7, 145.6, 169.4, 172.4, 175.0; HRMS (M+H) calcd. for  $\text{C}_{20}\text{H}_{30}\text{N}_2\text{O}_4$  363.2278, found: 363.2278;  $[\alpha]_{589}^{25} = +9.7$ .

# LC-MS Analysis Report

## General Information

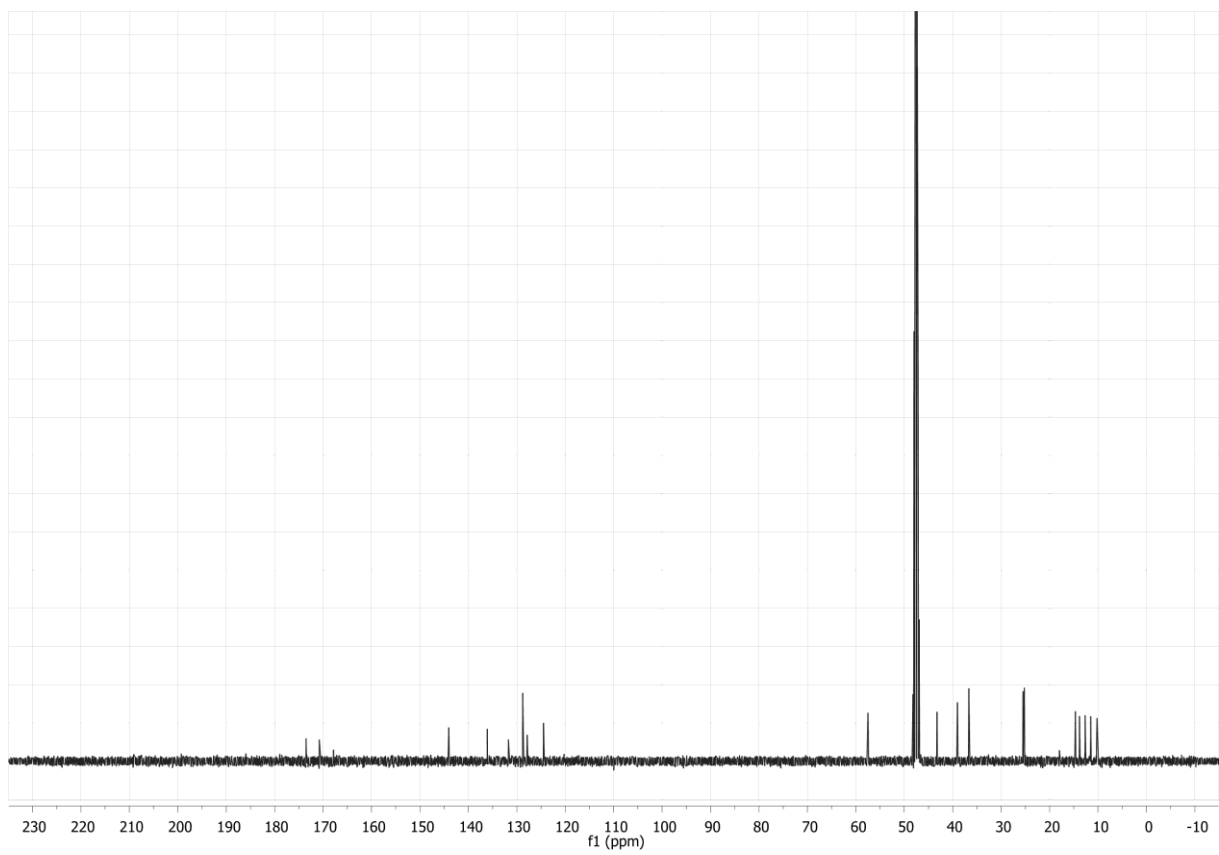
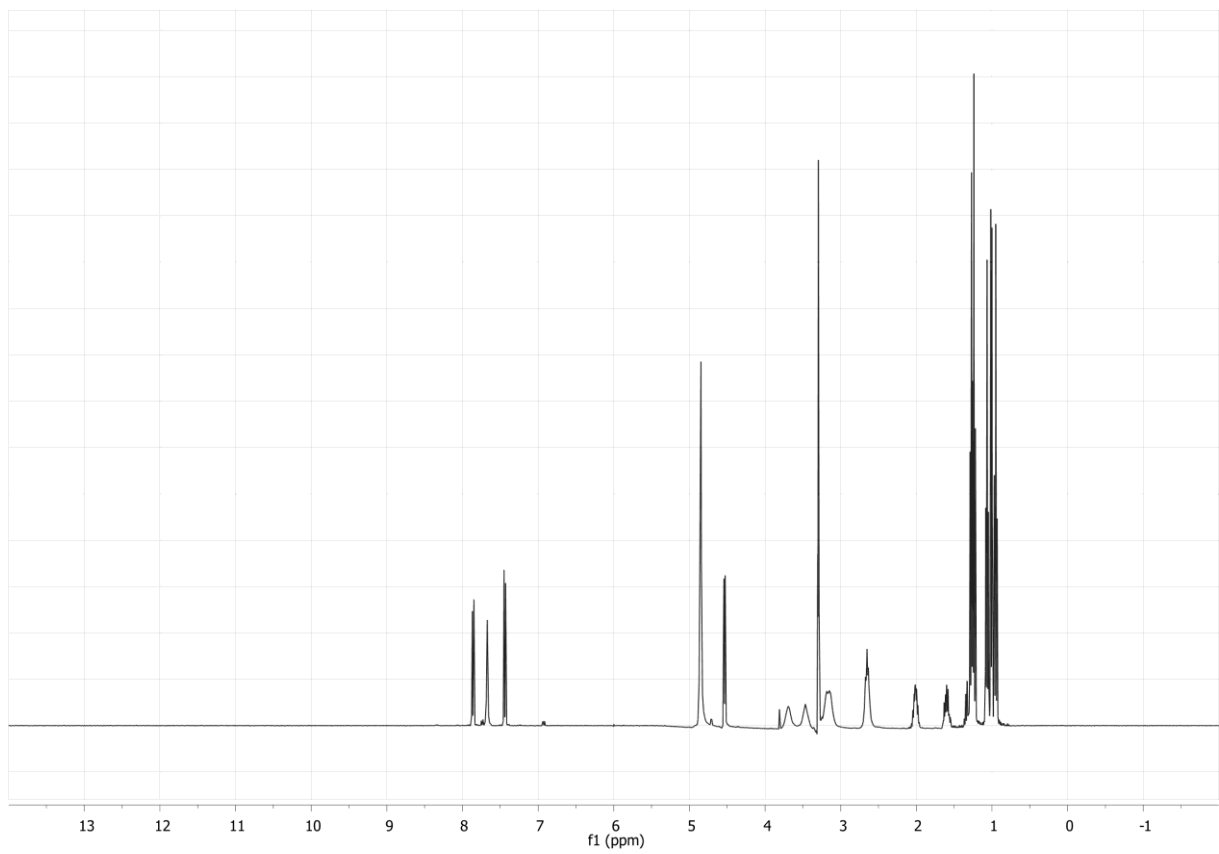
Sample ID: MAB 389  
Date & Time: 5/18/2011 11:54:30 AM  
Data File: B:\Malte\Results\benzamides\Acids purity\MAB 389\_10008.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

## Chromatogram



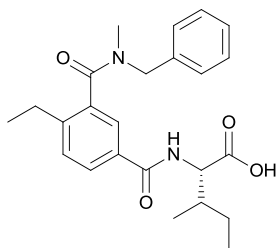
## Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 0.40 min	100.0





**(2S,3R)-2-(3-(Benzyl(methyl)carbamoyl)-4-ethylbenzamido)-3-methylpentanoic acid (62).**



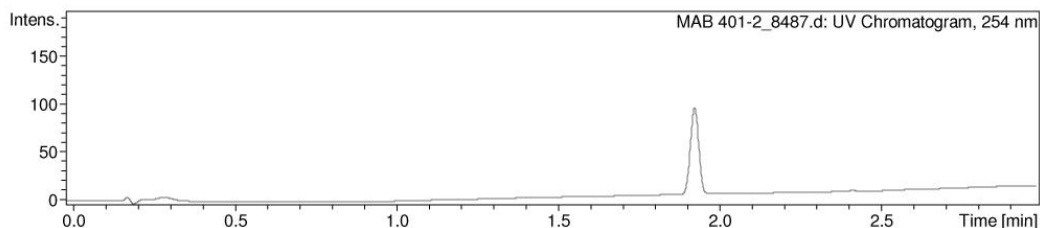
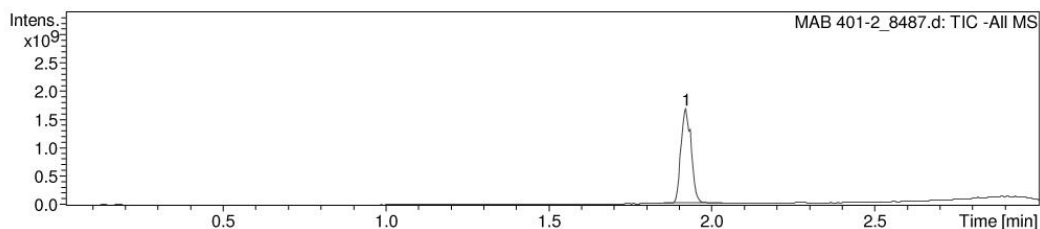
Yield: 45 mg (49%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J = 7.4$  Hz, 3H), 1.02 (d,  $J = 6.9$  Hz, 3H), 1.18 – 1.28 (m, 3H), 1.28 – 1.39 (m, 1H), 1.54 – 1.67 (m, 1H), 1.97 – 2.08 (m, 1H), 2.60 – 2.73 (m, 2H), 2.76 (s, 2H), 3.08 (s, 1H), 4.38 (s, 1H), 4.55 (d,  $J = 6.5$  Hz, 1H), 4.60 – 4.80 (m, 1H), 7.11 – 7.16 (m, 1H), 7.23 – 7.36 (m, 2H), 7.37 – 7.47 (m, 3H), 7.72 (t,  $J = 7.5$  Hz, 1H), 7.87 (dt,  $J = 8.1$  Hz, 1.8 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 15.5, 16.3, 26.8, 27.1, 33.5, 37.0, 38.2, 51.5, 56.0, 59.1, 126.4, 128.5, 129.0, 129.6, 129.8, 130.0, 130.1, 130.5, 133.6, 137.4, 138.2, 145.7, 169.6, 173.0, 175.1; HRMS ( $\text{M}+\text{H}$ ) calcd. for  $\text{C}_{24}\text{H}_{30}\text{N}_2\text{O}_4$  411.2284; found: 411.2276;  $[\alpha]_{589}^{25} = +4.2$ .

## LC-MS Analysis Report

### General Information

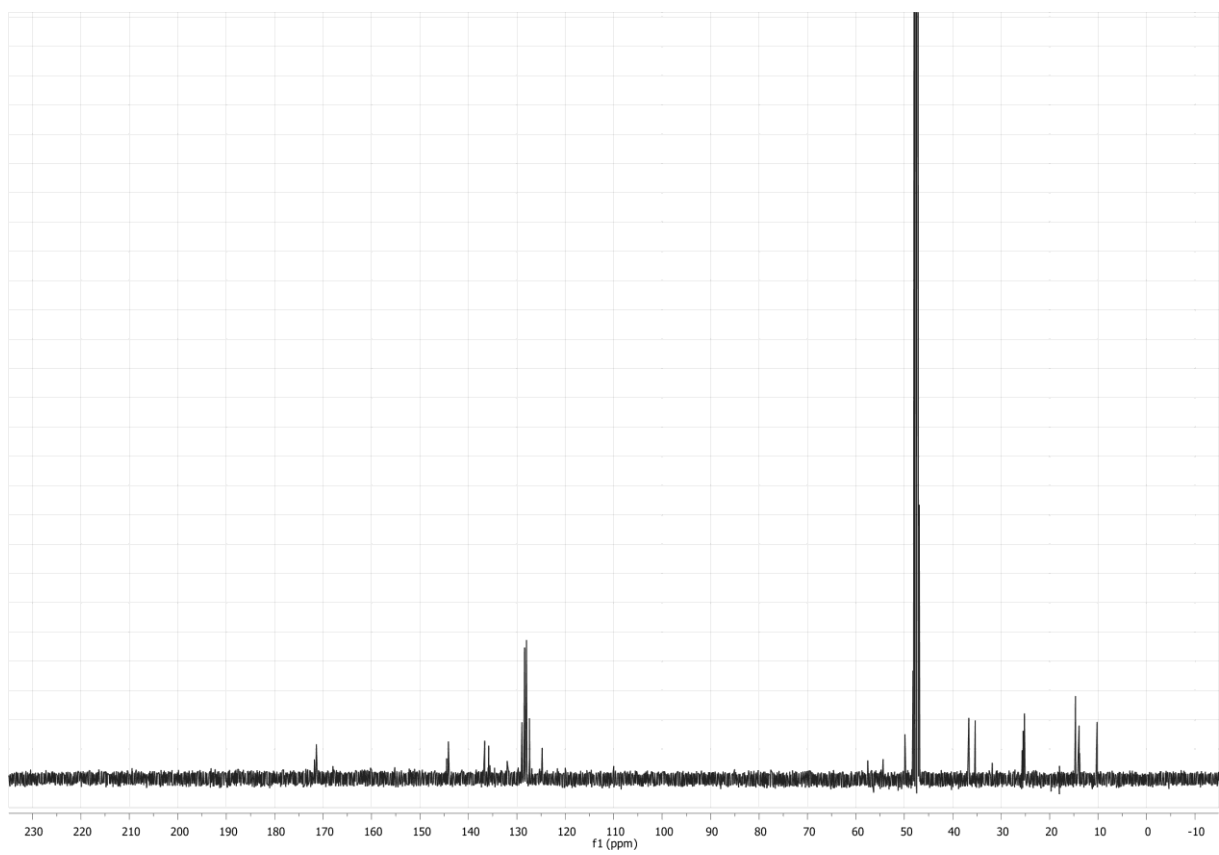
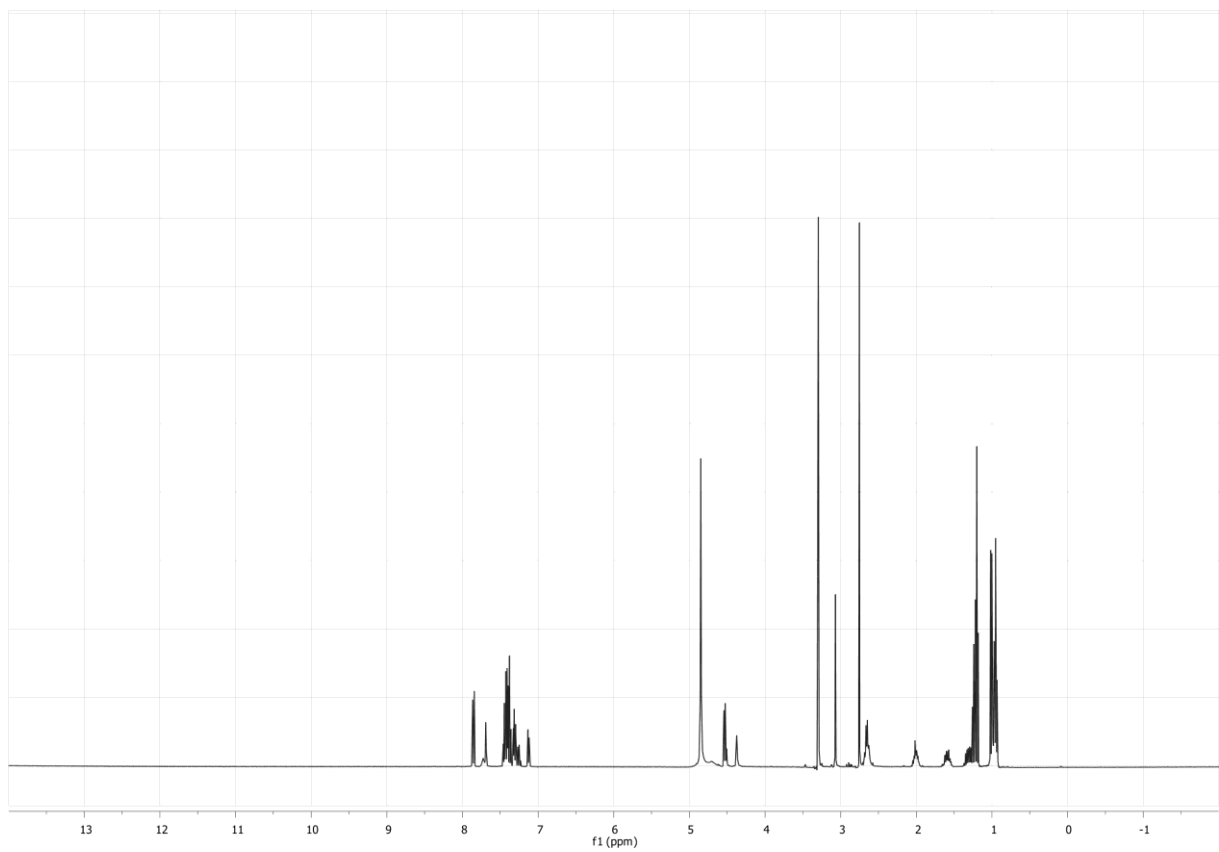
Sample ID: MAB 401-2  
Date & Time: 4/1/2011 5:49:18 PM  
Data File: B:\Malte\Results\benzamides\Acids purity\MAB 401-2\_8487.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram

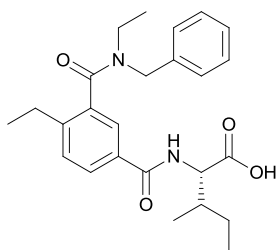


### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 1.92 min	100.0



**(2S,3R)-2-(3-(Benzyl(ethyl)carbamoyl)-4-ethylbenzamido)-3-methylpentanoic acid (63).**



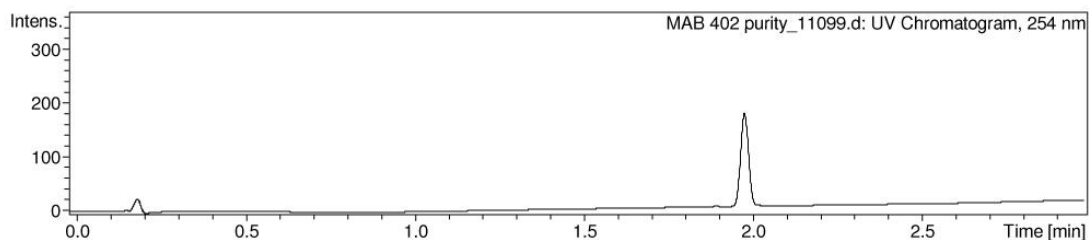
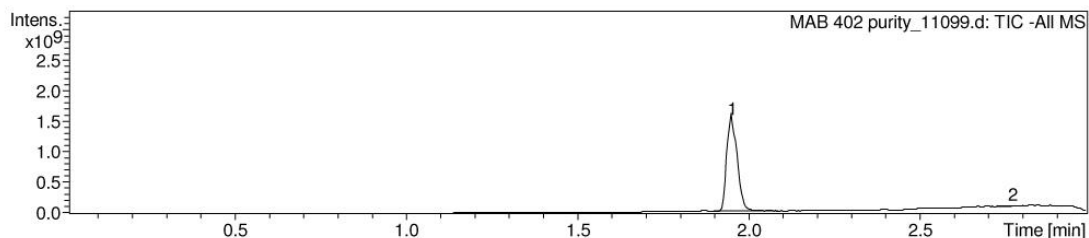
Yield: 46 mg (52%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.95 (t,  $J = 7.4$  Hz, 3H), 1.02 (t,  $J = 7.1$  Hz, 3H), 1.20 (t,  $J = 7.6$  Hz, 3H), 1.24 (t,  $J = 7.6$  Hz, 3H), 1.27 – 1.38 (m, 1H), 1.51 – 1.66 (m, 1H), 1.93 – 2.08 (m, 1H), 2.59 – 2.70 (m, 2H), 3.04 – 3.17 (m, 1H), 3.61 – 3.86 (m, 1H), 4.36 (d,  $J = 4.6$  Hz, 1H), 4.54 (d,  $J = 6.5$  Hz, 1H), 4.57 – 5.03 (m, 1H), 7.10 – 7.16 (m, 1H), 7.21 – 7.33 (m, 2H), 7.34 – 7.45 (m, 3H), 7.67 – 7.75 (m, 1H), 7.86 (dd,  $J = 8.1$  Hz, 2.0 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 12.6, 15.5, 16.3, 26.8, 27.2, 38.3, 41.3, 44.4, 48.1, 53.4, 59.1, 126.2, 128.6, 128.9, 129.0, 129.5, 129.9, 130.0, 130.5, 133.4, 137.4, 138.8, 145.7, 169.3, 173.0, 175.1; HRMS (M+H) calcd. for  $\text{C}_{25}\text{H}_{32}\text{N}_2\text{O}_4$  425.2440; found: 425.2442;  $[\alpha]_{589}^{25} = +5.7$ .

## LC-MS Analysis Report

### General Information

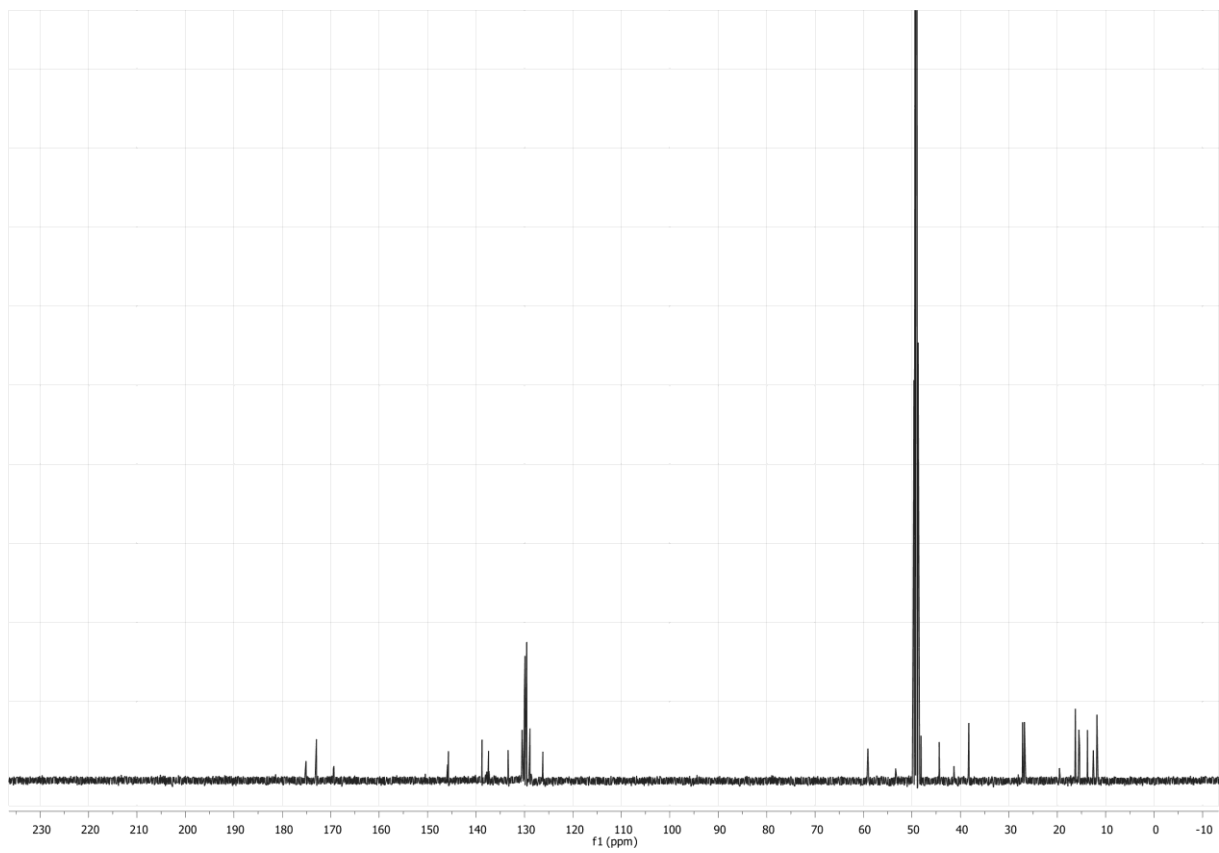
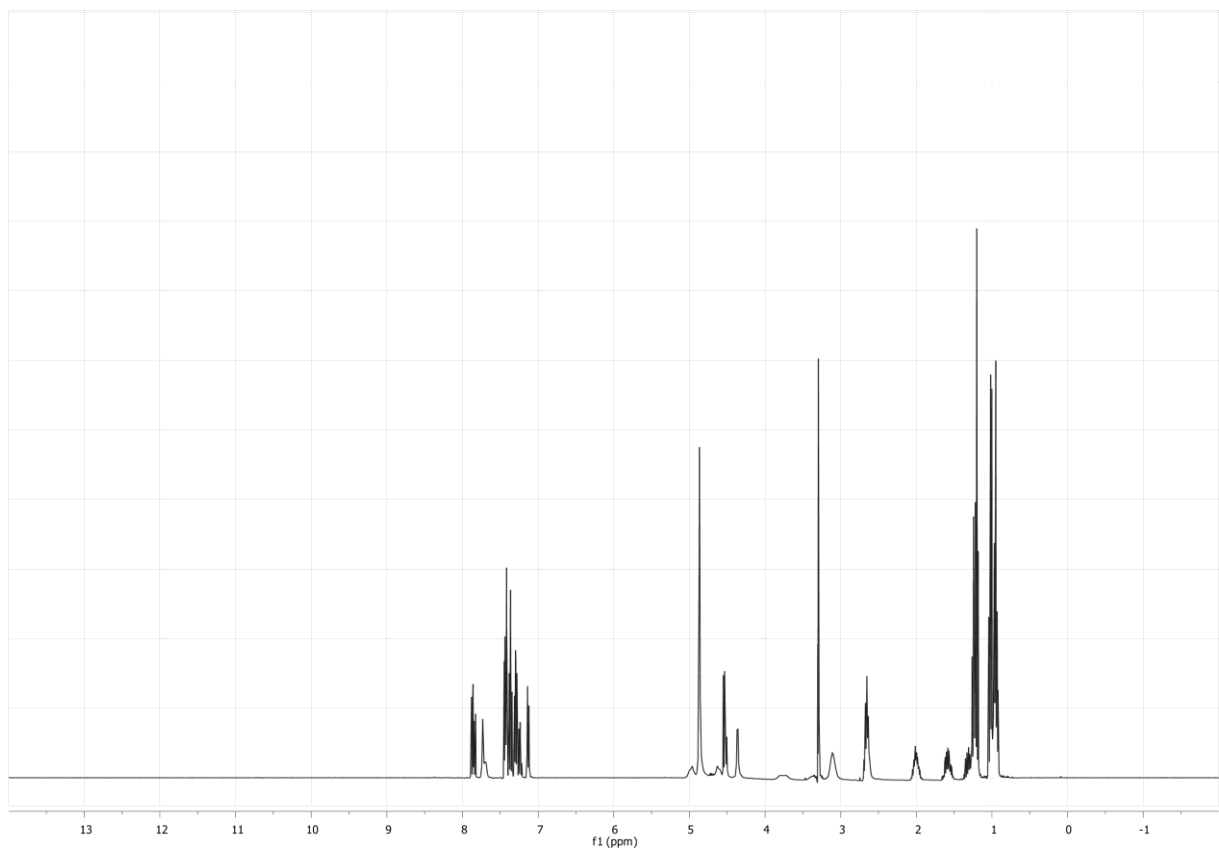
Sample ID: MAB 402 purity  
Date & Time: 8/8/2011 4:03:25 PM  
Data File: B:\Malte\Results\benzamides\Acids purity\MAB 402 purity\_11099.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram

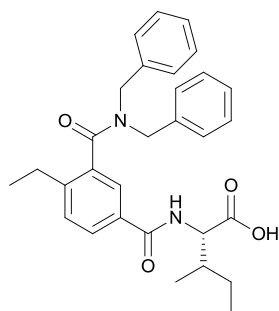


### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 1.95 min	98.8
Cmpd 2, 2.77 min	1.2



**(2S,3R)-2-(3-Dibenzylcarbamoyl)-4-ethylbenzamido)-3-methylpentanoic acid (64).**



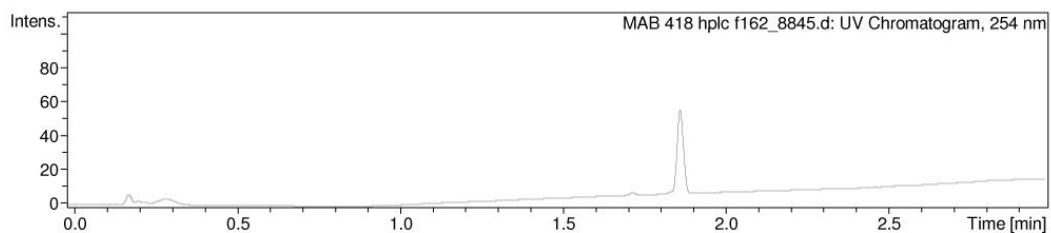
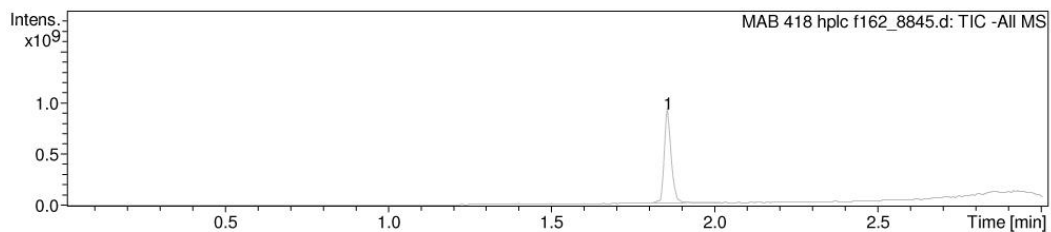
Yield: 138 mg (46%, white semi-solid);  $^1\text{H NMR}$  ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.96 (t,  $J = 7.4$  Hz, 3H), 0.98 – 1.03 (m, 3H), 1.20 (t,  $J = 7.6$  Hz, 3H), 1.23 – 1.36 (m, 1H), 1.53 – 1.65 (m, 1H), 1.95 – 2.06 (m, 1H), 2.63 (q,  $J = 7.4$  Hz, 2H), 4.29 (s, 2H), 4.33 – 4.49 (m, 1H), 4.52 (d,  $J = 6.3$  Hz, 1H), 4.90 – 5.22 (m, 1H), 7.10 (d,  $J = 7.2$  Hz, 2H), 7.22 – 7.41 (m, 8H), 7.43 (d,  $J = 8.1$  Hz, 1H), 7.76 (s br, 1H), 7.85 (dd,  $J = 8.1$  Hz, 1.9 Hz, 1H);  $^{13}\text{C NMR}$  ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.9, 15.5, 16.3, 26.7, 27.2, 38.5, 48.7, 53.0, 59.3, 126.2, 128.7, 129.05, 129.09, 129.7, 129.9, 130.0, 130.1, 130.6, 133.5, 137.0, 137.2, 138.2, 146.0, 169.2, 173.5, 175.5; HRMS (M+H) calcd. for  $\text{C}_{30}\text{H}_{34}\text{N}_2\text{O}_4$  487.2591, found: 487.2606;  $[\alpha]_{589}^{25} = +5.5$ .

## LC-MS Analysis Report

### General Information

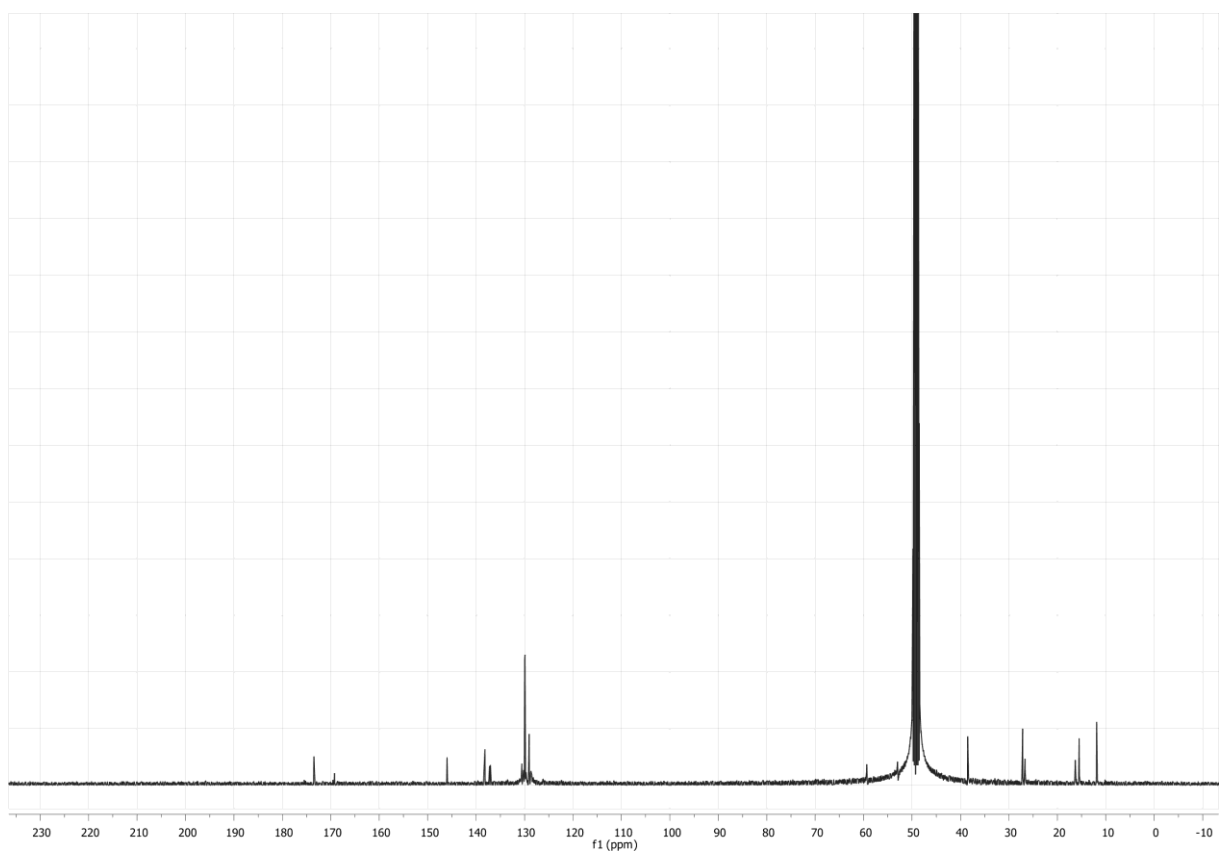
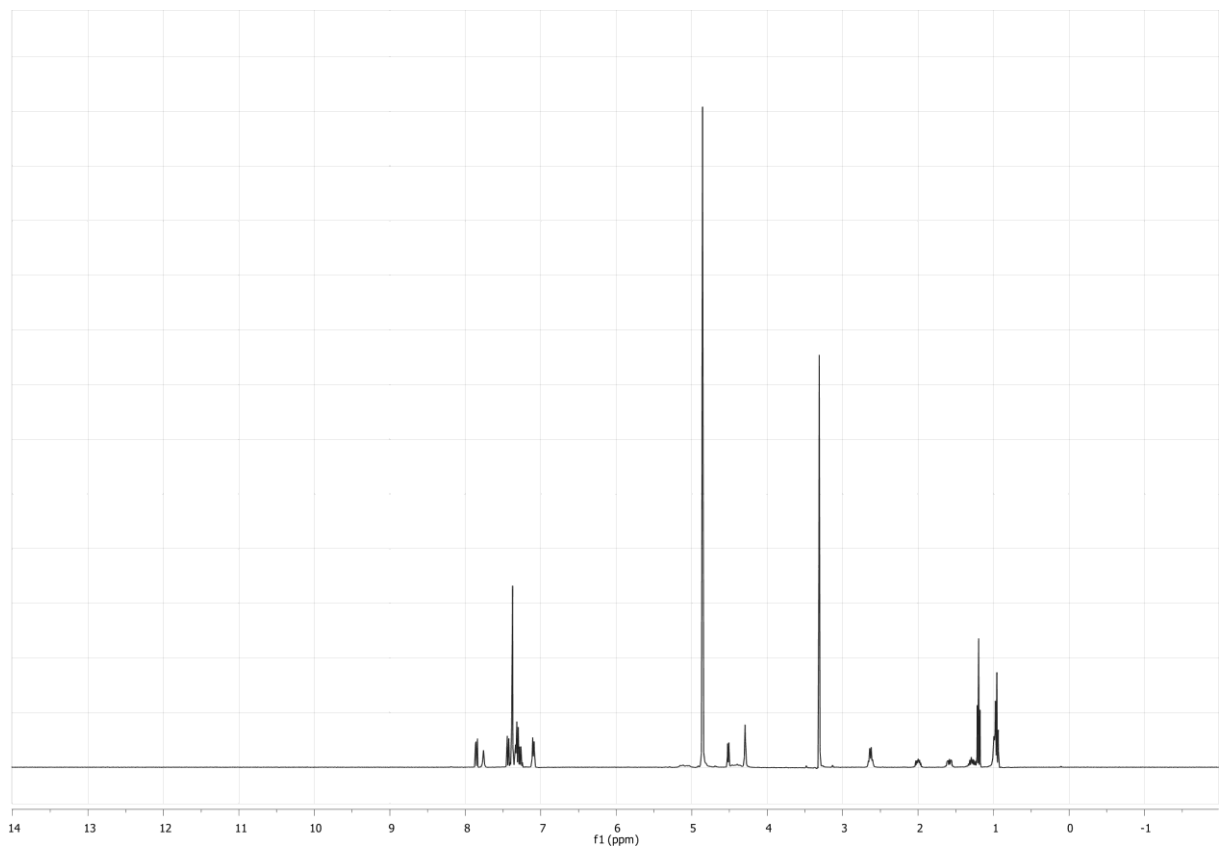
Sample ID: MAB 418 hplc f162  
Date & Time: 4/11/2011 4:00:16 PM  
Data File: B:\Malte\Results\benzamides\Acids purity\MAB 418 hplc f162\_8845.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram

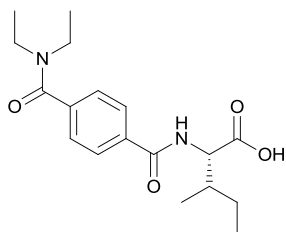


### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 1.86 min	100.0



**(2S,3R)-2-(4-(Diethylcarbamoyl)benzamido)-3-methylpentanoic acid (68).**



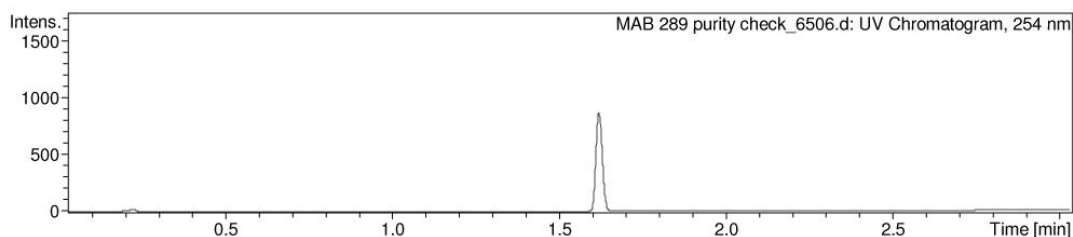
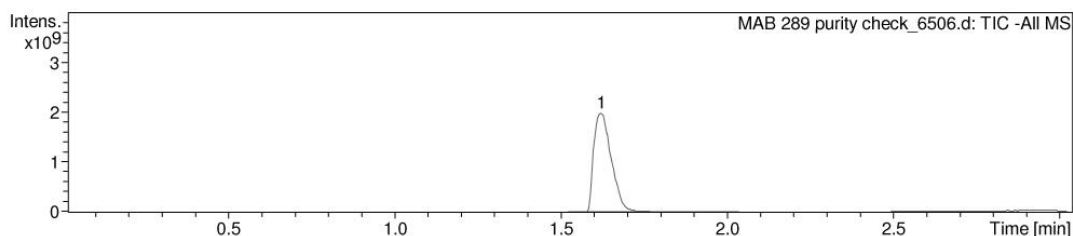
Yield: 70 mg (74%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.97 (t,  $J = 7.4$  Hz, 3H), 1.04 (d,  $J = 6.9$  Hz, 3H), 1.12 (t,  $J = 7.0$  Hz, 3H), 1.26 (t,  $J = 7.0$  Hz, 3H), 1.30 – 1.41 (m, 1H), 1.63 (ddq,  $J = 14.9$  Hz, 7.5 Hz, 4.3 Hz, 1H), 1.98 – 2.11 (m, 1H), 3.27 (q,  $J = 7.0$  Hz, 2H), 3.56 (q,  $J = 6.9$  Hz, 2H), 4.58 (d,  $J = 6.4$  Hz, 1H), 7.44 – 7.49 (m, 2H), 7.90 – 7.94 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.9, 13.2, 14.5, 16.3, 26.7, 38.3, 41.0, 45.0, 59.0, 127.5, 129.1, 136.6, 141.2, 169.7, 172.7, 174.9; HRMS (M+H) calcd. for  $\text{C}_{18}\text{H}_{26}\text{N}_2\text{O}_4$  335.1965, found: 335.1967;  $[\alpha]_{589}^{25} = +15.4$ .

## LC-MS Analysis Report

### General Information

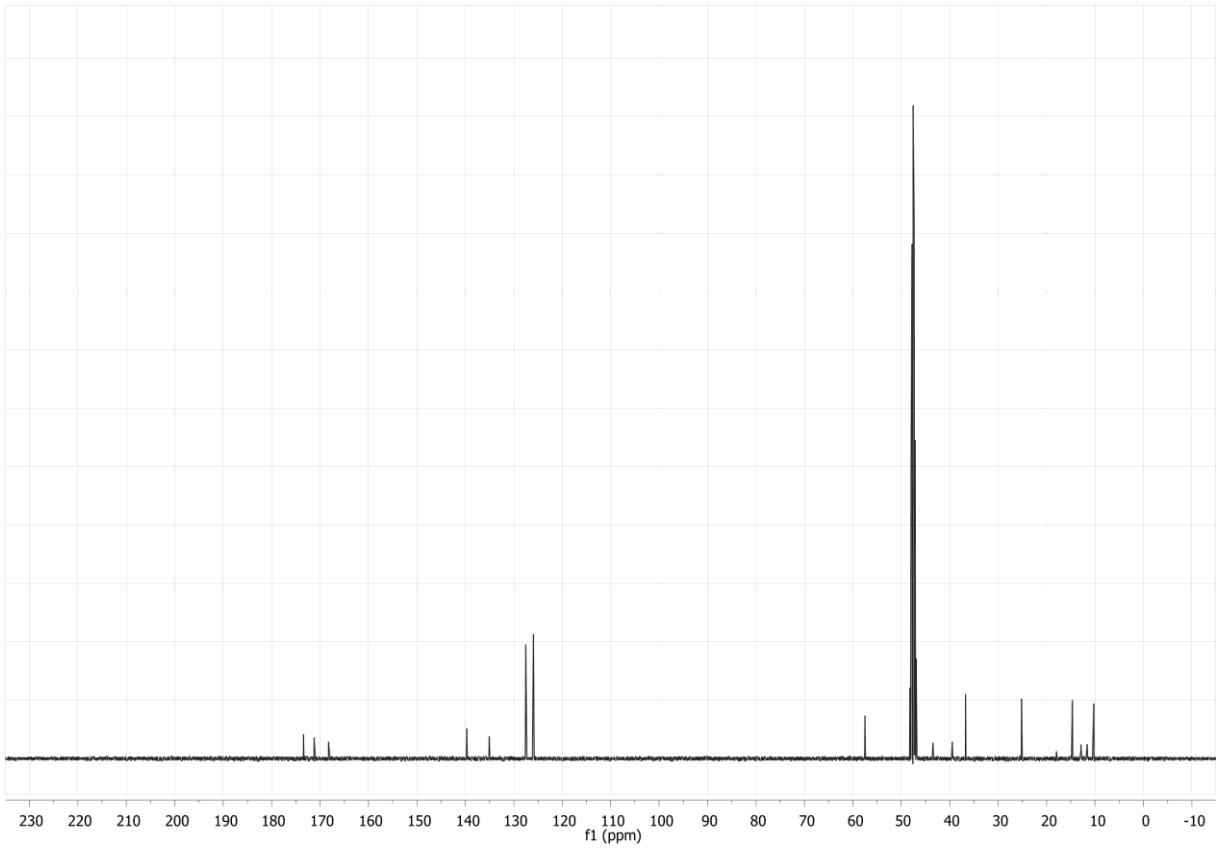
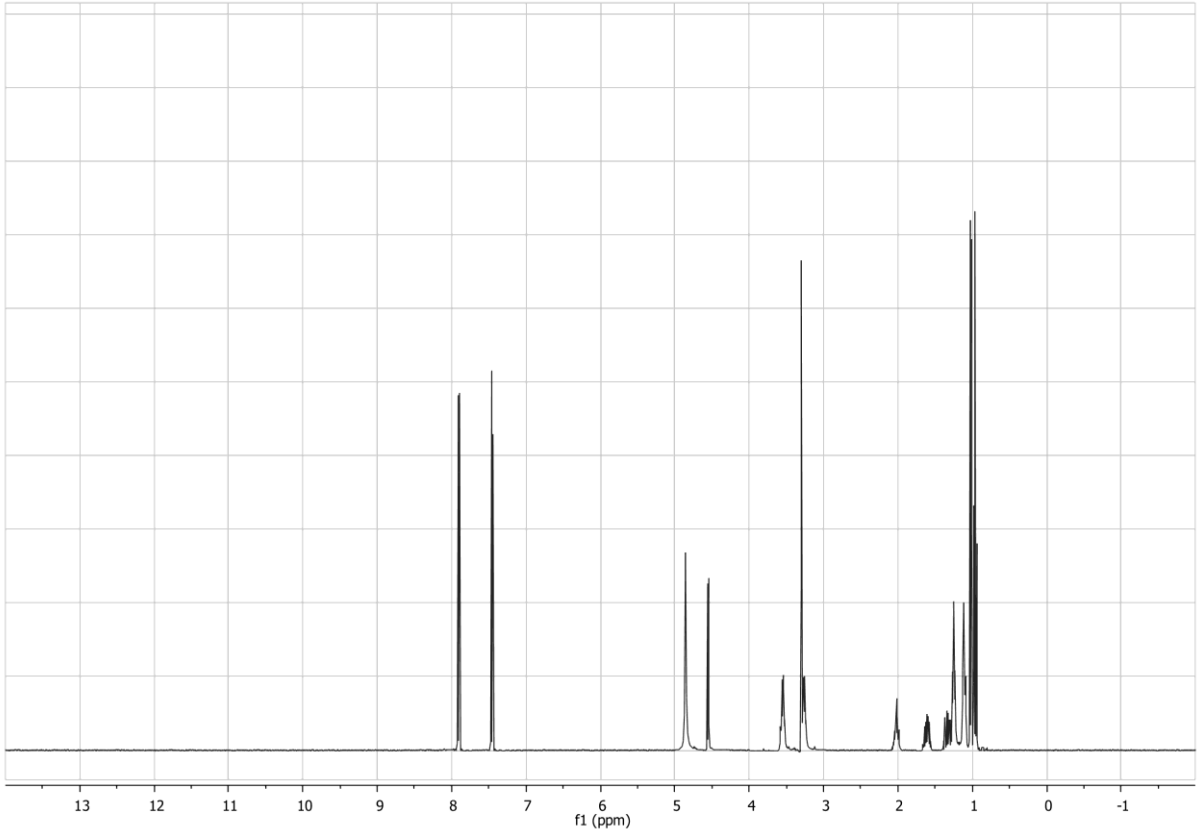
Sample ID: MAB 289 purity check  
Date & Time: 1/24/2011 2:38:53 PM  
Data File: B:\Malte\Results\benzamides\Acids purity\MAB 289 purity check\_6506.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram



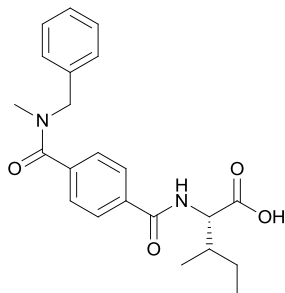
### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 1.62 min	100.0





**(2S,3R)-2-(4-(Benzyl(methyl)carbamoyl)benzamido)-3-methylpentanoic acid (69).**



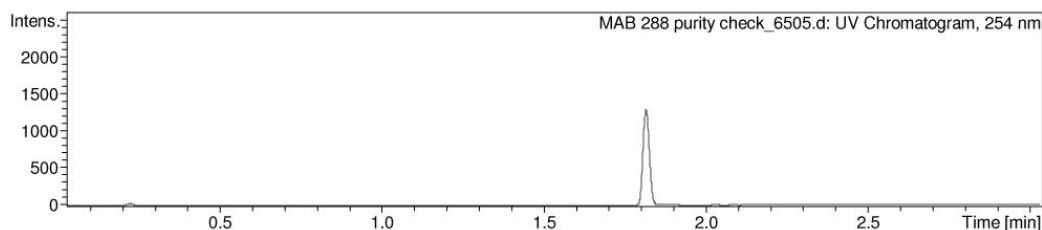
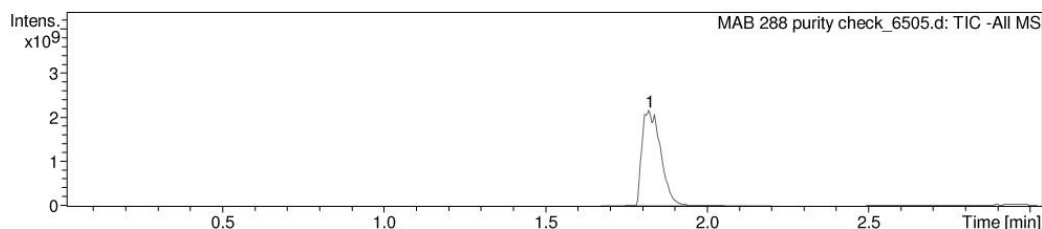
Yield: 42 mg (88%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.90 – 0.98 (m, 3H), 0.98 – 1.05 (m, 3H), 1.25 – 1.38 (m, 1H), 1.60 (dt,  $J = 12.4$  Hz, 7.5 Hz, 1H), 1.96 – 2.06 (m, 1H), 2.87 (s, 2H), 3.02 (s, 1H), 4.49 – 4.53 (m, 1H), 4.55 (d,  $J = 6.5$  Hz, 1H), 4.75 (s br, 1H), 7.16 (d,  $J = 7.4$  Hz, 1H), 7.24 – 7.39 (m, 4H), 7.53 (d,  $J = 8.1$  Hz, 2H), 7.86 (d,  $J = 7.9$  Hz, 1H), 7.92 (d,  $J = 8.1$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.8, 16.3, 26.7, 33.8, 37.7, 38.3, 51.9, 56.2, 59.1, 128.1, 128.2, 128.9, 129.0, 129.1, 129.3, 130.0, 130.1, 137.0, 137.7, 138.2, 140.5, 169.8, 173.0, 175.0; HRMS (M+H) calcd. for  $\text{C}_{22}\text{H}_{26}\text{N}_2\text{O}_4$  383.1965, found: 383.1968;  $[\alpha]_{589}^{25} = +13.2$ .

## LC-MS Analysis Report

### General Information

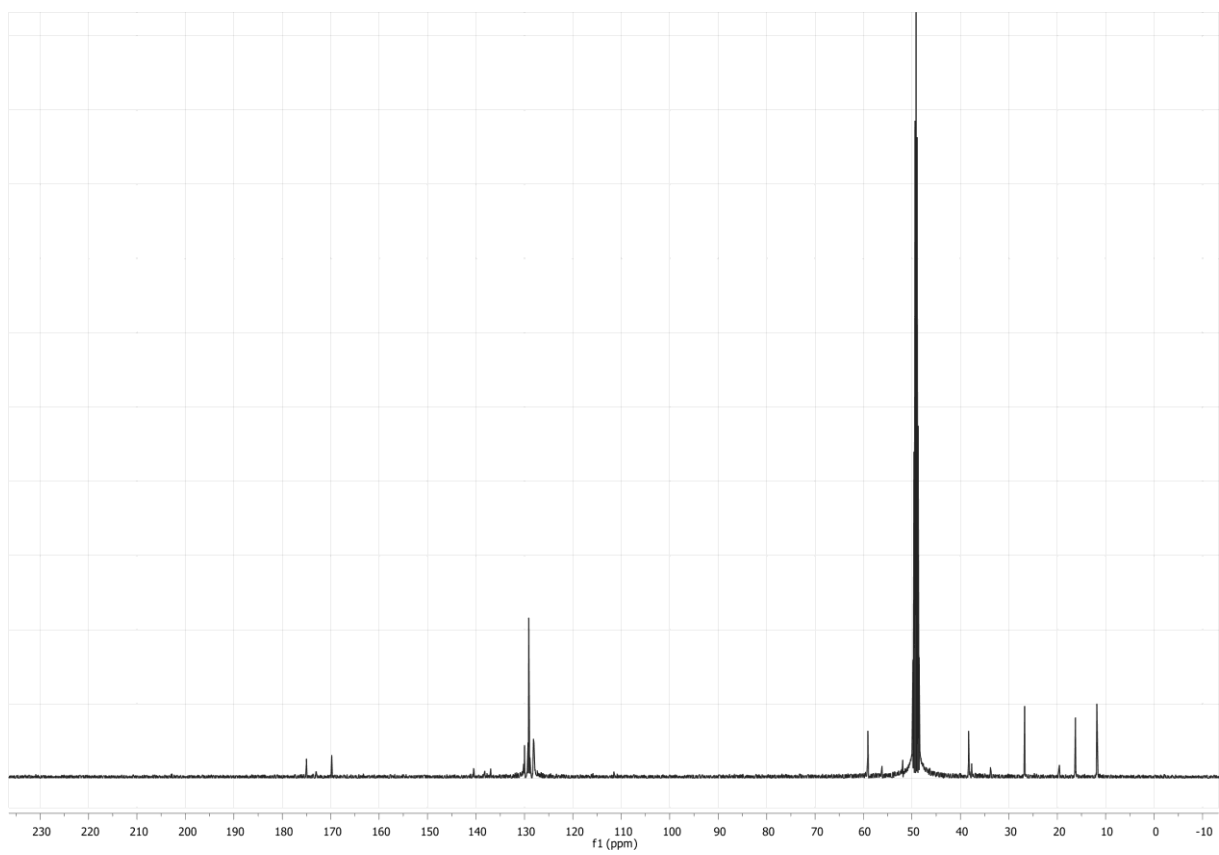
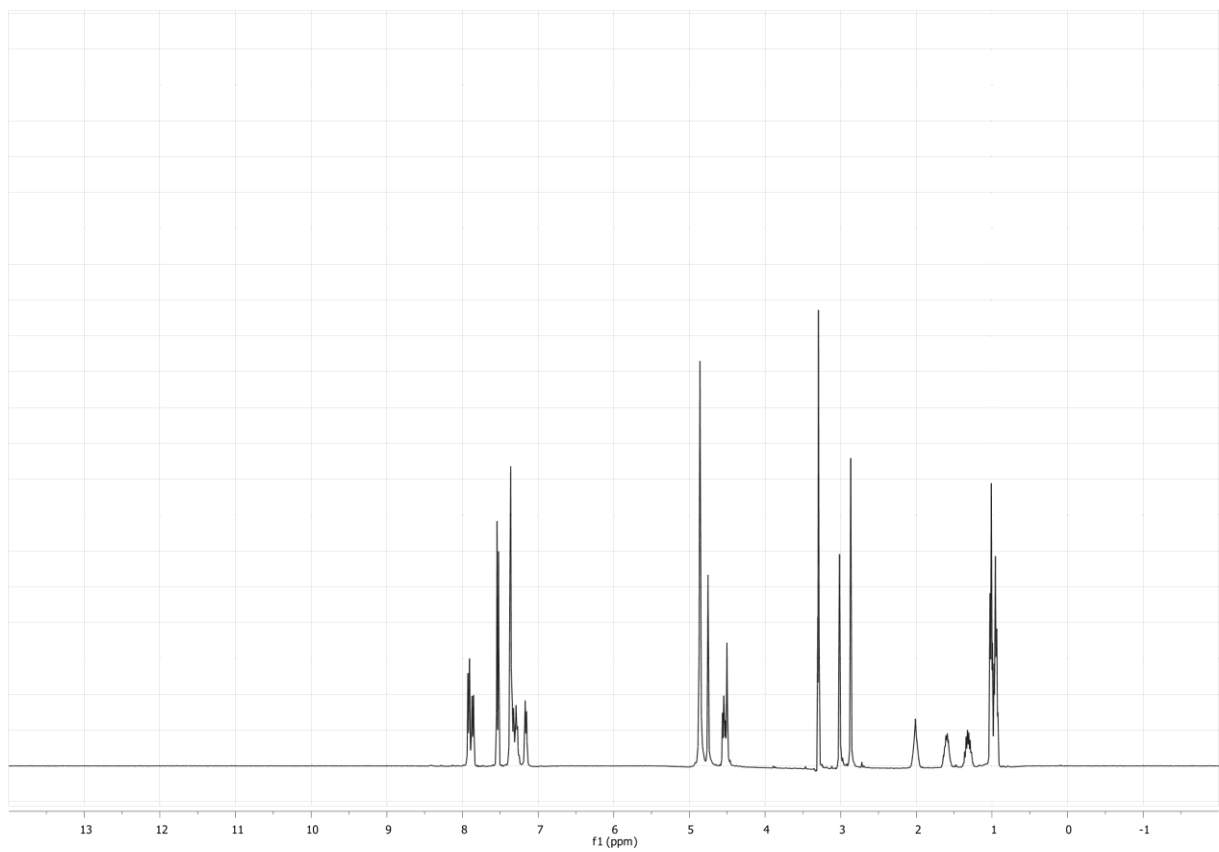
Sample ID: MAB 288 purity check  
Date & Time: 1/24/2011 2:34:29 PM  
Data File: B:\Malte\Results\benzamides\Acids purity\MAB 288 purity check\_6505.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram

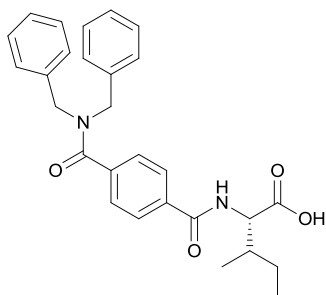


### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 1.82 min	100.0



**(2S,3R)-2-(4-(Dibenzylcarbamoyl)benzamido)-3-methylpentanoic acid (70).**



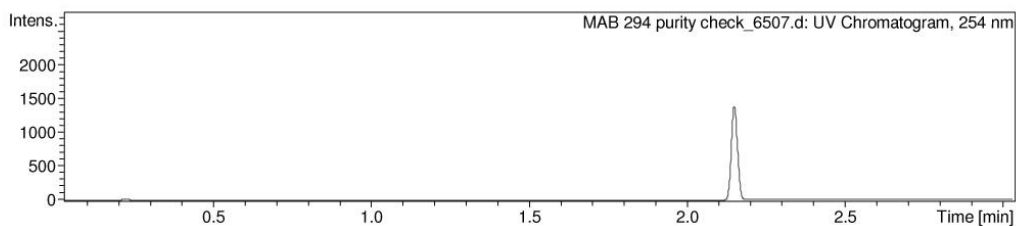
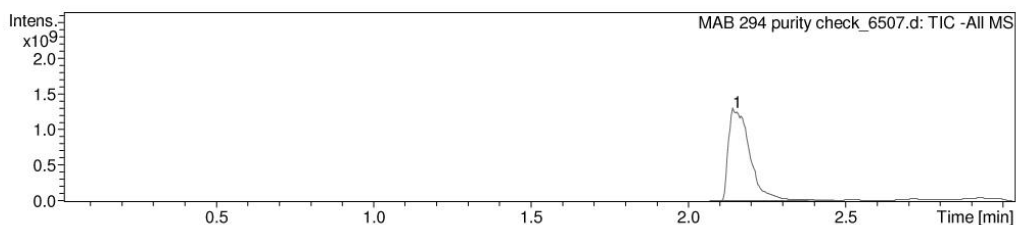
Yield: 20 mg (57%, white semi-solid);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 0.95 (t,  $J = 7.4$  Hz, 3H), 1.01 (d,  $J = 6.9$  Hz, 3H), 1.25 – 1.38 (m, 1H), 1.60 (ddq,  $J = 14.9$  Hz, 7.5 Hz, 4.3 Hz, 1H), 1.95 – 2.07 (m, 1H), 4.43 (s br, 2H), 4.54 (d,  $J = 6.3$  Hz, 1H), 4.71 (s br, 2H), 7.14 (d,  $J = 6.9$  Hz, 2H), 7.26 – 7.40 (m, 8H), 7.54 – 7.59 (m, 2H), 7.86 – 7.91 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ )  $\delta$ : 11.9, 16.3, 26.7, 38.4, 48.7, 53.2, 59.2, 127.9, 128.3, 128.9, 129.2, 129.4, 130.0, 130.1, 137.1, 137.5, 138.1, 140.4, 169.7, 173.8, 175.2; HRMS (M+H) calcd. for  $\text{C}_{28}\text{H}_{30}\text{N}_2\text{O}_4$  459.2278, found: 459.2289;  $[\alpha]_{589}^{25} = +11.9$ .

## LC-MS Analysis Report

### General Information

Sample ID: MAB 294 purity check  
Date & Time: 1/24/2011 2:43:15 PM  
Data File: B:\Malte\Results\benzamides\Acids purity\MAB 294 purity check\_6507.d  
Data Processing: TIC and UV chromatograms displayed (all wavelengths), 254 nm integrated

### Chromatogram



### Compound List (Area Frac. % of UV 254 nm)

Cmpd. Label	Area Frac. %
Cmpd 1, 2.15 min	100.0

